# Development of phosphor thermometry systems for use in development gas turbine engines

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## Contents

Contents	2
List of Tables	6
List of Figures	8
Nomenclature	18
Abstract	22
Declaration	23
Copyright Statement	23
Acknowledgements	24
About the Author	25
	26
1.1 Overview of the Engineering Doctorate Programme	26
1.2 Background	27
1.3 Aims and Objectives	27
1.4 Thesis Structure	28
2 MOTIVATIONS FOR PHOSPHOR THERMOMETRY IN AERO-ENGINES	30
<ul> <li>2.1 Temperature measurement in aero-engines</li> <li>2.1.1 Characterisation of Engine Sensors Types</li> <li>2.1.2 Research into improved engine efficiency</li> <li>2.1.3 The role of accurate temperature measurement</li> <li>2.1.4 Verification / Design tool</li> <li>2.1.5 Health Monitoring / Engine Control</li> <li>2.1.6 Summary</li> </ul>	<b>30</b> 30 32 35 38 38 39
2.2 Review of the existing methods for surface temperature measure	rement
Inside aeroengines2.2.1Introduction2.2.2Thermocouples and RTDs (contact sensors)2.2.3Radiation Pyrometry2.2.4Thermal Paints and Thermal Melts2.2.5Summary	<b>39</b> 39 41 43 44
2.3 Capability shortfalls, gaps & requirements analysis	47
3 LUMINESCENCE THERMOMETRY	57
3.1 Introduction	57
3.2 Historical Context	57
<b>3.3 Principles of Luminescence</b> 3.3.1 Generic Luminescence Behaviour 3.3.2 Luminescence in Phosphors	<b>59</b> 59 62

3.4	Different response modes	66
3.4.1	Introduction	66
342	Intensity Mode	67
343	Intensity Ratio	68
344	Temporal Analysis	71
345	Line shift/width method	71
346	Absorption/Excitation hand analysis	70
5.4.0	Absorption/ Excitation band analysis	70
3.5	Other factors	79
3.5.1	Introduction	79
3.5.2	Activator concentration	79
3.5.3	Particle Size	80
3.5.4	Oxygen and Pressure	81
		-
3.6	Generic phosphor thermometry system	82
3.6.1		82
3.6.2	Emission Detection	83
3.6.3	Excitation Sources	87
3.7	Consideration factors inside an engine environment	91
4 ET	RE ODTIC LASED DELIVERY CONSTDERATIONS	96
		50
4.1	Introduction	96
4.2	Consideration Factors and Damage Mechanisms	96
4.2.1	Gas Breakdown	97
4.2.2	Fibre Selection for UV transmission	98
4.2.3	Thermal Tolerance	99
4.2.4	End face preparation	100
4.2.5	Loss/damage in bends	101
4.2.6	Solarisation Effects / Luminescence in the fibre	101
43	Experimental Setur	102
/31	Fibre Dreparation	102
4.3.1	Boom and Launching conditions	103
4.3.2	Energy density, fluence, peak powers and power densities	104
4.3.3	Energy density, nuence, peak powers and power densities	100
4.4	Estimated Delivery Performance	111
4.5	Results and Discussions	114
4.5.1	Fused silica fibre	114
4.5.2	Investigating the variation of launching angle	118
4.5.3	Investigating wavelength dependency: 355nm vs. 266nm	121
4.5.4	Investigating the use of DOE homogenisers	123
4.5.5	Sapphire Fibres vs. Fused Silica Fibres	128
16	Further Decearch	125
4.6.1	Hollow Wayequides and PCEs	135
4.6.2	Tanered Fibres	135
4.0.2	Hollow Tanarad Pyray-Glass	135
4.0.5	Tonow Tapered Tyrex Glass	150
4.7	Conclusions	137
5 BC	NDING METHODS AND SPECTRAL ANALYSIS	138
5.1	Phosphor Bonding Methods	138
5.1.1	Introduction	138
5.1.2	Chemical Bonding	138
5.1.3	Vapour Deposition / Rf Sputtering	140
5.1.4	Flame and Plasma Spray	141
5.1.5	Discussion	142

<b>5.2 E</b> 5.2.1 5.2.2	mission Spectral Analysis. Introduction Experimental Methodology	<b>146</b> 146 146
5.2.4	Results and Discussions	140
6 INT	ENSITY MEASUREMENTS FOR PHOSPHORS	156
6.1 A 6.1.1 6.1.2 6.1.3 6.1.4 6.1.5 6.1.6 6.1.7	bsolute intensity measurements Introduction Methodology Effect of distance, detector area and detection angle Conversion to radiant flux and intensity Results, discussions and measurement uncertainty Further work Conclusions	<b>156</b> 156 157 161 163 168 176 177
6.2 Q 6.2.1 6.2.2 6.2.3 6.2.4	<b>uantum efficiencies and signal-to-blackbody-ratios</b> Introduction Methodology Results and Discussions Conclusions	<b>178</b> 178 178 181 186
7 DE1	ERMINATION OF PHOSPHOR LIFETIMES	188
7.1 I	ntroduction	188
7.2 M	lethodology	188
7.3 L	ata Processing	191
7.4.1 7.4.2 7.4.3 7.4.4 7.4.5	YAG:Tm Magnesium Manganese phosphors YAG:Tb Y <sub>2</sub> O <sub>3</sub> :Eu YAG:Dy	195 200 205 215 218
7.5 S	ummary and Conclusions	220
8 DES	SIGN FOR ROTATING ENGINE COMPONENTS	223
8.1 I	ntroduction	223
8.2 C	omponent rotational speeds and linear velocities	223
8.3 P 8.3.1 8.3.2 8.3.3	<b>hosphor selection criteria</b> Single Phosphor Solution Sample per revolution Multiple Phosphor Solution	<b>225</b> 225 229 230
8.4 P 8.4.1 8.4.2 8.4.3 8.4.3 8.4.4 8.4.5	robe System Design Discussion Operating laser wavelength Fibre optic Single fibre probes on stationary targets Dual fibre probes Probe performance	<b>231</b> 231 232 234 235
<b>8.5 S</b> 8.5.1	ingle fibre probe on rotating surfaces Collection efficiency	<b>237</b> 237

<b>8.6</b> 8. 8. 8.	<ul> <li>Experimental Testing on Rotating Components</li> <li>.6.1 Experimental procedure</li> <li>.6.2 Hot rotating disc</li> <li>.6.3 Rotating at a constant temperature</li> </ul>	<b>243</b> 243 245 248
8.7	Measurement Uncertainty	254
8.8	Conclusions	261
9	CONCLUSIONS, RECOMMENDATION AND FUTURE WORK	263
9.1	Summary	263
9.2	Recommendation	266
9.3	Future Work	266

Word Count: 74,748

## List of Tables

Table 1: Characteristics of different sensor types on the aeroengine.         Summarised from [2]         30
Table 2: Most common thermocouple types used in aeroengines40
Table 3: Pros/Cons and performance of existing temperature         measurement methods in gas turbines.         46
Table 4: Temperature range of various engine components
Table 5: Summary of typical process times from excitation to emission
Table 6: Elements in the a) lanthanide series; b) transition metals series63
Table 7: Comparison of the `conventional two camera' approach and the`filter wheel approach' detection for the two-mode intensity method
Table 8: Effect of increasing temperature on the probability of radiative $(P_r)$ and non-radiative $(P_{nr})$ decay72
Table 9: Comparison of different light detectors
Table 10: Factors for consideration for coupling laser radiation into fibres         97
Table 11: Correction factors required for different wavelengths         112
Table 12: Maximum estimated transmittable energy at variouswavelengths for different diameter fibres, based on 12ns pulses.112
Table 13: Comparison of maximum transmissions, fluence and powerdensities for f=50mm and 25mm coupling lens.121
Table 14 : Transmission % of energy with and without the DOE
Table 15: Effective energy falling on the fibre face         127
Table 16:         Recommended minimum bend radius [147]         131
Table 17: Summarised pros and cons of using sapphire fibres instead offused silica/quartz fibres.132
Table 18: Comparison of various high temperature chemical binders [166]         139
Table 19: Summary of the key issues related to different bonding           techniques         142
Table 20: Summary of key characteristics of commercially available           binders from ZYP Coatings
Table 21: Reference for common radiometric and photometric           terminology         158
Table 22: Determined PMT Ps.Ec values165
Table 23: Uncertainty budget for phosphor intensity

Table 24: Comparison of the estimated limit of measurement based on theoretical/ideal conditions and experimental conditions.	184
Table 25: Temperature when the signal to blackbody radiation = 1.Simulation using an input laser energy of x1, x10, x100 mJ/pulse.	186
Table 26: Uncertainty of furnace temperature	189
Table 27: Excitation and emission wavelength investigations for different           phosphors	190
Table 28: Charge balance of Mg:Mn phosphors cited in the literature	201
Table 29: Summary of the quenching temperature, maximum         temperature that was determinable and the combined standard         uncertainty.	222
Table 30: Summary of the useful temperature range determinable usingthe lifetime analysis for various phosphors	222
Table 31: Summary of combined measurement uncertainties on           stationary components	222
Table 32: Summary of linear velocities, distance to target for various           engine components at 13,000 RPM.	224
Table 33: Summary of the maximum acquisition time available for           measurement	227
Table 34: Key feature of the different probe designs	233
Table 35: Additional losses in the fibre optic detection system	236
Table 36: Required radial position to maintain a linear velocity of $0.048 mm/\mu s$ at various rotation speeds	244
Table 37. Collection time to lifetime ratios for YAG:Tm at various speeds	249
Table 38: Determined lifetime before and after corrections at various speeds.	249
Table 39: Lower temperature measurement capability limit for YAG:Tm for a range of lens sizes and collection to lifetime ratios. Values for half- FOV probes. Bracketed values are for full-FOV probes	257
Table 40: Lower temperature measurement capability limit for $Mg_3F_2GeO_4$ :Mn for a range of lens sizes and collection to lifetime ratios. Values for half-FOV probes. Bracketed values are for full-FOV probes	257
Table 41: Uncertainty of measurement for YAG:Tm phosphor	260

# List of Figures

Figure 1: EngD model, showing the relationship and benefits to the research Engineer, University and the sponsoring company
Figure 2: Analysis of the relative importance of factors for the three different engine sensor types
Figure 3: Reasons for engine research and development activities
Figure 4: Examples of factors that drive aeroengine research and development activities
Figure 5: Operational temperature trends [12]
Figure 6: Increase in turbine inlet temperatures [13]37
Figure 7: Influence of temperature on $CO_2$ and NOx emissions [13]37
Figure 8: Results from thermal paints applied to an engine component [32]44
Figure 9: Issues with existing methods45
Figure 10: Component locations in an aeroengine [13]48
Figure 11: Summarised capability gap matrix based on findings from the EVI-GTI [10]50
Figure 12: Various turbine section engine component [32]51
Figure 13: Complex internal cooling channels and holes for HP turbine blade and guide vanes. [13]52
Figure 14: Variation of pressure and temperature across the turbine section of the aeroengine [13]53
Figure 15: Relative importance of factors for high pressure, intermediate pressure, and low pressure turbine blade sections in gas turbines
Figure 16: High pressure drive cone [32]55
Figure 17: Jablonski energy level diagram showing the luminescence process
Figure 18: Chemical composition of thermographic phosphors63
Figure 19. Configuration co-ordinate diagram64
Figure 20: Configuration co-ordinate diagram showing the effect from the charge transfer state (CTS) curve
Figure 21: Different response modes for thermographic phosphors
Figure 22: Variation of emission intensity with increasing temperature. Taken from [42]67
Figure 23: Ideal intensity variations for the intensity ratio response

Figure 24: Energy level diagram for free ions of Dy and Sm. Taken from [73] cited in [70]69	)
Figure 25: Emission spectra at different temperatures. Left: YAG:Dy [71]; Right: $Y_2O_2S:Sm$ [70]69	)
Figure 26. Typical lifetime characteristics with increasing temperature73	;
Figure 27: Lifetime of phosphors vs. temperatures. Taken from [42]74	ŀ
Figure 28: Left: Architecture of the framing camera, showing the eight faced prism splitting the light equally to eight ICCD cameras [55]. Right: Curve fit for a single pixel from a series of images obtained from the separate detectors [79].	.)
Figure 29: Phase shifts for different lifetimes75	;
Figure 30: Energy levels of $Y_2O_3$ :Eu at symmetry sites $C_2$ ad $C_{3i}$ 76	,
Figure 31: Risetime variation with temperature. Taken from Allison et al. [95]77	,
Figure 32: Emmsion lineshift and linewidth variation with temperature [42]	;
Figure 33: The absorbtion spectra of $Y_2O_3$ :Eu [64]79	)
Figure 34: Left: Variation of absorption peak with temperature [42]79	)
Figure 35: Effects of reducing the particle size [97]80	)
Figure 36: Effects of different gases on the lifetime decay of different phosphors at different temperatures. Phosphors a) $La_2O_2S:Eu$ b) $Mg_4FGeO_6:Mn$ c) $Y_2O_3:Eu$ [100]81	•
Figure 37: Variation in lifetime decay time of La <sub>2</sub> O <sub>2</sub> S:Eu phosphor with increasing pressure [101]82	<u>,</u>
Figure 38: Generic layout for a thermographic phosphor system83	;
Figure 39: Comparison of CCD and CMOS cameras	,
Figure 40: Typical rectangular excimer laser profile. Taken from [112]	)
Figure 41: Sinusoidal wave used to excite phosphor. Taken from Allison <i>et</i> <i>al</i> . [89]90	)
Figure 42: Amount of background radiation (shaded area) collected using wide and narrow band filters	;
Figure 43: Different factors affecting phosphor selection and overall design of the phosphor thermometry system95	;
Figure 44: Fibre damage mechanisms. Taken from [120]97	,
Figure 45: Examples of commercially available fibre jackets	)

Figure 46: Fibre Transmission of 2.5 million high intensity 308nm laser pulses of 7.5mJ energy with a pulse width of 8ns. Taken from Delmdahl et al. [141]	2
Figure 47. Cohemotic for fibre optic coupling investigations	-
Figure 47: Schematic for fibre optic coupling investigations	<u> </u>
Figure 48: Converging and diverging beam 104	1
Figure 49: Launching angle	5
Figure 50: Variation of angle as functions of laser beam diameter and lens focal length	5
Figure 51: Beam area at various positions (x) from the focal point	5
Figure 52: Variation in beam diameter and beam area at various positions from the focal point. Simulation using an initial beam diameter of 10mm, and lens with a focal length of 50mm107	7
Figure 53: Variation in the peak power density, average power density and fluence at various positions from the focal point. Simulation using a 10mJ/pulse, and lens with a focal length of 50mm, and initial beam diameter of 10mm	7
Figure 54: Variation in the peak power density at various positions from the focal point, using various laser energies/pulse ranging from 5mJ to 50mJ	3
Figure 55: Variation in the peak power density at various positions from the focal point, using various laser energies/pulse	3
Figure 56: The total average power, total peak power and total energy experienced on the fibre's face of fibres with different diameters, at various distances relative to the focal point. Simulation using a laser pulse energy of 10mJ, with the focal length of lens = 50mm	C
Figure 57: The variation of laser beam area:fibre area ratio of various diameter fibres, at various positions relative focal point	1
Figure 58: The variation of estimated transmission of various diameter fibres, at various positions relative focal point	1
Figure 59: The variation of maximum energy at different pulse widths, based on 1mm diameter fibre at various wavelengths	3
Figure 60: The variation of maximum energy at various fibre diameters,	3
Figure 61: The variation of maximum energy at different wavelengths,	1
Figure 62: Transmitted pulse energy through a 1.00mm core diameter fibre at various locations relative to the focal point, for various input pulse energy levels. Wavelength = 355nm, Focal length of coupling lens = 50mm	5
Figure 63: Damage points at various positions relative to the focal point. Wavelength = $355$ nm, f = $50$ mm	5
Figure 64: Estimated fluence and peak power densities at the damage points	7

Figure 65: Transmission efficiency (%) based on the total input pulse energy. Wavelength = 355nm, f=50mm
Figure 66: Estimated pulse energy on fibre face at various positions relative to the focal point, for different input energy levels. Wavelength = 355nm, f = 50mm
Figure 67: Corrected transmission %, based on the estimated pulse energy falling onto the fibre's face. Wavelength = 355nm, f = 50mm
Figure 68: Transmitted pulse energy at various locations relative to the focal point, for various input pulse energy levels. Wavelength = 355nm, f= 25mm
Figure 69: Transmission efficiency (%) based on the total input pulse energy. Wavelength = 355nm, f=25mm
Figure 70: Estimated pulse energy on fibre's face at various positions relative to the focal point, for different input energy levels. Wavelength = 355nm, f = 25mm
Figure 71: Corrected transmission %, based on the estimated pulse energy falling onto the fibre's face. Wavelength = 355nm, f = 25mm
Figure 72: Estimated fluence and peak power densities at the damage points. f=25mm
Figure 73: Transmitted pulse energy at various locations relative to the focal point, for various input energy levels. Wavelength = 266nm, f= 50mm
Figure 74: Transmission % at various locations relative to the focal point, for various input energy levels. Wavelength = 266nm, f= 50mm
<ul> <li>Figure 75: Fluence profiles at the fibre entrance face: a.) Gaussian</li> <li>Nd:YAG laser focused with a simple plano-convex lens, b.) Using a DOE,</li> <li>c.) Nd:YAG focused with a plano-convex lens-Result of cavity changes</li> <li>and realignment. (Taken from Setchell [146])</li></ul>
Figure 76: Beam profiles with and without DOE
Figure 77: Injection optics using a DOE. Taken from Hand et al (1999)124
Figure 78: Lost energy when the DOE is placed too far away from the coupling lens
Figure 79: Zeroth order focus and diffractive focus at the same position
Figure 80: Location of the zeroth order focus and diffractive focus 125
Figure 81: Results with and without the DOE with input laser energies of:
Figure 82: Energy falling onto the fibre's face
Figure 83: Transmission of input energy, illustrating attenuation along the length of the fibre, for fused silica/quartz and sapphire fibres with different core diameters
Figure 84: Regions were a 1.0mm core sapphire fibre would be more suitable than fused silica/quartz fibres

Figure 85: Transmitted energy through FV Polymicro 425 $\mu m$ fused silica	
fibres	133
Figure 86: Transmitted energy through Photran 425 $\mu m$ sapphire fibres	133
Figure 87: Photonic Crystal Fiber [156]	135
Figure 88: Tapered fibre	136
Figure 89: The optical arrangement of a hollow taper for direct launching and delivery of UV light. Taken from Ilev et al.,[129]	137
Figure 90. Left: emission spectra of $Y_2O_3$ phosphor in Resbond binder after thermal exposure to 1,400°C [166]. Right: Intensity of the peak emission of $Y_2O_3$ phosphor after thermal exposure to 1,200 °C [167]	140
Figure 91: Intensity of thin coatings (0.1 $\mu$ m-3 $\mu$ m) after annealing at 1,200°C compared to thick film (10 $\mu$ m) and powdered Y <sub>2</sub> O <sub>3</sub> :Eu phosphor [167].	141
Figure 92: The variation in intensity with time (hours) with constant thermal exposure of 1,200°C for thin coatings (0.1 $\mu$ m-3 $\mu$ m) produced by RF sputtering and thick film coatings (10 $\mu$ m) produced by chemical binders [167].	141
Figure 93: Temperature variation across the section of a TBC deposited on a substrate. Taken from [169]	144
Figure 94: Schematic for spectral emission analysis.	147
Figure 95: Y <sub>2</sub> O <sub>3</sub> :Eu– Excitation and emission spectra – Obtained by Phosphor Technology Ltd	148
Figure 96: $Mg_3F_2GeO_4:Mn$ – Excitation and emission spectra – Obtained from Phosphor Technology Ltd.	148
Figure 97: YAG:Tm– Excitation and emission spectra. Obtained from Phosphor Technology Ltd	149
Figure 98: YAG:Tb - Normalised 2D plot of spectral response at various temperatures	149
Figure 99: YAG:Tb - Normalised 3D plot of spectral response at various temperatures	150
Figure 100: YAG:Tb – Normalised contour intensity plot throughout the entire temperature range	150
Figure 101: YAG:Tb – Contour intensity plot normalised to max intensity at a single temperature	151
Figure 102: YAG:Tb - Magnification contour plots of emission line	151
Figure 103: $Y_2O_3$ :Eu - Normalised 2D plot of spectral response at various temperatures	152
Figure 104: Y <sub>2</sub> O <sub>3</sub> :Eu - Normalised 3D plot of spectral response at various temperatures	152

Figure 105: $Y_2O_3$ :Eu - Contour intensity plot normalised to max intensity at a single temperature. 153
Figure 106: YAG:Tm - Normalised 2D plot of spectral response at various temperatures
Figure 107: YAG:Tm - Normalised 3D plot of spectral response at various temperatures
Figure 108: YAG:Tm – Normalised contour intensity plot throughout the entire temperature range
Figure 109: YAG:Tm – Contour intensity plot normalised to max intensity at a single temperature
Figure 110: Comparison of energy (area under curve) from phosphor emissions with: a.) intense, but relatively short lived lifetimes (faster decays), b.) less intense, but relatively long-lived lifetimes (slower decays)
Figure 111: Solid angle
Figure 112: Schematic for high temperature intensity investigation
Figure 113: Schematic for investigating the effect of distance and detector area
Figure 114: Radiant Flux- Effect of: a.)distance, b.)detector diameter, c.)detector area
Figure 115: Intensity- Effect of: a.)distance b.)detector diameter, c.)detector area
Figure 116: Angular distribution of emission at various laser angles. [64]163
Figure 117: PMT's equivalent circuit166
Figure 118: (a) output pulse shapes with an anode load $R = 50 \Omega$ , for a range of parallel capacitors. (b) pulse shapes for $C = 10 \ pF$ and a range of resistor values. Taken from [182]
Figure 119: Anode equivalent circuit with transimpedance amplifier 167
Figure 120: The effect of luminescent decay and blackbody radiation on the PMTs mean anode current
Figure 121: Intensity (mW/sr) with increasing temperature at various laser energies for a number of phosphors169
Figure 122: Effect of increasing laser energy at room temperature
Figure 123: Intensity per mJ (mW/sr.mJ) with increasing temperature at various laser energies for a number of phosphors
Figure 124: Emission intensity relatively to the intensity taken at 20°C at various temperatures using different laser energy levels
Figure 125: Variation of intensity per mJ of laser energy with increasing temperature

Figure 126: Variation of intensity per mJ of laser energy (semi-logscale) with increasing temperature
Figure 127: Variation of $I_0$ for slow and fast decays
Figure 128: Variation of the measured and potential intensity per mJ of laser energy with increasing temperature
Figure 129: Variation of the measured and potential intensity per mJ of laser energy (log scale) with increasing temperature
Figure 130: Area under phosphor decay curve = Energy
Figure 131: Blackbody Radiance with increasing temperature at various wavelengths
Figure 132: Lifetime values of various phosphors with increasing temperature
Figure 133: Peak phosphor power with peak laser power ratio
Figure 134: Quantum efficiencies of various phosphors with increasing temperature
Figure 135: Phosphor emission to blackbody radiation ratio, using theoretical blackbody values where source size equal to luminescence, and no contributions from reflections from furnace walls and other sources
Figure 136: Phosphor Emission to blackbody radiation based on experimental conditions
Figure 137: Schematic for high temperature lifetime ( $ au$ ) evaluations
Figure 138: Calibration procedure for lifetime determination with temperature
Figure 139: Simplified energy diagram for YAG:Tm from Cates et al. [195] (left). Additional emission lines (right) based on information taken from Zhang and Grattan [196]
Figure 140: YAG:Tm phosphor decays at various temperatures. Normalised intensity with time
Figure 141: YAG:Tm - Lifetime decay with increasing temperature, under different conditions
Figure 142 and Figure 143: Map of lifetime values indicating the effect of different start/end collection times (proportions of $\tau$ ). Emission from YAG:Tm at 700 °C (left) and 1200 °C (right). The sensitivity of the effect of the start/end times is reduced at higher temperatures
Figure 144: Percentage variation in lifetime values at various start and end collection times for YAG:Tm at 1200 °C
Figure 145: Example of YAG:Tm decay and curve fit at 1350 °C 198
Figure 146: YAG:Tm – Regression and residual analysis after the
quencining temperature

Figure 147: YAG:Tm –Residual error analysis in terms of temperature	Э
Figure 148: YAG:Tm – Regression and residual error analysis	C
Figure 149: Mg <sub>3</sub> GeFO <sub>4</sub> :Mn phosphor decays at various temperatures using 266nm illumination and 630nm emission. Normalised intensity with time. Temperature range: 20-500 °C (left) and 600-900 °C (right)	1
Figure 150: Mg <sub>3</sub> GeFO <sub>4</sub> :Mn - Lifetime decay with increasing temperature, under different conditions202	2
Figure 151: Decay and curve-fit for Mg <sub>3</sub> GeFO <sub>4</sub> :Mn phosphor at 20 °C(left) 600 °C (right), emission line - 630nm, excitation wavelength 266nm 203	3
Figure 152: Map of lifetime values indicating the effect of different start/end collection times (proportions of lifetime). Result from Mg <sub>3</sub> GeFO <sub>4</sub> :Mn phosphor, 266nm excitation, 630nm emission at: a.) 20 °C, at b.) 500 °C	3
Figure 153: Magnesium Manganese - Regression and residual analysis after the quenching temperature. (Linear polyfit)	4
Figure 154: Magnesium Manganese - Regression and residual analysis after the quenching temperature. (2 <sup>nd</sup> order polyfit)205	5
Figure 155: Magnesium Manganese - residual analysis in terms of temperature after the quenching temperature. (2 <sup>nd</sup> order polyfit)	5
Figure 156: YAG:Tb phosphor - Lifetime values with increasing temperature: Under 266nm excitation (left) and 355nm excitation (355nm)	6
Figure 157: Decay and single exponential decay curve-fit for YAG:Tb at 400 °C, 490nm emission line, using 266nm excitation wavelength. R <sup>2</sup> = 0.999	7
Figure 158: Decay and curve fit for YAG:Tb - 490nm using 266nm excitation, using single exponential decay relationship at 800 °C. When start decay values are t = 0 (left) and t = $\tau$ (right)207	7
Figure 159: Decay and curvefit for YAG:Tb - 490nm using 266nm(top), 355nm (bottom) excitation, using single exponential decay relationship at 1,000 °C	8
Figure 160: Map of lifetime values indicating the effect of different start/end collection times (proportions of lifetime). Result from YAG:Tb phosphor at 544nm emission using 355nm excitation at 20 °C	Э
Figure 161: YAG:Tb phosphor - Lifetime values using single and double exponential relationship for 544nm emission under 266nm excitation	1
Figure 162: Decay and single exponential decay curve-fit for YAG:Tb at 20 °C under various conditions	2
Figure 163: Regression and single order polyfit, taken after 700 °C 213	3
Figure 164: YAG:Tb - Residual analysis in terms of temperature for a linear polyfit	3

Figure 165: YAG:Tb - Residual analysis in terms of temperature for a 2 <sup>nd</sup> order polyfit
Figure 166: Emission spectra of YAG:Tb. Taken from Choe et al. [197] 214
Figure 167: Simplified energy diagram for Y <sub>2</sub> O <sub>3</sub> :Eu
Figure 168: Y <sub>2</sub> O <sub>3</sub> :Eu – Decay curves at various temperature
Figure 169: $Y_2O_3$ : Eu – Lifetime values with increasing temperature
Figure 170: Y <sub>2</sub> O <sub>3</sub> :Eu - Example of curve fits. It shows that the single exponential relationship fit the data well
Figure 171: Y <sub>2</sub> O <sub>3</sub> :Eu - Regression analysis after the quenching temperature
Figure 172: $Y_2O_3$ :Eu - Regression and residual in terms of temperature 218
Figure 173: YAG:Dy- Lifetime values with increasing temperature
Figure 174: YAG:Dy- Regression analysis after the quenching temperature
Figure 175: YAG:Dy- Residual analysis in terms of temperature
Figure 176: Lifetime values of various phosphors with increasing temperature
Figure 177: Figure to compare the different linear velocities (root to tip) of different turbine blades at various engine speeds
Figure 178: Effect of probe design and maximum detection time for lifetime determination at various engine speeds
Figure 179: Illustration of temporal phosphor selection criterion
Figure 180: Phosphor Selection for High Pressure Turbine Blade application
Figure 181: Phosphor Selection for Intermediate and Low Pressure turbine blade application
Figure 182: Phosphor Selection for drive cone application
Figure 183: Optical collection system for a mixed phosphor solution
Figure 184: Schematic for fibre optic delivery and collection
Figure 185: Returned emissions from the fibre
Figure 186: Detected signal of a moving target is a product of phosphor decay and collection factor
Figure 187: The collection efficiency factor of the fibre optic probe
Figure 188: Phosphor decays with short and large lifetimes
Figure 189: Detected signal of a moving target using a twin fibre probe configuration

Figure 190: Twin fibre probe configuration with the use of a dichroic mirror.	. 243
Figure 191: Schematic for rotating phosphor thermometry experiments	. 244
Figure 192: Thermal imaging - hot rotating disc using FLIR -thermal imaging camera	. 247
Figure 193: The interference caused by the flame torch	. 247
Figure 194: Experimental data showing the decay of YAG:Tm phosphor at different rotational speeds	. 249
Figure 195: YAG:Tm simulation at different rotational speeds	. 250
Figure 196: Determination of the collection factor using the ratio of stationary to rotational waveforms.	. 251
Figure 197: Moving focus image point as the disc rotates	. 252
Figure 198: Focused target image on the fibre	. 253
Figure 199: System to reduce the proportion of blackbody radiation	. 253
Figure 200: Alternative system to reduce the proportion of blackbody radiation	. 254
Figure 201: Temperature range (upper and lower limits) based	. 256
Figure 202: Lower temperature measurement capability limit for YAG:Tm for a range of lens sizes and collection to lifetime ratios. Left: Half- FOV; Right: Full-FOV	. 257
Figure 203: Lower temperature measurement capability limit for $Mg_3F_2GeO_4$ :Mn for a range of lens sizes and collection to lifetime ratios. Left: Half-FOV; Right: Full-FOV	. 258
Figure 204: Variation of the determined lifetime values for $Mg_3F_2GeO_4:Mn$ phosphor using the maximum available detection time	. 259
Figure 205: Variation of the determined lifetime values for YAG:Tm phosphor using the maximum available detection time	. 259
	<ul> <li>Figure 190: Twin fibre probe configuration with the use of a dichroic mirror.</li> <li>Figure 191: Schematic for rotating phosphor thermometry experiments</li></ul>

## Nomenclature

## Latin Symbols

atin Symbo a <sub>d</sub>	Detector area (mm)		
В	Blackbody radiation		
B <sub>w</sub>	bandwidth		
с	Speed of light		
С	PMT circuit capacitance (farads)		
C <sub>f</sub>	Collection factor		
CL	Load capacitance (farads)		
Co	Output capacitance (farads)		
E <sub>c</sub>	Collection efficiency (%)		
E <sub>RPM</sub>	Engine rotation speed (RPM)		
d	Diameter of beam		
$d_{bx}$	laser beam diameter at position x (mm)		
d <sub>f</sub>	fibre diameter (mm)		
df/dx <sub>i</sub>	Uncertainty sensitivity		
EL	Laser Energy/pulse (J)		
E <sub>f</sub>	Total energy on the fibre's face (mJ)		
F <sub>T</sub>	Filter transmission at central $\lambda$		
f	Focal length (mm)		
f <sub>L</sub>	Laser repetition rate (Hz)		
G <sub>d</sub>	Dynode gain		
Ga	Amplifier gain		
h	Planck's constant (J.s)		
I	Intensity		
I <sub>0</sub>	Intensity at time t=0		
It	Intensity at time t		
$\mathbf{I}_{\text{anode}}$	Current at the PMT anode (A)		
Ip	Photoelectric current (A)		
$\mathbf{I}_{\text{peak}}$	Intensity (peak)		
Iq	Input laser energy on fused silica fibre		
Is	Input laser energy on sapphire fibre		
k	Boltzmann's constant		
k <sub>r</sub>	Radiative rate of emission		
k <sub>nr</sub>	Non-radiative rate of emission		
L	Length of fibre		
n	Refractive index		
n <sub>core</sub>	Refractive index of fibre core		

$n_{clad}$	Refractive index of fibre clad	
Ν	Electron population	
N <sub>0</sub>	Electron population at time t=0	
$N_{\text{excited}}$	Electron population at excited state	
N <sub>ground</sub>	Electron population at ground state	
NA	Numerical aperture	
Os	Output energy from sapphire fibre	
Oq	Output energy from fused silica fibre	
P <sub>1,2,3</sub>	Constants from regression analysis	
$P_L$	Laser average power (W)	
$P_{LP}$	Laser peak power (W)	
P <sub>r</sub>	Probability of radiative emission	
P <sub>nr</sub>	Probability of non radiative emission	
Ps	Photocathode sensitivity (A/W)	
r	Radial distance	
R	PMT equivalent circuit resistance ( $\Omega$ )	
$R_L$	Load resistance $(\Omega)$	
Ro	Output resistance $(\Omega)$	
R <sub>f</sub>	optical power (radiant flux) (W)	
R <sub>a</sub>	Amplifier Load ( $\Omega$ )	
S <sub>A</sub>	Surface area for blackbody radiation	
S <sub>x</sub>	Electronic singlet states	
t	Time (s)	
t	Pulse duration (s)	
Т	Absolute temperature (K)	
Т	Electronic intermediate states	
T <sub>c</sub>	Time constant	
tL	Laser pulse duration (s)	
Τ <sub>q</sub>	transmission efficiency per meter	
T <sub>r</sub>	Rise time (s)	
Ts	transmission efficiency per meter	
u <sub>c</sub>	Combined uncertainty	
V	Linear velocity	
V <sub>d</sub>	Supplied voltage to PMT (V)	
$V_{\text{out}}$	Observed voltage on oscilloscope (V)	
х	Distance from target to PMT (mm)	
х	Distance from focal length (mm)	

### **Greek Symbols**

α	Detection angle (°)
α	Launch angle
lpha c	critical angle
3	Emissivity
$\eta$	Photocathode quantum efficiency (%)
Ω	solid angle (sr)
τ	Decay lifetime (s)
$ au_{c}$	Time constant (s)
$ au_{_d}$	Decay lifetime (s)
$ au_r$	Emission rise time (s)
ω	Angular velocity (rad/s)
λ	Decay constant
λ	Wavelength (nm)

#### List of Abbreviations

APD	Avalanche photo diodes
CCD	Charged couple device
CFD	Computational Fluid Dynamics
CMOS	Complementary metal-oxide-semiconductor
СТ	Charge transfer
CVD	Chemical vapour deposition
DOE	Diffractive optical element
DPSS	Diode pumped solid state
EFG	Edge-defined, film fed growth
EPSRC	Engineering and Physical Sciences Research Council
EVI-GTI	European Virtual Institute for Gas Turbine Instrumentation
FEA	Finite Element Analysis
FFT	Full frame transfer
FOV	Field of View
FWHM	Full-Width Half-Maximum
GBW	Gain bandwidth
HP	High pressure
HP-TB	High pressure turbine blade
ICCD	Intensified CCD
IED	Industrial Encoders Direct
IP	Intermediate pressure
IR	Infrared

LASER	Light Amplification by Stimulated Emission of Radiation		
LCD	Liquid crystal display		
LDA	Laser Doppler Anemometry		
LED	Light emitting diode		
LGO	Light guide optics, Germany		
LHPG	Laser Heated Pedestrial Growth		
LP	Low pressure		
MACE	School of Mechanical, Aerospace and Civil Engineering		
MCP-PMT	Micro channel plate photomultipliers.		
NA	Numerical aperture		
NGV	Nozzle guide vanes		
PCF Photonic crystal fibre			
PIV	Particle Image Velocimetry		
PIWG	Propulsion Instrumentation Working Group		
PMT	Photo-multiplier Tubes		
PLD	Pulsed laser deposition.		
PSP	Pressure sensitive paint		
PVD	Physical vapour deposition		
RF	Radiofrequency		
RTD	D Resistance temperature detectors		
SMA	Sub-Miniature version A connector		
SPM	Silicon photomultiplier		
ТВ	Turbine blade		
ТВС	Thermal Barrier Coatings		
TDC	Top dead centre		
UV	Ultra violet		

## Abstract

The pursuit for improved engine efficiency is driving the demand for accurate temperature measurement inside turbine engines. Accurate measurement can allow engines to be operated closer to their design limits to improve thermal efficiency. It can enable engineers to verify mechanical integrity, provide better prediction of component life, validate CFD and other design tools and aid the development for leaner more efficient engines. Unfortunately, experimentally measuring surface temperatures under harsh rotating conditions is challenging. This EnaD study conducted by Ashig Hussain Khalid at the University of Manchester and Rolls-Rovce plc, reviews the rationale of using phosphor thermometry over existing methods, including thermocouples, pyrometry and thermal paints/melts, which lack detail, accuracy, or are too expensive for continuous testing. Although phosphor thermometry exhibits desirable characteristics, the high temperature and fast rotating engine environment presents some challenges that would need to be addressed before a successful measurement system can be implemented. Examples of such issues include: rising blackbody radiation, restricted optical access, fibre optic constraints and limited time period to collect data. These factors will impose measurement limits and greatly influence the design philosophy of the system, including phosphor choice, phosphor lifetime characteristics, bonding technique, excitation/detection methodologies and probe design. Taking these into consideration, the research focuses on the development of phosphor thermometry systems for use in development gas turbine engines, with measurement solutions for specific engine components. The high pressure turbine blade was given research priority.

A number of phosphors including YAG:Tb, YAG:Tm. Y<sub>2</sub>O<sub>3</sub>:Eu and Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn were investigated and characterised in terms of intensity and lifetime decay, with increasing temperature up to 1500°C. Spectral analysis and absolute intensity measurements established emission peaks and permitted comparative quantitative analysis to optimise system setup. The intensity of phosphor emission relative to Planck's blackbody radiation was also performed. YAG:Tm under 355nm illumination was found to exhibit the highest emission intensity at high temperatures, and because its spectral emission peak at 458nm was the lowest, its advantage in terms of blackbody radiation was further amplified. For rotating components, an upper temperature limit is reached based on the emission intensity at rising blackbody radiation levels and the system's ability to detect fast decays. A lower limit is reached based on the quenching temperature, probe design and rotational velocity. There are different methods to correct the distorted decay waveform as it traverses through the acceptance cone of the fibre. A phosphor selection criterion, taking into consideration these limitations, was successfully applied for various rotating engine components. The optical layout was setup and tested on stationary and rotating cases under laboratory conditions using similar design constraints, including fibre choice, maximum permissible lens size and target distances. A series of tests validated design methodologies and assumptions to enable testing on full scale rotating engine components.

 $Mg_3F_2GeO_4$ :Mn, using 355nm illumination, was found to be the most suitable phosphor for the HP drive cone. The estimated performance under the expected rotational speeds was found to be 624-812°C with a standard uncertainty of ±0.99%. YAG:Tm, illuminated with 355nm, was found to be the most promising phosphor for high pressure turbine blade measurements. The performance under the expected rotational speeds was found to be 1117-1375°C with a standard uncertainty of ±0.97%. This is better than other competing technologies that are currently available for temperature measurement of rotating turbine blades.

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"Without struggle, there is no progress" Frederick Douglas

"Do not follow where the path may lead. Go, instead, where there is no path and leave a trail" Ralph Waldo Emerson

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## **1** Introduction

### 1.1 Overview of the Engineering Doctorate Programme

The Engineering Doctorate is a four year postgraduate programme developed by the Engineering and Physical Sciences Research Council (EPSRC). It was devised in the 1990's as a radical alternative to traditional PhDs, being more vocationally focussed and better suited to the needs of industry. The programme, managed by EngD Centres, is a partnership between the research engineer, University, and the sponsoring company, as illustrated in Figure 1. The research engineer undergoes intensive technical and management training and is awarded a doctoral qualification. The university benefits by generating stronger industrial links whilst increasing its research profile through publications in Journals and Conferences. The industry benefits with the research topic being directly related to industrial interests, access to university's facilities, expertise and knowledge transfer; The EngD is a cost effective solution for research and development activities, and the unique collaboration has shown to be making a valuable contribution to UK industries.



Long term partnership

Figure 1: EngD model, showing the relationship and benefits to the research Engineer, University and the sponsoring company

**Sponsoring Company:** Rolls-Royce plc is a global business engaged in providing power systems and services for the use on land, at sea and in the air. They operate on four global markets – civil aerospace, defence aerospace, marine and energy. With customers in 150 countries and an order book worth £35bn, the company has over 38,000 employees with offices, service centres and manufacturing bases located in over 50 countries. The world aeroengine market is expected to deliver 139,000

engines worth over \$810 billion over the next 20 years, powering 65,000 commercial aircraft and business jets [1]

To stay competitive, maintain leadership and reduce the environmental impact, a generous proportion is budgeted for research and development activities. During the past five years, the company has invested over £3.5bn. Currently they have 29 University Technology Centres (UTC) across the world undertaking intensive research to develop promising ideas that will be eventually transferred into the company for the future. High priority research themes include the development of technologies to reduce the environmental impact, namely noise and gaseous emissions. The research conducted in this doctoral research program is related to this theme, where improved temperature sensing capabilities will aid future designs with greater efficiency.

#### 1.2 Background

There are many motivations for temperature measurement inside development aeroengines which are highlighted in detail in the next chapter. Unfortunately, the accurate measurement of surface temperatures under harsh rotating conditions is challenging. Phosphor thermometry can provide distinct advantages over existing methods such as thermocouples, pyrometry and thermal paints/melts, in certain instances. There are many capability gaps for surface temperature measurements, especially the first stages of the turbine section, where phosphor thermometry may be able to fill. The idea of phosphor thermometry dates back to the late 1930's, and although it exhibits characteristics to make accurate measurements, it is still immature and its implementation to enable successful temperature measurements on rotating gas turbine environments presents some engineering challenges that requires further research. Rolls Royce plc wants to increase its knowledge base and confidence in using phosphor thermometry to measure surface temperatures components in such instances to help in the development of future engines.

#### 1.3 Aims and Objectives

The generic objective of this project is to further the research on thermographic phosphors so that a temperature measurement solution can be applied on hot regions of gas turbine engines, where conventional competing technologies and techniques are problematic. The research should include the design of such a system including phosphor choice with the expected performance in terms of measurement capability and uncertainty which are to be validated. As this project is partially funded by the RR Civil Aerospace market, there is focus on civil aeroengines. The key objectives to achieve this are:

- Understand commercial drivers for undertaking phosphor thermometry research, to ensure it is focused with an end purpose in mind.
- Identify limitations of existing and competing temperature measurement techniques. Identify opportunities and niche markets by analysing capability gaps and requirements where phosphor thermometry would be desirable over competing techniques.
- Understand the theory behind phosphor thermometry, review of the state of the art including techniques, system apparatus and applications.
- Identify limitations, consideration factors and requirement for the successful implementation of phosphor thermometry in gas turbines.
- Identify the key phosphors that would have desirable characteristics to permit measurement, including: spectral emission wavelengths, intensity, lifetime and operating temperature.
- Design and build systems with appropriate testing in laboratory conditions to validate and evaluate the performance in terms of temperature capability and uncertainty.
- Provide an estimation of performance (temperature range and uncertainty) at operating engine conditions with a recommendation to Rolls Royce plc.

## 1.4 Thesis Structure

A brief introduction to the engineering doctorate program and the sponsoring company (Rolls Royce plc) was provided and was followed by the aims and objectives of the research project. As this is an EngD project, opposed to a traditional PhD, the business element of the research is emphasised in the next chapter which discusses the motivations for undertaking such research. A comprehensive review of existing measurement technologies is undertaken, and the capability gaps and future requirements for temperature measurement inside turbine engines are also discussed.

Chapter three provides a literature review of phosphor thermometry, highlighting the state of the art and the generic system setup. The main issues and consideration factors are also reviewed in this chapter. Chapter four investigates the maximum energy that would be deliverable to a phosphor target. The amount of energy failing onto the phosphor has implications on the performance of the phosphor thermometry technique, with larger energies allowing greater emission intensities and a higher temperature capability. Chapter five highlights the details of spectral analysis and provides a review of different bonding techniques. Chapter six characterises the intensity of different phosphor emissions to allow estimations of radiant emission levels from different optical configurations, phosphors and input energies. The

emission intensity relative to the blackbody radiation is also discussed in this section. Chapter seven characterises the temporal characteristic (decay lifetimes) and establishes the quenching temperature and useful temperature range of different phosphors. Chapter eight builds on this taking into consideration various limiting factors under rotating conditions. For rotating components, an upper temperature limit is reached based on the lowering emission intensity at rising blackbody radiation levels and the system's ability to detect fast decays. A lower limit is reached based on the quenching temperature, probe design and rotational velocity of the component. A phosphor selection criterion is used to aid the selection of suitable phosphors for different engine components. The optical layout is tested on stationary and rotating cases under laboratory conditions using similar design constraints, including fibre choice, maximum permissible lens size and target distances. These tests validate design methodologies and assumptions to enable testing on full scale rotating engine components. Chapter nine concludes the investigation by providing a recommendation to Rolls-Royce with the most suitable phosphor with expected performance in terms of temperature capability and measurement uncertainty for the chosen engine components.

## 2 Motivations for Phosphor Thermometry in aeroengines

## 2.1 Temperature measurement in aero-engines

#### 2.1.1 Characterisation of Engine Sensors Types

Sensing technology has significantly evolved over the past few decades, becoming increasingly more sophisticated allowing improved performance in terms of: range, uncertainty, accuracy and responsiveness. In gas turbine engines, sensors are used to characterise, monitor and control the engine, performing a vital role in ensuring safe and efficient operation. Roberts [2] notes there are three distinct sensor groups in gas turbine engines, each possessing different requirements and constraints. Table 1 highlights the purpose and desirable characteristics for each group; and Figure 2 demonstrates the relative importance of these factors, which can aid instrumentation designers/suppliers to focus research budgets to ensure their products are suited for end use.

Туре	Purpose	Characteristics	Volume and Costing
Engine Control System	Engine control system. E.g. Shaft rotational speed, gas path temperature and pressure sensors, oil and fuel flow sensors.	<ul> <li>Extremely high reliability</li> <li>Long life is critical. (Expected to operate thousands of hours with little attention.)</li> <li>Robust with long calibration intervals</li> <li>For aeroengines, low weight is high importance</li> </ul>	<ul> <li>Small numbers per engine.</li> <li>Large business volume.</li> <li>Unit cost is of high importance.</li> </ul>
Engine Health Monitoring	Monitor the behaviour of the engine during service. This aids the scheduling of maintenance activities to minimise engine downtime. Tests will use additional sensors and the data from existing control system sensors. E.g. Vibration, oil quality sensors.	<ul> <li>High levels of reliability</li> <li>Long service life. However, less critical than engine control system sensors.</li> <li>Low weight is very important.</li> </ul>	
Engine Development & Certification Testing	<ul> <li>To demonstrate product compliance with regulatory/certification requirements.</li> <li>Fully understand product behaviour/performance of engine.</li> <li>Design method validation and development.</li> </ul>	<ul> <li>Measurements made on development test engines in test facilities. However, some flight tests are also carried out.</li> <li>Moderate reliability acceptable.</li> <li>Shorter lives and high mortality rates can be acceptable if they are unavoidable.</li> <li>Low uncertainty in measurement can be of high importance.</li> <li>Weight is of low importance at ground test facilities.</li> </ul>	<ul> <li>Large numbers per engine</li> <li>However, the total business volume is small (not needed in every engine).</li> <li>Therefore, the unit cost is of low importance.</li> <li>Cost can be reduced if reliability, flexibility and uncertainty can be improved.</li> </ul>



Sensors specific for development engines and test rigs are used to demonstrate product compliance with regulatory and mandatory certification requirements for safe operation. Structural, mechanical and aero-thermal measurements enable engineers to assess the performance, validate mechanical integrity, verify and aid the development of computational fluid dynamic (CFD) and structural/mechanical FEA models and other design tools. Given that the majority of tests are carried out on test rigs, the relative importance of specific requirements for this sensor type is different. For example, weight is of low importance at ground test facilities, with shorter lives with high mortality rates being acceptable. However, a high accuracy with a low measurement uncertainty can be of high importance since the total business volume is small as they are not needed in every engine. As the research focused in this thesis is primarily for development engines, factors associated with weight, long life and unit costs can therefore be ignored.



Figure 2: Analysis of the relative importance of factors for the three different engine sensor types

#### 2.1.2 Research into improved engine efficiency

Aircraft manufactures (e.g. Boeing and Airbus) would like their aircraft to be as efficient as possible for many competitive reasons. There are many ways in which this is achieved including:

- Improved aerodynamic performance for drag reduction
- Lower structural weight from carbon fibre composites
- Improved control systems
- Increased engine efficiency.

This thesis focuses on the last solution, increasing engine efficiency. Although the research is primarily focused on civil aeroengines, it could also applied to industrial gas turbine engines for power/electric generation. The gas turbine engine is already well designed for efficient operation, however, there are still many factor that drive designers to continually improve design and increase operational performance, and in recent years its importance has been greatly increased. In the past 5 years, Rolls-Royce plc have invested over £5bn for research and development activities. Some reasons justifying this, highlighted in Figure 3, include: to achieve product differentiation, lower operating and manufacturing costs, increase safety, performance, operational efficiencies and reliability. Many of these reasons are interlinked, for example increasing operational efficiencies also increases range whilst lowering emissions.



Figure 3: Reasons for engine research and development activities

The civil aviation market demands aircrafts to comply with stringent fuel consumption and emission regulations due to growing concerns over greenhouse gases, primarily  $CO_2$ . Unfortunately,  $CO_2$  is a product of the complete combustion of any hydrocarbon fuel, it is therefore unavoidable and is directly proportional to fuel burn; a reduction is only achievable through improvements in efficiency. It is predicted that by 2050, the  $CO_2$  produced by the UK's aviation industry could be four to ten times higher than in 1990 [3]. A government strategy for higher pricing and taxes can choke demand for civil flights, which could adversely restrict aviation expansion, and cause a further decline in economy. In the current economic downturn, this could have further disastrous global consequences. As climate change awareness and environmental importance continues to grow, policy makers are in a predicament balancing the benefits of environmental emission control against possible global economic consequences. A sensible solution reducing the magnitude of the compromise is the promotion of more efficient, greener technologies. This is currently being put in to practice with the government providing many incentives to encourage this.

Apart from environmental and political pressures, financial motives and other competitive reasons also contribute towards the driving force behind improved engine efficiency (Figure 4). It is claimed that an improvement of one percent in efficiency can save \$20 million in fuel costs over the complete lifetime of a typical industrial gas fired 400-500MW power plant [4]. With growing oil prices, these savings would be further amplified. For aviation, improved efficiency lowers specific fuel consumption, which not only reduces operational costs, it also increases an aircraft's flying range requiring fewer refuels and downtime, which is another competitive factor. In military aviation, there is a continual drive for increased flight performance parameters which can be achieved with more efficient engines with higher thrust-to-weight ratios.



Figure 4: Examples of factors that drive aeroengine research and development activities.

Rolls-Royce's main competitors in the civil aeroengine industry include Pratt and Whitney, General Electric and Safran. The market is not very contestable due to high costs and barriers to entry that exist due to rigorous aviation regulations and certification procedures and many years of continual research which has resulted in a wealth of technical experience, patent applications, technical expertise and reputation that can not be easily replicated.

From economic supply and demand theory, increasing efficiency in engines can:

- Increase the demand of aircrafts in general due to lower operating costs.
- Provide a range of performance enhancing features that can be used for product differentiation,
- Aid societal marketing campaigns to promote the company's green involvement.

For these reasons, improved engine efficiency is considered extremely competitive, and can influence customers (aircraft manufacturers) to switch engine suppliers, which can result in significant losses in revenue to the favoured supplier. Sloman and Sutcliffe [5] notes that oligopolies can not ignore the development of competitors. Significant business may be lost if competitive advantages, in this case engine efficiencies, are not maintained relative to competitors. Therefore, it is in the interest for all competitors to engage themselves in improving their engine design for better efficiency.

According to Sloman and Sutcliffe [5], organisations can work together in some aspects to maximise profits. The mutual involvement on competitive factors, such as engine efficiency and performance, can be difficult to agree upon. However, by treating some aspects, such as development test instrumentation, as pre-competitive, firms can work together by addressing common needs by creating joint ventures and collaborations. These are becoming increasingly more common - examples include the PIWG (*Propulsion Instrumentation Working Group*) and the EVI-GTI (*European Virtual Institute for Gas Turbine Instrumentation*), and more recently joint PIWG/EVI-GTI. These were formed in an attempt to maximise instrument development work by sharing research and development resources. A matrix of technology gaps published by the EVI-GTI is maintained by periodically capturing, analysing and reviewing common instrumentation needs. Their aim is to promote collaboration between gas turbine manufacturers, concentrate available funding on priority needs, and steer instrumentation supply chain and academic researchers.

#### 2.1.3 The role of accurate temperature measurement

The three most acknowledged ways to improve engine efficiency are by [6]:

- Increased compression through the compressor
- Improved combustion efficiency
- Temperature increase of the gases entering the turbine

Both fuel economy and thrust can be improved without increasing the size or weight of the aeroengine if higher turbine inlet temperatures are achieved [7]. The current trend, illustrated in Figure 5 and Figure 6 demonstrates the gradual evolution of increasing turbine inlet temperatures, which now exceeds the limits of the flow confining materials and has only been made possible through advances in material sciences and manufacturing technologies that have enabled the use of thermal barrier coatings (TBCs) and advanced film cooling. In the case of film cooling, air is drawn from the main gas path that consequently results in a loss in overall efficiency [8], and it is likely that the increase in airflow will result in an increase in NO<sub>x</sub> emissions [9] which is counter productive for cleaner environments. Therefore tighter emission control measures in the future would possibly limit the amount of cooling available for

this, necessitating the need for efficient thermal protection systems that would require accurate temperature measurement.

Unfortunately, operating at higher temperatures has severe consequences:-

The higher temperature leads to a harsher environment and places practical restrictions based on the metallurgy of turbine components. In general, increasing the temperature reduces the specific strength of materials, which reduces component life and increases the chance of failure. A surface temperature change or an uncertainty of 10K can result in 100% impact on the lifetime for creep predictions and 30% oxidation life prediction [10]. A few minutes of operation at over-temperature conditions can result in dramatic reductions in blade life [11]. This rapid loss of structural integrity places great importance on operating the turbine below their thermal design limit. Based on this practical issue alone, the optimum temperature is prescribed by the balance between the benefits of thermal efficiency at higher temperature operation and material stability and life.



Figure 5: Operational temperature trends [12]


Figure 6: Increase in turbine inlet temperatures [13]

A higher combustion temperature also increases the production of nitrous oxides  $(NO_X)$ , as shown in Figure 7, and in reality the effects  $NO_X$  emissions are also carefully considered. It can be appreciated that there is a complicated trade off between engine efficiency and  $CO_2$  reduction, the combustion and turbine inlet temperatures, the stability and life of the flow confining materials, the amount of cooling required and the resultant  $NO_X$  emission. This goes beyond the scope of this thesis, and therefore the effects of  $NO_X$  will not be discussed further.



Figure 7: Influence of temperature on CO<sub>2</sub> and NOx emissions [13]

If the temperature is not known accurately, the engine is forced to operate below its optimum to prevent failure. The ability to make accurate measurements with reduced uncertainty offers the potential to increase engine efficiency by allowing it to operate closer to its design limit [7].

## 2.1.4 Verification / Design tool

Accurate thermal maps can also help designers validate the structural integrity of components that may form a part of the safety requirements. It enables engineers to better understand the flow physics and thermal behaviour allowing them to assess the aero-thermal performance of the engine, verify computational CFD, FEA models and other design tools, which can be used to optimise and facilitate the development and testing of newer engine designs and thermal protection systems. It can also enable better predictions for material stability and life. Existing engine components may be over-engineered possessing either an increased weight or greater amount of cooling; optimisation would permit a leaner engine design that would require less thrust and hence less fuel.

### 2.1.5 Health Monitoring / Engine Control

The previous motivation focused on increasing engine efficiency and was limited for use in development test engines. Another motivation that is gaining more importance is maintenance. With new schemes such as 'power by the hour', sophisticated engine health monitoring systems that reduce maintenance schedules are regarded as highly competitive, being beneficial for both engine manufacturer and client, providing cost savings whilst minimising downtime. The accurate measurement of engine component temperatures allows for better predictions of component life, which can be used to optimise and prolong maintenance schedules. These can be based on component life instead of schedules based on hours of operation, and can prevent the premature replacement of expensive blades or other components. The reduced scrap rate is not only financially beneficial, it is also good for the environment.

Thermal barrier coatings can be modified to behave as thermographic phosphors and show temperature dependant properties [14]. The advantage of this procedure is that no additional phosphor layers are required. This concept was first proposed by Choy, Feist and Heyes [15] and is being further developed for monitoring blade temperatures, assessing the quality of thermal barrier coatings and blade life [14, 16-18]. Roberts [2] notes that sensor technology used for engine control systems and engine health monitoring, described in section 2.1.1, is often a subset and a developed form of the technology that was originally used for development test engines. It is envisaged that the research on phosphor thermometry for development engines presented in this thesis will be further developed in the future so that it becomes more compact, lightweight and cost effective to be used as health monitoring and engine

There are also other application areas outside of the aero engine where research into phosphor thermometry would benefit. Examples include the nuclear, fuel cells, automotive and process engineering industries.

## 2.1.6 Summary

Research into methods to provide accurate temperature measurement of engine components with a low uncertainty is a very important tool for providing thermal analysis that can be used for:

- Verifying the mechanical, structural integrity of components at elevated temperatures
- Assessment of aero-thermal performance
- Verify and help further develop CFD, FEA and other development tools.
- Aid the design and development for better, leaner more energy efficient engines with reduced cooling requirements.
- Provide insitu correction/validation for measurements made from other methods,
   e.g. pyrometry, thermal paints.
- Provide better component lifetime predictions, which can help to prolong maintenance schedules
- With a reduced temperature uncertainty, the engines can be allowed to be operated closer to their design limits, which can help to improve engine efficiency
- Provide a platform technology that can be further developed into a technology that could be used for health monitoring or engine control sensing purposes.
- Other application areas: automotive industry, fuel cells, processing industry.

# 2.2 Review of the existing methods for surface temperature measurement inside aeroengines

## 2.2.1 Introduction

Unfortunately experimentally measuring surface temperatures under harsh rotating engine conditions is challenging. This section provides a review of existing technologies used for temperature measurement in aeroengines. The most widely used methods include the use of thermal paints, thermocouples and radiation pyrometry. The principles, pros and cons, typical performance with uncertainty characteristics are compared and discussed. Given that each technique has strengths and weaknesses, the surface temperature measurement of different engine components may favour a different technique depending on the measurement objective.

### 2.2.2 Thermocouples and RTDs (contact sensors)

Thermocouples consist of two different metallic wires connected together. They rely on the Seebeck Effect where a voltage is created due to the diffusion of electrons along a temperature gradient. They provide an economical and accurate method for contact measurement and have been widely used to measure temperature of flows and components inside aeroengines. The most common types used in engine environments are highlighted in Table 2.

Тур	е	Chemical Composition Wire 1 (+)	Chemical Composition Wire 2 (-)	Typical Range (°C)	Comments
	Е	<i>nickel-</i> chromium alloy	<i>copper</i> -nickel alloy	-270 to 1000	Suited for low temperatures
Metals	к	<i>nickel-</i> chromium alloy	<i>nickel-</i> aluminium alloy	-270 to 1372	Cheap high temperature thermocouple. However suffers from high hysteresis.
Base	N	<i>nickel-</i> chromium- silicon	<i>nickel</i> -silicon- magnesium	-270 to 1300	Most suitable base metal thermocouple for high temperatures. Low hysteresis.
sle	R	<i>platinum</i> -13% rhodium	Platinum	-50 to 1768	R and S types are better suited for
Metä	s	<i>platinum</i> -10% rhodium	Platinum	-50 to 1768	temperatures below 1300°C
Nobel	в	<i>platinum</i> -30% rhodium	<i>platinum</i> -6% rhodium	0 to 1820	B is better suited for temperatures above 1300°C

Table 2: Most common thermocouple types used in aeroengines

Other more specific thermocouples that offer superior characteristics, include:

- Au/Pt thermocouples offers high accuracy below 1000 °C.
- Pt/Pd thermocouples offers high accuracy from 1000-1500 °C.

Thermocouples can undergo chemical and physical changes at higher temperatures, leading to loss of calibration. While thermocouples depend on the Seebeck effect to generate a voltage, RTD's (resistance temperature detectors) depend on changes in electrical resistivity and require power to operate. As electrons move through a metal, they are impeded by atomic thermal vibrations in the crystal lattice, with higher temperatures resulting in greater impedance and resistivity. Although RTDs can theoretically measure temperatures to about 1000°C, it becomes increasingly difficult to prevent contamination from impurities and therefore a practical upper temperature limit is reached at around 700°C.

Generally, RTD's can offer greater stability, accuracy and repeatability with low drift characteristics compared to thermocouples. However, RTDs are larger in size, have slower response times, have a reduced upper temperature limit and can be expensive compared to standard base metal thermocouples; noble metal thermocouples offering higher accuracy and stability can also be expensive.

Issues with both these devices are:

• They are intrusive contact sensors and have to withstand the harsh gas turbine environment.

- It is assumed that thermal equilibrium is maintained between the measuring surface. This can be a significant source of error with poor thermal contact increasing measurement uncertainty.
- They can distort the gas flow consequently changing the environmental conditions of that surface. The installation method is of vital importance, and devices may be embedded into a surface to minimise interference. This can be difficult and expensive with the routing of the wires also being problematic. In some cases, wires are embedded into machined grooves which can compromise component structural integrity. Wire thermocouples are commercially available as low as 0.25 mm diameter. More recently, thin film thermocouples and RTDs can be deposited directly onto the components surface via vapour disposition processes, possessing a total thickness of 10-20microns. These reduce the thermal inertia allowing faster response times with minimal intrusiveness, and are therefore becoming increasingly more common in gas turbine engines.
- As thermocouples and RTDs are electrical sensors, they are affected by electromagnetic interference in the surrounding environment that can induce additional uncertainty.
- When used on rotating components, slip rings that link rotating lead-outs to stationary wiring, or telemetry units that transmit wireless signals from a rotating assembly to a stationary system, are required, which further increases the measurement uncertainty.
- They lack measurement detail since they only provide discrete diagnosis.

## 2.2.3 Radiation Pyrometry

Pyrometry is a remote surface temperature measurement method that eliminates many problems associated with contact sensors, and has become very popular in turbo-machinery. It is governed by Planck's Law which relates the thermal radiation intensity, wavelength and temperature. The advantages of pyrometry include [19]:

- No upper temperature limit because radiation energy increases with temperature.
- Fast response as there is no inherent thermal inertia of thermocouples
- Non-intrusive. This avoids the thermal and fluid dynamic interference of turbo machinery flows in the gas stream.
- Reduced routing problems for rotating components
- Immunity to electromagnetic interferences from the surrounding environment
- Traversing the field of view, via a scanning system can enable 2D thermal maps on rotating components

There are a variety of pyrometry types including fixed turbine blade, fibre optic and scanning turbine pyrometers. Despite these advantages, there are limitations that confine their use. These include:

- Optical access is required to collect radiation from a defined target.
- Lower temperature limit where there is insufficient radiation to make a reading; for gas turbine pyrometers, this is about 500°C.
- Planck's relation is only valid for perfect radiators and the intensity for real surfaces is significantly reduced by the emissivity factor. Unfortunately this does not remain constant and is a function of the temperature, wavelength, observation angle and surface conditions which includes roughness, oxide layer and contamination [19]. This makes it difficult to specify accurately for corrections. In addition, thermal barrier coatings introduce new problems because they become translucent at high temperatures and also exhibit a variable emissivity due to contamination and ageing effects.
- Reflected radiation from other sources and surfaces make the temperature reading biased high. According to Nutter [20], the magnitude of the reflected radiation is function of geometry. Measurements indicate that up to 70% of detected energy collected by the detector between wavelengths 350-1050nm can be reflected energy [21]. Atkinson and Strange [22] reports this value to be over 75% in the first stage of the high pressure turbine.
- Combustion Gas stream/flame interference Errors are introduced if the sight path is not completely transparent at the pyrometers operating wavelength. The gas stream/flame interference[23] makes pyrometry very sensitive to the environment [19, 24]. Radiation from the target surface can be partially absorbed by the gases, making the temperature reading biased low. Conversely, radiation from the gases at high temperatures can make the reading biased high. These effects depend on the chemical constituents, temperature and gas emittance which can produce considerable levels of emission, reflection, absorptions and scattering phenomenon, which can introduce significant errors. Flames from the combustion chamber and hot particulates, such as carbon particles and soot can cause incandescent and chemi-luminescence spikes that introduce further measurement errors. Flames are an intense source of thermal radiation, which can saturate the detector preventing measurement. These problems are usually confined to the first stage of the turbine.
- Optical contamination Optical pyrometer mounts have sight tubes exposed in the gas stream. The target may become obscured because of particle disposition, such as soot and sand on the lens, leaving a temperature that is biased low. A purge air system with an air curtain configuration can minimise the effects of optical contamination [19].

## 2.2.4 Thermal Paints and Thermal Melts

Thermal paints contain blends of pigments held together by resin that undergo a series of permanent colour changes as the temperature is increased. They can be applied onto a surface and are widely used in engine development and testing environments. Indication spots and strips incorporate a similar technology. Thermal melts, containing various metal alloys, can also be used to determine temperatures by observing the appearance of the solid and molten phases on a surface. Thermal paints and thermal melts can be easily applied to large areas including complex shaped components, and provide an effective way to obtain a permanent visual record of the peak temperature variations over surfaces. A variety of multi-change paints are available covering a temperature range from 150-1350°C [25-28]. They are usually split up into two main categories;

- Low Temperature Paints: Using four or less colour changes and covering a temperature range from 120°C to 590°C, with an uncertainty in the region of ± 20°C
- High temperature Paints: Multi change paints covering a temperature range between 500 °C to over 1300 °C. The accuracy and measurement uncertainty of these paints is in the region of ± 4%

They are generally used for large area coverage where real-time readings are not required. A post-test example of thermal paints applied to an engine component is shown in Figure 8.

The main issues with these include:

- Accurate temperature determination is very difficult and highly dependant on the operator's skill and experience. Quantitative analysis is therefore very subjective.
- Automation using digital cameras can save time and cost. However, this can be difficult as non-uniform illumination and various lighting conditions cause significant sources of error. Another temperature indicative coating technology that is currently being developed undergoes permanent changes that affects its fluorescent properties under UV light, enabling peak temperature histories up to 1500°C [29]. Since this new technique is not visible and requires UV photoluminescence interrogation, the method can potentially eliminate human errors and errors caused by lighting conditions that prevent automation.
- Thermal paints are not only dependant on temperature; they are also dependant on the period of thermal exposure. Although paints are calibrated over a matrix of temperatures and exposure times, it can be difficult to determine whether a pigment change was a result of a peak temperature or a lower temperature with a longer exposure. Thermal melts are generally are

insensitive to the thermal exposure time, and are often used to calibrate thermal paints.

- Paints may react with components and should therefore be pre-tested. Paints may be applied by spray or brush; however care must be taken when they are applied over small cooling holes and other features.
- Since these coatings present changes that can not be reversed upon cooling, a
  permanent record of the peak temperatures can be visualised offline [30, 31].
  Although this is desirable in some instances, it makes the technique limited to
  a single dedicated test, with subsequent tests requiring engine disassembly,
  paint strip and reapplication, which can be expensive and time consuming.
- It does not provide real time data.
- Optical access is required, usually requiring strip down of components.

Rolls-Royce plc widely use thermal paints for development engine testing. However, full scale engine testing is very expensive and only reveals peak temperatures for tests that only last a period of approximately 3 minutes. The implementation of a system that can measure temperature with similar accuracy in real time under different operating conditions can not only provide very useful data for engineers; it is cost effective and can significantly reduce research and development time.



Figure 8: Results from thermal paints applied to an engine component [32].

## 2.2.5 Summary

In summary, it has been shown that established techniques include the use of thermocouples, pyrometry and thermal paints/melts. The methods either lack detail or do not have the desired accuracy with high levels of uncertainty, or are too expensive for continuous testing (Figure 9). Table 3 summarises the performance and main factors and limitations for each measurement technology. Although

thermocouples are typically cheap, accurate and easy to install, in complex flow and rotating environments, thermocouples can be intrusive, difficult to install with routing of the wires being problematic. The measurement also lacks detail since it only provides discrete diagnosis. Pyrometry offers many advantages over thermocouples including non-intrusiveness, immunity to electromagnetic radiation with reduced problems with routing [19]: However, there still remain many issues that limits their use, including: issues with emittance variation with temperature, reflected radiation [33, 34] and gas stream/flame interference[23], which makes them very sensitive to the environment [19, 24].



Figure 9: Issues with existing methods.

Phosphor thermometry has many attributes that make it better suited to competing/existing methodologies in many applications. It is accurate and like pyrometry, share many of the non-intrusive benefits over thermocouples (e.g. routing of cables and immunity to electromagnetic noise), while overcoming many of the shortfalls and issues related to it (e.g. emissivity variations, flame interferences, reflected radiation). Despite these advantages, the variation in component geometry, operational temperatures, optical access and rotation speeds makes it difficult to generalise a universal measurement solution for all engine components. The high temperature and fast rotating engine environment presents some unique challenges with interrelated issues that would need to be collectively considered before a successful phosphor thermometry measurement system can be implemented. Examples include rising blackbody radiation, restricted optical access and time to collect data, etc. These factors will impose temperature limits and greatly influence the design philosophy of the measurement system, including phosphor choice in terms of lifetime decay and absolute intensities, excitation/detection methodologies, and probe design. These factors will be considered throughout the rest of the thesis. The technology is not mature enough for direct use and needs further research before it is can reveal surface temperatures of components in aeroengines.

	Characteristics	Advantages	Limitations
Thermocouples	Range: N TYPE: 270-1300°C Nobel Metals -50- 1750°C Static component accuracy/uncertainty – 0.5% / ± 2°C RTDs: Up to 700°C	<ul> <li>They can provide accurate temperature measurements</li> <li>Offer real-time temperature monitoring</li> <li>Relatively cheap, however the installation costs may be costly, especially on rotating components</li> </ul>	<ul> <li>Intrusive</li> <li>Limited number</li> <li>Costly installation - rotating components will require telemetry or slip rings</li> <li>Bonding to ceramic surfaces</li> <li>Electromagnetic interference</li> <li>Lack detail since it provides discrete diagnosis.</li> </ul>
Thermal Paints/Melts	Range: Capability 120- 1300°C through a variety of paints that are available. Up to 1500°C underdevelopment. Uncertainty: ± 20°C up to 500°C ± 4% above 500°C Thermal Strips/tabs Up to 300°C, ± 3%	<ul> <li>Effective way to cover a larges, complex geometries</li> <li>Can be used on stationary and rotating components</li> <li>Provides a permanent visual record of peak temperatures: Can be viewed offline.</li> </ul>	<ul> <li>Irreversible</li> <li>Requires a dedicated test, with subsequent tests requiring engine disassembly, paint strip and reapplication, which can be expensive and time consuming</li> <li>No real time data</li> <li>Paints may react with components</li> <li>Highly dependant on operator's skill and experience.</li> <li>Automation difficult</li> <li>Thermal paints are also dependant on thermal exposure.</li> </ul>
Radiation Pyrometry	Range: 500-3000°C Uncertainty: approx 20°C Time resolution: approx 1ms	<ul> <li>No upper temperature limit</li> <li>Fast response - no inherent thermal inertia</li> <li>Non- intrusive</li> <li>Reduced routing problems for rotating components</li> <li>Immunity to electromagnetic interferences from the surrounding environment</li> <li>2D Surface map possible with scanning.</li> </ul>	<ul> <li>Optical access is required</li> <li>Lower temperature limit of 500°C</li> <li>Emissivity variation with temperature and surface condition.</li> <li>Translucency of ceramic coatings</li> <li>Sight path transmission</li> <li>Reflected radiation</li> <li>Combustion Gas stream and flame interference</li> <li>Optical contamination</li> </ul>
Phosphor thermometry	Range 20°C – 1700°C Achievable through various phosphors	<ul> <li>Immunity to electromagnetic interferences from the surrounding environment</li> <li>2D Surface map possible with scanning</li> <li>Immune to effects caused by emissivity, flame interference and reflections from components</li> <li>Non- intrusive.</li> <li>Reduced routing problems for rotating components</li> </ul>	<ul> <li>Optical access required</li> <li>Limit due to decreasing phosphor's intensity and faster decaying response with increasing temperature</li> <li>Rising levels of blackbody radiation</li> </ul>

Table 3: Pros/Cons and performance of existing temperature measurement methods in gas turbines.

## 2.3 Capability shortfalls, gaps & requirements analysis

It is difficult to generalise a "one-for-all" thermographic phosphor measurement solution for all engine components. Some components are rotating, whereas others remain stationary. There will also be significant variations in the operational environment including temperature range and optical-access. These factors will influence the design and methodology of the overall phosphor thermometry measurement system. Due to economic restrictions it is important to review existing measurement shortfalls, capability gaps and requirements inside gas turbines, and identify key areas/ engine components where research could be concentrated in order to address a real need. This section also highlights key information such as temperature range and rotation speeds that would be used to feed into the design for phosphor thermometry.

A study undertaken by the EVI-GTI, comparing the current available instrumentation capabilities and requirements from engine manufacturers, has revealed capability shortfalls and gaps that can be used to prioritise research activities to ensure new products meet a market need. The capability gaps are organised in a matrix based on engine location and measurement challenges/requirement as opposed to technology lines. Roberts [2] notes that organisation divided along technology lines are consequences of developer's specialist technology areas, e.g. optical sensors, chemical sensors, etc., and can result in 'solutions in search for problems' rather than real problems being identified with the most appropriate solution developed. A simplified version of the measurement matrix is shown in Figure 11, highlighting the existing capabilities and gaps based on application location and environment. It can be seen that there are shortfalls and capability gaps for measuring component surface temperatures, especially in hot sections of the engine. The surface temperatures inside different turbine engines types, (e.g. industrial, marine, turboprop, military and civil aero-engines vary depending on the engine design. Since, this project is partially funded by the Civil Aviation department, there is a focus on parameters and geometries for current and anticipated future civil aero-engines (Trent 900, 1000 and above).





Figure 10: Component locations in an aeroengine [13]

- 48 -

Measurand/ Measurement Challenges	Externals (-40 to 300C) 1 bar	Intake (-40 to 150C) 1 bar	Compressor (Front) and Fan (-40 to 350C) 1 – 20 bar	Compressor (rear) (350 to 700C) 10 – 45 bar	Combustion system (700 to 2400C) 30 – 45 bar	HP Turbine (1000 to 1800C) 30 -45 bar	LP Turbine (700 to 1000C) 1 -20 bar	Reheat/After Burner (Aero) (700 to 1800C)	Exhaust (0 to 700C) 1 bar	Shafts and bearings ()	Rigs and Cascades ( 0 to 600C)
Aerodynamic Measurements											
a.1 Airflow Metering											
Intake Airriow Intake Condensation /water content	ina	2	na	na	na	iva	na	na	na	ina	1
Core mass flow	Na	1	1	1	2	2	2	na	1	Na	1
Air systems pressures	Na	na	1	1	1	1	1	na	na	Na	1
Offtake/Cabin Bleed flows	1	na 1	1	<u>1</u>	na	Na 22	na	na	na	Na	1
breather flows	2	na	na	na	na	Na	na	na	na	2	na
a.2 Gas, Flame and Particle analysis	Nia					Niz				N	
CO2	Na	na	na	na	2	Na	na	na	2	Na	na
СО	Na	na	na	na	2	Na	na	na	2	Na	na
UHC	Na	na	na	na	2	Na	na	2	2	Na	na
Formaldyhides PM10	Na	na	na	na	3	Na	na	na 2	3	Na	na
02	2	2	na	na	2	Na	na	na	2	Na	na
Smoke measurement	Na	na	na	na	2	Na	na	2	2	Na	na
Cabin Bleed Air quality	3	na	3	3	na	Na	na	na	na	Na	na
Temperature by gas analysis	Na	na	na	na	2	1	na	2	na	Na	na
a.3 Boundary Layer											
Surface static pressure	Na	1	1	1	1	1	1	1	1	Na	1
Surface Tx	1	_∠ na	na	na	2	3	3	2	na	Na	1
Conductivity measurements of coatings	Na	na	na	na	3	3	na	3	3	Na	na
Surface hot film sensors	1	1	2	3	3	3	3	3	na	Na	1
a.4 Liquid Flow											
Fuel	3	na	na	na	3	Na	na	3	na	Na	na
Lubricant	3	na	na	na	na	Na	na	na	na	2	1
Water High turndown ratio flowmotors	1	na	na	na	na	Na	na	na	na	Na	na
Turbine Flowmeters	2	na	na	na	2	Na	na	na	na	Na	na
Positive Displacement	1	na	na	na	na	Na	na	na	na	Na	na
Coriolis flow meter	2	na	na	na	na	Na	na	na	na	Na	na
	<u> </u>	na	na	na	na	INd	na	na	na	INd	na
a.5 Gas Path Instrumentation											
Temperature rakes steady	Na	1	1	1	3	3	2	2	1	Na	1
Temperature Takes dynamic	Na Na	na 1	2	2	3	3 3	2	2	na 1	Na Na	2
Temperature LE instro dyn	Na	na	2	2	3	3	2	2	na	Na	2
Pressure rakes steady	Na	1	1	1	3	3	2	2	na	Na	1
Pressure rakes dynamic Pressure I F instro steady	Na	1	1	2	2	2	2	2	2	Na	1
Pressure LE instro dynamic	Na	1	1	2	2	2	2	2	2	Na	1
Traversible instrumentation	Na	na	na	na	na	3	3	3	1	Na	1
Hot Wire anemometry	Na	na 1	na	na	na	Na	na	na	na	Na	2
Laser Anemometry 3-D	Na	1	na	na	na	Na	na	na	1	Na	1
Seeding- single stage	Na	na	na	na	na	Na	na	na	na	Na	1
Seeding- Multistage	Na	na	na	na	na	Na	na	na	na	Na	2
	Na	na	na	па	па	Na	па	па	na	Na	1
a.6 Torque and Thrust											
Torque meters	1	na	na	na	na	Na	na	na	na	1	1
In-engine shaft torque	Na	na	na	na	na	Na	na	na	na	2	1
Load cells	Na	na	na	na	na	Na	na	na	na	na	1

Structural Measurements											
b.1 Aerodynamic Instabilities											
Dynamic gas pressure (wall tapping)	Na	1	1	2	3	3	3	3	2	na	1
b.2 Component Temperatures											
Thermocouples:- Thin film	Na	na	2	2	3	3	3	3	2	2	1
Thermocouples: Thin film under TBC	Na	na	na	na	3	3	na	na	na	na	Na
Thermocouples:- Embedded.	Na	na	1	1	2	2	2	2	na	1	1
Thermocouples surface temp	1	1	1	1	2	2	2	2	2	1	1
Resistance sensors	1	1	na	na	na	Na	na	na	na	na	1
Thermal paints (witness)	1	na	na	2	2	2	2	2	2	na	Na
Thermal Paint (reversible)	1	1	2	3	3	3	3	3	3	3	1
Thermal imaging	1	1	3	3	3	3	3	1	1	na	Na
Pyrometry	Na	na	na	na	na	2	2	na	na	na	2
Scanning Pyrometry	Na	na	na	na	na	2	2	na	na	na	Na
Journal J. J. Contemport			ma								
b.3 Fatigue & vibration											
Accelerometers	1	na	2	2	2	2	2	2	2	1	1
Foil straingauges (dynamic)	1	1	1	na	na	Na	na	na	na	1	1
Wire and cement gauges (dynamic)	Na	na	na	2	2	2	2	2	2	na	Na
Wire and ceramic gauges (dynamic)	Na	na	na		2	2	2	2	2	na	Na
Thin Film straingauges (dynamic)	1	1	1	2	2	2	2	2	2	1	1
TE gauges (Under TBC dynamic)	Na	1	1	2	2	2				1	Na
Tin Timing (fluttor)	Na	na	1	2		2	2	na	na	na	1
Tip Timing (nucler)	Na	na	2	2	na	 	3	na	na	na	2
Tip timing (synchronous modes)	Na	na	2	2	na	2	2	na	na	na	Na
	IN C	l lla	l lla	i la	l lla			i la	na	na	ING
The timing system for sin odded biddes											
h 4 Loads and Displacements											
b.4 Loads and Displacements	2	2	2		2	2			2	2	
b.4 Loads and Displacements Steady strain gauges	2	2	2	2	2	3	3	3	2	2	na
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection	2 Na	2 na	2 na	2 na	2 na	3 Na	3 na	3 na	2 na	2	na
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement	2 Na Na	2 na na	2 na 1	2 na 2	2 na na	3 Na 3	3 na 3	3 na na	2 na na	2 2 na	na na na
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance	2 Na Na Na	2 na na na	2 na 1 2	2 na 2 2	2 na na na	3 Na 3 2	3 na 3 2	3 na na na	2 na na na	2 2 na na	na na na 1
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance blade lean, untwist Ean blade sumping profile	Na Na Na Na Na	2 na na na na	2 na 1 2 2	2 na 2 2 2	2 na na na na	3 Na 3 2 Na	3 na 3 2 na	3 na na na na	2 na na na na	2 2 na na na	na na na 1 2
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance blade lean, untwist Fan blade running profile Shaft displacement	Na Na Na Na Na Na	2 na na na na na	2 na 1 2 2 2 2	2 na 2 2 2 na	2 na na na na na	3 Na 3 2 Na Na Na	3 na 3 2 na na	3 na na na na na	2 na na na na na	2 2 na na na na	na na na 1 2 2
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance blade lean, untwist Fan blade running profile Shaft displacement	Na Na Na Na Na Na	2 na na na na na na	2 na 1 2 2 2 2 na	2 na 2 2 2 na na	2 na na na na na na	3 Na 3 2 Na Na Na	3 na 3 2 na na na	3 na na na na na na	2 na na na na na na	2 2 na na na na 2	na na na 1 2 2 na
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance blade lean, untwist Fan blade running profile Shaft displacement b5 Oil systems	Na Na Na Na Na Na Na Na	2 na na na na na na	2 na 1 2 2 2 na	2 na 2 2 na na na	2 na na na na na na	3 Na 3 2 Na Na Na Na	3 na 3 2 na na na na	3 na na na na na na	2 na na na na na na	2 2 na na na na 2 2	na na na 1 2 2 na
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance blade lean, untwist Fan blade running profile Shaft displacement b.5 Oil systems Liawid Deservers	Na Na Na Na Na Na	2 na na na na na na	2 na 1 2 2 na	2 na 2 2 na na na	2 na na na na na	Na Na Na Na Na	3 na 3 2 na na na	3 na na na na na	2 na na na na na	2 2 na na na 2	na na 1 2 2 na
b.4 Loads and Displacements Steady strain gauges Bearing load & crossover detection Disc axial movement Tip clearance blade lean, untwist Fan blade running profile Shaft displacement b.5 Oil systems Liquid Pressures Oil/Air Seal delta pressures	Na Na Na Na Na Na Na	2 na na na na na na na na	2 na 1 2 2 na na	2 na 2 2 na na na	2 na na na na na na na na	3 Na 3 Na Na Na Na Na	3 na 3 na na na na na na	3 na na na na na na na na	2 na na na na na na	2 2 na na na 2	na na na 1 2 2 na 7
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Summary of Capability Gaps											
a.1 Airflow Metering	2	2	1	1	2	2	2		1	2	1
a.2 Gas, Flame & Particle analysis		2	3	3	3	1		2	3		
a.3 Boundary Layer	1	2	2	3	3	3	3	3	3		2
a.4 Liquid Flow	3				3			3		2	1
a.5 Gas Path Instrumentation		1	2	2	3	3	3	3	2		2
a.6 Torque and Thrust	1									2	1
b.1 Aerodynamic Instabilities		1	1	2	3	3	3	3	2		1
b.2 Component Temperatures	1	1	3	3	3	3	3	3	3	3	2
b.3 Fatigue & vibration	1	1	2	2	3	3	3	3	3	1	1
b.4 Loads and Displacements	2	2	2	2	2	3	3	3	2	2	2
b.5 Oil systems	2		2	2						2	2
Key:	_										
1 - Capability OK											
2 – Some shortfall											
3 – Capability Gap											

Figure 11: Summarised capability gap matrix based on findings from the EVI-GTI [10]

After discussions with various Rolls Royce plc engineers and stakeholders, a number of desirable temperature measurement locations were identified. One of the most desirable locations for temperature measurement is the turbine section, which is an assembly of alternate static nozzle guide vanes (NGVs) and rotating discs that are mounted with blades via the fir tree root joints. A 3D and 2D cross section of the turbine section of the engine is shown in Figure 10, and examples of individual components are shown in Figure 12. The section is located directly after the combustion chamber and extracts thermal energy from the gases to rotate the blades and shafts to drive the compressor and other accessories.



HP turbine blade with and without thermal barrier coating

HP Nozzle Guide Vanes



HP blade post in fir tree joints on HP disc



Fir Tree Joints on the HP discs



HP Disc

Figure 12: Various turbine section engine component [32]

As stated earlier, the optimum operating temperature of the turbine is prescribed by the balance between the benefits of a higher temperature operation and material stability and life. High Pressure turbine blades and nozzle guide vanes (NGVs) operate in harsh conditions where the flow temperatures is above their melting point, and the use of thermal barrier coatings (TBCs) and internal and external cooling reduce the temperature of the substrate, reducing thermally driven failure mechanisms including thermal fatigue, increasing component life. TBC's on top of bond coats also protects the components from oxidation driven failures. Cooling is provided by air from the HP compressor through internal channel systems that flows the air though rows of holes to produce an exterior film of cool air, as shown in Figure 13. The amount of cooling depends on many factors including the choice of material, TBCs, engine performance requirements and cost. Cooling reduces cycle efficiency and induces aerodynamic losses by reintroducing air through the holes. This increases aerodynamic design complexity, and restricts the freedom of aerofoil design due to structural and manufacturing constraints. As cooling holes must be cast, it also increases manufacturing complexity and cost.



Figure 13: Complex internal cooling channels and holes for HP turbine blade and guide vanes. [13]

TBCs alone provide no benefit in reducing metal temperatures on uncooled components. As cooling reduces cycle efficiency an increases the chances of  $NO_x$  production, it should be minimised. Advances in metallurgy and casting technologies have enabled the use of single crystal alloys, allowing components to be operated at

higher temperatures, reducing the cooling requirements. Nickel alloys have high temperature creep resistance and strength retention, provide better resistance to oxidation, and are commonly used for turbine blades and NGVs. Single crystal components offer superior metallurgical properties at greater costs, and therefore material selection is dependant on the balance between temperature, life and cost. Since the HP turbine blades and HP-NGVs operate in harsher environments, the design complexity and demand for accurate temperature measurement for design optimisation from a designer's viewpoint is higher than that for Intermediate Pressure (IP) and Low Pressure (LP) turbine components where there is a gradual reduction in temperature and pressure as power is extracted from the gas flow, as shown in

Figure 14. As IP and LP turbine blades are operated at lower temperatures, they would not require as much cooling or design sophistication; LP blades are designed to run un-cooled. However, as they are made using lower temperature capability alloys to lower costs, the design complexity and the demand for accurate temperature measurement can remain high. A summary of the relative importance of key factors is shown in Figure 15.



Position in turbine section

Figure 14: Variation of pressure and temperature across the turbine section of the aeroengine [13]



Figure 15: Relative importance of factors for high pressure, intermediate pressure, and low pressure turbine blade sections in gas turbines

Modern civil aeroengine turbines are expected to operate at 13,000 RPM. Using the blade and disc geometries illustrated in Figure 10, the calculated linear velocities for HP turbine blades range from 0.4 to 0.52  $mm/\mu s$  from root to tip, which equates to a blade tip speed in excess of 1000mph. Modern Civil aeroengine IP and LP turbine blades usually run on separate shafts and may possess different rotational speeds. However, if the same engine rotation speed as HP turbine blades is assumed, the linear velocities of IP and LP blades are expected to be higher due to their increased radial positions within the engine. In existing development engines (Trent 900, 1000), the HP Turbine blades and NGVs currently experience surface temperatures in the region of around 800-1300°C, and future engine development over the next decade anticipates temperatures reaching 1400°C. The component linear velocities, optical access and distance to target are important consideration factors for optical instrumentation design, and can indicate the available time period for a probe to deliver and collect emissions from a target, and would ultimately place a limit on the temperature that would be detectable using phosphor thermometry. This key information is shown in Table 4 for a variety of components that were selected for

phosphor thermometry research. Due to practical reasons and time and cost constraints, the decision was made to only consider the most desirable engine components. The relative importance for temperature measurement on stationary engine components, such as nozzle guide vanes and turbine housings, is less than that for rotating components, because there are fewer complications in design and thermal data is more easily obtainable using thermocouples without requiring expensive telemetry. An imaging system to provide thermal maps would be a more suitable for further research for stationary components. As thermal data for stationary components, hold a lesser need, rotating components were given research priority. After analysing various options and discussions with various engineers, the most design sophistication, is the HP turbine blades. Other components that were also considered were the HP compressor drive cone. These two rotating components were given the design priority for research.



Figure 16: High pressure drive cone [32]

Engine Component	Crude Geometry of component	Linear Velocities	Current and Future component temp range	Gas path Temp	Access consideration, additional notes
HP- Nozzle Guide Vanes (HP-NGV)	A single NGV has 2 stationary inner blades. Outer: 180x90mm Inner: 130x60mm, Inner Blade: 90x60mm	Stationary	Current: 850-1300 °C Future: 1400 °C	1550 °C - 1900 °C	
HP Turbine Blade (HP-TB) At Root, At Tip	Actual Blade: 100 x 45mm Radial distance to blade root: 295mm Radial distance to blade tip: 385mm	Current: 13,000 RPM Future - 14,000 RPM Linear velocities: Root: 0.4mm/µs Tip: 0.52mm/µs	Current: 850-1300 °C Future: 1400 °C	1550 °C - 1900 °C	Maximum permitted probe diameter: 10mm. Existing pyrometer probes have 5mm diam. lens. Throw – probe to target distance 90mm. 120mm with purge air requirements
HP Disc, HP Disc Post / Firtree Joint and Disc Rims/ Bucket groove	Starting radius: 90mm Outer radius excluding fir-tree joints: 235mm Outer radius including fir-tree joints: 280mm	Speed: 13000RPM Linear velocities at mid point of fir tree: 0.35mm/µs	Inner disc section: 700 °C (max) Disc Posts and fir tree joints: 750 °C (max)		Very limited access. A cover-plate is preventing optical access. Future designs suggest that cover-plate and rim plates will remain.
IP - NGV		Stationary	Current: 800-1150 °C Future: 1250		Similar to HP NGV
IP – TB	Disc radial positions: 330mm (start) 350nm (outer) Blade radial positions : 365mm (root) 475mm (tip)	Linear velocities range from 0.5- 0.65mm/µs	Current: 800-1150 °C Future: 1250 °C		Similar to HP TB
LP1NGV		Stationary	Current: 900-1050 °C		Similar to HP NGV
LP1TB	Blade radial positions : 360mm (root) 535mm (tip)	Linear velocities 0.5-0.73 mm/µs	Current: 800- 1020 °C		Similar to HP TB
LP2NGV		Stationary	750-1000 °C		Similar to HP NGV
LP2TB Combustion chamber,	Annular design	Stationary	250- 950 °C Surface temperatures on top of ceramic tiles: 1300 °C	600- 2100 °C. Mean approx 1700 °C	Similar to HP TB High background radiation. outside surface ceramic tiles- PVD TBC
HP Compressor Drive cone	External cone radius approx: 210mm to 150mm	13,000 RPM Linear Velocity Upstream: 0.285 mm/µs Downstream: 0.2 mm/µs	Heat by compressed air + heat from the combustion chamber can reach up to 700 °C 600-825 °C		Limited access. A small diameter (4mm) flexible probe e.g. Alstom pyrometer probe/similar design may be used. Probe- to-target throw: dependant on position. 40, 50, 90mm (assume 50mm)

Table 4: Temperature range of various engine components

## **3** Luminescence Thermometry

## 3.1 Introduction

The section starts off with a brief history of luminescence and phosphor thermometry and introduces the fundamental physics of luminescence, later specialising into the luminescence in phosphors. It will attempt to explain various responses that change with temperature, giving phosphors their sensing properties. It will start off with the Jablonski diagram which explains luminescence in general, and later moves on to the configurational coordinate diagram and the charge transfer curve model that aids in the understanding of the thermal properties of thermographic phosphors. The generic phosphor system and the current state of the art, including apparatus and different response modes and techniques are discussed.

## 3.2 Historical Context

Luminescence is created from sources apart from heat and is distinct from incandescence and blackbody radiation, or other effects that cause materials to glow at high temperatures. This phenomenon has been observed and reported throughout history. Early Indian and Chinese scriptures dating prior to 1,500 BC refer to light emission from fireflies and glow worms. Aristotle in the fourth century BC observed luminescence from bacteria, fungus and fish and reported the distinction from incandescence: "some things, though they are not in their nature fire, nor any species of fire, yet seem to produce light" [35-37]. Monardes, in the 16<sup>th</sup> century, observed blue emissions from a wood extract and great scientists including Boyle and Newton tried to explain its occurrence; however, it was Stokes who successfully explained this phenomenon as luminescence in 1852 [37].

Alchemists were the first to synthesize luminescent materials, mainly by accident in their attempts to make gold [35, 37]. In 1603, Cascariolo created a material that glowed purple at night having been exposed to sunlight during the day. Later, Galla in 1612 wrote the first publication on synthetic luminescent material [35, 37]. Another publication in 1640 termed the word "phosphor" to mean any 'microcrystalline solid luminescent material' [37]. Wiedemann introduced the term "luminescence" in 1888 to include all light emission including both fluorescence and phosphorescence [38]. The two terms are still open for discussion. Earlier literature refers to phosphorescence for emissions with lifetimes >  $10^{-3}$  s, whereas recent literature suggests lifetimes >  $10^{-8}$  s.

Phosphors have wide range of applications including cathode ray tubes, plasma displays, light bulbs and x-ray conversion screens. During the  $18^{th}$  and  $19^{th}$  centuries,

A. Khalid

phosphors were mainly used for detecting invisible particles (UV photons, cathode rays, x-rays and alpha particles) [37]. During this time, with many concurrent advances in other scientific fields such as vacuum science, ceramics, glass working, and electromagnetism, Braun introduced the idea of the cathode ray tube in 1897 and won the Nobel Prize in Physics in 1909 for his contributions [39]. After the introduction of the fluorescent lamp by GEC in 1938, the demand for efficient lighting increased. The need for better CRTs and efficient lighting accelerated research into properties of phosphors and luminescence. During the 19<sup>th</sup> century, Lenard and co workers synthesised phosphors by firing impurities that formed luminescent centres in the host [40]. Pohl and Sietz introduced the configurational coordinate model of luminescence centres, establishing the basis of modern-day luminescence physics [40]. Another figure that helped us understand luminescence is Jablonski whose work resulted in the Jablonski energy diagram, a tool that can be used to explain the kinetics and spectra of fluorescence, phosphorescence, and delayed fluorescence. Other key figures include Condon, Fonger and Struck, who have helped us to understand certain thermal properties.

Luminescence involves the promotion of electrons to higher energy states with subsequent emissions of light. The idea of emission analysis for sensing applications is not new. For the case of pressure measurements, the Stern-Volmer relationship between emission intensity and air pressure dates back to as early as 1919. The capture and analysis of luminescence emission profiles is rapidly becoming an important tool in sensing technology and is now very common for biomedical and aerospace applications for detecting oxygen levels, pressure, temperature and for fluorescence microscopy. The idea of using phosphors for temperature measurement was first cited in 1937 [41] during the development of the fluorescent lamp where a loss of luminescence was observed with increasing temperature. Although Neubert suggested the idea, it was not until the early 1950's when Urbach and Bradley, cited in Allison and Gillies [42], first utilised phosphors to obtain temperature distributions on a flat wedge. Czysz and Dixson carried out tests producing thermal maps of models in wind tunnels [43, 44]. By applying a phosphor at the tip of an optical fibre, Wickersheim and co-workers investigated many phosphors with a variety of applications and their work led to the commercialisation of fluorescence-based thermometry systems [45]. Grattan and associates also investigated a variety of fibre tip thermometry systems based on a variety of phosphors. Cates [46] and co-workers developed remote measurement systems by adhering a layer of phosphor onto a surface of interest rather than at the end of a fibre [47], offering the system greater flexibility and allowing remote measurements of moving surfaces.

Phosphor thermometry within turbine engines dates back at least 20 years [48], and a number of relevant studies have been undertaken since [8, 49-52]. There has also been some work by Bird at Rolls-Royce Plc in conjunction with Bradford University, and Nottingham Trent University, directly targeting this area. This technology has attributes that make it better suited than existing methodologies in many applications. Thermographic phosphors are accurate, and like pyrometry, share many of the nonintrusive benefits over thermocouples (e.g. routing of cables for rotating components and immunity to electromagnetic noise.), whilst overcoming many of the shortfalls and issues related to it (e.g. emissivity variations, flame interferences and reflected radiations). For these reasons, phosphor thermometry systems have also been demonstrated to be successful in a number of other applications ranging from measurements of internal combustion engines[53], burning materials and pyrolysis studies[54, 55], to supersonic[56] and hypersonic aerothermodynamics experiments[57-59]. An excellent review with a strong theoretical background to phosphor thermometry is provided by Allison and Gillies [42]. Khalid and Kontis [60] recently reviewed developments in the past 15 years.

## 3.3 Principles of Luminescence

## 3.3.1 Generic Luminescence Behaviour

Luminescent processes are governed by a few important events that occur on timescales that are orders of magnitude apart. In general, excitation causes the energy of luminescent molecules to jump to higher electronic states. This configuration state is not permanent; vibrational relaxation, internal conversion, intersystem crossing and emissions soon follow, resulting in the excited state returning back to the ground or an intermediate state. This process can be neatly summarised with a Jablonski energy-level diagram (Figure 17). Phosphors (or other luminescent materials) are thermographic if they exhibit changes in luminescent characteristics with temperature.

For any particular molecule, several electronic states exist. There are a combination of different available orbits (singlet states –  $S_0$ ,  $S_1$ ,  $S_2$ , ) and spin orientations (triplet/intermediate states –  $T_1$ ,  $T_2$ ), represented by thick lines, that are further divided into a number of vibrational and rotational energy levels, represented by the thinner lines in Figure 17.



Figure 17: Jablonski energy level diagram showing the luminescence process.

Excitation (e.g.  $S_0$  to  $S_1$ ,  $S_2$ ) involves the absorption of sufficient energy to raise a molecule's electrons into electronic states of  $S_1$  or  $S_2$ . This molecule does not remain excited continually. According to Bell *et al.* [61], the ground state ( $S_0$ ) is the only stable state with all other states decaying back to this state. Complying with the conversation of energy principle, the amount of energy absorbed must be released. This happens via:

- emissions of photons equal to the energy-level difference
- energy transfer via quantised vibrational exchange (phonons) in the material
- other complex energy transfer mechanisms [42].

These energy transfers are further detailed as follows, with typical timescales summarised in Table 5.

*Vibrational Relaxation:* Absorption can cause molecules to be excited into higher vibrational states within an excited electronic state (for example  $S_1^{\text{level 3}}$ ); in this case, the most likely transition will be the relaxation to the lowest vibrational energy level ( $S_1^{\text{level 0}}$ ). This can be seen as vibrations occurring in the crystal lattice, sometimes referred as the emission of phonons in quantum physical terms, so that energy is lost as heat [51].

*Internal conversion:* The lowest vibrational level from a excited state can be converted to the highest vibrational energy state of a lower electronic state (for example  $S_2$  level 0

can turn into  $S_1$  <sup>level 5</sup>) This usually occurs when two electronic energy levels are sufficiently close. According to Bell [61], internal conversion results in vibrational relaxation with energy eventually being lost as heat.

*Fluorescence:* This radiative transition from an excited state is accomplished by the emission of a photon. This is generally proceeded from a state of thermal equilibrium to various vibrational levels The emission wavelength, calculated by Planck's equation  $(dE = hc/\lambda)$ , is found to be less than the excitation wavelength due to energy level differences, resulting in emissions of longer wavelengths (Stokes shift).

*Quenching:* There are several non-radiative relaxation processes/transitions that compete with radiative processes. One such transition is quenching. This occurs when energy is transferred to another nearby molecule. Oxygen is an effective quencher. The probability of occurrence is dependent on the quenching substance and concentration. By increasing the probability of quenching, the probability of radiative emission (luminescence) will decrease. This principle forms the basis of oxygen and pressure sensitive paints [61].

Intersystem crossing: This is a transition from  $S_1$  to  $T_1$ . Intersystem transitions require changes in electron spin and generally have an extremely low probability of occurrence. According to Turro [62], molecular structure and higher atomic size increases this probability; therefore, molecules containing heavy atoms (e.g. transitional metals) often facilitate intersystem crossing, making these as common as internal conversions. Many efficient phosphors originate from a deliberately added impurity [42]. At this point if the molecule has not returned to its ground state, further possibilities may occur:

- *Phosphorescence* transition to  $S_0$ . This process is orders of magnitude slower than fluorescence. The energy level of  $T_1$  is lower than that of  $S_1$  and therefore the emission wavelength of phosphorescence is higher than that of fluorescence.
- Intersystem crossing from T<sub>1</sub> to S<sub>0</sub>
- Quenching and other non-radiative transitions
- Delayed Florescence This is when there is an intersystem transition back to  $S_1$ . At this point, the entire process of relaxation back to the ground state starts again. If fluorescence occurs after this (from  $S_1$  to  $S_0$ ), this is known as 'delayed florescence'. This has the spectrum of fluorescence but the time of phosphorescence.

From the description, one may think that every atom has the potential to exhibit luminescence; according to Sant and Merienne [6] practically all existing materials are luminescent. However, luminescent behaviour depends on relative probabilities of alternatives processes by which excited atoms can return to ground state. According to Heyes [51] the persistence of phosphorescence implies that electrons occupy excited energy levels for extended periods. This allows interactions between excited atoms and the surroundings to have an influence on the nature of the emission. Some influences are thermally driven, making them sensitive to temperature.

Transition Example	Process	Rate	Typical Timescale
$S_{\text{o}} \rightarrow S_{1}$	Excitation, Absorption	k(e)	Femtoseconds, 10 <sup>-15</sup> s
	Internal Conversion	k(ic)	Picoseconds, 10 <sup>-12</sup> s
	Vibrational Relaxation	k(vr)	Picoseconds, 10 <sup>-12</sup> s
$S_1 \rightarrow S_0$ (radiative)	Florescence	k(f)	Typically less than 10 <sup>-8</sup> s
$S_1 \rightarrow S_0$ (non	Quenching and other non	k(nr),	10 <sup>-7</sup> – 10 <sup>-5</sup> s
radiative)	radiative processes	k(q)	
$S_1 \rightarrow T_1$	Intersystem Crossing	k(pt)	10 <sup>-10</sup> - 10 <sup>-8</sup> s
$T_1 \to S_0$	Phosphorescence	k(p)	10 <sup>-3</sup> – 100 s (earlier literature)
			$> 10^{-8}$ s (recent literature)

Table 5: Summary of typical process times from excitation to emission

The Jablonski model is useful for understanding luminescence in general, and is sufficient to explain oxygen quenching behaviour for pressure sensitive paints (PSPs). However, to understand thermal behaviour, the chemical nature of the phosphor and the understanding of the configuration coordinate diagram is necessary.

## 3.3.2 Luminescence in Phosphors

Phosphors are usually white in appearance and can take a number of forms consisting of a host material doped with activator atoms, usually rare earth (lanthanides) ions or transition metals (Figure 18), seen in Table 6. Other luminescence ions include antinides, heavy metals, electron-holes and ZnS-type semiconductors. Most phosphors used for high temperature applications consist of rare-earth ions in ceramic hosts. Examples include YAG:Dy and  $Y_2O_3$ :Eu.

Example hosts include:

- Yttrium garnets e.g. Y<sub>3</sub>(Al,Ga)<sub>5</sub>0<sub>12</sub>, YAG
- Yttrium oxides e.g. Y<sub>2</sub>O<sub>3</sub>
- Oxysulfides e.g. La<sub>2</sub>O<sub>2</sub>S, Gd<sub>2</sub>O<sub>2</sub>S, Y<sub>2</sub>O<sub>2</sub>S
- Vanadates e.g. VO<sub>3</sub>, VO<sub>4</sub>, V<sub>2</sub>O<sub>7</sub>
- Yttrium/Lutetium phosphates e.g. YPO<sub>4</sub>, LuPO<sub>4</sub>
- Others include: Al<sub>2</sub>O<sub>3</sub>, ZnS:Ag:Cl, LiGdF<sub>4</sub>, BeAl<sub>2</sub>O<sub>4</sub>



Figure 18: Chemical composition of thermographic phosphors

a) Lanthanides (rare earth ions)			b) Transition Metals					
Ce	Cerium	Sc	Scandium	Cd	Cadmium			
Pr	Praseodymium	Ti	Titanium	Hf	Hafnium			
Nd	Neodymium	V	Vanadium	Та	Tantalum			
Pm	Promethium	Cr	Chromium	W	Tungsten			
Sm	Samarium	Mn	Manganese	Re	Rhenium			
Eu	Europium	Fe	Iron	Os	Osmium			
Gd	Gadolinium	Co	Cobalt	Ir	Iridium			
Tb	Terbium	Ni	Nickel	Pt	Platinum			
Dy	Dysprosium	Cu	Copper	Au	Gold			
Но	Holmium	Zn	Zinc	Hg	Mercury			
Er	Erbuim	Y	Yttrium	Rf	Rutherfordium			
Tm	Thulium	Zr	Zirconium	Db	Dubnium			
Yb	Ytterbium	Nb	Niobium	Gg	Seaborgium			
Lu	Lutetium	Мо	Molybdenum	Bh	Bohrium			
		Tc	Technetium	Hs	Hassium			
		Ru	Ruthenium	Mt	Meitnerium			
		Rh	Rhodium	Uun	Ununnilium			
		Pd	Palladium	Uuu	Unununium			
		Ag	Silver	Uub	Ununbium			

Table 6: Elements in the a) lanthanide series; b) transition metals series.

Lanthanide ions, found in the 6<sup>th</sup> period of the periodic table, are characterised by an incomplete 4*f* shell that is shielded from the effects of the crystal lattice by outer filled shells. Therefore, when ions are mixed into a host lattice in low concentrations they are considered isolated [42] and treated as free ions [51] Although this is the case, according to Heyes [51], the host lattice has a profound effect on the thermal response of the phosphor. The influence on the absorption and emission processes can be explained with the aid of a configurational coordinate diagram (Figure 19). The environment of a luminescent centre is not static and the diagram shows the potential energy curves as the function of the configuration coordinate (deviation from the equilibrium). Although the model is very simplistic and the shapes of the curves are not parabolic in reality, it shares many features of the Jablonski diagram which illustrates several physical phenomena including Stokes Shift. In addition, it can also illustrate:

- Absorption and emission band widths
- Understanding of thermal quenching

Energy potentials and vibrational energy levels are represented by horizontal lines; and absorption and emission transitions are indicated by vertical lines. After excitation (A to B), electrons occupying an upper vibrational level of an excited state (point B) will relax to the ground vibrational level of that state (point C) losing energy via the release of phonons (B-C) [51]. The electrons will reach a higher vibrational level of the ground state (D) via radiative emission (C to D), and will continue to lose energy (phonons) on their return to their ground state equilibrium (D to A). The difference in excitation and emission energy levels can be seen illustrating Stokes Shift. The Frank-Condon principle states that electronic state transition times are much shorter than vibrational relaxation and are therefore assumed to occur in static conditions. Based on this, excitation occurs to vibrationally excited levels of the excited electronic state. According to Royer [63], emissions occur from the lowest vibrational level of the excited state, because relaxation from excited vibrational states is much faster than emission.



Configurational Coordinate

Figure 19. Configuration co-ordinate diagram.

According to Heyes [51], at temperatures above 0 K, electrons are distributed over different vibrational levels according to the Boltzmann's law, shown in Equation 1.

$$n_{excited} = n_{ground} \left( \frac{\Delta E}{kT} \right)$$

Equation 1

where 'n' is the electron population at a given state; 'E' is the energy difference between these states; 'k' is the Boltzmann constant and 'T' is the temperature.

If the temperature is high enough, electrons in the excited state can intersect the ground state curve (point E) allowing vibrational relaxation via phonon release to the ground state without any radiative emission. Ranson [64] describes this as the absorption of thermal energy (phonon) from point C, which excites the electrons to the intersection point E. Since non-radiative processes can now also compete with the radiative processes, the observed luminescence intensity from a large quantity of excited ions will diminish, explaining the thermal quenching. Also as the temperature is elevated, electrons are spread over a number of vibrational levels in the excited state. Since radiative transitions that can take place between any of the vibrational states in the excited and ground states, a broadening of the of the emission lines is expected [51].

Photo excitation alone can sometimes promote electrons into high vibrational levels at points beyond the intersection point (E) which results in a purely non-radiative emission, with no luminescence being observed. In some cases this may explain why higher energy photons (lower wavelengths) can actually dampen observed luminescence.

A further development to the configurational coordinate model that explains quenching behaviour in different host materials is proposed by Fonger and Struck [65]. According to these authors, the outer crystal field, which is dependent on the chosen host, has another energy potential (charge transfer state) curve that can be added onto the existing configuration coordinate diagram, as shown in Figure 20. With this inclusion, excited electrons can also return to the ground state via the charge transfer (CT) curve. Suppose an excited electron reaches an excited state of E3; it would normally return to the ground state by radiative emission. However, if the electrons are further excited by elevated temperatures (thermal activation), the electrons can intersect the crossover point of the CT curve, enabling the transfer of electrons to a lower energy level of E2 without any radiative emission. Likewise, electrons in E2 or E1 states can also transfer their energy to the ground state in the same way. Different hosts will have different CT curves in different places, explaining the variation in behaviour from different hosts.



Configurational Coordinate

Figure 20: Configuration co-ordinate diagram showing the effect from the charge transfer state (CTS) curve.

## 3.4 Different response modes

## 3.4.1 Introduction

There exist numerous response modes, illustrated in Figure 21 in which phosphors can be calibrated to reveal temperature. These are all the response modes that are reported in the literature.



Figure 21: Different response modes for thermographic phosphors

#### 3.4.2 Intensity Mode

When a continuous light source is used to excite the phosphor, electron populations are constantly being promoted to higher states and returning back to their ground states via radiative emissions. An equilibrium level is reached indicated by a steady emission intensity level. Elevation of temperature increases the probability of deactivation via non-radiative processes which is observable by a reduction in intensity. Many authors investigating the variation of intensity for various phosphors and their emissions lines have confirmed this to be true for most cases. An example of this for  $La_2O_2S$ :Eu phosphor is shown in Figure 22 [42]. Although the intensity of most emission lines decrease with temperature, there are some cases where there is an increase. This may be due to increases in absorption energy at that temperature.



Figure 22: Variation of emission intensity with increasing temperature. Taken from [42]

The advantage of intensity based approaches is that 2D surface measurements can be easily made using CCD/CMOS cameras, with each pixel serving as a separate sensor. A common problem with intensity based techniques is that the observed intensity is also a function of other variables, and if they are not taken into account, large errors can remain. Examples of such factors include: non-homogenous illumination, light source instabilities, phosphor coating thickness and densities, distance and detector viewing angle, surface curvature, reflections and shadings. These problems are well documented, especially in literature relating to PSPs. Researchers have attempted to provide corrections by reference imaging and other mathematical means [66]. However, a better intensity approach that eliminates many of these issues is the intensity-ratio approach.

### 3.4.3 Intensity Ratio

The intensity ratio mode relies on taking a ratio of two emission lines. This eliminates a number of errors that are common in the standard intensity approach. In pressuresensitive-paint (PSP) literature, standard PSPs are added with pressure insensitive reference dyes to make binary paints. The insensitive dye acts as an intensity monitor. Bell *et al.* [61] reports this technique to be the most successful approach for illumination correction. The methodology can also be applied to thermographic phosphors. Some phosphors exhibit a multiple emission response with some emission lines being insensitive/less sensitive to temperature. Ideally, the intensity of one of the emission lines should be independent of temperature. Figure 23 shows an ideal intensity variation of the two emission lines with temperature. It is important that the reference can be excited with the same wavelength with emissions at different wavelengths to enable easy differentiation.



Figure 23: Ideal intensity variations for the intensity ratio response.

For low temperatures Chyu and Bizzak calibrated the intensity-ratio for La<sub>2</sub>O<sub>2</sub>S:Eu to make 2D surface heat transfer measurements on a hot jet impinging on a circular plate [67, 68]. The system reported a range of 292K-333K with an accuracy of 0.5K and repeatability of 0.15K. The cooling effectiveness was also determined from a row of cooling holes [69]. Until recently, dysprosium was the only known rare-earth activator to exhibit an intensity ratio response at high temperatures. Fiest and Heyes [70] showed similar response with samarium-doped phosphors. The main mechanism behind this phenomenon is thermailisation [71]. When two energy levels are closely separated by a difference of approximately 1,000 cm<sup>-1</sup>, the upper level will not fluorescence at low temperatures due to high multi-photon relaxation that quenches the energy. As the temperature increases, the upper level becomes more populated

and hence the fluorescence from this level gradually increases. Figure 24 illustrates the similarities between the energy diagram of free Dy and Sm ions. The diagram is only indicative of the physical principles, and in reality there will be host interactions that resulting in variations in the energy levels which could lead to energy splitting, line broadening and shifting [70]. Figure 24 illustrates the emission spectra of YAG:Dy and Y2O2S:Sm. For YAG:Dy, the absorption excites the dysprosium into an excited state which relaxes to the  ${}^{4}F_{9/2}$  level. This level undergoes fast thermal equilibrium and pumps a proportion of its population to the nearby  ${}^{4}I_{15/2}$  level. As the temperature increases, there is a gradual build-up of the population to this level, and radiative emissions from this level increase. However, above a certain temperature, luminescence slowly begins to decrease due to the charge transfer state (CTS) transitions [72]. The <sup>4</sup>F<sub>9/2</sub> level emission (496nm) almost stays constant with increasing temperature and can therefore be used as an internal reference allowing temperature to determined as a relative, rather than a absolute measurement eliminating significant sources of error [71].



Figure 24: Energy level diagram for free ions of Dy and Sm. Taken from [73] cited in [70]



Figure 25: Emission spectra at different temperatures. Left: YAG:Dy [71]; Right:  $Y_20_2S:Sm$  [70].

The intensity ratio technique using thermographic phosphors was first cited in Gross *et al.* [71] using YAG:Dy<sup>3</sup> who reported a temperature range of 300K-1,500K and an accuracy of  $\pm 9$  to  $\pm 50$ K. Kontis *et al.* [72], reported a similar system utilising two gated ICCD cameras. Temperature calibration was made between 295K-1,350K, with a reported accuracy and repeatability of  $\pm 2.5$ K and <0.3%. The system was used for thermal measurements on a ceramic plate exposed to an impinging jet flame [72], and surface heat transfer measurements in a supersonic combustor [74].

Heyes, Feist and Seedfeldt [75] investigated the intensity ratio for dysprosium using YAG and YSZ hosts. Temperature calibration was performed between 300-900K, with data repeatability around  $\pm 0.6\%$  [75]. The system was used for temperature measurement on ceramic and alloy plates that were heated by flame impingement. YSZ is currently used for making gas turbine thermal barrier coating; the tests demonstrated the capability of making 'smart TBCs' with instrumentation abilities. The same authors also investigated Y<sub>2</sub>O<sub>2</sub>S:Sm phosphors using the intensity ratio mode between 300-1,100K and showed an uncertainty of  $\pm 1\%$ ; they also tested the lifetime decay response mode from 900-1,425K which showed an uncertainty of  $\pm 1\%$  and 0.1% at higher temperatures [70]. Another phosphor that has been reported to exhibit a dual emission response and has been cited numerously in the past few years in a number of publications [54, 55, 76-81] is based on magnesium manganese phosphors. The drawback of intensity ratio response is that two separate detections are required. The conventional way to achieve this is by using two cameras with appropriate optical filters to detect the intensity of the desired wavelength. Another way to achieve this is by using a filter wheel. Table 7 compares these techniques.

	Two camera	Filter Wheel + Single camera
Schematic	Camera B Camera A	Camera
Signal capture	This system measures signals simultaneously.	This system measures both signals sequentially. Software is required to separate individual signals.
Alignment between images	Alignment is required. The variation in distance and angles can further induce errors. Separate cameras can have slightly different responses.	The same camera and its position can eliminate many errors caused by alignment and CCD defects.
Mechanics	No moving parts.	Reliable mechanical parts are required with good repeatability.
Other	Flat field correction is required	

 Table 7: Comparison of the `conventional two camera' approach and the `filter wheel approach' detection for the two-mode intensity method.

More recent approaches include the use of a cube beam splitter to ensure that the images are spatially identical. This approach was used by Kontis [72]. Stereoscopes with two apertures that allow two images to be independently filtered using a single camera, have also been used. This provides similar advantages to the 'filter wheel' approach with no moving parts. This approach was adopted by Heyes *et al.* [75] to image the dual emission ratio response of a YAG:Dy and YSZ:Dy. The system was later enhanced to also allow the simultaneous measurement of lifetime decay response, enabling the cross checking of temperature using the two methods. It also extended the dynamic range of measurement [82]. Similar two mode response systems have been reported by Omrane and Hasegawa [83].

## 3.4.4 Temporal Analysis

#### **Lifetime Decay Analysis**

Lifetime decay analysis is used in a number of scientific disciplines. It is an established method and is based on the decay mechanism of emission of fluorescent molecules. The temporal method eliminates many of the issues related with intensity based approaches, and is:

- Insensitive to non-uniform excitation
- Insensitive to dye concentrations/surface curvature/paint and thickness
- The approach can be used in high ambient light environments
- The system can also take into account photo-degradation [66].

Excitation promotes a large number of electrons into an excited state. When excitation is ceased, electrons return to their ground equilibrium level. For simplicity, this is either a radiative or non radiative transition. The rate of the electron population returning to the ground state can be expressed mathematically as:

Equation 2

$$\frac{dN}{dT} = \lambda N$$

with the solution yielding to

$$N_{o}e^{-\lambda i}$$

where N(t) is the quantity of electrons at a given time, N<sub>0</sub> is the initial quantity of excited electrons at t=0, and  $\lambda$  is the decay constant, the rate at which electrons make this transition. The mean lifetime ( $\tau$ ) of which an electron remains in the excited state can be determined from Equation 4.

Equation 3

4

$$au = rac{1}{\lambda}$$
 Equation

Since the two transition pathways (radiative and non-radiative) compete and are mutually exclusive, the decay constant can be written as the sum of the two possible rates of transitions. For simplicity, the analysis excludes the effects of interaction between activators, impurities in the host that can lead to further processes and change the simple exponential decay signature.

$$\lambda = k_r + k_{nr}$$
 Equation 5

The radiative rate  $(k_r)$  is a temperature independent term and can be considered as being a constant, whilst the non-radiative  $(k_{nr})$  transition becomes highly temperature dependant after the quenching temperature. For a given temperature, the probability of an single electron taking a transition pathway can be calculated from basic probability theory, resulting in:

Probability of radiative emissions: 
$$P_r = \frac{k_r}{k_r + k_{nr}}$$
 Equation 6

Probability of non-radiative emissions:  $P_{nr} = \frac{k_{nr}}{k_r + k_{nr}}$  Equation 7

If the temperature is increased, the decay rate via non-radiative means  $(k_{nr})$  also increases and has the following consequences highlighted in Table 8. In summary, the probability of radiative transition will decrease whilst the probability of non-radiative transition will increase.

Equation	Effect if the $k_{nr}$ value (or temperature) is increased
$\lambda = k_r + k_{nr}$ $\tau = \frac{1}{\lambda}$	$m{\lambda}$ (the decay rate constant) will be increased. Therefore, the decay lifetime of the transition will be decreased
$P_r = \frac{k_r}{k_r + k_{nr}}$	If the $k_{nr}$ term is increased, the probability of radiative transition will be decreased. If the temperature is very high, this probability will yield to zero. (impossible)
$P_{nr} = \frac{k_{nr}}{k_r + k_{nr}}$	If the $k_{\rm nr}$ term is increased, the probability of non-radiative transition will increase, yielding to 1 (certainty) at high temperatures.

Table 8: Effect of increasing temperature on the probability of radiative ( $P_r$ ) and non-radiative ( $P_{nr}$ ) decay.

By assuming the electron population is proportional to the observed luminescent intensity. The lifetime decay relation can be then represented as:
$$I(t) = I_o e^{-\frac{t}{\tau}}$$
 Equation 8

where  $I_o$  is the initial intensity at time t = 0, and  $\tau$  is the decay lifetime. Figure 26 illustrates typical lifetime characteristics with increasing temperature indicating faster decays with increasing temperature. The relation is only held after the quenching temperature. Researchers have also observed variation in intensity levels with temperature that is not shown in the figure. Figure 27 illustrates the decrease in lifetime decay with temperature for a range of phosphors. It also shows the quenching temperature for some of the phosphors.



Figure 26. Typical lifetime characteristics with increasing temperature.

Since the lifetime approach is independent of illumination energy, the problems associated with model deformation, movement, shading and uneven light distribution do not exist [66]. In terms of disadvantages, the lifetime method suffers a lack of signal strength [84] as the excitation light, in pulsed form, is only available for fractions of the time. To compensate for this, high-powered laser pulses are commonly used. However, increasing the pulse strength risks the destruction of the phosphor paint. Although, this is true for pressure sensitive paints; however, phosphors have much higher damage tolerances.



Figure 27: Lifetime of phosphors vs. temperatures. Taken from [42]

#### Lifetime Imaging

The principle drawback of measuring lifetime decay profiles in the past was instrumentation limitations only feasible to provide discrete spot measurements. The intensity method, despite its problems, was more attractive as 2D thermal maps could easily be obtained using CCD/CMOS imaging. Distribution maps using the lifetime approaches were built up using point measurements coupled with a XY scanning device. Examples include Davies [84] for pressure measurements, and by researchers at Imperial College [85] for temperature measurements in a laboratory combustion rig. A camera lens was used to focus the detection on a single point, and a traverse was used to successively scan an area of 8mm<sup>2</sup> with 128 point measurements. In recent years there have been many advances in imaging technologies making it practical for such temporal responses to be imaged to reveal temperature profiles over 2D surfaces. Lifetime imaging using CCD/CMOS cameras has seen considerable application in the biomedical industry, and was originally developed for oxygen detection[86]. This system was later modified for wind tunnel experiments [87], and the technique has been used intensively in the past few years using Mg₄FGeO<sub>6</sub>:Mn thermographic phosphors by researchers at Lund University who claim to be the first to obtain such 2D measurements using this approach [79]. A high speed framing camera, containing an eight faced prism was used to split light to eight independent intensified CCD cameras, where the exposure time and time separation between images could be precisely configured. This produced a complete 2D thermal map determined using lifetime decay analysis. Figure 28 shows an exponential curve fit for a single pixel derived from a series of images that were carefully triggered at different frames using a high speed camera.



Figure 28: Left: Architecture of the framing camera, showing the eight faced prism splitting the light equally to eight ICCD cameras [55]. Right: Curve fit for a single pixel from a series of images obtained from the separate detectors [79].

### **Frequency Domain Lifetime Decay**

It is possible to determine decay lifetimes in the frequency domain using a specimen excited by a continuous wave. The resulting wave will have a different amplitude and phase due to various time lags of certain luminescent processes. The advantage of this, opposed to a pulsing system, is that luminescent intensity is expected to be higher since the phosphor is being illuminated for 50% of the time. Figure 29 exemplifies the response for different lifetimes, indicating both changes in phase and amplitude. Phase lag is proportional to the lifetime and can be mathematically determined; an in-depth analysis can be found in Liu and Sullivan [66]. Burns and Sullivan [88] implemented this technique to map surface pressure measurements. Allison et al. [89] made temperature measurements using phosphors and blue LEDs.



Figure 29: Phase shifts for different lifetimes.

#### **Rise-time Analysis**

An investigation by Rhys-Williams and Fuller [90] noted that there are rise times associated with the response of thermographic phosphors. Their research showed that it was dependant on activator concentrations. The phosphor under investigation was  $Y_2O_3$ :Eu at room temperature. Ranson later analysed risetime characteristics in the late nineties and realised that it could be used for detecting temperature [91]. Ranson et al. [92], notes that the crystal structure of  $Y_2O_3$ :Eu has two sites of symmetry producing energy levels shown in Figure 30. They note the previous work of Heber et al. [93] who give evidence for three potential energy transfers (a,b and c) to level  $D_0$ . The energy transitions of paths 'a' and 'b' have been observed to be very fast compared to that of 'c' [94]. It is this transition that gives this phosphor the rise time characteristics.



Figure 30: Energy levels of  $Y_2O_3$ :Eu at symmetry sites  $C_2$  ad  $C_{3i}$ . Reconstructed from Ranson [64]

The emission of 611 nm (path d) follows the lifetime decay relation shown previously according to Equation 9,

$$N(t) = N_o e^{-\frac{t}{\tau^d}}$$
 Equation 9

where  $N_{0,}$  in this case, is the total number of electrons at  $D_0$ . This is not fixed and depends on the transition paths 'a', 'b' and 'c'. The fast transitions 'a' and 'b' can be modelled as being instantaneous; but the transition of 'c' is dependent on the decay of electrons from  $C_{3i}$  to  $D_0$  which decay at

$$N_{c3i}(t) = N_c e^{-\frac{t}{\tau^d}}$$
 Equation 10

Thus, the number of electrons accumulated from path 'c' as a function of time is:

$$N_c(t) = N_c - N_c e^{-\frac{t}{\tau^r}} = N_c \left(1 - e^{-\frac{t}{\tau^r}}\right)$$
 Equation 11

The total number of electrons at  $D_o$  is then:

$$N_0(t) = N_{ab} + N_c \left(1 - e^{-\frac{t}{\tau'}}\right)$$

Equation 12

Combining the equations yields the full characterisation of the decay, shown in Equation 13:

$$N(t) = \left[ N_{ab} + N_c \left( 1 - e^{-\frac{t}{\tau^r}} \right) \right] e^{-\frac{t}{\tau^d}}$$
Equation 13

where  $\tau_d$  = lifetime decay,  $\tau_r$  is the risetime, N<sub>ab</sub> and N<sub>c</sub> are the number of electrons by transitions a, b and c, respectively.

The investigations were carried out were carried out using  $Y_2O_3$ :Eu phosphor with approximately 3% Eu concentration. Previous investigations by Rhys-Williams and Fuller [90] noted that rise times ranged from 60  $\mu$ s at 5% mole concentration to 320  $\mu$ s at 0.27% mole concentration. Recent work by Allison *et al.* [95] underwent investigations at 0.5% Eu. The results shown in Figure 31 clearly demonstrate the effects of temperature on risetime, showing a noticeable decrease in risetime due to increasing temperatures. Another temperature related response is also shown; there is an increase in luminescence strength due to increasing temperatures; according to Allison *et al.* [95], this is due to increased phosphor absorption at the excitation wavelength (337 nm nitrogen laser).



Figure 31: Risetime variation with temperature. Taken from Allison et al. [95].

# 3.4.5 Line shift/width method

According to Gross *et al.* [71] temperature can cause the crystal lattice containing the rare-earth to vibrate creating a changing crystal field that produces a broadening of emission linewidths. Frequency shift of the spectral lines can also occur due to thermal expansion of the crystal lattice [71]. Both line shift variation and broadening can be calibrated to reveal temperature. However, these effects are usually small. The variation in the line shift at 1,000K is only 3 nm, making the temperature sensitivity very small and difficult to detect [51]. Kusama *et al.* [96] utilised this approach using  $Y_2O_2S$ :Eu phosphor for measuring temperature varying between -15°C and 72°C (Figure 32).



Figure 32: Emmsion lineshift and linewidth variation with temperature [42]

### 3.4.6 Absorption/Excitation band analysis

Various studies have shown a variation in the excitation and absorption band of some phosphors due to changes in temperature. When a nitrogen laser (337nm) or third harmonic Nd:YAG laser (355 nm) is used to excite a  $Y_2O_3$ :Eu phosphor, there is a gradual increase in the emission intensity with increasing temperature. According to Allison and Gillies [42], this is because the absorption, at these wavelengths, is weak at room temperatures and slowly increases with temperature. The absorption spectra for  $Y_2O_3$ :Eu at room temperature is shown in Figure 33, illustrating the weak absorption lines at 337 and 355 nm; Figure 34 illustrates the shift in absorption band due to increases in temperature.



Figure 33: The absorbtion spectra of Y<sub>2</sub>O<sub>3</sub>:Eu [64]



Figure 34: Left: Variation of absorption peak with temperature [42]

# 3.5 Other factors

# 3.5.1 Introduction

This section reviews other factors apart from temperature that can influence emissions from a phosphor.

## 3.5.2 Activator concentration

Activator concentration affects the shape and intensity of the emission.  $Y_2O_3$ :Eu concentrations less than 5% leads to strongest lines with narrowband wavelength [51] Greater concentrations leads to dispersion. It was found that reducing the dopant concentration increases the rise time for  $Y_2O_3$ :Eu phosphor[90], however this is not true for all phosphor and would require further research. With increasing

concentrations, the energy gap between lines is reduced so electrons reach lower levels from neighbouring ions by non-radiative means, indicated by a loss of radiative emissions. Allison and Gillies [42] notes that higher concentrations may alter the fluorescent decay so that it follows a multi-exponential rather than a simple exponential profile, making the lifetime more difficult to determine and prone to errors.

### 3.5.3 Particle Size

The decay lifetime and intensity of a phosphor changes with the size of phosphor particle. Investigations into nano-crystalline and coarse grain particles of  $Y_2O_3$ :Eu phosphors suggests that the excited state parabola on the configuration coordinate diagram may be affected. Konrad et al. [97], shown in Figure 35, explains that a there is a increasing slope of the excited parabola with reducing particle size. The implication of this is that the intersection point between the ground state is increased, therefore lifetime decays are expected to be higher with temperature quenching expected to occur at higher temperatures. Works by Christensen et al. [98], has shown an increase in lifetime (from 436-598<sub>µs</sub>) due to reductions in particle size (from 0.42 to 0.11<sub>µm</sub>). As different preparation and surface bonding techniques can alter sizes of particles, it seems reasonable to suggest that the decay lifetime method is not absolute, and it is important that calibration is performed for each phosphor



Figure 35: Effects of reducing the particle size [97]

# 3.5.4 Oxygen and Pressure

Pressure sensitive paints respond to both thermal and oxygen changes. Phosphors were originally thought to be insensitive to oxygen and pressure changes. However, recent investigations by Feist *et al.* [99] and Brubach *et al.* [100] suggest differently. Feist *et al.* [99] showed uncertainties due to changes in oxygen for  $Y_2O_3$ :Eu were an order of magnitude greater than those observed at fixed concentrations, providing a convincing case for oxygen quenching. In the investigation, volumetric percentage of oxygen was changed from 21% to 5% by flooding the furnace with nitrogen. A recent report by Brubach *et al.* [100] investigates the effects of various gas compositions. The results (Figure 36) show that different gas compositions do not influence the decay time of Mg<sub>4</sub>FGeO<sub>6</sub>:Mn and La<sub>2</sub>O<sub>2</sub>S:Eu phosphors, and are only influenced by thermal quenching. These phosphors would be suitable for environments where gas compositions are expected to change.  $Y_2O_3$ :Eu however showed high sensitivity to oxygen and different gas compositions.



Figure 36: Effects of different gases on the lifetime decay of different phosphors at different temperatures. Phosphors a)  $La_2O_2S:Eu \ b) Mg_4FGeO_6:Mn \ c) Y_2O_3:Eu \ [100]$ 

There is evidence that application of pressure/strain can affect luminescent properties of thermographic phosphor. This phenomenon is not very well understood but becomes relevant when extreme pressures are concerned. The application of pressure can be viewed as the imposition of compressive strain that can result in changes in chemical bonds and atomic level orbital configurations. The decay time of Gd<sub>2</sub>O<sub>2</sub>S:Tb decreased by an order of magnitude with application of 2GPa, while La<sub>2</sub>O<sub>2</sub>S:Eu increased by an order of magnitude with application to 3.5GPa [101]. Although some phosphors may not show oxygen sensitivity as in the case of La<sub>2</sub>O<sub>2</sub>S:Eu [100], they may show pressure sensitivity [101], and is important that both parameters are treated independently. Brubach et al. investigations showed no change in lifetime for La<sub>2</sub>O<sub>2</sub>S:Eu up to a pressure of 10 Bar (1MPa). However, Figure 37 illustrates a decrease in lifetime at higher pressure (0 - 50MPa). In very harsh flows, such as those experienced in gas turbine engines, the maximum pressure is around 50 Bar (5MPa), and the pressure effects may become relevant.



Figure 37: Variation in lifetime decay time of  $La_2O_2S$ :Eu phosphor with increasing pressure [101]

 $Y_2O_3$ :Eu showed sensitivity to oxygen and also showed irreversible changes after the absolute pressure was increased to 6 Bar. For these reasons,  $Y_2O_3$ :Eu, which has been a popular choice for phosphor thermometry in gas turbine engines, would be unsuitable for temperature measurement in such environments where the pressure and oxygen levels are changing.

# 3.6 Generic phosphor thermometry system

## 3.6.1 Introduction

The exact design of the phosphor thermometry system, in terms of phosphor choice, excitation and emission capture and measurement response methodology will depend

on the end application. There are many considerations factors that would need to be taken into account in the engine environment. The components of a generic phosphor thermometry system are illustrated in Figure 38. An excitation source, either a pulsed or continuous, dependant on the response methodology, is used to excite the phosphor that is bonded onto the surface of interest. Subsequent emissions are passed through an optical filter and are captured by a suitable detector (CCDs, PMTs). Data are stored, processed and compared to calibration curves to determine temperature. The entire system may be controlled by software that can enable synchronisation between detector, excitation source and the heat generating phenomena.



Figure 38: Generic layout for a thermographic phosphor system.

# 3.6.2 Emission Detection

This section compares typical characteristics and limitations of commercially available detectors that are used for point measurement and imaging. The findings and typical performance characteristics are shown in Table 9.

#### **Point Detection**

*Photomultiplier Tube (PMT):* The PMT has been the most widely used instrument for phosphor emission measurements. They are very sensitive and responsive, with typical rise and fall times in the 1ns regime. Photons strike a photo emissive cathode

which emits electrons that are collected at the anode. These electrons are then accelerated towards a series of additional electrodes (dynodes) that generate additional electrons. This cascading effect creates  $10^5$  to  $10^7$  electrons for each photon hitting the first cathode. The amplified signal is finally collected at the anode where it can be measured. PMTs have large detection areas and can offer a high gain and superior SNR compared to its competitors.

*Micro channel plate PMT:* A MCP-PMT contains an electron multiplier consisting of an array of millions of glass capillaries fused into a thin disk less than 1mm thick. The time between the generation of the primary emissive electron at the cathode and the arrival of the corresponding bunch of electrons at the anode is very small, with response times in the region of 100 picoseconds, making them around 10X faster than conventional PMTs. In the past MCPs were only available for the detection of UV, soft X-ray photons and neutrons. They have now been engineered for visible light detection [102].

Photodiodes: These feature excellent linearity with respect to incident light, have wide spectral response, are compact, mechanically rugged and have a long life. Response times typically vary from hundred nanoseconds to a few microseconds, making them slower than PMTs. However, recent developments enable them to operate at similar bandwidths. The signal generated by photodiodes is very small relative to noise inherent in the system, resulting in poor SNR, especially when they are operated at high bandwidths or low light levels. For low light levels, it is usual to increase the gain by increasing the feedback circuit resistor, which consequently lowers response speed and increases thermal noise [103]. A high-speed preamplifier can ensure a wide response speed and lower noise. Avalanche photodiodes have some intrinsic gain and offer lower noise characteristics than standard electronic amplification, making them more suited to lower light conditions [103]. As the shot noise is often higher; the SNR is not usually improved. However, for low-level detection, gain can be increased to improve SNR, whilst maintaining response speed, until shot noise reaches thermal noise level [103]. The quantum efficiency of these devices is much higher than that of PMT. However, as these detectors have much smaller detection areas, it is likely that more light will be lost in collection optics than gained by quantum efficiency.

*Si Photomultipliers (SPM):* These are relatively new solid-state devices and have received considerable amount of research over the past decade. Their performance is superior to that of standard and avalanche photodiodes in terms of sensitivity, and is approaching that of PMT detectors [104-106]. These detectors have a number of advantages over PMTs including: small size, low bias voltage operation, magnetic field

insensitivity, a higher degree of robustness and immunity to damage from high light condition overexposure.

	Standard Photo- diodes	APD	Conventional PMT	МСР-РМТ	Si- PMT (SPM, SiPM)
Gain	None	Low gain (x10-300)	High Gain (10 <sup>6</sup> )	High Gain (10 <sup>6</sup> ) Typical = 5 x10 <sup>5</sup>	High Gain (10 <sup>6</sup> ) Much higher gains than APDs. Same region as PMTs.
Bias V		High (800V)	High (kV)		Low (30V)
Sensitivity	Typically < 1 A/W	25 A/W @ 520 nm 30 A/W @ 1,064 nm	40,000 A/W @ 520 nm (SensL)[107] Sensitivity: 110 uA/lm Anode sens: 500 A/lm (nominal); 2,000 A/lm (max)	Very high Cathode sensitivity >1,200 uA/Im (min), 1,500 (typical) (Burle)[102]	60,000 A/W @ 520 nm (micro) 130,000 A/W @520 nm (mini) 1,000 A/W @ 1,064nm
Response to excess light	Some PIN damaged	Some APDs are damaged	Damage		Tolerant
Area	Small	Small	Large diameters- e.g. 46mm. Arrays are impossible		Small (1x1 mm <sup>2</sup> ) large areas up to 9mm <sup>2</sup> available. Building larger arrays is possible.
Rise/Fall Times - response	T <sub>rise</sub> = 0.1 microseconds	Can be operated at 2,000 MHz. (therefore ns)	Rise time: 1 ns.	Faster than PMTs. 100 picoseconds	<5ns
Quantum Efficiencies	Higher than PMTs	Higher than PMTs	20-30% at peak	>20% at peak	40% @ 520 nm at peak
SNR (signal to noise)	Low	Low.	High PMTs offer a higher gain, larger detection area and superior SNR compared to APDs		High – SNR to be similar, and in some cases better than PMTs.
Other notes	Robust	Robust	Fragile, affected by magnetic, electromagnetic interference.	Excellent for pulsed light.	Robust

Table 9: Comparison of different light detectors

#### Imaging

#### CCD-Charge-Coupled Devices and CMOS Imagers

CCDs contain photosensitive elements that convert photons into charge. Their quantum efficiency can be as large as 90% for back illuminated devices. The full-well capacity indicates the upper limit can be detected before electrons spill into neighbouring pixels, smearing the image. There are a number of different types of CCDs (including full frame, and interline transfer architectures) each with its own advantages and limitations that go beyond the scope of this thesis. More information can be found in [103]. Phosphor thermometry using intensity-based methods using continuous illumination, where fast transfers are not required, is relatively straightforward. However, fast transfers are required for unsteady cases, or when capturing decays for temporal based approaches. CCDs contain vertical and horizontal registers and an output section. The time to read the charge off the CCD can be approximated by clocking speeds (10-50 million pixels per second). As noise is proportional to clocking speeds, full frame transfer (FFT) devices are optimised for low noise operation by slowing the scan rate.

*CMOS Imagers:* Unlike CCDs, each pixel has its own integrated amplifier. Where CCDs pixels always transfer charge, CMOS pixels converts this to a voltage, enabling faster clocking speeds and hence frame rates. As there are readout transistors at every pixel, the active pixel area (fill factor) for CMOS imagers is reduced. CCDs have 5-10X the sensitivity of CMOS cameras, making them more suited for faint/low light conditions. CMOS imagers compensate for this by taking longer exposures, and stacking more frames. Binning is also often used to increase sensitivity by combing charge from adjacent pixels at the expense of spatial resolution. The relative advantages /disadvantages are described in Figure 39.

Advantages of CMOS	Disadvantages
Standard fabrication is	The fill factor refers to the
lower - on chip integration	percentage that is sensitive to light. This is lower in
Low power consumption	CMOS devices
Random access to pixel regions of interest. Adds flexibility	The noise is often more than CCD devices.
Intra pixel amplification and on chip conversion means faster frame rates.	

	CMOS	CCD
Windowing	Yes	No
Power	Low	High
Integration	High	Low
Speed	High	Low
Blooming	No	Yes
Cost	Low	High
Fill factor	Mid/Low	High
Noise	Mid/High	Low

Figure 39: Comparison of CCD and CMOS cameras.

*Multi-port/Multi-gate CCDs:* These devices use multiple amplifiers for parallel readouts that can significantly improve frame rates, and like CMOS cameras frame rates up to 10 KHz can be achieved.

*Intensified CCD (ICCD):* ICCDs utilise an image intensifier coupled to a CCD. They offer high sensitivity in ultra-low-light-level conditions. Since the intensity is increased, the exposure time can be reduced and gating methods can be utilized to provide better temporal resolution. These cameras are also suited for lifetime imaging.

*Time delay integration (TDI):* This is an effective method for imaging moving objects. Normally, the object must stay fixed during the integration time. If an object is moving, the image can become smeary. The TDI CCD has rows of pixels with charge transfers that are synchronized with the speed of the moving object. This technique allows clear imaging of objects moving at line rates up to 100kHz [103]. Future improvements of this technique may prove useful for applications such as high speed turbine blade imaging.

*Digital APDs/Photon Imagers:* Si-Photomultiplier technology has been combined with CMOS technology to form a new generation of low light cameras currently under development [107]. These systems will have the capability to photon count at the pixel level, making them very sensitive. This could allow for exposure times to be reduced, allowing the capture at finer timescales.

### 3.6.3 Excitation Sources

Excitation energy can be supplied to phosphors by a variety of ways, including electromagnetic radiation (lasers and LEDs), particle beams (electrons, neutron, ions) and electrical current. This section only reviews electromagnetic radiation, focusing mainly on UV lasers.

#### **Pulsed Laser Systems**

**Q** switched Nd:YAG Laser Systems: From the literature, most researchers have used nitrogen lasers (337nm) or Nd:YAG lasers (355 or 266nm) to produce high energy pulsed UV light to excite phosphors. The typical amount energy used is 5-10mJ, 10ns at 20Hz. The state of this technology has advanced in the past few decades, and present Q-switched solid state laser system can be expected to deliver around 500mJ at 355nm and 200mJ at 266 nm with repetition rates of around 20Hz and pulse duration of 10ns [108, 109]. High powered pulsed laser systems may not

be suited for phosphor illumination as the high fluence (energy/area) may be too great for the phosphor and issues such as sputtering and the breakdown of air may become more relevant at energies greater than 30mJ. However, high-energy laser beams could easily be expanded to reduce fluence and be used to produce large UV illumination areas suited for phosphor imaging purposes.

*Q switched diode pumped solid state (DPSS) laser:* These lasers use laser diodes instead of flash lamps to pump the solid gain medium. Pumping efficiencies are greater as the diode's narrowband wavelength is optimised for peak absorption. Although flash lamps generate broader wavelengths, all their emissions is not absorbed. DPSS lasers have replaced many flashlamp lasers in many scientific applications. Typical high energy systems deliver 1 mJ/pulse in the UV range, which is much lower than flashlamp-pumped alternatives. However NASA is currently developing a system that will deliver energies greater than 200mJ in the UV range [110]. DPSS lasers offer higher repetition rates (500 to 5,000Hz), compared to a typical 20Hz for flashlamp lasers. Lot-Oriel Group [111] have produced a DPSS Nd:YAG laser capable of delivering 250 mJ/pulse at 1064 nm with a repetition rate of 400Hz and a pulse width of 0.8ns. Assuming a typical reduction in power by an order of magnitude for generating UV wavelengths, this equates to approximately 25 mJ/pulse.

**Excimer Lasers:** These are gas lasers formed by a mixture of three different gases: a rare earth gas (e.g. Ar, Kr, Xe), a halogen (either F or Cl), and a bath gas (Ne or He). An advantage of excimer lasers is that they produce high power pulse outputs directly in UV range, and no frequency tripling/quadrupling is required that typically reduces the energy by an order of magnitude. Typical high energy systems produce 200mJ energy/pulse at wavelengths ranging from 157-351nm [112]. They typically operate with repetition rates much higher than standard Q-switched Nd:YAG systems, at 500-1000 Hz, with pulse duration near 10 ns. More powerful models, such as Coherent SX series, offer 1000 mJ/pulse at 300 Hz [113]. According to Junger and Schmidt [114], excimer lasers still remain unchallenged as the only source laser to deliver high pulse energies and high average powers at UV wavelengths.

In the past, excimer lasers had issues with working lifetimes, laser pulse stabilities and performance. However, these have improved in the past few decades with increased gas, tube life and pulse homogeneity [114]. Pulse stabilities have improved from  $\pm 12\%$  to  $\pm 2\%$ , with a jitter of less than 2ns. This is still slightly lower than Nd:YAG systems that has typical stabilities of 1% and jitter of 0.5 ns. However, recent improvements in Junger and Schmidt [114] have reported energy stabilities to have improved to  $\pm 0.5\%$ . Excimer lasers produce quasi-rectangular beams, typically 8×20 mm, with a near-Gaussian profile in the short axis, and a super-Gaussian profile in the

long axis (Figure 40). Due to the shape and intensity profile, transformations cannot be made using the same optical systems used for round Gaussian beams. Most applications require the beam to be modified by homogenizing and then reshaping the profile to match the application. This can add complexity in the optical system.



Figure 40: Typical rectangular excimer laser profile. Taken from [112]

#### **Continuous lasers/light sources**

Continuous lasers and other light sources are suitable for intensity mode measurements. Pulsing can be introduced to enable temporal mode (e.g. lifetime decay) analysis. One way is to use a mechanical shuttering mechanism. However, there are limits on how fast these can operate; older mechanisms are operative in the sub milliseconds regime, and are considered too slow and unsuitable to detect lifetimes New optical choppers/mechanical shutters can provide subshorter than this. microsecond pulses [115] and now there are a range of optical shutters (LCDs) that can provide responses faster than this. Electrical pulsing the power to the device can also be used. However there are rise-times and fall-times related to the apparatus that will limits its use. The problem with continuous excitation sources is that the pulse energy produced is relatively low. To obtain the same energy as typical 10ns 10mJ Q switched Nd:YAG laser pulse, you will need a continuous laser operating continually with 1 MW of power; whereas high energy laser systems can only operate with average powers of 5 KW (x200 reduction). This limitation has lead to researchers resorting to high peak power pulsed laser systems, even when they utilise the intensity response mode [70, 74, 80].

#### Fibre Lasers

Fibre lasers are increasing becoming more popular due to increased reliable up-time, beam quality, reduced running costs and servicing operation. In principal, fibre lasers are similar to DPSS lasers. The generic design includes: a.)laser diodes for pumping, b.)a scheme for coupling the pump energy into the gain medium, c.)a fibre based resonator configuration with brag gratings instead of mirrors, and d.)a method for

getting rid of excess heat. As the laser is created directly inside a fibre, there is no need for optical setup that requires the beam to be delivered to a target via a series of steering mirrors. It also eliminates the need for fibre optic coupling from a conventional laser. Fianium Ltd has recently created the worlds' first commercial high-powered 266 and 355 nm UV fibre laser [116]. The system is capable of producing 1W average power at 100 MHz, with a pulse width of 10ps. These yields to  $0.01 \,\mu$ J /pulse and a peak power of 1 KW, which is relatively low.

#### **UV LEDS**

Allison et al. [117] reports that high energy UV LEDs could be used to excite phosphors. They can be used in continuous or pulsed mode. Although they have relatively low powers, they can be operated with higher pulse-widths to increase the total amount of pulse energy. They have fall times of a few ns, and have emissions spectra much broader than that of a laser that may be better absorbed by the phosphor which may produce intensities of similar magnitude. Newly developed high powered UV LEDs can produce 450 mW of continuous 380 nm UV light [118]. In pulsed mode with a pulse-width of  $1\,\mu s$ , this yields to energies of around  $0.45 \,\mu J$  /pulse, which is relatively low (approx x20,000 reduction compared to typical q switched Nd:YAG laser pulses) and would therefore be unsuitable for high temperature measurements where the phosphors efficiency is low and blackbody radiation is high. However, it may be suitable in applications where relatively low temperatures are concerned. LEDs have replaced lasers in many applications including pressuresensitive-paints analysis and fluorescence detection for biological purposes [119]. Since the pulse-widths can be modulated, the decay lifetime can be determined using the frequency domain approach using sinusoidal wave. Allison et al. [89] reports using this approach using blue LEDs (Figure 41) to determine the phosphor's lifetime and temperature.



Figure 41: Sinusoidal wave used to excite phosphor. Taken from Allison et al. [89]

# 3.7 Consideration factors inside an engine environment

The idea of phosphor thermometry dates back to the late 1930's, This technology has attributes that make it better suited than existing methodologies and have also been demonstrated to be successful in a number of applications ranging from measurements of internal combustion engines[53], burning materials and pyrolysis 55], supersonic[56] and hypersonic aerothermodynamics studies[54, to experiments[57-59]. An excellent review with a strong theoretical background to phosphor thermometry is provided by Allison and Gillies [42]. Khalid and Kontis [60] recently reviewed developments in the past 15 years. Phosphor thermometry within turbine engines dates back at least 20 years [48], and a number of relevant studies have been undertaken since [8, 49-52]. Bird has also completed some work at the sponsoring company in conjunction with Bradford University and Nottingham Trent University. Although phosphor thermometry exhibits characteristics to make accurate measurements, it has limitations and is still immature for direct use. Variations between component geometries, operational temperatures, optical access, and rotation speeds make it difficult to generalize a universal measurement solution for all engine components. The high temperature and fast rotating engine environment presents some challenges with interrelated issues that would need to be collectively addressed before a successful measurement system can be implemented. Examples include rising blackbody radiation, restricted optical access and time to collect data, etc. These factors will impose temperature limits and greatly influence the design philosophy of the measurement system, including phosphor choice, bonding technique, excitation/detection methodologies, and probe design. The thermographic phosphor system solution will therefore be bespoke to suit the end desired application. Figure 43 highlights some of these issues related to the overall measurement system.

The indentified issues form the basis for the rest of thesis. In order to design a phosphor thermometry measurement solution for different engine components, it is important to understand the different component geometries, expected component temperature range, linear velocities and engine restrictions for optical access. This has already been completed in section 2.3 and Table 4. Different detection and excitation methodologies have already been discussed earlier. Adhering the phosphor to the surface of interest is vital for the successful application of phosphor thermometry. It should be durable and capable of surviving the exposed environmental conditions. The method should be chemically compatible with the phosphor, substrate, and the environment. A number of bonding techniques are discussed in section 5.1.

Due to engine restrictions, the only feasible solution for laser delivery to engine components is the use of fibre optics. They must be able to survive the thermal environment they will be exposed to. The maximum energy that would be delivered through them must be carefully considered. This has implications on the performance of the phosphor thermometry technique, with larger energies allowing greater emission intensities and a higher temperature capability. This is covered in chapter 4.

As there will be limited optical energy available to the phosphors and restricted emission collection, the effect on the phosphor's intensity performance were evaluated. Absolute intensity measurements can permit estimations of the resultant optical powers for different optical configurations and phosphors. This is covered in chapter 6.

Generally there is reducing phosphor emission intensity with increasing temperature, and a detection limit is reached when the signal eventually becomes too weak relative to the noise inherent in the detection system. In addition to this, rising blackbody radiation eventually becomes too large masking out phosphor emissions. This becomes increasingly more important at temperatures above 1000°C. There are a number of approaches that can be adopted to reduce the effects of this to maximize the performance of the technique. The detected radiation will be a modulation of both the blackbody radiation and the phosphor emission. This can be large relative to the contributions from the spiky phosphor emission if large band filters are used. The use of narrow band interference filters centred at phosphor emission peaks reduces the proportion of blackbody radiation passing through them, yielding better phosphor signal-to-blackbody radiation ratios. This is illustrated in Figure 42. This however results in lower transmission efficiencies, and therefore a compromise is made between the choice of filter and the system's ability to detect lowlight levels. The peak emission wavelength may be shifted with increasing temperature, therefore the emission spectra at increasing temperatures is required to ensure the choice of interference filters are correct and optimised for the given phosphor at the required temperature. This analysis is covered in section 5.2.



Figure 42: Amount of background radiation (shaded area) collected using wide and narrow band filters

The choice of phosphor in terms of emission wavelength is also another important factor to consider in combating the effects of rising blackbody levels. For a given temperature range that is likely to be experienced in turbine engines (500-1500°C) and spectral range (300 - 800nm), the blackbody radiation, predicted by Planck's law of radiation, is greater at longer wavelengths; therefore, it is more effective to use phosphors with emission peaks at lower wavelengths. Ranson [64] notes that the level of blackbody radiation at 544nm, the peak emission wavelength of YAG:Tb, is a factor of approximately 5x less than the peak emission wavelength for  $Y_2O_3$ :Eu, 611 nm, Allison and Gillies [42] notes that at 488nm, one of the peak emission wavelengths for YAG:Dy, has an order of magnitude less blackbody radiation than the peak emission wavelength for  $Y_2O_3$ : Eu at the same temperature. However, this should not be used in isolation, and the intensity response of the phosphor at given temperature must also be considered. Ranson later notes that the strong intensity exhibited by  $Y_2O_3$ :Eu at 611nm outweighs the advantage of YAG:Tb at 544nm in terms of blackbody radiation [64]. The phosphors signal to blackbody radiation to take both these factors into consideration was performed and is highlighted in section 6.2.

With temporal lifetime methods on stationary components, a lower temperature limit is based on the quenching temperature of the phosphor, while an upper limit is reached based on the phosphor's intensity at rising blackbody radiation levels and the systems ability to detect faster decaying signals. For rotating components there will be a restricted time window to make measurements, therefore the limit will also depend on the probe design and the rotational velocity of the component. In addition to this, as the phosphor's signal traverses through the acceptance cone of the fibre, the collection efficiency will vary as a function of rotation angle, with the maximum efficiency at the fibre axis varying to zero at limits of the probes field of view, and would consequently distort the detected phosphor decay waveform. Due to these limitations, a phosphor selection criterion for different engine components was devised in order to select the most suitable phosphors that would enable temperature determination. A fibre optic probe and optical layout was setup using design constraints, including fibre choice, maximum permissible lens size and target distances and was tested on stationary and rotating cases to validate design methodologies and assumptions that would be applied under full scale engine conditions. This is highlighted in chapters 7 and 8.



Figure 43: Different factors affecting phosphor selection and overall design of the phosphor thermometry system

# 4 Fibre optic laser delivery considerations

# 4.1 Introduction

In previous sections it was identified that phosphor thermometry requires an excitation source, commonly a laser, that will require optical access into the engine. An open laser beam delivery would be extremely difficult to install due to components being the optical path and engine vibrations and movements. A practical solution would be the use of optical fibres to deliver both the excitation energy and the return emissions to/from the target. A wide range of laser based measurement techniques, such as LDA and PIV, already make use of optical fibres to deliver laser beams. They have distinct advantages over bulky conventional optic systems, making them easy to align and available to use in confined areas. As the general theory and physics of light propagation through fibre optics is well known, it is not covered in this thesis. The main problem with fibre systems is the amount of power and energy that can be coupled and transmitted through them; various transmission losses and fibre damage mechanisms are responsible for this. This has significant implications on the performance of the technique, with larger energies allowing greater emission intensities and a higher temperature measurement capability.

This chapter discusses the main transmission losses, damage mechanisms, techniques and factors for consideration for optimal performance. This investigation builds on previous work undertaken by Bird at Rolls-Royce. A series of investigations were performed using different fibre materials, launching conditions, and with other new technologies, to establish the optimal setup that would allow the maximum transmission of energy through an optical fibre suitable for engine environments. This is important because the measurement systems design philosophy and subsequent testing of phosphors (e.g. intensity and lifetime decay) will be based around this energy limit.

# 4.2 Consideration Factors and Damage Mechanisms

Many authors have shown that the maximum energy transmittable through a fibre is limited by several damage mechanisms. Figure 44 demonstrates various locations and mechanisms where damage is likely to occur. Each mechanism is dependant on a number of parameters mainly attributed to laser, fibre and launching characteristics (sub-detailed in Table 10), with the overall transmission limited by the weakest threshold. "A chain is only as good as its weakest link". A number of these interrelated factors were investigated in this chapter. Unfortunately there is no standard damage threshold measurement or procedure and therefore manufacturer

guidelines are not always clear, and do not cover every combination. Quoted thresholds are usually for continuous wave infrared laser systems, and do not capture the complete electromagnetic light spectrum, especially in the ultraviolet (UV) range.



Figure 44: Fibre damage mechanisms. Taken from [120]

Fibre Characteristics	Light Source (laser) characteristics	Others
<ul> <li>Fibre Core/Clad materials</li> <li>Core diameter</li> <li>Multimode/Single mode</li> <li>NA/Acceptance angles</li> <li>Material imperfections</li> <li>Bend Radius</li> <li>Transmission characteristics, attenuation at desired wavelength</li> <li>Damage tolerance</li> <li>End surface quality /preparation</li> </ul>	<ul> <li>Wavelength</li> <li>Energy per pulse</li> <li>Average power</li> <li>Pulse duration</li> <li>Beam diameter</li> <li>Peak power density</li> <li>Energy density</li> <li>Pulsed / Continuous Wave</li> <li>Beam Uniformity</li> <li>Beam quality</li> <li>Spectral linewidth</li> <li>Transverse characteristics</li> </ul>	<ul> <li>Alignment to the beam</li> <li>Launching/injection conditions</li> <li>Transient Medium (eg air)</li> <li>Angle of fibre injection / Fibre acceptance angles</li> <li>Fibre routing - fibre bend radii and fixations</li> <li>Environmental conditions eg. Temperature.</li> </ul>

Table 10: Factors for consideration for coupling laser radiation into fibres

# 4.2.1 Gas Breakdown

Optical gas breakdown is caused by the creation of a transient plasma initiated by the intense focused light source. According to Soubacq et al [121] the breakdown mechanism involves the following three stages:

- 1.) Pre-ionisation phase
- 2.) Heating and expansion phase: where the medium becomes highly absorbent to the laser flux and is heated very rapidly  $(10^4 K)$  and is followed by a quick expansion.
- 3.) Post discharge phase; At the end of the laser pulse, the plasma is at high temperature  $(10^{5}K)$  and continues to expand [122]

This can constitute severe problems that limits fibre transmission [123]. It not only extract energy from the beam before it reaches the target, but also causes local changes in the refractive index, defocusing and scattering the beam, which could cause permanent damage to the fibre. Buscher et al [124] showed that the energy threshold for gas breakdown varies with pressure, gas type and wavelengths. Tambay and Thaeja [125] investigated breakdown using 355nm 532nm and 1064nm in different gases, and showed that the breakdown threshold is higher at greater wavelengths. Ranier [126] also confirms this stating that threshold fluences for breakdown and damage usually decrease with decreasing wavelengths and pulsewidths. Therefore the threshold for 266nm would be lower than 355nm. As the beam energy is focused towards the focal point with a coupling lens, the rising energy density and power density increases the probability of breakdown. Gas breakdown can be reduced by decreasing the power density by either lowering the laser energy level or by focusing the beam over a larger area. The latter would require a larger fibre to maximise the collection of all the energy as the beam would overfill the fibre with energy being lost.

The plasma can also cause thermal damage at the fibre's face due to the high, but short-lived, temperatures ( $10^5$  K). This is however not always negative; Setchell [127] notes plasma occurring at the fibre entrance face can result in subtle surface modifications that can leave the surface more resistant to further breakdown or damage events. Other forms of thermal damage have been associated with high average power lasers. Light escapes from the core and is absorbed by the core-cladding interface leading to rise in local temperature that can lead to subsequent damage. However, according to Nimmo [128] average power levels of pulsed laser systems are not considered high enough for thermal damage to be relevant. The average power of the current laser used in the investigation, operating at 10mJ/pulse at 15Hz is only approximately in the region of 150mW.

### 4.2.2 Fibre Selection for UV transmission

There are numerous fibres commercially available for laser delivery. However, problems are encountered when searching for suitable fibres that are both suited for high peak laser energies and UV transmissions. The UV region has some significant limitations related to high attenuation losses and dispersion [129]. Scattering results in energy loss, reducing efficiency[130]. UV absorbs and scatters light greater than visible or IR light; UV light can alter the substrate's chemical properties. According to Polymicro Technologies [131], UV transmission is limited by intrinsic attenuation (given by scattering, electronic transitions and non-structured OH absorption) and additional loses due to UV defects (resulting from the breaking of strained bonds under

UV light or gamma radiation), and becomes increasingly more relevant at low deep UV wavelengths below 250nm. The detailed physical damage behaviour and mechanism involved goes beyond the scope of this thesis.

Not all materials offer transparency in the UV region, therefore only transparent materials for efficient transmission can be used; examples include sapphire, fused silica (quartz) and fluoride glass. Although there are a number of commercially available UV fibres that can efficiently handle transmission of relatively low laser intensities, there still remains a commercial gap for the transmission of high powers and energies. The most widely used and cited fibre material for UV laser transmission is fused silica; it is affordable, readily available and offers excellent transmission characteristics with low attenuation losses.

### 4.2.3 Thermal Tolerance

For harsh engine environments, the thermal tolerance of the fibre is of vital importance. Standard silica fibres can only withstand temperatures up to 200°C. However, the melting temperature for fused silica is approximately between 1600-1800°C depending on different crystalline structures. The relatively low temperature tolerance for fibres is due to the fibre clad material and the additional protective PFA/polymide layers that have relatively low temperature tolerances. In addition, the thermal coefficient of different clad materials is problematic at high temperatures and would cause the fibre to fail because both materials would expand at different rates. Using an all silica fibre (core and clad) would eliminate this; however, the clad would need a different refractive index for total internal reflection and transmission to be effective. Equation 14 relates the light gathering ability of the fibre (NA) and the refractive indices of core and clad materials. An all-silica fibre yields an NA of approximately 0.2 which is lower than 0.38 that is typical for standard fused silica fibres. These fibres typically come with a low temperature capability polyimide coating that can only withstand temperatures up to  $350^{\circ}$ C. This coating can be replaced by a number of fibre jackets that can provide extremely high break strength and protection against chemical corrosion whilst extending the operating temperature range from cryogenic temperatures up to 800°C. Fiberguide Industries Gold jackets offers a temperature capability up to 750°C. CuBall Coatings from Oxford Electronics also offers similar thermal protection up to 600°C. Stainless steel jackets by Fiberquide and Oxford Electronics can provide protection up to 800°C. There may be a possibility for the temperature capability to be increased further as the melting temperature for the fibre is at least 1600°C. Stainless steel has high levels of oxidation at elevated temperatures, and if this is the only limitation preventing a further increase in temperature, then oxygen resistant alloys (such as nickel alloy) may be used to

enhance this temperature capability. However, there may be other limitations and problems regarding the fibres optical transmissions and structural strength that would limit the maximum temperature capability and would be subject to further research.



Silverflex (Fiberguide [132]) (up to 625 °C).



Stainless Steel (Oxford Elect. [133]) Monocoil (Fiberguide [132]) (160°C through 800°C)



Stainless Steel Braided Hose (Fiberguide [132]) (up to 525°C)

Figure 45: Examples of commercially available fibre jackets.

Only single fibres were considered in this investigation, because it was originally thought that fibre bundles made using numerous smaller fibres would compromise the temperature capability because bundles are generally bonded using epoxy adhesives, which has a maximum temperature capability of 300°C. Specialist companies offer fibres that could be used up to 400°C, and 500°C in the short term. There are also issues regarding the fibres transmission efficiency as there are inter-fibre spaces, defined by the packing ratio which reduces the overall transmission. However, recent advances in technology such as fused-end bundles produced by LGO-Germany[134] claim to eliminate inter-fibre spaces offering higher transmission with a temperature capability up to 1000°C.

# 4.2.4 End face preparation

A number of studies have indicated the fibre front surface preparation has vital importance to the transmission efficiency. According to Photran (2007), a better surface polish allows greater power handling capability with increased damage tolerance. Scratches or debris on a fibre surface can increase the chance of damage, and studies suggest that dust on a surface can reduce the threshold by a half [135]. Ultraviolet light is absorbed and scattered more than the visible light, and therefore even tiny scratches and material flaws can act as absorption/scatter points that can cause damage. The preparation and surface quality of the exit face is also important. Setchell [136] notes the damage mechanism at the exit face is different to that from the entrance face, and is mainly due to subsurface defects from polishing and fractures from the cleaving processes.

In investigations by Alaruri et al. [52] and previous work by Rolls Royce, fibres were prepared by cleaving and polishing both ends until no scratches were observed using a x20 microscope. An optically smooth surface may not be sufficient for high energy laser systems [137]. Producing a mirror-like roughness free surface for multimode

fibres greater than  $400\,\mu\text{m}$  fibres from cleaving alone can be very difficult and additional treatments such as flame polishing are required to eliminate sharp edges from cleaved surfaces [120]. Fiberguide Industries [138] notes that even after fine grinding subsurface cracks can extend up to 4-6x the peak-valley surface roughness depths, and therefore it is recommended that these are completely polished out.

### 4.2.5 Loss/damage in bends

Bending can cause permanent mechanical damage to a fibre, especially during installation and routing of the fibre, and is due to mechanical forces such as tension that can cause micro-cracks[139]. High laser energies and powers can also damage the fibres. When a fibre cable is bent excessively, the optical signal within the cable may refract and escape through the fibre cladding reducing the transmission efficiency. Excessive bends can cause asymmetric power mode distribution causing high fluencies in the outside proportion of the fibre cross section, leading to permanent damage. Whispering galley rays that only reflect from the outer core/clad is responsible for this[127], and investigations indicate damage is the most at the first bend in the fibre. This damage mechanism can be inhibited if a broader power distribution is used and by using launch angles that are close to the fibre's NA. Fibre manufacturers usually quote minimum bend radii to prevent this and mechanical damage from occurring.

### 4.2.6 Solarisation Effects / Luminescence in the fibre

Standard synthetic silica optical fibres with high-OH levels drop their transmission to 50% of their original value upon exposure to an unfiltered deuterium lamp (UV source) within 24 hours of continuous irradiation [140]. In addition to this, they can develop significant colour centres visible to the eye. This is solarising behaviour. Nonsolarising UV fibres are commercially available and offer high performance with high steady transmissions between 160-1200 nm without solarisation (Ceramoptics's Optran UVNS, and Ocean Optic's Xtreme Solarisation Resistant fibres). Although deuterium lamps are used for testing, Delmdahl et al. [141] notes that these relatively low intensities are insufficient and cannot describe the photo-degradation behaviour for high power pulsed applications, therefore solarisation tests for high power applications include the exposure of approximately 10,000 laser pulses at 193nm at fluences of approximately 50 MW/cm<sup>2</sup> [142]. Studies indicate that the main reason for the decrease in transmission after few thousand high intensity pulses is color-centre formation, with dopants and impurities that may have contributed to this. Nimmo[128] observed red fluorescence in silica fibre when illuminated at 266nm. According to the author, this could have been indicative of impurities in the fibre, which can cause interference problems with the thermographic phosphor signal that may be difficult to separate out, especially if a single fibre optic delivery and detection system is incorporated. However, material improvements in the past decade have led to significantly smaller degradations. Delmdahl et al. [141] showed that a 1m, 800  $\mu$ m diameter fused silica fibre successfully transmitted more than 2.5 million pulses of 308nm XeCl excimer laser with a pulse energy of 7.5mJ and pulsewidth of 8ns, equating to a fluence of 1.5J/cm<sup>2</sup> and peak power density of 410MW/cm<sup>2</sup>. Similar tests by a Japanese firm have shown damage tolerance to pulsed 193nm laser with fluences of 20mJ/cm<sup>2</sup> for over 10<sup>5</sup> pulses on DUV fibres[143]. According to Rayotek [144] sapphire fibres do not produce solarising behaviour.



Figure 46: Fibre Transmission of 2.5 million high intensity 308nm laser pulses of 7.5mJ energy with a pulse width of 8ns. Taken from Delmdahl et al. [141]



# 4.3 Experimental Setup

Figure 47: Schematic for fibre optic coupling investigations

An investigation was setup to establish the maximum energy that would be transmittable under various UV wavelengths, laser energies, and launch conditions. The generic experimental setup is shown in Figure 47. A Q-switched Nd:YAG laser

operating in the UV region (266nm, 355nm) was used as the laser source. Only UV wavelengths were investigated because the chosen phosphors under investigation are most efficient at these wavelengths. An SMA fibre mount connected with a micro positioner was used to hold the fibre. Fibres with diameters less than 750  $\mu$ m fibre were held in an SMA pinvice, and over 750  $\mu$ m were held in a SMA fibre connectorisor. A 50mm/25mm coupling lens was used to focus the beam, and the micro fibre positioner was used to finely vary the fibre's horizontal position to/from the focal point. A Gentec energy meter was used to monitor the transmission of energy at the fibre's exit. The fibre mount was carefully pre-aligned to the optical axis so that the laser beam passes through the centre position of the fibre for the full range of horizontal positions. The vertical and angular motions of the axis were fixed. To ensure the beam was collinear to this axis, and passed though the centre, a low powered He-Ne was positioned using mirrors and beam combiner as shown in Figure 47. This aided the alignment of optical components and helped to avoid any premature damage caused by misalignment.

1.0mm core all-silica 'Superguide G' fibres were used. Fiberguide Industries claims them to offer the lowest loss and highest laser damage resistance in the industry for UV-visible applications [132]. It has a 95% transmission at 266nm, and higher at 355nm. It contains a high hydroxyl ion content that provides high transmission efficiency from UV through visible and into IR. Fibres with high-OH silica cores for UV applications are now very common, and other manufacturers also offering similar products include Polymicro Technologies' – FV series fibres which were also investigated.

#### 4.3.1 Fibre Preparation

Approx 15-20cm lengths of fibre were used, with both ends prepared in the exactly the same manner. Fibres were initially scribed with a diamond scribe and snapped. Both ends were placed into blank SMA connectors (Thorlabs) with the appropriate fibre diameter drilled out. Candle wax was used to ensure fibre remained secure during the polishing and experimental procedures. Wax was used instead of epoxy as it allowed fibres to be easily re-prepared or replaced. The ends were attached to an SMA polishing disc and were polished using aluminium oxide lapping paper on a glass polishing plate, starting with  $30 \,\mu\text{m}$ ,  $10 \,\mu\text{m}$ ,  $3 \,\mu\text{m}$ ,  $1 \,\mu\text{m}$  and finally a  $0.1 \,\mu\text{m}$  to obtain a very high quality surface finish. A 200X magnification inspection fiberscope (Thorlabs) was used to ensure the finish was free from visible scratches. As the sapphire fibres were crystalline and harder than quartz, diamond lapping paper was used instead. Both ends of the fibre were cleaned in acetone prior to mounting onto the SMA fibre mount.

# 4.3.2 Beam and Launching conditions

A full range of fibre positions with the beam converging into the fibre, and with the beam diverging out from the focal point were investigated (shown in Figure 48)



Figure 48: Converging and diverging beam

The simplest injection approach is to use a plano-convex lens to focus the beam to less than the diameter of the fibre core, and then position the fibre face as far downstream as possible without overfilling the fibre core. Commercially available laser to fibre couplers are primarily designed for continuous powered lasers that have relatively low peak powers. They contain a fibre attachment that aligns the fibre to the focal point of the coupling lens. If these were used with high peak powered lasers, the peak energy and power densities would be extremely high resulting in damage to the fibre.

The unmodified beam diameter was approximated to be 7mm. This was focused using coupling lenses which had focal lengths of 25mm and 50mm. The launching angle ( $\alpha$ ) could be calculated using simple geometrical relations. To maximise coupling efficiency, the cone angle should also take into consideration the numerical aperture (NA) of the fibre. The NA is the light-gathering ability of a fibre and determines the maximum launching angle (critical angle- $\alpha_c$ ) at which light can be accepted and propagated through the fibre via total internal reflection. The derivation, found in many text books, reduces to Equation 14. It can be seen that the NA is dependent on the core-clad reflective indices of the fibre and is usually quoted in manufacturer specifications.

NA = n.sin 
$$\alpha_c = \sqrt{n_{core}^2 - n_{clad}^2}$$
 Equation 14



Figure 49: Launching angle

The NA of the fibre is also dependant on the refractive index of the medium it is in. However, when coupled in air ( $n_{air} = 1$ ), NA=sin $\alpha_c$ , and since sin $\alpha_c = \alpha_c$  for small angles, it can be assumed that NA= $\alpha_c$ .



Figure 50: Variation of angle as functions of laser beam diameter and lens focal length

The launching angle ( $\alpha$ ) is influenced by the beam diameter (d) and the focal length of the coupling lens (*f*), and therefore a number of combination pairs exist that can be used to obtain the same angle. Figure 50 shows all these possibilities for beam diameters ranging from 0.1-30mm, and focal lengths of 1-200mm, and can be used as simple design tool to quickly match up coupling lens/beam diameter requirement for a given fibre. To obtain good transmission efficiency it is important for the launching beam not to overfill the fibres face, and have a launch angle ( $\alpha$ ) that is smaller than the NA of the fibre. Quoted NA's for fused silica fibres were found to be as high as 0.4, and were highly dependent on the clad material used. A higher NA enables more efficient light coupling and transmissions at tight bends[132]. However, for an all silica fibre that would be suitable for high temperature applications, the differences in refractive indices between the core and clad materials are relatively small, yielding NAs of approx 0.15 and 0.22. In the investigation a coupling lens with a focal length

of 50mm and 25mm were used. This is expected to produce half cone angle of approximately 0.07 rad and 0.14 rad respectively. With the average fibre's NA of 0.185, this results in launch angles of 0.75NA and 0.38NA respectively. Fiberguide Industries [138] suggests launch angles between 0.3-0.9X the fibre's NA; both these lenses will produce angles that fall within these limits.

# 4.3.3 Energy density, fluence, peak powers and power densities

The fluence (energy/area) and power density (power/area) can be used to indicate the damage thresholds of fibres. Both parameters are different but equally important. Although the energy/area may not be very high, the peak power can be very high due to the short pulse durations that yields very high peak power densities. By having knowledge of the energy/pulse, pulse duration and variation of the beam diameter, the fluence and the power densities can be determined at different positions relative to the focal point.

The laser beam characteristics were as follows: Pulse duration : 12ns Pulse frequency: 15 Hz. Wavelength: 266nm or 355nm Average Energy /Pulse : Controllable from: 1-15 mJ/pulse (355nm) & 1–5 mJ (266nm)

The pulsewidth does not affect the energy or energy density (energy/area), however it does affect the power density (power/area) since power=energy/time. Having the same amount of energy transferred over shorter pulses yields greater peak powers, and peak densities. The beam area at various positions can be determined using geometrical relations highlighted in Equation 15 and Figure 51, and is illustrated in Figure 52. Based on an energy level of 10mJ/pulse, the fluence and power densities can also be found, illustrated in Figure 53.

 $BeamArea_{@x} = \pi.y.^{2} = \pi.(x\tan\alpha)^{2} = \pi.\left(\frac{xd}{2f}\right)^{2}$  Equation 15

Figure 51: Beam area at various positions (x) from the focal point





Figure 52: Variation in beam diameter and beam area at various positions from the focal point. Simulation using an initial beam diameter of 10mm, and lens with a focal length of 50mm.



Figure 53: Variation in the peak power density, average power density and fluence at various positions from the focal point. Simulation using a 10mJ/pulse, and lens with a focal length of 50mm, and initial beam diameter of 10mm.

Figure 54 and Figure 55 illustrate the variation in maximum peak density at various positions relative to the focal length at various laser energy/pulse levels. If a maximum peak power density threshold of 53MW/cm<sup>2</sup> is assumed, as described in a Rolls Royce internal technical report, the graphs can be used to predict how close to



the focal point a fibre can be positioned at various pulse energies before damage is likely to occur.

Figure 54: Variation in the peak power density at various positions from the focal point, using various laser energies/pulse ranging from 5mJ to 50mJ



Figure 55: Variation in the peak power density at various positions from the focal point, using various laser energies/pulse
Based on the relations highlighted in Equation 15, the beam diameter reduces to zero at the focal point, implying infinite fluence and power density. However, in reality there is a minimum beam waist that is dependent on the beam quality, wavelength and lens design which prevents the beam focusing to zero. Assuming that the minimum focus is much less than the fibre diameter, a linear relationship between the beam diameter and focal position can be assumed. The total amount of energy and power that will fall onto the fibre's face will depend on the fibres position relative to the focal point. If the beam diameter is greater than the fibre diameter (overfill), then some energy will be lost. Figure 56 shows the total energy and power that are experienced by fibres of different diameters at various positions relative to the focal point. A larger fibre diameter would experience the same level of energy at distances further away from the focal point, with the advantage of reduced fluence and power densities at that position. The ratio of beam area to fibre area, can indicate the proportion of beam energy falling onto the fibre, and can be used to estimate realistic transmissions % at various position. Figure 57 and Figure 58 shows the variation of laser beam to fibre area ratio for various fibre diameters at various positions relative to the focal point. An expression was derived relating these terms to determine the amount of total energy falling onto the fibre at various positions, shown in Equation 16. Equation 17 is derived by substituting in Equation 15 into Equation 16.

- $E_f$  = Total energy on the fibre's face (mJ)
- $E_L$  = Energy of laser (mJ)

 $d_f = fibre diameter (mm)$ 

f = focal length of lens (mm)

x = position from the focal point (mm)

2 \

 $d_{bx}$  = laser beam diameter at position x (mm)

$$E_{f} = E_{L} \frac{area_{fiber}}{area_{beam@x}}$$

Equation 16

$$E_{f} = E \cdot \frac{\left(\frac{\pi d_{f}^{2}}{4}\right)}{\left(\frac{\pi}{4}\right)\left(\frac{x \cdot d_{bx}}{f}\right)^{2}} = E \cdot \left(\frac{d_{f} \cdot f}{d_{bx} \cdot x}\right)^{2}$$

Equation 17

This term  $\left(\frac{d_f \cdot f}{d_{bx} \cdot x}\right)$  in Equation 17 determines the minimum distance (x) that is required for all the beam energy to fall on to the fibre's face. This critical distance is determinable when the term =1, shown Equation 18, and provides the theoretical optimum distance with the most energy falling onto the fibre, at the lowest fluence and power density levels. The term has to be  $\leq 1$  to ensure the energy falling on the fibre face is below the input energy. A value of unity should be used in Equation 17 when the value is > 1.

 $x = \left(\frac{d_f \cdot f}{d_{bx}}\right)$ 

Equation 18

A number of tests were performed when the launch angle of the beam was converging and when the launch angle was diverging out from the focal point and then into the fibre. The fibre position relative to the focal point and the amount of energy that was transmitted through the fibre were noted for post analysis.



Figure 56: The total average power, total peak power and total energy experienced on the fibre's face of fibres with different diameters, at various distances relative to the focal point. Simulation using a laser pulse energy of 10mJ, with the focal length of lens = 50mm.



Figure 57: The variation of laser beam area: fibre area ratio of various diameter fibres, at various positions relative focal point.



Figure 58: The variation of estimated transmission of various diameter fibres, at various positions relative focal point.

# 4.4 Estimated Delivery Performance

It is difficult to obtain information on fibre damage thresholds from manufacturers, which usually quote thresholds for continuous wave lasers and generally do not cover all the electromagnetic light spectrum, especially UV range, with the most common wavelengths quoted as 1064nm. Fiberguide Industries provides an empirical

relationship, shown in Equation 19, relating the fibre size, laser pulse duration and the maximum energy per pulse that is likely to be transmittable through a fibre. The relationship has limitations and does not account for different beam profiles and launching mediums and conditions, and is based on the recommended launch conditions of 0.7d for d<  $600 \,\mu\text{m}$  and  $200 \,\mu\text{m}$  for d> $600 \,\mu\text{m}$ , and a launch angle between 0.3-0.9 X the fibre's NA.

 $F = 440d^{0.95}t^{0.5}$ 

Equation 19

Where E= energy per pulse, J d = the fibre diameter, mm t = pulse duration, seconds

The relationship is only valid for the wavelength range 470-2145nm, however, the following correction factors are suggested for wavelengths below this.

Wavelength	400nm	350nm	300nm	250nm
Increase fibre diameter or decrease energy/pulse by a factor of:	2	3.5	7	20
Increase pulse duration by a factor of:	4	12	50	400

 Table 11: Correction factors required for different wavelengths

A simulation using these relationships was undertaken to illustrate the effects of fibre diameter, wavelength, and pulse duration, and make predictions of the maximum energy that can be transmitted under various conditions. The results of the simulations are shown in Figure 59 to Figure 61. Figure 59 shows the variation of maximum energy at different pulse widths, based on a 1mm diameter fibre at various wavelengths. Figure 60 shows that the maximum transmittable energy varies almost linearly with fibre diameter (power of 0.95), unlike damage tolerances of bulk materials that are dependant on area (or  $d^2$ ). e.g. energy/area or power/area. It is possible that this empirical relationship also takes into account other factors such as transmission losses and other damage mechanisms. Based on a 12ns pulse, the maximum pulse energies that can be transmitted are predicted (shown in Table 12) for fibres with various diameters. The likely performance for 355nm and 266nm were also estimated (x3.2 and x12.5). It is shown that for a 1.0mm fibre, the maximum energy transmittable using the existing laser system is 15mJ and 3.8mJ respectively.

	Transmittable energy per pulse				
Wavelengths	0.5 mm fibre	1.0 mm fibre	1.5 mm fibre	2.0 mm fibre	
> 470nm	24.95 mJ	48.20 mJ	70.85 mJ	93.12 mJ	
400 nm	12.47 mJ	24.10 mJ	35.42 mJ	46.56 mJ	
350 nm	7.13 mJ	13.77 mJ	20.24 mJ	26.60 mJ	
300 nm	3.56 mJ	6.89 mJ	10.12 mJ	13.30 mJ	
250 nm	1.25 mJ	2.41 mJ	3.54 mJ	4.66 mJ	
355 nm	7.8 m1	15 ml	22 m1	29 m1	
266 nm	2.0 mJ	3.8 mJ	5.6 mJ	7.5 mJ	

Table 12: Maximum estimated transmittable energy at various wavelengths for different diameter fibres, based on 12ns pulses.



Figure 59: The variation of maximum energy at different pulse widths, based on 1mm diameter fibre at various wavelengths.



Figure 60: The variation of maximum energy at various fibre diameters, at a pulsewidth of 12ns, at various wavelengths.



Figure 61: The variation of maximum energy at different wavelengths, at a pulsewidth of 12ns, for various fibre diameters

# 4.5 Results and Discussions

### 4.5.1 Fused silica fibre

The results of the investigation showing the laser transmission through Fiberquide Industries 1.00mm core SuperG fibre at various positions from the focal length, at various input energy levels, is shown in Figure 62. The experimental setup and procedure is highlighted in section 4.3. There was a loss of approximately 5% as it passed through the lens, and is accounted for in subsequent transmission % analysis. Figure 63 shows the damage points at the various positions vs. transmitted energy and input energies, and Figure 64 shows the corresponding fluence and power densities. The transmission level fell irreversibly when damage was observed and the position of the fibre when damage occurred was noted. It was found that the diverging beam allowed the fibre to be placed closer to the focal point. It also allowed a greater maximum energy before damage occurred. The estimated fluence and power density at the damage points for the converging beam was found to be 5mJ/mm<sup>2</sup> and 0.45MW/mm<sup>2</sup>, whereas it was 15mJ/mm<sup>2</sup> and 1.4MW/mm<sup>2</sup> for the diverging beam. This corresponds to a relative advantage of X3. However, in terms of pulse energy transmitted, the converging beam allowed a maximum energy of 6.42mJ/pulse whilst the diverging beam allowed 8.77mJ/pulse before damage was observed, resulting in an advantage of 36.6%. These findings are in agreement with Hand et al. [145] who noted the importance of focusing the beam in front of the fibre so that the beam is expanding to the fibre to avoid direct focusing of the beam inside the fibre. Another study has also shown that certain damage processes can be inhibited if the beam is diverging when it is entering the fibre [146]. At energies

greater than 7.8mJ/pulse, gas breakdown was observed. It is possible that any local peak fluences and local peak power densities caused by beam instabilities and beam profiling may lose some of their energy in this activity, reducing the peak power densities and fluences, and the probability of damage.

It may be possible for more energy to be transmittable through the fibre if greater input energies are used. According to the Fiberguide's relation, shown in Equation 19, it is estimated that 15mJ/pulse could be transmittable through a 1.0mm diameter fibre. Based on transmission efficiencies of approximately 70%, as a result of this investigation as shown in Figure 65, approximately 21.4mJ/pulse of input energy would be required to achieve this. Further research would be required to confirm this as the maximum input energy achievable using the current laser system was limited to approximately 15mJ/pulse.

In experiments involving lower pulse energies at approximately 2.5mJ/pulse, the fibre could be moved to 1.0mm within the focal point without any damage. This translates to a beam radius of 0.07mm and estimated peak density of 1.2GW/cm<sup>2</sup>. This however may not be the case because there will be a minimum beam waist preventing the beam diameter to be reduced further, limiting the maximum fluence and power densities that can be achievable.

The transmission of energy (as a % of initial laser energy) at various positions is compared, and is shown in Figure 65. The transmission increases when the distance is moved closer to the focal point. This is because energy is lost at distances where the beam overfills the fibre's face, and is not corrected for in the analysis. The beamarea/fibrearea ratio gives an indication of the proportion of total energy falling onto the fibre. By correcting for this at various positions, the actual transmission efficiency curve based on the predicted energy falling onto to the fibre's face can be deduced and is shown in Figure 67. It was found that some values transmitted more energy than predicted, by up to a 55%, indicated by values >100%. This was shown to be true at distances where the beam diameter > fibre diameter, and where there was overfill. The reason for this is because it is assumed that the energy distribution along the beam is uniform with the proportion of total energy falling on the fibre face directly related to the fibre area to beam area ratio. However in reality, laser beam profile distributions are near Gaussian where the centre of the beam is more concentrated than the outside. With the fibre positioned at this central position, the proportion of energy falling onto the fibre is expected to be more than estimated values based on uniform distribution, accounting for increased transmission values. As the fibre is moved closer to the focal point, a greater proportion of the total energy will fall onto the fibre, and the estimate of the total energy falling on to the fibre becomes closer to the real beam profile; when the beam diameter = fibre diameter, the estimate of the total energy falling on to the fibre face for a uniform beam profile is the same as the actual (or any other) beam profile. Further research with beam profile analysis would be required to provide further corrections.



Figure 62: Transmitted pulse energy through a 1.00mm core diameter fibre at various locations relative to the focal point, for various input pulse energy levels. Wavelength = 355nm, Focal length of coupling lens = 50mm



Figure 63: Damage points at various positions relative to the focal point. Wavelength = 355nm, f = 50mm.



Figure 64: Estimated fluence and peak power densities at the damage points.



Figure 65: Transmission efficiency (%) based on the total input pulse energy. Wavelength = 355nm, f=50mm



Figure 66: Estimated pulse energy on fibre face at various positions relative to the focal point, for different input energy levels. Wavelength = 355nm, f = 50mm.



Distance from the focal point (mm)

Figure 67: Corrected transmission %, based on the estimated pulse energy falling onto the fibre's face. Wavelength = 355nm, f = 50mm.

#### 4.5.2 Investigating the variation of launching angle

The investigation was repeated using a coupling lens with a focal length of 25mm. This produced a half cone launch angle of approximately 0.14 rads, opposed to 0.07 rads produced by the 50mm lens. With the average fibre NA of 0.185, this resulted in 0.75NA and 0.38NA for the 25mm and 50mm focal length lens respectively. Although

it was expected that the performance would be greater for the 25mm focal length lens, because it produced a launching angle of 0.75NA, which provided a better match for the fibre, the transmission results, shown in Figure 68 to Figure 72, indicate that 50mm focal length coupling lens (0.38NA) performed better. Damage threshold fluence and peak power density values using the 25mm lens were estimated to be in the region of 7 mJ/mm<sup>2</sup> and 0.6 MW/mm<sup>2</sup> for a diverging beam and approximately 4 mJ/mm<sup>2</sup> and 0.35 MW/mm<sup>2</sup> for the converging beam. This compares approximately 20% and 53% lower than those experienced using f=50mm. The transmission efficiencies were also approximately 26% and 31% lower than those experienced using f=50 mm. The reasoning for this could be accountable to minute x,y,z or even angular misalignment; it is rational to assume that greater the difference between the launch angle and NA of the fibre, the greater the tolerance to misalignment. This could be the reasoning for the improved performance from the f=50mm lens. Hand et al. [145] notes the optimum performance with conventional optics is achieved when the beam is focused to give 0.5 NA. Using this philosophy, it may be possible for the performance of the fibre to be increased further by using an coupling lens with the focal length of approximately 38mm.



Figure 68: Transmitted pulse energy at various locations relative to the focal point, for various input pulse energy levels. Wavelength = 355nm, f= 25mm



Figure 69: Transmission efficiency (%) based on the total input pulse energy. Wavelength = 355nm, f=25mm



Figure 70: Estimated pulse energy on fibre's face at various positions relative to the focal point, for different input energy levels. Wavelength = 355nm, f = 25mm.



Figure 71: Corrected transmission %, based on the estimated pulse energy falling onto the fibre's face. Wavelength = 355nm, f = 25mm.



Figure 72: Estimated fluence and peak power densities at the damage points. f=25mm.

	Max transı (mJ/pı	missions ılse)	Max flu (mJ/m	luence Max peak pov mm <sup>2</sup> ) (MW/m		wer density mm <sup>2</sup> )	
	Converging	Diverging	Converging	Diverging	Converging	Diverging	
f=50	6.42	8.77	5	15	0.45	1.4	
f=25	4.72	6	4	7	0.35	0.6	

Table 13: Comparison of maximum transmissions, fluence and power densities for f=50mm and 25mm coupling lens.

### 4.5.3 Investigating wavelength dependency: 355nm vs. 266nm

The amount of energy available at 266nm was variable between 0-5 mJ/pulse and between 0 - 15mJ/pulse for 355nm. The maximum energy was limited by the laser. The results using 266nm wavelength laser, using a 50mm focal length lens and a 1.0mm diameter core fibre optic at various input energy levels is shown in Figure 73. The maximum energy and likely performance that was transmittable using the existing

setup was approximately 3mJ and 8.7mJ for 266nm and 355nm. This relates to disadvantage of a factor of approximately x2.9 when using the 266nm wavelength. The transmission data, shown in Figure 74, indicates that the maximum transmission efficiency was approx 58% using 266nm, opposed to approximately 70% for 355nm, indicating a disadvantage of approximately x1.4 in terms of transmission efficiencies. Spectral data from bulk medium for fused silica and quartz indicates higher transmissions at 355nm compared to 266nm. The breakdown of air was experienced at around 3.8 mJ/pulse using 266nm, whereas it was approximately 7.8mJ using the 355nm indicating a difference by a factor of x2. The observation is in agreement with Tambay and Thaeja [125]and Ranier [126] who also showed that the breakdown fluence threshold is lower at lower wavelengths.



Figure 73: Transmitted pulse energy at various locations relative to the focal point, for various input energy levels. Wavelength = 266nm, f= 50mm



Figure 74: Transmission % at various locations relative to the focal point, for various input energy levels. Wavelength = 266nm, f= 50mm

#### 4.5.4 Investigating the use of DOE homogenisers

In addition to surface characteristics, damage thresholds are affected not only by the total energy and powers over the fibre surface, but also by the local peak fluences/power densities. Intense "hotspots" may be caused by non-uniform laser beam profiles, and laser instabilities that can affect breakdown and induce damage mechanisms with non-liner effects that limit the overall transmission through the fibre. An optimum design for injection optics would inhibit damage mechanisms by minimising peak fluences over the entrance face by generating a broad mode power distribution in the fibre. Beam-profiling can reveal the actual fluence distribution, and according to Setchell [146], a figure of merit for different beam profiles is established by measuring the ratio of peak to average fluences, with examples shown in Figure 75. This value should be as low as possible, and according to this relationship, a perfect uniform "top-hat" distribution over the fibre will experience a ratio of unity.





Figure 75: Fluence profiles at the fibre entrance face: a.) Gaussian Nd:YAG laser focused with a simple plano-convex lens, b.) Using a DOE, c.) Nd:YAG focused with a plano-convex lens-Result of cavity changes and realignment. (Taken from Setchell [146])

Damage in fibres can occur at peak intensities orders of magnitude lower than threshold values. According to Hand et al. [145], it is high beam quality that is responsible for this. Beam quality is often described by how tightly it can be focused. With a high beam quality, relatively few nodes in the fibre are excited, resulting in high optical intensities; non linear effects such as self focusing can lead to a further increase causing damage [145]. This can be avoided by coupling the beam to greater number of nodes within the fibre. However this requires the beam quality to be reduced by ideally conditioning the beam to produce a waist the size of the fibre and a divergence angle equal to the acceptance angle of the fibre. Unfortunately, this can not be achieved with normal Gaussian optics, as it requires a reduction in beam quality by an order of magnitude. This can be achieved by modifying the laser's fundamental frequency resonator. However, for efficient UV production by 1064nm Nd:YAG lasers, a high quality beam is required. Therefore, modifying the beam after the frequency conversion process is more rational, and can be achieved using a DOE diffuser. The field of diffractive optics has allowed a number of possibilities for optics design offering better beam characteristics than conventional lens optics. DOE are used to transform irregular incident laser beams into a uniform-intensity spot, as shown in Figure 76. Conventional diffusers disperse a collimated beam over a range of angles because they contain inhomogeneities that scatter light with angular distribution that cannot be controlled accurately. In contrast, a DOE diffuser has periodic patterns that diffract light over a closely controlled range of angles. Researchers at the Herriot Watt University were successfully able to transmit 10mJ of Nd:YAG frequency doubled 532nm Q-switched 10ns laser with a repetition rate of 10 Hz through a single fused silica fibre of 200  $\mu m$  core diameter [145]. Setchell[127] was able to transmit 110mJ of 1064nm Nd:YAG laser of approx 9ns through a 365  $\mu m$  fused silica fibre, corresponding to 11.2GW/cm<sup>2</sup>. However investigation in the UV range in the literature is limited.

Irregular beam profile

Smoothened uniform beam profile

Figure 76: Beam profiles with and without DOE

A DOE, provided by Rolls Royce plc, was aligned in front of the coupling lens, using the schematic shown in Figure 47. An input laser beam of 355nm wavelength, approximately 3.7 and 8.5mJ/pulse at 15Hz was used in the investigation. The laser beam was allowed to expand out of the DOE and onto the coupling lens (f=50mm), where it could be focused onto the fibre, as shown in Figure 77. The fibre used for the investigation was 1.0mm core Fiberguide Industries SuperG.



Figure 77: Injection optics using a DOE. Taken from Hand et al (1999)

The DOE only diffracts a fraction of the light (DOE first order diffraction angle), defined by diffraction efficiency (typically 85%); whilst the rest remains undiffracted with a divergence angle of 0°. It was found that the position of the DOE relative to the

coupling lens position did not affect the zeroth-order focus. This remained to be at the focal length of 50mm with or without the DOE. Positioning the DOE too far caused the beam to overfill the lens, losing energy, as shown in Figure 78. With the DOE too close to the lens, there was no significant shift of the first order focus, and it appeared to remain at the same position as the zeroth order focus, as shown in Figure 79. As the DOE was moved further away from the lens, the diffracted focus moved a few millimetre after the zeroth focus position, as shown in Figure 80. The zeroth order focus remains at high quality and can still form a narrow focus. However, since this focus is a few millimetres before the diffracted beam focus, it can be optimally focused infront of the fibre, rather than at the surface or inside it. There was minimum beam waist which was approximately 2.0mm in diameter. This size decreased slightly when the DOE was moved further away from the lens. This could be due to energy losses by overfilling the lens which decreases the number of divergent diffractive nodes, reducing the effectiveness of the DOE.



Figure 78: Lost energy when the DOE is placed too far away from the coupling lens.



Figure 79: Zeroth order focus and diffractive focus at the same position



Figure 80: Location of the zeroth order focus and diffractive focus The transmission results through the fibre with and without the DOE with input energies of approximately 3.7 and 8.5mJ/pulse are shown in Figure 81 and Table 14. Losses were incurred due to scattering and reflectance from surfaces and were measured to be in the region of 5% for the lens and 7.5% for the DOE, resulting in a total loss of approx 12.5% when the DOE was used. With these losses taken into account, the maximum transmission efficiency % with and without the DOE was found to be approximately 34% and 72% respectively. This is approximately a transmission efficiency disadvantage of a factor of X2 when the DOE is used.



Figure 81: Results with and without the DOE with input laser energies of: a.) 3.7mJ/pulse and b.) 8.5mJ/pulse

		Energy In	Corrected	Max energy	Transmission
		(mJ)	(mJ)	transmitted (mJ)	(%)
Case One	With DOE	3.7	3.23	1.03	32%
	Without DOE	3.7	3.6	2.69	74%
Case Two	With DOE	8.5	7.4	2.63	36%
	Without DOE	8.5	8	5.6	70%

Table 14 : Transmission % of energy with and without the DOE

The DOE eliminated the breakdown of air, and allowed the fibre to be moved considerably closer to the zeroth order focal point. Although it contains less energy, it

is still capable of causing damage. Purely based on the 15% undiffracted beam (1.11mJ/pulse) and ignoring the energy of the diffracted beam, the proximity of the fibre to the focal point translates to fluences and peak power density of approximately 18mJ/mm<sup>2</sup> and 1.5MW/mm<sup>2</sup> at 2mm, and 72mJ/mm<sup>2</sup> and 6.0MW/mm<sup>2</sup> at 1mm from the focal point.

As the beam waist of the first order focus was greater than the fibre area, a large proportion of the energy did not enter the fibre's face, shown in Figure 82, and has not been accounted for in the transmission % analysis. By assuming that all of the zeroth order undiffracted beam, and only a proportion of the diffracted energy (based on fibre to beam waist area ratios, shown in Figure 82) entered the fibre, an expression was derived (Equation 20) to determine the total amount of energy falling onto the fibre face. This can then be used to evaluate more realistic transmission efficiencies and benefit of having the DOE.

$$E_{f} = \left(1 - \frac{k_{DOE}}{100}\right)E + \left(\frac{k_{DOE}}{100}\right)\left(\frac{d_{f}}{d_{b}}\right)^{2}E$$
 Equation 20

Where:

 $E_f = \text{Total energy on the fibre face,} \\ E = \text{Input Energy} \\ k_{\text{DOE}} = \text{DOE diffraction efficiency, typically 85\%} \\ d_f = \text{effective diameter of fibre} \\ d_h = \text{diameter of diffractive beam waist.}$ 



Figure 82: Energy falling onto the fibre's face

	Effective Energy in (mJ)	Undiffracted Energy (mJ)	Diffracted Energy (mJ)	Diffracted Energy falling on the fibre face (mJ)	Total Energy on fibre face (mJ)
Case One	3.23	0.48	2.75	0.6875	1.1675
Case Two	7.4	1.11	6.29	1.57	2.7

Table 15: Effective energy falling on the fibre face

The transmission performance with the DOE with this correction calculates to an average of approximately 92%, compared to approximately 71% without the DOE. This indicates a transmission benefit using a DOE. However, based on transmission results from the existing setup, an input energy of approx X2.1 would be required with the DOE to produce the same level of output without the DOE. Although the %transmission may be lower, based on these findings pulse energies greater than 28mJ would be required to realise the benefit of using the DOE and enable higher energies to be transmitted through the fibre. This exceeded the maximum energy that could be provided by the existing laser system. Using a smaller fibre and lower laser energy levels and inferring the maximum power through a 1mm diameter fibre was not a sensible option because the minimum beam waist with the DOE would exceed the input area of the fibre incurring significant losses. It is predicted that if a larger fibre with a greater input surface area to capture more of the diffracted energy, or a different DOE that produced a smaller beam waist, is used, more energy would be coupled and transmitted through the fibre. The optimum solution would be to use the DOE that produces a diffusion angle less than the NA of the fibre with a beam waist (first order focus) that is the same size as the fibre.

#### 4.5.5 Sapphire Fibres vs. Fused Silica Fibres

The use of single crystal sapphire fibres may be an alternative to conventional fused silica fibres for delivering UV laser energy to the phosphor target inside the gas turbine engine. They have distinct advantages, making them very suited for this purpose. Sapphire, aluminium oxide ( $Al_2O_3$ ), is very hard with Mohs hardness scale rating of 9. It exhibits high mechanical strength, chemical resistance and thermal stability making them very suitable for harsh environments. With a melting temperature of 2053°C they can be inserted directly into the engine without additional thermal protection.

For fibre coupling and optical properties, they offer high transmissions from UV to IR wavelengths and the main perceived advantage compared to silica fibres is that their increased power handling performance with quoted damage threshold of  $1200J/cm^2$  [147] and experiments in the literature have demonstrated their ability to accept input energies of 750mJ/pulse at 10Hz. Nubling and Harrington [148] suggests a greater potential (>1J/pulse) from a typical 300 µm core fibre, and thus the performance from a 1.0mm core fibre is expected to be higher. However, these findings were from lasers with pulsewidths in the 500ns regime, which would result in significantly lower peak power densities compared to pulses in the 10-20 nanosecond regime. Other work using nanosecond pulsed lasers at IR wavelengths revealed 43mJ of energy

successfully transmitted through a 425 µm fibre. Assuming the same fluence and power density damage thresholds, this equates to 238mJ of energy for a 1mm fibre. Unfortunately, no relationship or guidelines were found in the literature or manufacturer guidelines to estimate the performance at UV wavelengths. Therefore the Fiberguide Industries corrections for UV wavelengths, shown in Table 11, was used, and yielded a maximum energy/pulse of 75mJ at 355nm and 19mJ at 266nm though a 1mm core sapphire fibre. This is a factor of x5 better than fused silica fibres. A study by Flourescience[149], found that sapphire fibre absorbed all the UV 355nm power supplied translating to approximately 20MW peak power and a power density of 3GW/cm<sup>2</sup>. According to Noel [149] this was a factor of 100 X better power handling than the best fused-silica fibres reported. Not many other tests have been conducted or reported in the literature for the transmissions of high powered UV lasers through sapphire fibres.

According to Photran[147], sapphire fibre's thermal conductivity is 30X higher, and the Rayleigh scattering is 30X lower, than that of fused silica. Some losses in the UV range (below 300nm) are attributed to Raleigh scattering[148], therefore UV transmissions are expected to be better than fused silica. However, transmission and attenuation curves from various manufacturers suggest differently, showing the transmission for sapphire is lower, at approximately 80% at 266nm and 85% at 355nm [144], compared to over 90% for fused silica. Fiberguide industries indicates transmission for fused silica fibres to be approximately 98%/m at 355nm and 95%/m at 266nm. This may be due to other losses that go beyond the scope of this thesis. The index of refraction for sapphire is 1.71, which results in reflection losses of 6.9%, in comparison with only 4% reflection losses from silica fibres (n<sub>fused silica=1.4</sub>) [150].

Due to the high transmission losses, long lengths of sapphire fibres should be avoided, despite being currently restricted by manufacturing processes at approximately 5m. Although the transmission is significantly lower than that for fused silica fibres, they are expected to handle more power densities and fluences, allowing higher energies to be transmitted when higher input energies are used. Figure 83 illustrates the attenuation of energy for sapphire and fused silica fibres at different input energies as the fibre length is increased. There is a region where the use of sapphire fibres would be more beneficial than fused silica fibres, and is dependant on: damage thresholds, length of the fibre and the amount of output energy that is to be delivered. The input energy required by the sapphire fibre to obtain the same output from fused silica fibre for various fibre lengths was mathematically derived, shown below in Equation 21 to Equation 24. Figure 84 illustrates this in graph form; by placing threshold limits based on fibre diameters, the region where sapphire fibre would be more suited is identified by the shaded area.

Equation 23

$$O_q = I_q T_q^L$$
 Equation 21

$$O_s = I_s T_s^L$$
 Equation 22

$$O_s = O_q = I_s T_s^L = I_q T_q^L$$

$$I_s = I_q \left(\frac{T_q}{T_s}\right)^L$$
 Equation 24

where:

 $I_a$  = Input laser energy on fused silica/quartz fibre  $O_q$  = Output energy from fused silica/quartz fibre L =length of fibre  $I_s$  = Input laser energy on sapphire fibre  $O_s$  = Output energy from sapphire fibre  $T_q$  = transmission efficiency per meter (95%)  $T_s$  = transmission efficiency per meter (85%)



Figure 83: Transmission of input energy, illustrating attenuation along the length of the fibre, for fused silica/quartz and sapphire fibres with different core diameters.



Figure 84: Regions were a 1.0mm core sapphire fibre would be more suitable than fused silica/quartz fibres

According to Rayotek [144] sapphire fibres do not produce solarising behaviour, and would therefore be advantageous over standard fused silica fibres that do.

A disadvantage of sapphire fibres is that they have a Young's Modulus of 65Mpsi (448GPa), making them 6x stiffer and less flexible, consequently requiring larger bend radii. The recommended minimum bend radius for sapphire, given by Photran [147] for various fibre diameter sizes is shown in Table 16. Based on this trend, it is estimated that approximately 20cm radius will be required for a 1mm fibre. This is X4 larger than the requirement for fused silica fibres, which is typically 100X fibre radius; therefore for a 1mm fused silica fibre, a radius of 5cm would be required. This recommendation for fused silica fibres is for the prevention from mechanical damage alone, and when high powered lasers are being transmitted, the recommended bend radius is 500X the fibre radius [133], yielding a bend radius of 25cm. For sapphire fibres, Photran [147] claim that the fibre would break by mechanical means before energy is lost around a bend. Therefore based on this, sapphire fibres are actually more advantageous by 25%.

Fibre diameter ( $\mu$ m)	150	250	325	425
Min. bend radius (cm)	2	3	6	8

Table 16: Recommended minimum bend radius [147]

The standard methods for cleaving and polishing do not apply because sapphire is crystalline and very hard (9 Mohs Scale, compared to 7 Mohs scale for quartz. Therefore, obtaining a good front quality surface can be very difficult; diamond abrasives and laser polishing are commonly used. The NA for sapphire fibres is in the region of 0.11, which is almost half the value for fused silica fibre (typically 0.2 for all silica fibres). This will make it more sensitive to misalignment and difficult to couple a beam into the tip of the fibre. Sapphire fibres are considerably more expensive than fused silica fibres, by a factor of X100 (estimated for 2m lengths). However, additional coatings and thermal protection that are required for fused silica fibres would also increase their costs. Currently there are two main techniques for manufacturing sapphire fibres (Edge-defined, film fed growth (EFG) and Laser Heated Pedestrial Growth (LHPG)). EFG allows multiple fibres to be grown simultaneously, making it more suitable for production scale-up, and therefore cost effective; however, according to Nubling and Harrington [148] transmission losses in these fibres are higher that than those created by LHPG, which remains to be the most promising method for developing high quality sapphire fibre. Table 17 summarises the main pros and cons of using sapphire compared to fused silica fibres.

Advantages	Disadvantages
Higher temperature capability. Simpler design with no thermal protection coatings required	Lower transmission, higher attenuation in the UV region. 85%/m (sapphire) compared to over 95%/m (fused silica)
Mohs Scale of 9.(quartz =7). More resistant to harsh environments	Stiffer – (less flexibility, requiring greater minimum bend radii.) However, for high power laser systems, the bend radius for fused silica is comparable to sapphire.
Chemically inert	Lower NA: -Sapphire NA= 0.11. - Quartz NA = 0.18-0.21
No solarisation behaviour	Expensive, approx x100 more expensive than fused silica
Estimated higher laser damage thresholds	Harder Sapphire: Mohs Scale: 9 Fused Silica: Mohs Scale: 7 Can be difficult to obtain a good surface finish.
	Limited length at 5m. However, longer lengths would experience high attenuation losses.
	Index of refraction :n <sub>sapphire</sub> =1.71 (reflection losses of 6.9%) :n <sub>fused silicationate</sub> = 1.4(reflection losses 4%)

 Table 17:
 Summarised pros and cons of using sapphire fibres instead of fused silica/quartz fibres.

Unfortunately, a 1.0mm diameter sapphire core fibre was unattainable that could be tested and directly compared against results obtained from the 1mm fused silica fibre. A  $425\,\mu\text{m}$  sapphire fibre was obtained from Photran, and to keep the investigation neutral, an all silica fibre with the same diameter (425-FV Series) was obtained from Polymicro Technologies. A SMA pinvice was used to hold the fibres in place during the investigation. Approximately 15cm length fibres were used. As sapphire is harder

than fused silica, diamond lapping paper was used in the polishing procedure. In both cases a 200X fiberscope was used ensure the finish was free from visible scratches. A f=50mm coupling lens was used to produce a launch angle of approx 0.07 rad, which was approximately 0.63NA and 0.38NA for sapphire and fused silica fibres respectively.



Figure 85: Transmitted energy through FV Polymicro 425  $\mu m$  fused silica fibres



Figure 86: Transmitted energy through Photran 425  $\mu m$  sapphire fibres

The results shown in Figure 85 and Figure 86 indicate the performance for sapphire fibres were not as superior as originally predicted. The maximum energy that was

transmitted through them using the existing setup was approximately 0.95mJ/pulse, compared to approximately 4mJ/pulse for the same sized fused silica fibre. This is approximately a x4 disadvantage. There seemed to be a mechanism that was altering the fibre face for sapphire, which was limiting the amount of energy that was being transmitted through it. Repolishing the sapphire fibre enabled more energy to be transmitted through the fibre. This surface modification mechanism was observable using the microscope, and was not present on the fused silica fibres. Despite polishing the fibres using a series of diamond lapping paper ( $30\,\mu m$ ,  $10\,\mu m$ ,  $3\,\mu m$ ,  $1\,\mu m$ ,  $0.1 \,\mu m$ ), sapphire fibres may require a finer quality polish; it would be of research interest to retest the fibres using laser polishing schedules that are meant to offer superior surface finishes and higher damage resistance compared to ordinary mechanical polishes [147]. According to Allison et al. [135] annealing the surface can raise the damage factor by 3-4. Itoh et al. [151] used a CW  $CO_2$  laser to do this and the damage threshold of 2-4 GW/cm<sup>2</sup> was observed for 248nm with 18ns pulse, corresponding to 40-80 J/cm<sup>2</sup>. Setchell [127] noted that a CO<sub>2</sub> laser conditioning following a good mechanical polish has produced surfaces that are very resistant to breakdown. Other experiments by Pini et al. [152] was able to increase the threshold to  $16J/cm^2$  by immersing the input end of the fibre in a few mm of water.

The fibres that were tested were made by the EFG method, and it would be of research interest to test sapphire fibres made by the LHPG method. Another reason that may account for the poor performance experienced by the sapphire fibres used in the investigation is that the fibre used air as the waveguiding structure (equivalent of the clad), for light propagation. From the relations presented in Equation 14, and due to high differences in refractive indices between air and sapphire, the NA of the fibre is characterised as 1.37 radians, which is many times the recommended NA of 0.11. The manufacturers claim that although the NA for the bare sapphire fibre is relatively high, launch angles > 0.11 will excite many inefficient multimode nodes reducing the overall transmission efficiency and will make the fibre very sensitive to bending. Unfortunately, adhering clad materials to the sapphire core is problematic, especially when exposed to environments of high temperatures and high thermal shock. For this reason, sapphire fibres adhered with a clad material are not yet commercially available. A study by Lee et al. [153] and Janney et al. [154] presents a method of producing and growing clad coatings top of the sapphire core. Although the work is in its infancy, this could allow greater coupling and transmission efficiency and would be subject to further research.

### 4.6 Further Research

A number of other ideas were identified and would be subject to further research. However, to fully realise the potential of such technologies and techniques to deliver more energy through the fibre, a higher laser energy system would be required.

#### 4.6.1 Hollow Waveguides and PCFs

A problem using high laser powers with solid core fibres is the damage threshold of the bulk material is reached[155]. An alternative option is to use a hollow core fibre whose core is formed by air, which according to Jelinkova, et al [155] can not be easily destroyed. These fibres exhibit extremely low losses and have proven to extremely effective at high wavelengths in the IR region. However, no known research has been undertaken at UV wavelengths. Another similar fibre that is quickly gaining popularity is Photonic Crystal Fibres (PCFs), also known as micro-structured or holey fibres [156]. They have a micro-structured arrangement of low index material (typically air voids) in the background material of higher refractive index (usually undoped silica), and employ a fundamentally different guiding mechanism. In PCF, this is achieved by incorporating holes into a glass matrix, shown in Figure 87. A photonic bandgap acts as a virtually loss-free mirror, confining light to a core. The fibres exhibit extremely low non-linearity, high breakdown threshold, zero dispersion, and negligible interface reflection [156]. They have been tested at IR and visible spectral wavelengths using high energy pulsed beams achieving superior results compared with conventional solid core fibres. No known tests have yet been reported at UV wavelengths.



Figure 87: Photonic Crystal Fiber [156]

#### 4.6.2 Tapered Fibres

Fibre optics offers convenience and flexibility, but are limited by damage thresholds caused by high power densities at the input face. Beam non-uniformity can also cause extreme intensity hotspots where the power/area can be 100x higher than surrounding areas. Some of these problems can be eased by increasing the diameter (and hence area) of the fibre, which can significantly reduce the power density and fluence by a theoretical factor of the diameter ratio squared; doubling the input diameter can reduce the power/area and fluence by 4. However, increasing the fibre diameter

through its total length increases cost, reduces mechanical flexibility requiring greater bend radiis. A tapered fibre consisting of tapered section and a constant pigtail section shown in Figure 88, allows the benefits of a greater input area, which could permit the transmission of greater energies, whilst retaining the mechanical flexibility throughout the rest of its length. Tapered fibres can allow the input face to experience a reduction in power/area up to factor of 10 [133]. They can also simplify the lateral alignment for fibre coupling because of the broader area to focus the beam. Oxford Electronics and Fiberguide industries provides tapered fibres with a maximum ratio of 4:1, with tapered lengths between 1-3 meters and pigtail section length upto 50m. To retain the optical propagation properties and efficient transmission, the launch angle must be reduced by the taper ratio, because the effective NA of the taped fibre is effectively reduced by this factor [133].



Figure 88: Tapered fibre

### 4.6.3 Hollow Tapered Pyrex-Glass

Ilev et al. [129], demonstrated that UV laser beams can be directly coupled into uncoated hollow tapers made from pyrex glass, as illustrated in Figure 89. The operating principle is based on the grazing-incidence effects when the reflectance coefficients at a dielectric boundary approach 100% at angles of incidence close to 90 degrees [129, 157]. The use of a hollow taper combines the advantages of tapered optics and hollow fibres [158]. The hollow taper possesses several advantages that offer direct launching of laser radiation providing a high coupling efficiency, which is also suitable for high energy laser systems. The taper also serves as homogenizer to provide a smooth laser output profile that can be directly coupled into optical fibres with high efficiencies [158]. In addition tapers reduce sensitivity due to misalignment. A 5mm diameter pulsed Nd:YAG laser (355nm and 266nm, 8ns) was launched directly into the taper, without any focusing elements, which produced a beam that could be directly coupled into a fibre with high efficiency and less resistant to damage when compared to coupling using a focusing lens [159]. Miller et al [160] found that a hollow pyrex glass taper enabled them to launch twice the maximum fluence achieved using lenses. The maximum fluence, using a 4-6ns, 266nm Nd:YAG laser transported through a 600  $\mu m$  core silica fibre without damage was reported to be between 3.5 and 7.5J/cm<sup>2</sup>, which results in a peak power density of 1.5GW/cm<sup>2</sup>; this agrees well

with the bulk damage threshold for high OH synthetic fused silica, which have been reported to be  $7.2 \text{ J/cm}^2$  [161].



Figure 89: The optical arrangement of a hollow taper for direct launching and delivery of UV light. Taken from Ilev et al.,[129]

## 4.7 Conclusions

Due to engine restrictions, the only feasible solution for laser delivery to engine components was through the use of fibre optics. Investigations were performed to establish the maximum energy that could be coupled and be delivered to the phosphor target. Various transmission losses and fibre damage mechanisms limit the amount of energy that would be transmittable through the fibre. This has significant implications on the performance of the phosphor thermometry technique, with larger energies allowing greater emission intensities and a higher temperature capability. These mechanisms include gas breakdown, surface preparation, fibre bending and thermal damage. The section investigated a number of variables, including the effect of the launch angle, effect of wavelength, fibre position, converging and diverging beams, beam diameter and the subsequent laser energy, fluence and peak power density experienced at the fibres face. During this task, a number of relations were derived for assessing the performance of the transmissions. The maximum amount of energy/pulse transmittable through a 1.0mm fused silica fibre was 8.7mJ for 355nm This relates advantage x2.9 when transmitting 355nm and 3mJ for 266nm. wavelengths. It may be possible for the fibre to transmit more energy if higher input energies were permissible. It was found that fused silica fibres performed better than sapphire fibres in delivering UV light. The use of a DOE beam homogeniser could offer the potential to allow higher transmissions, however it was estimated that a energy of 28mJ would be required, which exceeds the limits of the exiting laser system, and therefore in the present investigation, the DOE did not allow more energies to delivered. Using a smaller fibre and lower laser energy levels and inferring the maximum power through a 1mm diameter fibre was not a sensible option because the minimum beam waist that was achievable with the DOE would exceed the input area of the fibre. Based on the setup, the launching angle of approx 0.38NA performed better than the launch angle of 0.75NA, and more energy could be coupled in to the fibre when the launching beam was diverging when it entered the fibre.

# **5 Bonding Methods and Spectral Analysis**

## 5.1 Phosphor Bonding Methods

#### 5.1.1 Introduction

Adhering the phosphor to the surface of interest is vital for the successful application of phosphor thermometry. The method should be chemically compatible with the substrate and should not alter the spectral and thermographic properties of the phosphor. The method should be durable and capable of surviving the exposed environmental conditions, including the maximum operating the temperature. This section reviews various bonding techniques and highlights the key advantages and limitations for each method. A summary of each method is shown in Table 19.

## 5.1.2 Chemical Bonding

This process involves mixing powdered phosphors with chemical bonding agents to create a paint that can be either brushed or air-sprayed on to a surface. This allows large areas of various sizes and shapes to be covered easily. However, maintaining a uniform surface and controlling the thickness and roughness can be difficult. Tests indicate a variation in intensity across different test pieces [162]. The minimum coating size that can be produced is around 10  $\mu$ m (typical is around 30-60  $\mu$ m). This is relatively large compared to the vapour deposition and plasma spraying techniques. Greater thicknesses provide greater thermal gradients between the phosphor coating and substrate, constituting to a greater error in measurement. The differences in thermal expansion cause the paint and substrate to expand at different rates which causes the paint to fake off, especially at high temperatures. According to Allison *et al.* [163], one of the most challenging surface for bonding is high strength nickel alloy due to high differences in the thermal expansion coefficients.

With chemical binders there is a possibility of changing the phosphor's atomic configuration, and hence luminescence and thermographic properties. Ideally, chemical binders should suspend the phosphor without changing atomic properties. Chemically bonded phosphors require curing by raising the temperature to 700°C and slowly bringing it back down to room temperature. The exact curing method depends on the binders being used. Epoxy binders have a temperature limit that is reached at a few hundred degrees. Apart from high temperature capability, chemical binders must also allow optical transmission characteristics to enable phosphor excitation and emission detection.

In the past few years, a variety of commercially available binders have been investigated [163-165]. Propriety binders manufactured by thermal paints experts at Rolls-Royce Plc have been successfully tested up to 1,100°C [51]. Some of the higher surviving commercially available binders include ZYP-ZAP and Coltronics-Resbond, and have shown survivability with fluorescence detection up to 1,600°C [166]. Table 18 compares some of these binders.

Binder	Composition	Max temp (°C)
Sperex SP115	Silicone Resin	1,000 °C
Sauerisen thinning	Soluble sodium silicate	1,204 °C
liquid	Water based	
ZYP – BNSL	Glassy carbon and magnesium	
	aluminium silicate	
	Alcohol and acetone based	
ZYP – HPC	magnesium aluminium silicate	1,500 °C
	water based	
ZYP – LK	75% SiO2, 20% K2O, 5% LiO2.	1,100 °C
	Water based	
ZYP-LRC	Water based	1,400 °C
ZYP – ZAP	water-alcohol-based binder	1,600 °C
Coltronics Resbond	791, 792 Silicate Glass,	1,600 °C
	793 Silica Oxide	
	795 -Alumina Oxide	

Table 18: Comparison of various high temperature chemical binders [166]

A major problem associated with chemical binders is the effects due to thermal exposure. Figure 90 illustrates the reduction in emission intensity of  $Y_2O_3$ :Eu phosphor with Resbond 793 binder at 1,400°C after 4 hours of thermal exposure [166]. Similar results were reported by Ranson *et al.* [167] using a different chemical binder. The results showed a reduction in intensity to approximately 10% of their initial value following thermal exposure at 1,200°C for two hours. The reason for this could be simply due to the paint layer flaking off. Another explanation reports the paint transitioning to a yellow/brown colour, and is still unclear whether it is problems with the optical transmission of UV used to excite the phosphor, or the emissions from the phosphor, or a combination of the two. Thermal exposure for long periods may force chemical reactions within the phosphor changing its characteristics, which may be responsible for the further decline in intensity.



Figure 90. Left: emission spectra of  $Y_2O_3$  phosphor in Resbond binder after thermal exposure to 1,400°C [166]. Right: Intensity of the peak emission of  $Y_2O_3$  phosphor after thermal exposure to 1,200°C [167].

## 5.1.3 Vapour Deposition / Rf Sputtering

In this process, a coating is applied by vaporising the phosphor and allowing it to condense on the surface of interest. There are a variety of ways this can be achieved including electron beam (EB-PVD), pulsed laser disposition (PLD), chemical vapour deposition (CVD) and RF-frequency sputtering. The resulting coatings are very robust and long-lived with fluorescent intensity being constant throughout its life [167]. The coatings can be made very thin compared to chemical binder paints, and can be finely controlled to have a uniform surface finish. Unfortunately, deposition processes can be very expensive with limited coverage capability.

As no chemical binders are required, there are no chemical interference or problems concerning optical transmissions of phosphor excitation (UV) and emission detection. However, during vapour deposition process, dopant atoms can be situated in a variety of positions and rotations within the hosts crystal structure and therefore experience a variety of crystal field effects, leading to weaker and wider spectral emissions. Post annealing is required to realign the ions to restore crystalline quality and increase luminescent intensity. Allison *et al.* [42] notes that the high temperatures generated during vapour deposition can irreversibly break down some phosphors, such as oxysulphides with post annealing offering no benefit.

Ranson *et al.* [167] investigated thin coatings  $(0.1\mu$ m $-3\mu$ m) produced by RF sputtering and thick coatings (approx 10  $\mu$ m) produced by binder paints. They found that emission intensity from RF sputtering was related to coating thickness, and it was shown that 0.7  $\mu$ m post annealed coatings provided the same level of intensity as thick coatings (approx 10  $\mu$ m) produced by chemical binders (Figure 91). While thick coating produced by binder paints show declining intensity with thermal exposure,

Ranson *et al.* [167] showed that thin coatings produced by vapour deposition show no decline in intensity level. Figure 92 compares the variation in luminescent intensity as a function of hours of constant thermal exposure at 1,200°C. The results show that the binder paints showed a very quick decline in intensity, whilst thin coatings produced by vapour deposition showed no change.



Figure 91: Intensity of thin coatings (0.1  $\mu$ m-3  $\mu$ m) after annealing at 1,200°C compared to thick film (10  $\mu$ m) and powdered Y<sub>2</sub>O<sub>3</sub>:Eu phosphor [167].



Figure 92: The variation in intensity with time (hours) with constant thermal exposure of  $1,200^{\circ}$ C for thin coatings ( $0.1 \ \mu$ m- $3 \ \mu$ m) produced by RF sputtering and thick film coatings ( $10 \ \mu$ m) produced by chemical binders [167].

#### 5.1.4 Flame and Plasma Spray

Flame spraying is portable and applicable to objects of diverse geometries, but has a lower impact velocity than plasma spray [168]. Plasma spraying involves injecting powdered phosphors into an electrical discharge to liquefy them. The technique allows robust, well-adhered coatings made up from a multiplicity of solidified droplets [51]. It is usually used to produce thicker films than vapour deposition methods and can produce coatings exceeding a millimetre. Like vapour deposition, annealing is also necessary to restore crystalline quality and improve luminescent efficiency

# 5.1.5 Discussion

A summary of the main findings for each technique is shown in Table 19.

Chemical Bonding	<ul> <li>Mixing powdered phosphors with chemical bonding agents may change the phosphor's atomic configuration, and possibly thermographic properties.</li> <li>Usually requires curing to 700°C.</li> <li>Differences in thermal expansion cause paint and substrate to expand at different rates, causing the paint to flake off.</li> <li>Declining intensity with increased thermal exposure [46-48].</li> </ul>
Vapour Deposition (EB-PVD,PLD,CVD) / RF sputtering	<ul> <li>Coatings are very robust and long-lived</li> <li>Fluorescent intensity being constant throughout its life with no decline in intensity with increased thermal exposure [47].</li> <li>Coatings are thinner than chemical binders.</li> <li>Annealing necessary to restore crystalline quality. and improve luminescent efficiency.</li> <li>Expensive.</li> <li>Limited coating area coverage.</li> </ul>
Flame and Plasma Spraying	<ul> <li>Used to produce thicker films than vapour deposition techniques.</li> <li>Annealing necessary to restore crystalline quality and improve luminescent efficiency.</li> </ul>

Table 19: Summary of the key issues related to different bonding techniques

A number of binders were investigated and trialled for suitability with a number of phosphor including YAG:Tm, YAG:Tb and  $Y_2O_3$ :Eu phosphors. The binders were:

- Proprietary binders produced by the Thermal Paints Lab at Rolls Royce  $\operatorname{plc}$
- ZYP ZAP
- ZYP HPC
- Epoxy Binders

Key information, including the chemical composition and curing and outgas temperature for a variety of binders that have been previously used for phosphor thermometry are summarised in Table 20.

	ZAP	GEL-P	НРС	LRC	BNSL
Max Temp (°C)	1800	1500	1500	1800	1600
Hardness	HIGH	HIGH	LOW	LOW	MED
Water resistance	HIGH	HIGH	LOW	LOW	MED
Ability to suspend solids	LOW	HIGH	MED	HIGH	MED
Composition	53.5%: P <sub>2</sub> O <sub>5</sub> 46.5%: Al <sub>2</sub> O <sub>3</sub>	40%: P <sub>2</sub> O <sub>5</sub> 30%: Al <sub>2</sub> O <sub>3</sub> 30%: SiO <sub>2</sub>	100% Magnesium Silicate	100% Al <sub>2</sub> O <sub>3</sub>	Glassy Carbon/ Magnesium silicate
Liquid carrier	Water + Alcohol	Water + Alcohol	Water	Water	Alcohol
Cure temperature	350 °C	350 °C			
Outgas temp	600 °C	600 °C	150 °C	600 °C	150 °C
Out gas products	H <sub>2</sub> O/NO <sub>X</sub> /CO <sub>X</sub>	H <sub>2</sub> O/CO <sub>X</sub>	H <sub>2</sub> O	H <sub>2</sub> O/NO <sub>X</sub>	CO <sub>X</sub>
pH	2-3	2-3	7	2-3	2-4

Table 20: Summary of key characteristics of commercially available binders from ZYP Coatings A ratio of approximately 30:70 phosphor:binder by volume was used to make paints suitable for airspray, and approximately 50:50 to make phosphor pastes suitable for brushing. A minimum of 3g phosphor was required to make sufficient paint for mixing and airspray. After initial shaking for approximately 5 minutes, a magnetic stirrer was used to mix the paint for approximately 2 hours. Paints made by at Rolls Royce plc was pre-prepared by experts at the Thermal Paints Lab ready for air-spray. Prior application of the phosphor paint, the substrate's surface was roughened using P80 aluminium dioxide grit paper, and cleaned using acetone. A thin layer of paint was airbrushed on to the surface, and was allowed to dry for approximately 3 hours. The sample was then placed in a furnace heated to 100 °C at a slow rate of approximately 1 °C /min. The samples were then heated to the curing temperature of approximately 350 °C for 30mins, and to the outgas temperature (600 °C) at a rate of approximately 10°C/min. The temperature was further raised to 700 °C for 10 minutes before they were allowed to cool. It was found that raising the temperature too quickly caused bubbling as gas escaped from the paint too quickly, creating poor adhesion consequently making the coating to flake off quicker.

It was found that the furnace had to be slowly cooled by gradually decreasing the thermoset values to lower temperatures; if the paint were cooled too quickly, eg by opening the furnace door, the chance of the paint flaking was high. This is due to differences in the expansion thermal coefficient that caused the coating and substrate later to expand and contact at different rates. The likelihood of this failure was increased with thicker films produced by paint brushing.

When iron/steel substrates were used, the paints flaked off quicker compared to nickel alloy substrates. Compared to the other binders, it was found that ZYP-HPC binders was the most prone to this type of failure. The reason for this is oxide layers being formed between the substrate and the paint, weakening the adhesion to the surface. For stainless steel, this was true after approximately 900 °C. Heating in a oxygen free environment, or using furnaces filled with an inert gas could reduce this type of failure.

ZYP HP binder produced a coating that could be scraped off easily, and would therefore not survive harsh environments. The paints produced by RR binders and ZYP ZAP produced much harder wearing coatings. For ZYP-ZAP, after exposure to 1100°C, the paint was transitioning to a yellowy/brown colour. These irreversible effects reduced the intensity of the luminescence emissions. Although manufacturer guidelines suggest a temperature capability up to 1800 °C (Table 20), with other researchers reporting success with thermographic phosphor paints up to 1600 °C [166], tests undertaken on YAG:Tm on metallic surfaces had a maximum temperature capability with luminescence being detectable at approximately 1250 °C – 1300 °C. It was difficult to establish the cause of the failure, as opening the furnace door to inspect the surface caused the paint to flake off.

A phosphors intensity response usually decreases with increasing temperature. Because the binder paints also exhibited a decline in intensity that was a function of thermal exposure, it was difficult to discriminate whether it was the phosphors response or whether it was the caused by thermal exposure. Vapour deposition capability was not available at the in the University's department and was considered too expensive for initial investigation on different phosphors. For these reasons it was decided to that powdered samples pressed on to the substrate would be used in the investigation. These showed no variation in intensity with thermal exposure, and it is envisaged that the results would be similar to those produced by vapour deposition methods that are likely to be employed on engine components under engine environments. The results by Ranson [167] indicate that powdered samples only exhibited slightly higher intensity values (by approx 33%) compared to vapour deposition techniques with coatings greater than  $0.7 \,\mu m$ . It is interesting to note that although the intensity response of the phosphor changed using chemical binders, there was no change in lifetime decay constant values when compared to data from powdered samples under both 355 and 266nm illumination.



Figure 93: Temperature variation across the section of a TBC deposited on a substrate. Taken from [169]

The phosphor coating, regardless of thickness, may possess sufficient heat capacity and thermal conductivity to alter its thermal environment, exhibiting a certain level of intrusiveness. This may not be a problem at ambient temperatures, where heat fluxes are low and the effects of blackbody and emissivity are negligible. However, in gas turbine environments, it may be necessary to develop a thermal model to determine whether heat transfer will impose a limit to the accuracy of the measurement [28]. If
thick coatings are used then the temperature difference between the substrate and the phosphor top could be very large. The thickness of YSZ thermal barrier coatings on top of nickel alloys bondcoats are approximately  $200\,\mu m$ , and the surface temperature on top of the YSZ TBCs under maximum operating engine conditions with thin film cooling are estimated to be approximately 200 °C higher than the substrate temperature. Another reference estimates drop of 170°C over a TBC coating of  $150 \,\mu m$  [170] at the maximum operating conditions of the HP turbine. This equates to approximately  $1-1.2^{\circ}C/\mu m$  if a linear relationship is assumed. If the same thermal conductivity of TBC is assumed for thermographic phosphors, (  $\kappa$  for YAG and YSZ = 3.2 W/mK [171], and 2.5-4.0 W/mK [172]), then a  $1 \mu m$  coating produced via vapour deposition techniques will provide a temperature difference of approximately 1°C between the top and bottom surface. At 1400 °C (1673K), this is approximately 0.05%, and is considered to be very small. However, thick coatings of approx 30  $\mu m$ produced by binder paints will produce a temperature difference of approximately 30°C, (assuming the same thermal conductivity), which will produce a significant difference that is as large as 1.79%. Based on these assumptions, and due to the increased durability of bonding especially under harsh engine environments, the use of vapour deposition techniques to produce thin durable coatings of thickness of  $1 \mu m$ would be the most promising option.

## 5.2 Emission Spectral Analysis.

#### 5.2.1 Introduction

This section reports the spectral emissions of various thermographic phosphors following pulsed laser excitation at various temperatures. The purpose of the investigation was to understand the spectral response with increasing temperature, in order to optimise the measurement system setup. Although spectral analysis has been previously reported at relatively low temperatures, there has not been much work reporting the spectral analysis at high temperatures. As a result of the thermal expansion of the crystal lattice, there may be linewidth variation and peak emission wavelength shifts with increasing temperature, as illustrated in Kusama et al.[96]. If this is the case, then fixed wavelength analysis may not provide the optimum setup for detecting temperatures at higher temperatures. There may also be other emission peaks that may be dormant at low temperatures, and be more active at higher temperatures which could potentially be used to determine temperature. The aim of this investigation is to:

- Identify peak emission lines
- Observe wavelength shifts with increasing temperature
- Observe other emission lines that can be used to determine temperature

This information can provide a better understanding which would to help optimise system setup, enabling the use of correct narrow band interference filters, and improve the overall performance of a phosphor thermometry system.

#### 5.2.2 Experimental Methodology

The phosphors that were investigated were YAG:Tb,  $Y_2O_3$ :Eu and YAG:Tm. The schematic is shown in Figure 94. A third harmonic Nd:YAG, with an energy approximated to be 9mJ/pulse with a repetition rate of 15Hz, was used to excite the phosphor target. The phosphor emissions were focused onto a fibre optic tip, which delivered the light to an Princeton Instruments ICCD spectrometer. A grating spectrally dispersed the collected light onto the ICCD camera that was fitted on to the exit port of the spectrograph. The ICCD was nitrogen and water cooled for thermal stability. Experimental parameters such as the spectral window, exposure time, intensifier gain and triggering mode were all configurable using a combination of the spectrometer was calibrated using a tungsten light bulb, provided, and the diffraction sensitivity was set using an Hg/Ar calibration lamp.

A Vectsar VF1 furnace, modified for optical access, was used to heat the phosphor samples in 100°C intervals. A minimum of 30 minutes was allowed for the temperature to stabilise inside the furnace before readings were taken. The results from 200 pulses were added together using the cameras software. Matlab was used to normalise, analyse and present the results. Matlab also enabled the subtraction of the blackbody and dark current for each reading. 2D and 3D spectral response curves with increasing temperature were plotted. These were normalised to the maximum intensity that was observed throughout the entire temperature range. A 2D contour map was also created in Matlab to determine the emission intensities relative to the peak emission intensity at that temperature.



Figure 94: Schematic for spectral emission analysis.

The spectroscopy system had another 3D feature to allow the simultaneous spectral and temporal information from an individual event. This feature could allow the user obtain the lifetime decay curve from phosphor emission at each wavelength, and establish whether any emission profile at different wavelengths are decaying at different rates. Unfortunately, after an initial feasibility experiment, due to the low intensities and fast decay profiles of phosphor emissions, it was found that the apparatus was unsuitable to provide such data accurately.

### 5.2.3 Excitation and emission spectra

The following excitation and emission spectra, shown in Figure 95 to Figure 97, for various phosphors ( $Y_2O_3$ :Eu,  $Mg_3F_2GeO_4$ :Mn, YAG:Tm) were obtained at room temperatures for the University of Manchester by Phosphor Technology Ltd. However, these do not show the emission variation with increasing temperature. The next section discusses the results for the emission spectra for various phosphors with increasing temperatures.



Figure 95:  $Y_2O_3$ :Eu- Excitation and emission spectra – Obtained by Phosphor Technology Ltd



Figure 96:  $Mg_3F_2GeO_4$ : Mn – Excitation and emission spectra – Obtained from Phosphor Technology Ltd.



# 5.2.4 Results and Discussions



Figure 98: YAG:Tb - Normalised 2D plot of spectral response at various temperatures



Figure 99: YAG:Tb - Normalised 3D plot of spectral response at various temperatures



Figure 100: YAG:Tb – Normalised contour intensity plot throughout the entire temperature range



Figure 101: YAG:Tb – Contour intensity plot normalised to max intensity at a single temperature



Figure 102: YAG:Tb - Magnification contour plots of emission line



Figure 103: Y<sub>2</sub>O<sub>3</sub>:Eu - Normalised 2D plot of spectral response at various temperatures



Figure 104:  $Y_2O_3$ :Eu - Normalised 3D plot of spectral response at various temperatures



Figure 105:  $Y_2O_3$ :Eu - Contour intensity plot normalised to max intensity at a single temperature.



Figure 106: YAG:Tm - Normalised 2D plot of spectral response at various temperatures



Figure 107: YAG:Tm - Normalised 3D plot of spectral response at various temperatures



Figure 108: YAG:Tm – Normalised contour intensity plot throughout the entire temperature range



Figure 109: YAG:Tm – Contour intensity plot normalised to max intensity at a single temperature

The emission spectra results for YAG:Tb are shown in Figure 98 to Figure 102. For  $Y_2O_3$ :Eu they are shown in Figure 103 and Figure 105, and for YAG:Tm they are shown in Figure 106 to Figure 109. Peak emission for YAG:Tb,  $Y_2O_3$ :Eu and YAG:Tm was shown to be 544nm, 611nm and 458nm respectively. The spectral FWHM linewidth of the emissions was between 5-10nm for YAG:Tb and  $Y_2O_3$ :Eu and approximately 15nm for YAG:Tm.

The maximum temperature that was observable using the spectrometry system and existing setup for YAG:Tb,  $Y_2O_3$ :Eu and YAG:Tm was approximately 900 °C, 800 °C and 1200 °C respectively. It was found that there were no observable shifts in the emission wavelength with increasing temperature for any of the phosphors investigated. Researchers that have used YAG:Tb thermographic phosphors to measure temperate have only utilised the 544nm emission peak. However, it has been shown that YAG:Tb has another emission line at 490nm. Although this emission line is known to exist, no research has been conducted to reveal temperatures using this wavelength. Although the intensity of the 544nm emission line was greater than the 490nm emission line by approximately x2, the 490nm is worth investigation as it may provide benefits over the 544nm wavelength in terms of blackbody radiation.

# 6 Intensity measurements for phosphors

## 6.1 Absolute intensity measurements

### 6.1.1 Introduction

There are a number of issues related to high temperature phosphor thermometry measurement which includes: a.) decreasing phosphor intensity values, b.) faster decays, and c.) rising levels of blackbody radiation, that will impose limits on the maximum detectable temperature. A problem for design engineers attempting to use phosphor thermometry is the lack of phosphor characteristic information in the literature to help aid optimal setup. Although absolute lifetime decay values can be found, intensity values are only presented as relative arbitrary measurements, and in most cases are not presented at all. To the best of our knowledge there has been no work presenting a thermographic phosphor's absolute intensity response throughout its useful temperature thermographic phosphors, promoting the gathering of such data. Obtaining absolute values serves many interrelated useful purposes including:

- a.) Provides a judicious evaluation of the intensity performance of different phosphors to evaluate the real advantage of signal-to-blackbody radiation at high temperatures for various phosphors with different peak intensities and emission wavelengths. Although lower peak emission wavelengths are more beneficial in terms of blackbody radiation, a lower emission intensity may neutralise or even reverse this.
- b.) Facilitate the phosphor selection process.
- c.) Evaluate the performance of a detection system/probe design configuration, which can be used to estimate the resultant radiant power that would be observable at the detector's active area. Permit a better understanding of the scale and range of measurements that can help optimise system setup, and aid the in optical instrumentation selection process, taking into account both the phosphors temporal and intensity response to maximise performance. This can also be used to estimate the input laser power requirement that would be necessary to produce emissions of certain intensity.
- d.) Since measurements are not relative, absolute measurements can permit comparisons made from independent investigations from other scientific institutions using different optical configurations and apparatus.
- e.) Can provide an insight to the physical luminescence processes at high temperatures.

#### 6.1.2 Methodology

The lifetime decay mode relies on pulsed excitation source, usually delivered using high-energy pulsed laser sources because they provide peak powers that are orders of magnitude higher than continuous mode light sources. For this reason, researchers also investigating other response modes, such as the 'intensity ratio', also use high energy pulsed laser sources [70, 74, 80]. The authors reviewed numerous ways to measure the phosphor's intensity and quantum efficiency, with a number of sources suggesting the use of an integrating sphere that relies on a continuous light source [173] [64]. However, luminescence mechanics, energy and power scales from continuous illumination sources are usually different from those produced by pulsed sources, and therefore the knowledge gained from such approaches is not beneficial. Also, it is envisaged that continuous light sources would not produce enough phosphor emissive power to enable discrimination against the high blackbody radiation at higher temperatures.

A detection methodology may involve measuring the total optical energy from an emission, using energy meters. The total energy can be obtained by integrating under the luminescence decay curve. However, this method does not necessarily indicate a phosphor's capability of being detected. A less intense phosphor emission with a longer lifetime would result in higher energies, yet be less detectable compared to a very intense emission with relatively faster decay lifetimes, as demonstrated in Figure 110. A better method would be measure the radiant flux per steradian (intensity). This allows optical power levels to be estimated for any optical collection configuration, and permit assessment on whether the phosphor emission's optical power would be sufficient to enable detection.



Figure 110: Comparison of energy (area under curve) from phosphor emissions with: a.) intense, but relatively short lived lifetimes (faster decays), b.) less intense, but relatively long-lived lifetimes (slower decays)

The phosphors investigated were:

- YAG:Tm 0.1% atomic percentage activator concentration, Phosphor Technology Ltd, UK
- YAG:Tm 0.5% atomic percentage activator concentration, Phosphor Technology Ltd, UK
- YAG:Tb, 3% activator concentration
- Y<sub>2</sub>O<sub>3</sub>:Eu, 3% activator concentration
- Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn, Phosphor Technology Ltd, UK

There are many often confusing measurements units that are interchangeably used in the literature. A useful reference for the optical terminology is summarised in Table 21. Radiometry is concerned with the absolute amount of light; whereas photometry is the measurement of light as the human eye perceives it due its different wavelength sensitivities, and is mainly used for instruments designed for humans e.g. light bulbs, LEDs and displays. Only radiometric measurements are relevant for phosphor thermometry, and although it may appear for some phosphor emissions to be more intense to the human eye, it may not be the case, therefore photometric units should be avoided. The radiant flux is the amount of radiant optical power that is observable, whereas the intensity is the flux per unit solid angle. The solid angle, used for defining 3D angles, is similar in concept to the radian used for defining 2D angles, and is defined as the ratio of the surface area that subtends from the centre of a sphere to the radius<sup>2</sup>, illustrated in Figure 111.

	Radiometric	Photometric
<b>Power</b> Flux.	Radiant flux (Watts)	Luminous Flux (lumens)
<b>Illumination</b> Flux received per unit area.	Irradiance (W/m²)	Iluminance (lux or lumen/m <sup>2</sup> )
<b>Intensity</b> Flux emitted per unit solid angle.	Radiant Intensity (W/sr)	Luminous intensity (Candela or lumens/sr)
<b>Brightness</b> Flux emitted per unit solid angle per unit area.	Radiance (W/sr/m²)	Luminance (Candela/m <sup>2</sup> or lumens/ sr.m <sup>2</sup> )

Table 21: Reference for common radiometric and photometric terminology



Figure 111: Solid angle

The method employed in this section uses a PMT to measure the optical phosphor emission power, calibration and additional calculations, described in the next sections, were used to obtain absolute intensity values. The schematic shown in Figure 112 was used to collect the data. A 7mm, 12ns, 15Hz, third and fourth harmonic Q-switched Nd:YAG laser beam was used to excite the phosphor. The laser provided an adjustable energy up to 15mJ/pulse and 5mJ/pulse for 355nm and 266nm respectively. The resultant emissions were detected using an Electron Tubes 9954B PMT which had an active diameter of 52mm, and was located approximately 420mm from the phosphor. An adjustable iris was placed on top of the PMT, where the opening diameter could be altered between 2-40mm and be used to effectively alter the solid angle ( $\Omega$ ).

Narrowband interference filters (8nm FWHM, 45% transmission) centred at peak phosphor emission wavelengths were placed in front of the PMT to isolate signals from other unwanted emission wavelengths, reflected laser radiation, and blackbody radiation. A 200MHz USB Picotech 3206 oscilloscope was used to digitise and record the measurements. To prevent ringing effects, the input was terminated with a 50 $\Omega$ resistor to match the impedance from the output of the amplifier. A beam-splitter was used to reflect the UV laser beam to the target whilst allowing the transmission of phosphor emissions. This enabled the beam and the detector to remain on the same optical axis.

A modified Vecstar high temperature furnace, powered by six silicon carbide rods, with a maximum temperature capability of 1600°C was used to heat the phosphor. To obtain optical access, a 15mm diameter hole was drilled through the 150mm back wall insulation, and was sealed using a 5mm thick quartz window to reduce heat losses that could potentially affect thermal stability and limit the maximum temperature capability. The temperature was regulated using a closed loop PID controller located on the front panel of the furnace, and an internal thermocouple. Two independent thermocouples (Type K and Type N) were also installed to monitor the temperature to ensure thermal uniformity and stability. It was found that the factory fitted thermocouple used to control the furnace temperature had slow response times and consequently resulted in an overshoot in temperature that was measureable from the other faster more responsive thermocouples. However, after a period of approximately 15 minutes, temperature readings amongst the thermocouples were stabilised within 0.5% to each other, indicating good furnace uniformity and thermal stability. Since the furnace is designed for high temperature capability, it was difficult for the furnace to control/maintain temperatures below 300  $^{\circ}$ C.

Powdered phosphor samples were pressed onto a porous ceramic firebrick. The same volume was pressed over an area of approx 25x25mm to ensure the thickness and distribution of the samples was the same. The use of binder paints introduced irreversible effects with temperature making it difficult to monitor/measure intensity and was therefore not investigated. This was in agreement with tests conducted in references [64, 174, 175]. There were no observable irreversible effects using powdered samples, and it is envisaged that the results from this investigation would be similar to those produced by vapour deposition or plasma spraying methodologies.

Four different laser settings producing a different energy/pulse were used to observe the effects of increasing laser energy level. The laser energy was periodically monitored using a Gentec laser power meter, and throughout the experiment minor adjustments were required to ensure the energy/pulse remained constant. An average of 100 laser pulses was used to reduce the error caused by pulse to pulse variation, which can be over 14%. To estimate the repeatability of results and ensure concordance, tests were conducted using 3 independent samples. Tests were undertaken with the temperature incremented in 100°C intervals between the range 100- 700 °C, and in 50 °C intervals after this up to the maximum temperature where emissions were detectable using the setup. The furnace was allowed to stabilise for 20 minutes before readings were made. For each temperature, all the relevant information such as laser energy, distance to target, angle, PMT gain voltage, dark current, blackbody radiation and peak intensity were recorded for post processing. The determined values were then further processed to provide a unit of measurement that was 'intensity per mJ of laser excitation'.



Figure 112: Schematic for high temperature intensity investigation

#### 6.1.3 Effect of distance, detector area and detection angle

A number of pre-investigations were performed to analyse and understand the luminescent nature of thermographic phosphors, and establish whether emissions can be modelled as lambertian point sources where intensity readings are independent of distance and detection area. For point sources, the detected power is proportional to the detection area and follows the inverse square law with distance. Since lambertian or diffusively reflecting materials transmit with equal radiance in all directions, the radiant flux varies proportionally to the cosine of the angle subtended to the normal surface. The optical intensity is defined as the radiant power transmitted over one solid angle. The solid angle in this investigation was approximated to be:

Equation 25

 $\Omega = \frac{a_d}{x^2}$ 

As the solid angle takes in to account detection area and the distance, the radiant intensity is expected to remain constant. Both the effects of detector area and distance were investigated, with the setup shown in Figure 113. For simplicity the angle remained at  $0^{\circ}$ , where  $\cos(0)=1$ . The angle of the laser beam was set to the same optical axis using the beamsplitter. The investigation results, shown in Figure 114 and Figure 115, illustrate that the detected emissive power varies proportionally with the detectors collection area, and inversely proportional to the distance squared, as expected. By taking these effects into account using the solid angle, it was shown that the intensity (radiant flux per solid angle) remained constant; illustrating phosphor emissions created using a relatively small laser excitation area could be modelled as being point sources.



Figure 113: Schematic for investigating the effect of distance and detector area



Figure 114: Radiant Flux- Effect of: a.)distance, b.)detector diameter, c.)detector area



Figure 115: Intensity- Effect of: a.)distance b.)detector diameter, c.)detector area

Ranson [64] investigated the angular distribution of emission. The results indicated in Figure 116 show the observed emission power is proportional to the cosine of the detection. The effects of the laser's firing angle, and its effects on the angular distribution of emissions was also investigated. The results indicate that the laser's firing angle had no effect on the emission's angular distribution. As these effects have already been investigated, no further additional tests on this were conducted.



 $\Box 60^{\circ} \quad \diamond 50^{\circ} \quad \Delta 40^{\circ} \quad \times 30^{\circ} \quad \ast 20^{\circ} \quad \circ 10^{\circ} \quad + 0^{\circ}$ 

Figure 116: Angular distribution of emission at various laser angles. [64]

#### 6.1.4 Conversion to radiant flux and intensity

PMTs are extremely sensitive detectors and have been the most widely used instrument for phosphor thermometry. They have relatively large detection areas and can offer high gains with superior SNR. In order to effectively evaluate absolute radiant flux and intensity values, it is important to understand the PMT's physical behaviour. In general, photons strike a photo emissive cathode which emits electrons by the photoelectric effect. These electrons are focused and accelerated towards a series of additional electrodes (dynodes) that generate additional electrons that are collected at the anode. The cascading effect creates 10<sup>5</sup> to 10<sup>7</sup> electrons for each photon hitting the first cathode.

**Gain:** The PMT dynode gain ( $G_d$ ) is derived by current amplification given by the product of the individual dynode contributions, illustrated in Equation 26, and is related to the energy of the incident electrons, and hence to the inter-dynode voltage [176].

 $I_{anode} = d_1 d_2 d_3 \dots d_n I_{photocathode} = G_d. I_{photocathode}$ Equation 26

Using a constant light source at various wavelengths and observing the PMT output as the voltage was gradually increased revealed a relationship between the supplied dynode voltage (V<sub>d</sub>) and the dynode gain (G<sub>d</sub>), and was approximated to relation shown in Equation 27. This relationship closely matched the manufacturer's data sheet curves for that particular PMT. Experimental testing at different wavelengths of light, and taking into consideration instrument uncertainties, and statistical analysis revealed a standard uncertainty (confidence level of 68%) of approximately  $\pm 3.5\%$  at 1000V rising to approx  $\pm 7.1\%$  at 1400V.

 $G_d = 2.V_d^{11.175} .10^{-30}$ 

Equation 27

Equation 28

**Photocathode Sensitivity and Collection Efficiency:** The photocathode sensitivity describes the conversion efficiency for photons into photoelectrons at various wavelengths, and is determined using quantum efficiency spectral response curves provided by the manufacturer and the relation shown in Equation 28 [176].

$$P_s(\lambda) = \frac{\eta . \lambda}{1.24}$$

Quantum efficiencies (  $\eta$  ) from manufacturer datasheets also include optical transmission and reflection losses associated with the PMT window. However, it does not include all losses required for determining an absolute measurement. There are a number of scenarios, highlighted in Wright [177], showing electron trajectories that do not reach the first dynode. Also, quoted efficiencies are generally tested under diode operation. Therefore in reality the overall efficiencies are always lower, and given by the product of  $P_s$  and collection efficiency ( $E_c$ ).  $E_c$  is dependant on the structure and operating conditions of the PMT, and is normally not specified; Monte Carlo simulations predict  $E_c$  values to be in the region of 70-95% [177], and various research based on experimental measurements on the same PMT tube, have reported efficiencies of 64%[178], 86% [179], 91% [177] highlighting the difficulty in making these measurements. To permit confidence in measurements in this investigation as manufacturers datasheets do not capture all the efficiency losses along the path from light to voltage conversion, independent experimental tests using a calibrated meter or light source of known optical power, is required to scale and correct collected data to provide absolute measurements. The authors conducted independent experimental tests using a 24V DC constant current light source, a calibrated Molectron S103 Sigma optical power meter and a series of interference filters. This was used to initially characterise the absolute intensity of the white light source at various wavelengths and then used to evaluate the PMT's actual efficiency (Ps.Ec). A white light source was preferred to single narrowband LEDs because they cover a wider spectrum permitting the characterisation of all the wavelengths of interest. Using the relevant interference filter that matches the phosphor's peak emission permits the direct calibration of the PMTs response at these wavelengths directly, simplifying analysis and avoiding the need to produce a full spectral response of the PMT and using interpolation to determine the response.

Table 22 compares conversion efficiencies obtained from the calibration to those predicted by the manufacturers' datasheets. Although it is clear that the PMT's photocathode sensitivity (Ps) is wavelength dependent, as shown in Equation 28, it

appears that the collection ( $E_c$ ) may also be wavelength dependent. The uncertainty of the calibration, taking into consideration instrument uncertainties and statistical variations was found to be approximately 6.04%.

Phosphor	λ (nm)	$E_c.P_s$ (mA/W) Estimated using quoted QE values and $E_c = 0.6$	E <sub>c</sub> .P <sub>s</sub> (mA/W) Determined from experimentation
$Mg_3F_2GeO_4:Mn$	660	1.6	0.7
$Mg_3F_2GeO_4:Mn$	630	4.57	1.5
Y <sub>2</sub> O <sub>3</sub> :Eu	611	7.38	3.5
YAG:Tb	544	26.32	22
YAG:Tb	490	48.39	36
YAG:Tm	458	55.40	50

Table 22: Determined PMT Ps.Ec values

**PMT's time response:** The PMT's electronic behaviour must be understood to ensure waveforms are faithful representations of reality. PMTs are modelled as ideal current sources in parallel with an output resistance  $R_0$  (>10<sup>12</sup> ohms) and capacitance  $C_0$  (5-20pF – typical 10pF), as shown in Figure 117 [180, 181]. The measured output depends on the load resistance ( $R_L$ ) and capacitance ( $C_L$ ) in combination with  $R_0$  and  $C_0$ , illustrated in Equation 29 and Equation 30. The time constant of the circuit ( $\tau_c$ ) can be determined using Equation 31. The bandwidth ( $B_w$ ), rise time ( $T_r$ ) and time constants ( $T_c$ ) are closely related terms and are determined using the relations shown in Equation 32 to Equation 34.

$R = \frac{R_0 R_L}{R_0 + R_L}$	Equation 29
$C = C_0 + C_L$	Equation 30
$T_c = RC$	Equation 31

$$B_W = \frac{1}{2\pi T_c} = \frac{\ln(9)}{2\pi T_r}$$
 Equation 32

$T = T \ln(9) = \ln(9)$	
$I_r - I_c.m(9) - \frac{1}{2\pi B_W}$	Equation 33

$$T_c = \frac{1}{2\pi . B_w} = \frac{T_r}{\ln(9)}$$
Equation 34

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Figure 117: PMT's equivalent circuit.

This time response must be faster than that decay lifetime of the phosphor for successful lifetime determination. Although the rise time for the 9942B PMT is specified at 2ns, with the PMT connected to a  $1M\Omega$ , 20pF oscilloscope, a time constant that calculates to  $30\mu s$  and  $t_r$  of  $65\mu s$ , is considered slow making it unsuitable for measuring decay lifetimes faster than this. Terminating the connection with large resistor values will yield higher gains since V=I.R, but will consequently produce a slower temporal response as  $\tau_c = RC$ , and vice-versa. These effects are shown in Figure 118.



Figure 118: (a) output pulse shapes with an anode load  $R = 50 \Omega$ , for a range of parallel capacitors. (b) pulse shapes for C = 10 pF and a range of resistor values. Taken from [182]

A solution avoiding such compromise is the use of a transimpedance amplifier, where a feedback resistor is connected across the output and the inverting summing input of an operational amplifier. This effectively holds the input to zero. With the capacitance and input voltage connected to the virtual earth, the resistor is isolated from the unwanted influence of capacitance  $C_0$ , and only experiences circuit board capacitance of 0.1pf [182], which greatly improves response times. According to Johnson[183], the temporal response in this configuration is considerably faster and can be determined using Equation 35. The gain bandwidth product (GBW) provides frequency and open loop gain limitations of the operational amp. Using values of 5.4Mhz for standard CA3410 amplifiers and 1M $\Omega$  for R<sub>F</sub>, a bandwidth of 0.29MHz is achieved,

yielding  $t_{rise}$  and  $T_c$  of 1.19µs and 0.54 µs respectively, instead of 65µs and 30µs for the original setup, providing a temporal advantage of approximately x55. In the past, commercial operational amplifiers were unsuitable for fast pulse work or low noise applications, but have greatly improved [182]. For example, Texas Instrument JPA657 JFET amplifiers have GBW of 1.6GHz and would therefore provide a theoretical advantage of approximately x951. However, fast transimpedance amplifiers can be problematic because they can produce oscillating and unstable signals. Stabilisation can be achieved by adding a small capacitance  $C_F$  in parallel with  $R_F$  to reduce its transimpedance at high frequencies.



Figure 119: Anode equivalent circuit with transimpedance amplifier

Another solution is the use of fast feedback amplifiers, consisting of a few fast transistors configured in the current feedback mode. These amplifier, typically contain two to three fast transistors configured in the current feedback mode, and can be connected directly to the PMT base. These typically have  $50\Omega$  impedance and provide voltage gains from 10-100, whilst achieving temporal response rise times of 1ns [182, 184]. An A2 Thorn EMI fast pulse amplifier that used a VV100B wideband pulse amplifier providing a non-inverting gain of 10 was used in this study. The circuit had low noise and drift characteristics with an input resistive load of  $50\Omega$  and temporal response rise times specified at 2.5ns, yielding a time constant of 1.15ns and a bandwidth of 140MHz. This amplifier was found to be adequately fast with gain that required no additional amplification.

**PMT maximum current considerations:** PMTs are limited to maximum light and signal levels. High mean anode currents can result in non-linear performance and deteriorate the PMT. During experimentation, it was ensured that the mean anode current, averaged over one second, was below 100uA as recommended by the manufacturer [176]. For pulsed applications, an expression was derived to evaluate

Equation 36

the mean current, illustrated in Figure 120 and Equation 36, because the mean signal also depends on:- the laser repetition rate ( $f_L$ ), decay lifetime ( $\tau$ ), phosphor's peak radiant flux (R), and blackbody radiation levels (B) at different temperatures. The blackbody radiation (B), peak radiant flux (R) and phosphor decay lifetime ( $\tau$ ) are all functions of temperature. Increasing the temperature will increase the blackbody radiation, but reduce the lifetime decay and peak radiant flux in most cases. Therefore at high temperatures, when higher gains may be required to enable detection of weaker and faster phosphor signals, it is the increasing blackbody radiation that will impose a limit on the maximum PMT gain that can be allowed to ensure linearity without causing permanent damage to the PMT.

$$I_{anode} = E_c P_s G_d (f_L R. \tau + B)$$



Figure 120: The effect of luminescent decay and blackbody radiation on the PMTs mean anode current

#### 6.1.5 Results, discussions and measurement uncertainty

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With calibration data and the understanding of the PMT and electronic circuit behaviour, calculations were made to determine radiant flux, phosphors intensity Equation 37, and intensity per mJ of incident laser energy values (Equation 38) from observed voltages from the oscilloscope. These are shown in Equation 37 - Equation 39. Expanding Equation 38 reveals all its raw terms and is shown in Equation 39.

$$I = \frac{R}{\Omega} = \frac{V_{out}}{F_T \cdot F_\lambda \cdot E_c \cdot P_s \cdot G_d \cdot G_a \cdot R_a \cdot \Omega \cdot \cos \alpha}$$
Equation 37  
$$\frac{I}{E_L} = \frac{V_{out}}{F_T \cdot F_\lambda \cdot E_c \cdot P_s \cdot G_d \cdot G_a \cdot R_a \cdot \Omega \cdot E_L \cdot \cos \alpha}$$
Equation 38

$$\frac{I}{E_{L}} = \frac{(2.42)V_{out}x^{2}.f_{L}}{F_{T}.F_{\lambda}.E_{c}.\eta.\lambda.G_{a}.R_{a}.\pi.d^{2}.P_{L}.V_{dynode}}$$
Equation 39

The effect of the laser energy was investigated at different temperatures. Four different laser energy settings (approximately 1.7mJ, 3.2mJ, 5.5mJ and 8.5mJ) were used in the investigation. Results shown in Figure 121 indicate the phosphor's

emission intensity rises proportionally with increasing laser energy levels across the entire temperature range. Figure 122 indicates the effect of emission intensity at various laser energies for various phosphors at room temperature. For YAG:Tb phosphors, the emission response is almost linear. However, for YAG:Tm, there are signs of diminishing returns, which suggests maximum emission levels, where adding more laser energy yields no benefit in emission intensity. Further research at higher laser energies is required to establish these saturation points. Figure 123 indicates the phosphor emission intensities normalised to the input laser energy, and Figure 124 shows the emission intensity relatively to the intensity taken at 20°C at various temperatures, using different laser energy levels. Although the emission intensity varied with increasing temperature, for YAG:Tm and YAG:Tb the sensitivity to laser energy levels was relatively constant throughout the temperature range. However, the sensitivity for  $Y_2O_3$ : Eu varied with temperature. At low temperatures below 600°C, the phosphor produced proportionally higher emission intensities/mJ with increasing laser energy. However, when the temperature was increased, the relative gain became smaller and eventually reversed, where increasing the laser energy did not proportionally increase the emission intensity.



Figure 121: Intensity (mW/sr) with increasing temperature at various laser energies for a number of phosphors.



Figure 122: Effect of increasing laser energy at room temperature



Figure 123: Intensity per mJ (mW/sr.mJ) with increasing temperature at various laser energies for a number of phosphors.



Figure 124: Emission intensity relatively to the intensity taken at 20°C at various temperatures using different laser energy levels



Figure 125: Variation of intensity per mJ of laser energy with increasing temperature



Figure 126: Variation of intensity per mJ of laser energy (semi-logscale) with increasing temperature

The results shown in Figure 125 and Figure 126 clearly indicate a variation in phosphor intensity with temperature, which is considerably different amongst different phosphors, under different excitation wavelengths.  $Y_2O_3$ :Eu using 266nm excitation exhibited the highest intensity per mJ of laser excitation from all the phosphors investigated at room temperature. The intensity from the same phosphor under 355nm illumination was considerably lower, by a factor of approximately x100. However, the intensity from the 266nm decreased at a faster rate with increasing temperature. Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn showed an increase in emission intensity with increasing temperature for both excitation wavelengths. It is interesting to observe the efficiency of the phosphor was approximately the same for both excitation wavelengths. For YAG: Tb two emissions wavelengths were investigated (490nm and 544nm). The ratio between 544nm and 490nm remained the same throughout the temperature range, at approximately 2.3 for both excitation wavelengths. The intensity produced by the 266nm excitation produced a higher intensity by a factor of approximately x6.5 at room temperature. Both YAG:Tb and Y<sub>2</sub>O<sub>3</sub>:Eu experienced higher intensity emissions under 266nm illumination at 20°C. However, YAG:Tm produced a higher intensity under 355nm than 266nm illumination by a factor of approximately X3. It was shown that the 0.5% activator concentration produced almost double the intensity produced from 0.1% concentration throughout its entire temperature range. The intensity of YAG:Tm under 266nm illumination reduced with increasing temperature, whilst remaining relatively constant under 355nm illumination. From all the phosphors investigated, YAG:Tm experienced the highest intensity at temperatures over 900 °C. With a peak emission wavelength of 458nm, the phosphor also experiences the lowest amount of blackbody radiation; therefore its advantage at high temperatures is further amplified. At lower temperatures with relatively longer luminescence decays, some phosphors experienced observable rise-times, and it was the peak intensity that was used in the analysis, however, as the temperature increased, the rise times became faster eventually becoming undetectable. Using the optical setup, it became difficult to monitor intensity values over 900°C for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn phosphors, 1100 °C for Y2O3:Eu , 1150°C for YAG:Tb and, and 1400°C for YAG:Tm. The reason for this is the increasing blackbody radiation, faster decays and decreasing phosphor intensity signals levels.

There are many sources of uncertainty. A relative approach using the relationship described in the ISO GUM [185] together with guidelines from Bell[186] and Birch[187] were used for determining the combined expression of uncertainty. It was assumed the terms are independent and uncorrelated. As the relation shown in Equation 38 can be expressed in terms shown in Equation 41, with the exponents  $p_i$  being either positive or negative, the relative combined expression of uncertainty can be determined using Equation 42. A summarised uncertainty budget highlighting the major uncertainty sources from instrumentation, calibration and evaluations from statistical analysis is show in Table 23. The combined relative standard uncertainty (u) was found to be approximately  $\pm$  10.7%. The expanded uncertainty (U=ku) based on the standard certainty (u) multiplied by the coverage of k=2 providing a 95% confidence level was found to be approximately  $\pm$  21.4%.

$\left[u_{c}(y)\right]^{2} = \sum_{i=1}^{N} \left(\frac{df}{dx_{i}}u(x_{i})\right)^{2}$	Equation 40
$Y = cX_1^{p_1}X_2^{p_2}X_3^{p_3}X_N^{p_N}$	Equation 41
$[u_{c}(y)/y]^{2} = \sum_{i=1}^{N} [p_{i}u(x_{i})/x_{i}]^{2}$	Equation 42

Uncertainty elements	Fractional Standard Uncertainty [pː.u(xː)/xː]
V <sub>out</sub> : Instrument uncertainty (±3% with 95% confidence, k=2, p=1)	0.015
Solid angle: Instrumentation + human uncertainties (micrometer, ruler) U(x) = 0.05 + 3mm (p=2, k=1.73), U(d) = 0.01 + 0.1mm (p=2, k=1.73)	0.01
Dynode Gain: Instrumentation uncertainty: $U(V_{dynode}) = 1.5V$ (k=1.73, p=11.75) Statistical variations from experimental data (0.035 – 0.07)	0.0621
P <sub>s</sub> .E <sub>c</sub> : Calibration uncertainty Instrumentation - Moletron power meter (±5% with 95% confidence, k =2, p=1) Statistical variations from experimental data - 0.039-0.071 (0.055)	0.0604
Amplifier: Load (0.01) Amplifier gain (0.01)	0.0082
Filter transmission (±5%, k=1.71, p=1)	0.016
Laser Energy: Instrumentation - Gentec power meter ( $\pm 5\%$ , 95% con, k=2, p=1) Laser repetition rate (0.001) statistical variations of laser pulses (sd = $\pm 5.6\%$ , k = $\sqrt{100}$ , p =1)	0.026
Phosphor Intensity Statistical variation - repeatability (k = $\sqrt{4}$ , p = 1) Variation caused by the linearity of the laser (0.05)	0.053
<b>Combined relative standard uncertainty (u)</b> Root-sum square: $\sqrt{\sum_{i=1}^{N} [p_i u(x_i)/x_i]^2}$	0.107
coverage factor k=1	

Table 23: Uncertainty budget for phosphor intensity

Although the response of the PMT and electronic circuitry indicated by the time constant was relatively fast, there were some issues related to measuring the peak intensity of fast decaying phosphor emissions. According to Sarner [188], dynode chain PMTs suffer from afterpulses that last approximately 0.25µs which are caused by the initial discharge that ionises the gas inside the tube [189, 190]. There was also some electrical interference, possibly caused by the lasers Q-switching electrical circuitry. It appeared that some of these effects are wavelength dependant. Due to these effects, which were difficult to discriminate against, the peak intensity measurements were taken after a timed delay following the laser pulse. For 458nm, this was after approximately  $0.4\mu s$ ,  $0.3\mu s$  for 490nm and 544nm, and  $0.25\mu s$  for 611nm, 630nm and 660nm. Due to this, the first proportion of the phosphor decay was lost; the peak intensity may have been higher if detection was possible, and only becomes significant when the phosphors lifetimes decays become relatively fast. The difference in values for slow and fast decays can be seen in Figure 127. By modelling the luminescence decay as a single exponential, shown in Equation 43, it is possible to provide correction and predict the intensity at time t=0 (I<sub>0</sub>), shown in Equation 44, provided the phosphors decay lifetime  $(\tau)$  and the time of measurements (t) are

known. Figure 128 and Figure 129 show the potential intensity values based on these assumptions if detection was possible. It is important to note that this does not take into account any luminescence intrinsic delays and rise-times, and ignores any multi-exponential decay effects. Therefore there may be an element of uncertainty that becomes increasing more significant at these low timescales when decays get faster especially at higher temperatures. This is however omitted from the uncertainty analysis, but the magnitude of the differences between measured and potential can be seen.



Figure 127: Variation of  $I_0$  for slow and fast decays



Figure 128: Variation of the measured and potential intensity per mJ of laser energy with increasing temperature



Figure 129: Variation of the measured and potential intensity per mJ of laser energy (log scale) with increasing temperature

### 6.1.6 Further work

The unit of measurement provided in this section is the phosphors intensity (W/sr) per mJ of incident laser energy, which is a power:energy ratio. To completely provide a dimensionless intensity measurement, the measurement can be either a power:power or an energy:energy ratio. For the first case, the laser energy can be replaced by the peak laser power, which can be easily obtained by scaling the energy measurement by the pulse duration (12ns). The other useful dimensionless measurement taking the ratio of input to output energies requires the conversion of phosphors peak intensities (W/sr) into total energies, and would indicate the quantum yield for that phosphor. Another useful analysis extending from this study is the phosphor's signal to blackbody radiation ratio. This can be achieved using the results presented and blackbody radiation estimations at the phosphor's peak wavelength at high temperatures that could be predicted using Planck's radiation law. These are discussed in the next section.

Due to afterpulse interference from the PMT, it would be beneficial to undertake this study using a MCP-PMT or other instruments which are less prone or immune to these effects. Other future work could include the investigation using different % activator concentrations or using other high temperature thermographic phosphors.

The work presented in this section only considers peak emission wavelength of phosphors under 266nm and 355nm illumination, and does not take in to account the

absorption spectra of the phosphor; future work could also investigate emission intensity under different excitation sources of various energies, linewidths, pulsewidths and wavelengths - It would be particularly interesting to study the peak intensity of peak emissions under peak absorption excitation wavelengths.

### 6.1.7 Conclusions

It was shown that the detected emissive power of phosphor emissions varied proportionally with the detectors collection area, and inversely proportional to the distance squared, as expected. By taking these effects into account using the solid angle, it was shown that the intensity (radiant flux per solid angle) remained constant; illustrating phosphor emissions created using a relatively small laser excitation area could be modelled as being point sources. It was shown that there is clear variation in the phosphors peak emission intensity with increasing temperature. This variation was different amongst phosphors and the excitation wavelength. It was shown that both Y<sub>2</sub>O<sub>3</sub>:Eu and YAG:Tb produced a higher emission intensity per mJ using 266nm excitation than 355nm at lower temperatures. The efficiency of Mq<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn was approximately the same for both excitation wavelengths, both showing an increase in emission intensity with increasing temperature. YAG:Tm produced a higher intensity under 355nm than 266nm illumination by a factor of approximately X3. The intensity of YAG:Tm under 266nm illumination reduced with increasing temperature, whilst remaining relatively constant under 355nm illumination. From all the phosphors investigated, YAG:Tm experienced the highest intensity at higher temperatures (900  $^{\circ}C$  +) With a peak emission wavelength of 458nm, the phosphor also experiences the least amount of blackbody radiation; therefore its advantage at high temperatures would be further amplified. Using the existing optical setup, it became difficult to monitor intensity values over 900°C for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn phosphors, 1100°C for Y<sub>2</sub>O<sub>3</sub>:Eu, 1150°C for YAG:Tb, and over 1400°C for YAG:Tm. This is due to increasing blackbody radiation, faster decays and decreasing phosphor intensity signals levels. The work presented in this section is extended with further analysis to reveal quantum efficiencies with increasing temperatures and provide signal-to-blackbody radiation evaluations at high temperatures in the next section.

### 6.2 Quantum efficiencies and signal-to-blackbody-ratios

#### 6.2.1 Introduction

Following on from the previous section, this section provides additional measurements including an evaluation of the phosphors quantum efficiency and signal-to-blackbody radiation ratio at various temperatures. These terms are beneficial in a number of ways and may provide physicists additional insight to the physical luminescence processes of phosphors at high temperatures, and may provide additional aid to application design engineers wanting use a phosphor thermometry solution at high temperatures. To the best of knowledge no such work exists.

### 6.2.2 Methodology

#### **Quantum Efficiency:**

The unit of measurement provided from the radiant flux measurements shown in Equation 38 is the phosphors intensity (W/sr) per mJ of incident laser energy, which is a power:energy ratio. To completely provide a dimensionless measurement, it can either be a power:power or an energy:energy ratio. For the first case, the laser energy can be replaced by the peak laser power. The peak laser power ( $P_{LP}$ ) is obtained by the relations shown in Equation 45. The power ratio can now be easily obtained by scaling the intensity measurements per mJ obtained in Equation 38 by the pulse duration (12ns), and is shown in Equation 46.

$$P_{LP} = \frac{E_L}{t_L}$$
$$\frac{I}{P_{LP}} = \frac{I}{(E_L/t_L)} = \left(\frac{I}{E_L}\right) t_L$$

Equation 45

Equation 46

The other useful dimensionless measurement taking the ratio of input to output energies requires the conversion of peak intensity (W/sr) into total energy and would indicate the quantum yield for that phosphor at that wavelength. The energy is an integration of the intensity decay curve, illustrated in Figure 130. This is mathematically expressed in the form shown in Equation 47, and simplifies to the absolute energy determinable by multiplying the radiant power ( $I_0$ ) with the phosphors lifetime ( $\tau$ ).



Figure 130: Area under phosphor decay curve = Energy

Phosphor energy = 
$$\int_0^\infty I_o e^{\frac{-t}{\tau}} dt = I_0 \left[ -\tau e^{\frac{-t}{\tau}} \right]_0^\infty = I_0 \tau$$
 Equation 47

The new terms would still be expressed as radiant power or radiant energy per steradian. There are  $4\pi$  steradians in a sphere, and by assuming the phosphor emits into half a hemisphere, the total energy or power can be obtained by scaling the measurements by a factor of  $2\pi$ ; thus the equations for power:power ratios and energy:energy ratios are expressed in Equation 48 and Equation 49.

$$\frac{R_f}{P_{LP}} = \frac{2\pi I_o}{E_L} t_L$$
Equation 48  
$$\frac{E_P}{E_L} = \frac{2\pi I_o \tau}{E_L}$$
Equation 49

#### Signal to Blackbody radiation:

One of the biggest engineering challenges facing high temperature phosphor thermometry that limits it use is increasing blackbody radiation. Although absolute intensities, highlighted in the previous sections, can estimate the resultant radiant power observable at the detector's active area which can be used to facilitate instrument selection and optimise system setup, it does not indicate the amount and proportion of blackbody radiation that would be present. The choice of phosphor in terms of emission wavelength and absolute intensity are important consideration factors in combating the effects of blackbody radiation.

The spectral emissive blackbody radiance (B) from a surface at various wavelengths and temperatures can be estimated using Planck's law of radiation, shown in Equation 50, and illustrated in Figure 131. From the figures, it can be seen that for the temperature and emission wavelength range of interest, the blackbody radiation is always greater at higher temperatures. Therefore, provided that all other factors remain constant, a phosphor with a lower emission wavelength will experience lower blackbody radiation, and would thus permit a higher temperature capability before blackbody radiation becomes problematic. However, this assumes the intensity from different phosphors are the same. Although lower emission wavelengths are more beneficial in terms of blackbody radiation, a low emission intensity may neutralise or even reverse this. The ratio of phosphor signal intensity to blackbody radiation is a useful evaluation because it combines the effects of blackbody radiation and phosphor emission intensities at various temperatures, providing a valuable quantitative assessment that can be used as a design aid for phosphor selection.

$$B(\lambda,T) = \varepsilon \frac{2hc}{\lambda^5 [\exp(hc/\lambda kT)] - 1}$$

Equation 50

Radiance ( $W \cdot m^{-2} \cdot sr^{-1} \cdot m^{-1}$ )

where:

B = Spectral Radiance  $(W \cdot m^{-2} \cdot sr^{-1} \cdot m^{-1})$ 

- $\lambda$  = wavelength (m)
- T = absolute temperature (K)
- h = Planck's constant = 6.626068e-34 (J.s)

c = velocity of light = 299792458 (m.s<sup>-1</sup>)

 $\varepsilon$  = emissivity, (assume a constant of 0.7)

 $k = Boltzmann's constant = 1.3806503e-23 (J.K^{-1})$


Figure 131: Blackbody Radiance with increasing temperature at various wavelengths

## 6.2.3 Results and Discussions

## **Quantum Efficiency:**



Figure 132: Lifetime values of various phosphors with increasing temperature.

Figure 133 illustrates peak power ratios between the phosphor's peak wavelength emissions and the laser pulse. The characteristic shape of the graph remained the same as Figure 129 only being scaled by a constant factor of  $2\pi T_L$ . Since  $T_L$  is so small (12ns) indicating high laser peak powers (83.3MW/pulse), the power ratios between phosphor emissions are therefore very small.



Figure 133: Peak phosphor power with peak laser power ratio

Figure 132 shows Lifetime values of various phosphors with increasing temperature. Figure 134 indicates the energy ratio between the phosphor energy at its peak wavelength and the total laser energy falling onto its surface. This is effectively the quantum efficiency and is expressed in a percentage form. It was shown that both Y<sub>2</sub>O<sub>3</sub>:Eu and YAG:Tb yield similar levels of efficiency at lower temperatures when excited with a 266nm source. At a temperature above 900  $^{\circ}$ C, the quantum efficiency of the YAG:Tm phosphor excited using 355nm exceeds that of all the other phosphors investigated. The values indicate an efficiency of less than 0.1%. The determined quantum efficiency is not the not the overall efficiency of the phosphor covering the entire wavelength spectrum; it is phosphor's efficiency only at the at peak phosphor wavelength (10nm FWHM). It is important to note that the analysis did not take into account any reflected or scattered laser radiation that was not directly absorbed by the phosphor. However, this is not necessarily negative as the investigation provides results for a realistic test case. The work presented only considers peak phosphor emissions under 266nm and 355nm illumination, and does not take in to account the absorption spectra; It would be particularly interesting to study peak emissions under peak absorption excitation wavelengths.



Figure 134: Quantum efficiencies of various phosphors with increasing temperature

### Signal to Blackbody radiation:

The absolute intensity levels from the phosphor and the estimated blackbody radiation values using Planck's radiation law were used to make this assessment. Equation 50 predicts the radiant flux emitting from a unit area per unit wavelength per unit solid angle, and was therefore scaled using the emissivity factor, correct source size and wavelength band size, shown in Equation 51, to make a suitable assessment. By assuming the source area  $(S_A)$  is the same as laser illumination (or phosphor luminescence) area, a fairer comparison between the two signals (blackbody radiation and phosphor luminescence) can be made. The wavelength band was assumed to be 10nm corresponding to a standard interference filter with FWHM = 10nm. Although the emissivity had wavelength and temperature dependencies, the analysis assumed a constant emissivity of 0.7 (typical for ceramic materials) throughout the entire temperature and wavelength range of interest. As both ratio terms are expressed in terms of unit solid angle, the terms would cancel making the ratio independent of solid angle, and hence also distance. The minimum level of blackbody radiation was assumed to be  $1 \times 10^{-9}$ W/sr (1nW/sr) representative of noise in the measurement system; replacing it with zero made the ratio reach infinity. Figure 135 shows the ratio using theoretical blackbody radiation radiance based on these assumptions, with a constant emissivity and no additional contributions from reflections from the furnace.

$$Band = S_A \cdot \varepsilon \cdot \frac{2hc}{\lambda^5 [\exp(hc/\lambda kT)] - 1} \cdot \Delta \lambda \quad (W \cdot sr^{-1})$$
 Equation 51



Figure 135: Phosphor emission to blackbody radiation ratio, using theoretical blackbody values where source size equal to luminescence, and no contributions from reflections from furnace walls and other sources.

A figure of merit is the temperature when the phosphor emission per mJ of excitation energy equals the blackbody radiation (ratio = 1); this is the cross over temperature where the blackbody radiation rapidly starts to overtake masking out the phosphors signal; a measurement limit is soon reached. By assuming this happens when the ratio is 0.1, extrapolating the data reveals this limit for various phosphors, and is highlighted in Table 24. At a ratio of 0.1, emissions from blackbody radiation are 10X higher than peak emissions from the phosphor; to determine the lifetime decay accurately, at least  $3\tau$  would need to be captured representing a decay to  $36.8^3 = 5\%$ of the initial peak intensity value, indicating blackbody radiation values 200X the signal at this point.

	Based or ideal	n theoretical/ conditions	Based on experimental conditions	
Phosphor	Ratio = 1	Ratio = 0.1 (limit)	Ratio = 1	Ratio = 0.1 (limit)
611nm Y <sub>2</sub> O <sub>3</sub> :Eu using 266nm	1110 °C	1180 °C	1000 °C	1100 °C
544nm YAG:Tb using 355/266nm	1230 °C	1310 °C	1100 °C	1220 °C
458nm YAG:Tm using 266nm	1360 °C	1450 °C	1250 °C	1350 °C
458nm YAG:Tm using 355nm	1475 °C	1550 °C	1375 °C	1450 °C

Table 24: Comparison of the estimated limit of measurement based on theoretical/ideal conditions and experimental conditions.

Experimental blackbody data from the investigation was used to compare with the theoretical/ideal blackbody radiation values. The blackbody radiation values from experimental data were slightly higher than that from theoretical values. This is due to a number of reasons including: a.) the phosphor's emissivity may have been different, and may also a function of temperature and wavelength; b.) the source area of the collected blackbody radiation was equal to the optical access of the furnace, which was larger (15mm) than the assumed 7mm diameter; c.) there was additional contributions from reflected radiation from furnace walls and the silicon carbide heating rods. Another reason could be attributed to out of band leakage through the interference filters, which could be significant if the broadband blackbody is sufficiently intense. By generating a model and providing an adjustment/correction for the blackbody radiation source area size, the additional radiation from reflected radiation and other reasons was estimated to be in the region of X2.5. The model matched experimental values of temperature where the blackbody radiation equalled absolute signal levels. Figure 136 illustrates the signal to blackbody radiation ratios based on the existing experimental setup and limitations. Table 24 compares limits based on both theoretical and experimental blackbody radiation data, and illustrates the difference between them is approximately 75-100 °C.



Figure 136: Phosphor Emission to blackbody radiation based on experimental conditions

The ratio is an indication of the signal to blackbody ratio per mJ of laser energy. By simulating the laser energy by a factor of x10 and x100, and assuming a linear intensity response, the following temperatures are yield when the signal to blackbody ratio = 1, illustrated in Table 25. It is estimated a x10 and x100 increase in laser energy could improve the signal to blackbody radiation ratio and enhances the temperature capability by approximately 100 °C and 175°C respectively. In terms of a percentage, a 1000% (x10) increase in laser energy would only yield an advantage of approximately 6% in absolute temperatures (K). The table is based on the experimental blackbody values; thus there will be an estimated improvement of approximately 75°C using theoretical blackbody radiation values and ideal experimental conditions. Also, utilising the potential phosphor intensity curves predicts a further improvement of 100°C. However, in reality the advantage would be much lower than this because of non-linear phosphor responses and laser energy saturation that would limit phosphor emission intensity. A measurement limit may also be reached due to other reasons such as the lifetime decay becomes impossible to detect due to the detectors temporal limitations or discrimination against the laser pulse's fall curve.

Phosphor	Laser Energy X 1	Laser Energy x 10	Laser Energy x 100
611nm Y <sub>2</sub> O <sub>3</sub> :Eu using 266nm	1000 °C	1100 °C	1175 °C
544nm YAG: Tb using 355/266nm	1100 °C	1200 °C	1300 °C
458nm YAG:Tm using 266nm	1250 °C	1350 °C	1420 °C
458nm YAG:Tm using 355nm	1375 °C	1450 °C	1540 °C

Table 25: Temperature when the signal to blackbody radiation = 1. Simulation using an input laser energy of x1, x10, x100 mJ/pulse.

### 6.2.4 Conclusions

It was shown that there is a variation in the phosphors emission intensity with increasing temperature. This variation is different dependant on the phosphor type and the excitation wavelength. It was shown that both  $Y_2O_3$ :Eu and YAG:Tb produced a higher emission intensity per mJ using 266nm excitation than 355nm at lower temperatures. YAG:Tm produced a higher intensity under 355nm than 266nm illumination by a factor of approximately X3. The intensity of YAG:Tm under 266nm illumination reduced with increasing temperature, whilst remaining relatively constant under 355nm illumination. The quantum efficiency of the phosphor was obtained by multiplying the lifetime decay with the peak radiant power. Although YAG:Tm only had an efficiency below 0.1%, it was the most efficient phosphor at higher temperatures (900 °C+). Using the optical setup, it became difficult to monitor intensity values over 1100°C for  $Y_2O_3$ :Eu, 1150°C for YAG:Tb and, and over 1400°C for

YAG:Tm. This was due to increasing blackbody radiation, faster decays and decreasing phosphor intensity signals levels.

For the wavelengths and temperatures of interest, the blackbody radiation is always lower at lower wavelengths. With a peak emission wavelength of 458nm, YAG:Tm experienced the lowest proportion of blackbody radiation, and because it also exhibited the highest intensity at higher temperatures from all the other phosphors investigated, its advantage was further amplified. The phosphor signal intensity to blackbody radiation ratio is useful because it combines the effects of blackbody radiation and phosphor emission intensities at various temperatures, providing a valuable quantitative evaluation that can be an important design aid for phosphor selection. A figure of merit is when the blackbody radiation equals the phosphor emission (ratio =1). The assumed limit of measurement is when the ratio = 0.1. It was found by simulation that increasing the laser energy by x10 and x100 could increase the temperature limit by approximately 100 and 175 °C, indicating that a 1000% increase in laser energy could produce an advantage of approximately 6% in absolute temperatures (K). Based on the signal to blackbody ratio measurement limit analysis, YAG:Tm using 355nm illumination was found to exhibit the greatest temperature measurement capability at higher temperatures, and was found to offer an advantage of approximately +350 °C and +250 °C increased upper temperature capability compared to Y<sub>2</sub>O<sub>3</sub>:Eu and YAG:Tb phosphors. However, the quenching temperature was found to be approximately 1100 °C, making the lower temperature sensing capability poorer than the other two phosphors.

# 7 Determination of Phosphor Lifetimes

## 7.1 Introduction

This chapter focuses on luminescence decay characteristics of a number of potential phosphors that would be suitable for thermal analysis in engine environments. Unlike radiometric intensity measurements that are sensitive to the operating conditions, optical setup (including angle and distance) and thermal exposure times, lifetime decay measurements are absolute temporal measurements generally insensitive to these effects, and have shown to exhibit high levels of repeatability with low levels of uncertainty in the measurement. This section describes the methodology of how measurements were made, regression analysis and estimation of the measurement uncertainty. The results from this section will be used to select phosphors with suitable temporal characteristics for use on specific engine components.

## 7.2 Methodology

The decay lifetime ( $\tau$ ) of different phosphors at various temperatures were obtained using the schematic shown in Figure 112. A 7mm, 12ns, 15Hz, third and fourth harmonic (355nm, 266nm) Q-switched Nd:YAG laser beam was used to excite the phosphor. The laser provided an adjustable energy up to 15mJ/pulse and 5mJ/pulse for 355nm and 266nm respectively. Both excitation wavelengths were investigated. The subsequent emissions were collected using an Electron Tubes 9954B PMT after passing through a narrowband interference filter (8-10nm FWHM, 45% transmission) that was centred at the phosphor's peak emission wavelength, previously determined in the spectral analysis (Section 5.2), to isolate signals from other unwanted emission wavelengths, reflected laser radiation and blackbody radiation. PMTs are extremely sensitive detectors and have been the most widely used instrument for phosphor thermometry. An A2 Thorn EMI amplifier was used to condition the signal from the PMT. A 200MHz USB Picotech 3206 oscilloscope, terminated with  $50\Omega$  resistor, digitised and recorded the measurement for post analysis. A 45° 355nm reflective beamsplitter enabled the laser beam and the detector to remain on the same optical axis. The laser energy was monitored using a laser power meter, and minor adjustments were required to ensure the energy/pulse remained constant. An average of 150 laser pulses was used to reduce random errors and pulse to pulse variation, and reduce the noise to less than 1%. With a repetition rate of 15Hz, the complete acquisition of 150 pulses took 10s.

A modified Vecstar high temperature furnace (max capability of 1600°C), with a 15mm hole through the back insulation wall for optical access, was used as to heat the phosphor. The temperature of the furnace was controlled by a type R thermocouple (platinum-rhodium alloy based) and a PID eurotherm controller. To ensure the sample was at the same temperature, a second and a third thermocouple (Super OmegaCLAD - type K and type N) were placed in close proximity to the phosphors, and was fed into calibrated Picotech TC-08 thermocouple data logger. Tests were completed with the temperature incremented in 100 °C intervals before the quenching temperature and in 25 °C intervals after this up to the maximum temperature where emissions were detectable. For each temperature, the furnace was allowed to stabilise for 20 minutes for each 100 °C increment and approximately 10 mins for 25 °C increments, before readings were made. This was important because the difference between 3 independent thermocouple readings were otherwise significantly different. As the furnace utilises fast heating silicon carbide elements, designed to reach high temperature very quickly, the variation in temperature between thermocouples values was high, especially at lower temperatures. The built-in furnace thermocouple has a slow response times, consequently resulting in significant overshoot in temperature measured by the other thermocouples, which required time to settle and reach a temperature where all three thermocouples agreed. Despite this, there was yet some variation and uncertainty based on a number of tests, illustrated in Table 26, and was relatively larger at lower temperatures. The furnace had difficulty in maintaining a constant temperature at temperatures below 400 °C, and a longer time was required for the furnace to stabilise.

Temperature (°C)	% variation of absolute temp
100	1.20 %
200	1.06 %
300	0.87 %
400	0.45 %
500	0.39 %
600	0.34 %
700	0.21 %
800	0.19 %
900	0.18 %
1000	0.25 %
1100	0.22 %
1200	0.20 %
1300	0.25 %
1400	0.35 %

Table 26: Uncertainty of furnace temperature

The data acquisition system had memory limit of one million samples. For a maximum bandwidth of 200MHz, the minimum sample period is 5ns. However, hardware/software settings could enable 1GHz sampling for repeat signals, enabling a sampling period of 1ns. Operating at 200MHz with memory constraints meant that the maximum acquisition time was limited to approximately 5ms, and approximately 33 microseconds for the storage of 150 different waveforms. Due to this, the sampling rate was appropriately chosen to collect at least 6 lifetimes of the luminescent decay. For each test, all relevant information such as the PMT detection area, distance to target, angle, PMT gain voltage, background, blackbody radiation and peak intensity were recorded for post processing and analysis.



Figure 137: Schematic for high temperature lifetime ( au ) evaluations

Powdered phosphor samples were used because they showed no irreversible intensity behaviour. A number of phosphors with a combination of excitation and emission wavelengths were investigated, highlighted in Table 27.

Phosphor	Excitation wavelengths investigated (nm)	Emission wavelengths investigated (nm)
YAG:Tm - 0.5%	266nm, 355nm	458nm
YAG:Tm 0.1%	266nm, 355nm	458nm
YAG:Tb	266nm, 355nm	490nm, 544nm
Y <sub>2</sub> 0 <sub>3</sub> :Eu	266nm, 355nm	612nm
YAG:Dy	355nm	458nm, 490nm, 500nm
Mg:Mn	266nm, 355nm	630nm, 660nm

Table 27: Excitation and emission wavelength investigations for different phosphors

## 7.3 Data Processing

 $I = I_0 \cdot e^{-t/\tau} + c$ 

The luminescence decay lifetime ( $\tau$ ) was modelled as a single exponential function. The lifetime ( $\tau$ ) is the time it takes for the intensity to decay to approximately 36.8% of its original value. Raw data from the oscilloscope (150 time waveforms) were fed into Matlab and an averaging algorithm was used to average the waveforms to minimise the random errors and noise in the measurement. Taking more samples reduces this by  $\sqrt{n}$ . The noise from a single shot was approximately ±3mV, and taking 150 pulses reduced this to 0.24 mV which is approximately  $\pm 0.12\%$  from a typical reading of 200mV, or  $\pm 0.6\%$  from a typical low intensity reading of 40mV. As the lifetime ( $\tau$ ) represents a decrease in intensity to 36.8% of its original value, a minimum of 3 decays represents a total decrease to  $(0.368)^3$  which approximately 5% of its original value. This equates to approximately  $\pm 2.4\%$  and  $\pm 12\%$  at  $3\tau$ . Matlab was used to curvefit the data in the form shown in Equation 43 using the non-least squares method. After experimentation and familiarisation with decay curve, it soon became evident that the determined lifetimes ( au ) were sensitive to the collection time in which the curve fitting routine is applied. For YAG:Tb phosphors, a double exponential decay was also used to determine fits when the single exponential fit was not adequate.

Where 'c' is the offset caused by the blackbody radiation, and dark current by the detector. Although the theoretical response of the PMT and electronic circuitry was relatively fast, there were some issues related to measuring intensities of fast decays. According to Sarner [188], PMTs suffer from afterpulses that last approximately  $0.25\,\mu$  s, and are caused by the initial discharge that ionises the gas inside the tube and [189, 190]. There was also be some electrical interference, possibly caused by the laser's Q-switching electrical circuitry. Due to these effects that were difficult to discriminate against, the peak intensity measurements were taken after a timed delay after the laser pulse. The phosphor's peak intensity may have been higher if detection was possible. The interference from the laser's Q-switching power supply was reduced by electronic shielding and by moving the data collection electronics as far away as possible. Although the data acquisition was triggered at the start of the laser pulse, there was some intrinsic delay in the region of few tens of nanoseconds that varied from phosphor to phosphor, before the steep rise in luminescence was observed. The behaviour varied between phosphor and could be wavelength dependant, as the initial interference caused for  $Y_2O_3$ :Eu (at 611nm) was different to that of YAG:Tm (at 458nm), which was approximately  $0.1\,\mu s$  and  $0.25\,\mu s$  respectively. This could be due

to different photocathode sensitivities of the PMT, or delayed luminescence behaviour (or phosphorescence/delayed fluorescence) exhibited in the phosphors response.

It was found that the phosphors response were not pure single exponential decays. This unique decaying signature of the phosphor suggests additional luminescence mechanics that have not been accounted for. Non-linearity of the detection system was ruled out because: a.) testing with neutral density filters yielded the same results, and b.) this behaviour has also been reported by other authors. This can lead to a significant source of uncertainty when determining lifetimes of decay curves, which is shown to be sensitive to the start and total acquisition time of the decay. In many cases, there is an element of the decay that is only observable in the first decay ( $1\tau$ ) and is generally faster than the rest of the decay. Such signals have also been reported by Tobin et al [191] and Feist et al. [8]. Therefore, the shortening of the steeper region of the decay curve. A pure single exponential will yield the same lifetime independent of the time period during which the data was obtained. Feist et al. [8] and Dowell and Gillies [192] states that optimum time required to get  $\tau$  that is within 90% is when the collection period is between 0-  $6\tau$ .

Although these authors investigated the effects of increasing the collection time by increasing the end total time from time t=0, they did not investigate the effects of introducing a delay at the start of the decay curve. In the investigation presented in this chapter, the effects of both the start and end time, and inherently the total observation time period was investigated. The start and end times were made dimensionless by making them functions of the lifetime ( $\tau$ ). All the combination of start and finish times from time t=0 up to time=6 $\tau$  in 0.1 $\tau$  intervals were investigated. The variation of determined  $\tau$  values was mapped. It was found that good repeatable results were obtained when the initial delay was equal to the lifetime ( $\tau$ ). However, this is not straight forward as  $\tau$  is dependant on the initial delay; therefore an iteration process was programmed in Matlab to feedback the determined lifetime values into initial delays. It required approximately 4-6 iterations to converge to a single lifetime value.

Another factor realised during the investigation was the importance of specifying the offset value. The offset is caused by blackbody radiation and dark current produced by the PMT and electronic circuitry. Although Matlab's curve-fitting algorithm/toolkit is capable of determining this automatically, it occasionally does not determine it correctly, consequently resulting in different lifetime ( $\tau$ ) values. This is especially true when the observation time is much less than the lifetime of the phosphor.

Without specifying the offset values, the length of acquisition time (in terms of  $\tau$ ) had to be significantly increased to obtain the correct lifetime values. By manually providing the offset value, it was found that the collection time could be significantly reduced with the determined lifetime values being within a few percent to values if a longer collection time was allowed. It was found that using a acquisition time of  $1-3\tau$  (which is a total length of  $2\tau$ ) produced the same results provided by a collection time of  $1-6\tau$  (approximately within 5%), with the additional advantage of the analysis becoming quicker, and requiring less collection time. Infact, the stability and repeatability of measurements from  $1-3\tau$  was better than from  $1-6\tau$  because as the phosphor's intensity levels become relatively small, the total proportion of noise in the collection increases; at  $3\tau$  the phosphor's intensity decays to  $0.368^3 = 5\%$  of its original values, whereas at  $6\tau$  it reaches  $0.368^5 = 0.25\%$  of its original value. If the SNR was 100:1, then at  $3\tau$  a SNR of 5:1 is yielded, whereas it is 0.25:1 (1:4) indicating the noise is actually 4x larger than the signal at  $6\tau$ .

Specifying offset values (or waveforms) would be particularly suitable in situations where the background value is subject to change, for example in rotating gas turbine environment, where the effects of reflected blackbody radiation, flame radiation and the probes efficiency at various rotational positions may differ throughout the collection period. In this case, an offsetting waveform would have to be subtracted, before the curve-fitting routine could be applied successfully.

There were other components of noise apart from the shot noise that resulted in fluctuations that were not removed by taking more readings. There was electrical interference with the flashlamp and Q-switch on, but without the laser liasing, concluding the noise is not optically generated. There were dominant oscillations, amongst the shot noise, that decayed with time. The first peak amplitude at time  $t_0$ was in the region of  $\pm 100$  mV, and decreased by approximately x1.8 between subsequent peaks, therefore after 8 oscillations, the effect of this interference was less than 1%. The period of the oscillations was approximately  $0.05\,\mu s$ , making the total time with interference greater than 1% approximately  $0.4\,\mu s$ . It is most likely caused by the lasers high voltage Q-switching mechanism, where a change in electrical energy transferred over short durations can result in significant electromagnetic disturbances in BNC cables and connections. This interference was significantly removed by placing an electric shield, made from aluminium foil, around the PMT connectors, amplifier and near the oscilloscope connectors. Using shorter cables from the PMT to the amplifier connector and moving the experimental setup away from the laser power source significantly reduced this interference. The total effect of these changes reduced the interference by x2.5. As the majority of this interference was not random, an isolated waveform could be stored and be used to digitally remove from the convoluted signal by waveform subtraction. This worked well and reduced this significantly; however, there were other elements of interference that were optically generated.

With the laser switched on, there was interference in the signal caused by the UV laser that had possibly entered the PMT because of reflections and backscatter from the optics and phosphor target. The UV may have leaked through the interference filter that are only effective with collimated light beams. The laser beam has a pulse width of 15ns with rise and fall times. If the luminescence decay from the phosphor is shorter than the laser's fall curve, then it may be difficult to discriminate against. The fall curve from the current Spectron Nd:YAG is in the region of 2ns, and is relatively fast. The relatively intense UV light source may cause saturation of the PMT and the effects of fast signals (both rise and fall times) may cause problems with the recovery of the electronics within the PMT itself. This behaviour has also been noted by Sarner [188] who notes the interference of PMTs lasting approximately  $0.25\,\mu$ s during phosphor lifetime measurements.

The limits of the data acquisition may be reached when decays become relatively fast. In the investigation presented the sampling resolution was 5ns. Assuming that 15 samples are required for successful lifetime determination, representing a capture of  $3\tau$  with 5 samples/ $\tau$ , then the minimum lifetime detectable is 0.075 µs.

Other forms of interference may be caused by luminescence from other surfaces. Observation of the response without the phosphor's presence may provide a test for this. If there is luminescence at the phosphor's peak wavelength, it may have to be digitally removed by subtraction. However, it may also be temperature dependant and simple subtraction of the waveform at room temperature will not be sufficient at different temperatures. If the substrate, where the phosphor is bonded to, is also exhibiting luminescence, provided there are no chemical or physical changes causing behavioural changes, calibration of the substrates luminescence intensity and temporal characteristics against temperature may provide a correction for this. However, the intensities may be different because of the phosphors presence reducing the amount of UV excitation falling onto the substrate, and further research into advanced mathematical algorithms and routines to separate the different signals would be required.



Figure 138: Calibration procedure for lifetime determination with temperature

## 7.4 Results, Discussions and Regression Analysis

### 7.4.1 YAG:Tm

The peak emission intensity from the spectral analysis of this phosphor was found to be 458nm, which corresponds to transitions from  ${}^{1}D_{2}$  to  ${}^{3}F_{4}$ , shown in Figure 139. The phosphor has also been reported to exhibit emissions in the infrared range at central wavelengths of 1460, 1900, and 2300 nm[193, 194]. This could represent the number of other possible emissions of lower energy (longer wavelength) that have also been known to exist as shown in Figure 139.



Figure 139: Simplified energy diagram for YAG:Tm from Cates et al. [195] (left). Additional emission lines (right) based on information taken from Zhang and Grattan [196]



Figure 140: YAG:Tm phosphor decays at various temperatures. Normalised intensity with time.



Figure 141: YAG:Tm - Lifetime decay with increasing temperature, under different conditions

Although, there was a decrease in lifetime with increasing temperature, shown in Figure 140, the quenching temperature of this phosphor was found to be approximately 1100°C, shown in Figure 141. A number of different test cases with different illumination energy, wavelength (266nm, 355nm) and phosphor concentration (0.1%, 0.5%) revealed that the temporal response of the phosphor remained to be the same, despite these factors significantly affecting the phosphors intensity response. At low temperatures, the effects of the start delay significantly affected lifetime values that were different by approximately 30%. However, as the temperature increased, the decays exhibited a closer resemblance to a single exponential, where the sensitivity of the start and ending collection times was reduced with predicted lifetimes being within 5% to each other, as shown in Figure 143 to Figure 144.



Figure 142 and Figure 143: Map of lifetime values indicating the effect of different start/end collection times (proportions of  $\tau$ ). Emission from YAG:Tm at 700 °C (left) and 1200 °C (right). The sensitivity of the effect of the start/end times is reduced at higher temperatures.



Figure 144: Percentage variation in lifetime values at various start and end collection times for YAG:Tm at 1200  $^{\circ}\text{C}$ 



Figure 145: Example of YAG:Tm decay and curve fit at 1350 °C

#### **Regression and residual analysis:**

Regression analysis was performed in Matlab. The log of the lifetime values, shown in Figure 141 was curve-fitted after the quenching temperature using non least squares method, according to linear relationship shown in Equation 53.

 $\ln(\tau) = p_1 T + p_2$ Equation 53

where T is the temperature (°C), and  $p_1$  and  $p_2$  are constants, which were determined to be -0.01491 and 18.81 respectively. A plot of the regression is shown in Figure 146. The r<sup>2</sup> values of the curve fit was approximately 0.997. Residual analysis from all the tests taken, using different conditions revealed a variation in ln(t) of 0.3; in terms of temperature, which is determinable by multiplying the residual with  $p_1$ , this related to a variation of approximately ±10 °C, which is approximately ±1% in terms of absolute temperature (K) (Figure 147).



Figure 146: YAG:Tm – Regression and residual analysis after the quenching temperature.



Figure 147: YAG:Tm -Residual error analysis in terms of temperature.



Figure 148: YAG:Tm - Regression and residual error analysis

It was found that the residual error could be significantly improved if the curve fitting procedure, including start and end time positions, is predetermined and remains the same, for example calibration of the data from  $1\tau$ - $3\tau$ . It was found that the residual errors were significantly reduced from  $\pm$  1% to approximately  $\pm$  0.4% (6 °C) by adopting this approach, Figure 148. It was envisaged that at temperatures above 1350 °C, when the initial delay becomes greater than the lifetime, the analysis would result in greater errors. However, this was not shown to be true. At 1400  $^{\circ}$ C, a fixed delay of  $0.2\,\mu s$  represented a delay of almost  $2\,\tau$ . Although lifetime values may have been significantly different if the delay was lower (e.g. at t=0 or  $1\tau$ ), analysis showed that calibration using this method did not necessarily result in greater errors, presenting itself as a valuable option for lifetime determination with the additional advantage of avoiding the initial proportion of the decay, where de-convolution of the interference may result in greater errors. It was found that the collection period from  $2\tau$  -  $4\tau$  yielded similar results as the collection period of  $1\tau$  -  $3\tau$  (within 10%); this represented a temperature variation of 6 °C at 1370 °C, which is approximately a variation of 0.36% in terms of absolute temperature (K).

### 7.4.2 Magnesium Manganese phosphors

A number of Mg:Mn phosphors have been previously cited in the literature, and have been reported to exist in a number of various chemical compositions, shown in Table 28. A number of these were not easily available/manufacturable because of their charge imbalance, as shown in Table 28. The Mg:Mn phosphor used in this thesis was obtained from Phosphor Technology, UK; it has a similar chemical composition to the Mg<sub>3</sub>GeFO<sub>4</sub>:Mn phosphor, shown in reference [81], with an additional Fluorine (F) to balance the charge of the molecule.

 $Mg_3F_2GeO_4:Mn: 3Mg^{2+}$  and  $Ge^{4+}$  equals <u>10+</u>, 40<sup>2-</sup> and 2F<sup>-3</sup> equals <u>10-</u>

Phosphor Chemical composition	Charge Imbalance (I), Balance (B)		Methodology used, excitation and emission wavelengths	Ref	
Mg₄GeO <sub>6</sub> F∶Mn	$4Mg^{2+}$ and $Ge^{4+}$ equals <u>12+</u> ,	$60^{2-}$ and F <sup>-1</sup> equals <u>13-</u>	I	266nm excitation 631nm, 657nm emissions intensity ratio and lifetime method	[55] [79] [54] [77]
Mg₄GeO₄ :Mn	$4Mg^{2+}$ and $Ge^{4+}$ equals <u>12+</u> ,	40 <sup>2-</sup> equals 8-	Ι	Intensity	[80]
Mg₃Ge O₄ F:Mn	$3Mg^{2+}$ and $Ge^{4+}$ equals $10+$ ,	4O <sup>2-</sup> and F <sup>-1</sup> equals <u>9-</u>	Ι	266nm excitation 633nm and 659nm intensity ratio	[81]
Mg₄GeO <sub>5.5</sub> F:Mn	$4Mg^{2+}$ and $Ge^{4+}$ equals <u>12+</u> ,	5.5 O <sup>2-</sup> and F <sup>-1</sup> equals <u>12-</u>	В		[76]

Table 28: Charge balance of Mg:Mn phosphors cited in the literature.

From the spectral analysis, it was found that the emission wavelengths of interest were 630nm and 660nm. The phosphor was found to exhibit a decline in lifetime ( $\mathcal{T}$ ) with increasing temperature from room temperature, shown in Figure 149. However, the sensitivity was relatively small until after 500 °C (quenching temperature), shown in Figure 150. The results using various excitation (266nm and 355nm) and emissions wavelengths(630nm and 660nm), shown in Figure 150, indicates the lifetimes and temporal characteristics for both emission lines remained the same under both excitation wavelengths, suggesting the luminescence mechanics for both these emissions under both excitation wavelengths are occurring at the same rate. The difference between them was found to be less than  $\pm 2\%$ .



Figure 149: Mg<sub>3</sub>GeFO<sub>4</sub>:Mn phosphor decays at various temperatures using 266nm illumination and 630nm emission. Normalised intensity with time. Temperature range: 20-500 °C (left) and 600-900 °C (right)



Figure 150:  $Mg_3GeFO_4:Mn$  - Lifetime decay with increasing temperature, under different conditions

Figure 150 represents determined lifetime values when the curve was fit from time  $1 \tau - 4 \tau$ . The determined lifetime value was sensitive to the collection period, mainly the initial delay and the total acquisition length. Like the YAG:Tm phosphor, the first proportion of the decay was relatively faster than the rest of the decay, indicating that the phosphor's decaying signature is not purely exponential. By ignoring this first proportion, lifetime values based on a single exponential relationship, shown in Equation 43, proved to provide a good match to the data. Example results showing the average data from 150 pulses and curvefit is shown in Figure 151. It can be seen that with the curve fit starting at  $\tau$ , the first proportion of the curve is under predicted. Figure 152 maps the results of lifetimes values at various start and end time, as functions of  $\tau$ . The results indicate that the lifetimes were under-predicted, indicating faster decays, by a factor of approximately 10%-20% when the decay routine was stared before a time of  $1\tau$ .



Figure 151: Decay and curve-fit for  $Mg_3GeFO_4$ :Mn phosphor at 20 °C(left) 600 °C (right), emission line - 630nm, excitation wavelength 266nm



Figure 152: Map of lifetime values indicating the effect of different start/end collection times (proportions of lifetime). Result from Mg<sub>3</sub>GeFO<sub>4</sub>:Mn phosphor, 266nm excitation, 630nm emission at: a.) 20 °C, at b.) 500 °C

#### **Regression/residual analysis**

The same method for regression/residual analysis presented for YAG:Tm was performed for data from the magnesium manganese phosphors. A polyfit after the quenching temperature (500  $^{\circ}$ C) was performed in Matlab, according to Equation 53.

$$\ln(\tau) = p_1 T + p_2$$
Equation 53

where T is the temperature (°C), and  $p_1 = -0.02163$  and  $p_2 = 15.82$ . The fit is shown in Figure 153, with an  $r^2$  value of 0.98. Residual analysis, based on this linear relationship revealed a variation of approximately 25°C, which is approximately 2.5% error from of the absolute temperature (K). This was relatively high and therefore a second order polynomial, based on Equation 54, was used in the regression analysis where  $p_1$ ,  $p_2$  and  $p_3$  were determined to be 2.075 x 10<sup>-5</sup>, -0.05028 and 25.4 respectively. The overall fit was better than the original linear relationship, and had a  $r^2$  value of 99.89%, Figure 154. The residual in terms of temperature was determined by differentiating and solving Equation 54 with the solution shown in Equation 55. With this fit, the residual error in terms of temperature was approximately  $\pm$  2.5 °C, which is approximately  $\pm$  0.2 % at the temperatures concerned, Figure 155.

$$\ln(\tau) = p_1 T^2 + p_2 T + p_3$$
Equation 54
$$dT = \frac{d(\ln(\tau))}{p_1 T + p_2} = \frac{R}{p_1 T + p_2}$$
Equation 55



Figure 153: Magnesium Manganese - Regression and residual analysis after the quenching temperature. (Linear polyfit).



Figure 154: Magnesium Manganese - Regression and residual analysis after the quenching temperature. (2<sup>nd</sup> order polyfit)



Figure 155: Magnesium Manganese - residual analysis in terms of temperature after the quenching temperature. (2<sup>nd</sup> order polyfit)

## 7.4.3 YAG:Tb

From the spectral analysis performed on this phosphor, there were two emission peaks centred at approximately 544nm and 490nm. They represent the transition from  ${}^{5}D_{4} - {}^{7}F_{5}$  and  ${}^{5}D_{4} - {}^{7}F_{6}$  respectively. [197] Both these emission wavelengths were tested under 266nm and 355nm excitation wavelengths. The 490nm emission peak has not yet been previously investigated in the literature for phosphor thermometry. The quenching temperature of this phosphor was found to be in the region of 700 °C for both wavelengths. Figure 156 shows the lifetime values for the different emission peaks under 355nm and 266nm illumination. It was found that the temporal decay of the phosphor under all these conditions were found to be approximately the same.



Figure 156: YAG:Tb phosphor - Lifetime values with increasing temperature: Under 266nm excitation (left) and 355nm excitation (355nm)

It was found that the phosphor exhibited very good single exponential decay behaviour below 650 °C, where the curve fitting routine and equation modelled reality well ( $r^2 = 0.999$ ). Example plots and single exponential curve fits are shown in Figure 157. Above 650 °C, which happened to be around the quenching temperature of the phosphor, the decay relationship transitioned into a multi-exponential decay where curve fitting from a single exponential relation were not as good. Example of such fits with various delay intervals, shown in Figure 158, indicate the model used for lifetime determination may be subject to large errors. In the example given at 800°C, the variations between determined values were different by a factor of x2. At higher temperatures, this effect reduced and the variation between the two results was

approximately 15%, Figure 159. Figure 160 shows map of lifetime values indicating the effect of different start/end collection times for 544nm emission at 20  $^{\circ}$ C under 355nm illumination.



Figure 157: Decay and single exponential decay curve-fit for YAG:Tb at 400  $^{\circ}$ C, 490nm emission line, using 266nm excitation wavelength. R<sup>2</sup> = 0.999



Figure 158: Decay and curve fit for YAG:Tb - 490nm using 266nm excitation, using single exponential decay relationship at 800 °C. When start decay values are t = 0 (left) and t =  $\tau$  (right)



700 °C using 266nm double exponential fit.



Figure 159: Decay and curvefit for YAG:Tb - 490nm using 266nm(top), 355nm (bottom) excitation, using single exponential decay relationship at 1,000 °C. When start decay values are t = 0 (left) and t =  $\tau$  (right)

There were rise time associated with YAG:Tb phosphors, and have been previously noted in Ranson[64]. These risetimes could also be used for temperature determination. However, due to the risetimes being close to the interference times of the detection system, they were not investigated further. Detailed mechanics can be found in Ranson [64]. The risetime reduced the potential intensity of the phosphor and consequently the decay at the first proportion of the decay was slower than the rest of the decay. Examples of decay curves and fits are shown in Figure 162. This behaviour was only experienced at temperatures below 400 °C; at higher temperatures rise times became faster and less influential. This behaviour was also noted with  $Y_2O_3$ :Eu phosphor, and is opposite of what is experienced by YAG:Tm and Magnesium Manganese phosphors where the decay at the first proportion of the decay was found to be faster than the rest of the decay. However, above approximately 400 °C the behaviour for YAG:Tb was reversed and the first proportion of the decay became faster than the rest of the decay.



Figure 160: Map of lifetime values indicating the effect of different start/end collection times (proportions of lifetime). Result from YAG:Tb phosphor at 544nm emission using 355nm excitation at 20 °C.

Single and double exponential curve fits were applied at temperatures above 600 °C. The double exponential relation (Equation 56), fit the data extremely well, with  $r^2$  values in region of 0.99. With the double exponential plotted against temperature, the luminescence was modelled to have a faster and slower decaying exponential component. This could also explain why the decay is faster in the first proportion of the decay. The results for both lifetimes are shown in Figure 161. The difference between them was in the region of x5-10. The result from the single exponential relationship were also compared, and is shown in the same figure. Although the double exponential relationship fit the data well, there were issues with the method

and initial conditions for the curve fitting routine. There are many solutions that fit the decay curve, and extra conditions were forced onto the routine to ensure the results fit the chosen model, shown in Equation 56, where the decay is comprised of two separate decays. The conditions applied were 'a', 'b', ' $\tau_1$ ' and ' $\tau_2$ ' are positive and  $\tau_1 > \tau_2$ . The offset values caused by blackbody radiation and dark current were subtracted before the curve-fitting routine was applied. The double exponential may make it more accurate than the single exponential model but adds further complexity to the lifetime determination process. Residual analysis from the investigation from the double exponential relation revealed similar magnitude levels to the single exponential relationship, providing no real advantage in terms of improved uncertainty. However, further research is yet required to optimise the methodology and realise the full potential of using double exponentials to improve the uncertainty in measurement.

As the difference between the two decaying exponential is approximately x5, it is expected that by capturing the decay after a delay of  $1\tau$  using the single exponential approach would miss the effects of the faster decaying exponent, and would only curve fit most of the slower decay curve, providing a better single exponential curve fit, making the determination of lifetime more stable and less prone to errors. It was therefore expected for the single exponential curve to be closer to the slower decaying curve of a double exponential relationship. However, this does not take in to the consideration the amplitude (intensity) of the decays, and although it is expected that the faster decaying component would be completely missed out, significantly higher emission intensities from this exponent may influence the curve fit. It was found that the average of the two decay exponents was close to the value of the single exponential decay.

It was originally thought that the fast and slower exponents from the double exponential relation may be attributed to the different emission lines, (544nm and 490nm), represented by emissions from  ${}^{5}D_{4}-{}^{7}F_{5}$  and  ${}^{5}D_{4}-{}^{7}F_{6}$  respectively. However, testing at both these wavelengths proved this hypothesis to be false as the temporal analysis presented for both emission wavelengths revealed that both the events occur and decay at the same rate.

$$I = a.e^{-t/\tau_1} + b.e^{-t/\tau_2} + c$$

Equation 56



Figure 161: YAG:Tb phosphor - Lifetime values using single and double exponential relationship for 544nm emission under 266nm excitation.



Figure 162: Decay and single exponential decay curve-fit for YAG:Tb at 20 °C under various conditions

Regression and residual error analysis, taking data from different excitation and emission wavelengths revealed an error of approximately 3% using a linear polyfit regression relationship (Figure 164) and approximately 1.5% for a second order polyfit (Figure 165). Regression coefficient values were found to be -0.01607 and 18.87 (single order polynomial, shown in Equation 53), and 1.099e-5, -0.03786 and 29.53 (second order polynomial, shown in Equation 54) However, separating the data with a separate calibration for 355nm and 266nm revealed an residual error of approximately  $\pm 10$  °C (0.79% at 1000 °C).



Figure 163: Regression and single order polyfit, taken after 700 °C



Figure 164: YAG:Tb - Residual analysis in terms of temperature for a linear polyfit



Figure 165: YAG:Tb - Residual analysis in terms of temperature for a 2<sup>nd</sup> order polyfit

It is noted in Choe et al. [197] the spectral emission of YAG:Tb are separated in two distinct groups, as shown in Figure 166.

- a.) below 480nm (blue) representing transitions  ${}^{5}D_{3} {}^{7}F_{1}$  and
- b.) above 480nm (green) representing transitions  ${}^{5}D_{4} {}^{7}F_{3}$ , where J= 0-6

The intensity of blue emission lines is much smaller than that of green emission lines. It is noted that the spectral distribution of emissions is strongly dependant on the Tb concentrations [198-203] where it has been reported that green emissions increase with increasing terbium concentrations. The blue emission dominates at very low concentrations below 0.1%, and almost disappears above 2%. In Choe et al. [197], it was shown that the blue emission line of 421nm decreased by 82% when the Tb concentration was increased from 1% to 5%. It is of interest to investigate these blue emissions at 421nm, as this wavelength may provide greater advantages in terms of blackbody radiation that may prove useful for temperature determination at high temperatures. This is subject to further research and will require samples of YAG:Tb with Tb concentrations less than 0.1%.



Figure 166: Emission spectra of YAG:Tb. Taken from Choe et al. [197]

## 7.4.4 Y<sub>2</sub>O<sub>3</sub>:Eu

The 611nm emission line of the phosphor was investigated under 266nm and 355nm emissions. The simplified energy diagram of  $Y_2O_3$  phosphors, shown in Figure 167, shows that the peak emission, centred around 611nm, corresponding to transitions from  ${}^5D_0$  to  ${}^7F_2$ .



Figure 167: Simplified energy diagram for Y<sub>2</sub>O<sub>3</sub>:Eu

The quenching temperature of the phosphor was found to be in the region of 600 °C. It was found that the lifetime value under both excitation wavelengths was the same, indicating the same physical phenomena. This is shown in Figure 169. At low temperatures below 700 °C, the phosphor exhibited rise times that could also be used to determine temperature. The detailed analysis goes beyond the scope of this chapter and further details can be found detailed in Ranson [64]. It was found that a single exponential decay relationship taken with an initial delay of 1 $\tau$  proved to provide a good fit with repeatable result. Example of such fits at various temperatures is shown in Figure 170. Regression and residual analysis using a linear polyfit according to Equation 53, yielded p1 = -0.01667 and p<sub>2</sub> = 17.05 after the quenching temperature (Figure 171) indicating a variation of approximately ±8 °C, which is approximately ±0.63% at 1000 °C, shown in Figure 172. However, calibration using a single excitation wavelength of 355nm revealed a regression residual in the region of ±3 °C which is approximately ±0.32% at 1000 °C.







Figure 169: Y<sub>2</sub>O<sub>3</sub>:Eu – Lifetime values with increasing temperature


 $\label{eq:Figure 170: Y_2O_3:Eu - Example of curve fits. It shows that the single exponential relationship fit the data well.$ 



Figure 171: Y<sub>2</sub>O<sub>3</sub>:Eu - Regression analysis after the quenching temperature.



Figure 172: Y<sub>2</sub>O<sub>3</sub>:Eu - Regression and residual in terms of temperature

### 7.4.5 YAG:Dy

Spectral analysis revealed that there were a range of wavelengths that could have been used for decay lifetime analysis. The wavelengths that were investigated were 458nm, 490nm and 500nm. These represent emissions from  ${}^{4}F_{9/2}$  and  ${}^{4}I_{15/2}$  to  ${}^{6}H_{15/2}$ . Only 355nm phosphor was considered in this investigation because initial studies with 266nm yielded poor intensities at room temperature and was therefore neglected. The quenching temperature of this phosphor was found to be 1200 °C, and it was interesting to note that all three wavelengths exhibited the same lifetime decay, suggesting the luminescence mechanics are occurring at the same rate, despite them being from different emissions lines. As with YAG:Tb, YAG:Dy did not experience a pure exponential decay, making it difficult to curve fit accurately using a single exponential relationship, even when the initial delay was set to  $1\tau$ . According to Cates et al. [195], lower activator concentrations may result in more single

exponential behaviour because the activator ions would be more isolated in the crystal lattice of the matrix. However, low activator concentrations can result in reduced emission intensities, and therefore Cates et al. [195] suggests an optimum value of about 1.0%. In the investigation presented in this thesis, an activator concentration of 5% was used, and it would be of interest to test the phosphors using lower concentration values as it may yield better single exponential behaviour and improve uncertainty. Despite these issues, the regression analysis from a linear polyfit after 1200 °C showed that the residual errors in terms of temperature was in the region of  $\pm 10$  °C, which is approximately  $\pm 0.56\%$  at 1500 °C (1773K). The temporal characteristics of this phosphor fell outside the temporal requirement for the chosen engine applications, and was therefore not investigated further using temporal analysis.



Figure 173: YAG:Dy- Lifetime values with increasing temperature



Figure 174: YAG:Dy- Regression analysis after the quenching temperature



Figure 175: YAG:Dy- Residual analysis in terms of temperature

# 7.5 Summary and Conclusions

A plot of lifetimes for various phosphors with increasing temperatures is shown summarised in Figure 132 and Table 29 and Table 30. The quenching temperature for YAG:Tm, YAG:Tb,  $Y_2O_3$ :Eu, YAG:Dy and  $Mg_3F_2GeO_4$ :Mn was found to be in the region of 1100 °C, 650 °C, 600 °C, 1200 °C and 500 °C respectively. Using the existing optical setup, it became difficult to monitor emissions over 900 °C for  $Mg_3F_2GeO_4$ :Mn, 1100 °C for  $Y_2O_3$ :Eu, 1175 °C for YAG:Tb and, 1400 °C for YAG:Tm and 1500 °C for YAG:Dy. The reason for this is increasing blackbody radiation, and detector limitations for monitoring faster decays and decreasing phosphor intensities.



Figure 176: Lifetime values of various phosphors with increasing temperature.

It was found that the phosphor signals were not true exponentials, especially in the first proportion of the decay. At higher temperatures, all the phosphors investigated experienced a slightly faster decay than the rest of the decay. YAG:Tb and  $Y_2O_3$ :Eu at lower temperatures experienced a slightly slower decay values as they had relatively larger rise times. An acquisition time of  $1\tau$  -  $3\tau$  produced very stable and repeatable results for lifetime determination, and yielded similar/better results as 1 au -6 auprovided that the offset value (based on the dark current and the blackbody radiation) was predetermined. The results from acquisition times of  $2\tau$  -4 $\tau$  also yielded similar results, within 10% lifetime variation. Some phosphors for example YAG:Tm experiences truer exponential behaviour at temperatures above 1000 °C and could be calibrated using acquisition times lower than  $3\tau$ . At 1200 °C, YAG:Tm could be calibrated using  $2\tau$ , with results that were within 5-10% of lifetime values if higher acquisition time was used. Phosphor thermometry via lifetime analysis has been shown to determine temperatures with high accuracy with uncertainties typically around 0.5 %. The combined expression of uncertainty taking into consideration the errors from the regression analysis and other uncertainties sources such as the thermocouples, thermocouple module, and furnace temperature variation for each phosphor is summarised in Table 31.

	Quenching	Maximum	Standard
	temperature	temperature	Uncertainty %
		achieved using	
		existing setup	
YAG:Tm	1100 °C	1400 °C	0.39%
Y <sub>2</sub> O <sub>3</sub> :Eu	600 °C	1100 °C	0.38%
YAG:Tb	700 °C	1200 °C	0.51%
YAG:Dy	1200 °C	1500 °C	0.46%
$Mg_3F_2GeO_4:Mn$	500 °C	900 °C	0.31%

 Table 29: Summary of the quenching temperature, maximum temperature that was determinable and the combined standard uncertainty.

	20 °C	100 °C	200 °C	300 °C	400 °C	500 °C	000 °C	2° 007	800 °C	D° 000	1000 °C	1100 °C	1200 °C	1300 °C	1400 °C	1500 °C
YAG:Tm																
Y <sub>2</sub> O <sub>3</sub> :Eu																
YAG:Tb																
YAG:Dy																
$Mg_3F_2GeO_4:Mn$																

Table 30: Summary of the useful temperature range determinable using the lifetime<br/>analysis for various phosphors

	YAG:Tm	Y <sub>2</sub> O <sub>3</sub> :Eu	YAG:Tb	YAG:Dy	Mg <sub>:</sub> Mn
	at 1300 °C	at 1050 °C	at 1150 °C	at 1350 °C	at 750 °C
Uncertainty based on regression analysis (95% confidence levels)	0.4 %	0.32 %	0.79 %	0.56 %	0.2 %
Thermocouple	0.1 %	0.1 %	0.1 %	0.1 %	0.1 %
TC08 - module	0.2 %	0.2 %	0.2 %	0.2 %	0.2 %
Furnace Temp uncertainty (variation in thermocouple readings)	0.25 %	0.23 %	0.22 %	0.3 %	0.2 %
Combined standard uncertainty (%)	0.39%	0.38%	0.51%	0.46%	0.31%
Standard uncertainty in terms of temperature	6.1 °C	4.7 °C	6.9 °C	7.5 ℃	3.2 °C

Table 31: Summary of combined measurement uncertainties on stationary components

# 8 Design for rotating engine components

### 8.1 Introduction

This chapter analyses the temporal characteristics of phosphors taken from previous chapters and attempts to select the most suitable phosphors and details the design for a suitable fibre optic probe and optical setup for specific engine components, taking into consideration the components rotational speed and temperature range. The design and optical layout will be tested for verification, taking into consideration likely physical constraints, realistic probe to target distances and maximum probe diameter sizes that would be permissible in such environments. It discusses the design philosophies and where possible suggests ways in which the system could be further improved for future work.

## 8.2 Component rotational speeds and linear velocities

It is important to select phosphors that can be calibrated at the expected temperature range of the engine component under investigation. With temporal lifetime methods on stationary components, a lower temperature limit is based on the quenching temperature of the phosphor, while the upper limit is based on the phosphor's intensity at rising blackbody radiation levels and the systems ability to detect faster decaying signals. For rotating components it is also important to consider probe design and phosphors lifetime characteristics, at the temperature range of interest to allow the probe's field-of-view adequate time to capture the luminescent decay. A defined time and detection limit will be imposed based on the rotational linear velocity and the probes field-of-view. The probe field of view is the target size that would be imaged onto the fibre, and would ultimately define the total acquisition time from a rotating surface. The linear velocity (v) can be calculated for a range of rotational speeds for engine components using Equation 57, where  $E_{RPM}$  is the engine speed in RPM, and r is the radial position in meters. A summary of linear velocities for suitable components is shown in Table 32; the calculated velocities have been rescaled to  $mm/\mu s$ . The linear velocities at several engine rotation speeds for turbine blades are shown in Figure 1. From the previous chapter it was shown that the acquisition times of 3 au yielded same results as acquiring data to 6 au , provided the offset values are pre-determined. By assuming that this is the minimum time required for successful lifetime determination, a limit is reached based on the linear velocity of the component and the probe's field-of-view. Figure 178 illustrates maximum detection time and lifetimes that can be determined using probe designs with different fields-of-view, at various engine speeds. It illustrates that increasing rotation speed limits the maximum lifetime measurement that can be made, and since decays are slower (higher lifetime values) at lower temperatures, a lower temperature capability limit is set.

$$v = \frac{2\pi \cdot E_{RPM}}{60} r$$

Equation	57
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Component	Radial positions	Linear Velocity	Distance to target	Diameter of probe
High Pressure Turbine Blade	0.295 - 0.385	0.4 - 0.52 <i>mm / μs</i> (root-tip)	120mm	10mm
Intermediate Pressure turbine blade	0.36 - 0.475	0.5–065 <i>mm / μs</i> (root-tip)	Assume same as HPTB	Assume same as HPTB
Low pressure turbine blade	0.36 - 0.535	0.5 – 0.73 <i>mm / μs</i> (root-tip)	Assume same as HPTB	Assume same as HPTB
Drive cone	0.15 - 0.21	0.2 – 0.285 mm/ μs	40mm	5mm

Table 32: Summary of linear velocities, distance to target for various engine<br/>components at 13,000 RPM.



Figure 177: Figure to compare the different linear velocities (root to tip) of different turbine blades at various engine speeds



Maximum time available for collection (microseconds)



Figure 178: Effect of probe design and maximum detection time for lifetime determination at various engine speeds

# 8.3 Phosphor selection criteria

## 8.3.1 Single Phosphor Solution

The upper and lower detection limits, together with the temperature range will set a criterion for ideal phosphor characteristics that can be used for phosphor selection. These limits can be plotted on top of existing phosphor lifetime with temperature plots. For simplicity and illustration purposes, a selection window is shown in Figure 179 which permits the comparison and suitability of different phosphors. For example, characteristics of phosphors B and D lie outside the desired temperature range. Although phosphor A would be most sensitive phosphor, the upper and lower limits of

measurement would prevent detection over the entire desired temperature range. In this instance phosphor C features the most suitable temporal phosphor characteristics. It is important to note that this temporal analysis should not be used alone, and effects of blackbody radiation, phosphor quantum efficiencies and the intensity of the phosphor to enable detection along with other issues must also be taken into consideration.



Figure 179: Illustration of temporal phosphor selection criterion

The field-of-view will depend on the distance to target and the probe design including lens size, focal length and position of the fibre. The probe should ideally incorporate the largest lens size and be placed as close as possible to the target to maximise signal collection. However, placing the lens too close to the gas stream would result in carbon build up that could interfere and obstruct the signal, therefore extra allowance is accommodated by purge air requirements. For HP turbine blade measurements, the distance to target from the turbine housing is 90mm, and therefore the total distance from the lens including purge air requirements is approximately 120mm. For HP turbine blade measurements, a maximum external hole diameter of 10mm yields a maximum allowable lens diameter of 8mm if an additional 1mm is used for housing and mounting purposes. For the drive cone application, a maximum lens diameter of 4mm is assumed with a target distance of approximately 50mm.

Using a fibre probe system with the fibre positioned at the focal point of the lens, and with the angle of collection less <NA of the fibre, the full FOV image can be assumed to be the same as useful diameter as the lens. This is 8mm for the HP turbine blade application and 4mm for the drive cone application. For the HP turbine blade application, an engine speed of 13,000 rpm and full FOV of 8mm results with the maximum acquisition time of  $17.4 \,\mu\text{s}$ , enabling the lifetime determination of approximately  $5.8 \,\mu\text{s}$ , assuming a collection of  $3 \,\tau$ . For IP-TB this is  $14 \,\mu\text{s}$  and  $4.6 \,\mu\text{s}$ ,

and  $13.3 \,\mu s$  and  $4.43 \,\mu s$  for LP turbines blades. The times for IP and LP blades are slightly lower than times for HP turbine blades, as expected, due to their increased radial positions within the engine. For the drive cone application, assuming an full FOV (field-of-view) of 4mm and a lower radial position, the maximum allowable time calculates to approximately  $16.32 \,\mu s$  and  $5.45 \,\mu s$  for successful lifetime determination. A summary of this is shown in Table 33. Coincidentally, the time window for the drive cone application is approximately the same as for the HP turbine blade despite them having different radial positions and FOV's. An upper temperature is reached due to rising blackbody radiation and the systems ability to detect lowering phosphor intensity signals and faster decays. From experimental results, this occurred when the lifetime of the phosphor was approximately  $0.08 \,\mu s$ . By placing these limits together with the expected temperature range requirements for different engine components, a phosphor selection criterion, as illustrated in Figure 179, can be applied on top of existing phosphor characteristic curves. This is shown in Figure 180 to Figure 182 for different engine components.

	Linear Velocities (mid point)	Full Field Of View	Max acquisition time	Maximum lifetime determinable (ratio 2.5)	Maximum lifetime determinable (ratio = 3)
HP Turbine Blade	0.4-0.52 mm / μs (root-tip)	8mm	17.4 μs	7 μs	5.8 μs
IP Turbine Blade	0.57 mm/μs	8mm	14 µs	5.6 μs	4.6 μs
LP Turbine Blade	0.60 mm/μs	8mm	13.3 μs	5.25 μs	4.43 μs
Drive Cone	0.245 mm/μs	4mm	16.32 μs	6.5 μs	5.45 μs

Table 33: Summary of the maximum acquisition time available for measurement



Figure 180: Phosphor Selection for High Pressure Turbine Blade application



Figure 181: Phosphor Selection for Intermediate and Low Pressure turbine blade application



Figure 182: Phosphor Selection for drive cone application

From Figure 182, it can be seen that the temporal characteristics of magnesium manganese phosphors would almost fit the requirements the high pressure compressor drive cone application in terms of temperature range (600-850 °C). However, from Figure 180, it can be seen that from all the phosphor tested, no single phosphor would cover the full temperature range requirements for the HP turbine blade (850-1400 °C). Rolls Royce have previously conducted tests using  $Y_2O_3$ :Eu and YAG:Tb at temperatures below 1100 °C. YAG:Tm would cover temperatures from about 1150 °C to 1400 °C, and would therefore be an invaluable design and verification tool for surface temperatures for future turbine blades. Consultation with Rolls Royce engineers has revealed that this would be the most critical temperature range of interest for turbine blade testing. The same probe could also be used for stationary NGVs as they share the same temperature limit.

#### 8.3.2 Sample per revolution

Another variation to the technique would be the possibility to sample the phosphor's decay per engine revolution. At 13,000 RPM, the time for a single revolution is approximately  $4615_{\mu s}$ . Sampling at this rate requires phosphor lifetimes to be at least 23ms at the temperature range of interest, assuming 15 samples are required for

successful lifetime determination representing a capture of 3  $\tau$  with at least 5 samples per  $\tau$ . A solution to extend from this would be to have additional collection probes at every ½ revolution (total of 2 equally spaced collection probes), or every ¼ revolution (4 probes). This would reduce the minimum lifetime decay requirement by a factor of the number of probes; therefore a 4 probe solution would require a minimum phosphor lifetime of 23/4 = 5.75ms, which is still relatively large. All the phosphors tested in this thesis had lifetimes less than  $1000\,\mu s$  (1ms) at the temperature range of interest, and therefore this possibility was ruled out in this instance. Further research into phosphors with longer decay lifetimes would be required to explore this possibility further.

### 8.3.3 Multiple Phosphor Solution

A solution to improve the temperature range for HP turbine blade application would be to use a combination of phosphors, for example YAG:Tb or Y<sub>2</sub>O<sub>3</sub>:Eu mixed together with YAG:Tm. This would extend the lower temperature capability of the system from 900 °C to 1400 °C covering most of temperature requirements for that component. The method relies on the different emission wavelengths, making it possible to detect both lifetimes from different phosphor simultaneously using a beam splitter, as shown in Figure 183. The technique assumes the phosphors do not chemically or physically interfere to change their thermographic or luminescence characteristics, in which case another calibration may be necessary. As the phosphor's coverage is reduced by a half if they are mixed in a 1:1 ratio, it is expected that the intensity from different phosphors will also be reduced by a half. Initial investigation using a single PMT and changing the interference filter presents this solution to be feasible; however, problems were encountered when the intensity of one phosphor was much greater than the other, and when detector sensitivities at the peak emission wavelengths were significantly different (eg. for the current detector, the QE for the peak emission of YAG:Tm (458nm) is approximately x5 of that for  $Y_2O_3$ :Eu (611nm). When higher PMT gains were necessary to detect emissions from the less intense phosphor (and/or less sensitive wavelength), the PMT also detected emission from the other phosphor which may have leaked through the filter causing interference. Further research is required to prevent this from happening, and/or into methods or techniques to digitally separate out the phosphors signals, as the dominant emission would be known. Another advantage of mixing phosphors is that if the decay lifetime of one phosphor is significantly higher than the other, then the long decaying phosphor can be used to correct for changes in the collection efficiency  $(c_f)$  as the target sweeps through the acceptance cone of the fibre; more details on this is covered later in this chapter. Another feasible solution would be to use different phosphors coated on different blades. By controlling the repetition rate, the collected signals from each blade could

be digitally separated using software, extending the temperature detection range of that phosphor. The solution assumes the blades are identical and share the same design and are exposed to the same environmental conditions.



Figure 183: Optical collection system for a mixed phosphor solution

## 8.4 Probe System Design Discussion

#### 8.4.1 Operating laser wavelength

A schematic of the phosphor thermometry system used for fibre coupling and collection optics is shown in Figure 184. From the Nd:YAG laser, only UV 355nm laser was considered for use with the phosphors of interest for the following three reasons. Firstly, the energy per pulse was higher from 355nm because of higher efficiencies of harmonic tripling as opposed to quadrupling of Nd:YAG (266nm) frequencies. The energy difference between the two wavelengths using the same laser voltage was found to be approximately x2.9. Secondly, laser to fibre coupling efficiencies were found to be 1.4x greater and less prone to damage at 355nm. Thirdly, although the quantum efficiencies for some phosphor were significantly higher at room temperature using 266nm, the advantage was greatly reduced at higher temperatures, and for some cases (e.g.  $Y_2O_3$ :Eu) it even reversed. For YAG:Tm, the phosphor's quantum efficiency was always higher using 355nm lasers by at least a factor of x3. Therefore there were no other advantages using 266nm.

### 8.4.2 Fibre optic

The fibre optic used in the investigation was Fiberguide's 1.0mm core Superguide G. An attempt was made to remove the polyimide coating to enable the fibre to be used at higher temperatures. However, the fibre became extremely inflexible and brittle, requiring extremely large bending radius. However, for short relatively straight sections, approximately 10cm, this option was feasible with the fibre being exposable to high temperatures for short periods. As there are many fibre jackets coating options that are commercially available to extend the temperature range to 800 °C, and probe designs that incorporate nitrogen cooling that have been previously used by Rolls Royce plc and Alaruri, et al. [52] that could be used to extend the temperature range further, no further attempt was made to protect the fibre from thermal damage, and therefore only the optical performance for laser delivery and emission collection was considered in this study. Sapphire fibres were initially considered due to their superior performance at higher temperature. However, following investigations highlighted in chapter 4, they were discarded because the investigation revealed relatively poor performance in delivering UV light.



Figure 184: Schematic for fibre optic delivery and collection

### 8.4.3 Single fibre probes on stationary targets

The probe design in terms of the size and choice of focusing lens is dictated by the maximum allowable hole size in the engine, and the NA of the fibre. The position of the fibre from the lens is also important. For an 8mm lens and NA of 0.19 for quartz fibre, the required focal length for a lens calculates to 20mm. A fused silica lens was chosen in the investigation, opposed to sapphire, for its superior UV transmission

characteristics and lower refractive index that would cause fewer reflections and backscatter. Two probes were made for testing: One was suited for the HP drive cone application utilising  $Mg_3F_2GeO_4$ :Mn phosphor, and one for HP turbine blade application utilising YAG:Tm phosphor. Key geometric features for both probe designs are shown in Table 34.

	HP turbine application	HP Compressor Drive Cone application
Size of lens (mm)	10	5
Useful diameter (mm)	8	4
Area (mm2)	50.27	12.57
Distance to target (mm)	120	50
solid angle	0.003491	0.005028
NA (quartz)	0.2	0.2
focal length of the lens (mm)	20	10

Table	34:	Key	feature	of the	different	probe	designs

A single fibre probe was initially setup and tested on a stationary target. The main issues related to this configuration were found to be:

- During laser to fibre coupling, the optimum fibre position from previous experimentation was found when the beam was diverging. However, with this configuration the beam's focus was a few mm in front of the fibre, which resulted in the breakdown of air causing intense spikes that lasted a few microseconds that disrupted/interfered with emission detection when high pulse energies were concerned. It is envisaged that prolonged use may also damage the PMT. The chance of breakdown was higher when the fibre was closer to the focus point, therefore the distance between them was increased, which consequently reduced the laser coupling efficiency from approximately 0.65 to 0.53.
- As the fibre's coupling lens was not at the focal position, the collected emissions returning to the PMT were not perfectly collimated, and it was thought it may affect the performance of the filter. However, testing using a separate collection fibre at the focal point did not produce different results; therefore these effects were neglected.
- At the probe's target-side setup, the fibre was positioned at the focal point of the lens. With this configuration, the laser output to the target was expected to be collimated. In reality the beam was diverging slightly (1.1°); for the distances concerned (120mm), this was assumed to be negligible. The fibre positioned at this focal point may not be the optimum position for emission collection because this position assumes a pick up of emissions from a collimated light source, whereas in reality emissions from the phosphor were

diverging as expected from a point source; therefore the position of the real focus may be further away. However, as a general rule, if the target size is less than the X5 the distance, then the focused position can be assumed to be at the same position as from collimated sources. Because of these issues, the probe was modified to allow minor adjustments to be made to the fibre's position whilst the output is observed; this simultaneously takes into account laser output to the target and the collection of emissions from the target into the fibre and PMT. The optimum position was found when the observed emission intensity from the phosphor was the greatest.

• There was significant UV backscatter that was being collected by PMT causing interference. This includes scatter and reflections off optical surfaces, lenses, fibre optic surfaces, mounts and other surfaces that may have leaked through the filter causing interference that lasted over  $0.7 \mu s$ . It was thought that there may have been significant scatter from the target side of the probe; however, interference was still present when the target probe lens was removed with the laser diverging and not pointing at anything suggesting that most of the interference was caused by scatter from laser to fibre coupling, opposed to scatter from the target side. There was a possibility of internal fluorescence within the fibre itself. Placing an iris in front of the PMT and by sealing the PMT from the sides reduced the interference to  $0.3 \mu s$ , which was the same as when separate fibres for delivery and collection were used.

### 8.4.4 Dual fibre probes

A separate illumination and collection probe would eliminate many issues related to single fibres. Separate fibres could allow better coupling of the laser with minimal interference to emission collection. The returning collection fibre could also be closely connected to the light detector, lowering optical losses present in a single fibre setup. However, the system would require two holes in the engine for separate illumination and collection probes, which may not be permissible for structural/mechanical reasons. The optical alignment for both the illumination and collection areas to be the same can be challenging, especially if target distances are expected to change, losing the probes flexibility to map temperatures over a surface, especially if a scanning probe head is later to be developed. A twin fibre, single lens probe that utilises a single hole in the engine would be preferable, but will not eliminate the scatter from the target probe end, and again with this arrangement, the illumination and collection areas would still be different. A single fibre solution ensures that the illumination and emission collection are optically collinear. Although there will be increased backscatter with reduced efficiencies, it is more flexible and can reduce the complexity of the system, especially when obtaining results from different probe to target distances. However, it is important to note that only half the probe's FOV (half-FOV) can be utilised for emission collection because the laser beam will be delivered from the central position of the probe. This consequently reduces the collection time and ultimately the minimum temperature measurement capability of the system.

For the probe used for the drive cone application, it would be of interest to investigate tapered fibres for laser delivery and collection. This can eliminate the need for a lens, which can reduce complexity whilst increasing flexibility of the probe. However, it expected for the NA to be reduced for total internal reflection to be effective, as previously stated in section 4.6.2. This may be advantageous because it will not require collimating lens for laser delivery, and there will be a reduced chance for stray radiation to be collected from other sources. An all silica tapered fibre with a taper ratio up to 1:10 is available from a number of manufacturers, however, the maximum size usually limited to 5mm diameter, and therefore a requirement of 4mm input and 1mm pigtail section would be commercially available. With this configuration, the NA of the fibres expected to reduce to 0.2/4 = 0.05. This is relatively small, and there may issues if the target is rotating as the reduced NA will consequently reduce the collection efficiency and the effective time period to capture luminescence decays, significantly affecting the performance of the technique. For these reasons, this option was ruled out, and would be subject to further research.

### 8.4.5 Probe performance

Using the appropriate laser energy levels, distances, lens diameter and collection solid angles, the expected intensity based on the values predicted from experimental testing were determined. The intensity per mJ for YAG:Tm and Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn at room temperature was estimated to be approximately 1.5mW/Sr.mJ and 1mW/Sr.mJ respectively. For a 5mJ of laser source on the target, and using the appropriate solid angles, this calculates to approximately  $26_{\mu W}$  and  $25_{\mu W}$  of optical power respectively. However, there were additional losses with the inclusion of the fibre optic system that are not taken into account in this prediction. These include:

- Reflection losses from the collection lens and the fibres face itself. For quartz this is estimated to be 4%[150], which reduces the amount of energy by  $0.96^2 = 0.92$ .
- The attenuation of energy from fibre, which is estimated to be in the region of 0.95 per meter, which equates to  $0.95^5$  (0.77) for a 5m fibre cable.
- At the collection end of the fibre, additional reflection losses from the collimation lens, beam splitter and the filter, equates to approx  $0.96^3$ . = 0.88.
- The iris in front of the PMT was used to reduce the amount of reflection energy from the UV wavelengths reaching the PMT that can cause signal interference

effects. Reducing the iris size reduced these effects, but consequently reduced the total amount of emission reaching the PMT.

The diameter of the returned emission beam from the fibre with an NA = 0.2 should be less than the diameter of the lens, otherwise, emissions would overfill the lens and energy would be lost. At a distance of approximately 55mm from the lens, this was approximately 2x55tan0.2 = 22mm, which was less than 25mm, the diameter of the lens, and therefore no losses due to this was assumed. (Figure 185)

reflection losses from fibre	0.96
reflection losses from lens	0.96
transmission loses per m	$0.95^5 = 0.774$
collimating lens reflections	0.96
beam splitter reflection	0.96
reflection from filter	0.96
losses by the iris	0.95
	0.599

Table 35: Additional losses in the fibre optic detection system



Figure 185: Returned emissions from the fibre

With these losses taken into account, shown in Table 35, together with a filter transmission of 0.5, the expected optical signals at the detector was expected to be approximately  $7.5_{\mu W}$ . However, the observed detected signals were approximately  $3.5_{\mu W}$  for YAG:Tm. Other losses not taken in to account in the analysis were later identified to be caused by the following:

The focused emissions from the probe lens to the fibre using the geometries presented were greater than the fibre size. A slot was made in to the probe housing, in order to observe the focus in relation to the fibre size to optimise the fibres position to increase collection. Even at the fibres optimum position, the beam was overfilling the fibre core. A 1.0 mm fibre core and a minimum target waist diameter of 1.1mm represents an overfill of x1.21, and is therefore only expected to capture 0.83x of the signal. Using a sharper focus by changing the

lens or a using a fibre with a larger diameter will help to increase the collection of emissions.

- Losses due to the NA of the fibre The NA is the fibres ability to capture emissions from the maximum angle to enable successful propagation of light via total internal reflection. Fortunately, the focal length of the lens using the optical setup was chosen to match the fibres NA, and therefore there is no reason to assume additional losses from this.
- The intensity from the chemically bonded phosphor was reduced by approximately
   0.52X compared to that from powdered phosphor samples.

By taking these additional losses into account, experimentation values agreed well with estimation. The reduction in optical power being delivered to the detector caused no issues for YAG:Tm phosphors as the gain from the PMT could be increased without significantly increasing noise and causing non-linearity effects within the PMT. The gain of the PMT was set at 1100V, and a single shot noise of ±11mV was observed. Taking 150 samples reduced this to approximately  $\frac{11}{\sqrt{150}} = \pm 0.9$ mV which

is 0.45% at 200mV. At time  $3\tau$  of a luminescent decay curve, the estimated noise would be approximately  $(100x0.9/200x0.367^3) = 9\%$ . However, for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn, the lower optical power had more profound effects. The observed noise levels for a single shot was approximately  $\pm 20$ mV, which was reduced to approximately  $\pm 1.63$ mV for 150 samples, which is  $\pm 1.36\%$  at 120mV. However, at  $3\tau$ , this would be  $(100x1.63/120x0.367^3) = 27.5\%$ , Increasing the dynode gain from the PMT increased the noise levels significantly where measurement were not possible. Although the expected optical power levels was similar in magnitude to YAG:Tm, the detector's radiant sensitivity at 630nm and 660nm was extremely low for the E-tubes PMT light detector, by a factor of at least x20 compared to that for the peak wavelengths of YAG:Tm (458nm). Although measurements were possible, a quantum efficiency of 1% or less at 660nm at the expected optical levels using the existing PMT is not the optimum choice for measurements from this phosphor.

## 8.5 Single fibre probe on rotating surfaces

#### 8.5.1 Collection efficiency

Apart from a limited time window to make measurements, there was another issue with the detection of luminescence lifetimes from rotating surfaces. As the phosphor's signal from a rotating surface traverses through the acceptance cone of the fibre, the collection efficiency varied as a function of rotation angle, with the maximum efficiency occurring at the fibre axis and varying to zero at limits of the probes field-of-view.

Therefore, following pulsed laser excitation, the detected signal  $(S_1)$  was a product of the collection factor  $(C_f)$  and luminescent phosphor decay as illustrated in Figure 186, Equation 58 and Equation 59. Details on how the phosphor decay waveform  $(decay_1)$  was discriminated and used to determine temperature is discussed further.



Figure 186: Detected signal of a moving target is a product of phosphor decay and collection factor

$S_1(t) = C_f(t)$ . decay <sub>1</sub> (t)	Equation 58
--	-------------

Therefore,

 $decay_1(t) = S_1(t) / C_f(t)$ Equation 59

There are a number of ways to determine the collection factor  $C_{f}(t)$ .

With a constant intensity source that does not vary with time, the collection factor ( $C_f$ ) is simply the normalised detected signal ( $S_{2-normalised}$ ), as shown in Equation 60 and Equation 61, where the constant (c) is maximum intensity value, which occurs at the central position. By substituting  $C_f$  from Equation 61 into Equation 59, the real phosphor decay waveform can be obtained by taking ratios of the two detected signals. (Equation 62).

$S_{2}(t) = C_{f}(t). c$	Equation 60
therefore	
$C_{f}(t) = S_{2}(t)/c = S_{2-\text{ normalised}}(t)$	Equation 61
$decay_1(t) = S_1(t) / S_{2-normalised}(t) = c. S_1(t) / S_2(t)$	Equation 62

In the investigation the collection factor of the probe was found by using a LED, serving as a constant light, mounted on to the surface of the rotating disc with the variation of intensity collected by the probe at realistic target distances. A battery pack, mounted at the centre of rotation, was used to power the LED. This reduced complexity by avoiding the need for rotary pin-outs from the shaft. The fibre's output was coupled into the PMT, and the measured intensity was normalised by dividing through by the maximum intensity levels found at the central position. Since the PMT measurements were collected against time, the linear velocity of the rotating disc, using Equation 57, was used to determine the displacement in mm, shown in Equation 63. This could then be used to determine a time waveform that could be used to correct a rotating phosphor decay waveform for any given speed. A number of tests at different rotating speeds yielded the same result, providing validation and suggesting that the corrected waveform and method is very repeatable and could therefore be used as an accurate method of providing such corrections. An example of the collection factor taken at a rotating speed of 2700rpm is shown in Figure 187. It can be seen that the maximum time for collection (and hence distance) equals that of the lens diameter as predicted.



Figure 187: The collection efficiency factor of the fibre optic probe. Collected using an PMT and a LED source mounted onto a rotating disc.

Another technique that can be used to determine the  $C_f$  (t) is by using another phosphor, or another emission lines from the same phosphor, that has lifetimes large enough to assume constant intensity throughout the collection period. An illustration is provided in Figure 188. This can enable in-situ calibration for any rotating surface, which could be advantageous in situations when a probe is used over surfaces with varying distances and angles during the probes collection period; this can avoid the

need for pre-determination of the collection factor at various distances and angles. This would be of particular interest for scanning probes, where finding the collection factor at different position would be time consuming. A number of methods could be utilised, for example the mixing of phosphors together and using the apparatus shown in Figure 183 to enable the simultaneous discrimination against emissions from the different phosphors. Alternatively, different phosphors could be applied to different rotating components that are the identical in design and exposed to the same environmental conditions where software and appropriate triggerable hardware can enable the capture of signals from both phosphors using the same probe.



Figure 188: Phosphor decays with short and large lifetimes.

This collection factor ( $C_f$ ) depends on the optical setup and probe design including distance to targets, fibre's NA and focal length of the lens. The  $C_f$ , shown in Figure 182, was experimentally determined using the constant intensity LED approach. The collection factor ( $C_f$ ), as a function of distance, can be mathematically expressed as a Gaussian distribution function that takes the form shown in Equation 59, where x is the distance in mm, and a,b and c are constants. This obtained results were curve fit in Matlab; for the probe design and optical setup used in the investigation, the curve fit determined: a = 1, b = 0.09 and c = 3.24. The equation can be expressed in terms of time, and will also depend on the linear velocity of the rotating component, shown in Equation 65, where  $\omega$  is the angular velocity, and r is radial position of the target.

$$c_f(x) = ae^{-\left(\frac{x-b}{c}\right)^2}$$

$$c_f(t) = ae^{-\left(\frac{\omega r t - b}{c}\right)^2}$$

Equation 65

Equation 64

By combing Equation 65 and the phosphor's decaying signal from a stationary target given by Equation 66, an expression can be derived, that can predict the signal from a rotating phosphor target with a specified lifetime ( $\tau$ ), at any given engine speed and radial position, shown in Equation 67. With the phosphors decay signal from a stationary target and the collection factor being normalised ( $I_0'$  and a' = 1) further simplifies Equation 67 to Equation 69. This equation relates the angular velocity, radial position and lifetime decay (au) of the phosphor. However, further research would be required to determine the how the constants b and c relate to the properties of the optical setup and probe design. By replacing au with the polynomial relation found during calibration in the form shown in Equation 70 and Equation 71, Equation 69 can be expanded to relate the observed signal to the absolute temperature, shown in Equation 72. This equation relates the intensity waveform of the rotating phosphor signal to the temperature of different phosphors at any given rotation speed for any given probe configuration.

$I_{stationary}(t) = I_0 e^{-\left(\frac{t}{\tau}\right)}$	Equation 66
$I_{(rotating)}(t) = \left(ae^{-\left(\frac{\omega rt-b}{c}\right)^2}\right) \left(I_0 e^{-\left(\frac{t}{\tau}\right)}\right)$	Equation 67
$I_{(rotating)}(t) = \left(e^{-\left(\frac{\omega \tau - b}{c}\right)^2}\right) \left(e^{-\left(\frac{t}{\tau}\right)}\right)$	Equation 68
$I_{(rotating)}(t) = e^{-\left[\left(\frac{\omega rt-b}{c}\right)^2 + \left(\frac{t}{\tau}\right)\right]}$	Equation 69
$\ln \tau = p_1 T + p_2$	Equation 70
$\tau = e^{p_1 T + p_2} = e^{p_1 T} e^{p_2}$	Equation 71
$I_{(rotating)} = e^{-\left[\left(\frac{\omega t - b}{c}\right)^2 + \left(\frac{t}{e^{p_1 T} e^{p_2}}\right)\right]}$	Equation 72

In single fibre probe configurations, where the fibre delivers the laser beam and collects emission, the peak emission intensity and maximum transmission based on the collection factor, occurs immediately after excitation. This may not be the optimal choice because the detected signal becomes weaker as emissions decay and the target sweeps away from the fibre axis, which may impose further limits depending on the minimum amount of signal that can be detected. Also, as the laser beam will be delivered from the central position of the probe, only half of the probe's FOV can be used to collect emissions (Half FOV). This has significant implications on the maximum lifetime value that can be detected and hence the lower temperature capability of the system. A carefully designed dual fibre configuration would increase the amount of signal received, and may increase the performance of the system, shown in Figure 189. Following illumination, the excited phosphor target will move and enter the axis of the collection fibre where the collection efficiency is greatest. This will occur after a delay that depends on the probe design and engine speed, and would yield greater signals at time t; it will also allow a lengthier collection period, which may permit an increased temperature measurement range capability. This would however increase the complexity of the system, and the system may not be able to cover the entire temperature range, especially if the decays are extremely fast relative to the engine speed, where it will be sensible to have the collection fibre at the same axis as the laser delivery fibre.



Figure 189: Detected signal of a moving target using a twin fibre probe configuration.

Double fibre configurations were considered, where it may be possible to have the laser delivery and collection on the same point, by using a number of methodologies. Bird had suggests the use of a prism [204] to separate the UV and emission wavelengths. Another solution could use dichotic mirrors/beam splitters that could be

used before or after the lens, shown in Figure 190. If the beam splitter can be rotated, then the delivery laser could be offset to match the speed of rotation and maximise the collection time of decay.



Figure 190: Twin fibre probe configuration with the use of a dichroic mirror.

## 8.6 Experimental Testing on Rotating Components

### 8.6.1 Experimental procedure

A number of tests were taken using thermographic phosphor at different rotational speeds. A disc with a number of thermographic phosphors adhered to using HPC binder (volume ratio of 1:1), representing blades, were used for simplicity and safety, as illustrated in Figure 191. A Parvalux 120W 24V DC motor was used to rotate the disc and an IED rotary encoder was used determine the rotation speed that would be used for further analysis. It is noted that variation of repetition rates for the Nd:YAG laser could damage/reduce its life, therefore only rotational speeds that were multiples of 15Hz were considered. A digital counter and software was used to count TDC (top dead centre) pulses from the rotary encoder for synchronised triggering of the laser (multiples of 15Hz) in order to excite the exact spot on the rotating disc at the required repetition rate. The IED encoder was capable of producing up to 10,000 pulses/rev. However, it was limited to a maximum operating frequency of 600kHz. At the maximum anticipated speed of approximately 6000RPM (100Hz), the maximum number of pulses/rev that would be acceptable is 6000; therefore the encoder was reprogrammed using a SICK encoder programmer to reflect this change. The counter was able to count pulses after the TDC pulse and be used to offset the laser trigger to any rotational position on the disc, where a single degree variation was represented by 16.68 (6000/360) pulses/degree. Using this system, it was possible to trigger the laser to any rotational position, with high repeatability and accuracy (approximately  $0.06^{\circ}$ , representing a distance of approximately 126tan $0.06^{\circ} = 0.13$  mm). Although rotational and linear velocities of the system were significantly lower than that of real gas turbine aero engines, the system was used to verify the design methodologies that could be applied at higher rotational speeds. As it was unfeasible to obtain similar linear velocities using the existing apparatus, the linear velocities were scaled down by a factor of x10; this would be used to test lifetimes that are x10 higher, to maintain

similar linear velocity-to-lifetime-decay ratios, where the limiting ratio is determined by the maximum available time period/lifetime ( $\tau$ ) = 3. The required radial distances to obtain the linear velocity of 0.048  $_{mm/\mu s}$ , representing x10 reduction of HPTB blade's mid position linear velocity of 0.48  $_{mm/\mu s}$  (1700km/h) at various rotational speeds is shown in Table 36.

Revs/s	DDM	rad/a	Radial distance
(ПZ)		Tau/S	
15	900	94	0.506
30	1800	188	0.253
45	2700	283	0.169
60	3600	377	0.126
75	4500	471	0.101

Table 36: Required radial position to maintain a linear velocity of  $0.048 mm/\mu s$  at various rotation speeds.



Figure 191: Schematic for rotating phosphor thermometry experiments

To test the repeatability of the laser hitting the target, the probe was aligned to a small phosphor spot (7mm) painted on the disc, and was allowed to rotate for at least 5 minutes at 3600RPM. Once the speed of the motor was ramped up to the desired speed, the speed was monitored and showed no significant variation. It was found that the laser hit the same target every time with no shift and drift in position with time. At 3600RPM (60 Hz), the standard deviation was monitored to be less than 0.2 Hz (0.33%). The encoder's TDC pulse has a resolution of 10,000 pulses/revolution, representing a deviation of 1/10000<sup>th</sup> turn. At a radial distance of 126mm, this is a movement of 0.08mm and is considered negligible. With an offset pulse also included in the calculation, the maximum deviation is approximately 0.21mm. The Spectron SL404 Nd:YAG laser external triggering system has a quoted jitter time of 0.5ns. At the maximum rotation speed of 4500RPM, this relates to a movement less than 0.20003mm, which is considered negligible.

#### 8.6.2 Hot rotating disc

Tests were conducted on different phosphors at different temperatures. The heat source on the rotating disc was provided by a Rothenberger MAPP flame torch. The temperature of the rotating disc was measured using an FLIR ThermaCAM E45 camera, which possessed a quoted accuracy of  $\pm$  2%. The emissivity, which is the amount of radiation emitted compared to that of a perfect emitter had to be found and set for correction. Generally, the emissivity of metals is low and increases with temperature, and for non-metals it is usually high and decreases with temperature. The analysis does not take into account these changes and assumes fixed values throughout. The phosphor's emissivity was estimated using the procedure highlighted by FLIR, by simultaneously comparing values from surfaces of known emissivity values. A steel block, half painted with black paint and the other half with HPC bonded thermographic phosphor was heated to approximately 300 °C. The temperature of the paint was observed, assuming an emissivity in region of 0.97. The emissivity of the phosphor was found by altering the emissivity values on the camera until the temperature matched, and was found to be in the region of 0.89, which was approximately 30% higher than originally thought for ceramic/refractory materials. Rotating tests were performed using the E45 thermal imaging camera and Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn phosphor between a temperature range of approximately 300°C-670 °C at 2700RPM. A single phosphor had to be painted on the entire disc because changes in emissivity between different surface areas (with/without phosphor and the black paint), significantly affected measurement from the thermal imaging camera. After applying the correction for the collection factor at the speed and radial distance, the temperature based on the thermographic phosphors and the thermal imaging camera agreed with

differences of approximately 2.46% at 600°C. This was relatively higher than expected because it was difficult to maintain a constant temperature over the surface, with temperatures constantly rising/cooling with flame instabilities, highlighting the difficulty in measuring surface temperature in such environments. There would be uncertainties in measuring the emissivity values for the phosphor, which may also be changing with increasing temperature. When the heat source was removed, the temperature changed very rapidly during the acquisition time of 150 pulses, which represented a total time of 10 seconds at a repetition rate of 15Hz. This time was too slow with observed measurements between pulses having significant differences in decay profiles; where the averaging of such data would produce erroneous results. The number of pulses for acquisition was changed to 10, representing a temperature reading that could be acquired in less than one second. With the flame on, the stray radiation from the flame and the air was also being picked up by the thermal imaging camera, shown in Figure 193, highlighting the limitations of infrared technology. Phosphor thermometry was immune to this highlighting the advantages of such systems. The effect was only present at temperatures lower than 500°C with its effects reducing with increasing temperature. Because of these effects the flame was position behind the disk. As the disc cooled, collected waveforms became slower until they reached values close to the waveform of  $C_{f}$ , because of the relative large lifetimes  $(3000_{\text{LS}}$  at room temperature) representing a collection/lifetime ratio of 0.05 at 3600RPM, where the intensity could be assumed be constant throughout the collection period.

There was no observed interference from the laser beam and phosphor emissions on the infrared thermal imaging camera, for both  $Mg_3F_2GeO_4$ :Mn and YAG:Tm phosphors even though YAG:Tm is known to emit emissions in the IR region of the spectrum [193, 194]. Also there was no observed temperature increase on the IR camera with the laser firing on the phosphor target.



Figure 192: Thermal imaging - hot rotating disc using FLIR -thermal imaging camera



Figure 193: The interference caused by the flame torch

#### 8.6.3 Rotating at a constant temperature

As the temperature of the disc varied considerably, it was difficult to monitor the temperature profile of the disc using the thermal imaging camera with low uncertainty. Because of this, the accurate assessment and performance of the thermographic system, including the performance of using the correction relations could not be made. As there were many changing variables, fixing the temperature would allow a better assessment of determine phosphor lifetime value for the given optical setup at the given rotation speeds and temperatures. It would be of interest to test the rotating disc under constant temperatures, possibly inside a closed furnace, and is suggested as future work. Testing at room temperature is a suitable solution where a constant temperature is maintained. YAG:Tm was selected because it possessed the fastest decaying phosphor characteristics at this temperature.  $Mg_3F_2GeO_4:Mn$ , YAG:Tb and  $Y_2O_3:Eu$  all had lifetimes greater than  $1000 \,\mu s$ , which would represents a collection time to lifetime ratio of approximately 0.17 for  $Y_2O_3:Eu$  and 0.05 for YAG:Tb and  $Mg_3F_2GeO_4:Mn$  phosphor, that were considered too small to provide adequate readings.

The normalised results based on experimental data for different rotation speeds are shown in Figure 194. The results show the variation in decay profiles obtained from a stationary target and decay profiles obtained at various rotation speeds. It can be seen that an increase in linear velocity decreases the lifetime of the collected waveform profile. For YAG:Tm at 900RPM, the profiles are almost the same, at this speed the ratio of acquisition time and lifetime values was 18, which meant that 18 lifetime decays could be collected during this time, indicating there will be no significant variation in lifetime values when the collection is only to  $3\tau$ . The ratios for other speeds are shown in Table 37, indicating that as the speed increases, the ratio decreases. A simulation of the variation in collection factor and variation in lifetime decay values at various speeds is shown in Figure 195, and agreed well to experimental data. The lifetimes of collected emissions from rotating surfaces before correction were faster than the actual phosphors decay. The determined lifetime values by curve fitting before and after correction is shown in Table 38, and illustrates that the application of the correction was successful. A test with the collection time to lifetime ratio of 3 would confirm the design philosophy and measurement limits that could be applied at higher rotational speed for use in aeroengine; however the maximum speed the Parvalux motor could be operated at was 4500RPM, which gave a ratio of 3.59. There was a variation of approximately 6% between actual and corrected lifetime values, by assuming the same variation, this would relate to an variation of approximately 4.4 °C (0.28% of absolute temperature) at 1300 °C.

RPM	900	1800	2700	3600	4500	6000
Linear velocity	0.0119	0.0238	0.0356	0.0475	0.0594	0.0713
(mm/us)						
Maximum available	673	336	225	168	135	112
collection time (us)						
Useful acquisition	539	269	180	135	108	90
time (x0.8)						
Time/lifetime ratio	18	8.9	6	4.49	3.59	2.99

Table 37. Collection time to lifetime ratios for YAG:Tm at various speeds.

RPM	900	1800	2700	3600	4500
Determined lifetime	31.15	27	23.96	21.34	18.9
before correction					
Determined lifetime	31.9	30.4	29.8	32	32.2
after correction					

Table 38: Determined lifetime before and after corrections at various speeds.



Figure 194: Experimental data showing the decay of YAG:Tm phosphor at different rotational speeds



Figure 195: YAG:Tm simulation at different rotational speeds.

From Equation 58, the observed signal  $(s_1)$  is the product of the stationary signal and the collection factor. Another form of this equation is shown in Equation 73. By knowing the phosphor's waveform from a stationary target, it is possible to determine the collection factor  $(c_f)$  by dividing the observed rotating signal waveform  $(I_{rotating})$  by the stationary  $(I_{stationary})$  waveform, shown in Equation 74. The temperature must however remain constant. By undertaking test for YAG:Tm at a constant temperature, for both the stationary and rotating cases, the collection factor was determined. An example this is shown in Figure 196. The results agreed well with  $c_f$  values originally based on the LED measurement approach. It is important to note that  $c_f$  produced by this method were a lot nosier, especially when both decaying signals were approaching



zero, where the ratio would result in increased noise. Knowledge of  $C_f$  enables the correction of decay at any rotational speed and radial distance.

Figure 196: Determination of the collection factor using the ratio of stationary to rotational waveforms.

 $I_{(rotating)} = I_{(stationary)} \cdot C_f$  $C_f = \frac{I_{(rotating)}}{I_{(stationary)}}$ 

Equation 73

Equation 74

The probe has been successfully tested on stationary YAG:Tm targets at stable temperatures up to 1300 °C. This was achieved by placing the probe between the insulation walls of the Vecstar furnace for short periods (10 seconds). Experiments were also undertaken using the probe on stationary components using the flame torch with detected lifetimes that were found to less than  $0.3 \mu s$ , indicating a temperature greater than 1350 °C. As the quenching temperature of this phosphor occurs at 1100°C, it was not possible to test the effects of varying lifetime decay values for YAG:Tm on rotating components because these high temperatures were unachievable while the disc was rotating using existing apparatus.

There are issues with the collection efficiency that could have further implication in obtaining the decay waveform of the phosphor. As the target sweeps through the acceptance cone of the fibre, the collected intensity from the target drops, therefore the detected intensity of the phosphor, which is already decaying, is lowered even

further. A slit was made in the mock up detection probe to view the internal optics of the probe system. In a darkened room, the collected target focus on the fibre became visible when the probe was pointing to an LED on the disc. The minimum focus produced by the lens was found to be in the same region as the fibre core. As the disc rotated the position of the focused image moved in the opposite direction of the rotating disc, eventually dropping outside the fibres view, as shown in Figure 197. As the target moves, portions of the focused image will fall out of the fibre, which may cause unwanted consequences at high temperatures. By assuming that the rest of the fibre's area will still be illuminated with blackbody radiation, there will be a significantly greater proportion of observed blackbody radiation which may impose an upper temperature limit sooner than expected. This proportion of the blackbody will depend on the probe design, fibre area, temperature, rotation speed and lifetime of the phosphor.



Figure 197: Moving focus image point as the disc rotates

There are a number of solutions, subject to further research, that can be applied to reduce this. A larger fibre will allow a greater time period for the moving target image to be collected by the fibre, however, the proportion of blackbody radiation would still be increased, especially at the fibres collection limits, as shown in Figure 198. Although there will be gains in the collection efficiency by having fibre bundle that follows the path of illumination during the rotation period, it will be still limited by the increased proportion of blackbody radiation at the collection limits if the light from the ends of the bundle fibres are connected to the same detector. It may be possible to separate the fibre bundles to individual detectors, where software could be used to recreate the decay profile from individual signals, and be used to determine the temperature. Figure 199 exemplifies this for a 3 fibre bundle probe (more fibres could be used); in this case, the top fibre, at the central position of the probe, will also be the excitation fibre for phosphor illumination. This technique would effectively block
out any blackbody radiation from other fibres where the focused luminescent target image is not reached. A modified solution to this would include the use of a shuttering gate system at the collection end of the fibre (possibly LCD) that is synchronised to the rotational speed. The advantage of this would be that only one detector is required. However, it will increase the complexity of the system, and multiple pulses will be required to enable an overlap of curves from individual fibres. A schematic is shown in Figure 200.







Figure 199: System to reduce the proportion of blackbody radiation



Figure 200: Alternative system to reduce the proportion of blackbody radiation

# 8.7 Measurement Uncertainty

The interference due to the back scatter of the UV and electrical interferences can become significantly important ultimately placing a lower detection limit. Interference levels have been shown to last  $0.35\,\mu s$  when the probe was used. It was shown in the previous chapter that determination of lifetimes was still possible providing good calibration to temperature, even when the first two decays were omitted from the analysis. It was shown that a collection period from  $2\tau - 4\tau$  yielded the same results, within 10%, as from  $1\tau - 3\tau$ , which is a temperature variation of 6°C at 1370°C (0.36%) for YAG:Tm phosphors. For Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn, this was estimated to be 0.55%. If the interference time of  $0.35\,\mu s$  represents  $2\tau$ , then the minimum lifetime that could be successfully determined using this approach would be  $0.175\,\mu s$ . For YAG:Tm this would be at a temperature of approximately 1375°C, and 812°C for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn. For rotating components there will be an additional decrease in intensity due to the probes collection factor. However, this effect of this would

gradually become less significant at higher temperatures when the collection time to lifetime ratio increases.

The uncertainty caused by the thickness of the phosphor layer is also considered. For YSZ thermal barrier coatings, there is an estimated drop of 170°C over a TBC coating of  $150 \,\mu m$  [170]. Other examples indicate a drop of  $200^\circ C$  over a thickness of  $200 \,\mu m$ , which indicates a variation of approximately  $1^{\circ}C/\mu m$  at the maximum operating conditions of the HP turbine. The thermal conductivity of bulk YAG has been reported to be approximately 3.2 W/mK [171], and YSZ has been reported to vary between 2.5-4.0 W/mK [172]. By assuming the phosphors have the same thermal conductivity as the YSZ-TBC, and assuming a linear thermal gradient of 1°C/ $\mu m$ , then a  $1 \mu m$ phosphor deposited on top of the TBC would indicate a temperature uncertainty of approximately 1°C, 0.064% at 1300°C. Although a  $1\,\mu m$  coating can be acceptable, a  $5\,\mu m$  coating would more sensible with increased durability and survivability for the harsh environment that it will be subjected to, relating to an uncertainty of approximately 5°C (0.3%). For the high pressure drive cone application, with a relatively thin layer of phosphor, the temperature between the top and bottom of the phosphor layer is likely to remain be the same because temperatures are not expected to be as high, and will not be exposed to any film cooling.

#### Extending the lower temperature capability

The maximum determinable lifetime based on the lens size, rotation speed and a minimum collection to  $3\tau$ , was estimated to be approximately  $5.8 \mu s$  and  $5.45 \mu s$  for the HPTB and drive cone application respectively. This yields to a lower temperature capability of approx 1144°C for YAG:Tm and 641°C for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn. This is only valid if the full probe's FOV can be utilised. Using a single fibre probe for laser delivery and collection results in a situation where only half the probe's FOV can be used for emission collection. This consequently reduces the collection time and hence the minimum temperature measurement capability, which is then 1190°C for YAG:Tm and 671°C for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn. The full temperature range for both probe types is shown in Figure 201. The upper temperature range remains unchanged at approximately 812°C and 1375°C for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn and YAG:Tm respectively.



Figure 201: Temperature range (upper and lower limits) based

The lower temperature measurement limit, based on the minimum required collection time to lifetime ratio of 3, can be extended by either lowering this ratio, or by increasing the size of the lens. Although the probe is assumed to have a lens with a useful diameter of 8mm for the HPTB and 4mm for the drive cone applications, increasing its diameter will have dual benefits:

- Increased radiometric collection efficiency, that will help to extend the upper temperature capability of the phosphor.
- Increased collection time for rotating components, which will help to extend the lower temperature capability of the phosphor.

Based on this, the minimum detectable temperature limit was determined using the estimated rotation speed and calibration data curves (Equation 53 and Equation 54) for a combinations of different collection time to lifetime ratios and a range of lens sizes. This information is shown in Table 39 and Table 40, and graphically in Figure 202 and Figure 203. Note, the quenching temperate for the YAG:Tm phosphor is approximately 1100°C and imposes another lower temperature limit which is taken into account in the analysis.

		Probe lens diameter (mm)				
		8mm	9mm	10mm	11mm	12mm
	1.0	8.7μs - 1117°C	9.79μs - 1109°C	10.87μs - 1102°C	11.96μs - 1100°C	13.05μs - 1100°C
		(17.4µs - 1100°C)	(19.57µs - 1100°C)	(21.74µs - 1100°C)	(23.92µs - 1100°C)	(26.09µs - 1100°C)
Collection time to lifetime ratio	1.5	5.8μs - 1144°C	6.53µs - 1136°C	7.25μs - 1129°C	7.98µs - 1123°C	8.7μs - 1117°C
		(11.6µs - 1100°C)	(13.05µs - 1100°C)	(14.5µs - 1100°C)	(15.95µs - 1100°C)	(17.4µs - 1100°C)
	2.0	4.35µs - 1163°C	4.9μs - 1155°C	5.44µs - 1148°C	5.98μs - 1142°C	6.53µs - 1136°C
		(8.7μs - 1117°C)	(9.79µs - 1109°C)	(10.87µs - 1102°C)	(11.96µs - 1100°C)	(13.05µs - 1100°C)
	2.5	3.48µs - 1178°C	3.92µs - 1170°C	4.35µs - 1163°C	4.79μs - 1157°C	5.22μs - 1151°C
		(6.96µs - 1132°C)	(7.83µs - 1124°C)	(8.7μs - 1117°C)	(9.57µs - 1110°C)	(10.44µs - 1104°C)
	3.0	2.9μs - 1190°C	3.27µs - 1182°C	3.63µs - 1175°C	3.99μs - 1169°C	4.35µs - 1163℃
		(5.8µs - 1144°C)	(6.53µs - 1136°C)	(7.25µs - 1129°C)	(7.98µs - 1123°C)	(8.7μs - 1117°C)

Table 39: Lower temperature measurement capability limit for YAG:Tm for a range of lens sizes and collection to lifetime ratios. Values for half-FOV probes. Bracketed values are for full-FOV probes.



Figure 202: Lower temperature measurement capability limit for YAG:Tm for a range of lens sizes and collection to lifetime ratios. Left: Half-FOV; Right: Full-FOV

		Probe lens diameter (mm)					
		3mm	4mm	5mm	6mm	7mm	
	10	6.13μs - 636°C	8.17μs - 624°C	10.21μs - 615°C	12.25μs - 608°C	14.29μs - 602°C	
	1.0	(12.25µs - 608°C)	(16.33µs - 596°C)	(20.41µs - 588°C)	(24.49µs - 581°C)	(28.58µs - 575°C)	
Collection time to lifetime ratio	1.5	4.09μs - 653°C	5.45μs - 641°C	6.81μs - 632°C	8.17μs - 624°C	9.53μs - 618°C	
		(8.17µs - 624°C)	(10.89µs - 613°C)	(13.61µs - 604°C)	(16.33µs - 596°C)	(19.05µs - 590°C)	
	2.0	3.07μs - 666°C	4.09μs - 653°C	5.11μs - 644°C	6.13μs - 636°C	7.15μs - 630°C	
		(6.13µs - 636°C)	(8.17µs - 624°C)	(10.21µs - 615°C)	(12.25µs - 608°C)	(14.29µs - 602°C)	
	2.5	2.45μs - 676°C	3.27μs - 663°C	4.09μs - 653°C	4.9μs - 646°C	5.72μs - 639°C	
		(4.9µs - 646°C)	(6.54µs - 633°C)	(8.17µs - 624°C)	(9.8µs - 617°C)	(11.43µs - 611°C)	
	3.0	2.05μs - 684°C	2.73μs - 671°C	3.41μs - 661°C	4.09μs - 653°C	4.77μs - 647°C	
		(4.09µs - 653°C)	(5.45µs - 641°C)	(6.81µs - 632°C)	(8.17µs - 624°C)	(9.53µs - 618°C)	

Table 40: Lower temperature measurement capability limit for  $Mg_3F_2GeO_4$ :Mn for a range of lens sizes and collection to lifetime ratios. Values for half-FOV probes. Bracketed values are for full-FOV probes.



Figure 203: Lower temperature measurement capability limit for  $Mg_3F_2GeO_4$ :Mn for a range of lens sizes and collection to lifetime ratios. Left: Half-FOV; Right: Full-FOV

For YAG:Tm, it is shown that using a lens size of 10mm extends the lower temperature limit to 1175°C for half-FOV probe and 1129°C for full-FOV probe. By also lowering the collection time to lifetime ratio to 2.5 further extends this to 1163°C and 1117°C respectively. Lowering the ratio further, further enhances the lower temperature measurement capability limit. Similarly for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn phosphors, lowering the collection time to lifetime ratio to 2, extends the lower measurement capability to 653°C and 624°C for half and full FOV probes respectively. Decreasing the collection to lifetime ratio has consequences on the measurement uncertainty. As the determined lifetime ( au ) is dependant on the collection time, a re-calibration was performed using maximum allowable acquisition times that are based on the probes field of view and operating speed. For a time window of  $16\,\mu s$  for both the HPTB and drive cone applications (Table 33), recalibration data, shown in Figure 204 and Figure 205 were compared to the original calibration data curve for  $Mg_3F_2GeO_4$ :Mn and YAG:Tm phosphors. Also shown are the collection time to lifetime ratios for a probe that utilises the full FOV. It is shown that the error between the actual lifetime decay values (determined using no time restrictions) and lifetimes determined using a limited time window can be very high at lower temperatures, especially when the ratio between the collection and lifetime is less than 1. For  $Mg_3F_2GeO_4$ : Mn phosphors, the difference in value is a factor of x100 at room temperature. For all cases, it was found that the determined lifetimes from this are lower (faster) than the actual decay, biasing a higher temperature.

For Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn, where the collection time to lifetime ratio is approximately 1, the error in temperature measurement was approximately 8°C, which approximates to an additional uncertainty of 0.89% at this temperature. When the ratio is 2, the additional uncertainty is approximated to be 0.55%; at temperatures above 670°C, where the ratio is > 3, the variation was virtually none.

For YAG:Tm, when the collection time to lifetime ratio is 1, there is a variation of approximately 15% between lifetime values, which approximates to an additional uncertainty of 0.8% in terms of temperature. When the ratio is 2 at approximately 1162°C, a 12% variation approximates to an additional uncertainty of 8°C (0.55%) at this temperature. From this analysis, it can be seen that it is possible to extend the lower temperature capability of the system by reducing requirement for the minimum collection time to lifetime ratio with the penalty of increased uncertainty.



Figure 204: Variation of the determined lifetime values for  $Mg_3F_2GeO_4{:}Mn$  phosphor using the maximum available detection time



Figure 205: Variation of the determined lifetime values for YAG:Tm phosphor using the maximum available detection time

	Temperature range (mid-point) Collection time to lifetime ratio (R)			
	1117-1163°C (1140°C) 1 < R < 2	1163-1190 °C (1176 °C) 2 < R < 3	1190-1330°C (1260°C) R > 3	1330-1375°C (1353°C) τ < Interference < 2 τ
Uncertainty based on regression analysis (95% confidence levels)	0.4 (0.2)%	0.4 (0.2)%	0.4 (0.2)%	0.4 (0.2)%
Thermocouple	0.1%	0.1%	0.1%	0.1%
TC08 - thermocouple module	0.2%	0.2%	0.2%	0.2%
Furnace temp uncertainty (variation in thermocouple readings)	0.21%	0.21%	0.22%	0.25%
Estimated uncertainty for phosphor coating (5 microns)	0.3%	0.3%	0.3%	0.3%
Uncertainty for lifetime determination using c <sub>f</sub> corrections	0.28%	0.28%	0.25%	0.1%
Uncertainty for using R < 3	0.8%	0.55%	0	0
Uncertainty due to initial interference	0	0	0	0.36%
Standard uncertainty (1σ) In terms of temperature / K or °C	0.97% (13.71 °C)	0.78% (11.30 °C)	0.54% (8.28 °C)	0.62% (10.08 °C)

Table 41: Uncertainty of measurement for YAG:Tm phosphor

	Temperature range (mid-point) Collection time to lifetime ratio (R)			
	624-653 °C (639 °C)	653-671 °C (662 °C)	671-772 °C (721 °C)	772-812 °C (792 °C)
	1 < R < 2	2 < R < 3	R > 3	τ < Interference < 2 τ
Uncertainty based on regression analysis (95% confidence levels)	0.2 (0.1)%	0.2 (0.1)%	0.2 (0.1)%	0.2 (0.1)%
Thermocouple	0.1%	0.1%	0.1%	0.1%
TC08 - thermocouple module	0.2%	0.2%	0.2%	0.2%
Furnace Temp uncertainty (variation in thermocouple readings)	0.2%	0.2%	0.2%	0.2%
Uncertainty for lifetime determination using c <sub>f</sub> corrections	0.28%	0.28%	0.25%	0.1%
Uncertainty for using R < 3	0.89%	0.57%	0	0
Uncertainty due to initial interference	0	0	0	0.55%
Standard uncertainty (1ơ) In terms of temperature / K or °C	0.99% (9.02 °C)	0.71% (6.64 °C)	0.4% (3.98 °C)	0.64% (6.81 °C)

Table 42: Uncertainty of measurement for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn phosphor

All these variations can be built into the combined expression of measurement uncertainty. This combines the uncertainties found on stationary conditions, previously shown in Table 31, with additional uncertainties caused by rotating conditions discussed in this section. The performance of the system and measurement uncertainty is analysed into 4 distinct categories based on collection times. This is shown in Table 41 and Table 42 for YAG:Tm and Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn.

The standard uncertainty for YAG:Tm to measure surface temperatures of HP turbine blades is approximately  $\pm 0.97\%$  ( $\pm 13.71^{\circ}$ C) between the temperature range of 1117-

1163°C, ±0.78% (±11.3°C) between 1163-1190°C, ±0.54% (±8.28°C) between 1190-1330°C and ±0.62% (±10.08°C) between 1330-1375°C. The standard uncertainty for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn to measure temperatures of the drive cone is approximately ±0.99% (±9.02°C) between the temperature range of 624-653°C, ±0.71% (±6.64°C) between 653-671°C, ±0.4% (±3.98°C) between 671-772°C and ±0.64% (±6.81°C) between 772-812°C.

In summary, the measureable temperature range for YAG:Tm for HP turbine blade measurements is 1117-1375°C, and 624-812°C for  $Mg_3F_2GeO_4$ :Mn for the drive cone application. In all cases, the standard uncertainty (u, 1 $\sigma$ ) at the temperature range specified is estimated to be less than 1%. These uncertainties assumes a useful collection lens diameter of 8mm for HP turbine blade application, and 4mm for the drive cone application, and at least 5mJ of 355nm laser excitation is achievable at the phosphor target.

### 8.8 Conclusions

This section has discussed the design philosophy of the fibre optic probe and its effect on temperature measurement on rotating surfaces. Engine components are expected to rotate at 13,000 RPM, with linear velocities of approximately  $0.45 \text{ mm}/\mu s$ . By taking into consideration the lower detection limit of the system, the probes field of view, expected temperature range and the rotational speeds (and linear velocities), a phosphor selection criteria for engine components was applied on the different phosphors. It is important to consider the phosphor lifetime at the range of expected temperatures to ensure that there is adequate time for the probe to capture the luminescent decay. The two engine components that were given research priority were the HP turbine blade and the HP compressor drive cone. Unfortunately, no single phosphor could map the complete temperature region for HP turbine blades which ranges from 850-1400°C. However, YAG:Tm was found to be the most promising phosphor at temperatures between 1150-1400°C, which was noted to be the most For the HP drive cone application, critical temperature range of interest.  $Mq_3F_2GeO_4$ : Mn phosphor was found to be the most suitable phosphor that could map the complete temperature range of interest.

A fibre optic probe and optical layout was setup using design constraints that would be present in real engine environments, including fibre choice for tolerance to high temperatures and high power laser transmission, maximum permissible hole size and lens to target distances. Only the optical performance of phosphor thermometry was considered; the thermal performance of the probe was not tested as there are already designs with cooling that are currently being used for pyrometer applications. A

discussion of different probe designs including dual probe and dual fibres was presented. A rotating phosphor thermometry system was setup using a DC motor connected to rotating disc and an encoder that was used to trigger the excitation source to ensure the same position on the target is being illuminated. Unfortunately the system could not operate at the expected linear velocities of real aero-engines, and the design was chosen to operate the test case at exactly one tenth of this speed. The analysis could then be scaled with the appropriate corrections to verify design methodologies and enable the predictions for actual speeds. It was found that as the phosphors signal traversed through the acceptance cone of the fibre, the collection efficiency varied as a function of rotation angle, with the maximum efficiency occurring at the fibre axis and varying to zero at limits of the probes field of view. Therefore, following pulsed laser excitation, the detected signal was a product of the collection factor (C<sub>f</sub>) and luminescent phosphor decay. Different methods for determining the collection factor were discussed, including the use of a constant light source, a mixed phosphor solution, and taking ratios of stationary and rotating targets. The determined C<sub>f</sub> was curve-fitted and related to the angular velocity and radial position of the target, which could then be used to correct lifetime values from rotating surfaces at any speed. This methodology was tested on YAG:Tm phosphors, and showed promising results with a variation of approximately 6% between actual and corrected lifetime value; this related to an variation of approximately 4.4°C at 1300°C which is approximately 0.28% variation in terms of temperature. A number of suggestions were also made for future work that could help further develop such a phosphor thermometry system.

Only half the probe's FOV can be utilised for emission collection for a single fibre probe that is used for laser delivery and emission collection. This consequently reduces the collection time and hence the minimum temperature measurement capability of the system. It was found that the lower temperature capability can be increased by decreasing the collection ratio with the penalty of increased measurement uncertainty, or by increasing the size of the lens. Increasing the size of lens increases radiometric collection efficiency and the temporal collection period. The overall performance of the system for YAG:Tm is a temperature range of  $1117-1375^{\circ}C$  and  $624-812^{\circ}C$  for Mg<sub>3</sub>F<sub>2</sub>GeO<sub>4</sub>:Mn phosphors, both experiencing combined standard uncertainties lower than  $\pm 1\%$ .

# 9 Conclusions, Recommendation and Future Work

## 9.1 Summary

The main objective of this project was to further the research on thermographic phosphors so that a temperature measurement solution can be applied on hot regions of gas turbine engines, where conventional competing technologies and techniques prove to be problematic. The commercial drivers for undertaking such research to provide accurate temperature measurements of engine components were identified as:

- Verifying the mechanical, structural integrity of components at high temperatures
- Assessment of aero-thermal performance
- Verify and further development of CFD, FEA analysis, and other design tools
- Aid the design for better, leaner more energy efficient engines
- Provide insitu correction/validation for measurements made from pyrometry.
- Provide better component lifetime predictions, which can help prolong maintenance schedules
- With a reduced temperature uncertainty, it can allow engines to be operated closer to its design limits, and improve engine efficiency
- Provide a platform technology that can be further developed into a technology that could be used for health monitoring or engine control sensing purposes.

Unfortunately, experimentally measuring surface temperatures under harsh rotating engine conditions is challenging. Existing methods lack detail, do not have the desired accuracy, or are too expensive for continuous testing. Established techniques include the use of thermocouples, pyrometry and thermal paints/melts. Although thermocouples are typically cheap, accurate and easy to install, in complex flow and rotating environments, they can be intrusive, difficult to install with routing of the wires being problematic. The measurement also lacks detail since it only provides discrete diagnosis. Thermal paints are subjective and their response is also a function of thermal exposure, which requires operator's skill and experience to make accurate measurements. They are irreversible and can only provide measurements from a single test, making it very expensive. Although pyrometry offers many advantages over thermocouples including non-intrusiveness, immunity to electromagnetic radiation and reduced problems with routing, there still remain many issues that limits their use. These include: issues with emittance variation with temperature, reflected radiation and gas stream/flame interference, which makes them very sensitive to the environment.

Phosphor thermometry relies on luminescence properties of phosphors that change with temperature. A review of the state of the art was performed. Thermographic phosphors have many attributes that make them better suited for such environments; they are accurate and like pyrometry, share many of the non-intrusive benefits over thermocouples (e.g. routing of cables and immunity to electromagnetic noise), while overcoming many of the shortfalls and issues related to it (e.g. emissivity variations, flame interferences, reflected radiation). Despite these advantages, there are limitations and consideration factors that were identified that would limit their use. The variation in component geometry, operational temperatures, optical access and rotation speeds makes it difficult to generalise a universal measurement solution for all engine components. The high temperature and fast rotating engine environment presents some challenges that need to be addressed before a successful phosphor thermometry measurement system can be implemented. Examples include rising blackbody radiation, restricted optical access, fibre optic constraints and limited time period to collect data. These factors will impose measurement limits and greatly influence the design philosophy of the system, including phosphor choice in terms of lifetime characteristics, excitation/detection methodologies and probe design. Because of these issues and time constraints, only two engine components were given research priority. From the capability gaps analysis and requirements from Rolls Royce plc, the HP turbine blade was given priority; the HP compressor drive cone was also considered.

Due to engine restrictions, the only feasible solution for laser delivery to engine components was through the use of fibre optics. Fibres were chosen with the thermal tolerance in mind to withstand the thermal environment (800°C). A number of tests were conducted to establish the maximum amount of energy, using different fibres, wavelengths and launching conditions, that could be coupled and delivered to the phosphor target. This has implications on the performance of the phosphor thermometry technique, with larger energies allowing greater emission intensities and a higher temperature capability. The maximum amount of energy/pulse transmittable through a 1.0mm core fused-silica fibre was found to be approximately 8.7mJ for 355nm and 3mJ for 266nm. This relates to an advantage x2.9 when transmitting 355nm wavelengths. It may be possible to transmit more energy if higher input energies were permissible. In the tests performed, fused silica fibres performed better than sapphire fibres in delivering UV light.

A number of high temperature phosphors were selected for testing, including YAG:Tb, YAG:Tm.  $Y_2O_3$ :Eu and  $Mg_3F_2GeO_4$ :Mn. A phosphor thermometry calibration system was built in order to characterise spectral emissions, intensity and the lifetime of different phosphors, under different conditions. Spectral analysis was performed to

observe the phosphors emission peaks and shifts with increasing temperature, in order to optimise the collection system and improve the overall performance of a thermometry system. Different bonding techniques including chemical bonding and vapour deposition methods were discussed. It was found that the intensity from the phosphors was also sensitive to thermal exposure when chemical binders were used. Vapour deposition methods are expected to produce robust and durable coats with intensity levels that are independent of thermal exposure. However due to the elevated cost of using vapour deposition methods, powered phosphor samples were used instead, which are reported to yield similar results.

Absolute intensity measurements of various phosphors at various temperatures, excitation wavelengths and energy levels were investigated to permit quantitative comparisons and allow estimations of radiant emission levels for different optical configurations. The emission intensities relative to the blackbody radiation, predicted by Planck's equation was also performed; this gave a indication of phosphors signal strength at high temperatures. From all the phosphors investigated, YAG:Tm under 355nm illumination was found to produce the highest emission intensity per mJ of laser excitation at high temperatures (>900 °C), and because its emission wavelength at 458nm is lower than other investigated phosphor's emission lines, its advantage in terms of blackbody radiation was further amplified.

The lifetime of emissions with increasing temperature was investigated and characterised for a number of phosphors. It was found that the decay emissions following excitation were not purely exponential, where emissions from the first proportion of the decay were generally faster than the rest of the decay. Because of this, there was increased uncertainty that was dependent on the collection time used to make the lifetime measurement. It was found that a collection period from 1  $\tau$  -3  $\tau$ produced repeatable results provided that the offset value (based on the dark current and the blackbody radiation) was predetermined. Details of the algorithm used to provide lifetime values were provided. With temporal lifetime methods on stationary components, a lower temperature limit is reached based on the guenching temperature of the phosphor, while an upper limit is reached based on the phosphor's intensity at rising blackbody radiation levels and the systems ability to detect faster decaying signals. For rotating components the limit also depends on the probe design and the rotational velocity of the component. In addition to this, as the phosphor's signal traverses through the acceptance cone of the fibre, the collection efficiency varies as a function of rotation angle, with the maximum efficiency occurring at the fibre axis varying to zero at limits of the probes field-of-view, consequently distorting the detected phosphor decay waveform. Different methods for correcting this were proposed. A relation based on the rotation speed, radial distance, probes field of view, temperature and phosphor's lifetime characteristics was derived to estimate the expected phosphor waveform at any rotational speed and temperature. Based on various limitations, a phosphor selection criterion for different engine components was successfully applied in order to select the most suitable phosphors that would enable temperature determination. A fibre optic probe and optical layout was setup in laboratory conditions using design constraints, including fibre choice, maximum permissible lens size and target distances. A number of designs were considered and tested on stationary and rotating cases. The tests validated design methodologies and assumptions that could be applied under full scale engine conditions.

### 9.2 Recommendation

For the HP drive cone application,  $Mg_3F_2GeO_4$ : Mn phosphor, using 355nm illumination, was found to be the most suitable phosphor that could almost map the complete required temperature range of interest. The estimated temperature range and performance capability under the components expected rotational speeds is estimated to be 624-812°C with a standard measurement uncertainty of ±0.99%.

For the high pressure turbine blade application, no single phosphor was found to map the complete required temperature region which ranged from 850-1400°C. However, YAG:Tm (0.5% activator concentration), illuminated with 355nm laser, was found to be the most promising phosphor at higher temperatures (above 1150°C) which was noted to be most critical temperature range of interest. The estimated temperature range and performance from this phosphor under the expected rotational speeds, and other limitations experienced in engine conditions (including distance to target, maximum lens size and purge air requirements) is estimated to be 1117–1375°C with a standard measurement uncertainty of  $\pm 0.97\%$ . This is better than other competing technologies that are currently commercially available for rotating turbine blade temperature measurements.

### 9.3 Future Work

Temperature measurements were performed at atmospheric pressure under no flow conditions. Some phosphors e.g. Y<sub>2</sub>O<sub>3</sub>:Eu have been reported to show irreversible changes open exposure to high pressures, and have shown to exhibit greater uncertainty values when exposed to different oxygen levels. Magnesium Manganese phosphors have been previously tested to show no dependence to pressure or oxygen. However, it would be of interest to test the performance of YAG:Tm and other phosphors at higher pressures with varying levels of oxygen, possibly inside a pressurised chamber.

- Testing and calibration of rotating components in thermally stable environments, e.g. inside the furnace.
- Only temporal lifetime values were considered in the analysis because they have been proven to be the most accurate method for temperature measurement. It would be desirable to investigate the intensity ratio approach for  $Mg_3F_2GeO_4$ :Mn and YAG:Dy phosphors.
- Fibre bundles were not considered because of their limited temperature capability, and reduced transmission efficiency because of the inter-fibre spaces. Recent advances in technology such as fused-end bundles produced by LGO-Germany[134] claim to eliminate inter-fibre spaces offering higher transmission with a temperature capability up to 1000°C. It would be interest to test these fibres for high power laser delivery.
- Investigate tapered fibres and hollow tapered Pyrex glass for laser coupling.
- Other solutions to extend the temperature measurement range of the phosphor thermometry system discussed in the thesis include:
  - Multiple phosphor coatings
  - Increasing lens diameter
  - Dual fibre/probe systems
  - Fibre bundles with individual fibres routed to different detectors, or gated to a single detector.

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