Quantification of Microstructural Homogeneity and the Mechanisms of Particle Refinement During FSP of Al-Si Alloys

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Abstract: In the present study, the mechanisms of particle refinement during FSP were investigated in a gravity die cast Al-Si LM24/A380 alloy. The homogeneity of distribution of the refined particles was also evaluated in relation to the processing parameters. Detailed image analysis and Dirichlet tessellation were carried out to quantify particle refinement and clustering. ‘Stop-action’ experiments were used to study the mechanisms of particle break up, by following the behaviour along flow lines through the deformation zone surrounding the tool. A computer model was also used to predict the temperature profile during FSP, within the process zone, which was related to the refinement process.

Introduction

Al-Si alloys are widely used in automotive applications because of their low cost, attractive density, and high temperature capability, combined with excellent castability. However, in order to increase engine efficiencies, there are demands for ever more severe operating conditions, which will require improvements to the performance of current materials. Friction Stir Processing (FSP) [1-11] is a variant of the Friction Stir Welding (FSW) process, where the intense deformation generated by traversing a rotating welding tool, has been exploited as a technique of microstructural refinement in a range of materials [1]. Investigations into the use of FSP have shown that it can lead to a dramatic reduction in grain size and a high level of second phase particle refinement [1,3,4]. By refining the microstructure of cast Al-Si alloys, the high temperature mechanical performance and, in particular, fatigue properties should be improved [1,4,5]. The technique thus has potential for locally enhancing the microstructure in highly stressed areas of cast Al-Si engine components, such as piston crowns or cylinder heads.

Previous investigations into the effect of FSP on Al-Si casting alloys have shown that high levels of microstructural refinement can be achieved, with the size of the Si particles being reduced to less than 3 µm in sand castings, or even finer, to less than 1 µm in chemically modified castings containing a fibrous eutectic Si [6-10]. At the same time, there is a reduction in porosity, and the grain size can be reduced to below 5 µm. By subjecting Al-Si alloys to FSP, the tensile and fatigue properties have also been found to improve [6-9]. Several researchers have reported the effect of the processing parameters on the level of refinement that can be achieved (e.g. [1,9-11]). Observations to date include an increase in homogeneity [10] and a slight
reduction in particle size with tool RPM [11]. However, the mechanisms of particle refinement in Al-Si alloys during FSP have not been previously thoroughly investigated.

In the present study the microstructure of a hypo-eutectic gravity die cast Al-Si A380 alloy, subjected to a range of FSP conditions, was investigated. Image analysis of particle size distributions and Dirichlet tessellation were used to quantify the level of particle refinement and the homogeneity of the second phase spatial distribution, as a function of location within the processed zone, as well as the relationship to the processing conditions (rotation speed and transverse speed). Further, ‘Stop-action’ experiments were conducted to study in detail the particle break up, by examining the refinement behaviour through the deformation zone surrounding the tool. Computer modelling was also used to predict the temperature profile during FSP, in order to assist in the modelling of the refinement mechanisms.

Experimental

A commercial hypo-eutectic LM24/A380 Al-Si alloy (Al-8.9Si wt%), gravity die cast in a steel mould, to produce 296 mm x 210 mm by 26 mm thick plates, was used in the FSP experiments. Prior to FSP, the plates were scalped by milling off 2 mm each side to generate flat surfaces for processing. Friction stir processing was carried out using a CS Powerstir Friction Stir Welding machine using a H13 steel tool, with a 20 mm diameter shoulder and threaded, tapered, tri-flat pin, 5.8 mm in length. To investigate the effect of the FSP parameters, the cast slabs were first processed at a fixed transverse speed of 200 mm/min, using a range of tool rotation speeds from 300 rpm to 900 rpm, increasing in increments of 200 rpm. The effect of tool travel speed was also studied from 100 to 400 mm min⁻¹ using a matrix of rotation speeds, shown in Table 1. The plate temperature was monitored in all the tests with embedded thermocouples placed close to the tool at four different locations.

‘Stop-action’ samples were produced using a 200 mm min⁻¹ travel speed. This involved immediately stopping the FSW machine with the pin still embedded within the plate. The processed zone was then carefully cut out of the plate and the sample sectioned with the tool in-situ. Particle size distributions, the average particle sizes, and the particles spatial distribution were quantified by optical microscopy using ImagePro analysis software. The average particle size, in a given field of view, was defined in terms of the equivalent circular diameter (ECD) and the particle’s spatial distribution was characterised by the Dirichlet tessellation method [12].

A FE heat transfer model was used to reproduce the thermal field for each FSP condition, in order to better understand the effects of the process parameters on particle refinement. The model was implemented in ABAQUS with a translated heat source, designed to represent the tool, described in a user defined subroutine. The tool was represented by a circular surface source to simulate the shoulder, with the power distributed radially depending on the angular velocity, and a uniform cylindrical source to describe the pin. Full details are given in; [13,14]. The model used thermal-physical data measured for the processed alloy and was calibrated against thermo-couple measurements, positioned at four different locations, collected during each FSP run.

<table>
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Table 1: Matrix of FSP conditions used in the investigation, with the peak temperature (ºC) predicted at 1 mm below the tool shoulder at a radial distance of 5 mm’s.
Results and Discussion

Particle Size Distributions

The microstructure of the LM24 Al-Si casting alloy in the as-received condition is shown in Fig. 1(a). The starting material contained irregular eutectic Si, with a coarse flake morphology, as well as a significant volume fraction of finer α-AlFeMnSi and Al$_2$Cu eutectic colonies, which were uniformly distributed throughout the Al matrix. The initial second phase average particle size was 8 to 10 µm (ECD), and the average thickness of the Si flakes were 3 to 4 µm.

![Fig. 1](image)

Fig. 1: The cast starting material (a) showing coarse flake eutectic Si (dark), α-AlFeMnSi (medium grey), and Al$_2$Cu (light grey) phases and the typical refined particles present in the centre of the PZ after FSP with a travel speed of 200 mm min$^{-1}$ for (b) 300 RPM and (c) 900 RPM.

![Fig. 2](image)

Fig. 2: In (a) a typical particle size distribution is shown from the 300 RPM sample. Average particle sizes (ECD) measured (b) across and (c) down the centre of the PZ, after FSP at 200 mm min$^{-1}$ with a range of rotation speeds, and (d) across the PZ for a range of travel speeds, with the rotation speed kept constant at 500 RPM.

Fig. 1(b) and 1(c) show micrographs from the centre of typical friction stir processed zones (PZ) with two different rotation speeds. After FSP the microstructure of the cast Al-Si alloy was
greatly refined within the PZ. The large high aspect ratio plate like eutectic Si particles and other intermetallic phases were broken up into low aspect ratio, fine, angular-particles, with an average size ranging from 2 to 3 µm. Porosity, which existed within the original cast material, was also removed by FSP. However, the particle size distributions showed a large spread (illustrated in Fig. 2a) with larger and less refined particles found throughout the PZ, which tended to be fragments from the coarser Si eutectic flakes.

In order to quantify the homogeneity of the microstructure after FSP, the second phase particles within the PZ was further analysed as a functional of position for different processing conditions plotted in Fig. 2(b) and 2(c). It can be seen that the average particle size across the PZ was not entirely uniform, tending to increasing towards the retreating side of the PZ and reducing from top to bottom down the centre line. The reduction in particle size towards the bottom of the PZ is most likely due to the thermal gradient though the plate. The temperature at the bottom, of what is effectively a 6 mm deep partial penetration weld, was determined to be at least 100 °C colder than the top by the thermal model (Fig. 3). This is due to the heat sink effect of the 25 mm thick plate and the higher heat input from the tool shoulder. In addition, nearer to the plate surface, for some conditions, a zone of less refined particles was found to have been dragged into the PZ behind the pin by the material displaced by the rotating tool, within a 'flow arm'. The differences in refinement across the PZ will be discussed further below when the stop-action sample is considered.

Interestingly, changing the tool rotation speed within a large range, with a fixed travel speed, was found to have an insignificant effect on the average size of the refined particles. The particle size averaged over the whole of the PZ was found to be ~2.5 µm ± 0.2 µm for all the samples with any variation being within the experimental error. On the other hand, altering the travel speed appeared to have a greater effect on particle refinement. As seen in Fig. 2(d), at a constant rotation speed of 500 RPM, the average size of the particles was found to increase from ~2.2 µm at 100 mm min⁻¹ to ~2.7 µm at 400 mm min⁻¹.

![Fig. 3: Images from the FE thermal model, predicting the temperature field during FSP at (a) 300 RPM and (b) 700 RPM with a travel speed of 200 mm min⁻¹. The dotted line illustrates the position of the tool pin.](image)

**Homogeneity of Particle Distribution**

A tessellation technique was used to measure the homogeneity of the particle’s spatial distribution. The method used was based on the work of Murphy et al., who has evaluated the best statistical approach for characterising the spatial distribution of SiC particles in metal matrix composites (MMCs) [15]. Each particle was first expanded until impingement to generate a skeletal cellular image. Analysis of the resulting cell size distribution could then be used to
quantify the homogeneity of the particle’s spatial distribution, as a function of their position and the processing condition. To eliminate errors caused by differences in average particle size and particle density between images, the measured cell size distributions were first normalized against a random distribution of the same particle density. From Fig. 4 it is possible to see that the refined particles are not uniformly distributed throughout the PZ and there is a more significant effect of the processing conditions on the homogeneity of the particles spatial distribution than their size.

Following the recommended method of Murphy et al. [15] the homogeneity of the particle’s spatial distribution was quantified using the variance ratio of the normalized cell area distribution across the process zone; this data is summarized in Fig. 5. From this analysis it is clear that the degree of clustering, or inhomogeneity of the spatial distribution generally increases across the PZ from the advancing to the retreating side, where particle rich and particle free bands start to develop. A similar effect can be observed in Fig. 5(c), vertically through the centre of the PZ, where greater homogeneity is achieved closer to the base of the PZ than nearer to the surface of the plate.

This behaviour is less pronounced when a higher rotation speed is used and the particle’s spacing becomes more homogeneous across the processed zone, resulting in reduced variance values and a reduction in the noise in the data in Fig 5a, indicating a more uniform particle distribution. Ma et al. have also reported more uniform particle distributions at higher rotation rates, or a smaller pitch [11]. On the other hand, changing the travel speed appears to result in a less clear trend in the homogeneity of the spatial distribution of the particles (Fig. 5c).

Fig. 4: Micrographs from the sample processed at 200 mm min⁻¹ and 500 RPM on (a) the advancing side, and (b) the retreating side, with the tessellated cells overlaid. (c) Examples of the effect of tool rotation speed on cells constructed from images taken at 1 mm from the centre line on the retreating side of the PZ, where the particles were less homogeneously distributed, all with a travel speed of 200 mm min⁻¹.
Fig. 5: The normalised cell size variance ratio measured across the centre of the PZ after FSP with a range of ration speeds, at 200 mm min$^{-1}$, and (b) a range of travel speeds, with the rotation speed kept constant at 500 RPM; in (c) the variance is shown down the centre of the PZ with a travel speed of 200 RPM for increasing rotation speeds.

Fig. 6: Micrographs taken from the 'stop-action' samples processed at (a) 300 RPM and (b) 500 RPM, and both with a travel speed of 200 mm min$^{-1}$. The extent of the deformation zone around the pin was measured from the surface of the pin.
‘Stop-Action’ Experiment

The ‘stop-action’ samples were sectioned in plan view at a depth of 4 mm below the plate surface, where the material flow is dominated by the pin. Unfortunately, defects were found around the tool pin stop position. This is believed to be associated with a lag between the tool’s rotational and transverse motions as the machine were immediately stopped, causing the material around the pin to tear. Nevertheless, these specimens reveal several interesting features. Fig. 7 shows an example of the typical behavior of the particles as they break up following a flow line around the pin, whereas Fig. 9 summarises how the average particle size chances along four flow lines, starting at different positions round the tool (shown in Fig. 8). In Fig. 8 it can be seen that the direction of flow always follows the direction of tool rotation, even on the advancing side. The deformation zone is also very narrow and closely confined to the tool surface. At a rotation rate of 300 RPM, the deformation zone on the advancing side of the pin was 0.45 mm wide, and increased to 0.85 mm on the retreating side. However, when the tool rotation rate was increased to 500 RPM, the deformation layer on the advancing and retreating side were reduced to only 0.15 mm and 0.65mm respectively (Fig. 6). Particle break up occurs within the shear layer, most effectively in material originating from ahead of the tool on the advancing side, which experiences the highest strain rate and strain.

In Fig. 7 silicon and the intermetallic eutectic particles can be observed to already begin to fracture just ahead of the shear layer in advance of the tool, but the fragments have not been greatly dispersed and are still largely associated with the original particle/colony from which they fractured. This is primarily because, although the load on the particles is high, the plastic strain is still very small. At this stage particle fracture can be seen to be associated with cavities forming in the matrix within cracked silicon flakes and intermetallic eutectic colonies. The particles are then rapidly further broken up as they enter the shear zone, which introduces additional voids into the matrix. These refined particles are then re-distributed as the material continues to flow around the tool pin, and the voids are healed as the fragments become more dispersed within the Al matrix. In fact particle refinement occurs very quickly as the particles enter the shear zone close to the tool surface, where an intense shear strain develops, and the minimum particle size bottoms out at quite an early stage (Fig. 7, 9a). This occurs within a short distant of ~1.5 mm into the shear path which is less than a quarter of a revolution of the tool. In comparison, the important processes of void healing and particle redistribution appear to require higher strains and continue to improve all the way around the pin.

If the behaviour of the four flow paths in Fig 8 is compared (Fig. 9) it is apparent that, because particle fracture bottoms out relatively quickly, the particles all reach a similar minimum average size irrespective of the flow path unless the path is on the boarder of the shear zone round the tool (e.g. the outermost data set in Fig. 9). This is in agreement with the average particle size distributions across the PZ shown in Fig. 2, which are relatively flat until you reach close to the edge of the pin width on the advancing side of the tool. There is thus a significant difference in the level of refinement only near the boundaries of the PZ, even though the material experiences a progressively lower strain rate and total strain across the nugget zone from the advancing to the retreating side [16]. This asymmetric flow behaviour has been modeled by several groups and the strain rate of material entering the shear layer on the advancing side is generally predicted to be of the order of 10-100 times higher than on the retreating side (e.g. [17,18]).

Particle break up largely occurs by load transfer from the soft plastically deforming matrix to the hard non-deformable second phases, with few particles actually interacting directly with the tool surface. The load on the particles causing their fracture will therefore be a strong function of the matrix flow stress, which is controlled by the Zener Holloman parameter and will reduce at higher temperatures and lower strain rates [19]. However, the stop action samples and the relatively small change in particle sizes with position and processing parameters, show that the strain level reached in FSP is far higher than needed for the refined particles to approach a
lower size limit, pretty much everywhere in the PZ, irrespective of the parameters. This minimum particle size is thus probably dictated by the smallest dimension and, or defect density, (e.g. caused by branching, twinning, or other stress concentrations) in the original particles in the cast starring material.

Fig. 7: Micrographs showing the particle refinement and distribution along a typical flow path around the pin from a ‘stop-action’ sample processed at 200 mm min$^{-1}$ and 300 RPM.
Fig. 8: Micrograph of the ‘stop-action’ sample processed at 200 mm min$^{-1}$ and 500 RPM. The flow lines in which particle break up had been extensively studied are shown, and these are from the advancing side (-1.5 mm), the centre line (0 mm), the retreating side (1.5 mm), and the edge (2.5 mm).

Fig. 9: Average particle sizes (ECD) measured along the different flow paths around the tool pin within the stop action samples processed at (a) 300 RPM and (b) 500 RPM.
Conclusions
In the present study the homogeneity of particle refinement and second phase spatial
distribution, within the process zone developed during FSP, was investigated in a gravity die
cast A380 Al-Si alloy. The high level of refinement that can be achieved by FSP resulted in an
average particle size of 2 to 3 µm within the PZ. There was a tendency for the average particle
size and inhomogeneity to reduce from top to bottom, and increase across the centre of the PZ,
from the advancing to retreating side, but there was a very week influence of the processing
conditions and position in the PZ on the refined particle size. The homogeneity of the particle’s
spatial distribution was assessed from the variance of normalised tessellated cell size
distributions. The spatial distribution of the particles became significantly more random with a
reduction in tool pitch and was also greatest on the advancing side of the PZ. Particle refinement
was shown to occur very rapidly when particles enter the shear zone ahead of the tool and
bottom out at a much lower strain than is generated in the FSP process; i.e. it is extremely
difficult to reduce the particle size below a lower limit, which is probably related to the defect
density and minimum dimensions of the original phases within the cast material. This
combination of factors is primarily responsible for the insensitivity of the refinement level to the
FSP parameters and position within the PZ. The fracture and break up of the particles was
associated with voids forming in the matrix, which were progressively healed as the particles
became dispersed, with increasing strain along a given flow line round the tool.

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