# Using a Focused Ion Beam (FIB) Instrument to Investigate the Three-Dimensional (3D) Morphology of β-AIFeSi Intermetallic Particles

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### Abstract

For this research, the focused ion beam (FIB) instrument was used to examine  $\beta$ -AIFeSi intermetallic particles in as-cast samples of 6063 aluminum alloys. This paper will present a description of the FIB and the techniques used to examine detailed three-dimensional (3D) microstructures. Sequential electron micrographs are acquired after thin layers of material have been precisely removed via ion beam milling. The image from each section is digitized and exported to generate a 3D reconstruction of the microstructure. In addition, by extracting the particle-matrix interface of the  $\beta$ -AIFeSi phase, it is possible to perform quantitative analyses, such as roundness measurements.

### 1. Introduction

When casting 6xxx aluminum alloys, AlFeSi intermetallics form at the grain boundaries and interdendritic regions. These particles have a large influence on the workability of the material during subsequent deformation processing. During extrusion,  $\beta$ -Al<sub>9</sub>Fe<sub>2</sub>Si<sub>2</sub> has been linked to the presence of the pick-up defect [1-3], where surface material tears away and is later redeposited. Although, the  $\beta$ -phase is easily visible using standard imaging techniques, such as light optical microscopy (LOM) or scanning electron microscopy (SEM), several particle characteristics may be improperly described by these twodimensional (2D) views. Planar slices of AA 6xxx microstructures display discrete, needlelike  $\beta$ -particles; however, three-dimensional (3D) examinations reveal a plate morphology. The  $\beta$ -phase forms a partially interconnected network and exhibits branching – a preferential nucleation of smaller plates on larger "parent" particles [4].

Historically, investigations of AIFeSi intermetallics have been predominantly restricted to 2D. Tirel et. al. performed some 3D experiments using serial sectioning (via mechanical polishing) and subsequent LOM [5]. While practical, this technique has several shortcomings. Most notably, it is time consuming and laborious, repeatedly polishing small amounts of surface material as accurately as possible. (Particle contrast may be enhanced by a light etch, but should not cause significant surface relief.) Areas of interest are located in the LOM and captured; microstructural images are then aligned using image editing software, such as Adobe Photoshop. Microhardness (M-H) indents are used as reference position markers and also to determine the depth of material that was removed via polishing. The sections are then reconstructed to form 3D images. The limited spatial resolution of the LOM may be overcome by using SEM with its higher magnification

capabilities. However, this process will dramatically increase the required experiment time as the SEM sample chamber must be evacuated between each polishing step.

The aforementioned limitations may be overcome by using the dual-beam focused ion beam (FIB) instrument; (an FEI DB-235 was used in this research). A diagram of the system is displayed in Figure 1. As its name suggests, the dual-beam FIB is comprised of two columns: an SEM (Schottky field emission gun source) operates in conjunction with a focused ion beam (Gallium ion source), which is displaced 52° away from the electron column's vertical axis. 3D imaging is obtained by acquiring 2D electron (e<sup>-</sup>) images while the ion (i<sup>+</sup>) beam concurrently "mills" (removes) precise layers of material from the examined face. The spacings between sections are set using the instrument controls. Images are also collected from the signal generated by the i<sup>+</sup> beam. However, this beam should only be scanned for the minimum time necessary since it inflicts damage to the specimen surface. Figure 2 displays an e<sup>-</sup> image of an area that was damaged due to previous extended imaging at higher magnifications using the i<sup>+</sup> beam. (The e<sup>-</sup> image is foreshortened because of the 52° tilt angle.).



Figure 1: Schematic illustration of dual-beam FIB.



Figure 2: e<sup>-</sup> image displays damage from prior lengthy imaging using the i<sup>+</sup> beam.

## 2. Experimental Procedure

A section of a direct-chill (DC) cast billet of AA 6063 was obtained directly from an aluminum extruder with casting facilities. This alloy was chosen due to its large industrial significance as one of the most popular aluminum extrusion alloys [6]. In addition, 6063 has a relatively simple chemistry with a wide range of compositions limits. To ensure the presence of the  $\beta$ -AIFeSi phase following casting, the billet selected for examination was required to have a low Mn content and no Sr additions; these elements modify the AIFeSi intermetallics during casting [3,7]. Cr, Ti, and Zn were also kept to a minimum. The composition of the alloy used is presented in Table 1.

Samples were prepared via traditional metallographic procedures with a final polish of 0.05  $\mu$ m colloidal silica (SiO<sub>2</sub>). An etchant of 0.5% HF for 30 seconds improves the contrast between the AIFeSi phase and the aluminum matrix during LOM imaging. In addition, this concentration/time combination preferentially removes AI, yielding surface relief of any second phase particles. This topographical variation, as shown in the upper-left region of Figure 2, enables easier identification of the AIFeSi particles using the FIB.

Table 1: Comp	osition of AA	6063 billet used in this research.	(in wt.%, remainder A	J)

Mg	Si	Fe	Cu	Cr	Mn	Ti	Zn
0.552	0.442	0.118	0.003	0.001	0.005	0.006	0.011

The AIFeSi particles possess a higher average atomic number than the surrounding aluminum matrix and are therefore discernable in the SEM due to z-contrast. Although secondary electron images are used, this signal is still influenced by the diverse backscattered electron yield values. To maximize the contrast, surface detail, and resolution, a 10kV electron beam with an intermediate spot size was used. The ion beam is set to 30kV with a range of beam currents depending on the particular milling process.

To begin 3D observations, the e<sup>-</sup> beam is scanned across the sample to locate an area of interest [that contains AIFeSi particles]. (It is better when the feature is oriented vertically on the screen, though scan rotation may be enabled.) The eucentric height is set while tilting the sample to  $52^{\circ}$  – perpendicular to the i<sup>+</sup> beam, as shown in Figure 1. Focused images are acquired with both sources.

A wedge-shaped trench is milled from the sample near the area of interest using the "regular cross section" function. A high i<sup>+</sup> beam current (7 nA) minimizes the required time; for this stage, precision is not necessary. Due to beam damage, it is useful to move away from the feature when initially focusing the beam. The FIB mills the trench in a staircase pattern, illustrated by the side-view schematic in Figure 3a. When finished, the trench appears smooth as depicted in Figure 3b. The dimensions vary – they are made slightly larger than the feature to be examined because material that is removed during milling redeposits on the sides and bottom of the trench. Due to geometry, the length (y-dimension) must be at least 1.28 (tan 52°) times greater than the depth (z); (typically y = 2\*z). Figure 4a displays an e<sup>-</sup> image of the trench milling process in progress.

The next step is to "clean" possibly damaged material from the previous trench milling. The i<sup>+</sup> current (and hence the size of the beam) is reduced. The "cleaning cross section" function is utilized to mill thin layers of material, starting at the exposed face [within the trench] and moving towards the feature(s) of interest. The rectangular volume that is removed has the same width (x) and depth (z) as the previous wedge. When completed, images are acquired with both sources; the exposed face is the first section for 3D examinations. This process is repeated to obtain subsequent images; the y-dimension defines the space between sections. Serial sectioning performed by the FIB is illustrated by Figure 3c and shown by the i<sup>+</sup> beam image of Figure 4b. The size of the ion-milled volume may be continuously adjusted (in the x and/or z) to conserve time. Subsurface features have unpredictable shapes and redeposited material often blocks part of the field-of-view. Figure 4c, the final e<sup>-</sup> image for this investigation, displays several adjustments to the trench width.

This method of "shaving" off thin layers has many advantages over milling perpendicular to the surface, as done with the initial wedge-shaped trench. First, it is more efficient (faster) because the sputtered material can escape through the open trench, rather than having to eject exclusively via the surface. Secondly, this process imparts less deformation into the sample. Thirdly, when milling perpendicularly, only the i<sup>+</sup> beam can be used to image the entire area, since the e<sup>-</sup> beam would be partially blocked. The previously described technique provides a method to use both beams concurrently.



Figure 3: Illustration of how the i<sup>+</sup> beam mills material and the e<sup>-</sup> beam is used for imaging.



Figure 4: a) e<sup>-</sup> image of initial wedge-shaped trench partially milled (~30% completed).

- b) i<sup>+</sup> image with box positioned for next volume of material to be removed.
- c) e<sup>-</sup> image of the completed sectioning displays redeposited material on sides and bottom of trench, and continuous adjustments to the volume width.

When all of the micrographs had been collected, they were cropped to align the surface of the material with the top of the images. Since the sample was not moved and no drift occurred, subsequent sections were consistent in the x-axis. The particles' shapes are defined by the particle-matrix interface, which were extrapolated from the atomic number contrast at the boundary between the two regions. After repeating this process for each image, MatLab was used to reconstruct the intermetallics' morphology in 3D.

MatLab was also programmed to measure the perimeter (P) and area (A) for the particles' projections in 2D in order to calculate roundness (R), defined in equation (1). This descriptor was used instead of surface area-to-volume ratios (S/V), which depend upon particle size.

$$R = \frac{4\pi A}{P^2} \times 100 \tag{1}$$

#### 3. Results

Figure 5 presents multiple electron images of  $\beta$ -AIFeSi intermetallic particles from an ascast 6063 sample. When viewed in 2D at the surface, the  $\beta$ -phase appears acicular. The plate morphology cannot be viewed in cross-section due to its extremely small thickness dimension. However, thin plates are observable in the FIB micrographs, which display two perpendicular planes. Both Figures 5a and 5d – above and below the surface, respectively – display branching of the  $\beta$ -AIFeSi intermetallics. In total, forty sequential images were acquired in 0.5  $\mu$ m increments to examine the 3D morphology of the  $\beta$ -AlFeSi particles in the selected volume. Figure 6a presents the reconstructed  $\beta$ -plates in 3D; Figure 6b shows the projection of the particles when looking down the y-axis, the sectioning direction.



Figure 5: Images from sections of β-AIFeSi intermetallic particles. (2.5 µm spacing between images)

Average roundness for all 2D particle projections was calculated to be around 5.0, with high variance. The extremely small values (for comparison, R = 100 for circles) are consistent with the  $\beta$ -phase's thin plate morphology possessing very high aspect ratios.

### 4. Discussion

The FIB technique presented in this paper provides a useful method for investigating microstructural features in 3D. It has many advantages over serial mechanical polishing and LOM imaging. First, the SEM images provide higher spatial resolution and accurately measures the distance between sections. Secondly, the technique is simple to execute, and because the sample is never moved, subsequent images are automatically aligned. Thirdly, it conserves time; the investigation of a volume containing AIFeSi particles (such as the one shown in Figure 6) can be completed in less than 3 hours.

However, there are several limitations to this technique. First, the volume element examined is quite small, thus the FIB is most useful for concentrating on micron-sized features; larger volumes should be reconstructed using LOM techniques. Secondly, grayscale levels vary considerably across the e<sup>-</sup> image of the exposed face because the signal is generated within a trench. Therefore, it is non-trivial to automatically extrapolate

the particle boundaries using computer software; this task is currently being investigated. Presently, for this research, the boundary interface was traced using Adobe Photoshop.



Figure 6: a) 3D reconstruction of  $\beta$ -AIFeSi particles from volume previously displayed in Figure 5. b) 2D projection along y-axis. (34 pixels = 1  $\mu$ m)

The FIB provides a useful tool for investigating the shapes of particles in 3D. The work presented here was the beginning of research examining the morphological changes that occur to AIFeSi particles during the homogenization process. In this paper, the  $\beta$ -particles were used as an example to demonstrate the technique and how false assumptions are often made when looking at planar, 2D, microstructures. A fully homogenized AA 6063 sample containing  $\alpha$ -AIFeSi phase has also been previously investigated in 3D [8]. Currently, the authors are examining partially homogenized samples. In addition, the computer programs that were written to plot the 3D shapes and perform quantitative analysis are being revised. In the future, sequential energy dispersive spectroscopy (EDS) mapping will provide 3D elemental distributions surrounding the particles.

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