On the development of a pseudo micro-truss intermetallic microstructure in a high pressure die cast AZ91 alloy

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Abstract. The three dimensional features of the intermetallic microstructure that develop across the thickness of a 1 mm thick casting has been studied using SEM and dual beam FIB. The intermetallics form a closely interconnected spatial network which resembles a scaffold or micro-truss structure near the casting surface. The degree of interconnection decreases, and the overall scale of the microstructure is coarser at the core of the casting. The contribution of this pseudo micro-truss structure to the overall strength of the casting is discussed.

1. Introduction
The yield strength of high pressure die cast (hpdc) AZ91 alloy increases with a decrease in section thickness [1-6]. The average grain size of the cast material decreases with decreasing section thickness, and the increased strength of the thinner sections is normally ascribed to Hall-Petch strengthening effects [1, 7, 8-11]. However, the decrease in casting thickness may affect the other contributions to the strength, particularly dispersion strengthening [5, 9], so the underlying reasons for the increased strength of the thinner sections are not completely clear. It has been suggested [5] that the closely interconnected structure of the intergranular intermetallics formed during the solidification behaves similarly to a ceramic cellular solid [5], akin to a pseudo micro-truss structure. Highly segmented ceramic structures exhibit increased strength due to a Weibull modulus effect [10], and it seems likely that this additional strengthening effect adds to the thickness effects on the strength [5]. In order to assess this possibility, it is necessary to determine the extent of the interconnection in the intermetallic structure as well as its relative scale, i.e., the size of the intermetallic cells. With these goals in mind, 2D and 3D microstructural studies of the intermetallic structure have been carried out at two locations of the cross section of a cast to shape tensile specimen of hpdc AZ91 alloy.

2. Experimental
A tensile specimen of AZ91 (1 mm thick) alloy from earlier studies [4, 5] was used for this study. Several sections were obtained from the gauge length and metallographically polished by standard methods, down to 0.5 μm colloidal silica. One of the polished sections was etched with glycol. Figure 1 shows the sites of interest in the cross-section. These sites were selected as they represent the regions of minimum (core) and maximum (corner) solidification rates. Earlier work indicates that the core and
corner regions exhibit the lowest and highest hardness values on the cross section [4, 5] as well as the coarsest and finest scale of Mg$_{17}$Al$_{12}$ intermetallics [11], respectively. The 2D analysis was carried out on a JEOL 6460 LA Scanning Electron Microscope, on the etched cross-section (plane x-z in Fig 1), whereas the serial sectioning for the 3D analysis was performed on a FEI xT Nova NanoLab 200 DualBeam FIB system, on the un-etched cross-section. The procedure involves digging a small square trench, and subsequently removing slices of given thickness from one of the trench’s walls. See Fig. 1 for details on the geometry of the sectioning. Further details on the sectioning and imaging procedure have been published elsewhere [12]. Specific details to the sequential sectioning are given in Table I. The Amira® 3D reconstruction software [13] was used to create 3D images from the sequence of secondary electron images.

**Figure 1.** Identification of the sites for the microstructural study. The overall dimensions of the cross section were 10 mm by 1 mm. The sequential sectioning for the FIB study was carried out on y-z sections as indicated by the parallel lines and arrows. The x, z directions match those of Figures 2 and 3.

**Table I.** Details of the FIB sequential sectioning for the 3D images.

| Location | Resolution | Slice thickness (µm) | HFW (µm) |
|----------|------------|----------------------|----------|
| Core     | 1024x884   | 0.18                 | 36.6     |
| Corner   | 1024x884   | 0.2                  | 51.2     |

3. Results
SEM secondary electron images (x-z plane in Fig 1) from the core and corner regions are shown in Fig. 2. The bright regions are the Mg$_{17}$Al$_{12}$ intermetallic. The scale of the intermetallic appears relatively coarse at the core, Fig. 2a, whereas it is very fine at the corner, Fig. 2b. Some amount of interconnection is apparent in both microstructures, but especially at the corner. Figure 3 shows the reconstructed 3D images of the intermetallic. Each reconstructed image in Fig. 3 has been created from a series of 100 SEM micrographs. Comparison with sectioning perpendicular to those depicted in Fig 2 shows that the staggered appearance is an artefact of the sequential slicing. The solidification direction lies along the z-direction at the core, and away from the x-z vertex at the corner. It is seen that the intermetallics exhibit a high degree of interconnection at the corner, indeed resembling a micro-truss structure. On the contrary, at the core the degree of interconnection is significantly lower and the scale of the microstructure is much larger, as anticipated from Fig. 2. The
latter is to be expected since the core contains many externally solidified large grains which keep apart the intermetallic-rich regions that form as the small grains solidify inside the die cavity.

**Figure 2.** SEM secondary electron images showing the distribution of intermetallic at the core (a) and corner (b), respectively, of the specimen studied.

**Figure 3.** Different views of the intermetallic structure at the core and corner regions of the specimen of Fig. 2.
4. Discussion
Detailed quantitative analyses by Cáceres et al. [4, 5, 14] have shown that the strength of the as-cast structure of hpdc alloy AZ91 stems from three dominant mechanisms, namely: Hall-Petch hardening, solid solution hardening and dispersion hardening. The small grain size provides the largest strengthening, followed by solid solution hardening. The volume fraction of intermetallic is small (less than 10%) and dispersion hardening results in a minor reinforcement when the reinforcing particles are isolated and enable plastic deformation to bypass them after a small plastic strain. When a significant amount of interconnection is present, as in the present case, the intermetallic frame can be expected to behave like a rigid scaffold: plastic deformation can only occur by cracking the whole intermetallic structure. Extensive interconnection should thus result in a more rigid and harder material than when the intermetallic particles are isolated from each other, and it seems safe to ascribe the increased strength of thinner sections to the pseudo micro-truss structural reinforcement which in thin castings covers a larger share of the cross section [5]. It is noted that similar reinforcing effects stemming from the interconnected intermetallic network have recently been suggested with regards to the creep resistance of a thixomolded MRI 230D alloy [15].

5. Conclusions
The intermetallic microstructure at the core and corner regions of the cross section of hpdc AZ91 alloy specimens, of thickness 1 mm, has been characterised in 2D and 3D by using SEM and Dual Beam FIB. 2D images show that the scale of the intermetallics is relatively coarse at the core but appears very fine at the corner. 3D reconstructed images show that the intermetallic structure is finer and more profusely interconnected at the corner regions, resembling a micro-truss structure. The difference in scale and degree of interconnection is largely due to the presence of large dendritic grains at the core region. The highly interconnected intermetallic microstructure is likely to account for a measurable fraction of the alloys’ strength, especially in thinner sections in which it is more prevalent.

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