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FIB and SEM-STEM Studies of Friction-Stir Processed AM60 Magnesium Alloy

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In the present work, we present the microstructural study of a cast magnesium alloy AM60B processed by Friction Stir Processing (FSP) in order to achieve Ultra-Fine Grain (UFG) size (200-700nm) in the stirred zone. Focused ion beam (FIB) has been used for sample preparation, and transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM) and selected area electron diffraction (SAED) for the structural characterization. To explain the differences induced by the FSP, two lamellas obtained by FIB from the bottom and the upper part of the cross-section of the nugget are compared to one extracted from the parent material (unaffected zone A). The obtained results show the remarkable homogenization and grain refinement produced in the structure in the processed zone, as previous reported works indicate [1,2]. The microstructure of the as cast base material reveals the presence of dendrites of solid solution of Al in Mg (α-phase), with an average grain size of ~400-500 μm, and a partially divorced eutectic structure at the interdendritic spaces, where the massive and lamellar β-$Mg_{17}Al_{12}$ intermetallic phase can be observed (Figure 1). The SAED on one particle of β-phase shows a diffraction pattern that fits the crystalline structure of the cubic I -4 3 m space group (Nº 217) [3]. Also Mn containing phases were homogeneously distributed throughout the section. The performed analyses demonstrate that these particles are basically Al-Mn binary phases containing trace elements as Si or Fe, and the measured Mn/Al ratio allow to classify them into two types previously reported [2,4]: Type I are particles of equiaxed or almost rounded shape and type II are needle like or flowerlike particles, with a lower Mn/Al ratio.

The comparison of the two lamellas extracted from the nugget zone (Figure 2) shows the grain size variation of the α-phase matrix from the bottom (average size ~ 120 nm to 550 nm) to the top of the stirred zone (average size ~ 400 nm to 1100 nm), accordingly with the temperature variation during the process. This effect has been previously reported for FSP aluminium alloys [5]. From the SEM-STEM DF images it is also clear that this temperature profile strongly affects the dissolution and reprecipitation of second phases. As it can be seen in the image b, representative of the upper part of the nugget, the Al_xMn_y and $Mg_{17}Al_{12}$ intermetallic particles are clearly less concentrated than in the lower part of the processed section. The determined composition of the Mn-rich particles precipitated in the processed zone is consistent with that of Al_4Mn compound.

Conclusion: The microstructural refinement of the AM60B alloy has been accomplished by FSP. A difference in grain size and precipitation density between different zones of the nugget zone has been determined, consistent with the thermal cycle in the processed material.

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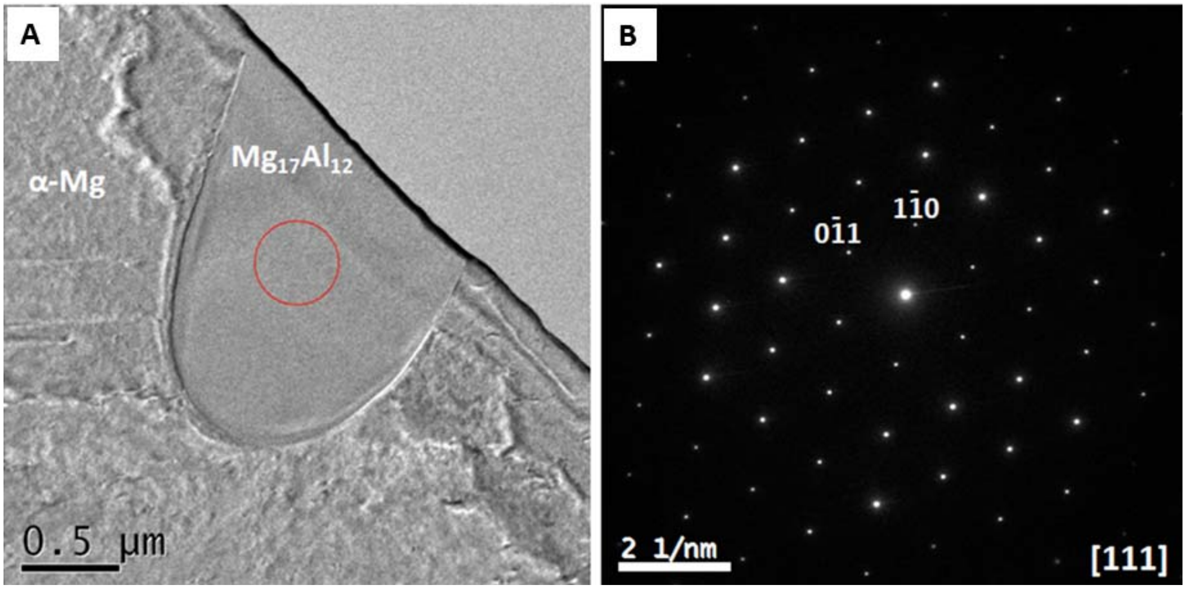

Figure 1. Unaffected AM60: (a) TEM BF image showing lamellar β-Mg17Al12 intermetallic phase in α-Mg matrix. (b) SAED pattern from the area marked in (a) showing the [111] zone axis.

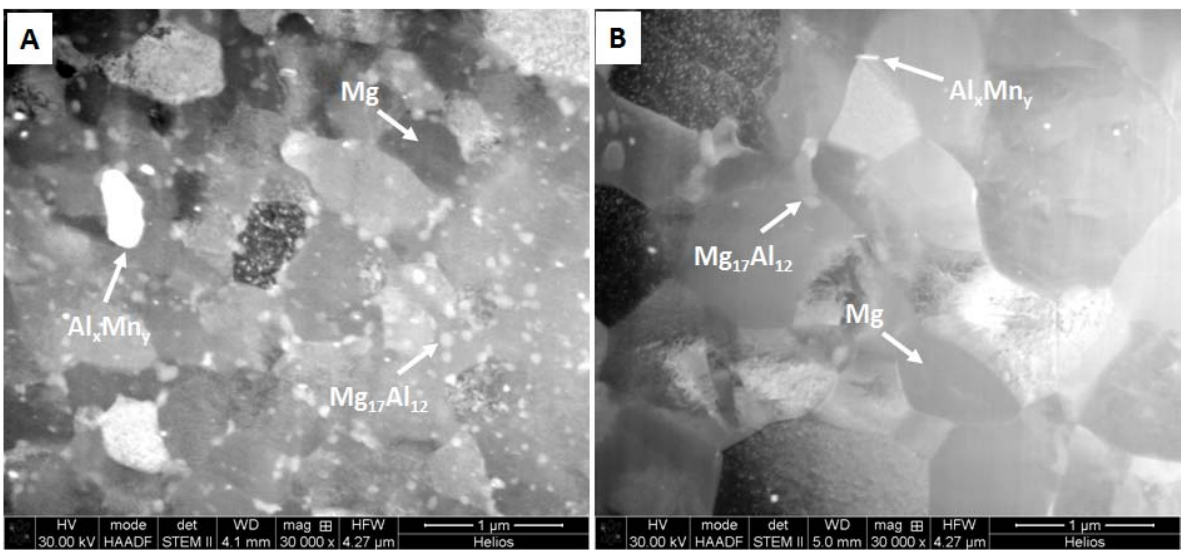

Figure 2. SEM-STEM DF images of two lamellas from the nugget zone of the processed sample. (a) Extracted from the bottom. (b) Extracted from the top.

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