Magnesium Technology 2011
Edited by: Wim H. Sillekens, Sean R. Agnew, Neale R. Neelameggham, and Suveen N. Mathaudhu
TMS (The Minerals, Metals & Materials Society), 2011

MICROSTRUCTURE EVOLUTION IN AZ61L DURING TTMP AND SUBSEQUENT ANNEALING TREATMENTS

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Keywords: AZ61, Microstructures, Texture, Mechanical Properties, Thixomolding, Thermomechanical Processing

Abstract

Microstructure evolution is studied in Thixomolded® Thermomechanical Processed (TTMP) AZ61L sheet at various stages of processing. Transmission electron microscopy (TEM) is utilized to examine (1) grain refinement and recrystallization and (2) refinement and re-distribution of the β-$Mg_{17}Al_{12}$ phase in the as-Thixomolded, as-TTMP, and annealed conditions. Electron backscatter diffraction (EBSD) is used to study texture evolution through TTMP and annealing. The influence of microstructure produced by TTMP and annealing on the mechanical properties will be discussed.

Introduction

The high cost and poor formability of magnesium sheet has limited its commercial application [1]. If, through materials design and processing, formability could be enhanced, several markets, especially automotive, would benefit. The emerging consensus is that deformation modes required for high ductility materials are more easily activated in fine, micro- or nanoscale grains [2]. Several different processes to achieve fine scale microstructure have been developed, producing high strength, high ductility materials [3–10]. Thixomolding Thermal Mechanical Processing (TTMP) builds upon the fine grains, isotropy, and low porosity of Thixomolded Mg alloys containing eutectic phases. Intense thermomechanical processing is applied to further refine grain size and eutectic phases. Furthermore, additional thermal treatments can be applied to optimize strength, ductility and formability.

Experimental

Thixomolded and TTMP samples of AZ61L were obtained from Thixomat, Inc., composition shown in Table I. A rolling pass yielded a sheet thickness of 1.5 mm for the TTMP samples, a 50% reduction in the thickness of the as-Thixomolded plates. Annealing temperatures of 250 °C and 300 °C were chosen to explore effects of annealing on microstructure and mechanical properties.

Table I: Composition of received AZ61L in wt %

Al	Zn	Mn	Si	Fe	Mg
6.5	0.46	0.14	0.01	0.003	bal.

Dogbone tensile specimens with a gauge length of 31.75 mm and cross section of 7.94 mm, were machined from the sheets with the tensile axes parallel to the rolling direction. Room temperature tensile tests were performed with a displacement rate of 0.71 mm/min. An extensonometer was used to measure tensile elongation.

Samples for microscopy were removed from the grip ends of the tensile bars in the plane of the sheet. TEM specimens were prepared by electropolishing with a 8% perchloric acid in methanol electrolyte or dimpled and ion milled using a Gatan Precision Ion Polishing System (PIPS). TEM was conducted with a JEOL2000FX and a Phillips CM12 AEM systems. EBSD specimens were prepared by polishing with 1 μm diamond followed by ion polishing in a Gatan PIPS. EBSD examination was conducted on a JEOL 840A SEM system equipped with a HKL EBSD system. A step size of 0.5 μm was used.

Results

The as-Thixomolded material reveals equiaxed grains, regions of high dislocation density, and both agglomerated (indicated by the arrow) and isolated β particles (Fig. 1). In addition to agglomerated and isolated, equiaxed β grains, regions of eutectic β are also found in the as-Thixomolded condition. Fig. 2 highlights a region of eutectic β, likely composed of a series of small β grains along a boundary.

The TTMP-induced microstructure demonstrates significant grain refinement from the Thixomolded condition (Fig. 3). Highly elongated,deformed grain structures are seen (region A) in the TTMP condition. A collection of nanoscale grains, that may indicate local dynamic recrystallization occurred, are shown in region B. The elongated β structures and clumps of β particles

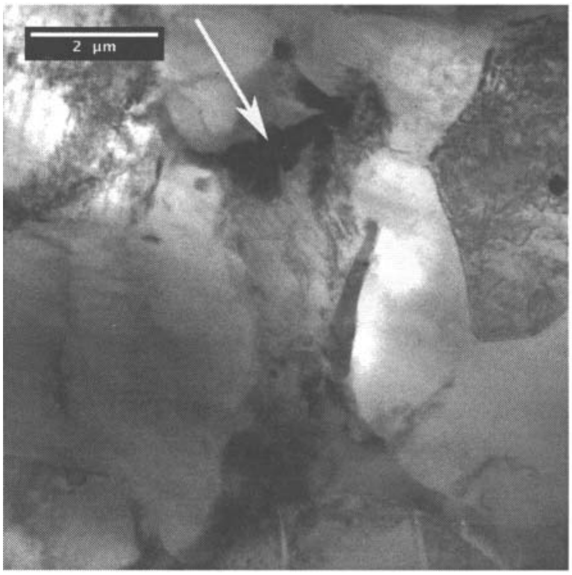

Figure 1: Microstructure of Thixomolded AZ61L. The arrow highlights a region of agglomerated β particles.

observed in the as-Thixomolded material are dissipated. β grains are heterogeneously distributed throughout the as-TMP microstructure.

Annealing treatments allow for further recrystallization. At 250 °C the material initially retains a fine grain microstructure. Grain coarsening becomes evident after 20 minutes at 250 °C (top row Fig. 4). Annealing at 300 °C initiates more rapid grain growth, evident even at the shortest annealing time (bottom row Fig. 4). Heterogeneous distribution of β grains is evident in Figs. 4(c-e). Nano-scale β particles in the TMP + annealed material were equiaxed and often exhibited a rounded morphology as can be seen in Fig. 5 and Fig. 6.

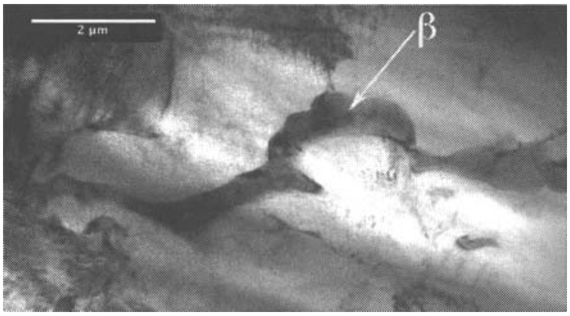

Figure 2: TEM micrograph showing the morphology of the eutectic β particles in the as-Thixomolded condition. Identification of phase was determined by EDAX measurements.

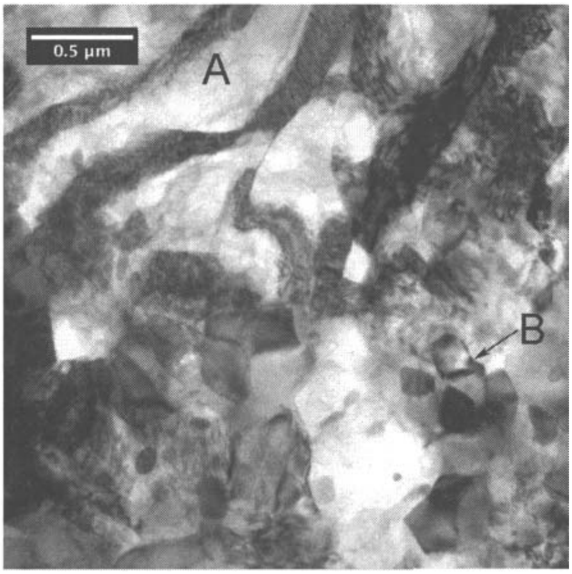

Figure 3: Elongated grains (A) and nanoscale α-Mg and β grains (B) are found in the as-TTMP condition.

β particles were frequently observed in the interior of grains and along α-Mg grain boundaries. Nano-scale recrystalized α-Mg grains tend to coincide with the β phase. Clusters of β particles and recrystallized α-Mg grains suggests that the β particles play a significant role in retarding grain growth.

A subset of the samples are analyzed by EBSD. As-TTMP and TTMP samples annealed for short times exhibit many (~75%) non-indexable EBSD diffraction patterns due to high deformation. The TMP samples annealed for longer durations yield fewer (~10%) non-indexable patterns. Band contrast images, with re-constructed grain boundaries (>5% misorientation), are shown in Fig. 7. Finer grains with a bimodal size distribution is evident for the TTMP + 300 °C 20 min anneal sample, demonstrating the effect of recrystallization. The maximum grain diameters for the as-Thixomolded and TTMP + 300 °C 20 min anneal are 36 μm and 14 μm respectively.

Pole figures were also obtained from the EBSD analysis. Nearly random texture patterns are observed in the as-Thixomolded and TTMP + 300 °C 20 min anneal sample (Fig. 8(a and d)). Strong (0001) texture patterns were observed for the as-TTMP sample(Fig. 8(b)) and the TTMP + 250 °C samples annealed for short times (Fig. 8(c)). In addition to the (0001) component, a weaker $\langle\bar{1}100\rangle$ texture was observed to follow the same trend parallel to the rolling direction.

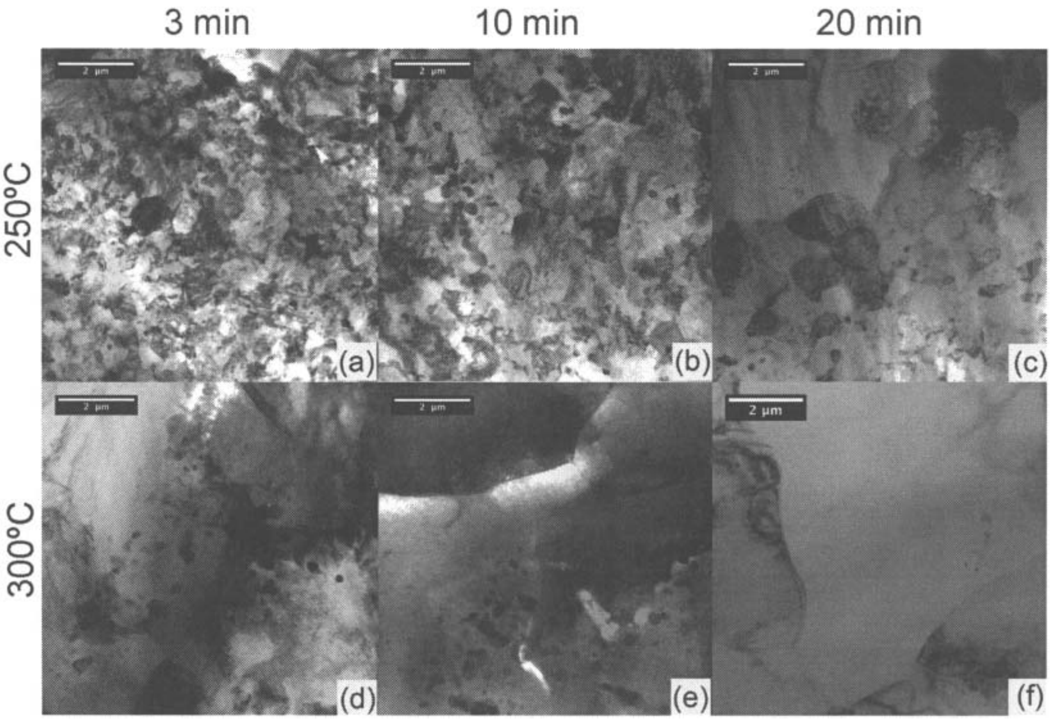

Figure 4: Microstructure evolution resulting from post TTMP annealing treatments (a) TTMP + 250 °C 3 min anneal, (b) TTMP + 250 °C 10 min anneal, (c) TTMP + 250 °C 20 min anneal, (d) TTMP + 300 °C 3 min anneal, (e) TTMP + 300 °C 10 min anneal, (f)TTMP + 300 °C 20 min anneal.

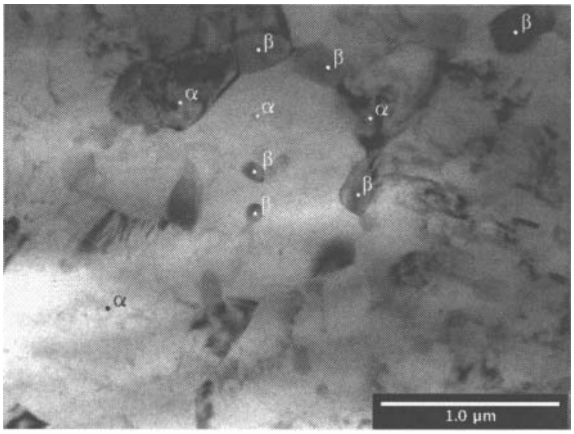

Figure 5: TEM micrograph of a sample annealed at 250 °C for 20 min with small α-Mg grains and β grains. Identification of the β phase was determined by EDAX measurements.

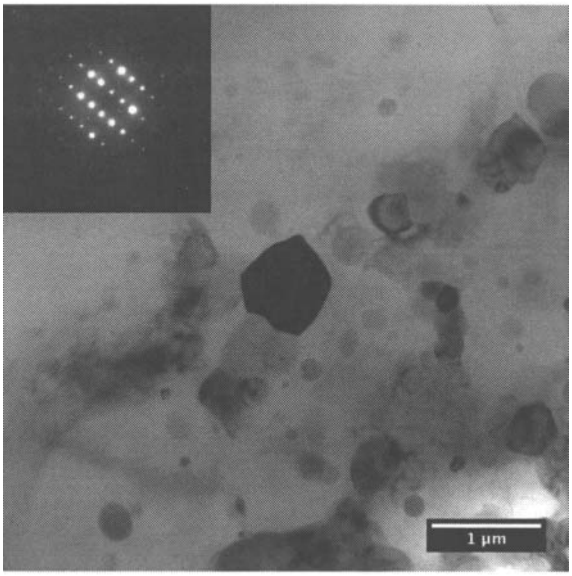

Figure 6: A larger β particle (dark) in a cluster of smaller, presumably β particles in the sample annealed at 300 °C for 20 min. SAED pattern from the $[\bar{1}13]$ zone axis of the β crystal.

Table II: Mechanical properties of the AZ61L samples examined in this study

Sample	YS (MPa)	UTS (MPa)	El (%)
as-Thixomolded	127.3 ± 14.7	191.5 ± 16.1	5.1 ± 0.4
as-TTMP	315.8 ± 6.8	370.7 ± 4.3	6.7 ± 3.9
TTMP + 250 °C 3 min Anneal	331.5 ± 6.4	372.3 ± 5.4	5.5 ± 2.4
TTMP + 250 °C 10 min Anneal	313.0 ± 17.2	365.3 ± 8.0	5.6 ± 3.4
TTMP + 250 °C 20 min Anneal	321.7 ± 3.1	361.3 ± 2.3	7.2 ± 1.4
TTMP + 300 °C 3 min Anneal	236.7 ± 4.1	315.3 ± 1.3	15.5 ± 2.1
TTMP + 300 °C 10 min Anneal	223.6 ± 1.2	308.5 ± 4.8	16.6 ± 3.0
TTMP + 300 °C 20 min Anneal	216.5 ± 5.7	307.6 ± 1.4	22.5 ± 1.5

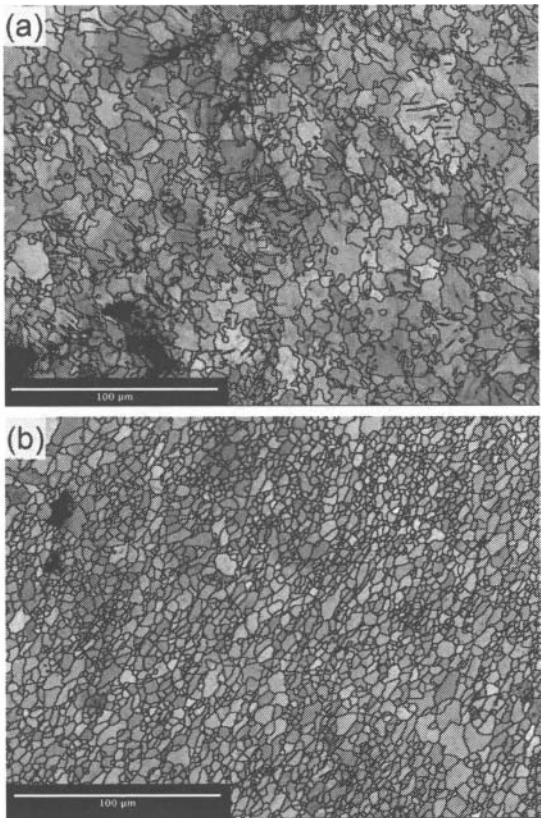

Figure 7: EBSD maps of (a) as-Thixomolded and (b) TTMP + 300 °C 20 min anneal

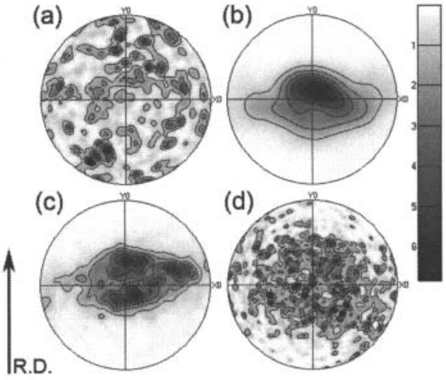

Figure 8: (0001) Pole figures of (a) as-Thixomolded, (b) as-TTMP, (c) TTMP + 250 °C 3 min anneal, and (d) TTMP + 300 °C 20 min anneal. Mapping was performed in the plane of the sheet with the rolling direction pointing up.

Discussion

It has previously been demonstrated that the effect of TMP on Thixomolded AZ61L is a marked increase in the yield strength and ultimate tensile strength of the material, without reduction of tensile ductility[3, 11, 12]. A summary of the mechanical characteristics of the material can be found in Table II. The TMP approximately doubled yield and tensile strength over that of the as-Thixomolded sample. Annealing at 250 °C had little affect on the tensile properties. As grains coarsen during the 300 °C annealing treatments, yield and tensile strength decreases . For the times and temperatures selected, annealing temperature had a more significant affect on tensile properties than annealing duration. An increase in ductility of samples annealed at 300 °C may result from a loss of deleterious texture as recrystallization occurs.

Low R value measurements taken in similarly processed AM60, indicate a decreased anisotropy from the as-TTMP condition, corresponding with the demonstrated loss of texture after the 300 °C 20 min anneal [11]. Texture evolution and R value measurements in TTMP AZ61L with anneal and/or aging treatments will be an area of future exploration. To our knowledge, non-textured magnesium sheet has not been seen in conventional rolled material.

Future investigations will further explore methods to (1) minimize texture after TTMP, (2) control and refine the distribution of β particles, and (3) optimize the heat-treatment to better control grain size, optimizing the mechanical properties.

Conclusions

TMP of Thixomolded AZ61L results in very fine grain sheet with a twofold increase in strength with no loss of ductility. Annealing at 250 °C had little affect on the mechanical properties from the as-TTMP condition and evidence of grain growth is not seen until 20 minutes. Grain coarsening and a drop in strength is seen after annealing the TTMP material at 300 °C. Annealing at 300 °C for 20 min was shown to lead to a nearly non-textured sheet with ductility several times greater than in the As-TTMP material. These results show the promise of optimizing the TTMP and post treatments to develop fine grain, non-textured magnesium sheet with high strength and formability.

Acknowledgement

This material is based upon work supported by the National Science Foundation under Grant No. 0847198.

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