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On Mechanical Properties & Microstructure of TTMP Wrought Mg Alloys

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Abstract

Thixomat has developed a Thixomolded® Thermomechanical Processing (TTMP) route for producing high strength-to-density Mg alloy sheet. The TTMP process leverages the inherent fine grain microstructure of rapidly solidified Thixomolded Mg alloys, e.g. AM60 and AZ61L, and the subsequent high strain rate thermomechanical processing to create fine well-dispersed grain structure. Likewise, eutectic phase in the molded microstructure can play a beneficial role in retaining fine grains and strengthening by a solution/division/re-precipitation mechanism. Further thermal treatments can be applied to optimize strength, ductility and formability. Some up-to-date results on mechanical properties and microstructure of TTMP Mg alloys are presented.

Introduction

Light weight-high strength Mg sheet would be in high demand in the automobile, aerospace, truck / trailer, battery, military, wind energy and electronic/communication markets were it not for two factors, 1) high cost, and 2) poor formability. If cost could be reduced and if formability could be enhanced, the aforementioned markets could enjoy the benefits of lightweight Mg on fuel economy and reduction of pollutants.

Inherent Deformation Problems of State-of-The-Art Commercial Practice – Despite significant efforts and the ever increasing need for light weight magnesium wrought products, limited commercial application of forming processes for magnesium alloys has been realized. This can be attributed to: (1) limited operative slip systems at room temperature in the hcp crystal structure [Hart1968, Ree1960, Kel1968, Bart1980, Kim2003, Bar(2007a, 2007b, 2004), Koi(2005a,2005b), Agn(2001,2003,2004,2005, 2006), Jai(2007,2008)], where for Mg the CRSS for basal slip has been shown to be significantly less than for prismatic or pyramidal slip, as reviewed by Koike [Koi2003b]; (2) the tendency to form strong textures during deformation as described in recent years by Agnew and others [Agn(2001,2003,2004,2005,2006), Jai(2007,2008), Kim2003]; (3) the highly anisotropic deformation behavior of textured microstructures that lead to macroscopically anisotropic mechanical properties [Kel1968, Agn2006, Kle2004, Koi2005a]; and (4) the prevalence of significant twinning during deformation that can lead to premature fracture, as described by Barnett [Bar2007a, 2007b,2007c), Jai(2007,2008)]. For these reasons, metal forming, which involves complex deformation paths to produce a component, is virtually impossible for ordinary grain size Mg alloys at ambient temperatures and at strain rates that are commercially viable. Microscopically, it has been shown that twinning becomes more prevalent as grain size increases, temperature decreases and strain rate increases [Yan2006, Jai2008) Mey2001] and it can cause fracture at low strains. An example of this is illustrated from the work of Barnett [Bar2007c]. Figure 1 shows crack formation associated with $\{10\bar{1}1\}$ double twins in room temperature deformation of rolled and annealed sheet derived from DC (direct cast) billet. Double twins are more favorably oriented for slip of <a> type dislocations than the parent grains from which they form, and large strains in the twins can lead to incompatibility stress and fracture [Hart1968, Ree1960, Bar2004, Yoo1981, Jai2008]. It has also been shown that activation of <c + a> slip rather than <a> slip on non basal planes is responsible in limiting ductility. Obara [Oba1973] has reasoned that this is because the <c + a> dislocations quickly dissociate into glissile <a> and sessile <c> segments greatly increasing work hardening and leading rapidly to fracture.

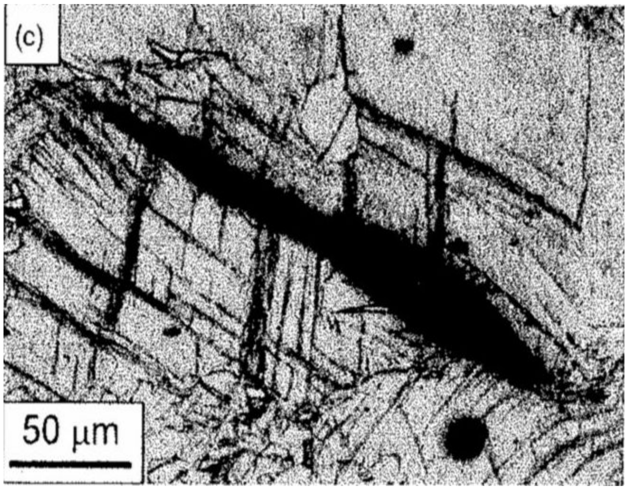

Figure 1. Crack initiation and voids in coarse grained commercial AZ31 sheet (Bar 2007c).

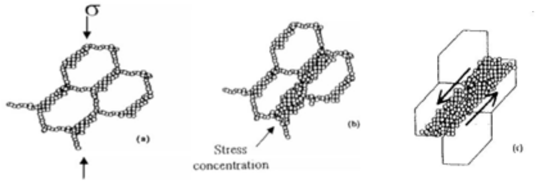

Figure 2. Twin induced heterogeneous re-crystallization leading to shear banding and hot cracking in coarse-grained commercial Mg alloys (Ion1982).

Attempts to produce finer-grained stock from commercially cast alloys as a means to avoid twin-induced fracture are fundamentally limited by the microstructure and deformation behavior described above. In particular, warm deformation of coarse grained material can, through both basal slip and twinning, cause local dynamic re-crystallization to form fine grains favorably oriented for deformation while at the same time leaving large grains between these soft regions where shear bands form and cause catastrophic cracking [Ion1982, del(2003, 2005b,

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2008)], as illustrated from Ion's work in Figure 2. To avoid this inherent propensity, many small reductions and reheats are required to gradually coax the original coarse-grained cast material down to sheet. These small increments of reduction of large grained stock limit the ability to refine grain size and to refine eutectic intermetallic size. Such wrought commercial stock retains a grain size of 15 to 90 µm, strong texture and brittle behavior well above ambient temperature. Hence formability and mechanical properties suffer.

Therefore, an NSF STTR Phase II Project has been undertaken to commercialize new technology in Mg processing that promised to overcome the cost and formability obstacles. In the STTR, the technical approach was to generate fine grain size uniformly to improve strength, ductility and formability in a process of few steps, compared to the costly 27 step procedure now required in commercial Direct Cast (DC) Mg sheet production. At the same time, the uniform fine grain structure was also postulated to overcome the large grain size and segregation problems associated with the relatively new developmental Twin Roll Cast (TRC) process. The project targets were yield strength of 250 MPa and elongation of 10%.

The process, Thixomolding Thermal Mechanical Processing (TTMP), builds upon the fine grained, isotropic, low porosity of Thixomolded Mg alloys which contain eutectic phases. Intense thermomechanical processing is applied to further refine the grain size and eutectic phases. Furthermore, additional thermal treatments can be applied to optimize strength, ductility and formability.

Experimental Procedures

As the starting stock for TTMP, Thixomolded® sheet bar was selected for its isotropic and fine grain structure, with minimum segregation and minimum porosity [Dec2008]. TMP (Thermal-Mechanical Processing) by heated rolling generated continuous dynamic recrystallization in this Thixomolded material. In addition, Twin Roll Cast (TRC) and Direct Cast + Extruded AZ31 were subjected to the same TMP process as the Thixomolded stock. Thermal treatments were applied in air after TMP.

The starting Thixomolded sheet bar was in the form of 100 mm x 150 mm panels of 3 mm thickness. These panels were molded on Thixomat's 280 commercial machine in AM60 (6 wt% Al, 0% Zn, 0.13% min Mn) and AZ61L (6 wt% Al, 1% Zn, low Mn) Mg alloys. Alloy content was varied in the Thixomolder by adding granules of AM60 or AZ61L. Other compositions were molded by Thixomats Thixoblending technique [Nan2007]. Thixomolding is an environmentally friendly and worker safe process, yielding stock that solidifies and cools at rates faster than 80°C/second – thus producing isotropic grain size < 10 µm with less segregation and much smaller grain size than Direct Cast and Twin Roll Cast starting stock. Additional panels 209 mm x 209 mm x 3 mm were molded on Thixomats 750 ton commercial machine to ascertain the porosity level and to produce larger sheet stock.

The Thixomolding operating parameters included (1) screw RPM of 158 to 170, (2) graphite die spray, (3) cycle time of 30 seconds, (4) shot pressure of 1541 to 2000 psi, (5) nozzle temperature of 500ºC to 530ºC, (6) peak barrel temperature of 615ºC to 640ºC, (7) die temperature of 230ºC and (8) shot time of 50 msec.

After preheating the above blanks, TMP was performed on heated 160 mm diameter rolls. Reductions were 50% in 1 pass. Post-TTMP thermal treatments were applied to some samples to rebalance the strength and ductility.

Room temperature tensile tests were run at a strain rate of 0.5 in/in/sec. Tensile elongation was measured between gauge marks on the samples. R values were derived from width and thickness of the broken tensile samples. Bendability was evaluated by free bending of sheet coupons upon themselves at room temperature.

Metallographic specimens were mounted, fine ground and mechanically polished and were later etched with an acetic- picral solution - containing 4.2 g picric acid, 70 ml ethanol, 10 ml acetic acid and 10 ml distilled water. Cross sections of the processed and tested materials were examined by optical and scanning electron microscopes.

Results

Tensile Properties. The mechanical properties of Thixomolded and TTMP AM60 are listed in **Table 1,** as are those of AZ61L in **Table II.** Yield strength is more than doubled by TMP for both alloys - to exceed 300 MPa. Post-TTMP thermal treatment A improved the combination of yield strength (328-343 MPA) and elongation (8-10 %). Thermal treatment B improves elongation to 17-21 %, at the expense of strength of 227-244 MPa.

Table I. Effect of Processing on TTMP AM60

Processing	YS, MPa	UTS, MPa	Elong, %
As Thixomolded	134	240	10
As TTMP	316	368	9
TTMP+Thermal Treatment A	328	371	10
TTMP+Thermal Treatment B	244	312	21

Table II. Effect of Processing on TTMP AZ61L

Processing	YS, MPa	UTS, MPa	Elong., %
As Thixomolded	132	240	10
As TTMP	305	362	6
TTMP+Thermal Treatment A	343	380	8
TTMP+Thermal Treatment B	227	314	17
TTMP+Thermal Treatment C	219	307	20

In **Table III,** it is apparent that neither TRC nor DC+extruded stock of AZ31 responded to TMP strengthening to the magnitude that Thixomolded AM 60 and AZ61L did.

Table III. Effect of Composition and Processing on Mechanical Properties of TMP Mg Alloys (50% reduction in One Pass)

Alloy	Processing	YS, MPa	UTS, MPa	Elong., %
AZ31	TRC+TMP	187	291	10

AZ31	DC+Extrude+TMP	174	282	17
AM60	Thixomold + TMP	316	368	9
AZ61L	Thixomold + TMP	305	362	6

Furthermore, edge cracking during TMP was minimal in Thixomolded material compared to medium edge cracking at 45° angles in DC extruded material and severe deep edge cracking in TRC stock.

Formability. Thermal treatment increased room temperature bendability of TTMP AM60 and AZ61L as well as Thixoblended/TTMP AZ6-1.5, AZ62, AZ63, AZ64, AZ55, ZA 65 and ZA 75.

R values for 6 sheets of AM60 in the TTMP plus Thermal Treatment condition are listed in **Table IV**. These average values range from 1.0 to 1.3, with the 45° samples on the high side of this range.

Table IV. Average R Values of TTMP + Thermal Treated AM60

Sheet	No. of Samples	Direction	R Value
1	4	Transverse	1.2
1	3	45°	1.2
1	5	Longitudinal	1.4
2	5	Transverse	0.9
2	5	45°	1.3
2	5	Longitudinal	1.1
3	5	Transverse	0.9
3	4	45°	1.1
3	4	Longitudinal	1.0
4	5	Transverse	1.0
4	6	45°	1.3
4	6	Longitudinal	1.1
5	6	Transverse	0.9
5	4	45°	1.4
5	5	Longitudinal	1.2
6	4	Transverse	1.1
6	5	45°	1.3
6	5	Longitudinal	1.2
Average	29	Transverse	1.0
Average	27	45°	1.3
Average	30	Longitudinal	1.2

Microstructures. The microstructure of as-Thixomolded AZ61L is shown in **Figure 3.** A uniformly fine proeutectic α phase is surrounded by β ($Mg_{17}Al_{12}$) particles that have been derived from the Liquid to α + β eutectic reaction. Dendrite arm spacing is less than 10 µm. The microstructure of AZ61L after TTMP is revealed in **Figure 4**. The α phase has been refined to a substructure. .The β particles are not distinguished in Figure 4; but appear as a dark cluster of fine particles. Twinning is not evident in the α phase. Sub-micron particles of β are evident within the α grains and at their boundaries.

Discussion

Many of the shortcomings of DC and TRC Mg sheet have been attributable to their proclivity to shear banding during hot working and to twinning - and to the limitations in slip systems. A fall out of these mechanisms is generation of strong textures, anisotropic properties, cracking during hot working and poor room temperature formability. This study confirmed the hot cracking at 45° during hot working and associated twinning in large grained TRC AZ31.

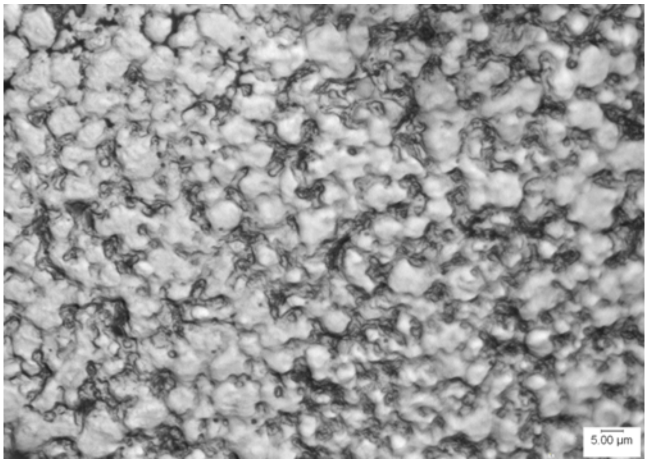

Figure 3. Optical micrograph of the as-Thixomolded microstructure of AM60. Fine α phase (white) surrounded by divorced eutectic of dark β phase ($Mg_{17}Al_{12}$) and grey α phase.

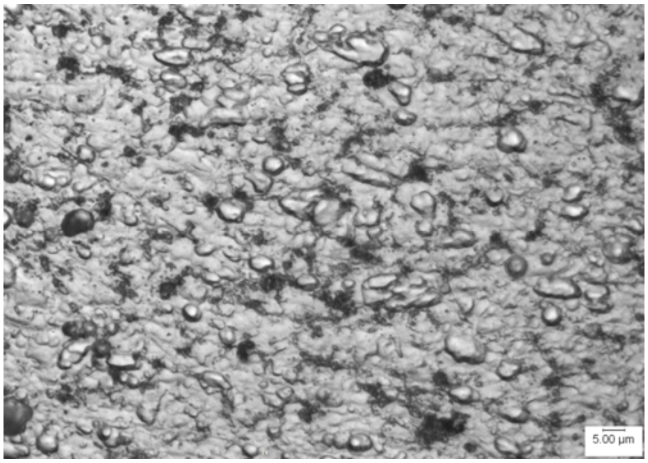

Figure 4. Optical micrograph of the TTMP and Thermally Treated microstructure of AM60. White α phase with fine grains; dark particles of refined β phase.

The technical hypothesis of this project was that initial fine grained, isotropic, low porosity molded sheet bar would alleviate the above-mentioned deformation problems and, rather, would favor more uniform slip deformation. This study confirmed that this is the case since twinning and shear banding were minimized in the TTMP process.

Both strength and ductility were enhanced by TTMP and appropriate thermal treatments. It is our belief that such response can be a result of grain refinement, intermetallic refinement and increase of residual dislocation content. The morphology of β particles as Thixomolded and after TMP indicated that some elongated eutectic β particles were subdivided by TMP into smaller, more equiaxed particles. In addition, some β particles were dissolved and reprecipitated as nano particles by TMP. Some of these fine β particles reported to grain boundaries, thus possibly retarding grain growth. Nano sized β particles within the grains would inhibit twinning as would the fine grains._Further studies are underway to confirm these hypotheses.

AM 60 was successfully fortified by Thixoblending® to add Zn; purposely designed to take advantage of the TTMP process and to increase strength. The scope for further alloy development is promising – both for optimizing the Zn/Al ratio and for experimenting with additional alloying elements by means of Thixoblending. In Thixoblending, chips of different alloy compositions and master alloys are premixed before feeding to the machine. These differing compositions then melt together and homogenize under the heat of the barrel and the mechanical action of the screw. Previous studies by Nandy [Nan2007] showed that these blended alloys follow the rule of mixtures, rendering the same mechanical properties as an alloy of the same average composition. A further aspect of Thixoblending is manufacturing agility, wherein purposely designed alloys can be ordered and delivered upon short notice. This degree of agility is undoubtedly not open to large scale DC and TRC plants. Thixoblending is likened to ordering from the "paint store".

The virtues of fine grain size in Mg alloys have been demonstrated in numerous papers. On strength, the Hall-Petch relationship holds - with strength being proportional to $d^{-1/2}$ (where d = grain diameter). In fact, this effect is stronger in Mg than in other structural alloys. On fatigue strength, a sister paper by Chen [Che2010] in this symposium shows fatigue strength of 150 MPa in fine grain TTMP plus thermally treated AM60. Nagata [Nag2007] and Kamakova [Kam2005] found enhancement of fatigue strength by grain refinement as did Kulvasova [Kul2009], who related fatigue strength to the Hall-Petch relationship. Somekawa [Som2005] and Liao [Lia2009] measured enhanced fracture toughness (K_{IC}) when grains were refined to less than 3 µm. Fine grain size lowers the ductile-brittle temperature of Mg and increases the compressive to tensile yield stress ratio [Eml1966].

Refining the size of β particles should increase ductility, toughness and formability since cracking stress is proportional to d^{-1}. Li2007 found that large β particles were subdivided during extrusion thus improving elongation. Lapovok [Lap2009] attributed void formation during superplastic forming to β particles; Reduction of β size should decrease this degradation during forming. Stress corrosion has been related to β phase cracking and hydrogen charging of β .by Chen [Che2009] and Winzer [Win2009]. Again, refinement of β should improve resistance to these environmental effects.

Further studies of fracture toughness, formability, corrosion, stress corrosion, creep, texture now are in order, to be related to processing and microstructure.

The TTMP process is a safe and low cost route to nanostructures. Such micro/nano-structure is generated in situ in the bulk by inexpensive processing, and fine nanopowders need not be handled – obviating safety concerns. The adoption of Thixomolded sheet stock on heated rolls affords fast and inexpensive commercial production - with few steps - on extant commercial equipment.

Conclusions

1. Thixomolding provides a fine grain low porosity microstructure amenable to thermomechanical processing to produce high strength/density Mg alloy sheet exceeding the targets of 250MPa yield strength and 10 % elongation..
2. Additional thermal treatment can be applied to attain high strength with good ductility.
3. Sheet so produced demonstrated good room temperature bendability in a range of AM and AZ Mg alloys, some of which were Thixoblended.
4. Low R values were found in AM60 sheet which had been subjected to TTMP plus thermal treatment.
5. Coarse twinning and shear banding were not evident in the microstructure of the fine grained TTMP sheet.

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