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PRECIPITATE FORMATION AND GRAIN REFINEMENT OF MG-AL-SN ALLOY DURING HOT DEFORMATION

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Abstract

Magnesium alloys are very popular in the automobile industry due to its high strength to weight ratio. However, the use of commercial magnesium alloys as sheet is limited by room temperature ductility. One way to improve the ductility may be to form precipitates during hot rolling. These may delay dynamic recrystallization, possibly leading to grain refinement, which is known to improve ductility. Equilibrium diagrams obtained from thermodynamic modeling software FactSage were used to design Mg-3Al-2Sn alloy, which should form Mg_2Sn precipitates during hot rolling temperatures around 300 °C. To investigate this prediction, the alloy was cast in a copper mould and precipitates formation characteristics were studied by using optical microscope (OM), scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectroscopy (EDS).

Introduction

The reduction of energy consumption and environmental pollution are driving forces in the automotive industry to improve fuel efficiency through light weight automobiles. Magnesium, being the eighth most abundant element in the earth crust and third most dissolved in seawater [1], is the lightest structural metal with the highest specific strength and a potential candidate to reduce the most weight of the vehicle. Although 36 % and 78 % lighter than aluminum and steel [2], respectively, very limited wrought magnesium alloys are commercially available in comparison to aluminum and steel [3].

To be used as a structural component in the automobile, alloys should have sufficient room temperature ductility and specific strength. However, magnesium shows brittle fracture with less than 10 % elongation at room temperature due to its HCP structure [4]. For improved formability at room temperature, a refined grain magnesium alloy not only shows better strength and ductility but also a lower ductile brittle transition temperature (DBTT) [5]. It is well established that strength of polycrystalline metals at room temperature can be improved by grain refinement according to the Hall-Petch relationship [6] as the higher grain boundary area impedes dislocation movement [5].

In Mg alloys, grain refinement has been accomplished by thermomechanical processing. For example, Mohri *et al.* [7] reported improved strength and ductility of Mg-4Y-3RE alloy due to grain refinement (as small as 1.5 μm) by hot extrusion and, Kumar *et al.* [6] produced a grain size less than 5 μm for AZ91 alloy by dynamic recrystallization (DRX) during high temperature extrusion. Recrystallization is an important mechanism by which grain refinement can be achieved in metals with low or medium stacking fault energy, such as magnesium [8]. Recrystallization is a restoration and usually thermally activated process by which relatively strain free grains are formed within the deformed structure consuming the deformed grains [9]. During the hot deformation process the term 'dynamic' is used to distinguish from the 'static' recrystallization process, which takes place after the deformation or during the post deformation annealing process [10]. When a critical deformation condition, i.e. critical dislocation density, is reached, dynamic recrystallization is initiated by the bulging of pre-existing grain boundaries at low strain rate [11]. Recrystallization can be retarded by the pinning of precipitates at the grain and sub-boundaries and also individual dislocations. For grain boundary pinning to occur, it is necessary to have sufficient amount of precipitates and the precipitates size should be smaller than a critical value [12].

In this study, an Mg-3Al-2Sn alloy was thermomechanically processed to generate precipitates during hot deformation with a view to affecting dynamic recrystallization (DRX). Aluminum is the most widely used alloying element with magnesium as it improves the strength, hardness, corrosion resistance and castability of pure magnesium [13]. Tin with a small amount of aluminum increases the ductility of the magnesium alloy, and Sn in any magnesium alloy reduces the risk of hot tearing during the extrusion and forging process [3]. Finally, the addition of Sn in magnesium alloy produces thermally stable Mg_2Sn precipitates [3].

Experimental Procedures

Thermodynamics software FactSage [14] was used to design candidate Mg alloys with the alloying elements Al and Sn. Commercially pure Mg, Al and Sn were melted using an induction furnace (20 kW, 5 kHz) in a graphite crucible and five ingots were cast in a copper mold preheated to 250 °C. The alloy addition and casting were done at 690 °C and 730 °C respectively with a mixture of CO_2 (99.5 %) and SF_6 (0.5 %) gases used to protect the liquid metal. The cast ingot was homogenized to dissolve all the precipitates. The aim is to form the precipitates again during the hot deformation process. The temperature for homogenization was selected using the FactSage generated equilibrium diagram.

Compression samples of 11.4 mm in height and 7.6 mm in diameter (h/d ratio of 1.5) were machined from the homogenized bar. Isothermal compression testing was done at 300 °C with a strain rate of 0.01 s^{-1} to strains of 0.2, 0.3, 0.4, 0.5, 0.7, and 0.9. Uniaxial compression test was performed using an MTS 810 coupled with a radiant furnace. The specimens were held at 300 °C for 10 minutes prior to deformation and water quenched immediately after the deformation.

Specimens for metallurgical examination were cut along the compression axis. Polished specimens were etched in a fresh solution of 10 ml acetic acid, 4.2 gm picric acid, 10 ml water and 70 ml alcohol for 3-8 seconds. Microstructural examination was carried out using a Nikon Epiphot 200 optical microscope with Clemex image analysis software. Grain size has been measured by linear intercept method. A Philips XL30 FEG SEM with energy dispersive X-ray spectroscopy (EDS) was used to examine the microstructure and characterization of the second phases. Microindentation hardness was measured by Clark CM-100AT machine with a load of 50 gram and a dwell time of 10 seconds.

Results and Discussion

Alloy design

The Mg-Al-Sn alloy was designed to target the homogenization and deformation temperatures, which are around 400 °C and 300 °C, respectively. Figure 1 shows the equilibrium phase fraction obtained from FactSage indicating the types and amount of precipitates formed during the equilibrium cooling of these two alloys. According to this diagram, a suitable homogenization (solution treatment) temperature is around 400 °C with secondary particles Mg_2Sn and $Mg_{17}Al_{12}$ starting to precipitate out from the Mg matrix below 340 °C and 210 °C, respectively. However, the exponential dependence of nucleation rate on undercooling indicates that the precipitation phenomena usually requires a minimum amount of undercooling to produce the necessary driving force to nucleate a particle [15]. Consequently, the deformation temperature was selected at 300 °C to allow about 40-50 °C of undercooling. More uniform and fine precipitates are formed with plastic deformation, which can generate high energy sites like dislocations, twins and deformation bands. These defects can accelerate the diffusion of solute atoms enhancing the subsequent aging kinetics and are the preferential location for the solute atoms to gather due to the minimum of energy needed. Second phases are preferentially formed at the grain boundaries during the thermomechanical processing but may also form inside the grain when annealing treatment is associated with the thermomechanical processing [16].

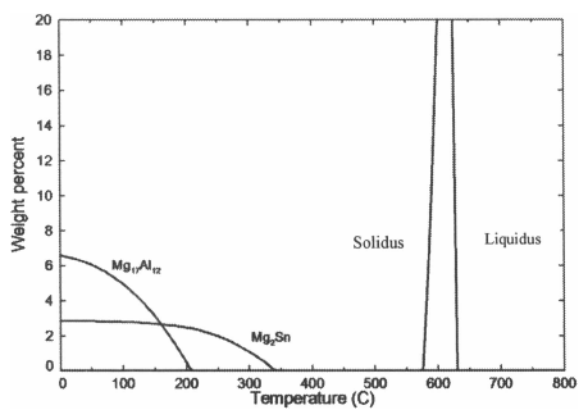

Figure 1: Equilibrium phase fraction of Mg-3Al-2Sn alloy predicted by FactSage.

Microstructure

Figures 2 and 3 show the as-cast optical and SEM microstructures of the alloy and EDS analysis of precipitates. The microstructure consists of primary α-Mg, and secondary particles between dendritic arms. The average grain size was measured as 78 µm. The precipitates are coarse having the size around 2-5 µm. EDS analysis confirms that the precipitates are mainly composed of Mg, Al, and Sn.

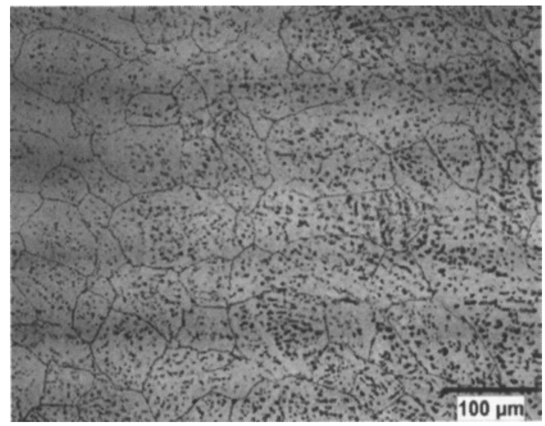

Figure 2: As-cast optical microstructure of Mg-3Al-2Sn alloy

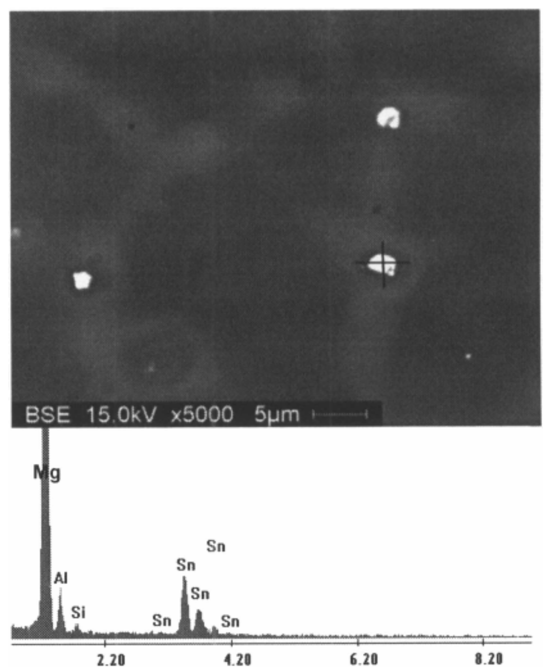

Figure 3: As-cast SEM microstructure of Mg-3Al-2Sn alloy and EDS quantitative analysis of the precipitates.

Although thermodynamically the precipitates were supposed to be dissolved above 340 °C, the dissolution of the precipitates in the matrix at this temperature is slow. In order to promote the homogenization, the temperature was selected as 420 °C. The samples were homogenized for 2, 4, 6, 8, 10 and 12 h to observe the dissolution behavior of the precipitates. No precipitate has been found after homogenization at 420 °C and for 10 h, as shown by the SEM microstructure in Figure 4-a. The average grain size of the homogenized sample is measured as 80 μm, which displayed almost no grain growth during the homogenization treatment. Before compression, the homogenized samples were held at the deformation temperature of 300 °C for 10 minutes to ensure a uniform temperature. Figure 4-b shows the SEM microstructure of the sample quenched immediately after the 10 minute hold and before compression. It is seen that the precipitates have started to form at the grain boundaries.

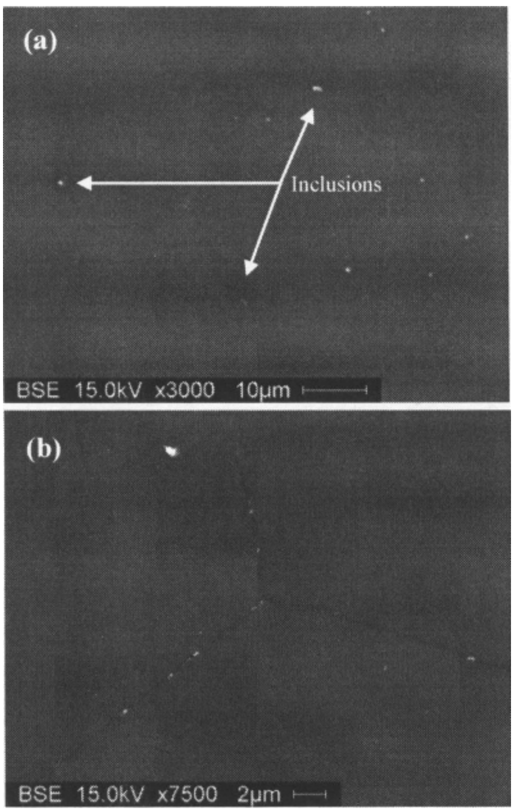

Figure 4: Homogenized and aged microstructure of Mg-3Al-2Sn alloy (a) Homogenized (b) Aging at 300 °C for 10 min and quenched

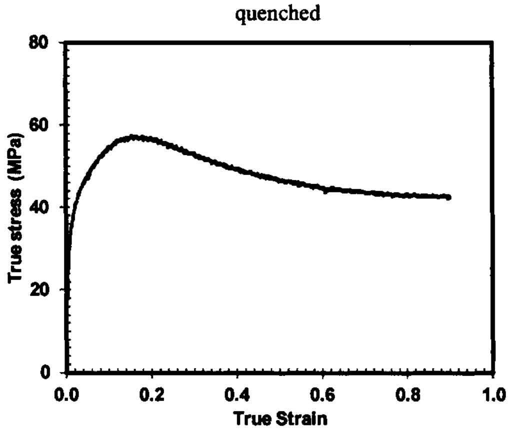

Figure 5: Flow behavior of Mg-3Al-2Sn alloy at 300 °C and strain rate of 0.01 s^{-1}.

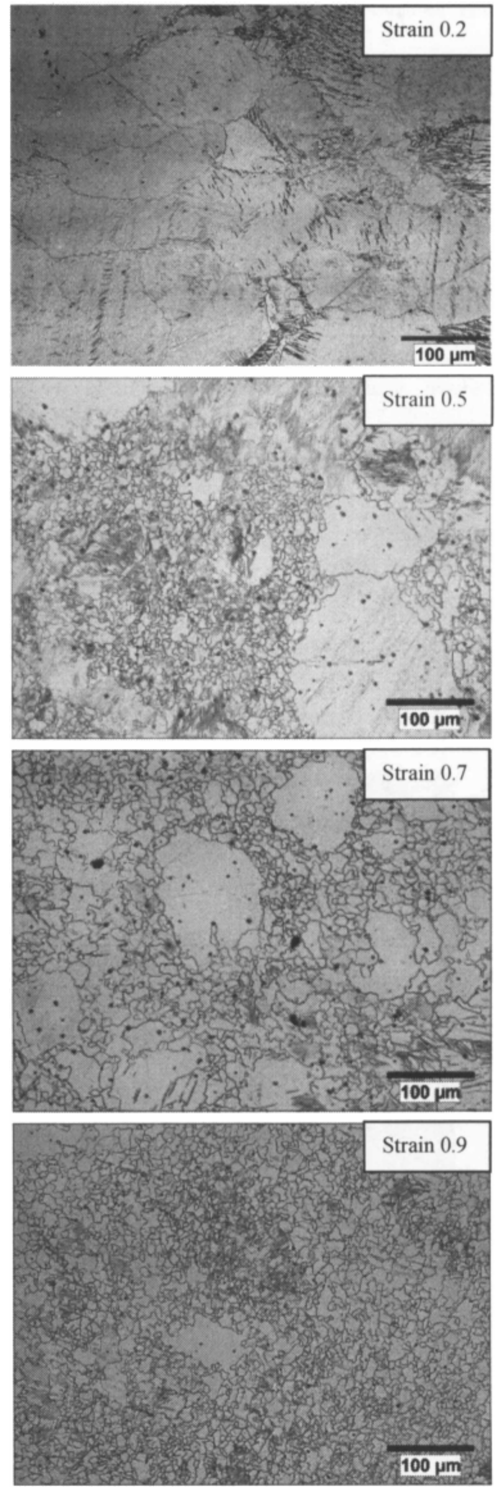

Figure 6: Microstructure evolution with different amount of deformation

Deformation behavior

The typical true stress and true strain curve obtained at a strain of 0.9 is shown in Figure 5. At 300 °C, the material exhibits work hardening, a single peak at low strain, followed by flow softening. The softening behavior after peak stress is attributed to dynamic recrystallization phenomena [10].

Microstructure evolution during deformation

Figure 6 shows the microstructures of hot deformed alloys subjected to various strains. A necklace type structure was observed with recrystallized grains distributed along pre-existing boundaries. The microstructures are characterized by dynamic recrystallization and grains are significantly refined. The recrystallized grains evolved dynamically as the static recrystallization was prevented by quenching the samples immediately after deformation. Dynamic recrystallization is attributed to magnesium alloy because of its low stacking fault energy (~ 78 mJ m^{-2}) and limited slip systems [8]. In general, for the hot working process, the dynamic recrystallization grain size depends on the deformation amount (strain), strain rate, and deformation temperature [6, 17, 18].

As can be seen in Figure 7, the DRX grain size increases with increasing strain up to strain of 0.7. The result is consistent with the results reported by Beer [19] for AZ31 alloy. Beer mentioned an initial increase in grain size with strain but reported to be fairly constant for strains more than 0.6 for AZ31 alloy deformed at a temperature of 400 °C and a strain rate of 0.1 s^{-1}. Figure 7 also shows that the % DRX increases with the increase in strain which indicates not only the occurrence of dynamic recrystallization but also the development of dynamic recrystallization with increasing the strain.

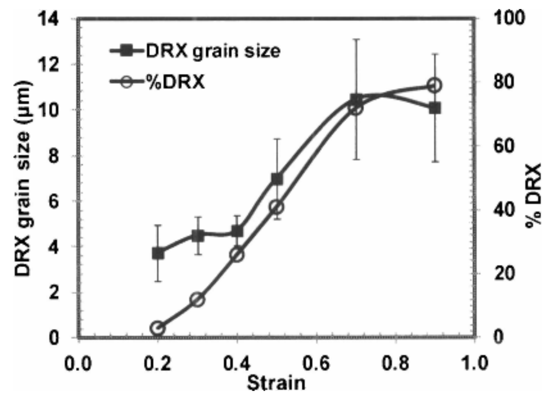

Figure 7: Variation of % DRX and DRX grain size with different strain.

Figure 8 shows the distribution of precipitates in the deformed alloy with precipitates 100-300 nm in size and mainly observed along the grain boundaries. The fine dispersion of precipitates may restrain grain growth by pinning the boundaries and dislocations.

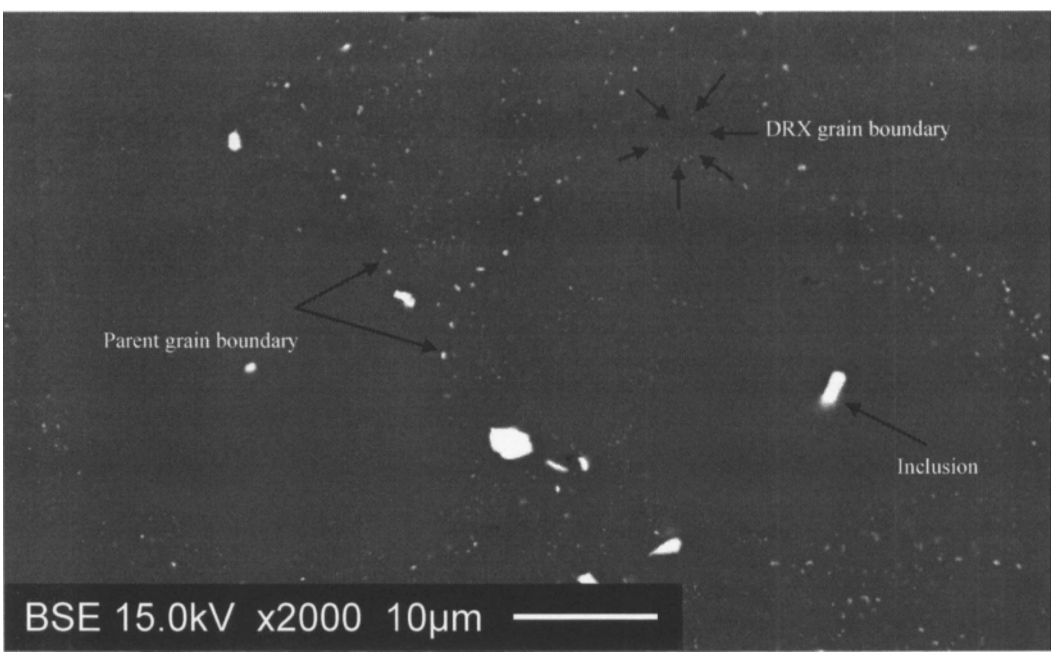

Figure 8: Grain boundary pinning effect by precipitates (300 °C, strain rate 0.01s^{-1}, strain 0.9).

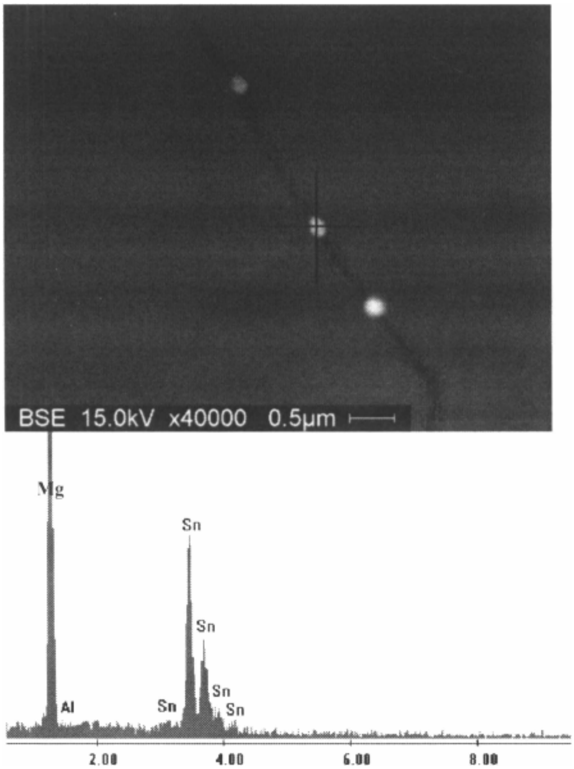

Figure 9: Precipitate formation at DRX grain boundary and EDS qualitative analysis of the precipitate.

The EDS study (Figure 9) confirms that the precipitates are mostly composed of Mg and Sn, which is consistent with the Mg_2Sn precipitate predicted by the thermodynamic calculation results from FactSage at 300 °C. The onset of dynamic recrystallization occurs during deformation when the strain exceeds a critical value, referred to as the critical strain for dynamic recrystallization [20], which is slightly lower than the peak strain on the flow diagram [21]. The formation of precipitates at the DRX grain boundary begins at ~0.4 strain in this study which is higher than the critical strain for the recrystallization (~0.15-0.18). In order to start the precipitate formation at the critical strain for DRX, future work includes deformation at a lower temperature such as 250 °C. At this temperature, the weight fraction of precipitate is expected to be double than that obtained at 300 °C (Figure 1).

Figure 10 shows the variation of room temperature hardness with strains. Yang *et al.* [22] reported the same trend of hardness change with strain for AZ31 alloy but deformed at different temperature and strain rate. The increase in hardness is due to the increase in vol % of DRX grains i.e. more fine grains development due to DRX, according to the Hall-Petch relationship.

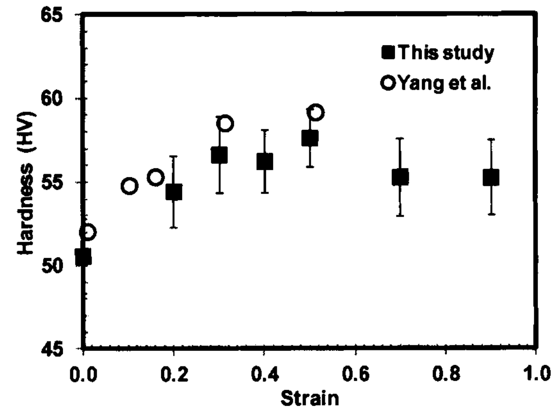

Figure 10: Variation of room temperature hardness with strain.

Summary

1. The experimental result supports the FactSage calculation in terms of homogenization temperature and precipitation prediction during deformation at 300 °C.
2. Amount of (volume percent) dynamic increases with increasing amount of deformation i.e. strain.
3. DRX grain size increases up to a certain strain then remain fairly constant with increasing strain.
4. Precipitation formation along the DRX grain may be associated with the grain refinement.
5. DRX grain boundary can be pinned by Mg_2Sn precipitates formed during deformation and dynamic recrystallization.
6. Microindentation hardness increases with the increase in deformation.

Acknowledgement

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