MATERIALS CHARACTERIZATION 62 (2011) 742–750

available at www.sciencedirect.com

ScienceDirect

www.elsevier.com/locate/matchar

Microstructure, mechanical properties and texture evolution of AZ31 alloy containing trace levels of strontium

*Alireza Sadeghi**, *Mihriban Pekguleryuz*

McGill University, Department of Mining and Materials Science, Montreal, Quebec, Canada

A R T I C L E D A T A

Article history:
Received 11 February 2011
Received in revised form 12 May 2011
Accepted 16 May 2011

Keywords:
Mg alloys
Sr addition
Grain refinement
Electron probe micro analysis
Hot deformation
Texture

A B S T R A C T

The effect of low levels of Sr (0.01, 0.03, 0.05 wt.%) on the microstructure, mechanical properties and texture of AZ31 magnesium alloy has been investigated. Thermodynamic modeling has been used to study the effect of Sr on phase precipitation at different temperatures. Cooling curve analysis reveals a decrease in solidification superheat with the addition of 0.03 wt.% Sr to AZ31. The as-cast microstructures of the alloys have been studied using optical microscopy (OM) and electron probe micro analysis (EPMA). Results show the refining effect of Sr on the grain size and on the β-$Mg_{17}Al_{12}$ precipitates through growth poisoning and inoculation, respectively. To investigate the mechanical properties of the alloys, as-cast samples were compression tested at elevated temperatures. Hot compression peak stress (σ_{max}) and critical strain before recrystallization (ε_c) initially drop but then increase. σ_{max} first decreases due to the depletion of Al from solid solution and then increases when the amount of the Al–Sr precipitates reach a significant amount. ε_c drops due to the acceleration of dynamic recrystallization kinetics as a result of grain refinement. It increases with increasing Sr when the concentration Sr in solid solution is increased leading to dislocation pinning and retardation of recrystallization. X-ray texture measurements on the hot compressed and extruded samples show a decrease in maximum intensity of the basal pole figures with increasing Sr as a result of reduced twining and the changes in Al and Sr concentrations in solid solution.

1. Introduction

Strontium is known to be an effective grain refining element in Mg alloys containing high levels of Al [1,2]. Having a low equilibrium solid solubility, Sr segregates to the surface of Mg grains and slows down the grain growth [3]. It has also been shown that Sr refines the beta ($Mg_{17}Al_{12}$) precipitates in AZ31 [4]. The effects of Sr in AZ31 are dependent on the concentration of Sr in the alloy and the type of second phase which is precipitated. When added to AZ31, Sr forms Al–Sr, Mg–Al–Sr, Mg–Sr and Sr–Zn precipitates. In the Mg–Al–Zn system Sr prefers to bind with Al rather than the other elements. Because of this high affinity of Sr to Al, all the precipitates in the AZ+Sr system could be classified into two groups: precipitates with Al and precipitates without Al. Sr-containing precipitates without Al ($Mg_{17}Sr_2$ and $SrZn_5$) only form at high concentrations of Sr where the affinity of free Al is decreased when most of it is captured in Al–Sr intermetallics. Sr precipitates with Al (Al_4Sr and Al_2Sr) are formed when Sr meets a high concentration of Al in the alloy. When the concentration of Al is low or the solidification condition slows down the diffusion of Al, Al–Mg–Sr precipitates form. In different alloying systems, many different stoichiometries for Al–Mg–Sr precipitates have been suggested [5–9]. Recently Janz et al. [10] have shown that τ-$Al_{38}Mg_{58}Sr_4$ is the only equilibrium ternary precipitate in the Mg–Al–Sr system and all

* *Corresponding author at:* Rm 2140 Wong Building, 3610 University Street, Montreal, Quebec, Canada, H3A 2B2. Tel.: +1 514 933 2348; fax: +1 514 398 4492.
E-mail address: alireza.sadeghi@mail.mcgill.ca (A. Sadeghi).

1044-5803/$ – see front matter
doi:10.1016/j.matchar.2011.05.006

the other reported stoichiometries are non-equlibrium precipitates which depend on the alloy composition and solidification conditions. L'Esperance et al. [9] have shown the decomposition of the non equilibrium Al–Mg–Sr ternary precipitate into Al_4Sr and Mg, by rejecting its excess Mg and absorbing Al from the matrix.

The addition of low levels of Sr into Mg–3Al–1Zn will result in a high Al/Sr ratio, in which case, Sr will find a high concentration of Al atoms and a precipitate with high Al/Sr ratio (Al_4Sr) will form. Although, the amount of precipitate is very small, it has significant effects of microstructure, mechanical properties and deformation texture [11–14]. However, research concerning the effect of trace levels of Sr on the most commonly used wrought AZ31 has not been extensively studied. In this work, the formation of new phases resulting from the addition of trace levels of Sr in AZ31 and its effects on mechanical properties and texture were investigated in as-cast and extruded samples by cooling curve analysis, thermodynamic predictions, electron probe micro-analysis (EPMA), hot compression and texture measurement.

2. Experimental Procedure

AZ31 alloys containing 0.01, 0.03 and 0.05 wt.% Sr were prepared using AZ31 extruded bars supplied by Applied Magnesium (Denver, CO) and Sr–10 wt.%Al master alloy (from Timminco, Haley, ON). AZ31 was melted in a graphite crucible using a high frequency induction furnace (NORAX). The melt was kept at 700 °C for 15 min under a gas mixture of CO_2-SF_6 to allow for sufficient dissolution and mixing of the alloying additions. The melt was cast at 720 °C into preheated (400 °C) cylindrical steel dies. The chemical compositions of the alloys have been determined by inductively coupled plasma (ICP) and are summarized in Table 1. Cooling curve analysis has been performed using sand cups and exposed junction thermocouples. The details of this analysis have been reported elsewhere [11].

Optical microscopy (OM) specimens were etched in a picric–acetic solution. Grain size was measured using the line intercept method. Electron probe microanalysis (EPMA) was performed on polished samples to identify the phase compositions and to investigate the element distribution across the microstructure. Pseudo-ternary phase diagrams of the Mg–Al–Sr–1 wt.%Zn–0.4 wt.%Mn were calculated and plotted using the thermodynamic system introduced into the FactSage software.

Hot compression samples were machined out of the cast bars and compressed using an MTS universal machine equipped with a radiation furnace and a quartz tube conducting Ar gas around the heated sample to avoid oxidation at elevated temperatures. The effect of friction on the contact surfaces between the samples and the anvils was reduced by using boron nitride powder and mica plates. Samples containing different levels of Sr were compressed to strain of 0.8 at a strain rate of 0.01 s^{-1} at 250 °C and 350 °C. In order to retain the as-deformed microstructure, samples were quenched in water immediately after deformation. The hot extrusion of cast cylinders was conducted using a 100 T hydraulic press and a forward extrusion die set. Samples were extruded at 250 °C and 350 °C with a 3:1 extrusion ratio to 10 mm bars with a ram speed of 4 mm s^{-1}. The details of extrusion trials are explained elsewhere [12].

Texture measurements on the hot compressed and extruded samples were performed using a Bruker D8 X-ray diffractometer. Hot compressed samples were sectioned parallel to the cylinder base (section was perpendicular to the compression direction). On the other hand, the deformation zone of the extruded samples was sectioned perpendicular to the circular cross section (section contains the extrusion direction). TextTools texture analysis software was used to calculate the orientation distribution function (ODF), to generate the pole figures, and to measure the volume fraction of prismatic planes in various directions.

Table 1 – Chemical composition of the alloys.

Alloy	Chemical composition (wt.%)			
	Al	Zn	Mn	Sr
AZ31	3.15	0.89	0.52	–
AZ31+0.01%Sr	2.94	0.87	0.49	0.012
AZ31+0.03%Sr	2.96	0.86	0.52	0.038
AZ31+0.05%Sr	3.19	0.82	0.51	0.053

3. Results and Discussion

3.1. Microstructure

One of the important questions in this study was related to how trace levels of Sr added to AZ31 would be incorporated into the microstructure during solidification. There are four possible scenarios for Sr atoms to be located in different regions and phases. They could (i) dissolve in α-Mg as a solid solution element, (ii) dissolve in the β-$Mg_{17}Al_{12}$ precipitates, (iii) segregate at the grain boundaries, or (iv) precipitate either as Al–Sr or Al–Mg–Sr second phases. Large atomic radius difference (r_{Sr}/r_{Mg}=34) [15] between Sr and Mg results in low maximum equilibrium solid solubility (0.04 at.% [16]). Solute trapping and extension of solid solution have also been reported for Sr atoms in Mg [16]. However, these latter happen at very high cooling rates (10^5–10^7 K s^{-1}) which is quite different from in the cooling rates observed in conventional casting. Janz et al. [10] have reported that there is negligible Sr-solubility in Mg and in $Mg_{17}Al_{12}$. They have shown that in samples containing high levels of Sr (>2 at.%), Sr forms a distinct ternary phase τ-$Al_{38}Mg_{58}Sr_4$ instead of dissolving in β-$Mg_{17}Al_{12}$.

According to the thermodynamic calculations and phase diagrams shown in Fig. 1, Al_4Sr is the only Al–Sr phase in the AZ31+(0.01–0.05) wt.%Sr composition range which precipitates after Al_8Mn_5 and α-Mg. Isothermal sections of the pseudo-ternary phase diagram (Fig. 1b–d) show that from 350 °C to 250 °C and then down to 25 °C, the Al–Sr precipitates are stable while Al–Mn precipitates switch their stoichiometry toward a lower Al/Mn ratio [17]. In Fig. 3, the grain size of as-cast samples is plotted versus Sr level. It can be seen that the grain size is refined with increasing Sr. The fact that Sr