Effect of thermomechanical treatment on microstructure and hardness behavior of AZ63 magnesium alloy

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Due to their high specific strength and low density, magnesium alloys are widely used in many weight-saving applications. This research is aimed at investigating the microstructure and hardness of commercial AZ63 alloy specimens subjected to two different thermomechanical treatments (TMTs). For the first TMT, after solution treated at the temperature of 380 °C for 20 h, AZ63 alloy specimens were 5% cold worked by rolling process followed by ageing at the temperatures of 150 °C and 250 °C for 3, 9 and 25 h. In the second TMT, the specimens were solution treated at the temperature of 380 °C for 20 h, underwent 2% cold worked and quenched in water of 0 °C. Half of the specimens were then 2% cold worked whilst the rest were rolled to 8% cold worked. All the specimens were then aged at the temperatures of 150 °C and 250 °C for 3, 9 and 25 h. Optical microscope was used to analyze the microstructures of the specimens. Hardness test was too conducted to measure the effect of the treatments on the specimens. Results show that two-step aging enhances the hardness of the specimens due to the distribution of fine β -phase ($\text{Mg}_{17}\text{Al}_{12}$) in the alloy matrix. The results also reveal that, the best hardness from the first TMT was produced by specimen that was pre-aged at 150 °C whereas, in the second TMT, aging at 250 °C exhibited the best hardness values.

KEY WORDS Thermomechanical treatment; Magnesium alloys; Cold work; Aging; AZ63

1 Introduction

In recent years, due to technological developments and concern on efficient usage of energy, magnesium alloys have attracted significant interest to be used in many applications. This is due to its excellent advantages such as low density and cost and abundant supply^[1], high strength-to-weight ratio^[2,3], high damping capacity, dimensional stability, good machinability and recyclability^[4]. The usage of magnesium alloys is increasing in industries including automotive^[5–10], transportation, aerospace, microelectronics, power-tools and new energy sources^[1,4]. However, on account of its poor ductility^[6,9] and due

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to the difficulty in dislocations movement in non-basal slip systems^[11] has limits magnesium and its alloys to certain applications. According to the Hall-Petch relationship, the mechanical properties of polycrystals at ambient temperatures can be enhanced through grain refinement. TMT may be considered as a process which produces fine grains, but for magnesium alloys detail studies are rarely conducted.

Most of magnesium-aluminum alloys contains aluminum with small amounts of zinc^[12]. These alloys which depend on the concentration of alloying elements are susceptible to age-hardening. So, applying prestrain prior to ageing may modify the morphology of the precipitates during subsequent ageing and hence improve mechanical properties of the alloy^[13].

AZ63 alloy is one of the most extensively used magnesium alloys^[1]. The only existing phase in this alloy, which precipitates continuously and discontinuously, is the intermetallic β -phase ($\text{Mg}_{17}\text{Al}_{12}$)^[14].

The aim of this investigation is to identify the effect of two different TMTs on the microstructure and hardness of a commercial AZ63 alloy. These TMTs include pre and post-cold working prior to and after artificial ageing.

2 Experimental Procedures

Approximately 150 test specimens were prepared by machining a commercial AZ63 ingot into a size of 100 mm×10 mm×10 mm each. The chemical composition (wt pct) of the machined ingots was Al 5.96, Zn 3.43, Mn 0.18, Ni 0.003, Fe 0.004, Si 0.04, Mg balance.

The prepared specimens were divided into 3 main groups. The first were the untreated specimens meant to be as a control specimens. The other two groups was subjected to two different TMTs. The second group was solution treated by heating the specimens in a resistance furnace to 380 °C for 20 h and quenched in cold water of 0 °C. The heating furnace used was capable of controlling the temperature to the accuracy of 3 °C. The specimens were then subjected to 5% cold worked by cold rolling the specimens at room temperature. Subsequently, a number of specimens were aged at 150 °C at 3 different aging times, namely 3, 9 and 25 h. The rest of the specimens were aged at 250 °C for 3, 9 and 25 h. The cold working and aging procedure were repeated before the specimens were analyzed. Cold working (pre or post) promotes precipitations during aging due to the plastic deformation. Moreover, two-step aging results in a higher density of precipitates^[15]. The specimens undergone the 1st TMT is denoted as in Table 1.

The third group of specimens were 2% cold rolled at room temperature and solution treated by heating the specimens to 380 °C for 20 h. At the end of the 20 h the specimens were quenched in cold water of 0 °C. The specimens were then 2% and 8% cold rolled at

Table 1 Treatments applied to the different specimens in the first TMT

Specimens	Condition					
	Post-aging		Post cold working	Pre-aging		Pre-cold working
	<i>t</i> /h	<i>T</i> /°C		<i>t</i> /h	<i>T</i> /°C	
1	3	150	5%	3	150	5%
2	9	150	5%	9	150	5%
3	25	150	5%	25	150	5%
4	3	250	5%	3	250	5%
5	9	250	5%	9	250	5%
6	25	250	5%	25	250	5%

room temperature. Half of the 2% cold rolled specimens were then subjected to an aging temperature of 150 °C for 3, 9 and 25 h. The rest of the specimens were aged at the temperature of 250 °C divided for the periods of 3, 9 and 25 h. Due to the brittleness of the alloy, the rolling processes should be performed carefully. To ensure more accurate and reliable results, each cycle is repeated twice. The overall TMT procedures for the two groups of specimens in the investigation are shown in Figs.1a and 1b. The thermo-mechanical processing was applied to specimens with the aim of obtaining homogenized distribution of precipitations in microstructure to achieve higher hardness values for the alloy.

Fig.1 Two different TMT procedures conducted on group 2 (a) and group 3 (b) specimens.

All the untreated and treated specimens were mechanically ground and polished using 6, 3 and 1 micrometer diamond powders and etched using 0.5% nital solution. Nikon optical microscope was used for microstructure analysis. Brinell hardness tests were conducted at ambient temperature using hardness test machine with an indenter diameter of 2.5 mm and applied load of 1838.7 N. The test was conducted at various stages of the TMTs, *i.e* at the pre-cold work, pre-aging, post cold work and post-aging.

3 Results and Discussion

Hardness of the untreated specimens machined from commercial AZ63 alloy was measured as 66 HB. The hardness of homogenized and 5% pre-cold worked specimens stood at 54 and 77 HB, respectively. Fig.2 demonstrates the result of the hardness test on some specimens (Table 2), which were experienced the first TMT. It is clear that pre-ageing at 150 °C results in increasing the hardness of the specimen Nos.1, 2 and 3.

In general, the increase in hardness of the alloy specimens is likely due to the preci-

precipitation of β -phase in the alloy matrix^[16]. Fig.3a shows the microstructure of untreated specimens in which β -phases is found to be distributed heterogeneously in the primary equiaxed dendrites of α -magnesium matrix whilst Fig.3b shows the β -phase at a higher magnification. A lamellar structure with alternately arranged β -phase and α -magnesium solid solution supersaturated with aluminum^[17] along with large intermetallic β -phase is observed in Fig.3c. It also reveals that the discontinuous β -phases precipitated at grain boundaries.

In contrast, pre-ageing at the temperature of 250 °C causes a decrease in the hardness of the specimens Nos.4, 5 and 6, comp-

Fig.2 Hardness test curves of different specimens underwent the first TMT (A: Untreated, B: Homogenized, C: Pre-cold worked, D: Pre-aged, E: Post-cold worked, F: Post-aged).

Fig.3 (a) Optical micrograph of untreated and etched AZ63; (b) optical micrograph of un-etched and untreated AZ63; (c) detailed view of the lamellar structure (etchant: 0.5% nital solution).

ared to those pre-aged at 150 °C with the same amount of pre-cold working. In TMT treated processes two phenomena, namely hardness decrease due to recovery^[13] and hardness increase due to the β -phase precipitation, compete together. By extending aging time, the softening effect becomes more dominant than hardening^[18], so the hardness of the specimens after aging for 9 h starts to decrease in the second TMT process.

Fig.4 shows the microstructures of the specimens which were pre-aged at the temperatures of 150 °C and 250 °C for 9 h. It can be observed from Fig.4 that by increasing the pre-aging temperature from 150 °C to 250 °C coarsening of the precipitates (β -phase) occurs and they tend to form along grain boundaries.

Fig.2 also shows that work hardening increases the hardness of all TMT treated specimens. The tendency in the increase in hardness is slightly more in specimens that have been pre-aged at the temperature of 250 °C, because it allows recovery and has higher potential to be work hardened. Cold working enables an increase in the density of mechanical twins, where it has a great effect on the ageing behavior of the Mg alloy. Mechanical twins are commonly observed in the microstructure of magnesium alloys^[19] (Figs.6c and 6d). Due to post-ageing and coarsening of the β -phase particles, which consequently increases

Fig.4 Optical micrograph of specimens pre-aged at temperatures of 150 °C (a) and (b), 250 °C (c) and (d) for 9 h.

Fig.5 Influence of ageing time and temperature on the hardness of the specimens work hardened to 2% (a) and 8% (b) before ageing of the second TMT.

the particles distance, dislocations move more conveniently resulting in the decrease of the hardness of the specimens.

Figs.5a and 5b show the hardness of specimens that has been 2% and 8% post-cold worked in the second TMT treatment procedures. It is observed that ageing at the temperatures of 150 °C and 250 °C for 3 h decreases the hardness of the specimens. However, extending the ageing time to 9 h increases the hardness of the specimen. This phenomenon may be due to the development of fine β -phase precipitates. Increasing the ageing time to 25 h reduces the hardness of the specimens owing to the over-ageing phenomenon. From

Fig.6 (a) Optical micrograph of specimens aged at the temperature of 150 °C for 9 h in the first TMT; (b) the same specimen but etched; (c) for 25 h in the second TMT and etched; (d) micrograph of (c) in higher magnification (etchant: 0.5% nital solution).

both Figs.5a and 5b it can be concluded that the second TMT treatment does not increase the hardness of the specimens considerably, however slightly higher hardness values obtains at the ageing temperature of 250 °C.

The solution treatment at 380 °C for 20 h leads to a complete dissolution of the β -phase into the α -phase grains. Aging of the solution treated specimens changes considerably the microstructure of AZ63 alloy. It causes the lamellar eutectic structure to be more homogeneous and the β -phases distribute in the eutectic structure.

Figs.6a and 6b show an optical micrograph of the first TMT treated specimen aged at 150 °C for 9 h, which consists of fine β -phase distributed in matrix and a well-homogenized eutectic microstructure.

From Fig.5, it can be seen that the optimum hardness can be obtained by a definite aging time. It means that the coarsening of the β -phases occurs due to over-aging and consequently the hardness of the alloy decreases.

By increasing cold working or strain hardening on the second TMT treated specimens, twinning lines appear in the microstructure due to the limited slip system of magnesium HCP structure for the temperature below 225 °C^[20]. Fig.6c shows an optical micrograph of the second TMT treated specimen, aged at 150 °C for 25 h, in which β -phase and mechanical twins are revealed. Fig.6d demonstrates mechanical twins more clearly.

The effectiveness of precipitation depends on the aging temperature. Continuous precipitation usually immerses in matrix at high temperatures, approximately 350 °C, whereas the formation of discontinuous precipitation, which occurs at grain boundaries, dominates

at lower temperatures^[5]. To improve the hardness of the specimens, continuous precipitation should be enhanced and discontinuous precipitation should be prohibited by aging^[17]. However, Figs.4b and 4d reveal that discontinuous precipitates distribute heterogeneously at grain boundaries. So, in the tested specimens discontinuous precipitates are responsible for increasing hardness rather than continuous precipitates.

4 Conclusions

By taking into account the above discussion the following results can be concluded:

- (1) Two-step aging increases the hardness of the specimens.
- (2) Pre-strain enhances the hardness of the TMT-processed specimens.
- (3) For the first TMT, pre-aging at 150 °C increases the hardness to a greater degree than pre-aging at 250 °C.
- (4) For the second TMT, aging at 250 °C increases the hardness to a greater degree than aging at 150 °C.
- (5) Distribution of fine β -phase in the microstructure due to ageing increases the hardness of the specimens.

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