Microstructure and Aging Behavior of Nonflammable AZ91D Mg Alloy

Seok Hong Min, Tae Kwon Ha

Abstract—Phase equilibria of AZ91D Mg alloys for nonflammable use, containing Ca and Y, were carried out by using FactSage® and FTLite database, which revealed that solid solution treatment could be performed at temperatures from 400 to 450°C. Solid solution treatment of AZ91D Mg alloy without Ca and Y was successfully conducted at 420°C and supersaturated microstructure with all beta phase resolved into matrix was obtained. In the case of AZ91D Mg alloy with some Ca and Y; however, a little amount of intermetallic particles were observed after solid solution treatment. After solid solution treatment, each alloy was annealed at temperatures of 180 and 200°C for time intervals from 1 min to 48 hrs and hardness of each condition was measured by micro-Vickers method. Peak aging conditions were deduced as at the temperature of 200°C for 10 hrs.

Keywords—Mg alloy, AZ91D, nonflammable alloy, phase equilibrium, peak aging.

I. INTRODUCTION

AZ91D magnesium alloy has good mechanical properties and excellent casting properties and is typical die-casting alloy with great application potential [1]-[3]. Many studies have shown that alloying is a useful means of improving the performance of AZ91D magnesium alloy, in which rare earth's function such as refining structures, solid solution strengthening and dispersion strengthening have been widely recognized. AZ91D magnesium alloy belongs to an aging alloy, so that heat treatment is also an effective way to improve the performance of AZ91D magnesium alloy. If combining alloying and aging treatment properly, therefore, the performance of AZ91D magnesium alloy could be more improved.

The need for weight reduction, particularly in portable electronics, telecommunications, aerospace and automobile industries has stimulated engineers to be more creative in their choice of materials. Magnesium and its alloys, with very low density and high strength to weight ratio are very good candidate. Although a wide variety of applications can be realized, use of Mg alloys at present is limited mainly due to formability and corrosion resistance. Magnesium alloys with Al and Zn have been widely applied to the automobile industry. The addition of rare earth elements and Ca is an effective way

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to improve the mechanical properties of magnesium alloys at elevated temperatures [4], [5].

Researches on alloying of AZ91D magnesium alloy mainly have so far focused on improving microstructure and performance of as-cast alloy, with less attention paid to effect of alloying on the aging process. Actually the aging effect relies largely on the original microstructure and aging process, microstructure, and performance of alloy are evidently relative to the added alloying elements. It has been reported that addition of rare earth element could weaken the aging hardening effect and could delay the peak aging time of AZ91D alloy [6].

The effect of the addition of beryllium and rare earth on the ignition-proofing the AZ91D Mg alloy was confirmed by Zeng et al. [7]. While the tensile properties of Mg alloy containing Be were deteriorated, after addition of RE, the tensile properties of the AZ91 alloy containing Be were enhanced approaching those of AZ91 alloy. The addition of calcium to Mg alloys retards oxidation during heating. A thin dense CaO film forms on the surface of the melt. The ignition temperature increases by 250 K after an addition of 5wt.% Ca added into pure magnesium. It is confirmed that a dense and compact protective MgO/CaO layer formed at elevated temperature [8]. When the ass ration of Ca-Al was higher than 0.8, the Mg alloys showed a good heat resistance. Precipitates in the Mg_2Ca phase were more stable and heat-resistant than that in the $Mg_{17}Al_{12}$ phase.

In the present study, aging behavior of AZ91D magnesium alloy and ignition proof AZ91D Mg alloy containing 0.3wt.% Ca and 0.2 wt.% Y. Peak aging conditions were deduced by solution treatment at 420°C for 2 hrs followed by aging at temperatures of 180 and 200°C for up to 48 hrs.

II. EXPERIMENTAL PROCEDURES

Fig. 1 Appearance of the AZ91D Mg alloy ingot used in this study

The materials used in this study were ingots of commercial grade AZ91D Mg alloy and ingots with basically the same

compositions containing 0.3wt.% calcium and 0.2wt.% yttrium for non-flammability [9]. Appearance of the ingot used in this study was given in Fig. 1. Phase equilibria of AZ91D Mg alloy and AZ91D Mg alloy containing 0.3wt.% calcium and 0.2wt.% yttrium were calculated using FactSage®, a commercial thermodynamic simulation software, and FTLite database. Solid solution treatment was carried out on both alloys at 420°C for 2 hrs followed by water quenching. For comparison, air cooling and furnace cooling were also performed after solid solution treatment. Aging treatment was carried out at 180°C and 200°C for up to 48 hrs. Microstructure evolution and variation of Vickers hardness after aging treatment were investigated in this study.
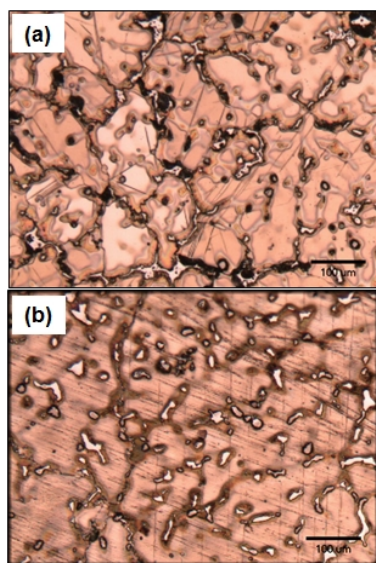

Fig. 2 Microstructure of as-cast ingots of AZ91D alloy (a) and nonflammable alloy (b)

III. RESULTS AND DISCUSSION

Fig. 3 shows microstructures of as-cast ingots of AZ91D alloy and nonflammable AZ91D alloy used in this study. In every charge of die-casting, two samples of 3 mm thickness and two of 0.8 mm thickness were produced. As shown in Fig. 3 (b), fabricated tensile specimen is very sound and has no defect on surface. A large amount of second phases, presumably considered to be β-$Mg_{17}Al_{12}$ phases, was observed in both alloys.

Fig. 4 shows equilibrium phases and their weight fractions as a function of temperature calculated in this study. As shown in Fig. 4 (a), liquid phase would disappear at 450°C on cooling and precipitation of β-$Mg_{17}Al_{12}$ phase is expected from temperature of 395°C. It is obvious from this figure that supersaturated solid solution can be obtained by annealing the ingot at temperatures from 400 to 450°C. In the case of nonflammable alloy as shown in Fig. 3 (b), on the other hand, purely supersaturated solid solution cannot be obtained within this temperature range. Due to yttrium addition, precipitates such as Al_3Y, M_2Y, Al_4MgY, and Mg_3YZn_8 were expectedly formed from liquid phase. As calculated in this study, relative

amount of β-phase of AZ91D alloy is a little lower than those of nonflammable alloy, which can be confirmed by microstructure observation given in Fig. 2. Using these results, solid solution treatment temperature was taken as 420°C to obtain supersaturated α solid solution.
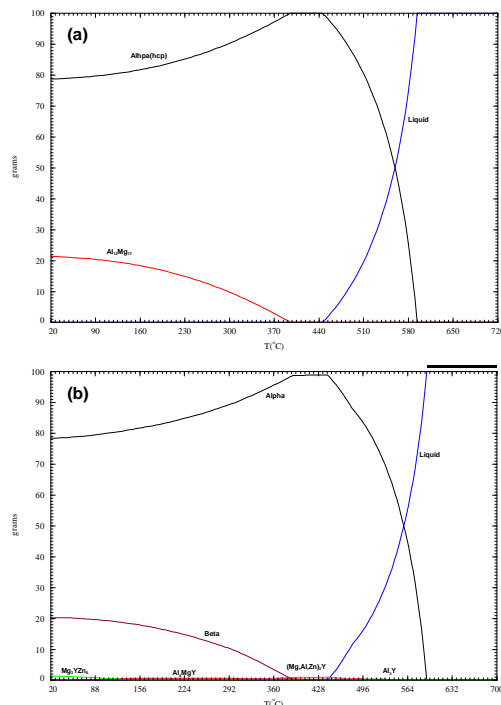

Fig. 3 Phase equilibria of AZ91D alloy (a) and nonflammable alloy (b) fabricated in this study

In Fig. 4, microstructure obtained after solution treatment followed by water quenching has been illustrated, in which almost all β-phases appeared to dissolve into matrix. In the case of nonflammable alloy, some precipitates still remained after solid solution treatment as shown in Fig. 4 (b).
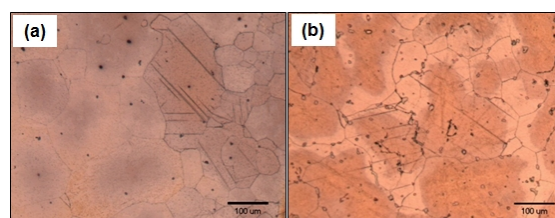

Fig. 4 Typical microstructure of AZ91D alloy (a) and nonflammable alloy (b) obtained after solid solution treatment followed by water quenching

The effect of cooling rate on the microstructure after solid solution treatment was investigated by employing air cooling and furnace cooling after solution treatment and the result is given in Fig. 5. With cooling rate decreased, precipitation of β-phase and lamellar formation were prominent.
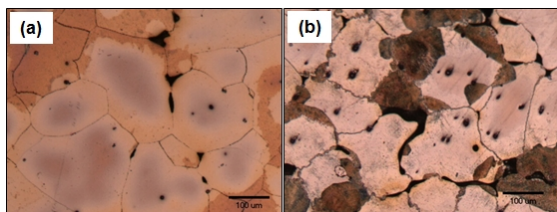
Fig. 5 Microstructure of AZ91D alloy obtained after solution treatment followed by air cooling (a) and furnace cooling (b)
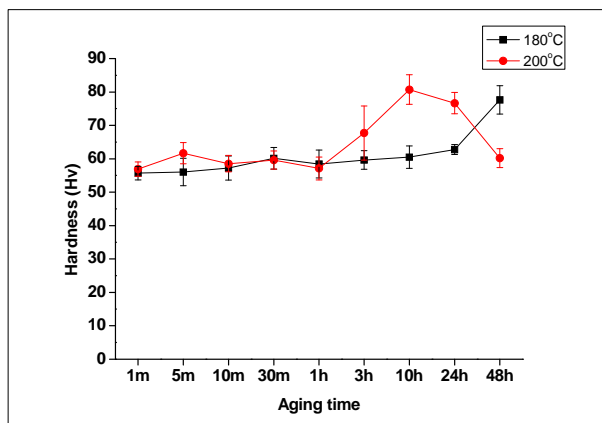

Fig. 6 Hardness of AZ91D alloy as a function of aging time obtained after aging treatment at 180 and 200°C
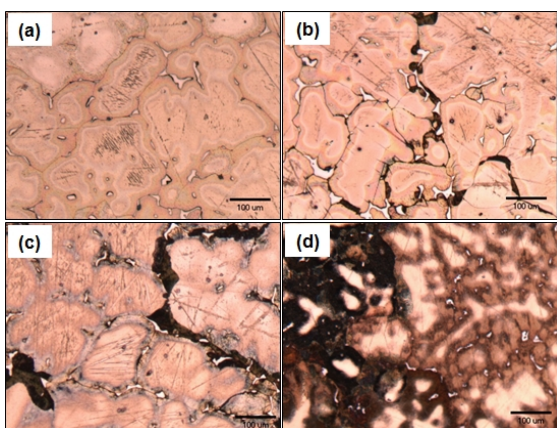

Fig. 7 Microstructure of AZ91D alloy aged at 180°C for various times of 10 min (a), 1 hr (b), 10 hrs (c), and 48 hrs (d), respectively

Fig. 6 shows variation of hardness as a function of aging time obtained from aging treatment at 180 and 200°C conducted on AZ91D Mg alloy. Hardness of AZ91D alloy was found to increase monotonically with aging time at 180°C, while, at 200°C, after increased until 10 hrs and reached maximum value, then hardness decreased with further aging time. Typical aging curve has been obtained at 200°C and peak aging conditions were deduced as 200°C for 10 hrs. As shown in Fig. 7, with aging time increased at 180°C, β-phase precipitated along the grain boundaries and the amount appeared to increase monotonically. Similar result was obtained at aging treatment at 200°C. With aging time increased, however, β-phase was

found to precipitate not only along the grain boundaries but also within the matrices and even lamellar structure could be observed after aging for 48 hrs.
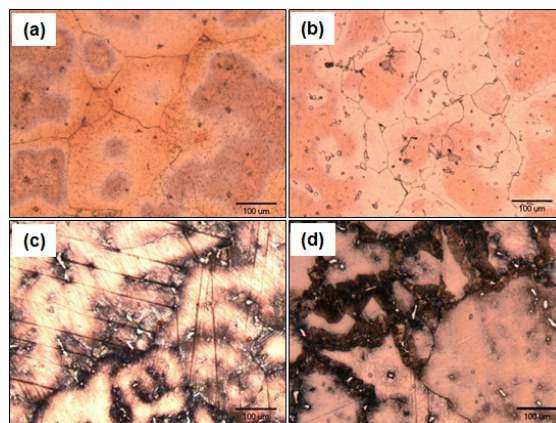

Fig. 8 Microstructure of AZ91D alloy aged at 200°C for various times of 10 min (a), 1 hr (b), 10 hrs (c), and 48 hrs (d), respectively
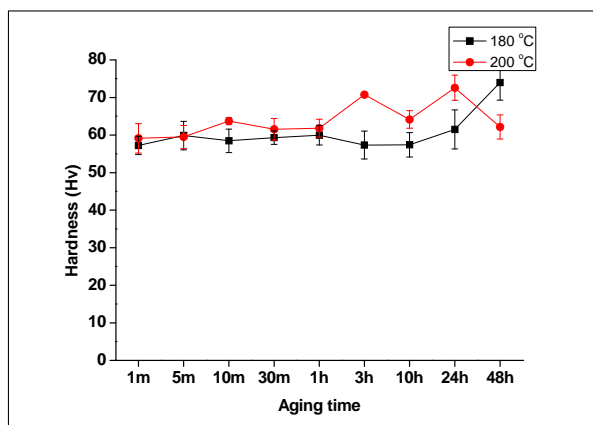

Fig. 9 Hardness of nonflammable AZ91D alloy as a function of aging time obtained after aging treatment at 180 and 200°C

Fig. 9 shows variation of hardness as a function of aging time obtained from aging treatment at 180 and 200°C conducted on nonflammable AZ91D Mg alloy. Hardness of nonflammable alloy was very similar to that of AZ91D alloy given in Fig. 6. Interestingly double peak in hardness of nonflammable alloy aged 200°C was observed, which should be more elucidated by further investigation. Microstructure observed by optical microscopy as given in Fig. 10 showed very similar feature to that of AZ91D alloy given in Fig. 8.

It is well known that small amount of rare earth elements can exist in the β phase in the form of solid solution besides great amount of yttrium form intermetallic compounds [3]. Because the electronegativity difference between yttrium and aluminum is larger than that between magnesium and aluminum, there is a stronger chemical affinity between yttrium and aluminum. Thus yttrium in β phase would enhance the chemical stability of the β phase, and accordingly, would inhibit decomposition of

β phase during solution treatment, which is presumably attributed to somewhat lower hardness of nonflammable alloy.
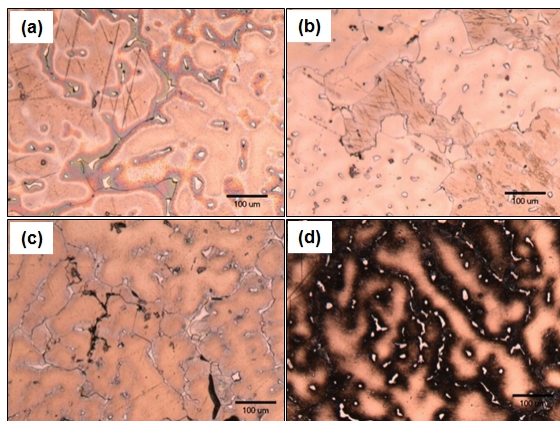

Fig. 10 Microstructure of nonflammable AZ91D alloy aged at 200°C for various times of 10 min (a), 1 hr (b), 10 hrs (c), and 48 hrs (d), respectively

IV. CONCLUSIONS

In the present study, aging behavior of AZ91D magnesium alloy and ignition proof AZ91D Mg alloy containing 0.3wt.% Ca and 0.2 wt.% Y. Supersaturated solid solution can be obtained by annealing the ingot at temperatures from 400 to 450°C. Solid solution treatment conditions were deduced by solution treatment at 420°C for 2 hrs. Aging treatment was carried out at temperatures of 180 and 200°C for up to 48 hrs. Typical aging curve has been obtained at 200°C and peak aging conditions were deduced as 200°C for 10 hrs.

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