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Effect of Aging Treatment on Mechanical Properties of Non-Flammable AZ91D Mg Alloy

Ju Hyun Won, Hyun Woo Lee, Seok Hong Min, Tae Kwon Ha

Abstract—Microstructure and mechanical properties of AZ91D Mg alloys for nonflammable use, containing Ca and Y, were investigated in this study. Solid solution treatment of AZ91D Mg alloy with Ca and Y was successfully conducted at 420°C and supersaturated microstructure with almost all beta phases resolved into matrix was obtained. After solid solution treatment, the 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 from the results as at the temperature of 200°C for 10 hrs. Hot rolling was also carried out at 400°C by the reduction ratio of 0.6 through 5 passes followed by recrystallization treatment. Tensile and compressive properties were measured at room temperature on the specimens of each process, i.e. as-cast, solution treatment, hot rolling, and recrystallization.

Keywords—Mg alloy, AZ91D, nonflammable alloy, hot rolling, peak aging, tensile test.

I. INTRODUCTION

MAGNESIUM belongs to alkaline earth metals, which occupy the second main group of the periodical table of elements. It was discovered in the eighteenth century and named after the ancient Greek district of Magnesia in Tehessaly. This silvery-white metal is the eighth most abundant element, comprising 2.7% of earth's crust. Due to high reactivity, magnesium is not found in elemental form in the nature but only in chemical complexes, widely distributed in rock structures, seawater and lake brines.

The inherent advantages of magnesium include a unique blend of low density, high specific strength, stiffness, electrical conductivity, heat dissipation, and absorption of vibration. When combined with easy machining, casting, forming and recycling, magnesium is seen as a very attractive material for a large volume of applications. In recent years, the interest in magnesium has grown dramatically, which has spurred academic research and industrial trials to identify more efficient ways of manufacturing the primary metal, as well as a search for new alloys and extending areas of their application.

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 [4]-[6]. 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 addition of rare earth elements and Ca is an effective way to improve the mechanical properties of magnesium alloys at elevated temperatures [4], [5]. 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-proof AZ91D Mg alloy was confirmed by [7]. 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].

In the present study, tensile and compressive behaviors of ignition-proof AZ91D Mg alloy containing 0.3wt.% Ca and 0.2 wt.% Y were investigated after various manufacturing processes such as casing, solid solution treatment at 420°C for 12 hrs, hot rolling at 400°C by the thickness reduction of 0.6 through 5 passes, and recrystallization at 400°C for 3 hrs. 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

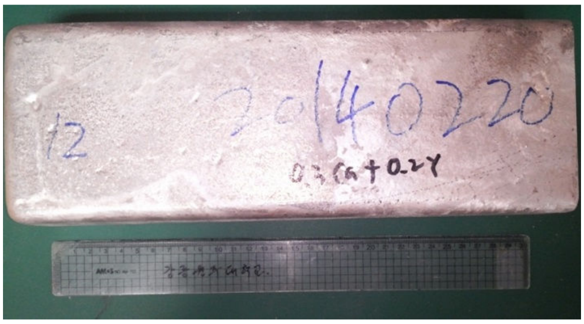

Fig. 1 Appearance of the AZ91D Mg alloy ingot used in this study

J. H. Won and H. W. Lee are with the Department of Advanced Metal and Materials Engineering, Gangneung-Wonju National University, 120 Gangneung-daehangno, Gangneung, Gangwon 210-702, South Korea.

S. H. Min and T. K. Ha are with the Department of Advanced Metal and Materials Engineering, Gangneung-Wonju National University, 120 Gangneung-daehangno, Gangneung, Gangwon 210-702, South Korea (phone: 82-10-2367-1989; e-mail: tkha@gwnu.ac.kr).

The materials used in this study were ingots of AZ91D Mg alloy containing 0.3wt.% calcium and 0.2wt.% yttrium for non-flammability [9]. Appearance of the as-cast ingot used in this study was given in Fig. 1 and typical microstructure was given in Fig. 2. Phase equilibria of the non-flammable AZ91D Mg alloy were calculated using FactSage®, a commercial thermodynamic simulation software, and FTLite database, from which conditions for solid solution treatment was taken as at 420°C for 2 hrs followed by water quenching. 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. After solid solution treatment, hot rolling was conducted on the ingot at 400°C by the thickness reduction of 0.6 through 5 passes followed by recrystallization at 400°C for up to 3 hrs. After each process, tensile and compression tests were carried out at room temperature under the strain rate of 5×10^{-4} s^{-1} and microstructures were observed.

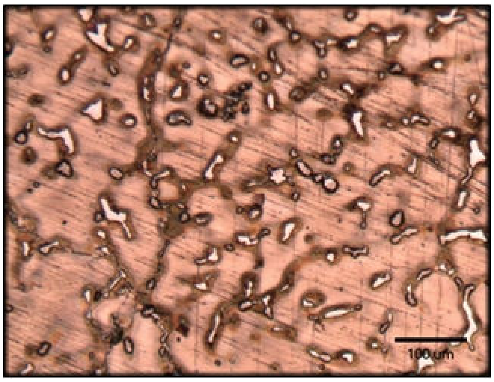

Fig. 2 Microstructure of as-cast nonflammable AZ91D alloy used in this study

III. RESULTS AND DISCUSSION

As shown in Fig. 2, microstructure of as-cast non-flammable AZ91D alloy used in this study revealed that a large amount of second phases, presumably considered to be β-$Mg_{17}Al_{12}$ phases, as expected from the result of phase equilibria given in Fig. 3. Fig. 3 shows equilibrium phases and their weight fractions as a function of temperature calculated in this study. 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, however, 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. Using these results, solid solution treatment temperature was taken as 420°C to obtain supersaturated α solid solution.

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. Some precipitates still remained after solid solution treatment as expected from calculated phase equilibria given in Fig. 3.

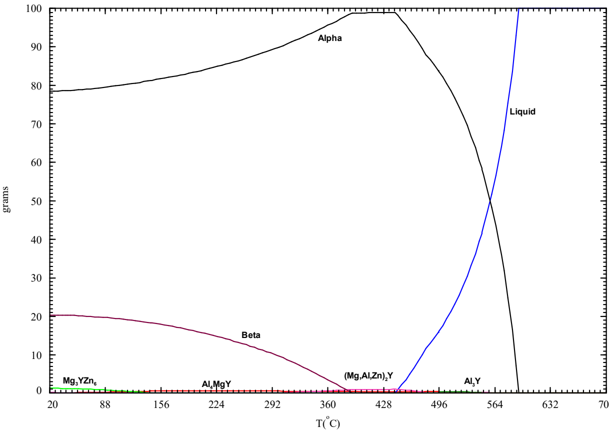

Fig. 3 Calculated phase equilibria of AZ91D alloy containing Ca and Y fabricated in this study

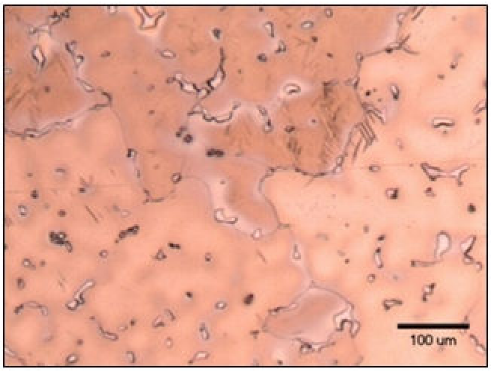

Fig. 4 Typical microstructure of nonflammable AZ91D alloy obtained after solid solution treatment followed by water quenching

Fig. 5 shows variation of hardness as a function of aging time obtained from aging treatment at 180 and 200°C. Hardness of nonflammable 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 treatment. Typical aging curve has been obtained at 200°C and peak aging conditions were deduced as 200°C for 10 hrs. Interestingly double peak in hardness of nonflammable alloy aged 200°C was observed, which should be more elucidated by further investigation. 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.

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.

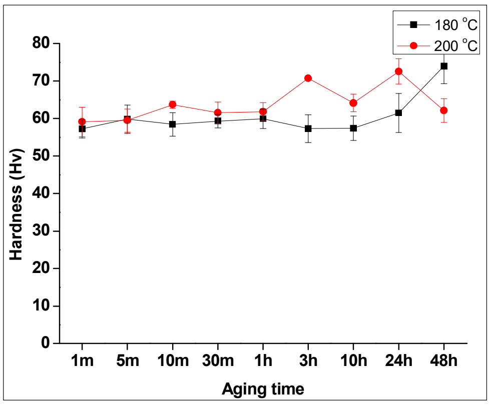

Fig. 5 Hardness of nonflammable AZ91D alloy as a function of aging time obtained after aging treatment at 180 and 200°C

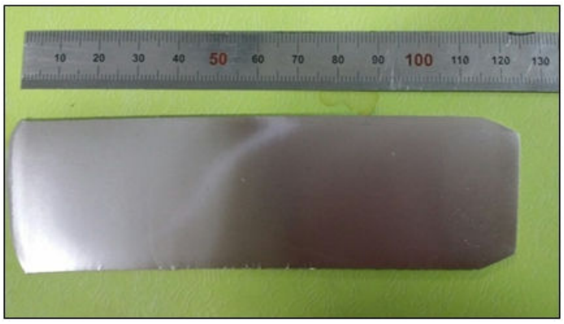

Fig. 6 Appearance of hot rolled sheet of nonflammable AZ91D alloy obtained at 400°C through 5 passes. Final thickness was 0.9mm

Fig. 6 shows an appearance of hot-rolled sheet, of which the thickness was 0.9 mm, obtained at 400°C through 5 passes. Although some edge cracks were observed, the surface of sheet shows excellent state without any cracks.

Fig. 7 shows typical microstructure of hot-rolled sheets, in which a large amount of deformation twins together with slip bands can be observed. Comparing with commercial AZ91D as shown in Fig. 7 (b), interestingly, grain size of nonflammable alloy was much smaller and density of slip band was much higher. During hot rolling process, it is expected that dynamic recrystallization would occur. The rolling temperature of 400°C is sufficiently high enough for dynamic recrystallization to occur. From Fig. 7, it is apparent that dynamic recrystallization more rapidly occurs in nonflammable AZ91D alloy than commercial one.

Fig. 8 shows tensile test results conducted on nonflammable AZ91D alloy after each fabrication processes such as casting, solid solution treatment, hot rolling, and recrystallization. Due to resolution of second phases after solid solution treatment, strength of nonflammable AZ91D alloy decreased a little. Severe strain hardening caused by the hot rolling process increased strength abruptly and decreased elongation. Subsequent recrystallization annealing process apparently enhanced strength and elongation simultaneously. Yield strength was obtained as 117 MPa, tensile strength 256 MPa, and elongation was obtained as 6.1 % in annealed specimen. Fig. 9 shows compression test results conducted on nonflammable AZ91D alloy after each fabrication processes such as casting, solid solution treatment, hot rolling, and recrystallization. It is very interesting to note that the compressive yield strength of nonflammable AZ91D Mg alloy showed the lowest value of 82 MPa after recrystallization annealing, which is much lower than that form tensile test result.

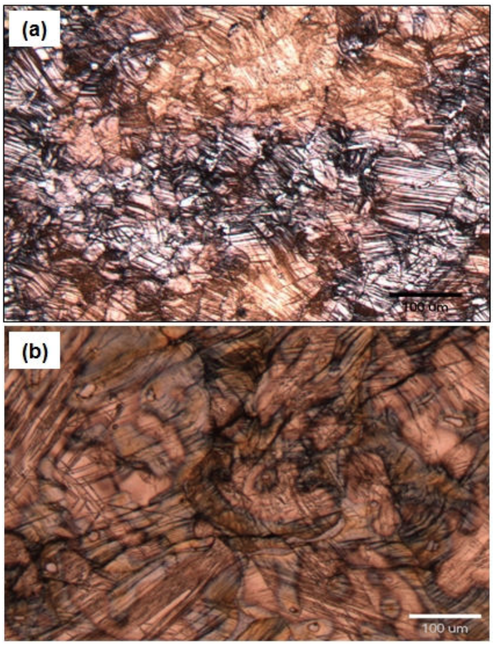

Fig. 7 Microstructures of hot-rolled nonflammable AZ91D alloy sheet (a) and commercial AZ91D alloy sheet (b)

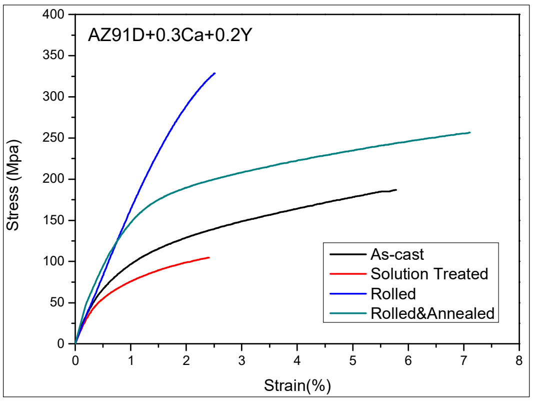

Fig. 8 Tensile test results on the nonflammable AZ91D alloy sheet conducted after casting, solid solution treatment, hot rolling, and recrystallization annealing.

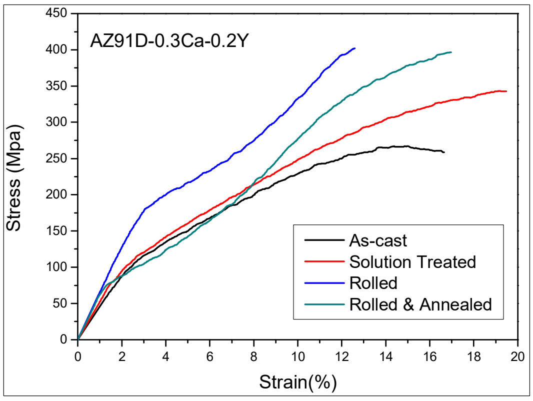

Fig. 9 Compression test results on the nonflammable AZ91D alloy sheet conducted after casting, solid solution treatment, hot rolling, and recrystallization annealing

IV. CONCLUSIONS

In the present study, tensile and compressive behaviors of ignition-proof AZ91D Mg alloy containing 0.3wt.% Ca and 0.2 wt.% Y were investigated after various manufacturing processes such as casing, solid solution treatment at 420°C for 12 hrs, hot rolling at 400°C by the thickness reduction of 0.6 through 5 passes, and recrystallization at 400°C for 3 hrs. 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. A large amount of deformation twins together with slip bands were observed. The grain size of nonflammable alloy was much smaller and density of slip band was much higher. Yield strength was obtained as 117 MPa, tensile strength 256 MPa, and elongation was obtained as 6.1% in annealed specimen. Compressive yield strength of nonflammable AZ91D Mg alloy showed the lowest value of 82 MPa after recrystallization annealing, which is much lower than that form tensile test result.

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REFERENCES

[1] B. L. Mordike and T. Ebert, Mater. Sci. Eng. A, vol. 302, p. 37, 2001.
[2] X. Cui, H. Liu, J. Meng, and D. Zhang, Trans. Nonferr. Met. Soc. Of China, vol. 20, p. 435, 2010.
[3] F. Guo, P, Li, X. Gao, and J. Xu, J. Rare Earth, vol. 28, p. 948, 2010.
[4] M. O. Pekguleryuz, and E. Baril, Mater. Trans., vol. 42, p. 1258, 2001.
[5] B. R. Powell, A. A. Lou, V. Rezhets, J. J. Bommarito, and B. L. Tiwari, SAE Tech. Paper 2001-01-0422, Soc. Automotive Eng., p. 406, 2001.
[6] G. Wu, F. Yu, H. Gao, C. Zhai, and Y. P. Zhu, Mater. Sci. Eng. A, vol. 408, p. 255, 2005.
[7] X. Q. Zheng, Q. D. Wang, Y. Z. Lu, W. J. Ding, C. Lu, Y. P. Zhu, C. Q. Zhai, and X. P. Xu, Scripta Mater., vol. 43, p. 403, 2003.
[8] B. S. You, W. W. Park, and L. S. Chung, Scripta Mater., vol. 42, p. 403, 2002.
[9] T. S. shih, J.-H. Wang, and K.-Z. Chong, Mater. Chem. & Phys., vol. 85, p. 302, 2004.