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EFFECTS OF CROSS ROLLING AND CALCIUM ADDITIONS ON THE MICROSTRUCTURE AND THERMAL PROPERTIES OF AZ31 MAGNESIUM WROUGHT ALLOY DURING HOT DEFORMATION

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ABSTRACT

Magnesium is the lightest structural metal and magnesium alloys are therefore obvious candidates in weight critical applications. The hexagonal close-packed structure of magnesium results in poor room temperature formability. However, on heating, several magnesium alloys show superior forming properties, with the ability to deform to very high strains. In the current work, effects of cross rolling and 0.7% Ca additions during hot deformation on the microstructure and thermal properties of AZ31 magnesium wrought alloys were investigated. Grain refinement in AZ31 alloy containing initial coarse grains was successfully achieved by thermos-mechanical processing (TMP) by dynamic recrystallization. After hot rolling normal and bimodal grain-size distribution were observed in AZ31 and AZ31-0.7%Ca alloys respectively. SEM and EDX results confirmed that the number and size of β-$Mg_{17}Al_{12}$ particles were increased due to the addition of small amount of calcium. Indeed, during hot deformation, the discontinuous precipitation of β-$Mg_{17}Al_{12}$ phase strongly influences the microstructural changes. Dynamic recrystallization was favored in grains having β-$Mg_{17}Al_{12}$ phase as close neighbor. DSC was also carried out to compare thermal properties of both AZ31 and AZ31-0.7%Ca alloys.

Keywords: AZ31; Hot rolling; Dynamic recrystallization; Microstructure and Thermal properties

1. INTRODUCTION

Magnesium alloys are the lightest structural alloys and becoming increasingly attractive for engineering applications because of their low density, excellent damping capacity, good recycling capacity and machinability [1, 2]. Magnesium is currently widely used in car components and physical electronic items [1-3]. Currently, these components have to be made by casting, which results in modest strength and is not suitable for large thin structure. Because magnesium suffers poor formability and limited ductility because of its hexagonal crystal structure with limited slip systems [2, 4]. Accordingly, many researchers have been tried to improve the shape forming properties of wrought Mg alloys [2, 5]. Microstructural refinement is an effective way for increasing both ductility and strength of these alloys [2].

The deformation behavior of Mg is well studied and there have been a number of studies that aim to reduce the grain size of magnesium below the values produced by conventional processing. Equal channel angle extrusion (ECAE), severe hot rolling, accumulated roll bonding, and biaxial reverse corrugation have all been shown to be capable of producing grain sizes in AZ31 of less than 3μm [6-10]. Some scientists deformed AZ31 magnesium alloy at different elevated temperatures to get desired properties [11]. Yet another alternative practical approach is using alloying and micro-alloying elements with the purpose of further improving the mechanical properties and processing performances of Mg–Al-based alloys [2,12-14].

Grain refinement by a factor of 10 to 80 times commonly occurs during hot rolling. The hot rolling process involves multiple cycles of rolling followed by reheating, and in a number of Mg alloys it has been reported that even finer structures can be achieved if a small number of high strain rolling passes are used [15-17]. On the other hand, addition of small amount of calcium has huge impact on dynamic recrystallization during hot rolling [2].

The aim of this present work is to investigate the combined effects of cross rolling and 0.7% Ca additions during hot deformation on the microstructure and thermal properties of AZ31 magnesium wrought alloys

2. EXPERIMENTAL

To prepare AZ31 and AZ31-0.7% Ca magnesium alloys, melting was conducted in a induction furnace at 750°C and melts were then poured into a preheated (250°C) metal mold in the presence of argon atmosphere. After casting x-ray fluorescence (XRF) of alloys were carried out for elemental analysis to ensure desired composition. As-cast alloys were then homogenized in Protherm resistance heating furnace at 350°C temperature for 8 hours and then quenched in water. Then homogenized alloys were cut into 10 mm thick plate for rolling which were carried out at 400°C.

During rolling both AZ31 and AZ31-0.7%Ca alloys were rolled to strain 24% reduction in 3 passes in order to analyze the effect of calcium on dynamic recrystallization. After AZ31 alloy was rolled from 10mm to 3.6mm to strain 64% reduction in 16 passes.

Optical metallography was carried out in order to examine the microstructural characteristics. Sample preparation for optical metallography consisted on grinding on emery paper with increasingly finer grits, followed by polishing with 3µ and 1µ diamond paste and final polishing using colloidal silica. The grain structure was revealed by subsequent etching using a solution of ethanol (70 ml), picric acid (4.2 g), acetic acid (10 ml) and distilled water (10 ml).

. Scanning electron microscopy (SEM) of as-homogenized AZ31 and as-homogenized AZ31-0.7%Ca alloys were conducted and microanalysis was also carried out by X-ray dispersive spectroscopy (EDS) system of SEM. Differential scanning calorimetry (DSC) of 21 mg of both as-homogenized AZ31 and as-homogenized AZ31-0.7%Ca samples were also carried out.

3. RESULTS AND DISCUSSION

3.1 MICROSTRUCTURAL OBSERVATIONS

Fig. 1 shows the optical microstructures of A) as-cast B) as-homogenized C) as-rolled [24% reduction and 3 passes] D) as-rolled [64% reduction and 16 passes] AZ31 alloys.

Average grain size of as-cast AZ31 alloy is 121µm. This alloy was then homogenized at 350°C temperature for 8 hours in order to minimize segregation and produce more homogenized structure. Grain growth occurred after homogenization and average grain size of as-homogenized AZ31 alloy is 138 µm. After that, rolling to strain 24% reduction in 3 passes refinement of grain structure occurred and the more equiaxed grains were indicative of dynamic recrystallization occurring during hot deformation processes [3]. The possible cause could be the result of repeated heating associated with the multiple rolling passes [18]. After 3 passes average grain size of as- homogenized AZ31 alloy was reduced from 138µm to 11.89µm. Still, the specimen receiving rolling reduction by 64% in 16 passes and average grain size was then reduced from 11.89µm to 6.56µm. As a result, major grain refinement was accomplished in the initial passes, and the final passes were merely effective in slight migration of grain boundaries to change the shape of the grains. After 16 passes grains are more uniform and average grain size is below 100µm and so AZ31 alloy might exhibit super-plasticity [18].

Fig. 2 shows the optical microstructures of as-rolled [24% reduction and 3 passes] AZ31-0.7%Ca alloy. Generally, due to the suppression of the dynamic recrystallization by Ca-containing particles during hot deformation process, the addition of calcium in AZ31 results refinement of grain structure [1-2]. However, in our work due to the presence of 0.7% Ca average grain size of AZ31 alloy increased from 11.89µm to 15.11µm. This is due to bimodal grain size distribution that is observed in AZ31-0.7%Ca alloy. Fig. 2 shows that in some regions grains are very small and grains in other regions are very large. The bimodal distribution may be due to the inhomogeneous deformation near particles which favors the particle stimulated nucleation during recrystallization and these Ca-containing particles can preferentially hinder the grain growth after recrystallization [2]. Thus, grains near Ca-containing particles are very small in size.

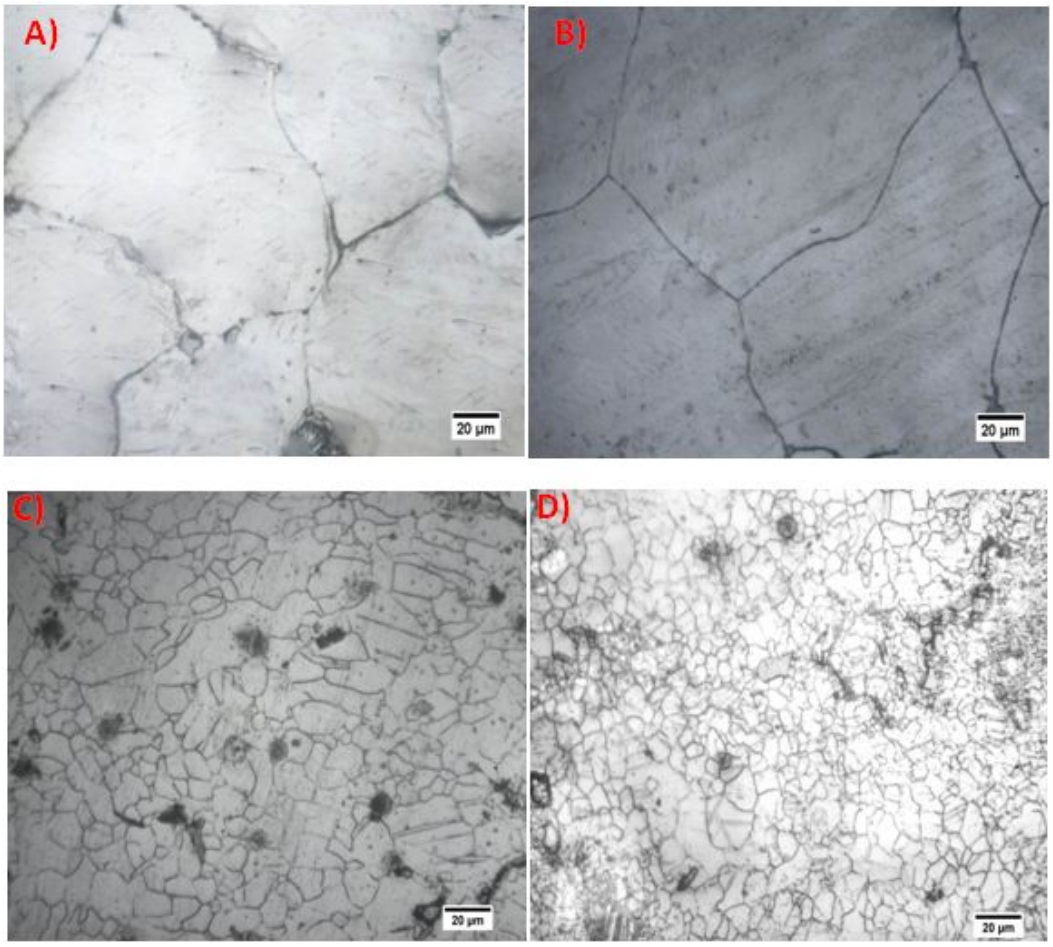

FIG.1: OPTICAL MICROGRAPHS OF A) AS-CAST B) AS-HOMOGENIZED C) AS-ROLLED (24% REDUCTION AND 3 PASSES) D) AS-ROLLED (64% REDUCTION AND 16 PASSES) AZ31 ALLOYS.

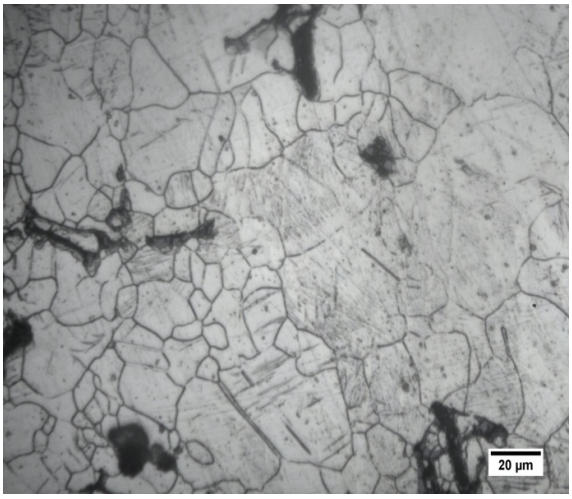

FIG.2: OPTICAL MICROGRAPHS OF AS-ROLLED (24% REDUCTION AND 3 PASSES) AZ31-0.7%CA ALLOYS.

3.2 SEM AND EDS ANALYSIS

Fig. 3 illustrates the microstructures of the as-homogenized AZ31 and AZ31-0.7%Ca alloys. It can be observed that both the microstructures consist of α-Mg Matrix and β-Mg17Al12 particles. However, microstructure in as-homogenized AZ31-0.7%Ca alloy, β-Mg17Al12 particles contain significant amount of calcium. Another important thing is that the size and number of particles in AZ31-0.7%Ca alloy are greater than AZ31 alloy due to the presence of calcium.

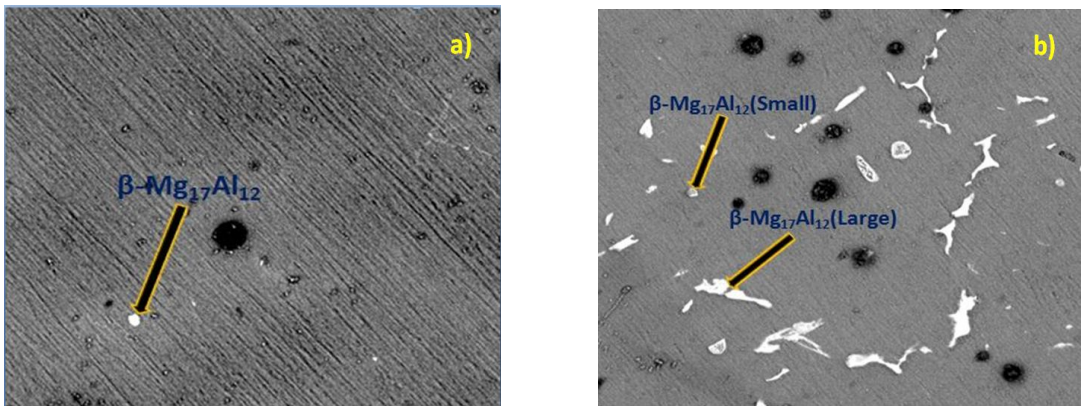

FIG.3: SEM MICROGRAPHS OF AS-HOMOGENIZED A) AZ31 B) AZ31-0.7%CA ALLOYS.

On the other hand, in SEM microstructure of AZ31-0.7%Ca alloy, usually two types of precipitates are observed and their compositions are different. In larger particles amount of calcium is three times higher than smaller particles.

Table 1: EDS Analysis (wt%) of Matrix and Particles Shown in Fig. 3

Materials	Phase	Al	Zn	Ca	Mg
AZ31	Matrix	4.86	1.45	-	93.69
	$Mg_{17}Al_{12}$	35.39	9.79	-	54.82
AZ31-0.7% Ca	Matrix	5.18	1.18	0.55	93.09
	$Mg_{17}Al_{12}$ (Large)	51.56	2.55	29.29	16.59
	$Mg_{17}Al_{12}$ (Small)	29.78	4.74	9.26	56.22

3.3 DSC ANALYSIS

Differential scanning calorimetry (DSC) analysis of both alloys is shown in Fig. 4. The first endothermic peak appeared at approximately 69°C for both alloys. The exothermic reaction of AZ31-0.7% Ca at approximately 140°C was larger in comparison with AZ31 alloy. Fig. 4(b) showed the temperatures of melting reaction at approximately 425°C when the alloy precipitates started after the addition of Ca.

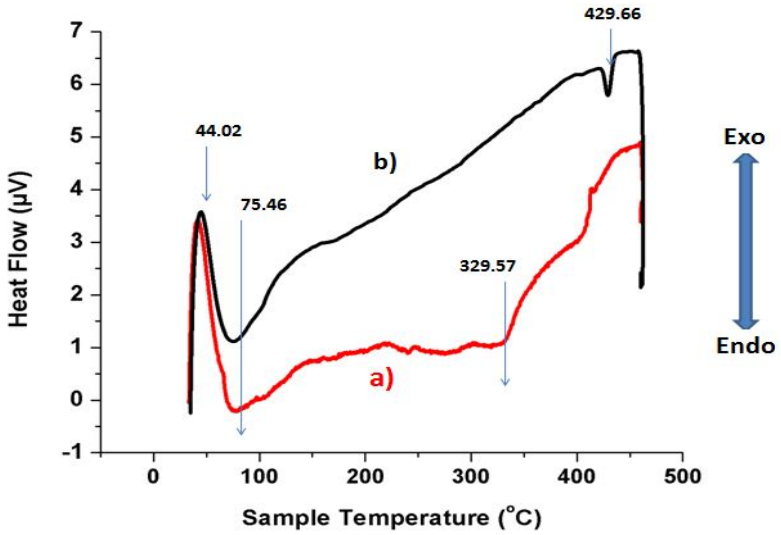

FIG.4: DSC HEATING CURVES OF A) AZ31 B) AZ31-0.7% CA ALLOYS

4. CONCLUSION

After homogenization of AZ31 alloy, grain size of that particular alloy had increased to a certain amount. More uniform and refined grains were observed with increasing amount of reduction and number of passes during hot rolling. However, bimodal grain size distribution was observed during high temperature deformation of AZ31-0.7%Ca alloy due to the presence of Ca- containing β-$Mg_{17}Al_{12}$ particles and the size and number of such particles in AZ31-0.7%Ca alloy are greater than AZ31 alloy.

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6. REFERENCES

[1] M. Yang, C. Duan, H. Li, T. Guo and J. Zhang, "Effects of minor Ca addition on as-cast microstructure and mechanical properties of Mg–4Y–1.2 Mn–1Zn (wt.%) magnesium alloy," Journal of Alloys and Compounds, vol. 574, pp.165-173, 2013.

[2] S.M. Masoudpanah and R. Mahmudi, "Effects Of Rare-Earth Elements And Ca Additions On The Microstructure And Mechanical Properties Of AZ31 Magnesium Alloy Processed By ECAP," Materials Science and Engineering, vol. A 526.1-2, pp. 22-30, 2009.

[3] G. Wu, Y. Fan, H. Gao, C. Zhai and Y.P. Zhu, "The effect of Ca and rare earth elements on the microstructure, mechanical properties and corrosion behavior of AZ91D," Materials Science and Engineering, vol. A 408(1), pp. 255-263, 2005.

[4] Maksoud, Ismael Abdel, H. Ahmed, and Johannes Rödel, "Investigation Of The Effect Of Strain Rate And Temperature On The Deformability And Microstructure Evolution Of AZ31 Magnesium Alloy," Materials Science and Engineering, vol. A 504.1-2, pp. 40-48, 2009.

[5] B.L. Mordike and Tü Ebert. "Magnesium: properties—applications—potential." Materials Science and Engineering, vol. A 302, no. 1, pp. 37-45, 2001.

[6] Stanford, Nicole, and Matthew R. Barnett. "Fine grained AZ31 produced by conventional thermo-mechanical processing." *Journal of Alloys and Compounds* 466, no. 1 (2008): 182-188.

[7] Kim, Woo Jin, and Hyo Tae Jeong. "Grain-size strengthening in equal-channel-angular-pressing processed AZ31 Mg alloys with a constant texture." *Materials transactions* 46, no. 2 (2005): 251-258.

[8] Eddahbi, M., J. A. Del Valle, María Teresa Pérez-Prado, and Oscar Antonio Ruano. "Comparison of the microstructure and thermal stability of an AZ31 alloy processed by ECAP and large strain hot rolling." *Materials Science and Engineering: A* 410 (2005): 308-311.

[9] Pérez-Prado, María Teresa, and O. A. Ruano. "Grain refinement of Mg–Al–Zn alloys via accumulative roll bonding." *Scripta materialia* 51, no. 11 (2004): 1093-1097.

[10] Yang, Q., and A. K. Ghosh. "Production of ultrafine-grain microstructure in Mg alloy by alternate biaxial reverse corrugation." *Acta Materialia* 54, no. 19 (2006): 5147-5158.

[11] Kawalla, Rudolf, and Alexei Stolnikov. "Deformation Behaviour and Microstructure Development of Magnesium AZ 31 Alloy During Hot and Semi-hot Deformation." *Advanced Engineering Materials* 6, no. 7 (2004): 525-529.

[12] Wu, Lu, Fusheng Pan, Mingbo Yang, and Renju Cheng. "An investigation of second phases in as-cast AZ31 magnesium alloys with different Sr contents." *Journal of Materials Science* 48, no. 16 (2013): 5456-5469.

[13] Zeng, Xiaoqin, Yingxin Wang, Wenjiang Ding, Alan A. Luo, and Anil K. Sachdev. "Effect of strontium on the microstructure, mechanical properties, and fracture behavior of AZ31 magnesium alloy." *Metallurgical and materials transactions A* 37, no. 4 (2006): 1333-1341.

[14] Wang, Yingxin, Xiaoqin Zeng, and Wenjiang Ding. "Effect of Al–4Ti–5B master alloy on the grain refinement of AZ31 magnesium alloy." *Scripta Materialia* 54, no. 2 (2006): 269-273.

[15] Barnett, M. R., Zohreh Keshavarz, and M. D. Nave. "Microstructural features of rolled Mg-3Al-1Zn." *Metallurgical and Materials Transactions A* 36, no. 7 (2005): 1697-1704.

[16] Del Valle, J. A., M. T. Pérez-Prado, and O. A. Ruano. "Accumulative roll bonding of a Mg-based AZ61 alloy." *Materials Science and Engineering: A*410 (2005): 353-357.

[17] A. Galiyev, R. Kaibyshev, and D. Voronin: *Proc. 6th Int. Conf. Magnesium Alloys and Their Applications*, K.U. Kainer, ed., Wiley-VCH, Weinheim, 2004, pp. 266-71

[18] Chang, Tien-Chan, Jian-Yi Wang, O. Chia-Ming, and Shyong Lee. "Grain refining of magnesium alloy AZ31 by rolling." *Journal of Materials Processing Technology* 140, no. 1 (2003): 588-591.