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## Effect of Microstructural Refinement on Tensile Properties of AZ80 Magnesium Alloy via Ca Addition and Extrusion Process

S. H. Allameh<sup>a</sup>, M. Emany<sup>a,\*</sup>, E. Maleki<sup>b</sup>, B. Pourbahari<sup>a</sup><sup>a</sup>*School of Metallurgy and Materials, College of Engineering, University of Tehran, Tehran, Iran*<sup>b</sup>*Iran University of Industries & Mines, Tehran, Iran*

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### Abstract

The microstructure and tensile properties of AZ80+X%Ca (X=0, 0.1, 0.5) magnesium alloy have investigated after applying extrusion process at 280 °C and 340 °C. Optical and scanning electron micrographs parallel to extrusion direction at 280 °C showed dynamically recrystallized grains. There were also initial grains elongated in extrusion direction in the AZ80+X%Ca alloy. Finer microstructures were observed by increasing calcium content due to the formation of some precipitates during grain growth. EDS analysis determined the newly formed precipitates as Al<sub>2</sub>Ca. The grain size was reduced from 90 μm to 9 μm by extrusion process in the sample with 0.5% Ca. At higher extrusion temperature (340 °C), similar microstructure was observed, except that the grain size was increased and there was no initial grains left in the structure anymore. From tensile testing, ultimate tensile strength (UTS) value was increased from 304 MPa to 329 MPa in extruded AZ80+0.5%Ca alloy at 280 °C.

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\* Corresponding author. Tel.: (+98-21) 82084083; fax: (+98-21) 82084083.

E-mail address: [ememy@ut.ac.ir](mailto:ememy@ut.ac.ir)

## 1. Introduction

Magnesium is the lightest structural metal which is a third lighter than aluminum. Magnesium alloys have attracted significant interests as a mass-saving replacement for steel and aluminum alloys. They have numerous applications in vehicles and electronic products because of their attractive features such as low density, high specific strength, and excellent shock resistance, Watarai (2006), Yi et al. (2007).

Magnesium high-pressure die castings are already being used in automobiles. Among magnesium alloys, AZ series contain about 90% of all magnesium cast products, Shang et al. (2011). Wrought magnesium products, however, only represent a small fraction of total magnesium consumption despite having the advantages of a higher strength and ductility than die-cast products. This can be directly ascribed to the high cost of extrusion for existing magnesium alloys when compared to aluminum alloys which results from comparatively slow extrusion speed of magnesium alloys, Beer (2012).

Grain size refinement is an effective way to improve strength and elongation of magnesium alloys simultaneously. One of the used methods to achieve this purpose is grain refinement through adding modifier elements to alloy, Liang et al. (2008). The addition of different modifiers such as rare earth elements, Meshinchi Asl et al. (2010) and Nd, Wang et al. (2012) has been investigated on AZ91 and AZ80 magnesium alloys. Calcium has also found to be effective to improve the compressive creep behavior of Mg-4Al-RE magnesium alloy, Yaocheng et al. (2014).

AZ80 magnesium alloy is a wrought commercial alloy and has attracted great attention of many researchers and scientists. However, it exhibits poor workability at room temperature because of the hexagonal close-packed crystal structure with only two active and independent base slip systems, Zhang et al. (2011), Tan et al. (2007). Fortunately, prismatic and pyramidal slip systems activated at high temperatures do facilitate dislocation creep, which is critical to ductility enhancement of Mg alloys with less-refined grains, and to production of complex components that cannot be economically formed at room temperature, Jun et al. (2013). So, high temperature and triaxial tension are the key parameters to increase the formability of AZ80 alloy which are prepared by hot extrusion process.

The aim of this work is to investigate the effect of Ca addition on the microstructure and tensile properties of extruded AZ80 alloy.

## 2. Experimental

All alloys were prepared in a graphite crucible with a thermal- barrier ceramic shield using an induction furnace. Commercial pure magnesium (99.95%) and aluminum (99.99%) ingots were melted in the crucible. The weight losses of Mg and Al were selected to be 10% and 3%, respectively. Manganese, zinc and calcium were added as Al - 20 wt% Mn, Mg - 50 wt% Zn and Mg - 20 wt% Ca alloys successively when the temperature of molten metal was about 850 °C. During the melting process, surface of the melt was shielded by a protective gas containing 95% CO<sub>2</sub> and 5% SF<sub>6</sub>. Finally, the melt was stirred and after defluxing, it was poured into a cylindrical permanent mold. Table 1 shows the compositions of as-cast alloys.

Cylindrical castings were cut into billets (30 mm in diameter and 30 mm in height) in order to fit into the extrusion container. These billets were preheated to 280 °C and 340 °C for 30 min and then extruded using a hydraulic press at a ram speed of 10 mm.s<sup>-1</sup> with the extrusion ratio of 12:1.

Table 1. Chemical compositions of experimental alloys.

Alloy	Al	Zn	Mn	Ca	Mg
AZ80	8.10	0.49	0.21	-	Bal.
AZ80-0.1%Ca	7.95	0.50	0.22	0.1	Bal.
AZ80-0.5%Ca	8.07	0.48	0.24	0.5	Bal.

Extrusion process was carried out applying graphite based oil between metal, die and container. All samples were quenched in water immediately after extrusion process. Round tensile samples were machined along the extrusion direction according to ASTM: E8/E8 M-11. The sketch of a tensile test specimen is seen in Fig. 1. Tensile tests were

carried out using a computerized testing machine (SANTAM STM-20) at a strain rate of  $0.1 \text{ mm}\cdot\text{min}^{-1}$  at room temperature.

The microstructures were observed by optical microscopy (OM) and scanning electron microscopy (SEM). A solution of 70 mL ethanol, 4 g picric, 10 mL acetic acid and 10 mL distilled water was used to reveal the extruded microstructure. The phase composition of different phases was characterized by energy-dispersive X-ray spectroscopy (EDS).

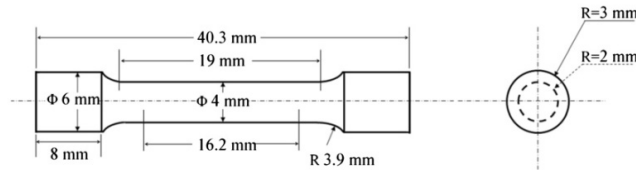


Fig. 1. Schematic of tensile test samples.

### 3. Results and Discussion

#### 3.1. Microstructure

Figure 2 shows the microstructure of extruded alloys at  $280 \text{ }^\circ\text{C}$  and  $340 \text{ }^\circ\text{C}$ . As it can be seen, the grains become finer with increase of Ca content. With addition of 0.5%Ca, the grain size of extruded alloy decreases from  $14 \text{ }\mu\text{m}$  to  $9 \text{ }\mu\text{m}$  at  $280 \text{ }^\circ\text{C}$  and from  $22 \text{ }\mu\text{m}$  to  $11 \text{ }\mu\text{m}$  at  $340 \text{ }^\circ\text{C}$ . The initial grain size for the samples with 0, 0.1 and 0.5% Ca was  $110 \text{ }\mu\text{m}$ ,  $97 \text{ }\mu\text{m}$  and  $90 \text{ }\mu\text{m}$  respectively. During the extrusion of some metals such as aluminum alloys, a fibrous microstructure (consisting of original as-cast grains elongated in the extrusion direction) is commonly developed due to the operation of dynamic recovery (DRV). In magnesium alloys, however, dynamic recrystallization (DRX) operates during hot deformation and a fine-grained microstructure is progressively developed, Beer (2012).

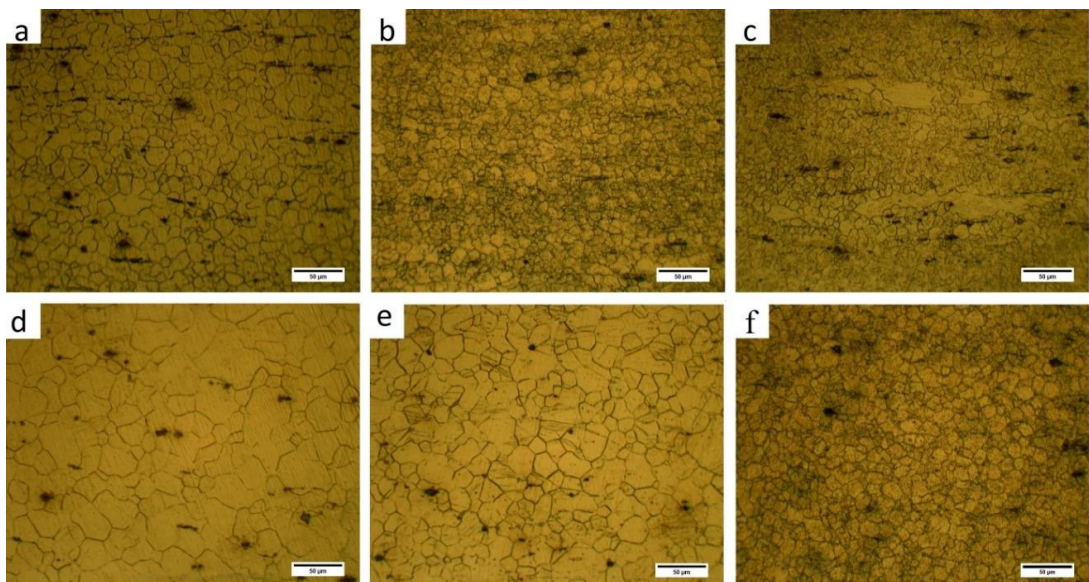


Fig. 2. Optical micrographs of extruded AZ80 alloy at  $280 \text{ }^\circ\text{C}$  with (a) 0% Ca; (b) 0.1% Ca; (c) 0.5% Ca and extruded at  $340 \text{ }^\circ\text{C}$  with (d) 0% Ca; (e) 0.1% Ca; (f) 0.5% Ca.

The microstructure of extruded Ca added AZ80 alloys also show refined precipitates mainly distributed at grain boundaries which clarifies the pinning effect of precipitates for the them. The mentioned grain refinement is the direct effect of this pinning effect. There is a much fraction of precipitates at 280 °C as a result of less solubility of alloying elements in comparison with 340 °C (fig. 3c). This figure also shows elongated initial grains in addition to dynamically recrystallized grains. This shows the effect of newly formed precipitates in hindering the movement of grain boundaries during recrystallization which prevents new grains from consuming initial grains.

Figure 3 shows the SEM images of extruded AZ80 alloy without and with 0.5% Ca at 280 °C. Table 2 represents the EDS analysis of the corresponding points on Fig. 3. Points A and C indicate the magnesium solid solution as the base alloy. The chemical composition of points B and D corresponds to  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> and Al<sub>2</sub>Ca respectively. Al<sub>2</sub>Ca is an effective compound for entangling of grain boundaries. Indeed, Al<sub>2</sub>Ca has a more preventing effect than  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> for dynamically recrystallized grains to grow and the grain boundaries are not free to move simply within the original grains. Thus a finer dynamically recrystallized structure is obtained in comparison with the base alloy. The larger size of Al<sub>2</sub>Ca particles than that of  $\beta$ -Mg<sub>17</sub>Al<sub>12</sub> particles after extrusion process also indicates their higher hardness and rigidity.

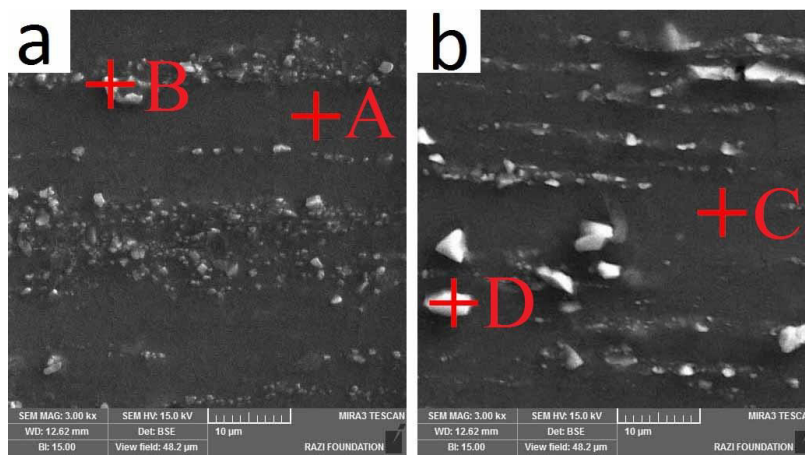


Fig. 3. SEM images of extruded AZ80 alloy at 280 °C with (a) 0% Ca; (b) 0.5% Ca.

Table 2. Chemical compositions of several regions in Fig. 3 identified by EDS.

Locations	A	B	C	D
Mg (at.%)	87.72	73.10	92.44	15.73
Al (at.%)	11.62	25.83	7.13	55.70
Ca (at.%)	0.03	0.04	0.10	27.63
Mn (at.%)	0.10	0.10	0.09	0.15
Zn (at.%)	0.52	0.93	0.37	0.79
	100.00	100.00	100.00	100.00

### 3.2. Tensile properties

The summary of tensile experiments is shown in Table 2. The UTS and elongation values show an incremental trend with Ca content. This increase is more distinct at 280 °C which shows UTS value of 329 MPa and elongation of 19%. This is because of grain refinement and production of more fine precipitates with a higher hardness in the structure. Theoretically, both Al<sub>2</sub>Ca and Mg<sub>2</sub>Ca could be formed in the alloy, but Al<sub>2</sub>Ca with melting point of 1079 °C is more effective in strengthening than Mg<sub>2</sub>Ca with melting point of 715 °C.

Table 3. Tensile properties of extruded AZ80 alloy at 280 °C and 340 °C with different Ca contents.

Extrusion Temperature	280 °C		340 °C	
Alloy	UTS(MPa)	Elongation (%)	UTS(MPa)	Elongation (%)
AZ80	304±7	12±2	305±6	11±4
AZ80-0.1%Ca	320±4	15±3	311±3	12±5
AZ80-0.5%Ca	329±3	19±5	312±2	8±3

### 4. Conclusion

- With addition of 0.5%Ca, the grain size of extruded AZ80 alloy decreased from 14 μm to 9 μm at 280 °C and from 22 μm to 11 μm at 340 °C.
- The microstructure of Ca added AZ80 alloy after extrusion at 280 °C showed refined precipitates distributed at grain boundaries which clarified the pinning effect of precipitates for the them. There was also a much fraction of precipitates at 280 °C as a result of less solubility of alloying elements in magnesium in comparison with 340 °C.
- There were elongated initial grains in addition to dynamically recrystallized grains in AZ80-0.5%Ca alloy which showed the effect of newly formed precipitates in hindering the movement of grain boundaries during recrystallization. EDS analysis determined the strengthening phases as β-Mg<sub>17</sub>Al<sub>12</sub> and Al<sub>2</sub>Ca in the base alloy and Ca-containing alloy respectively. Al<sub>2</sub>Ca is more effective for entangling of grain boundaries during recrystallization than β-Mg<sub>17</sub>Al<sub>12</sub>.
- Tensile testing revealed that the UTS and elongation values of AZ80 alloy extruded at 280 °C and 340 °C increased with Ca content. The UTS of extruded AZ80 alloy at 280 °C increased from 304 MPa to 329 MPa by addition of 0.5%Ca.

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