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The Effects of Yttrium Element on Microstructure and Mechanical Properties of Mg-5 mass%Al-3 mass%Ca Based Alloys Fabricated by Gravity Casting and Extrusion Process

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The as-cast microstructure of Mg-5Al-3Ca-xY alloy consists of dendritic α-Mg matrix, $(Mg, Al)_2Ca$ eutectic phase and Al_2Y intermetallic compounds. These two kind of $(Mg, Al)_2Ca$ compounds were observed: coarse irregular-shape structure at grain boundary and fine needle-shape structure in the α-Mg matrix grain. This $(Mg, Al)_2Ca$ phase of the extruded alloys was elongated to extrusion direction and size of this phase was refined comparing with that of as-cast alloys because of severe deformation during hot extrusion. Maximum yield and ultimate strength value of extruded alloys was 326 and 331 MPa at Mg-5Al-3Ca-3Y alloy, respectively. From these results, it is conclusively demonstrated that Y additions on Mg-5Al-3Ca alloy have more effect to improve mechanical properties. [doi:10.2320/matertrans.MC200777]

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Keywords: *magnesium-aluminum-calcium alloys, yttrium (Y), rare earth element, casting, extrusion*

1. Introduction

Magnesium alloys are the lightest structural and hence suitable for application in the automotive industry, which has increased attention to the vehicle weight and fuel economy. Magnesium alloys based on the Mg-Al system have been studied extensively for use in vehicle due to the weight savings they provide and also for their excellent castability.[1,2] The most widely used magnesium alloys is AZ91, in which Al is the major alloying element. However, commercial magnesium alloys such as AZ91 and AM50 are limited because of poor creep resistance and poor mechanical properties at elevated temperature of 120°C.[2–4] The cause of this phenomenon is grain boundary phase $Mg_{17}Al_{12}$. This phase softens at high temperature. Therefore, it is important to reduce the amount of $Mg_{17}Al_{12}$ phase and introduce thermally stable precipitates at grain boundaries as well as in the grain interior by adding proper alloying elements. It is well known that alloys of Mg-Al-Ca systems may provide significant improvement in elevated temperature properties due to reduction of volume fraction of $Mg_{17}Al_{12}$ phase.[5–10] Moreover, it has been reported that the content variation of rare earth elements (RE) and element Y in the magnesium alloys can influence the mechanical properties greatly,[11–16] which is mainly ascribed to the Orowan mechanism.[17,18] And, high performance Mg-Zn based alloys containing Y have been developed by Kawamura *et al.*[19] This alloy with Y addition, prepared by extrusion process, reveals excellent mechanical properties including maximum tensile yield strength about 600 MPa and elongation about 5% at room temperature.

In this research, Mg-Al-Ca based alloys with yttrium (Y) were produced by gravity casting and extrusion process and effects on Nd additions on microstructure and mechanical properties of Mg-Al-Ca based alloys were investigated.

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Table 1 The nominal comopisitions of the studied alloys (mass%).

Al	Ca	Y	Mg
5	3	—	Bal.
5	3	1	Bal.
5	3	2	Bal.
5	3	3	Bal.

2. Experimental Procedures

The nominal compositions of the four studied alloys are Mg-5Al-3Ca, Mg-5Al-3Ca-1Y, Mg-5Al-3Ca-2Y and Mg-5Al-3Ca-3Y as listed Table 1. Commercially pure Mg, Al and Ca (> 99.9%) were used to prepare these alloys and yttrium (Y) was added as Mg-20 mass%Y master alloy. The Mg-5Al-3Ca-xY ($x = 0$, 1, 2 and 3 mass%) alloys were fabricated under SF_6 and CO_2 atmosphere in a steel crucible. The alloy melts were cast into steel mold (D50 mm × H100 mm), which was heated at 200°C, at pouring temperature of 750°C. As-cast alloys was held for 1 h at 380°C and hot extruded into a rod of 12 mm diameter with a reduction ratio of 20 : 1. The extrusion speed is 5 mm/s and temperature of the extrusion container and die is 380°C. Microstructures of as-cast and extruded alloys were examined by optical microscope (OM; Nikon) and scanning electron microscope (SEM; JSM7000F) equipped with energy-dispersive X-ray spectrometer (EDS) system. Samples were cut and ground mechanically to a mirror like surface using abrasive papers and diamond pastes, and surfaces were then etched by immersing for 15–20 s in a solution 10 ml acetic acid, 4.2 g picric acid, 10 ml distilled water and 70 ml ethanol. Tensile tests were performed using universal material test machine (SHIMAZU AG-IS) at room temperature. All the tests were carried out at a initial strain rate of $1.0 \times 10^{-3}\,S^{-1}$.

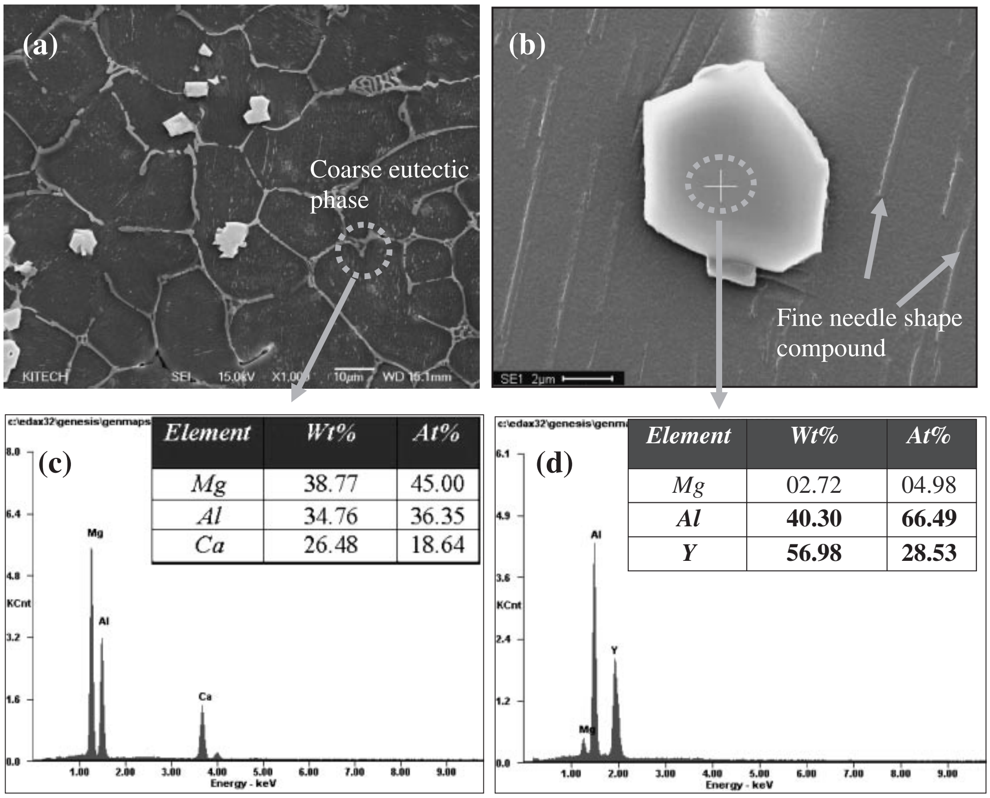

Fig. 1 SEM-EDS analysis of as-cast Mg-5Al-3Ca-3Y alloys; (a), (b) SEM image, (c) EDS result of eutectic phase and (d) EDS result of Y containing intermetallic compounds.

3. Results and Discussions

3.1 Microstructure of the as-cast alloys

Figure 1 shows SEM images and EDS analysis of Mg-5Al-3Ca-3Y alloy. Microstructure of as-cast Mg-Al-Ca-Y alloy consists of dendritic α-Mg matrix, eutectic phase and Al-Y rich intermetallic compounds as shown in Fig. 1(a). These eutectic phases were identified to (Mg, $Al)_2Ca$ phase by SEM-EDX analysis. For the eutectic phase, result of EDX shows that it is mainly composed of Al, Mg and Ca elements and atomic ratio of $Mg : Al : Ca = 2.4 : 1.9 : 1$, which is well in agreement with the stoichiometric ratio 2 : 2 : 1 of (Mg, $Al)_2Ca$. The previous work carried out by Szuki *et al.* reported[9,10] that both Mg_2Ca (C14) and (Mg, $Al)_2Ca$ (C36) were observed in Mg-Al-Ca based alloys. The crystal structure of C14 and C36 are similar, both hexagonal, and lattice parameter of C36 is twice of that of C14 along [0001] direction. However, no C14 structure, which was found in some Mg-Al-Ca alloys, has been observed in the present investigation. Higher magnification SEM observation exhibits fine needle shape particles distributing in the α-Mg grains, as shown in Fig. 1(b). In the recent, B. Jing *et al.* reported that these fine intermetallic compounds in α-Mg matrix were close to that of (Mg, $Al)_2Ca$ phases.[7] As shown in Fig. 1(b), the coarse intermetallic compounds with bright contrast were identified to Al_2Y phase by SEM-EDX analysis (Fig. (d)). It is found that atomic ratio of Al and Y in phase was about 2.3 : 1, which is well in agreement with the stoichiometric ratio 2 : 1. These Al_2Y intermetallic compounds with bright contrast were observed at both grain boundary and α-Mg matrix as shown in Fig. 1(a).

Influence of Y element on microstructure of as-cast Mg-Al-Ca-Y alloys was observed as shown in Fig. 2. All samples show a similar dendritic structure of varying size. With increasing of Y addition, grain size is sharply refined.

3.2 Microstructure of the as-extruded alloys

The optical micrographs of the as-extruded Mg-5Al-3Ca-xY alloys taken from the longitudinal direction are shown in Fig. 3. It is apparent that the band structure of eutectic phases with dark grey contrast was arranged approximately parallel to the extrusion direction. The dispersoids were fragmented into eutectic phase particles by means of extrusion. The agglomerated particles were tended to align parallel to the extrusion direction. The eutectic particle size uniform and the size was decreased than that of the as-cast alloys. It was demonstrated that that the extrusion process is useful to break down the cast structure of the ingot. In microstructure of Y containing alloys, the interval between the eutectic intermetallic compounds toward the transverse direction of extrusion was lower than that of the as-extruded Mg-5Al-3Ca alloys. It shows that grain size decreases with addition of Y element. For Mg-5Al-3Ca alloy, the average grain size is 4.8 μm, whereas the average grain size of Y containing alloys is 3.6, 2.8 and 2.9 μm with increasing of Y additions, respectively. Specially, comparing with as-cast alloys, grain size of Y containing alloys was greatly refined because of dynamic recrystallization during hot extrusion.

SEM-BEI observations demonstrate the detailed microstructure such as eutectic phase and Al-Y rich intermetallic compound behavior during extrusion as shown in Fig. 4. Eutectic phase phases distributing at grain boundaries in as-

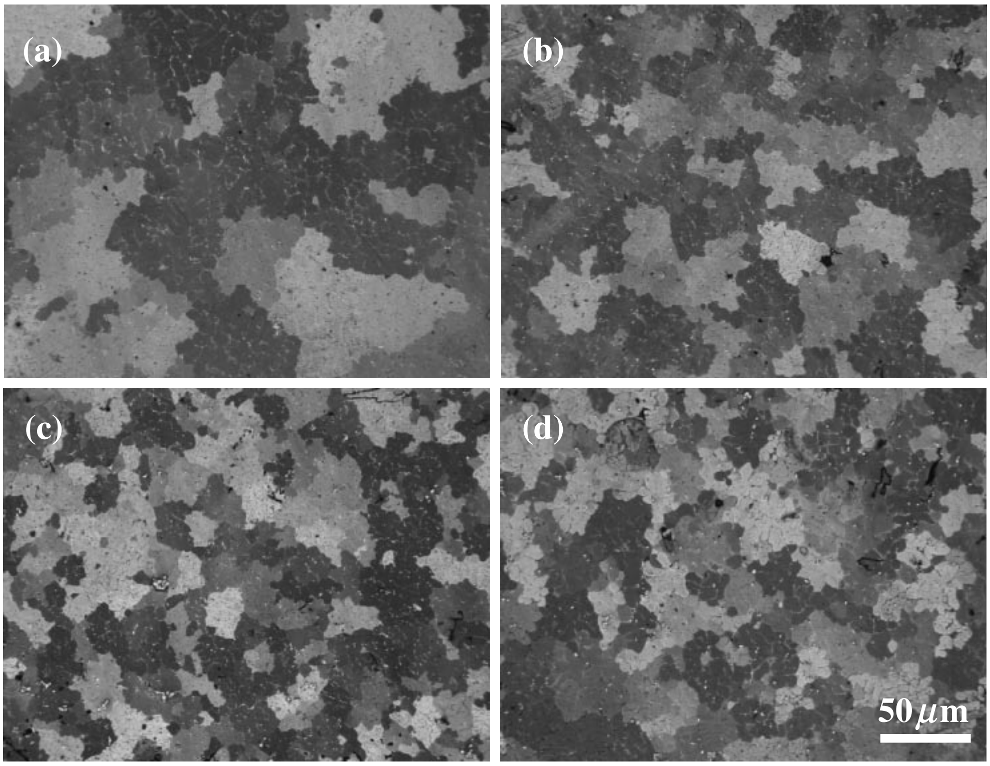

Fig. 2 Macrostructure of as-cast Mg-5Al-3Ca-xY alloys; $x = 0$ (a), 1 (b), 2 (c) and 3 mass% (d).

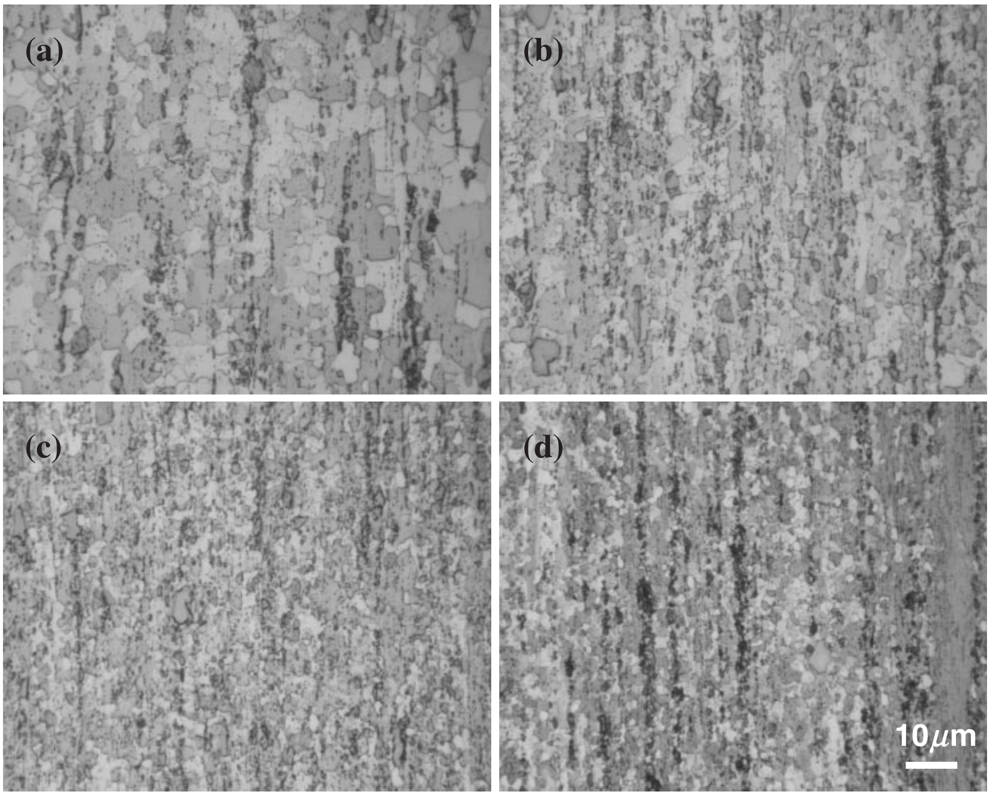

Fig. 3 Optical micrographs of the as-extruded Mg-5Al-3Ca-xY alloys: $x = 0$ (a), 1 (b), 2 (c) and 3 mass% (d).

cast samples have been converted band structure along the extruded direction. It can be seen that the eutectic phase was broken into small particles and elongated after hot extrusion. The transition of eutectic phase morpholorgy indicates that (Mg, $Al)_2Ca$ phases are brittle and easily broken during extrusion process. While, comparing with (Mg, $Al)_2Ca$ eutectic phase broken during extrusion, Al_2Y intermetallic compounds were not crushed. The eutectic phase and Al_2Y

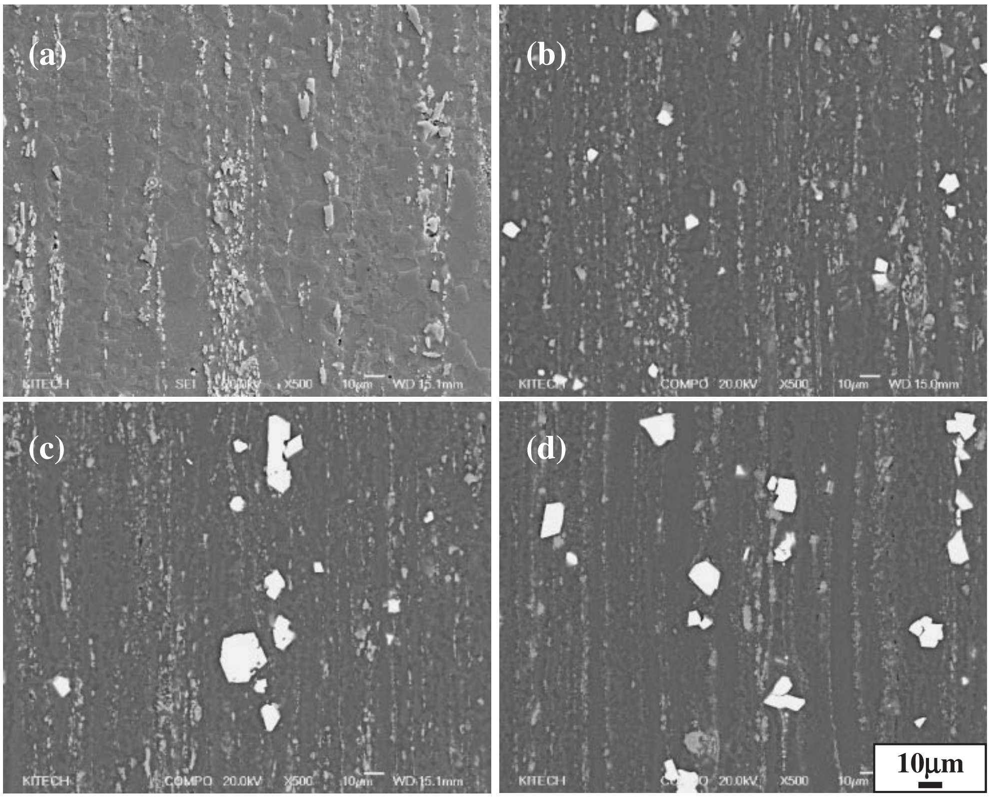

Fig. 4 SEM images of the as-extruded Mg-5Al-3Ca-xY alloys: $x = 0$ (a), 1 (b), 2 (c) and 3 mass% (d).

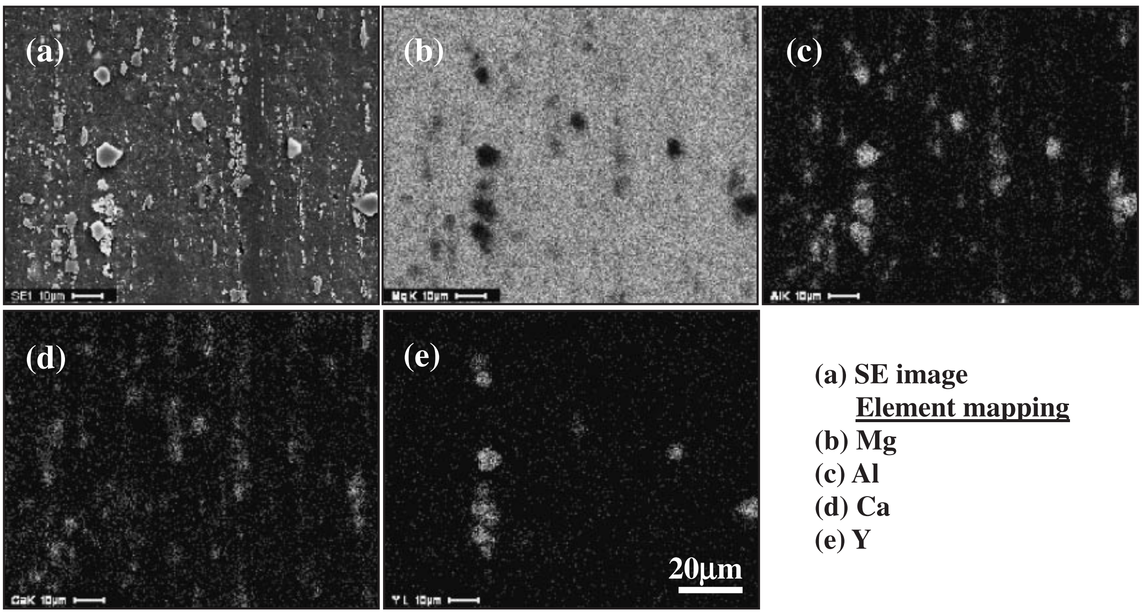

Fig. 5 SEM-EDS element mapping images of the as-extruded Mg-5Al-3Ca-2Y alloy.

intermetallic compounds of as-extruded alloys have same composition as that in the as-cast samples. With increasing Y addition, volume faction of Al_2Y intermetallic compounds is increased and size of this phase is coarsened.

In order to analyze phase distribution during extrusion, EDX element mapping was conducted as shown in Fig. 5. Eutectic phase regions were observed Mg, Al and Ca elements and the bright intermetallic compounds in SEM image were exhibited mainly Al and Y elements.

Higher magnification SEM observation exhibits fine needle shape particles distributing in the α-Mg grains after extrusion, as shown in Fig. 6. The fine needle shape compound of the as-cast alloys observed in Fig. 1(b) was elongated to extrusion direction and the length to needle shape of this phase was finer than that of the as-cast alloys because of severe deformation by hot extrusion. As shown in Fig. 6(b), the thickness of fine eutectic compound was less than 100 nm and these phases were homogeneously dis-

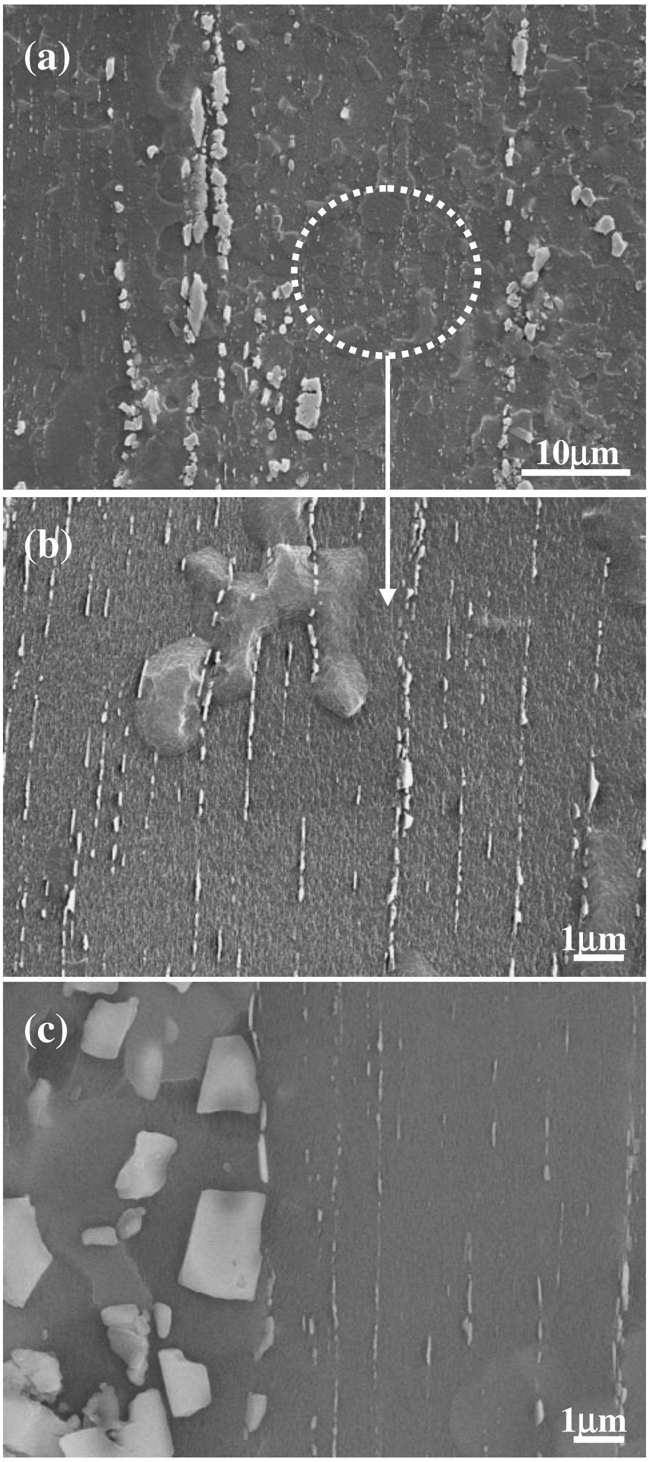

Fig. 6 SEM images in high magnification of the as-extruded alloy: (a) lower magnification, (b) higher magnification and (c) the backscattered image (BEI).

tributed in α-matrix. In the backscattered SEM image (Fig. 6(c), the contrast of fine needle shape compound is similar to that of coarse eutectic phase.

3.3 Mechanical properties of the as-extruded alloys

Figure 7 shows tensile properties of the as-extruded Mg-5Al-3Ca-xY at room temperature. With increasing of addition of Y addition, yield strength and ultimate tensile strength was increased and maximum value of yield strength and ultimate strength was 326 MPa and 331 MPa at Mg-5Al-3Ca-3Y alloy, respectively. The yield strength and ultimate tensile strength of the as-extruded alloys was improved by addition of Y. The excellent mechanical properties of hot extruded Mg-Al-Ca-Y alloys may be due to the following factor. It refines the grain size and improves the strength at room temperature via Hall-Petch behavior. Moreover, some researchers reported that grain can be refined by dynamic recrystallization in magnesium alloys, and the mechanical properties are improved by grain refinement.[20,21] However, the elongation of as-extrude Mg-Al-Ca-Y alloys after hot extrusion decreased in spite of the microstructure being refined as Y content increased. The effect of Al_2Y intermetallic compounds on elongation seems to depend on it size and volume fraction. With increasing of Y additions, the volume fraction and size of Al_2Y intermetallic compounds was increased sharply and their cutting effect on the matrix precipitates the initiation or propagation of cracks. These large particles may become the crack source during tensile test, in consistency with the decrease of elongation of as-extruded Mg-5Al-3Ca-3Y alloy.

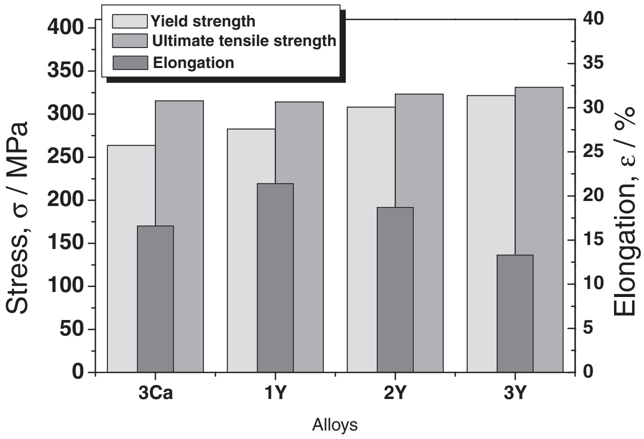

Fig. 7 Tensile properties of the as-extruded Mg-5Al-3Ca-xY alloys at room temperature.

Cleavage fracture, quasi-cleavage fracture and inner-granular fracture are the main fracture modes of magnesium alloys. Since most magnesium alloys have a hexagonal crystal structure and fewer slip systems, two or more slip systems are scarcely activated at the same time. However, dimples can be formed only by the operations two or more slip. Therefore, ductile fracture, characteristic of dimples, is not the main fracture mode of magnesium alloys. To research the influence of Y additions on the fracture characteristics, secondary electron imaging (SEI) and back-scatter electron imaging (BEI) of the tensile fracture were sown in Figs. 8 and 9, respectively. In the SEM fractographs (Fig. 8), fine scale dimple, cleavage facet sand cracks can be clearly observed. As shown in Fig. 8(a), fine scale dimples and cracks were observed in the as-extruded Mg-5Al-3Ca alloy. As Y addition was increased, the cleavage planes on the fracture surfaces were increased gradually, fine scale dimples were decreased obviously. Therefore, it indicates that with Y content increasing, the failure modes of the tensile samples change from ductile failure to fragile failure. In the fracture observation of Y containing alloys, some

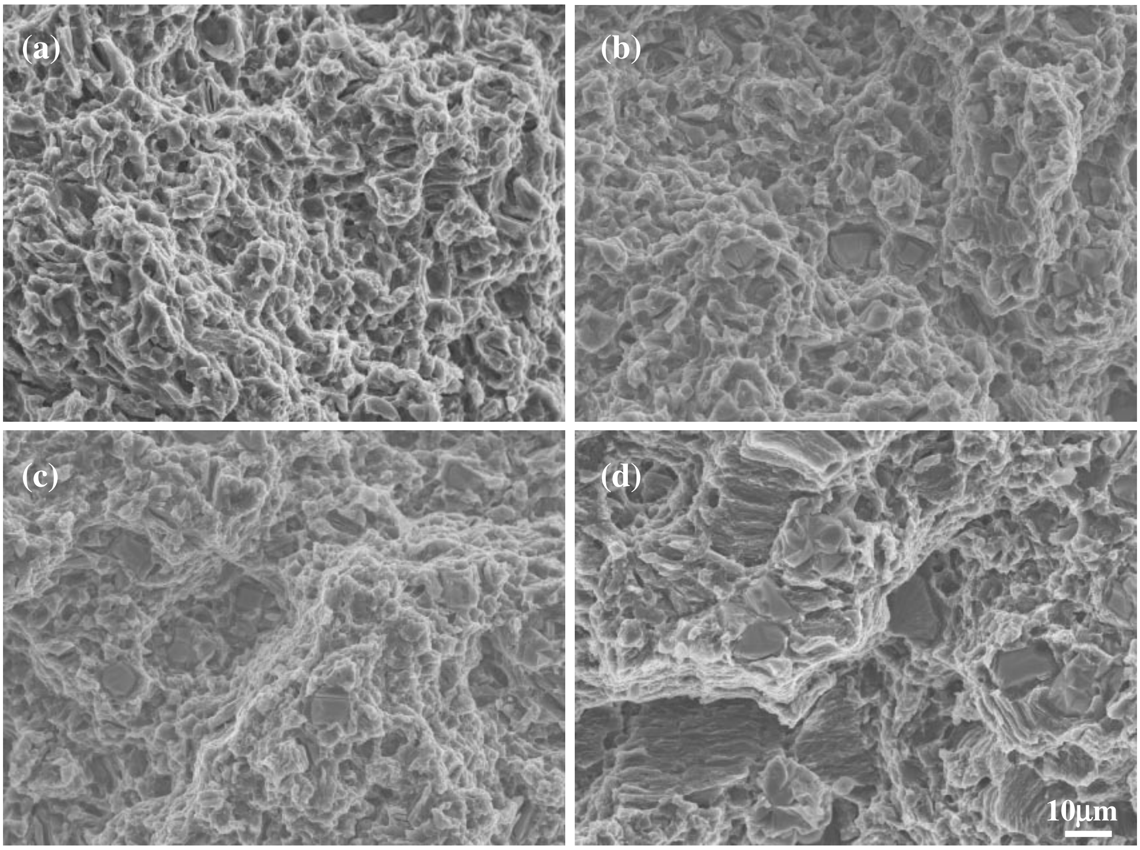

Fig. 8 SEM fracture images of the as-extruded Mg-5Al-3Ca-xY alloys obtained from the tensile test at room temperature: $x = 0$ (a), 1 (b), 2 (c) and 3 mass% (d).

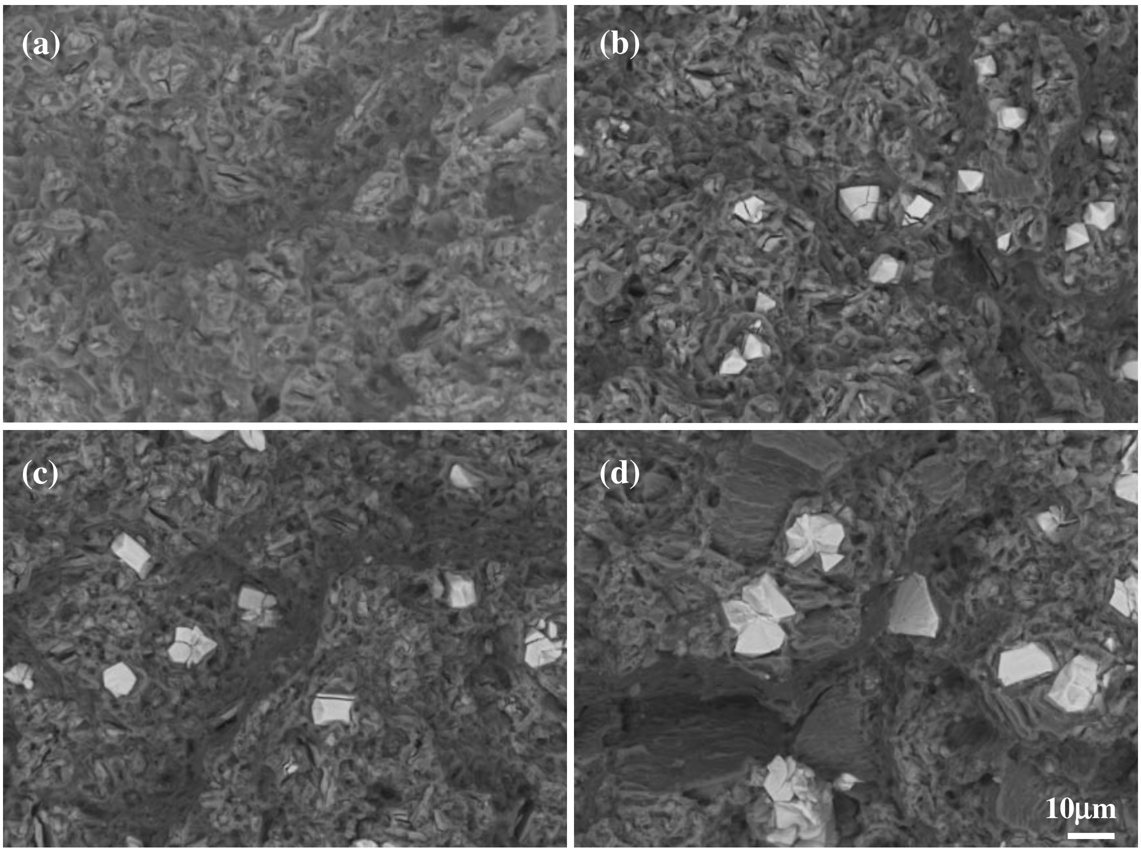

Fig. 9 The backscaater SEM fracture images of the as-extruded Mg-5Al-3Ca-xY alloys obtained from the tensile test at room temperature: $x = 0$ (a), 1 (b), 2 (c) and 3 mass% (d).

coarse Al_2Y phase particles can be observed lying in the larger dimple as shown in Fig. 8(b), (c) and (d). It is indicates that Al_2Y intermetallic compounds bring to the cutting effects on α-Mg matrix and finally become the origin of the microcracks.

Figure 9 shows backscatter electron images of the fracture surface. It also shows that Y contents increasing, the quantity and size of Al_2Y intermetallic compounds increase obviously. Most of the microcracks originated at the interface between the hard Al_2Y intermetallic compounds and the matrix due to severe strains. Some of the cracks were observed in coarse eutectic phase and Al_2Y intermetallic compounds. Therefore, coarse $(Mg, Al)_2Ca$ eutectic lumps in the some grain boundary area is regarded as an adverse factor to tensile property of Mg-5Al-3Ca-3Y alloy, this accompanied with cutting effect of Al_2Y intermetallic compounds contributes to decreased ductility and strength, regardless of high Y content.

4. Conclusions

In this research, effects of Y addition on microstructure and mechanical properties of Mg-5Al-3Ca based alloys fabricated by gravity casting and hot extrusion process have been investigated. The results are concluded as follows:

(1) Y addition to Mg-5Al-3Ca based alloys results in the formation Al_2Y intermetallic compounds at grain boundarys and α-Mg matrix grains.

(2) Microstructure of as-cast alloys contains dendritic structure of varying size and average grain size was greatly refined with increasing of Y addition.

(3) In the microstructure of the as-cast alloys, two kinds of $(Mg, Al)_2Ca$ compounds were obserbed; coarse irregular-shape structure at grain boundary and fine needle-shape structure in the α-Mg matrix grain.

(4) This eutectic phase of the extruded alloys was elongated to extrusion direction and crushed into small bloks because of severe deformation during hot extrusion.

(5) With increasing of addition of Y addition, yield strength and ultimate tensile strength was increased and maximum value of yield strength and ultimate strength was 326 MPa and 331 MPa at Mg-5Al-3Ca-3Y alloy, respectively.

Acknowlegements

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