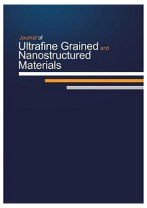

Journal of Ultrafine Grained and Nanostructured Materials
https://jufgnsm.ut.ac.ir
Vol. 53, No.2, December 2020, pp. 177-182
Print ISSN: 2423-6845 Online ISSN: 2423-6837
DOI: 10.22059/jufgnsm.2020.02.09

Microstructural refinement and tensile properties enhancement of the Mg-9Al-1Zn alloy after hot extrusion

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Recieved: 16 September 2020; Accepted: 9 November 2020
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ABSTRACT

The impact of hot extrusion process on the grain refinement and tensile properties for the rare earth elements (RE) and strontium (Sr)-containing Mg-9Al-1Zn alloys was studied. Based on the microstructural analysis, the grain size values reduced after the hot extrusion process, compared to the cast alloys. Moreover, the tensile properties of the extruded alloys were remarkably higher than the cast alloys. These extraordinary results were related to the grain refinement by the dynamic recrystallization phenomenon, and fragmentation and distribution of the intermetallics. Moreover, the RE/Sr additions improved the grain size reduction, owing to the presence of fragmented $Al_{11}RE_3$ intermetallics.

***Keywords**: Mg alloys; Hot extrusion; Mechanical properties; Microstructure; Grain refinement.*

1. Introduction

Magnesium is the lightest commercially-used alloy in the automobile industry and attracted so much attention due to its desirable features such as low density and high specific strength [1-3]. Mg alloys have already found many applications in power trains, steering wheels, instrument panels, seat framework, and steering columns [4]. Among different sorts of Mg alloys, the AZ91 alloy is known to be one of the most beneficial alloys in the industry, because of its excellent combination of die castability, mechanical, and corrosion properties [5]. However, due to semi-continuous networks of $Mg_{17}Al_{12}$ intermetallics, the tensile properties, especially the ductility of this alloy is not high enough and needs to be further improved [5]. Adding alloying elements [6,7] and implementing various thermomechanical processes [8,9] are the most practical means to achieve a refined microstructure and hence ameliorated mechanical properties. Recent research studies investigated the effects of various alloying elements such as Li [10,11], Pb [12,13], B [14,15], Bi [16], Ti [17], Sr [17,18], Ce [6,19] on the microstructure refinement and enhancement of the tensile properties of the AZ91 alloy. Among them, RE elements have been recognized for their profound influences on the texture development, grain refinement during the thermo-mechanical processing, and improvement of tensile properties [20]. Moreover, the synergetic effects of RE elements with other alloying elements were studied in recent years [20-22]. Zhang et al. [22] demonstrated that the combined addition of Ca and Ce/La mischmetal resulted in more effective grain refinement and better tensile properties than those of separate additions. Adding 0.2 wt.% Sr refined the grain structure, decreased the amounts of the β phase, and improved the tensile properties of the AZ91-0.5RE alloy [20].

On the other hand, the thermo-mechanical methods are used to achieve a fine grain structure [23,24]. It has been well-known that grain size plays a remarkable role in the improvement of tensile properties, especially for the Mg alloys with the HCP structure [23]. Furthermore, thermo-mechanical processes reduced casting defects such as porosity and shrinkage [25]. Moreover, the semi-continuous networks of cast alloys are fragmented and distributed during thermo-mechanical processing by the existence of high temperature and pressure. Former research studies [26,27] revealed that the intermetallics promoted the dynamic recrystallization (DRX) via particles stimulated nucleation (PSN) during the hot extrusion process. On the other hand, the coarse particles have been fragmented into finer dispersed intermetallics, which play an important role in hindering dislocation motions and grain boundary sliding at elevated temperatures [27].

In our previous study, the simultaneous use of RE/Sr additions on as-cast microstructure and mechanical properties of Mg-9Al-1Zn alloy was investigated [17]. In that study, the semi-continuous network of the $Mg_{17}Al_{12}$ intermetallics was fragmented and changed into a spherical shape. Furthermore, the mechanical properties of these alloys significantly improved. This work aims to investigate the hot extrusion process on the grain refinement and tensile properties of the Mg-9Al-1Zn-RE/Sr alloys.

2. Experimental

The pure Mg (99.97%), Al (99.95%), Al-50 wt% Zn, Al-10 wt% Sr, and Ce-rich mischmetal (48.7Ce-26.4La-19.6Nd-5.3Pr wt% [17]) master alloys were used and melted in an induction furnace with a graphite-ceramic crucible under the protection of 95% CO_2 and 5% SF_6 gas. At 750 °C, the prepared molten alloys were poured into a preheated mold at 250 °C with a height of 45 mm and an inner diameter of 34 mm. The nominal chemical compositions of the studied alloys are ordered in Table 1. After that, the homogenization heat treatment at 270 °C for 2 h followed by 415 °C for 18 h, and the subsequent air cooling was performed [28,29]. The hot extrusion process was conducted at 420 °C and with the extrusion ratio of 12:1. For microstructure analysis, the prepared samples were etched by acetic Picral solution (10 ml acetic acid + 4.2 g picric acid +10 ml H_2O + 10 ml ethanol). Then they are characterized by an optical microscope (OM) and FEI QUANTA 450 SEM equipped with Bruker QUANTAX XFlash 6 EDS detector. The room temperature tensile test samples were prepared according to the ASTM E8-04 standard and tested at a strain rate of 10^{-3} s^{-1}. The tensile tests of as-cast and as-extruded alloys were repeated twice to ensure the reproducibility of the results.

3. Results and discussion

Figures 1(a)-(d) explain the optical and SEM micrographs of the cast Mg-9Al-1Zn alloy with different amounts of RE/Sr additions. Figures 1(a) and (a1) represent the presence of the α-Mg phase surrounded by the dark and semi-continuous networks of β-$Mg_{17}Al_{12}$ intermetallics. By adding RE/Sr additions, the connection of β intermetallics reduced, and their morphology changed to a more spheroid form (Figures 1(b), (b1), (c), (c1), (d), and (d1)). Moreover, increasing the RE/Sr ratio results in the appearance of new intermetallics with different morphologies and distribution, which can be related to the Al_4Sr and $Al_{11}RE_3$ intermetallics [17]. Based on our previous investigation, the Al_4Sr and $Al_{11}RE_3$ compounds were intergranular and needle-like intermetallics, respectively [17]. Moreover, with more RE/Sr additions, the volume fraction of Al_4Sr and $Al_{11}RE_3$ intermetallics decreased and increased, respectively. The dendrite arm spacing (DAS) of the cast alloys was calculated and listed as 16.29 ± 1.52, 14.59 ± 1.01, 10.65 ± 0.95, and 11.46 ± 0.73 μm. This can represent the influence of the RE and Sr additions on the formation of a constitutional undercooling zone on the head of the solid/liquid interface, which promotes the nucleation and hinders the fast grain growth [30].

Generally, the as-cast alloys have large grain structures with semi-continuous networks of intermetallics, resulting in the deterioration of their mechanical properties [31-35]. According to the literature [32-35], the grain size of the as-cast AZ91 alloy was more than 100 μm. Therefore, to improve their properties and further practical applications, applying the thermo-mechanical

Table 1- Chemical composition of the studied alloys

Alloy	Al	Zn	RE	Sr	Mg
Base	9	1	0	0	balance
RE0.1	9	1	0.1	0.9	balance
RE0.5	9	1	0.5	0.5	balance
RE0.9	9	1	0.9	0.1	balance

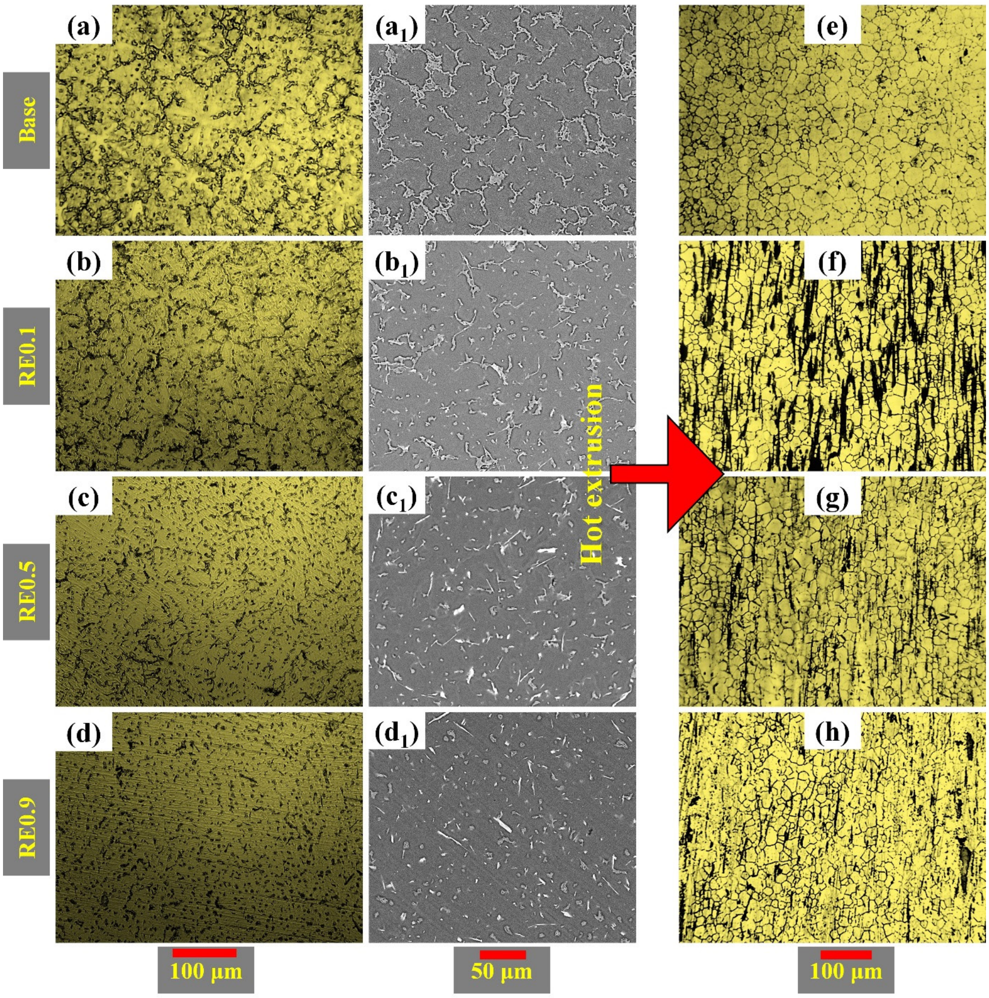

Fig. 1- Optical and SEM micrographs of the Mg-9Al-1Zn-RE/Sr alloys.

process is vital. Figures 1(e)-(h) illustrate the optical micrographs of the extruded alloys. As can be seen, the hot extrusion process results in the meaningful grain refinement and fracturing and distribution of intermetallics along the extrusion direction. Based on Figure 1(e), the Base alloy is almost without any signs of the β intermetallics, relating to the dissolution of these intermetallics during the homogenization and hot extrusion processes. However, other Figures depict the presence of the Al_4Sr and $Al_{11}RE_3$ intermetallics after these processes. The grain size values of the Base, RE0.1, RE0.5, and RE0.9 alloys are 22, 19, 14, 15 µm, respectively. The grain size variations of the extruded alloys are relevant to the changes in DAS values. The noticeable grain refinement by the hot extrusion process is related to the occurrence of the dynamic recrystallization process (DRX) [36,37]. Moreover, more RE/Sr addition reduced the grain size, compared to that of the Base alloy. Figure 2 shows the EDS mapping and point analysis for as-extruded RE0.1 and RE0.9 alloys, which reveals the distribution of Al_4Sr and $Al_{11}RE_3$ intermetallics and their related elements. As can be seen in Figure 2 and Table 2, the RE0.1 and RE0.9 alloys have mostly consisted of Sr-containing (Al_4Sr) and RE-containing ($Al_{11}R_{E3}$) intermetallics, respectively.

The presence of new intermetallics stimulates the DRX phenomenon during the hot extrusion by the Particle Stimulated Nucleation (PSN) mechanism. Based on Robson et al. [27], the deformation zone around the large (> 1 µm diameter) and hard particles during deformation leads to the formation of new high-angle grain boundaries (HAGBs), and hence the DRXed grains. In the present work, the size of the particles in the extruded RE0.1 and

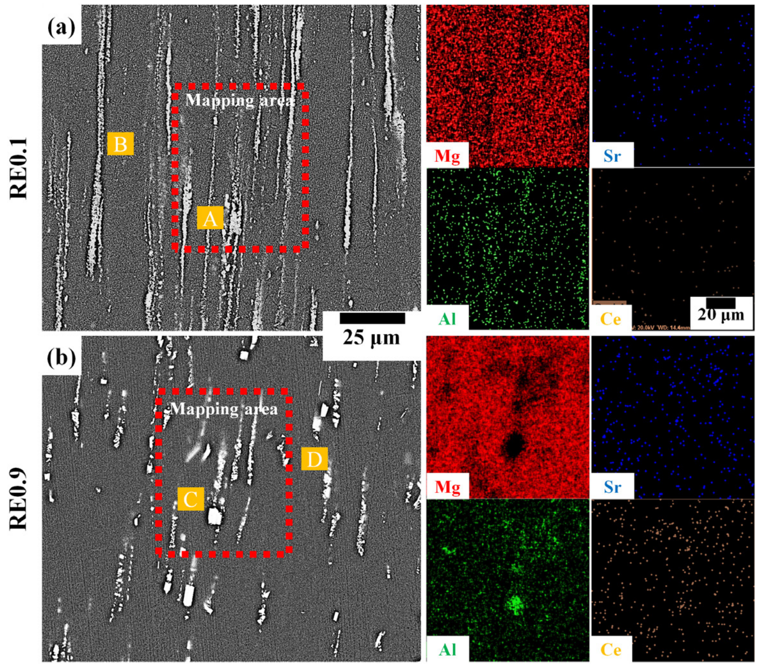

Fig. 2- SEM micrographs with the EDS analysis of the extruded RE0.1 and RE0.9 alloys.

Table 2- EDS analysis results related to the points in Figure 2

Point	Atomic percent (at.%)				
	Mg	Al	Zn	Sr	Ce
A	77.52	5.47	0.01	16.99	0.01
B	83.39	14.41	1.04	1.13	0.02
C	48.97	39.73	2.81	0.23	8.26
D	59.06	35.97	1.32	0.00	3.65

RE0.9 alloys is approximately 2.0 ± 0.1 and 5.0 ± 0.3 µm. These values show that the deformation zone around the RE-containing intermetallics is larger than around the Sr-containing intermetallics and hence these intermetallics have more potential for the formation of more DRXed grains. Furthermore, the intermetallics can act as inhibitors for grain boundaries motion during the grain growth of the DRXed grains. By considering the lower grain size values of more RE/Sr-containing alloys, the $Al_{11}RE_3$ intermetallics have a more beneficial impact on the grain refinement, compared to the Al_4Sr intermetallics. This incident can be related to the influence of RE-containing intermetallics on the formation of a more DRXed grain structure.

Figure 3 illustrates the tensile stress-strain curves for the as-cast and as-extruded alloys. According to this Figure, the ultimate tensile strength (UTS) and tensile elongation (%El) values of the cast Base alloy are ~130 MPa and ~2%, respectively. The RE/Sr additions significantly improve the UTS and %El values. In this regard, the UTS and %El values of the RE0.9 alloy are ~225 MPa and ~10.5%, respectively. The improvements are related to the presence of new Al_4Sr and $Al_{11}RE_3$ intermetallics, and changing the morphology of the semi-continuous β phase to a more discontinuous and fragmented one.

After the hot extrusion process, the UTS and El% values of the Base alloy improve significantly to 323 MPa and 17.2%, respectively. With the addition of 0.1% RE and 0.9% Sr, the UTS and El% values of the extruded Base alloy increase to 349 MPa, 19.7%. Furthermore, the extruded RE0.5 alloy is the optimum alloy with the UTS and El% values of 350 MPa and 29.1%. However, more RE/Sr additions deteriorated the UTS and El% values to 335 MPa and 19.8% for the RE0.9 alloy. The meaningful improvement of the tensile properties

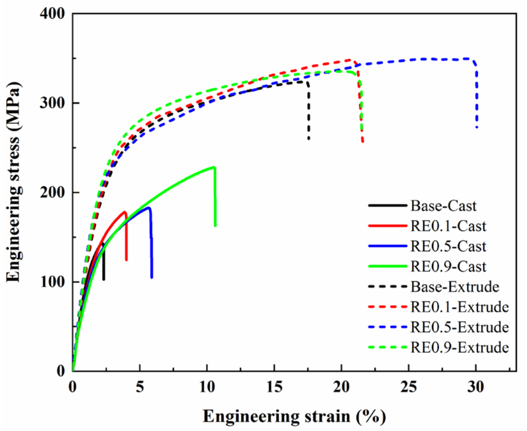

Fig. 3- Tensile stress-strain curves of the as-cast and as-extruded alloys.

by the hot extrusion process is related to different factors. (I) Grain refinement due to the occurrence of dynamic recrystallization during the hot extrusion and hence the enhancement of tensile properties the extruded alloys [36,37]. Moreover, with more RE/Sr addition, the grain size decreases from 22 to about 14 μm, which is a relevant issue for the improvement of tensile properties in the extruded RE0.5 alloy. (II) Hot extrusion process could properly break and distribute the semi-continuous intermetallics of the cast samples along the extrusion direction. However, 9RE/Sr addition creates larger needle-like $Al_{11}RE_3$ intermetallics, which may be a reason for the ductility deterioration of the extruded RE0.9 alloy. (III) Dissolution of the β phase and distribution of the Al atoms during homogenization heat treatment and hot extrusion [16,36]. (IV) The present of RE elements for the RE0.1 and RE0.5 alloys significantly increased the ductility, which can be related to their effect on the decrement of the texture intensity and development of a new texture component (RE texture) [38-40]. Moreover, the RE elements may affect the activation of non-basal slip systems by the reduction of c/a ratio or changing the stacking fault energy [38].

4. Conclusions

The effect of the hot extrusion process on the significant grain refinement and enhancement of tensile properties of Mg-9Al-1Zn-RE/Sr alloys were studied. The following conclusions can be drawn:

1- After the extrusion process, the grain size of the as-extruded alloys significantly decreased in comparison with the as-cast samples. Moreover, the intermetallics were fragmented and distributed along the extrusion direction. The formation fine DRXed grain structure after hot extrusion was related to the occurrence of the PSN mechanism.

2- By adding more RE/Sr, the grain size value reduced compared to the Base alloy. The lowest grain size value was related to the RE0.5 and RE0.9 alloys owing to the effect of RE elements on the formation of more DRXed grains.

3- Compared to the cast alloys, the tensile properties (especially the ductility) of the alloys significantly increased after the hot extrusion process, which can be related to the remarkable grain refinement and fragmentation, and distribution of the intermetallics.

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