Microstructure and texture evolution with Sm addition in extruded Mg-Gd-Sm-Zr alloy

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Abstract
We prepared as-extruded Mg-10Gd-xSm (x = 1, 3, 5)-0.5Zr alloys by indirect extrusion, and the microstructure, texture and mechanical properties of as-extruded Mg-10Gd-xSm-0.5Zr alloys were investigated. The results indicate that the microstructure, texture and strength of as-extruded Mg-10Gd-xSm-0.5Zr alloys take a great variation with Sm addition. Dynamic recrystallized grain size is decreased with increasing Sm content, and the average grain size rapidly reduced to 1.4 μm in as-extruded Mg-10Gd-5Sm-0.5Zr alloys due to abundant Mg5(Gd, Sm)5 precipitates and their pinning effect on grain boundaries. With an increasing Sm content, the texture has a significant variation. The fiber texture with ⟨10-10⟩ axis of Mg matrix parallel to extrusion direction (ED) in as-extruded Mg-10Gd-1Sm-0.5Zr alloy transformed into an abnormal texture with ⟨0001⟩ axis of Mg matrix parallel to ED in as-extruded Mg-10Gd-3Sm-0.5Zr alloy. The tensile yield strength and Brinell hardness gradually increase with Sm addition due to solid solution strengthening, refined grain boundaries strength and numerous precipitates strengthening. However, the as-extruded Mg-10Gd-5Sm-0.5Zr alloy exhibits the poor ductility due to stress concentration caused by abundant precipitates.

1. Introduction

Due to their lightweight, Magnesium and its alloys have been paid extensive attention in auto, aerospace, 3C production and other fields in recent decades [1–3]. However, Mg alloys usually exhibit poor ductility at room temperature due to their hexagonal close-packed crystal structure. Numerous investigations were dedicated to concentrate on improving ductility, and thermo-mechanical processing (including extrusion, rolling, forging) accompanied by recrystallization behavior is sufficient to improve the workability at elevated temperature [4]. Extrusion process is an easy manipulation with just a single pass compared with other wrought methods. Furthermore, extrusion Mg alloys exhibited the superior performance to cast counterparts [5]. However, their worldwide application is still limited due to strong basal texture for extrusion alloys.

Grain refinement and weakening texture are two feasible modification mechanism for improvement of ductility. Addition of rare earth (RE) elements is an effective access to realize above objectives [6, 7]. The addition of RE into extrusion alloys have been extensively studied for modifying the texture and refining grains. Stanford et al [8] reported that the addition of 1.55 wt% Gd to pure Mg effectively reduced the angle between the c-axis and ED. Moreover, a trace of Y, Ce and La addition remarkably changed the basal texture, and produced the ‘RE texture’ component, that is, ⟨11-21⟩ of Mg matrix parallels to ED [9]. Hadorn et al [10] also reported a weak ‘RE texture’ in an extruded Mg-0.39Gd (wt.%) alloy. In addition to the ‘RE texture’ component, an abnormal texture with ⟨0001⟩ direction parallel to ED have also been frequently reported in Mg-RE alloys during hot extrusion [11–13]. The formation of abnormal texture was reported to be associated with the different stored energy and grain boundary misorientation during annealing [14]. In recent studies, it is even related to extrusion parameters...
including extrusion ratio, extrusion speed and extrusion temperature during hot extrusion [11]. These factors influenced the final texture through solute drag effect [9, 10], nuclei at local deformation region [15, 16] and activation of extra deformation modes [11, 17]. Although RE elements have been reported to modify the texture and promote the formation of RE texture, the underlying formation mechanisms are still controversial in extrusion Mg alloys due to many manipulation factors which could resulting in the final formation of texture. In contrast to commercial Mg alloy with strong texture, numerous studies reported RE-containing Mg alloy generally have weak texture with relative randomized crystallographic orientation resulting in the good ductility [18].

Gd belongs to yttrium subgroup and has a maximum solubility of 23.6% in solid Mg, indicating that dissolved Gd in Mg shows a great favorable effect on solid solution [19]. In the studies of Mg-RE alloy, Gd exhibited more excellent solute strengthening in Mg than other RE elements such as Ho, Nd, Dy and Y [20]. Hence, plenty of Gd added into Mg with supersaturated state could produce significant microstructure and texture variation. Up to date, Mg alloys with high Gd contents (over 10 wt.%) have been focused on improvement of high strength of extruded alloys by combination of Y and other alloying elements [21, 22]. For example, the Mg-11.7Gd-4.9Y-0.3Zr (wt.%) alloy had an ultra-high strength, and it exhibited ultimate tensile strength of 539 MPa and yield strength of 500 MPa by pre-deformation and extrusion [23]. Wrought Mg-11Gd-4.5Y-1Nd-1.5Zn-0.5Zr (wt.%) alloys exhibited the ultimate tensile strength of 547 MPa and yield strength of 502 MPa [24]. The Zr element is a common grain refiner for Mg-RE alloys due to their similar crystal structure to Mg, and addition of Zr normally could improve strength of Mg alloys [25].

Sm has a maximum solubility of 5.8% in solid Mg [19]. Furthermore, Sm and Gd could reduce their solubility in Mg each other, indicating that an enhanced solute dissolution and precipitation might occur in Mg alloys [26]. This means that a significant evolution of microstructure and texture would be generated with variation of Sm content, theoretically. It is worth mentioning that Sm has a cheaper price than Y in the current RE market, showing that Mg–Gd–Sm system alloys are more attractive compared to Mg–Gd–Y alloy in commercial viability aspects. However, most of the Mg–Gd–Sm–Zr alloys were focused on ageing precipitation behavior in casting [26, 27], and extremely limited research were carried out on extruded Mg–Gd–Sm–Zr alloys compared to casting counterparts. Hence, it is very necessary to investigate the microstructure and texture evolution of extrusion Mg–Gd–Sm–Zr system alloys. Here, the effect of selected Sm on microstructure, texture and mechanical properties of as-extruded Mg-10Gd-xSm-Zr alloy was focused in this paper. Interestingly, the grain size, precipitates, texture and strength of as-extruded Mg-10Gd-xSm-0.5Zr alloy had an extraordinary change with Sm addition.

2. Experiment producers

The alloy ingots with nominal compositions of Mg-10Gd-xSm (x = 1, 3, 5)-0.5Zr were fabricated by melting high purity Mg (99.95 wt%), Mg-30 wt% X (X = Gd, Sm and Zr) master alloys in a medium electric resistance furnace under protective gas atmosphere (CO2 and SF6 with ratio 99:1). The Mg-10Gd-1Sm-0.5Zr, Mg-10Gd-3Sm-0.5Zr and Mg-10Gd-5Sm-0.5Zr alloys are named as GS1, GS3 and GS5 alloys. The actual chemical compositions of Mg-Gd-Sm-Zr alloy detected by inductively coupled plasma (Optima 8000) were listed in table 1. Melt at 750 °C was held for 5 min, then pouring the melt into a pre-heated metal mold. Cylindrical billets with a diameter of 50 mm and a height of 40 mm were cut from ingots, and then were homogenized treatment at 515 °C for 10 h followed by hot water quenching. Before indirect extrusion, the billets were pre-heated for 2 h at 500 °C, and the mold were preheated to 350 °C. Extrusion was conducted at ram speed of 5.4 mm s\(^{-1}\), with extrusion ratio of 9.67, and then the extruded rod was quenched in hot water. The sheet-shaped samples with a gauge length of 15 mm, width of 3 mm and thickness of 2 mm were processed for tensile tests at room temperature. Figure 1 shows the fabricated extruded samples and schematic diagram of tensile specimen. The tensile tests were executed via SHIMADZU AG-1250 N universal testing machine with a speed of 1 mm min\(^{-1}\). The values of Brinell hardness were measured along the extrusion direction (ED) under a load of 250 Kgf for 30 s.
Phase structures were measured by the x-ray diffractometry (XRD) at 40 kV and 40 mA with a Cu K-α radiation. The microstructure was observed using JSM-7800F scanning electron microscope (SEM), and Oxford C-nano electron backscatter diffraction (EBSD) at 15 kV. The EBSD method with step size 0.3 μm is used for characterization of microstructure and texture. The relevant EBSD data are analyzed using Aztec Crystal software.

3. Results and discussion

The SEM images and EDS results of as-homogenized Mg-10Gd-xSm (x = 1, 3, 5)-0.5Zr alloys are depicted in figure 2. It is clearly seen that the second phase formed in the solidification process is almost dissolved into the Mg matrix after homogenization treatment except for a small amount of cubic phase in all specimens. The corresponding EDS results show that the ample RE element are enriched in cubic particles, and a certain amount of Gd solute atoms are present in the matrix showing in figures 2(d)–(f). These particles with varied from 0.1–3 μm in size might be Mg-RE compounds which have been reported in early studies [28]. Figure 3 shows the XRD patterns of as-homogenized Mg-10Gd-xSm-0.5Zr alloys, and only α-Mg and GdH2 diffraction peak are detected. Combined with SEM images, these cubic phases can be identified as GdH2 compounds in as-homogenized alloys which is inconsistent with the other observations [28]. Recently, the formation of hydrides is related to the
reaction of Mg-RE alloys with water during the preparations with water cleaning in recent report by Huang et al [29]. However, the formation mechanism is still to be discussed. It is worth mentioning that the RE hydrides are considered to have an unfavorable effect on mechanical properties in some studied [29, 30].

The EBSD inverse pole figure (IPF) maps and pole figures of as-homogenized Mg-10Gd-xSm-0.5Zr alloys are depicted in figure 4. The average grain size gradually decreases with the increase of Sm, and the average grain size is 66.7 μm, 64.8 μm, 55.3 μm, respectively. Each alloy shows a uniform near-equiaxed grain structure with relatively uniform distribution. The pole figure of each alloy is shown in figures 4(a)–(f) indicating that the grains all exhibit the randomized texture in Mg-10Gd-xSm-0.5Zr alloys which means that the effect of initial texture on recrystallization behavior does not require consideration.

The SEM images and EDS results of as-extruded Mg-10Gd-xSm-0.5Zr alloys are depicted in figure 5. It is obvious that cubic phases formed in homogenization treatment are broken up, and the fair-shaped solute has a redistribution along ED. Quite few precipitates are observed in GS1 alloy, while the GS3 alloy has a certain quantity of the precipitates as shown in figure 5(b). Interestingly, a considerable amount of dynamic precipitates with 0.1 um - 5 um in size are observed. Some of them are dispersed inside the matrix in addition to some fair-shaped dynamic precipitates. The quantitative statistics results of the second phase are approximately 0.7% for

![Figure 3. XRD patterns of as-homogenized of Mg-10Gd-xSm-0.5Zr alloys.](image)

![Figure 4. IPF maps and corresponding pole figures of as-homogenized Mg-10Gd-xSm-0.5Zr alloys; (a), (d) GS1 alloys, (b), (e) GS3 alloys, (c), (f) GS5 alloys.](image)
GS1 alloys, 6.6% for GS3 alloys, and 25.4% for GS5 alloys, respectively. Subsequent EDS analysis results illustrate that the precipitates in marked region contain the luxuriant Gd, Sm element as shown in figures 5(a)–(f).

Figure 6 shows the XRD patterns of as-extruded Mg-10Gd-xSm-0.5Zr alloys, and matched XRD results indicates that the diffraction peak of second phases is not observed in as-extruded GS1 and as-extruded GS3 alloys. This means that a small amount of the precipitates do not reach the critical content for XRD detection in as-extruded GS1 and as-extruded GS3 alloys [31]. Interestingly, dynamic precipitates including Mg5Gd, Mg41Sm5 type intermetallic compound are detected in as-extruded GS5 alloy which is in consistent with other findings in Mg–Gd–Sm–Zr alloys [32]. Combined with the Mg–Gd ternary phase diagram, the equilibrium solubility of Gd element is approximately 19% in Mg at 500 °C, which is greater than the current Gd content in Mg [19]. However, it is worth mentioning that mold temperature and heat conduction could reduce the actual extrusion temperature. In the meanwhile, a mutual reduction of the solid solution of two rare earth elements in Mg is definite with Sm content increase. The formation of dynamic precipitates may be related to above-mentioned factors. It is worth noting that the large precipitated particles over 2 μm are distributed along the ED.
These fair-shaped regions are usually easy to result in nucleation and growth of precipitated particles, due to the fact that these regions with intense deformation could induce particles nucleation and Gd segregation. The IPF maps, pole figures and inverse pole figures of as-extruded Mg-10Gd-\(x\)Sm-0.5Zr alloys are shown in the figure 7, indicating that as-extruded GS1 alloys exhibit the bimodal grain structures which is consist of 79.2% DRXed and 21.8% unDRXed grains, while the full recrystallized grains are obtained in as-extruded GS3 and as-extruded GS5 alloys. The average grain size gradually decreases with addition of Sm, and a significant drop in grain size in as-extruded GS5 alloy. The average grain size is 8.5\(\mu\)m, 5.1\(\mu\)m and 1.4\(\mu\)m for as-extruded GS1, as-extruded GS3 and as-extruded GS5 alloy, respectively. The promoted dynamic recrystallized grains may be attributed to particle stimulation nucleation (PSN) mechanism, that is, precipitated particles could act as nucleation sites for DRX by the accumulated dislocation density [33]. Furthermore, grain boundaries are subjected to the precipitated particles pinning dynamically during hot extrusion [16, 33]. It is considered that numerous fine precipitates can provide the effective obstacle to growth of newly recrystallized grains via the Zener pinning effect. It is clearly expounded the significant grain refinement of as-extruded GS5 alloy. The most obvious evidence is the presence of abundant fine recrystallized grains in the extruded fair-shaped region which contains the precipitated particles in the band contrast maps in figure 7(b).

Pole figures and inverse pole figures from EBSD analysis results are simultaneously obtained in figure 7, showing that the texture of as-extruded alloys have a notable variation with Sm addition. For as-extruded GS1 alloy, the strong texture with \(\{10-10\}\) of Mg matrix parallel to the ED are obtained which agree with typical fiber texture in most Mg alloys. However, the texture has dramatically transformed into an abnormal strong texture with \(\{0001\}\) of Mg matrix parallel to the ED in as-extruded GS3 alloy which is the opposite of the typical orientation with c-axes are perpendicular to the ED in extruded Mg alloys. This abnormal texture with \(\{0001\}\) direction parallel to ED has also been reported in Mg-RE alloys during hot extrusion in recent years [11–13], and the related mechanism concerning the abnormal texture will be discussed subsequently. With an increasing Sm content, the texture orientation changes again from \(\{0001\}\) to \(\{11-21\}\) direction. This means that the extraordinary rotation of crystallographic orientation can be achieved in Mg–Gd–Sm–Zr alloy with addition of different Sm content.

Abundant studies have shown that the RE-containing Mg alloys did exhibit the weaker texture compared to extruded commercial Mg alloys with strong basal texture [9–13]. Texture modification triggered by Sm content...
may be related to solute drag effect \cite{8, 34, 35}, shear band induced nucleation (SBIN) \cite{15, 36}, deformation twin induced nucleation (DTIN) \cite{37, 38}, and activation of other deformation modes \cite{11, 34, 39}. Stanford \cite{8} and Hadorn et al \cite{10} demonstrated the Gd atoms are segregated to grain boundaries in an extruded Mg-Gd alloy by using atom probe tomography technique and high-angle annular dark-field scanning transmission microscopy. Barrett et al \cite{35} combining EBSD and molecular dynamics simulation demonstrated that RE segregation could homogenize the energies of fiber grain boundaries in curtailing recrystallization of RE-containing Mg alloy. Grain boundary segregation are believed to be relative to strong interactions between the solute and grain boundaries via hindering the migration of grain boundaries and dislocations. It might lead to a texture modification by means of transformation of recrystallization mode or preferential growth of oriented grains. It is worth noting that the nature of this behavior is a solute drag effect due to the very low diffusion capacity of RE elements in Mg alloys. In the present work, the grain boundary segregation of Gd and Sm atoms has reason to be currently regarded as the underlying texture modification mechanism. In addition, the addition of RE elements has been reported to be capable to promote the formation of more homogeneous shear bands during hot extrusion, indicating that the more heterogeneous nucleation sites for recrystallized grain are obtained, resulting in a recrystallized grains orientations in Mg alloys \cite{15, 36}. It is recently reported that the recrystallized grains with RE texture are formed at shear bands in Mg-RE alloy \cite{36}. Therefore, nuclei induced by shear band are considered as an underlying mechanism for texture modification although the shear zones are not introduced in this paper in a judicious manner. In addition to SBN, the DTIN also plays a significant role for texture modification in Mg-RE alloy. In the early stage of deformation, the twin behavior is not only well known to coordinate the deformation, but also providing a favorable location for the nuclei of recrystallized grains \cite{37, 38}. Guan et al reported \cite{37} that 10-11 compression twins and 10-11 - 10-12 double twins with the abundant internal stored energy could promote subsequent recrystallization and grain growth with randomized texture deriving from twins. It is worth mentioning that pinning effect of precipitated particles on grain boundaries or oriented rotation of the local lattice around the precipitated particles may result in the formation of RE texture. Combined with SEM images as shown in the figure 5(c), the nucleated particles certainly have a retardation effect on moving of grain boundaries through Zener effect during grain growth. In addition to inhibiting the growth of grains, these particles induced nucleation of recrystallized grains with randomized orientation resulting in the formation of RE texture during recrystallisation.

Most of RE elements added to Mg alloys can promote the formation of RE texture, while the abnormal texture with \langle 0001 \rangle of Mg matrix parallel to ED is formed which recently has been reported in Mg–RE alloy including Mg–Gd–Y–Zr, Mg–Y–Sm–Zr alloys \cite{11-13}. Jin et al claimed \cite{11} that promoted continuous operation of non-basal slip by induced RE is easier to promote the continuous accumulation of dislocation, and further would rotate subgrains with c axis parallel to ED. Lyu et al\cite{12} reported that drag effect of Y and Sm, the sufficient energy for growth of the specific orientation and the transformation of deformation modes might be the main reason for the formation of abnormal texture in a Mg–Y–Sm–Zr alloy. Nevertheless, the formation mechanism of the abnormal texture is still controversial and debatable in Mg–RE alloys. It can be determined that pinning effect of precipitated particles is an unrecognized modification mechanism in current study. It is clearly seen that most grains in the precipitation-free region oriented with a \langle 0001 \rangle of Mg matrix parallel to the ED, while the fine recrystallized grains in the fair-shaped region have the same orientation characteristics combined with band contrast map of figure 7(b). It is indicating that the nucleated particles have a weak or futile effect on growth orientation of grain although these particles widely induced nucleation of recrystallized grains. Therefore, it is reasonable to assume that prominent texture evolution can be attributed to the solute drag effect, SBN, TDIN, and non-basal slip activation with an increasing Sm content. The formation of an abnormal texture with \langle 0001 \rangle of Mg matrix parallel to ED in as-extruded GS3 alloy is not related to the pinning effect of precipitated particles.

Figure 8 shows the \langle 0001 \rangle \langle 1120 \rangle and \langle 10-10 \rangle \langle 1120 \rangle Schmid factors (SF) histograms of the as-extruded Mg-10Gd-xSm-0.5Zr alloys when the tensile stress is applied along the ED. It is well-known that the activity of a slip mode is strongly dependent on the crystallographic orientation of the metal, and value of SF is normally employed to evaluate activation capacity of slipping systems \cite{40}. For as-extruded GS1 alloy, un-recrystallized grains exhibit the strong fiber texture, leading to a distinct low SF for basal slip as shown in figure 8 (a). As Sm content increase to 3%, the average Schmid factors of basal slip have a slight increase, while the prismatic slip has a great decline. This means that basal slip dominates deformation mechanism for as-extruded GS3 alloys under tension along the ED at room temperature. With an increasing of Sm content, such an anomalous crystallographic orientation disappears and the average Schmid factor for basal slip as well as prismatic slip returns to normal levels.

Figure 9 shows the nominal stress-strain curves, Brinell hardness and the corresponding fracture morphologies of as-extruded Mg-10Gd-xSm-0.5Zr alloys at room temperature. The as-extruded GS1 and GS3 alloy all exhibit the good ductility with an elongation of 13.4%, 13.9%, respectively. It is mainly related to extremely few dynamic precipitates for as-extruded GS1 alloy, and the energetic activation of basal slip for as-
extruded GS3 alloy. As shown in figures 9(d), (e), the fracture morphologies of as-extruded GS1 and GS3 alloys contain abundant dimples and tearing ridges indicating ductile fracture is the main fracture mechanism for two alloys. With an increasing of Sm content, the yield strength gradually increases, and its value is 159 MPa for as-extruded GS1 alloy, 180 MPa for as-extruded GS3 alloy, 230 MPa for as-extruded GS5 alloy, respectively. In the meanwhile, the Brinell hardness also increase with increasing Sm content as shown in figure 9(c), the Brinell hardness value of as-extruded Mg-10Gd-xSm-0.5Zr alloys is 89.9HBW, 96.7HBW and 100.67HBW, respectively. While ultimate tensile strength not follow the such a law with increasing Sm. The improved yield strength and hardness with addition of 3% Sm is attributed to solid solution strengthening and a limited precipitations strengthening. Especially for as-extruded GS5 alloy, the extreme fine-grains (1.4 μm), abundant dynamic precipitated $\text{Mg}_5(\text{Gd, Sm})$, $\text{Mg}_4(\text{Gd, Sm})_5$ particles are responsible for significant improvement of yield strength. It is worth mentioning that as-extruded GS5 alloys exhibit the relatively poor elongation with 4.9%, and the deep cracks are observed at the fractured surface as shown in figure 9(f). Although precipitates provide the effective obstacles on slip of dislocation, they typically act as a damaged interface and source of crack.

Figure 8. Schmid factor distribution histograms of the as-extruded Mg-10Gd-xSm-0.5Zr alloys when the tensile stress is applied along the ED: (a), (b) as-extruded GS1 alloy, (c), (d) as-extruded GS3 alloy, (e), (f) as-extruded GS5 alloy.

Figure 9. (a) The nominal stress-strain curves of as-extruded Mg-10Gd-xSm-0.5Zr alloys, (b) the corresponding mechanical parameters of as-extruded Mg-10Gd-xSm-0.5Zr alloys, (c) Brinell hardness value of as-extruded Mg-10Gd-xSm-0.5Zr alloys, and fracture morphologies of (d) as-extruded GS1 alloy, (e) as-extruded GS3 and (f) as-extruded GS5 alloy at room temperature.
initiation due to stress concentration during plastic deformation. As a result, the crack tip goes through the precipitates mainly along the plastic deformation path and result in brittle fracture.

4. Conclusion

In this work, the microstructure, texture and mechanical properties of as-extruded Mg-10Gd-xSm-0.5Zr (x = 1, 3, 5) alloy are investigated comprehensively. The grain refinement is achieved with an increasing Sm, and the extreme fine-grains with approximate 1.4 μm in size are obtained in as-extruded GS5 alloy due to the Zener effect of precipitated particles on grain boundaries. Texture also takes a significant transformation, that fiber texture for as-extruded GS1 alloy changes into an abnormal texture with (0001) of Mg matrix parallel to ED in as-extruded GS3 alloy, and then the (11-21) texture is formed with addition of 5% Sm. The yield strength and hardness gradually increase with an increasing Sm content, and the as-extruded GS5 alloy has a yield strength of 230 MPa which is attributed to fine grains strengthening and numerous Mg5(Sm, Gd)3 precipitation strengthening. However, the as-extruded GS5 alloy exhibit the poor ductility due to crack initiation at precipitates.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of competing interest

The authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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