Original article

Effect of addition of small amounts of samarium on microstructural evolution and mechanical properties enhancement of an as-extruded ZK60 magnesium alloy sheet

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A B S T R A C T

The microstructure, texture evolution and mechanical properties of as-extruded Mg-5.5Zn-0.8Zr alloy with 1 % Sm addition were investigated. The results showed that the as-extruded ZK60-1Sm alloy consisted of α-Mg, Mg2Zn13, Mg7Zn3, Mg41Sm3 and (Mg, Zn)3Sm phases. The addition of 1 %Sm precipitated the Mg41Sm3 and (Mg, Zn)3Sm phases, which promoted the dynamic recrystallization (DRX) in the subsequent extrusion process and resulted in grain refinement. The microtexture and the orientation distribution characteristic implied that large amounts of <01–10> and <−12–10> slips were activated in ZK60-1Sm alloy during extrusion process, while only <01–10> basal slip was observed in ZK60 alloy. The non-basal slips led to more potential nucleation sites and a better coordination for the deformation, resulting in the better comprehensive mechanical properties of the as-extruded ZK60-1Sm sheet. Meanwhile, the as-extruded ZK60-1Sm alloy showed higher strength and ductility compared to ZK60 alloy. The ultimate tensile strength (UTS), the yield strength (YS) and the elongation to failure (EL) along the extrusion direction (ED) were enhanced by 11.7 %, 24.5 % and 26.2 %, respectively. The influence of fine grain microstructure, nanoscale Mg41Sm3 phase, micron scale (Mg, Zn)3Sm phase and texture evolution, contributed mainly to the strength of as-extruded ZK60-1Sm alloy.

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1. Introduction

Recently, numerous researchers have focused on wrought Mg-Zn-Zr based alloys due to their excellent strength and great potential for further development. They are widely used in automobile, high-speed train, electronic communication, aerospace, national defense and military [1–3]. The addition of Zn element can lead to lattice distortion, hinder dislocation movement, improve strength and play a role of solution strengthening. In addition, Zr element plays an important role in grain refinement, which can increase grain boundaries, slow diffusion rate of alloys, and effectively prevent grain growth.

Up to now, many investigations have been performed on Mg-Zn-Zr based alloys with the aim of gaining desired mechanical performance by some methods. On the one hand, the grain refinement, precipitation and texture control are realized through the microstructure modification of rare earth element alloying. On the other hand, the required mechanical properties are obtained by various thermal-mechanical treatments such as hot extrusion and aging after deformation [4]. At present, these two methods are widely used in improving the properties of alloys [5–7]. Zengin H et al. [8] revealed that La addition to ZK60 alloy resulted in the formation of Mg-Zn-La ternary phase and these fragmented particles led to nucleation of new grains around them i.e. particle stimulated nucleation (PSN), and promoted DRX during extrusion. The grain size of the alloy was significantly refined, which played an important role in improving the strength of the alloy. In addition, rare earth elements have the functions of forming dispersion phase with high melting point and changing the number, morphology and distribution of the second phase. It plays the role of solid solution strengthening and second phase strengthening, as-reported in Zheng [9] and Xu [10]. The addition of rare earth element can also improve the mechanical properties at room and high temperatures and the casting formability of magnesium alloys.

In previous studies, Sm element has a good solid solution strengthening effect, and it is easy to combine with Mg to form Mg24Sm4 phase with good thermal stability, which can play a second phase strengthening role in as-cast alloy [11,12]. Now, more and more researchers used Sm to improve the properties of magnesium alloys. For example, Lyu et al. [13] fabricated the high strength Mg-Y-Sm-Zn-Zr alloy by conventional hot extrusion and aging, Xia et al. [14] reported that the Mg-Sm-Zn-Zr alloy exhibits significant precipitation hardening effect. The addition of Sm can refine the grain size of magnesium alloy and make the microstructure more uniform, as-reported in Wang et al. [15] and Gui et al. [16]. In addition, the intermetallic compounds formed by the combination of Sm and Mg can play the role of second phase strengthening. Yuan et al. [17] found that (Mg, Zn)2(Sm, Gd)1 eutectic phase exists in as-cast Mg-2.6Sm-1.3Gd-0.6Zn-0.5Zr alloy. Huang et al. [18] ascertained the Mg2ZnSm and Mg24Sm phase in Mg6Zn4Sm-0.4Zr alloy, which significantly improve the mechanical properties of the alloy. Recently, Cao et al. [19] demonstrated that cold plastic pre-deformation before aging treatment can significantly improve the aging hardening response of Mg-5Sm-1.4Nd-0.4Zn-0.5Zr alloy.

2. Experimental procedures

2.1. Alloy preparation

The chemical compositions of ZK60 and ZK60-1 wt.%Sm (All compositions quoted in this work are in wt.% unless otherwise stated) were analyzed by inductively coupled plasma mass spectrometry (ICP-MS), and the results were given in Table 1. Experimental alloy ingots were prepared from high-purity Mg and Zn (>99.95%), Mg-30.17 %Zr and Mg-29.45 %Sm master alloys by melting in a KGYS175/3 vacuum induction melting furnace. Before the experiment, the mold and raw materials were preheated at 200 °C for 1h. Before extrusion, cast billets were homogenized at 450 °C for 10h with the protection of argon. During the extrusion process, extrusion temperature, extrusion ratio and extrusion ram speed were 400 °C, 20 and 1.6 cm/s, respectively. Finally, a 100 mm (width) × 4 mm (thickness) sheet was obtained. The processing of tensile specimens (along the ED) conforms to the international standard ISO 6892-1: 2016. Metallographic specimens (10 mm × 10 mm × 4 mm) and preliminary transmission electron microscopy (TEM) specimens (10 mm × 10 mm × 0.5 mm) were prepared by wire cutting from the as-extruded sheet.

2.2. Performance test

Table 1 - Chemical compositions of the investigated alloys.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Composition (wt.%)</th>
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<tbody>
<tr>
<td></td>
<td>Zn</td>
</tr>
<tr>
<td>ZK60</td>
<td>5.49</td>
</tr>
<tr>
<td>ZK60-1Sm</td>
<td>5.62</td>
</tr>
</tbody>
</table>

Although a large number of experiments have been carried out to improve the mechanical properties of Mg-Zn-Zr based magnesium alloys by RE additions and different processing routes, a fewer study referred about the effect of Sm on strengthening mechanism for as-extruded ZK60 alloy. Moreover, it remains unclear the anisotropy of alloy after processing deformation due to the Sm addition. Previously, we studied DRX behavior, second phase changes and microtexture evolution of as-extruded ZK60 alloy by 4 %Sm addition [20]. ZK60-4Sm alloy with good ductility was fabricated, and its EL increased by nearly 100 % compared with ZK60 alloy, but the improvement of UTS and YS was not significant. Based on above description, we added 1 % Sm into ZK60 alloy to study its effect on the microstructure, microstructure changes and mechanical properties of as-extruded ZK60 alloy, aiming to improve the UTS and YS of the alloy.

X-ray diffraction measurements (XRD; XRD-6000 X-ray diffractometer) were obtained for the phase analysis by Cu-Kα radiation with scanning angle from 20° to 80° and a scanning rate of 5°/min. Extruded samples for microstructural analysis were etched in a solution of 10 ml acetic acid, 4.2 g picric acid, 70 ml ethanol and 10 ml distilled water after grinding and mechanical polishing. The microstructural morphology...
and compound composition of the alloys were examined through optical microscopy (OM; Axio Scope. A1 Zeiss), scanning electron microscopy (SEM; FEI Quanta 250) equipped with energy-dispersive X-ray spectroscopy (EDS), and field emission transmission electron microscope (FETEM; Tecnai G2 F20 S-TWIN). The average grain size was measured using the linear intercept method by Image-Pro Plus 6.0. Thin foil specimens for TEM observation were prepared by mechanical polishing from 500 μm to 50–60 μm in thickness and then electrolytic double spray milling using TenuPol-5 Double jet electrolytic thinning instrument with 25 V at approximately 25 °C.

Moreover, to characterize the microtexture for samples, electron backscatter diffraction (EBSD) analyses were carried out using a SEM (Sirion-200) equipped with an Oxford Instruments-HKL Channel 5 EBSD system at an accelerating voltage of 20 kV, a working distance of 15 mm, a step size of 0.3 μm and a sample tilt angle of 70°. The samples (10 mm × 5 mm × 4 mm) were cut along the ED. After grinding the samples with different granularity of sand paper, the samples were mechanically polished and electrolyzed. The solution for electrolytic polishing was AC2, polishing temperature was –10 °C and the working voltage was 20 V.

The tensile tests were performed at room temperature with a crosshead speed of 1.0 mm/min in an Instron 5969 tensile testing machine, and the extensometer length was 25 mm. Each test condition was repeated at least three times. After tensile tests, the fracture surfaces were examined on a SEM.

### 3. Results and discussion

#### 3.1. Microstructure of extruded alloys

XRD analysis in Fig. 1 reveals that the addition of 1% Sm promotes the precipitation of new second phases in the alloy. In as-extruded ZK60 alloy, only a small amount of Mg2Zn3 and Mg7Zn3 phases can be detected. With the addition of 1% Sm, the new characteristic peaks become evident at 41° and 66°, which correspond to Mg6Sm3 and (Mg, Zn)5Sm phases.

The microstructure of as-extruded ZK60 and ZK60-1Sm alloys exhibits distinctly fine grains in the ED-ND plane in Fig. 2, the ND is the normal direction. It shows that extrusion process results in a significant grain refinement because of DRX in the two alloys and the DRXed grains are distributed along the ED. Furthermore, the un-DRXed grains are broken into small particles after extrusion deformation and elongated along the ED. In Fig. 2a and b, the grain size of ZK60 alloy is not uniform, while the grain size is relatively uniform in ZK60-1Sm alloy. Calculated by the linear intercept method, the average grain size of ZK60 and ZK60-1Sm alloys is 5.81 μm and 3.67 μm, respectively. This phenomenon indicates that the grain size of the extruded alloy decreases obviously with the addition of Sm element. Other than that, a fragmentation of eutectic phases is aligned along the ED which in ZK60 and ZK60-1Sm alloys on extrusion process. SEM micrographs (Fig. 2c and d) show that the particles of second phases which mainly distributed along grain boundaries in ZK60 alloy are smaller and fewer than those in ZK60-1Sm alloy. What is more, there are many second phases which are not completely dissolved in ZK60-1Sm alloy, a new (Mg, Zn)5Sm phase in the ZK60-1Sm alloy is found in EDS analysis. However, due to the detection limit for SEM, TEM observations are needed to characterize the small amount of tiny precipitates.

As mentioned above, the composition, size and morphology of the second phase particles obtained in ZK60 and ZK60-1Sm alloys are different. In order to investigate the detailed structure information, the intermetallic compounds in the extruded samples were observed and analyzed by TEM. The phases and their characteristics of the investigated alloys studied are shown in Table 2. Fig. 3 shows the TEM observation micrographs of as-extruded ZK60 alloy. The bright field TEM image (Fig. 3a) shows that some precipitates are observed along the grain boundaries. In addition, some point-like, rod-shaped particle are also observed which distributed in Mg matrix. High-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) images along with the selected area electron diffraction (SAED) patterns were taken in Fig. 3b. The fast fourier transform (FFT) patterns indicate a triclinic crystal structure for the second phase, and this rod-shaped particle is identified to be Mg2Zn3 phase, in the top right corner of Fig. 3b. In addition, the low right corner of Fig. 3b displays the SAED patterns image with the incident electron beam parallel to [−100], the analysis shows that the point-like particle is also Mg2Zn3 phase. Fig. 3c and d show the TEM light field images along with SAED patterns and the

### Table 2 – The phases and their characteristics of the investigated alloys.

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Phases</th>
<th>Characteristics (structure and size)</th>
</tr>
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<tbody>
<tr>
<td>ZK60</td>
<td>Mg2Zn3, Mg7Zn3</td>
<td>Triclinic, 60–300 nm</td>
</tr>
<tr>
<td>ZK60-1Sm</td>
<td>Mg2Zn3, Mg7Zn3, Mg6Sm3</td>
<td>Cubic, 10–40 nm</td>
</tr>
<tr>
<td></td>
<td>Mg2Zn3, Mg6Sm3, (Mg, Zn)5Sm</td>
<td>Cubic, 20–200 nm</td>
</tr>
</tbody>
</table>

*Fig. 1 – XRD patterns of the as-extruded alloys: (a) ZK60; (b) ZK60-1Sm.*
EDS results recorded from the nanometer phase. By SAED, we can find that those fine dispersed ellipsoidal precipitates are Mg2Zn3 phases. From the EDS results, we can see that the precipitates are mainly composed of Mg and Zn elements, which also indicates a stoichiometry of precipitated phase near Mg2Zn3. In general, there are point-like and rod-shaped micron Mg2Zn3 phases and ellipsoidal nanometer Mg2Zn3 precipitation phases in the extruded ZK60 alloy.

The grain of ZK60-1Sm alloy are clearly observable in TEM micrographs in Fig. 4a. The rod-shaped and petal-like precipitates at grain boundaries are Mg2Zn3 phase, confirmed by using SAED patterns with the electron beam parallel to the [100]. The SAED pattern (h = [1–1–1]) of bulk eutectic phase (0.5 μm) in Fig. 4b indicates that it is similar to (Mg, Zn)2RE phase [21,22] in structure and is considered to be (Mg, Zn)3Sm phase, which has a cubic structure with lattice parameters of a = 0.736 nm. The EDS map scanning indicates bulk eutectic phase has the composition of 41.85 ± 0.98Mg–43.09 ± 0.83Zn–1.24 ± 4.98Zr–13.82 ± 2.01Sm, is also coincident with (Mg, Zn)3Sm phase. In the extrusion process, in addition to the second phase which is not dissolved into matrix is broken into fine particles, the elements in the matrix also precipitate dynamically with the decrease of solid solubility. A large number of precipitate phases (20–200 nm) are found in HAADF-STEM images (Fig. 4c). In the images, there are some high-contrast point particles and gray-white ellipsoidal nanoscale precipitates. Take the SAED patterns with the electron beam parallel to the [1–31], those high-contrast point particles are confirmed as Mg41Sm5 phase, which has the tetragonal structure (a = 1.477 nm, c = 1.032 nm) and good thermal stability. Moreover, gray-white ellipsoidal nanoscale precipitates were analyzed by high resolution transmission electron microscopy (HR-TEM) images (in Fig. 4d). Those precipitates are considered to be Mg2Zn3 phase after taking the digital FFT patterns, which displays an image with the incident electron beam parallel to [01–1]. In a word, in addition to Mg2Zn3 and Mg2Zn3 phases, micron (Mg, Zn)3Sm phases and nanoscale precipitation Mg41Sm5 phase were also formed, after an addition of 1 % Sm element.

In this experiment, the supersaturated solute elements precipitate dynamically in the form of the second phase, resulting in a large number of nanoscale Mg41Sm5 particles with high thermal stability, and large (Mg, Zn)3Sm particles breaking up in the extrusion process, which distribute along the ED. On the one hand, these second phase particles provide the substrate for DRX nucleation by particle stimulated nucleation (PSN) effect, promote the DRX process. On the other hand, according to “Zener effect” [23], this second phase particles can also pin dislocations, effectively hinder the movement of grain boundary during grain growth, and further refine the grain size. It is concluded that the refining of grain size is due to the DRX during extrusion and the inhibition of grain growth by the newly formed Sm-containing second phase particles. According to Hall–Petch formula [24], the fine grains would play an important role in enhancing strength, especially the YS. Meanwhile fine grains can contribute to improving plasticity. The addition of Sm atoms also contributes to solid solution enhancement.

Fig. 2 – Optical microstructure and SEM micrographs of as-extruded alloys: (a) and (c) ZK60; (b) and (d) ZK60-1Sm.
3.2. Microtexture of extruded alloys

In magnesium alloys, high density fine precipitates are reported to be as nucleation sites for recrystallized grains with random directions, which may lead to the weakening of recrystallized microstructure, PSN may contribute to texture modification of alloys [25]. It can randomize DRXed grain orientation and weaken basal texture in magnesium alloys. In order to further study the effect of adding Sm on the microstructure of extruded alloy, the experimental alloys were characterized by EBSD. Fig. 5 shows the EBSD microstructure reconstruction of the longitudinal section (along the ED) of the extruded samples. The upper-right corner shows the degree of recrystallization and the average grain size of the alloy. As can be seen from Fig. 5, both ZK60 and ZK60-1Sm alloys undergo DRX with an average grain size of 5.28 μm and 2.96 μm, respectively, which is not significantly different from the average grain size measured by linear intercept method (in Fig. 2). In addition, the percentage of DRX in ZK60 alloy is only 26.20 %. In ZK60-1Sm alloy, finer DRX grains can be observed, and the DRX ratio is up to 45.80 %. This indicates that the increase of fine precipitates with high density after the addition of Sm element is conducive to refine the microstructure of extruded alloy. However, ZK60-1Sm alloy shows obvious bimodal structure, with obvious differences in the size of deformation grains and DRXed grains. This will affect the average texture strength of the alloy.

In EBSD microstructure reconstruction, each color represents a crystallographic orientation, and the grain orientation is similar when the color is similar. Fig. 5a is mainly marked with blue grain. According to orientation identification analysis, the normal direction of {01–10} planes of most grains after extrusion is parallel to the ED. That is, {01–10} fiber texture is formed in the ZK60 sheet as the extrusion process progresses. In ZK60-1Sm alloy (Fig. 5b), the grains are mainly marked blue and green, that is, some grains are oriented <01–10>//ED and <−12−10)//ED. The results show that the addition of Sm is beneficial to obtain fine structure and weak texture. In addition, twins are not found in Fig. 5, which can exclude the effect of twins on the mechanical properties of the alloys.

Fig. 6 shows the inverse pole figure (IPF) of as-extruded ZK60 and ZK60-1Sm alloy along the ED. It can be found that the maximum texture strength of ZK60 alloy (Fig. 6a) is 26.82, and the ED is basically parallel to the crystallization direction.
Fig. 4 – The as-extruded ZK60-1Sm alloy: TEM micrographs (a) along with the corresponding SAED patterns, TEM micrographs (b) of the second phase along with the corresponding SAED patterns and the EDS analysis results, HAADF-STEM images (c) along with the corresponding SAED patterns and HR-TEM images (d) along with the digital FFT patterns.

<01−10>. However, in ZK60-1Sm alloy (Fig. 6b), the strongest texture strength is distributed between {01−10} and {−12−10} basal texture arc, and the fiber texture begins to weaken to 7.25. It can be seen that the addition of Sm plays an important role in the texture weakening of ZK60 alloy. Moreover, the addition of Sm can change the bond energy between Sm

Fig. 5 – IPF maps obtained by EBSD (The figure includes the average grain size and recrystallization ratio of alloys): (a) ZK60; (b) ZK60-1Sm.
Fig. 6 – Inverse pole figure (IPF) of as-extruded alloys: (a) ZK60; (b) ZK60-1Sm.

and Mg atom, change the bond energy of Mg-Mg atoms around Sm, promote the non-base plane slip, thereby weaken the base plane slip, and improve the plasticity of ZK60 alloy [26].

Fig. 7 shows the distributions of Schmid factors (SF) of as-extruded ZK60 and ZK60-1Sm alloys, which are calculated based on EBSD data. It is interesting that the SF increases from 0.074 to 0.165 with Sm addition. In addition, the distribution of the SF for the experimental alloys is very different, the value for ZK60-1Sm alloy is relatively uniform than that in ZK60 alloy. In addition, we know that, when the applied stress on the basal slip system with high SF, the dislocation slip is easier to start and then the alloy has good plasticity. On the contrary, the plastic deforming ability of magnesium alloy is poor when the interfacial slip system has low SF [26]. Obviously, the dislocation slip of ZK60-1Sm alloy is easier to start and the plasticity is better.

3.3. Mechanical properties of extruded alloys

The tensile stress versus strain curves for as-extruded samples (along the ED) are depicted in Fig. 8. The UTS, YS and EL drawn from these curves are also plotted in Fig. 9. It can be seen that Sm addition has an observable effect on comprehensive mechanical properties of the alloy. The UTS, YS and EL of as-extruded ZK60-1Sm alloy reach to 335 MPa, 269 MPa and 17.8 %, respectively, which increased by 11.7 %, 24.5 % and 26.2 %, respectively, compared to those in ZK60 alloy. This indicates that the addition of Sm element has a significant positive effect on the comprehensive mechanical properties of ZK60 alloy.

Fig. 7 – Distribution of Schmid factors in as-extruded ZK60 and ZK60-1Sm alloys.

Fig. 8 – Tensile stress-strain curves of as-extruded ZK60 and ZK60-1Sm alloys at room temperature.

Fig. 9 – Mechanical properties of as-extruded ZK60 and ZK60-1Sm alloys.
3.4. Discussion

The strengthening of as-extruded ZK60-1Sm alloy is primarily attributed to grain refinement, second phase particles and texture.

1) Grain refinement strengthening: The broken second phase particles of alloys after extrusion can be used as the substrate of DRX nucleation, which can promote the progress of DRX. Furthermore, it can effectively hinder the grain boundary movement in the process of grain growth. According to Hall–Petch formula, there is an inverse proportional relationship between the YS and the grain size of the alloy, so the grain refinement can improve the YS of the alloy. Assume that the extruded ZK60-1Sm alloy and ZK60 alloy have the same ε0 and Hall–Petch coefficients, which can be calculated by the following formula. The addition of 1 % Sm results in the increase and appreciation of the YS caused by grain refinement: \( \Delta \sigma = k(d_{ZK60-1Sm}^{-1/2} - d_{ZK60}^{-1/2}) \). For extruded magnesium alloys, the \( k \) is 173 MPa·μm^{-1/2} [27]. According to the average grain size in Fig. 5, grain refinement will theoretically increase the YS of ZK60-1Sm alloy by about 25 MPa, which is nearly half of the YS difference (53 MPa) between ZK60-1Sm and ZK60 alloy. It indicates that the grain refinement effect caused by the addition of Sm element plays an important role in the contribution of the YS.

2) Second phase strengthening: After extrusion, the second phase of ZK60-1Sm alloy, which was not completely dissolved in Mg matrix, was broken, with different sizes and zonal distribution along the ED. These high thermal stability micron (Mg, Zn)3Sm and nanoscale Mg41Sm5 phases can effectively promote nucleation, hinder grain growth, refine grain size and sharply increase the number of grain boundaries. When deformed by external forces, the stress coordination effect near the grain boundary can promote the non-basal slip system and inhibit the basal slip, effectively reduce the stress concentration at the grain boundary, hinder dislocation movement, and improve the strength of the alloy [28,29].

3) Texture weakening: The randomization of recrystallized grain orientation of extruded ZK60-1Sm alloy helps to improve the anisotropy, significantly weaken the texture strength and greatly improve the plasticity. Moreover, the average SF of extruded ZK60-1Sm alloy is higher than that of ZK60 alloy, the dislocation slip is easier to start, and ZK60-1Sm alloy has better plasticity.

4. Conclusions

In the present study, a new Mg-5.5Zn-0.8Zr alloy with high strength and moderate ductility was fabricated by Sm addition and conventional extrusion. The conclusions are summarized as follows:

1) The microstructure of as-extruded Mg-5.5Zn-0.8Zr alloy is mainly composed of \( \alpha \)-Mg, \( \alpha \)-Mg2Zn3 and \( \alpha \)-Mg4Zn3 phases. With the addition of 1 % Sm, the micron scale (Mg, Zn)3Sm phase and nanoscale Mg41Sm5 phase mainly distributed along the ED are generated. These fine second phases can effectively pin dislocations and block slip.

2) After extrusion process, the DRX occurs both in ZK60 and ZK60-1Sm alloys, and the grains are refined remarkably. The second phase is extruded into fine granular phase and distributed in a strip along the ED. With the addition of Sm, the recrystallization grain orientation of the alloy is randomized and the texture weakened, which is helpful to improve the anisotropy and plasticity of alloy.

3) The as-extruded ZK60-1Sm alloy exhibits excellent mechanical properties with UTS of 335 MPa, YS of 269 MPa and elongation to failure of 17.8 % at room temperature.

4) The addition of 1 % Sm can effectively improve the mechanical properties of as-extruded Mg-5.5Zn-0.8Zr alloy. The
stabilization method is mainly for the second phase strengthening, fine grain strengthening and rare earth elements weaken texture also play a role.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

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