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Microstructure and mechanical properties of Mg-2Sn-1.95Ca-0.5Ce alloy with different extrusion speeds

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Abstract
Effects of extrusion speeds (0.3, 1.2, 1.8 and 3.5 mm s⁻¹) on the microstructure and mechanical properties of the Mg–2Sn–1.95Ca–0.5Ce (TXCe220) alloy were analyzed by various detection methods in this article. The results have revealed that extrusion speed exerts a prominent influence towards grain size, recrystallization fraction and texture. As the extrusion speed increases, the microstructure characteristics transform from bimodal grain morphology to the near complete dynamical recrystallization (DRX). The tensile yield strength of TXCe220 decreased from 365 MPa to 180 MPa accompanied by the elongation increasing from 4.0 % to 19.4 % when the ram speed rises from 0.3 mm s⁻¹ to 3.5 mm s⁻¹. The high yield strength of low-speed extruded alloys should be mainly ascribed to the nano-scale Mg₂Ca with diameter of 30–80 nm precipitates, fine DRX grains and strong texture.

1. Introduction

In recent years, structural lightweight has gradually become one of the strategies for energy saving and emission reduction, and related research has been vigorously promoted by a large number of scholars. For example, metal matrix nanocomposites are regarded as promising materials in the aviation and automotive industries due to their high strength, high vibration damping capacity, excellent electromagnetic shielding performance, good recycle ability, large hydrogen storage capacity and biocompatibility. Based on these superior properties, magnesium alloys exhibit broad development prospects in the fields of aerospace, automotive industry, electronic 3 C and biomedical sectors. However, the insufficient independent slip systems at ambient temperature due to the hexagonal close-packed (hcp) crystal structure will severely reduce the formability of magnesium alloys, which limits the extrusion speed of as-extruded magnesium alloys. Generally, conventional commercial wrought alloys such as AZ61, AZ80 and ZK60 can only be extruded at die-exit speeds approximately 0.5–2.5 m min⁻¹, much lower than that of aluminum alloy. Low extrusion speed will reduce productivity and increase final product cost, therefore restricting the further application of extruded Mg alloys.

In the diversified systems of Mg alloys, Mg-Sn based alloys have become strong candidates for achieving sufficient extrudability and comprehensive mechanical performance simultaneously. For instance, Park et al. suggested Mg–7Sn–1Al–1Zn (TAZ711) specimen still had good surface quality at high exit speeds, and no surface cracks appear even at speed as high as 27 m min⁻¹. Cheng et al. developed a novel Mg–8Sn–1Al–1Zn alloy extruded at 250 °C and 2–10 m min⁻¹ which showed the moderate tensile yield strength (TYS) from 199 MPa to 244 MPa with the elongation from 14.8 % to 17.5 %. The high-speed extrudability can be explained by the existence of the high thermally stable Mg₅Sn phase (1043K). This thermally stable phase with suitable size...
and morphology can effectively limit the size of grains and prevent the occurrence of hot cracks during high-speed extrusion [10].

However, even if the extruded Mg-Sn based alloys exhibit excellent extrudability, the highly concentrated Sn (not less than 7 wt.%) still hinders their further applications, after all, Sn will result in poor cost effectiveness of mass producing such alloy. Therefore, it is challenging to maintain high extrudability even when the Sn content reduces. In this case, adding other suitable low-cost elements that can form thermally stable phases to low-tin-content Mg alloys is a promising choice. Ca is considered to be one of the most ideal elements to enhance the mechanical performance of Mg-Sn alloys as a result of the formation of CaMgSn, Mg2Ca or Mg2Sn phase [11–17]. It has been confirmed that the types of the secondary phases in the Mg-Sn-Ca ternary alloy systems mainly depends on the mass ratio of Sn to Ca: the ratio lower than about 2.5 forms CaMgSn and Mg2Ca particles; the ratio of 3 only forms CaMgSn particles; much higher Sn/Ca ratio like 5 will form CaMgSn and Mg2Sn particles [12, 13]. The mass ratio of Sn to Ca in this paper is unity. The Mg2Ca phase therefore may also be formed at the grain boundaries with the exception of the formation of CaMgSn phase. Both of these phases, Mg2Ca (987K) and CaMgSn (1457K) have high thermal stability, which can effectively pin the grain boundaries during the high-speed extrusion process, thereby refine the grain size and ameliorate the extrudability [12]. Nevertheless, the CaMgSn phase usually appears as coarse needle-like in the Mg-Sn-Ca system, which will deteriorate the pinning effect. Yang et al [18–20] found that some rare earth elements such as Ce, Y and Gd could refine the CaMgSn phase very well. Meanwhile, Cerium can also refine the as-cast grains by increasing compositional undercooling of the alloy. Therefore, this paper considers adding a small amount of Ce due to its refinement effect and low cost.

Obviously, extrusion speed is one of the key parameters in determining the recrystallization behavior and dynamic precipitation during the deformation process. At present, few scholars have studied about the influence of extrusion speed on the microstructure and mechanical properties of Mg-Sn-Ca alloy. Although Pan et al [21] had done about the influence of extrusion speeds on the Mg-2Sn-2Ca alloy, the research focused on the relatively slow die-exit speeds (0.3~1.3 m min\(^{-1}\)). Therefore, this work is designed to extrude Mg-2Sn-1.95Ca-0.5Ce (TXCe220) alloy at higher die-exit speeds of 0.45~5.25 m min\(^{-1}\) to understand the effects of extrusion speeds on the microstructure and mechanical properties of TXCe220 alloy.

2. Experimental procedures

Cast ingots of Mg-2Sn-1.95Ca-0.5Ce (TXCe220) alloy was prepared by melting commercial pure Mg, Sn, Mg-25 wt.% Ca, Mg-30 wt.% Ce master alloys in a well-type electric resistance furnace. The melt was held at 750 °C for 40 min in a mixed protective atmosphere, which was composed of CO2 and SF6 with a flow ratio of 40. Then the alloy liquid was stirred well and poured into a preheated steel mold at 250 °C for obtaining the desired cast. Prior to extrusion processing, the TXCe220 as-cast billets with 25 mm in diameter and 20 mm in length were homogenized at 500 °C for 20 h followed by water quenching. The as-homogenized billets were placed in a homemade die coated with graphite lubricant, kept at 350 °C for half an hour, and finally directly extruded with a 100-ton extrusion press. The extrusion ratio was set to 25 and the extrusion speeds were arranged as 0.3, 1.2, 1.8, and 3.5 mm s\(^{-1}\) (named RS-0, RS-1, RS-2 and RS-3, respectively), giving corresponding die-exit speeds of 0.45, 1.8, 2.7, and 5.25 m min\(^{-1}\), respectively. The extruded rods were cooled in air after exiting the die.

The microstructural details of the alloys in different states were characterized via optical microscopy (OM, Optec MDS400), x-ray diffraction (XRD), scanning electron microscopy (SEM, Hitachi S-4800) and transmission electron microscopy (TEM, FEI Tecnai G2 F30), where both SEM and TEM were equipped with
energy dispersive spectrometers (EDS). The samples used to test the macroscale texture were sawed from the extruded bars and ensure that the reflecting surfaces of each composition samples were orthogonal to extrusion direction (ED). The (0002) and (10−10) pole figures in this article were measured by XRD and then calculated by MTEX 3.5.0 software. The analysis method of the DRX fractions and average DRX grain size of the as-extruded rods was mentioned in the previous literature [22]. For microscopic analysis, the samples were ground by using two different abrasive papers (800 and 1200 grits) respectively, polished to mirror surface and finally etched in a nitric acid alcohol solution and a mixed picric acid solution (5 ml distilled water + 5 ml acetic acid + 2.2 g picric acid + 35 ml ethyl alcohol) successively.

Figure 1 presents the dimension of the national standard tensile specimen, and the diameter and the gauge length of these specimens was 2.5±0.5 mm and 25 mm, respectively, which was machined in the light of the GB/T 16865–2013 testing standard. All the tested specimens were cut from the middle sections of rods and the axis of samples was parallel to ED. The tensile experiments of the tested samples were carried on a computer-controlled universal testing machine (Instron-5569) at ambient temperature with a strain rate of 0.010−3 s−1. The tensile tests of specimen corresponding to each extrusion speed were repeated not less than three times for the purpose of guaranteeing the accuracy of final results. The microhardness (HV) tests were conducted by using the microhardness tester (HSV-1000) with load of 500 g. The corresponding calculation data came from the average

![Figure 2. OM (a) and SEM (b) results of TXCe220 alloys and respective EDS results: as-cast (a1 and b1) and as-homogenized (a2 and b2).](image-url)
value of scattered ten data points (the maximum and minimum values are removed from the total of 12 points) obtained by the tests.

3. Results and discussion

3.1. Microstructure

Figure 2 shows the representative OM and SEM images of as-cast and homogenized TXCe220 alloys. Compared with cast samples, the average grain size of the homogenized samples has a certain degree of increase. Moreover, DRX denotes the area percentage of recrystallized grains.

Figure 3. OM (a) and SEM (b) results of the as-extruded TXCe220 rods: RS-0 (a1, b1), RS-1 (a2, b2), RS-2 (a3, b3), and RS-3 (a4, b4). FRX denotes the area percentage of recrystallized grains.
it can be seen clearly on the micrographs that the phases in the cast samples mainly include a large volume of irregular phases distributed in the grain boundaries (point A, which are divorced eutectic phases), interior of the grains (point C), and some needle-like (point B and D) phases distributed in the $\alpha$-Mg matrix. These two phases can be preliminarily inferred as Mg$_2$Ca and CaMgSn based on the corresponding results of EDS in figure 2. After homogenization treatment, the Mg$_2$Ca phases are almost completely dissolved, while the other phases are partially dissolved in the matrix and distribute more uniformly. These secondary phase precipitates with a diameter larger than 1 micrometer can serve as nucleation sites for DRX grains in the process of extrusion based on a famous particle stimulating nucleation (PSN) mechanism [23–29].

Figure 3 illustrates the corresponding OM and SEM results of the TXCe220 alloys extruded at the four studied speeds. The extruded bars exhibit a representative structural feature which contains both the fine DRX grains and coarse elongated grains. Compared with the as-cast and homogenized billets, the DRX grain size of the as-extruded rods has been significantly refined. The detailed microstructural characteristics of the extruded rods with various extrusion speeds are listed in table 1. The different extrusion parameters significantly affect the degree of DRX and grain size as shown in figure 3. Specifically, the average size ($d_{DRX}$) and area fraction ($f_{DRX}$) of recrystallized grains of RS-0 alloy are estimated to be $\sim0.88 \mu$m and $\sim78$ %. With the increase of ram speed, the $d_{DRX}$ and $f_{DRX}$ of recrystallized grains increase gradually. When the ram speed reaches 1.8 and 3.5 mm s$^{-1}$, the microstructure almost achieves complete dynamic recrystallization ($\sim95.8$ % and $\sim97.6$ %), while the $d_{DRX}$ has grown to be $\sim2.19 \mu$m and $\sim3.75 \mu$m. The reason is that as the increase of ram speed, excess heat is generated during the extrusion process accounted by the prevailing plastic deformation as well as friction, resulting in temperature increment of respective billet. In fact, grain growth and dynamic recrystallization during extrusion both require thermal activation, thus the driving force to trigger grain growth and dynamic recrystallization will increase as the temperature of the billet increases [7]. In addition, with increasing ram speed, the volume fraction of the CaMgSn phase gradually decreases, while the size gradually increases. In this case, its pinning force on the

### Table 1. Microstructure characteristics and Mechanical properties of the as-extruded TXCe220 rods.

| Ram Speed (mm s$^{-1}$) | $f_{DRX}$ (%) | $d_{DRX}$ (µm) | Texture level (Max) | Mechanical properties |
|-------------------------|---------------|----------------|--------------------|-----------------------|
| 0.3                     | $\sim78.0$    | $\sim0.88$    | 7.169              | YS (MPa) 365, UTS (MPa) 374, EL (%) 4.0, HV 85.3 |
| 1.2                     | $\sim83.1$    | $\sim1.67$    | 5.834              | YS (MPa) 310, UTS (MPa) 311, EL (%) 6.4, HV 77.4 |
| 1.8                     | $\sim95.8$    | $\sim2.19$    | 3.305              | YS (MPa) 270, UTS (MPa) 277, EL (%) 15.6, HV 71.7 |
| 3.5                     | $\sim97.6$    | $\sim3.75$    | 2.535              | YS (MPa) 180, UTS (MPa) 220, EL (%) 19.4, HV 65.5 |

$d_{DRX}$ refers to the average size of the DRX grains in the alloy. YS, UTS, EL and HV refer to the tensile yield strength, ultimate tensile strength, elongation and Vickers microhardness, respectively.
grain boundary is also weakened. Combining with these reasons, the final grain size and $f_{\text{DRX}}$ increase with the increase of ram speed.

Figure 4 presents the representative macroscale pole figures in which the reflective surface of the alloy is orthogonal to extrusion axis. As seen from the pole figures, the extruded samples display rod textures with (0002) preferentially parallel to the ED, which is a classical circumstances in the as-extruded Mg alloy [30]. As the increase of ram speed, the texture intensities gradually decreases. Specifically, a maximum texture intensity of 7.169 is obtained in the RS-0 alloy. While the maximum texture intensity of RS-2 and RS-3 alloys are only 3.305 and 2.535, which reduce by 53.9 % and 64.6 % respectively compared to RS-0 alloy. It should be noticed from above observation results that the DRX area fraction increase with the ram speed increases. As we all know, in extruded magnesium alloys, DRX grains generally have weaker texture than unDRX grains due to the more randomized DRX grains [31].

In other words, the weakening of the texture as the ram speed increases can be attributed to the increased area fraction of DRX. Li et al [32] also suggested that the texture with $<10\bar{1}0>$ // ED of Mg-Ca alloy is significantly weakened due to a high degree of recrystallization.

TEM analysis is performed so as to clarify more details about the secondary phase particles in the alloys and the TEM photos of the RS-0 alloy are demonstrated in figure 5. As the figure 5(a) shows, there are many dispersed secondary phase particles distributed either in grain interiors or at grain boundaries uniformly in the DRX region. These numerous secondary phase particles can effectively oppose the movement of dislocations and pin the grain boundaries during plastic deformation, thereby increasing the yield strength of the alloy.
The low-magnification bright-field image (figure 5(b)) presents a small amount of sub-micron coarse blocky precipitation (represented by the red circle A) with the length of such coarse precipitates approximately 400 ∼ 800 nm. This representative sub-micron precipitate is determined to be CaMgSn phase combining derived from the results (figure 5(e), (f)) acquired from selected area electron diffraction (SAED) and EDS techniques. The enlarged bright filed image (figure 5(c)) shows the fine spherical nanometer scale secondary phase particles (indicated by blue circles) and exhibiting a relatively uniform distribution in the α-Mg matrix. The diameter of these globular precipitates is approximately 30 ∼ 80 nm. According to the high-resolution transmission electron microscope (HRTEM), the diffraction pattern obtained by fast Fourier

Figure 6. The XRD results of the TXCe220 alloy.

Figure 7. The tensile experiment results of the extruded TXCe220 alloys (inset: microhardness histogram).
transform (FFT) and the corresponding EDS result (figure 5(h), (i)), this representative nano-scale particle (indicated by the yellow circle B) can be identified as the Mg2Ca phase.

The XRD images of the TXCe220 alloy in casted, homogenized and extruded conditions are presented in figure 6. According to the XRD results, all diffraction peaks only contain α-Mg, CaMgSn and Mg2Ca, which confirms the results of the above EDS. In addition, this also demonstrates that adding 0.5 wt.% Ce to Mg-2Sn-1.95Ca alloy does not cause the formation of any new phases. After homogenization treatment, the peak intensities corresponding to CaMgSn and Mg2Ca decreased, which is in keeping with the results of the above observation.

3.2. Mechanical properties
Figure 7 illustrates the engineering stress-strain curves of the four extruded TXCe220 alloys and the respective mechanical property values are presented in table 1. As compared with the RS-1, RS-2 and RS-3, the low-speed as-extruded RS-0 rod owns the maximum strength and microhardness, with the YS, UTS, and HV of 365 MPa, 374 MPa, and 85.3, respectively. As seen in figure 7, it demonstrates that both the strength and microhardness of the as-extruded rods decrease continuously with the increase of ram speed, but their ductility has improved. Especially when the ram speed is from 1.2 to 1.8 mm s\(^{-1}\), the TYS of the RS-2 alloy does not decrease much, while the elongation is significantly improved (from 6.4 % to 15.6 %). The elongation of RS-3 alloy has been further increased to 19.4 %.

The high tensile strength of RS-0 alloy extruded at low speed can be mainly explained by grain refinement strengthening, texture strengthening and precipitation strengthening. First of all, there is an inseparable relationship between the material yield strength and the size of the grains in accordance with the classic Hall-Petch equation, which defined as

\[
s = s_0 + kd^{\frac{1}{2}}
\]

where \(s\) represents the material yield strength, \(s_0\) represents lattice frictional stress, \(k\) represents stress intensity coefficient and \(d\) represents the mean of grain diameter. It is easy to know that the yield strength is inversely related to the grain size [14, 22, 35]. With the grain size decreases, the number of grain boundaries will increase, which will impede the movement of dislocations more powerfully and bring about an increase in yield strength eventually [36, 37]. The RS-0 alloy extruded at 0.3 mm s\(^{-1}\) owns the finest DRX grain (~0.88 \(\mu\)m) among all researched alloys. Therefore, for RS-0 alloy, the contribution of fine-grain strengthening to strength is the largest. In addition, texture also plays a prominent role on YS of wrought Mg alloys. Through unidirectional deformation, grain growth or recrystallization (static and/or dynamic), these alloys usually form grains with a
preferred crystallographic orientation [10]. Such grains with a specific orientation will also increase the activation stress for the basal slip under tension along the ED and induce higher strength of the alloy. The pole figure (figure 4(a)) of RS-0 rod exhibit a common rod texture of as-extruded Mg alloys. As the ram speed increases from 0.3 mm s\(^{-1}\) to 3.5 mm s\(^{-1}\), the texture intensity level continues to decline, so RS-0 alloy has the most significant texture hardening effect. Moreover, the \(k\) value in equation (1) is also correlated with the texture intensity closely. Yu et al. [38] indicated that a strong texture intensity own higher \(k\) value than those with a weak one in Mg alloys. Therefore, the RS-0 alloy with the finest grains and the highest texture intensity exhibits the most significant improvement in strength by the fine grain strengthening mechanism.

Finally yet importantly, precipitation strengthening is another key factor that exerts influence on yield strength strongly. As shown in figure 5(a), RS-0 rod contains a large amount of tiny globular precipitates (mainly Mg\(_2\)Ca phase of 30 ～ 80 nm) distributed homogeneously in grain interiors, which can increase yield strength via impeding the movement of dislocations and causing dislocation pile-ups during extrusion process [39, 40].

The tensile elongation of the extruded TXCe220 rods increases from 4.0 % to 19.4 % as the ram speed improves from 0.3 mm s\(^{-1}\) to 3.5 mm s\(^{-1}\). Especially when the ram speed is from 1.2 mm s\(^{-1}\) till 1.8 mm s\(^{-1}\), the ductility has more than double. The significant improvement in elongation depends on the degree of dynamic recrystallization. It has been known that the area percentage of DRX increases along with increasing ram speed. RS-2 and RS-3 alloys are almost completely recrystallized. As shown in figure 4, the high proportion of DRX result in the lower texture intensity. In randomized grains, either basal or non-basal slip systems are more beneficial to be activated and the noted von mises criterion as a mechanical concept in elastoplastic mechanics would be fulfill easier. Therefore, the randomization of texture plays a crucial role on optimizing the ductility of the magnesium alloy [32].

3.3. Fracture behavior

Figure 8 presents four SEM micrographs of the surface morphology of the tensile fractured TXCe220 alloys. The tensile fracture surface of all alloys is rough and contains many dimples of different sizes, which is the distinctive feature of ductile fracture. As the ram speed increases, the small dimples on the fracture surface of the samples become deeper and the big dimples become denser. The existence of secondary phase precipitates can be seen at the bottom of the dimples. In RS-0 alloy and RS-1 alloy, it can be found that some second phase particles have produced microcracks. The reason is that the deformation in between of the secondary phase particles and matrix does not match during the tensile deformation process, microcracks will initiate on the interface firstly [41]. Then they will further gather to grow up and multiply quickly, and finally bring about fracture failure of the alloy. Although these second phase particles can actively obstruct the dislocation slide and heighten the strength, they are also easy to be broken during tensile process and turn into cracks sources, which result in a decline in the elongation-to-fracture eventually [7, 35, 42].

4. Conclusions

This study aims on investigating the specific performance of the microstructural characteristics and tensile properties of the Mg-2Sn-1.95Ca-0.5Ce alloy at different extrusion speeds. The conclusions in this article can be stated as follows:

1. With the ram speed increases, the dynamic recrystallization degree in the extruded alloys are getting higher and higher. The RS-2 and RS-3 alloys have almost achieved complete dynamic recrystallization, but the dynamic recrystallization grains have also been coarsened and the texture is also weakened.

2. RS-0 alloy owns the highest YS, UTS and HV of 365, 374 MPa and 85.3, respectively. RS-2 alloy shows the best overall mechanical properties with a YS of 270 MPa and EL of 15.6%. When the ram speed reaches the highest, the yield strength of the alloy has been reduced to a certain extent, but the elongation has been significantly improved. RS-3 alloy exhibits a maximum elongation of 19.4%.

3. The high absolute strength of the low-speed extruded alloy benefits from the ultra-fine grain size (∼0.88μm), dispersed nano-precipitates and strong texture. The coarsening of the DRX grains, growth of the secondary phase particles and the randomization of the texture are the main inducements for the decrease of the strength of the RS-3 alloy. The weakening of texture intensity owing to the increase in the degree of DRX can explain the improvement in ductility.

Data availability statement

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