Materials Research Express

PAPER

The influence of extrusion temperature on the structures and mechanical properties of Mg–Al–4Y alloys

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Keywords: Mg–Al–Y alloy, hot extrusion, texture deformation, mechanical properties

Abstract

In this study, Mg–Al–4Y alloys were successfully prepared via the melting and casting method. The alloys were then extruded at 450 °C, 500 °C, and 550 °C. The influence of the extrusion temperature on the microstructures and mechanical properties of the alloys was investigated. The results indicated that the Al1Y phase was coarse and agglomerated in the Mg matrix. A strong {0001} basal fiber texture was formed in the alloys extruded at 450 °C; meanwhile, the alloy grains were refined, and the Al1Y phase was distributed uniformly in the matrix; these combined occurrences dramatically enhanced the strength and hardness of the alloys. As the extrusion temperature was increased from 450 °C to 550 °C, the yield strength, tensile strength, and hardness of the alloys gradually decreased, but the ductility significantly increased. The yield strength reduced from 209.6 MPa to 197.8 MPa, the tensile strength reduced from 311.5 MPa to 271.1 MPa, and the hardness reduced from 77.5 HB to 62.2 HB, while the ductility increased from 2.9% to 15.0%. Based on the experimental results, the high strength and hardness of the alloys were mainly due to fine grain strengthening, second phase strengthening, and {0001} basal fiber texture strengthening.

1. Introduction

Magnesium (Mg) alloys are the lightest structural metal materials. Owing to their high specific strength and hardness, large elastic modulus, superior electromagnetic shielding, and damping performance, they are widely applied in several fields, including the aerospace and automobile industries [1, 2]. Within the family of traditional Mg alloys, the Mg–Al alloys were first applied in the casting industry and have been the most used so far [1, 3]. However, their low mechanical strength and poor elasticity hinder their further application [4, 5]. In addition, the low thermal stability of the easily formed Mg17Al12 also hinders its application, especially under high temperatures [6]. Therefore, Mg–Al alloys require the addition of other alloying elements, and the alloys have to be dramatically deformed to refine their grain size and improve their mechanical properties.

Rare-earth elements, as promising Mg–Al alloy supplement candidates, are considered to have great potential for enhancing the mechanical performance of the traditional Mg–Al alloys. In previous studies, adding rare-earth elements (including lanthanum (La), cerium (Ce), neodymium (Nd), gadolinium (Gd), dysprosium (Dy), yttrium (Y), and scandium (Sc)) to Mg–Al–RE alloys greatly promoted grain refinement via solution and precipitation strengthening and improved the strength and corrosion resistance of Mg alloys [1, 7, 8]. The formation of the Al–RE thermal stable phase suppressed the formation of the thermally unstable Mg17Al12 phase, thus improving the creep resistance [9]. Willbold et al [10] found that adding La and Ce effectively improved the corrosion resistance and elasticity of the alloy. Stanford et al [11] demonstrated that the addition of Y to AZ31 Mg alloys not only enhanced the alloy strength and elasticity but also weakened the {0001} basal fiber texture. The weak basal texture led to reduced yield strength and enhanced elasticity of rare-earth Mg alloys. Other previous studies [12, 13] have also investigated the influence of different Y amounts on the
2. Experimental methods

2.1. Alloy preparation
Industrial-level pure Mg (>99.92 wt.%) ingots, pure Al (>99.99 wt.%) ingots, and Mg-19 wt.% Y (>99.5 wt.%) alloy ingots were used as raw materials. Based on the required composition of Mg, a certain amount of Mg ingots was weighed and placed in a stainless-steel crucible. The crucible was placed in a resistance-type furnace, which alloy ingots were used as raw materials. To ensure the reproducibility and reliability of the results, each sample was measured three times, and the averaged value of three measurements was taken as the final result. The BH3000 Brinell hardness testing machine was used to measure the hardness of the castings and extrusions. The KingScan IV probe and testing software were used to measure the Brinell hardness. Each sample was measured three to five times, and the averaged value was taken as the final result. The Bruker D8-Advance x-ray diffractometer (XRD) was used to analyze the second phase, with a testing voltage of 40 kV, current of 40 mA, scan range of 20°–90°, and scan step of 0.2°/s. The Leica CM3000I metalloscopy and the Zeiss-Merlin 6035 compact scanning electron microscope were used to observe the alloy microstructure, and energy-dispersive x-ray spectroscopy was used to verify the element and phase types of each micro-phase. The cross sections of Mg–1Al–4Y alloy samples extruded at different temperatures were subjected to electron backscatter diffraction (EBSD) testing, and the EBSD data were analyzed using the program Channel 5 from the Oxford Instruments HKL software.

Table 1. The major chemical compositions (wt.%) of the Mg–1Al–4Y alloys.

| Element | Mg  | Al  | Y  |
|---------|-----|-----|----|
| Composition | 94.6 | 0.9 | 4.5 |

microstructures and mechanical properties of Mg–Al alloys. In these studies, the addition of rare-earth element Y greatly enhanced the mechanical performance of Mg–Al alloys mainly owing to three reasons: (1) The Y element and Al element formed the Al17Y phase, which strengthened the alloys. (2) The Y atom, as a point for heterogeneous nucleation, could lead to grain refinement. (3) The formation of the Al17Y phase suppressed the precipitation of the unstable Mg17Al12 phase. However, few studies on the microstructure deformation and mechanical properties of the Mg–Al–RE alloys in the transformation process.

Grain size has a profound impact on the mechanical properties of Mg alloys. The Hall-Petch relationship demonstrates that a finer grain size can dramatically enhance the strength of metal materials. This is because the Taylor coefficient (Kt) of Mg alloys is higher than those of other metal materials, which makes the fine grain strengthening effects more dramatic [14, 15]. In previous studies, the extrusion and rolling methods were typically used to refine the material grain size [16–18]. Among these methods, extrusion, as a highly efficient, high-quality, low-consumption precision casting method, has been widely adopted in the field of molding of metal materials. Process parameters such as extrusion temperature, speed, and ratio have profound impacts on the material structures and properties.

Hence, in this study, Mg–Al–4Y alloys were prepared using the melting and casting method, followed by hot extrusion. The mechanical properties of the alloy are significantly improved at room temperature. Meanwhile, the influences of the extrusion temperature on the mechanical properties, microstructures, and texture evolution were also investigated.
3. Experimental results and discussion

3.1. Microstructure analysis

Figure 1 presents the x-ray diffraction (XRD) spectra of the as-cast and extruded Mg–1Al–4Y alloys. The influence of extrusion temperature on phase composition was minor. The major phases in the Mg–1Al–4Y alloys were the α-Mg phase and the Al₂Y phase. In addition, as the extrusion temperature increased, no significant intensity change of each diffraction peak was observed, which indicates that the influence of the extrusion temperature on the alloy texture was also minor.

Figure 2 presents the metallographic microstructures of the (a) as-cast Mg–1Al–4Y alloys and the extruded Mg–1Al–4Y alloys at (b) 450 °C, (c) 500 °C, and (d) 550 °C.

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Figure 2 presents the metallographic microstructures of the as-cast Mg–1Al–4Y alloys and the extruded Mg–1Al–4Y at different temperatures. The black grains denote the Al₂Y phase, and the grey matrix denotes the α-Mg phase. Figure 2(a) shows that most of the Al₂Y phase was aggregated. Figure 2(b) shows that after
extrusion at 450 °C, the large coarse grains were broken into fine subgrains, which refined the overall texture. Additionally, the Al2Y phase was distributed uniformly in the Mg matrix along the ED, and the texture became more homogeneous. Figures 2(c), (d) indicates that as the extrusion temperature further increased to 500 °C and 550 °C, the condensed Al2Y phase was significantly refined, broken into finer grains, and distributed evenly around the crystallization boundary along the ED.

To further clarify the distribution of the precipitated phase, figure 3 presents the microstructures of the Mg–1Al–4Y alloys at different extrusion temperatures, characterized by scanning electron microscopy (SEM). As illustrated in figure 3(a), after extrusion at 450 °C, partially coarse precipitated Al2Y phase was distributed near the grain boundary along the ED in the Mg–1Al–4Y alloys. Moreover, partially fine Al2Y precipitated phase was distributed within the grains. As presented in figure 3(b), after extrusion at 500 °C, the coarse Al2Y precipitated phase became less, and the grain size became finer than it was after extrusion at 450 °C. As the extrusion temperature further increased to 550 °C, as presented in figure 3(c), the composition, as well as the size, of the large Al2Y precipitated phase further decreased. Additionally, as the extrusion temperature increased, the fine Al2Y phase composition decreased dramatically. This was mainly because the high extrusion temperature benefited the dissolution of the Al2Y phase into the Mg matrix, which is consistent with the results in figure 2.

3.2. Mg–1Al–4Y alloy EBSD analysis

Figure 4 presents the EBSD images and the corresponding misorientation angle distribution maps of the Mg–1Al–4Y alloys at different extrusion temperatures. As illustrated in figures 4(a) and (d), at 450 °C, the grains showed clear boundaries and orientations. The grains were also fine and flat, with an average size of 0.88 μm. The corresponding misorientation angles were concentrated at the low-angle grain boundaries, with a percentage of 69.1%. Such a phenomenon indicates that texture existed in the extruded alloys. As illustrated in figures 4(c), (d), as the extrusion temperature further increased to 500 °C, the grain size of the alloys gradually increased to an average value of 1.52 μm, and the corresponding misorientation angle transitioned from low-angle boundaries to high-angle boundaries. The percentage of low-angle boundaries dramatically decreased to 35.3%, and the percentage of high-angle boundaries remarkably increased to 60.5%. As illustrated in figures 4(e) and (f), as the extrusion temperature further increased to 550 °C, the grain size of the alloys increased to 2.92 μm, and the grains exhibited an equiaxed shape. In addition, the corresponding high-angle grain boundaries (HAGBs) increased to a percentage of 74.3%. Such a phenomenon indicates that as the extrusion temperature increased, the recrystallized structures were enriched in the alloys. This was due to the formation of HAGBs, which provided potential nucleation sites for dynamically recrystallized (DRXed) grains [19]. Additionally, the high temperature accelerated the migration and expansion of grain boundaries, which led to mutual melting and further growth of DRXed grains [20]. Hence, the higher extrusion temperature benefited the gradual growth of flat grains to form an equiaxed shape.

3.3. Mg–1Al–4Y alloy microtexture analysis

Figure 5 presents the microtextures of the Mg–1Al–4Y alloys at different extrusion temperatures. The ED and TD denote extrusion direction and transverse direction, respectively. As illustrated in figure 5(a), in the alloys extruded at 450 °C, the pole density points of the [0001] pole figure were distributed along the TD, and the strongest pole density points were distributed along the normal direction (ND) to the TD with an angle of approximately 40°. The corresponding pole density points of the [11–20] and [10–10] were distributed along the edge of the pole figures and shifted toward the center. This indicates that a [0001] basal fiber texture existed in the alloys; that is, the (0001) axes of most grains were formed perpendicular to the ED, and the texture strength was 9.02. The occurrence of the [0001] basal fiber texture was mainly due to the low critical shear stress
(CRSS) required to initiate the basal plane slip \[21, 22\] for Mg alloys, which led to the basal slip being the main process during extrusion. As illustrated in figure 5(b), in the alloys extruded at 500 °C, the pole density points of the \{0001\} pole figures were still distributed along the TD, but the distribution was dispersed. The corresponding pole density points of the \{11\–20\} and \{10\–10\} were distributed along the edge of the pole figures and both sides of the ED. This indicates that the \{0001\} basal fiber texture still existed. Moreover, the texture strength decreased dramatically to 4.49. That was due to the increased amount of recrystallized structure as a result of the higher extrusion temperature, which decreased the number of grains with preferred orientation and ultimately reduced the texture strength \[23\]. Such results are also consistent with figures 4(a)–(d). As the extrusion temperature was further increased to 550 °C, as presented in figure 5(c), the pole density points of \{0001\} pole figures were distributed along both sides of the TD. The pole density points of the \{11\–20\} and \{10\–10\} pole figures were distributed along the ED. This indicates that the \{0001\} axes of most grains were parallel to the TD, and their textures were still \{0001\} basal fiber texture. However, the texture strength increased abnormally. One possible explanation for such results could be speculated from figure 4(e). In the

Figure 4. The EBSD images and the misorientation angle distribution maps of the Mg–1Al–4Y alloys extruded at (a), (b) 450 °C, (c), (d) 500 °C, and (e), (f) 550 °C.
alloys extruded at 550 °C, the grains became larger; therefore, fewer grains were observed within a certain observation area.

3.4. Mg–1Al–4Y alloy mechanical property analysis

Figure’s 6 and 7 present the stress-strain curves and hardness values of as-cast and extruded Mg–1Al–4Y alloys, respectively. Their detailed mechanical properties are listed in table 2. The stress-strain curve of the as-cast alloy showed fair elasticity and weak hardening. Its ductility and hardness were 16.4% and 47.5 HB, respectively.
Three factors might contribute to such phenomena: strength, tensile strength, and hardness were increased to 209.6 MPa, 311.5 MPa, and 77.5 HB, respectively, and the ductility increased to 15.0%. These results were all connected to the higher extrusion temperature. Under the same extrusion ratio, a higher extrusion temperature accelerated the migration rate of the coarse Al2Y distributed near the grain boundaries hindered the grain basal slip, which led to enhancements in the yield strength, tensile strength, and hardness slightly reduced to 202.7 MPa, 274.7 MPa and 66.1 HB, respectively, and the ductility increased to 7.3%. When the extrusion temperature further increased to 550 °C, the yield strength, tensile strength, and hardness decreased to 197.8 MPa, 271.1 MPa and 62.2 HB, respectively, while the ductility decreased to 2.9%. Three factors might contribute to such phenomena: (1) grain refinement, (2) second phase distribution and (3) texture formation. First, the extruded alloys showed finer grains compared with the as-cast alloy (figure 4(a)). According to the Hall-Petch formula, finer grains lead to higher strength and hardness of a material [24]. Second, in the as-cast alloy, the Al2Y phase was coarse and aggregated in the Mg matrix (figure 2(a)). In the extruded alloys, the Al2Y phase was uniformly distributed in the Mg matrix (figure 3(a)). During extrusion, the fine distributed Al2Y hindered the misorientation movement. Meanwhile, the coarse Al2Y distributed near the grain boundaries hindered the grain basal slip, which led to enhancements in strength and hardness but a reduction in elasticity. Moreover, no texture was generally observed in the as-cast alloy, but the {10–10} basal plane and the {0001} prismatic plane experienced compressive and tensile stresses, respectively. Therefore, the {0001} (11–20) basal slip and the {10–11} (10–12) tensile twinning were hindered [22, 26], and the CRSS of the {10–10} (11–20) prismatic slip was lower than the {10–11} (10–12) compression twinning [27, 28]. Hence, prismatic slip was the major deformation format. The prismatic slip was also associated with a high CRSS, which further improved the alloy mechanical strength. As the extrusion temperature increased to 500 °C, the strength and hardness of the alloys decreased gradually, whereas the ductility improved. The yield strength, tensile strength, and hardness slightly reduced to 202.7 MPa, 274.7 MPa and 66.1 HB, respectively, and the ductility increased to 7.3%. When the extrusion temperature further increased to 550 °C, the alloy strength and hardness decreased slightly, whereas the elasticity improved dramatically. The yield strength, tensile strength, and hardness decreased to 197.8 MPa, 271.1 MPa and 62.2 HB, respectively, and the ductility increased to 15.0%. These results were all connected to the higher extrusion temperature. Under the same extrusion ratio, a higher extrusion temperature accelerated the migration rate of the grain boundary and increased the amount and size of the recrystallized grains (figures 4(c) and (e)). The higher extrusion temperature also accelerated the dissolution of the Al2Y phase in the matrix and reduced its composition (figures 3(b) and (c)). All these occurrences led to a decrease in the strength and hardness of alloys. In addition, the stress-strain curves became flatter as the extrusion temperature increased. The strain hardening rate also decreased, but the elasticity was significantly improved. This was because at 450 °C, un-recrystallized grains were the major components. A large amount of hard Al2Y phase also existed in the Mg matrix, which

| Material | Yield strength (MPa) | Tensile strength (MPa) | Ductility (%) | Hardness (HB) |
|----------|----------------------|-----------------------|---------------|---------------|
| As-cast  | 65.7                 | 152.8                 | 16.4          | 47.5          |
| Extruded | 450 °C               | 209.6                 | 2.9           | 77.5          |
|          | 500 °C               | 202.7                 | 7.3           | 66.1          |
|          | 550 °C               | 197.8                 | 15.0          | 66.2          |

Figure 7. The hardness of the as-cast Mg–1Al–4Y alloy and the alloys extruded at different temperatures.
made the stress concentrated with the texture and led to severe hardening during the extrusion process. The tensile samples then broke with low elasticity. When the extrusion temperature was increased to 500°C, the amount of recrystallized grains increased, and the average grain size increased. The softening of the recrystallized grains partially offset the hardening and increased elasticity. When the extrusion temperature was further increased to 550°C, recrystallized grains constituted the entire structure. The Al\textsubscript{5}Y phase composition further decreased, and the recrystallized grain softening was the major deformation format, which further improved elasticity.

4. Conclusions

Based on the experimental results, the following conclusions were derived from this study:

(1) In the Mg–1Al–4Y alloys, a substantial amount of Al\textsubscript{5}Y phase was present. As the extrusion temperature increased, the size and composition of the precipitated phase decreased.

(2) In the extruded Mg–1Al–4Y alloys, as the extrusion temperature increased, the alloy grains gradually transitioned from non-DRXed grains to DRXed grains, and the grain size gradually increased. Additionally, the \{0001\} basal fiber texture appeared in the extruded Mg–1Al–4Y alloys.

(3) In the extruded Mg–1Al–4Y alloys, as the extrusion temperature increased, the alloy yield strength, tensile strength, and hardness gradually decreased, but the elasticity increased dramatically. When the extrusion temperature was 450°C, the alloy presented the highest strength and hardness. The yield strength, tensile strength, and hardness were 209.6 MPa, 311.5 MPa, and 77.5 HB, respectively. When the extrusion temperature was 550°C, the alloy presented fair elasticity; the extensibility reached 15.0%.

Acknowledgments

This work was supported by the Qinghai Department of Science and Technology (Grant No. 2020-ZJ-707).

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