Effect of Heat Treatment on Mg-9Gd-3Y-0.3Zr Alloy Microstructure and Properties

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Abstract. The paper explored microstructure, mechanical properties, and fracture characteristics of the cast and extruded Mg-9Gd-3Y-0.3Zr alloys on the different heat treatment systems, analyzed the phase composition and the grain crystal growth of the investigated alloys. By the way of optimizing the heat treatment method, the mechanical properties of the alloys both at room and high temperature was Significantly increased. The research shows that the Vickers hardness of the cast alloys on T6 heat treatment system is obviously higher than that on T5 system. The second-phrase particles rich in Gd, Y and Zr, and the Zr elemental particles dispersing in the grains and its boundaries can effectively fine the grain crystal. As the temperature increases from room temperature to 300°C, the fracture mode changed from typical cleavage to mixture of quasi-cleavage and tough dimple. Meanwhile, the Vickers hardness of the extruded alloys on T5 heat treatment system is slightly higher than that on T6 system, though the tensile strength is significantly higher. The fracture characteristics of the extruded alloys change from cleavage initiated from the heart plus tough dimple surrounding to ductile fracture of microspore aggregation, as the temperature increases from room temperature to 300°C. On conclusion, the tensile strength of the Mg-9Gd-3Y-0.3Zr extruded alloys at 300°C is about 327MPa and suitable for high temperature resistant alloy.

1. Introduction

Magnesium alloy are widely used in aerospace, automobile industry due to its higher specific strength and stiffness than steel and aluminum. The cast magnesium alloy is mainly applied for engine block and housing, gearbox and gearbox casing, missile pods and seat frame, instrumentation bracket and housing, aircraft wing ribs and other parts. The extruded magnesium alloy is chiefly used to forge
various lightweight forgings, high-strength panels, cabin, siding, reinforcing frame, skin and other parts particularly in the spacecraft missiles, satellites and space shuttle. However, the defects of low resistance to high temperature, poor creep resistance, and poor stability limit its scope of application. Therefore, the research and development of high-strength heat-resistant magnesium alloy has become a hot topics.

Researchers have developed a variety of binary alloys such as Mg-Al, Mg-Zn and Mg-Re alloys, as also as some Mg-Gd-Y, Mg-Gd-Sc, Mg-Sc-Mn ternary alloys[1-4]. This paper explored microstructure, mechanical properties, and fracture characteristics of the cast and extruded Mg-9Gd-3Y-0.3Zr alloys on the different heat treatment systems, analyzed the phase composition and the grain crystal growth of the investigated alloys. By the way of optimizing the heat treatment method, we try to improve the mechanical properties of the alloys at room and high temperature.

2. Materials and methods

2.1. Alloy preparation

The binary Mg-Sc, Mg-Gd, Mg-Y master alloys, preheated to 250°C, rather than its simple metal were mixed into the magnesium melt which has been heated in graphite crucibles to 800°C. Then, the temperature should be held for at least 10 minutes. In the meantime, the mixed melt should be stir up for flux refining. To avoid burning loss, the whole preparation process should be in an inactive gas shield. After the fining, the alloy melt was poured into a pattern die preheated to 200°C and water-quenched quickly to achieve Mg-9Gd-3Y-0.3Zr alloys[5-7]. The microelement contents can significantly impact the alloy’s microstructure and properties. Consequently, reducing burning loss is of uppermost priority.

2.2. Heat treatment

The T6 and T5 heat treatment systems of as cast ingots were distinguished so that the relations between temperature, property and phase structure were explored. The as cast ingots in T6 systems were treated by different aging time at 250°C after solution treatment at 500°C for 6 hours. Meanwhile, The as cast ingots in T5 systems were treated by different aging time at 250°C artificially and directly.

The as cast ingots were homogenized at 500°C for 6 hours and then extruded at 420°C. The T6 and T5 systems of extruded alloys were also explored. The extruded alloys in T6 systems were treated by different aging time at 200°C, 225°C, 250°C, respectively, after solution treatment at 500°C for 2 hours. Meanwhile, The extruded alloys in T5 systems were treated by different aging time at 200°C, 225°C, 250°C, respectively, artificially and directly[8].

2.3. Characterization of microstructure

The conformation, distribution and size of inclusions as well as fracture surface cracking area of the Mg-9Gd-3Y-0.3Zr alloys were explored by means of optical microscope (OM) and scanning electron microscopy (SEM, Philips 505, Holland). Specimens for OM were polished, cleaned, dried and then etched with different etching agent according to different heat treatment systems[9]. The liner intercept method was used to measure the average grain size. Fracture surface were investigated by SEM. The specimen fracture surface was observed so that to characterize the initiation and propagation behavior of the alloy’s fatigue crack in the condition of high cycle fatigue. Meanwhile, an X-ray diffract meter (XRD) was used to scan the alloy’s phase composition.

2.4. Hardness and mechanical property

The hardness of the alloys were determined with Vickers’s hardness tester after grinding and
polishing, using a 49 N load and holding time of 30 s.

The tensile testing were performed using cylindrical specimen on SUN10 tensile (Italy), with the rate of 2 mm/min at room temperature, 200°C, 250°C, 300°C respectively.

3. Results and discussion

3.1. Microstructure and hardness

3.1.1. Cast alloys

Fig. 1 shows the hardness trends of cast alloys over time. The hardness significantly increased with increasing aging time both in T6 and T5 systems. However, the alloys’ hardness in T6 systems is higher than that in T5 systems. And the hardness peak, which appears after 12 h aging, is earlier. It can be inferred that the solution treatment in T6 system increases the solid solubility of microelement such as gadolinium(Gd), yttrium(Y) and zirconium(Zr). Meanwhile, with the increase of aging time, supersaturated solid solution shedding also increases the alloy’s hardness. The trend can also be confirmed through the microstructure.

Fig. 1 Trendy of hardness-time of the cast alloy

Fig. 2 Trendy of hardness-time of the extruded alloys

The crystalline grains of unheated treatment are almost all equiax crystal, while there are main dendrites, few secondary dendrites, inner each grain. Meanwhile, the size and arm spacing of the crystals are small, with a large number second-phase particles dispersing in the grains and its boundaries. After solution heat treating(500°C, 6h), the main dendrites inner each grain have disappeared while a significant number of twin crystals appear inner some grain. The numbers of the second-phase particles reduce markedly. Moreover, after aging heat
treatment (T6: 500°C, 6h; 250°C, 12h), there are more twin crystals appearance inner the grains, without the grain growth. However, after aging heat treatment (T5: 250°C, 12h) directly, without solution, the number of twin crystals is smaller, but the number of the second-phase particles is more than that of T6 systems.

The secondary electron phase of the cast alloys indicates that the particles are rich in Gd, Y. And there is a core particle, namely α-Zr Simple particle, at each grain center. These α-Zr particles are close-packed hexagonal structure presenting very similar lattice constant with α-Mg. according the size-structure matching principle, these α-Zr particles separate out primarily from the melt and become the core of heterogeneous nucleation of α-Mg. The MgGd₃ grains are also detected as reinforced phases. These MgGd₃ grains distributing at boundary or inner the grains can restrain its growth with the result of hardness increasing.

3.1.2. Extruded alloys

The crystalline grains of extruded alloys are almost all fine equiax crystal, while are deformed, broken under pressure, compared with the cast alloys. Meanwhile, dynamic recrystallization of the crystalline grains, because of the comprehensive effect of extrusion temperature and speed, reduce the grain size further.

The Vickers hardness of extruded alloys, shown in Fig.2, is higher than that of cast alloys. On the one hand, the grain crystals are refined to nearly 20um compared about 80um in cast, which enhances the hardness. Simultaneously, the grain boundary sliding resistance strengthened by the increasing grain boundary number enhances the hardness also. On the other hand, there are massive metastructure emerging during the extrusion deformation process, which strengthen the hardness. The highest hardness is about HV120, which occurs both in the T5 (200°C, 75h) and T5 (225,24h) systems. Considering economy, the T5 (225,24h) system is accepted. However, the hardness has a significantly reducing after solution heat treating because of the grain growth in T6 systems. Fig.3 shows microstructure of the extruded alloys in T6 (500°C, 2h; 225°C,24h) and T5 (225°C, 24h) systems. Compared with that of unheated alloy, the grain size increases obviously after solution treatment in T6. Meanwhile, the size aged directly in T5 is nearly same with that of unheated treatment alloys.

3.2. Mechanical and fracture property

3.2.1. Cast alloys

Table 1 displays the mechanical property at room temperature, 200°C, 250°C, 300°C of the cast alloys heated in T6. The Tensile strength decreases from 311.71Mpa to 267.15 as the temperature increases from room temperature to 300°C, while elongation from 4.8% to 11.64%.
Table 1 Mechanical property of the cast alloys

| Temperature | Room temperature | 200°C | 250°C | 300°C |
|-------------|------------------|-------|-------|-------|
| Tensile strength, σb/Mpa | 311.71 | 285.75 | 279.09 | 267.15 |
| Elongation, δ/% | 4.8 | 5.6 | 10.68 | 11.64 |

Micro-morphology, shown in Fig.4, indicate the fracture characteristic of the cast alloys. The fracture appearance is flattened with lots of white spots at room temperature. It is a typical cleavage fracture mode. The longitudinal cross-section shows that a mass of particles rich in lanthanon scatter inner the grain and its boundary. There are a few twin crystal at an angle of 45 degrees to the extruded direction. While the temperature increases to 200-300°C, the fracture appearance become more and more irregular, and a biconical with tearing edge and lots of cavities. As the temperature increasing, the quantity of the cavities become more and more, and the dimension larger. Beyond all doubt, the fractograph exhibited much more typical ductile dimple fracture pattern. However, there are still a few cleavage planes existing on the fracture surface, especially where there are second-phase particles. Altogether, a ductile-brittle mixed fracture mode rather than onefold leads to the the fracture characteristic of the cast alloys.

3.2.2. Extruded alloys

Table 2 displays the mechanical property at room temperature, 200°C, 250°C, 300°C of the extruded alloys heat treated by T5(225°C,24h), T6(500°C,2h,225°C,24h) respectively. The tensile strength of the extruded alloys heat treated by T5 are higher than that by T6. Meanwhile, the elongation have an even better performance. The trend can be proofed through the microstructure in Fig.8. The size of the grain in T5 is nearly 10-15um, thus, it perform more excellent mechanical
property than that in T6 with the size of about 30-50um. As the temperature increases from room temperature to 300°C in T5, the tensile strength decrease from 375.44MPa to 327.17, reduced by 12.86%, while elongation increases from 5% to 13.32%, increased by 166.4%.

Table 2 Mechanical property of the extruded alloys

| Temperature | Room temperature | 200°C | 250°C | 300°C |
|-------------|------------------|-------|-------|-------|
| Heat treatment system | T5  | T6  | T5  | T6  | T5  | T6  | T5  | T6  |
| Tensile strength, σb/Mpa | 375.44 | 349.63 | 364.01 | 336.00 | 329.21 | 327.68 | 327.17 | 314.17 |
| Elongation, δ/% | 5.00 | 4.06 | 8.98 | 5.85 | 12.23 | 8.28 | 13.32 | 9.38 |

Fig.5 displays the micro-morphology of the extruded alloys’ fracture. Different from the cast alloy, the fracture characteristics of the extruded alloys behave the similar ductile-brittle mixed fracture mode at room temperature. There are both cleavage planes and dimples, which are mutually inclusive on the fracture surface. The fracture trajectory at 200°C is similar with that at room temperature, which occurs from cleavage fracture in the center, and extends outward. However, there are more dimples and less cleavage planes at 200°C than those at room temperature. The common characteristics at 250°C and 300°C show a ductile fracture of microspore aggregation, which break due to dimples spreading begin on the grain boundary [9,10].

4. Conclusions

Since the grain crystals are fined after the alloys extruded, the hardness, tensile strength at room and high temperature, and elongation of extruded alloys are all higher than that of cast alloys. The heat treatment system has an important influence on the microstructure, mechanical and fracture properties of the alloys. The optimized heat treatment system of the cast alloys is solution at 500°C for 6 h, then aging at 250°C for 12 h. Meanwhile, the extruded alloys would better to age directly at 225°C for 24 h. The tensile strength of the Mg-9Gd-3Y-0.3Zr extruded alloys at 300°C is about 327MPa and suitable for high temperature resistant alloy.

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