ANALYSIS AND RESEARCH ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AEZ641 Mg ALLOY

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Abstract - The microstructure and mechanical properties of AEZ641 magnesium alloy were studied and analyzed using OM, SEM, XRD and tensile test. The results show that β-Mg17Al12 phase, a few Mg3Al18Mn2 phase and needle and/or block phases (Al3Nd, Al4Ce) in the as-cast alloy are transformed into broken needle and/or block (Al2Nd, Al4Ce) phases after extrusion preforming, while β-Mg17Al12 phase and Mg3Al18Mn2 phase are dissolved in α-Mg matrix; after die-forging, in addition to the broken (Al2Nd, Al4Ce) phases distributed along the die-forging pressure, β-Mg17Al12 phase and a few Mg3Al18Mn2 phases reprecipitated with a few Al10Mn7Ce2 phase. During the aging heat treatment of as-forged AEZ641 Mg alloy, the morphology and distribution of (Al2Nd, Al4Ce) phases in the alloy basically remained unchanged. When the heat treatment technology was 200℃×4h, the grain boundary of the alloy was refined, and β-Mg17Al12 phase was reprecipitated, and Mg3Al18Mn2 was dissolved in the matrix, which significantly improved the mechanical properties of the alloy. During the deformation process, the fracture mechanism of the alloy changes from as-cast brittle fracture to ductile-based ductile and brittle mixed fracture mechanism.

1.Introduction

Mg alloys meet the requirements of lightweight, miniaturization, high integration and environmental protection in automobile, aviation, electronics, military industry and other fields due to its outstanding advantages of low density, high specific strength, high specific stiffness, good electromagnetic shielding, damping and shock absorption, and easy recovery. Therefore, as a green engineering material in the 21st century, Mg alloy is increasingly favored by relevant scientific research and engineering personnel[1-4]. Compared with cast Mg alloy, wrought Mg alloy can produce various kinds of plate, tube, bar and forging products through deformation processing, and obtain products with better properties than cast Mg alloy through material structure control and heat treatment, so as to meet the needs of more structural products[5]. Relevant research shows that rare earth elements have high solid solubility in Mg alloy. After adding rare earth elements to Mg alloy, obvious strengthening
phase can be precipitated after deformation, and there is a certain phase relationship with Mg alloy matrix. Therefore, the plasticity of the alloy will be significantly reduced, which is not conducive to the improvement of the structure and mechanical properties of magnesium alloy[6-11]. There are many kinds of extruded Mg alloy products, which are easy to realize continuous and automatic production. However, the continuous network $\beta$-Mg$_{17}$Al$_{12}$ phase of $\alpha$-Mg grain boundary distribution in AZ system alloy forms crack source in the deformation process, which limits the improvement of plasticity of Mg alloy, and also limits the application of Mg alloy. Therefore, there are lots of researches on heat treatment technology of this kind of Mg alloy, which makes $\beta$-Mg$_{17}$Al$_{12}$ phase dissolves in $\alpha$-Mg matrix, forms single-phase saturated solid solution, improves the alloy performance. There are also many research reports on the influence of adding rare earth in the alloy on Mg-Al series Mg alloys, such as AZ31[12], AZ61[13], AZ81 and AZ80[14]. By adding rare earth elements and heat treatment, it can play a good solid solution and precipitation strengthening effect, there are few reports on the strengthening mechanism of wrought rare earth Mg alloy after extrusion-isothermal die forging composite forming. Therefore, the paper takes AEZ641 Mg alloy as research object, and studies the evolution of the structure and properties of the alloy after extrusion-isothermal die forging forming and heat treatment, and provides a certain technical reference for the further research and development of the structure and mechanical properties of deformed rare earth Mg alloy.

2. Sample Preparation and Experimental Method

The main raw materials are pure Mg ingot, pure Zn ingot, common Mn, Mg-30Nd master alloy and Ce rich mixed rare earth (85%Ce, 8%~12%Nd and a small amount of La). Melting in crucible, when the melting temperature reaches 720℃, add 2.8%Ce+1.2%Nd rare earth to the AZ61 Mg alloy solution. After mechanical stirring and Ar gas refining for 15min, let stand for 30min, and then cooling to 710℃ for continuous casting to obtain semi-continuous ingots. The obtained ingots were cut, and the surface layer were turned into extruded billets of Φ112mm×125mm. After solid solution treatment at 410℃×12h, the billets were extruded into plates by the 8000KN extruder, the extrusion ratio was 32:1 and the extrusion temperature was 410℃, The cross section of extrusion profile is shown in Fig.1. Then take a billet with a large volume blank equal to the final tensile sample for isothermal die forging. The die forging direction is perpendicular to the cross section of the extrusion profile. The isothermal die forging temperature is 420℃, the deformation speed is 17mm/s, the deformation degree is 60%, and the holding time is 2~3s. The tensile sample is formed by ASTM standard die forging as shown in Fig.1. The composition of the tested alloy is shown in Tab.1.

| Tab.1 Chemical composition of AZE614 Mg alloy |
|---------------------------------------------|
| Alloy | Chemical composition (wt%) |
| AZE614 | Al | Zn | Si | Fe | Mn | Ni | Re | Mg |
|       | 5.96 | 0.74 | 0.02 | 0.004 | 0.24 | <0.00 | 3.85 | Bal. |

Take the position as shown Fig.1 as the heat treatment sample with the size 6mm×3.5mm×6mm.
The artificial treatment temperature is 175℃, 200℃ and 220℃, and the holding time is 10min, 30min, 1h, 2h, 4h, 8h, 18h, 24h, respectively, then air cooling. After rough grinding, fine grinding and polishing, 5g picric acid+5g acetic acid+110ml ethanol were used as the metallographic corrosion solution. The microstructure and element analysis were carried out by OM, SEM and EDS, and the phase analysis was carried out by XRD after heat treatment. The hardness and mechanical properties were tested by hardness testing and universal tensile tester.

3. Experimental results and analysis

3.1 Change and analysis of microstructure

The microstructure of as-cast AEZ641 Mg alloy consist of α-Mg matrix and β-Mg12Al17 network phase with discontinuous distribution along the grain boundaries, and a large number of needle like, regular or irregular block rare earth phases can also be seen, which are mainly composed of Al3Nd, Al4Ce, etc.[13], the average grain size is about 47μm, as shown in Fig.2(a). After extrusion preforming, the alloy undergoes obvious dynamic recrystallization. Because the alloy has undergone deformation at high temperature and high extrusion ratios, no intermittent network β-Mg12Al17 network can be seen in the alloy’s microstructure, indicating that β-Mg12Al17 phases are broken or dissolved during the extrusion deformation, the grains were obviously refined but the grain size was uneven, the average gain size is 2μm, and the grain boundary was relatively coarse. The hardness and brittleness of the acicular and massive rare earth phases in the as-cast alloy are high, which are broken or rotated under 3-dimensional pressure, and the broken rare earth phases are distributed as shown in Fig.2(b); The Al3Nd phase in the as cast alloy is an unstable phase, which decomposes during high temperature extrusion deformation to form Al2Nd phase. It is found by XRD that the alloy is mainly composed of Al2Nd, Al4Ce, Al10Mn7Ce2, Mg3Al18Mn2, and there are also uncertain phases which may be Al2La phase, but in the die forging state and aging heat treatment process, the uncertain phase does not appear in the XRD, and the discontinuous β-Mg12Al17 phase in the as-cast state is dissolved in the Mg matrix, so it is not detected in XRD, as shown in Fig.5(a). After die forging, the extrusion fiber streamline disappears, the alloy undergoes a second dynamic recrystallization, the grain boundary are further refined, the crushed block or needle like second phase in the extrusion state is further crushed under the action of three-dimensional stress, and distributed along the direction of die forging deformation, as shown in Fig.2(c).

![Fig.2 Microstructure of AEZ641 Mg alloy in different states](image)

It can be seen from the macro morphology of as-forged AEZ641 Mg alloy that there are obvious white particles distributed along the die forging deformation direction, and the black areas are all α-Mg matrix, such as Fig.3(a). Enlarge the “L” area, as shown in Fig.3(b), a large number of broken particles and a small number of bulk phases are distributed on the Mg matrix. It can be seen from the Fig.3(c) which is enlarge the “S” area that there are a large number of broken particles, regular blocks, short rods and irregular small pieces or broken needle phases on the grain boundary or Mg matrix. These precipitates have obvious crushing traces. At the same time, circular particles are also observed, and the average grain size is about 1.47μm. EDS analysis is performed on the A, B, C, D and E positions in Fig.3(c), as shown in Fig.4, among which elements at positions A, C and F mainly include...
Mg, Al, Ce, Nd and Mn, and the atomic ratio of the elements is similar; elements at position B mainly include Mg, Al, Ce, Nd, while elements at position D mainly include Mg, Al, Nd; elements at position E mainly include Mg, Al, Nd, Mg, Al elements; therefore, it can be seen that A, B, C is a mixture of multiple phases, with the largest phase size of about 16.12μm. According to the XRD analysis of AEZ641 Mg alloy in Fig.5(b), the main phases in the alloy are Al2Nd, Al4Ce, Mg3Al18Mn2, Al10Mn7Ce2 and β-Mg12Al17; In Fig.3(c), the D region is mainly composed of β-Mg12Al17 and Al2Nd phases, and the “E” region is mainly composed of β-Mg12Al17, which indicates that β-Mg12Al17 precipitates again during deformation, which is also an important factor to improve the mechanical properties of as-forged Mg alloy; in the detected region, the minimum precipitated phase is about 0.15μm. No mesophase formed between Mg-RE was found in the alloy, according to Hume Rothery theory[15], Al is easy to form intermediate phase with Nd and Ce in priority, and then a small amount of Mg3Al18Mn2, Al10Mn7Ce2 and β-Mg12Al17 phases are formed with the surplus Mn, Al and unused Mg, Al and Ce in the alloy, while the formation of intermediate phase between Mg and rare earth is relatively weak, and no Mg-RE intermediate phase is observed in Fig.5. In addition, the solid solubility of Ce in Mg is smaller than that of Nd in Mg, so the precipitation kinetics is larger than that of Nd. Therefore, Ce is easy to form Al4Ce phase in Mg alloy, while Al4Ce phase is relatively brittle, and it is easy to form cracks and break in the process of three-dimensional stress deformation. The broken Al4Ce phase forms short needle or granular solid solution and magnesium matrix; as shown in Fig.5, in the XRD analysis of the extruded and die-forged states, Al1Nd phase in as-cast state is not found[13], the main reason is that Al1Nd phase is an unstable phase. After two high-temperature plastic deformations of extrusion and die forging, this phase decomposes and forms stable Al2Nd phase. However, it is believed in relevant literature that Al3Nd phase decomposes to form Al2Nd phase and Al11Nd3 phase, while Al11Nd3 phase is easy to decompose into Al2Nd and Al single phase[16] during high-temperature deformation, therefore, although β-Mg12Al17 dissolved during extrusion, a small amount of new Mg3Al18Mn2 phase precipitated. During high temperature die forging, not only Mg3Al18Mn2 phase precipitated, but also β-Mg12Al17 phase reprecipitated, and a small amount of Al10Mn7Ce2 phase formed. These phases play an important role improving the properties of the alloy. In addition, a small amount of La in the mixed rare earth, and the difference between the radii of La and Mg is more than 15%. Therefore, the solid solubility of La in Mg is very small, so no precipitation phase related to La is found in EDS and XRD analysis.

![Fig.3 SEM of as-forged AEZ641 Mg alloy](image-url)
Fig. 4 EDS of points A~F in Fig. 3 (c)

Fig. 5 XRD of AEZ641 Mg alloy

Fig. 6 shows the hardness change curve of as-forged AEZ641 Mg alloy after artificial aging heat treatment. With the increase of aging holding time, the hardness of the alloy rises to the peak in a oscillating way, and then decreases in a oscillating way. The main reason may be that although the grains of the alloy are obviously refined during the heat treatment, the rare earth phase, Al_{10}Mn_{7}Ce_{2} phase and dispersed β-Mg_{12}Al_{17} phase which precipitated at the beginning do not play a role of nail rolling, which makes the hardness of the alloy decrease at the initial stage (175°C). With the increase of the holding time, the shape of the precipitated phase in the alloy changes gradually from the massive rare earth phase to granular and/or short needle phase and point precipitated phase, which are distributed in the crystal or on the grain boundary along the stress direction, which leads to the distortion of the alloy crystal lattice, resulting in the strengthening of the solid solution of the alloy and the increase of the hardness. After aging treatment 200°C×4h, there is intermittent β-Mg_{12}Al_{17} phase in addition to the rare earth phase in alloy, the continuous network is distributed in the grain boundaries,
which hinders the movement of the dislocations and makes the hardness of the alloy reached the peak value. Then with the prolongation of holding time, the alloy grain grows, the grain boundary becomes coarser and $\beta$-Mg$_{12}$Al$_{17}$ phase precipitates and dissolves in the parent phase. At the aging temperature of (200℃, 220℃), there is no initial softening phenomenon. With the increase of aging temperature, the time of peak hardness becomes shorter; under the condition of a certain aging time, the hardness value increases with the aging temperature, and then decreases after reaching the peak value. In the aging heat treatment process, it was found that the best hardness heat treatment process is 200℃×4h.

![Fig.6](image1)

Fig.6 Hardness of as-forged AEZ641 Mg alloy in different heat treatment states

Select the best hardness sample of three aging process for microstructure comparison, as shown in Fig.7. After heat treatment, there are massive and/or rod like rare earth phases in the alloy. They are not dissolved in the magnesium matrix and do not precipitated like $\beta$-Mg$_{12}$Al$_{17}$ phase. After 175℃×18h aging heat treatment, the grain refinement is not obvious, and there is a certain lack of aging, resulting in a significant decrease in hardness compared with that of die forging samples, as shown in Fig.7(a); after 220℃×2h aging treatment, the grain size tends to increase, and the distribution of black particle phase is uneven, and there is a certain over aging. After aging treatment at 200℃×4h, the black point phase in the crystal or on the grain boundary distributes along the die forging deformation direction, with less block phase, obvious grain refinement and finer grain boundary, as shown in Fig.7(b).

![Fig.7](image2)

Fig.7 Effect of heat treatment on Microstructure of as-forged AEZ641 Mg alloy

Fig. 8 shows the SEM morphology of as-forged AEZ641 Mg alloy after aging treatment. It can be seen from the picture that there are a large number of white blocks, broken needle and round particles are distributed in the intragranular or grain boundary of the alloy. After the artificial aging heat treatment at 175℃×18h, there are a lot of massive and granular phases in the alloy. The grain boundary is relatively coarse, and the grain size is about 1.93 μm, as shown in Fig.8(a); after aging treatment at 220℃×2h, a large number of acicular particles are distributed in the alloy, and there is no obvious phase relationship with the magnesium matrix, the bulk phase is relatively small, and the alloy
The grain size is about 1.68μm, as shown in Fig.8(c); according to the XRD pattern in Fig.9(a) and Fig.9(c) and the EDS element analysis in Fig.8, only a small amount of β-Mg₁₂Al₁₇ and Mg₃Al₁₈Mn₂ were found in the alloy after heat treatment, and the distribution of rare earth phases is uneven in the SEM morphology of Fig.8(a) and (c), and the stress concentration of the alloy is caused by the aggregation of rare phases, so the harness of the alloy decreases significantly; after 200°C×4h heat treatment, there are many massive, broken acicular and granular phases in the alloy, but they are relatively uniform, there are more round particles on the grain boundary and in the crystal, with the grain size of about 1.45μm; according to the EDS analysis in Fig.9(c) XRD and Fig.8(b), it can be seen that in addition to Al₃Nd, Al₄Ce and Al₁₀Mn₇Ce₂ phases, the secondary phase β-Mg₁₂Al₁₇ precipitates in discontinuous network, the Mg₃Al₁₈Mn₂ phase is dissolved, and the grain boundary becomes finer, at the same time, the Al₃Nd and Al₄Ce phases are rare earth phases with high melting point and good stability. During the heat treatment process, dissolution or precipitation like β-Mg₁₂Al₁₇ and Mg₃Al₁₈Mn₂ will not occur. To some extent, the rare earth phase can hinder the diffusion of the phases of β-Mg₁₂Al₁₇ and Mg₃Al₁₈Mn₂, which leads to the lack of sufficient Al atoms in the phases of β-Mg₁₂Al₁₇ and Mg₃Al₁₈Mn₂ and stops further growth[17]. In addition, it is known that microhardness of the rare earth phase is higher than β-Mg₁₂Al₁₇ phase[19], which leads to the hardness of alloy increase.

3.2. Evolution of mechanical properties.
Fig.10 shows the mechanical properties of AEZ641 Mg alloy in different states. After extrusion-forging compound forming, the tensile strength σₘ, yield strength σₖ and elongation δ of AEZ641 Mg alloy increased by 47.6%, 137.2% and 86% respectively; after die forging forming, the mechanical properties of AEZ641 Mg alloy after heat treatment at 200°C×4h are best. The tensile strength σₘ and yield strength σₖ increased by 4.8% and 9.1% respectively compared with those of AEZ641 Mg alloy without heat treatment, while the elongation δ is basically unchanged. The main reason is that the dynamic recrystallization, grain size refinement, and the distribution of the crushed rare earth phases on the Mg alloy matrix or grain boundary under the action of three-dimensional stress during the extrusion-forging compound forming process of AEZ641 Mg alloy, as well as the
precipitation and dissolution of the second phase in the crystal or on the grain boundary, the change of the shape and the rotation the gain [18]. During the process from as-cast to as-forged, AEZ641 Mg alloy experienced two times of high temperature and large plastic deformation, the grains of the alloy occurred obvious dynamic recrystallization, and the grains were greatly refined, at the same time, under the action of 3-dimensional compressive stress, the rare earth phases Al$_4$Ce and Al$_2$Nd were crushed to form small block or short needle phase like A-RE phases, which were distributed on the matrix or gain boundary of the alloy, playing a role in the formation cracks, at the same time, β-Mg$_{12}$Al$_{17}$ and Mg$_3$Al$_{18}$Mn$_2$ peak phases were precipitated in the alloy after die forging forming. So the comprehensive mechanical properties of as-forged AEZ641 at room temperature were improved significantly.

![Fig.10 Mechanical properties of different states of AEZ641 Mg alloy](image)

After heat treatment at 200℃×4h, the Mg$_3$Al$_{18}$Mn$_2$ phase of as forged AEZ641 Mg alloy dissolved, retained the precipitation of β-Mg$_{12}$Al$_{17}$ and Al$_{10}$Mn$_7$Ce$_2$ strengthening phases, changed the lattice structure of magnesium matrix, at the same time, the rare earth phase also restricted the growth of magnesium matrix phase, made the grain refined, so its mechanical properties have been improved to some extent. After the heat treatment of 175 ℃×18h, there is a certain under age, so the properties of the alloy not only have not been improved, but also significantly decreased; after the heat treatment of 220℃×2h, the alloy grain has a growing trend, there is a certain over age, and the mechanical properties of the alloy also decline

3.3. Analysis and discussion on fracture morphology and mechanism

Fig.11 shows the microstructure of the fracture of as-forged AEZ641 Mg alloy after tensile test. It can be seen from the figure that the fracture of as-cast AEZ641 Mg alloy is a flat fracture 90°with the tensile direction, and the fracture surface shows obvious cleavage river patterns, and many small cracks can be seen. During the tensile process, the small crack growth causes the alloy to fracture, as shown in Fig.11(a); Fig.11(b) The macro fracture morphology of the extruded alloy. From this morphology, it can be seen that the alloy fracture has obvious extrusion flow lines and obvious necking. A small number of fracture cracks are observed at the edge of the alloy fracture. There is no obvious radiation area, and the cut lips are small; Fig.11(c) shows the macro morphology of the fracture of the die forging alloy, and it can be seen from the figure that the tensile fracture is zigzag at room temperature, with an angle of 45°with the tensile direction, with obvious necking, and the
fracture morphology is obvious die forging flow line, and it is obvious on the side of the fracture. Obvious crack, the crack is not fully welded during die forging forming, which leads to the crack source of alloy fracture. Fig.11(d)–(f) shows the fracture morphology of alloy after heat treatment of die forging alloy at different grain temperatures. After T5 heat treatment, the fracture has obvious necking characteristics and the crack source of specimen side not welded, and the distribution of small holes is obvious. Around the fracture, the cracks can be seen. The discontinuous intergranular regions are seen, and the intergranular regions are connected by tearing ridge.

Fig.11 Microscope fracture pattern of as-forged AEZ641 Mg alloy after T5 heat treatment

Fig.12 shows the micro fracture morphology of AEZ641 Mg alloy in different states. Fig.12(a) shows a large number of dimples in the fracture, and the number of dimples is more and deeper than that of the extrusion fracture, at the same time, there are a large number of white particles, short needle like particles and small particles of rare earth phases are distributed in the dimple of the fracture surface of the die forging sample, which are obviously more than that of the extrusion fracture surface. Although there are still a small amount of cleavage steps, they still belong to the mixed fracture of toughness and embrittlement. However, the cleavage composition of the fracture surface of the die forging AEZ641 Mg alloy is obvious after the artificial aging heat treatment at 175°C×18h. The fracture mechanism belongs to cleavage fracture with certain ductile fracture characteristics, as shown in Fig.12(d), after 200°C×4h heat treatment, the fracture morphology mainly presents the distribution of equiaxed dimples, and a large number of broken rare earth phase particles are distributed at the bottom of the dimple, while after 220°C×2h heat treatment, the fracture morphology of the sample...
shows the parabola dimple, and the alloy in two states. The fracture mechanism of fracture belongs to the characteristics of ductile fracture, as shown in Fig. 12(f).

![Fig. 12 SEM fracture morphology of AEZ641 Mg alloy in different states](image)

**4. Conclusion**

By analyzing the microstructure and properties of as-cast AEZ641 Mg alloy by extrusion and forging compound forming, the main conclusions are as follows:

1. In the process of alloy extrusion forging, the acicular and massive rare earth phases in the as-cast state are broken into small, short acicular or granular phases under the action of three-dimensional compressive stress. The main phases are composed of α-Mg, Al₂Nd and Al₄Ce, accompanied by a small amount of Mg₃Al₁₈Mn₂, Al₁₀Mn₇Ce₂ and β Mg₁₂Al₁₇ phases. After heat treatment of as-forged AEZ641 Mg alloy by different processes, the morphology of rare earth phase did not change significantly. However, after aging heat treatment at 200°C×4h, in addition to grain refinement and a large number of broken Al₂Nd and Al₄Ce rare earth phase distribution, there are a large amount of round particles of β-Mg₁₂Al₁₇ phase reprecipitated, while Mg₃Al₁₈Mn₂ phase dissolved, making the comprehensive mechanical properties of the alloy the best.

2. In the process of extrusion forging, the fracture morphology of AEZ641 Mg alloy changed from cleavage fracture in casting state to mixed fracture mechanism of toughness and embrittlement in die forging state; after heat treatment, the fracture mechanism of AEZ641 Mg alloy was ductile fracture.

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