

Microstructure evolution and mechanical properties of Mg-12Zn-2Y alloy containing quasicrystal phase fabricated by different casting processes

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Abstract: Although icosahedral quasicrystal phase (denoted as I-phase) has been verified as an outstanding reinforcing phase, the mechanical properties of quasicrystal-reinforced Mg-Zn-Y alloys fabricated by traditional casting processes are still unsatisfactory due to the serious segregation of intermetallic compounds. In this study, the microstructure and mechanical properties of Mg-12Zn-2Y alloy fabricated by different casting processes, including permanent mold casting, squeeze casting and rheo-squeeze casting with ultrasonic vibration, were systematically investigated and compared. The results show that massive, large-sized I-phase and Mg₇Zn₃ phase gather together in the permanent mold cast sample, while the squeeze casting process leads to the transformation of I-phase into fine lamellar morphology and the amount of Mg₇Zn₃ decreases. As to the rheo-squeeze casting process, when the ultrasonic vibration is exerted with power from 800 W to 1,600 W, the α -Mg grains are refined and spheroidized to a large extent, and the lamellar spacing of the eutectic structure is significantly reduced, accompanied by some tiny granular I-phase scattering in the α -Mg matrix. However, when the ultrasonic power continuously increases to 2,400 W, the eutectic structure becomes coarse. The best mechanical properties of the rheo-squeeze cast alloy are obtained when the ultrasonic power is 1,600 W. The microhardness, yield strength, ultimate tensile strength and elongation are 79.9 HV, 140 MPa, 236 MPa, and 3.25%, which are 44.1%, 26.1%, 25.5%, 132.1% respectively higher than the corresponding values of the squeeze casting sample, and are 47.6%, 44.3%, 69.8%, and 253.3% respectively higher than the corresponding values of the permanent mold casting sample.

Key words: Mg-Zn-Y alloy; quasicrystal; rheo-squeeze casting; ultrasonic vibration treatment

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1 Introduction

Magnesium alloy has the characteristics of low density, high specific strength and large elastic modulus, and is widely used in electronic communication, transportation, aerospace, and other fields [1-3]. However, compared with other cast alloys, thermomechanical properties and heat resistance of conventional cast magnesium alloy are lower, which limit the industrial application of magnesium alloy [4, 5]. Some researchers have studied the effect of the quasicrystal phase on the mechanical

properties of alloys. In 1993, Luo et al. [6-9] reported that the stable quasicrystal phase was found in as-cast Mg-Zn-RE (Y, Gd, Nd, La, Ce, etc) alloys, which exhibited some excellent properties such as high hardness, low interface energy, outstanding corrosion resistance and high temperature stability. However, when Mg-Zn-Y alloy was melted in an electric furnace and slowly cooled in air, the quasicrystal phases existed in the form of icosahedron and bulky intermetallic compounds as well as aggregated at grain boundaries, which is harmful to the improvement of mechanical properties [6]. It was reported that the small-sized and uniformly-distributed quasicrystals could improve the properties of alloys significantly [10]. Zhang et al. [11] reported that fine I-phase precipitates formed in the hot extrusion process of Mg-Zn-Y-Zr alloys, which provided an obvious strengthening effect on the alloys, the ultimate tensile strength was improved to 342 MPa when it was extruded at 400 °C. In as-extruded Mg-Li

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Zn-Gd alloys, the dynamic recrystallization in Mg-8Li-3Zn-0.6Y alloy induced the generation of I-phase, which greatly improved the properties of alloys [12].

Different from the thermoplastic forming process, the casting processes are widely used to produce complex castings with high quality, such as permanent mold casting, squeeze casting and rheo-squeeze casting [13]. Among them, the rheo-squeeze casting combines the advantages of traditional die casting and forging technology. During the rheo-squeeze casting, a semi-solid slurry is usually prepared by mechanical stirring, electromagnetic stirring or ultrasonic vibration processes, and then solidified and crystallized under pressure, so as to refine grains and gain a uniform alloy structure [14]. Among the above slurry-preparing processes, ultrasonic vibration has the characteristics of high efficiency, low cost and no pollution. It is widely acknowledged that the cavitation and acoustic streaming induced by ultrasonic vibration can remarkably refine primary grains. The intermetallic compound (δ -Al₄FeSi₂ phase) of hypereutectic Al-Si alloy with 2% Fe was refined effectively by the ultrasonic vibration, which was attributed to the effects of cavitation and acoustic streaming [15]. The eutectic Si of A356 alloy was refined when ultrasonic power was in a certain range, and coarsened with the further increase of ultrasonic power because of the affiliated heating effect [16]. Chen et al. [17] found that the ultimate tensile strength (UTS), yield strength (YS) and elongation of 2024 wrought aluminum alloy using the ultrasonic vibration were 372 MPa, 246 MPa and 8.5%, respectively, which were increased by 20.8%, 21.2% and 84.8%, respectively, compared to the traditional squeeze casting sample. Based on the above investigations, it can be concluded that ultrasonic vibration is one of the simple and effective physical methods to refine alloy grain, and improves mechanical properties of alloys.

In this work, the effects of different casting processes (permanent mold casting, squeeze casting and rheo-squeeze casting with ultrasonic vibration) on microstructure evolution and mechanical properties of Mg-12Zn-2Y alloy were investigated. Meanwhile, the solidification precipitation behavior of quasicrystals I-phase in these three casting processes was discussed.

2 Experimental procedure

The Mg-12Zn-2Y magnesium alloy was prepared by commercial pure Mg (99.9%, purity), pure Zn (99.9%, purity) and Mg-30Y (wt.%) master alloy. An ultrasonic vibration equipment (Scientz-J4T) for fabricating semisolid slurry was applied, which included a heating furnace, ultrasonic generator and ultrasonic vibrator. The maximum power of this equipment was 2,600 W and the frequency was maintained at 20 kHz. To prevent the rise of melt temperature caused by the thermal effect of ultrasonic vibration, the rest-work ratio R_t was set to 1, i.e., $R_t = T_r/T_w$, $T_r = 1.0$ s and $T_w = 1.0$ s. The JMatPro software was used to calculate the equilibrium phase diagram during solidification of Mg-12Zn-2Y alloy to determine the pouring

temperature, and the results are shown in Fig. 1. The pouring temperature of the permanent mold casting and squeeze casting was 640 °C, and for rheo-squeeze casting, the ultrasonic processing temperature was from 640 °C to 540 °C.

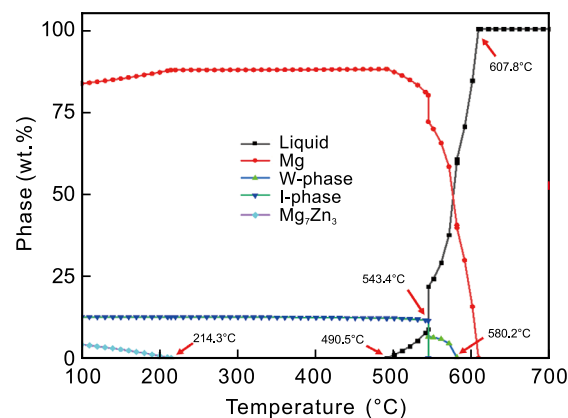


Fig. 1: Equilibrium phase diagram during solidification of Mg-12Zn-2Y alloy calculated by JMatPro software

Firstly, all the raw materials were put in a stainless-steel crucible with a well-type resistance furnace (SYDL SG2-5-12) for 30 min at 300 °C to remove water and oil. Then, in an atmosphere mixed with 1% SF₆ and 99% N₂, the temperature was increased slowly to 750 °C until the raw materials were completely melted, in which the argon was pumped into the melt for refining. Table 1 shows the five groups of Mg-12Zn-2Y alloy samples fabricated by permanent mold casting (I) squeeze casting (II) and rheo-squeeze casting (III-V), respectively. When the temperature of the melt decreased to 640 °C, the alloy of group I was poured into a metal mold preheated to 200 °C. The alloy of group II was poured into squeeze casting mold preheated to 200 °C at 640 °C, and was squeezed with the squeeze pressure of 50 MPa immediately. For groups III, IV and V, the 200 mL of melt was taken into a metal cup preheated to 600 °C (When the temperature difference between the melt temperature and the preheating temperature of the container is 20–50 °C, the ultrasonic effect is better, so the preheating temperature of the container is set at 600 °C). Then, the ultrasonic vibration head was inserted 10 cm below the surface of the melt. When the melt temperature decreased to 640 °C, the ultrasonic generator would work for nearly 80 s. During this process, the ultrasonic power was set to 800 W, 1,600 W and 2,400 W, respectively. Because the magnesium melt is easily oxidized, the atmosphere mixed 1% SF₆ and 99% N₂ was inlet to metal cup as the protective gas during the vibration process. Following, the melt was poured into an extrusion mold preheated to 200 °C, and was squeezed with the squeeze pressure of 50 MPa. After holding for 1 min, a cylindrical ingot with $\Phi 35$ mm \times 80 mm was obtained.

The specimens were cut from a position about 10 mm away from the bottom of the cylindrical ingots, then were grinded and polished and etched with 4vol.% nital. The microstructures were observed by an optical microscope (OM, Jiangnan MV-4000) and scanning electron microscope (SEM, ZEISS SIGMA

300) equipped with an energy-dispersive X-ray spectrum analyzer (EDS). The specimens were thinned by ion milling, then characterized by a transmission electron microscope (TEM, FEI talos200x). The phase constitution of specimens was detected by the X-Ray diffractometer (XRD, PANalytical

X-Pert PRO MPD). The tensile properties were evaluated using a Shimadzu AG-IC vertical electronic universal testing machine at the strain rate of $1.0 \text{ mm} \cdot \text{min}^{-1}$, and the hardness tests were carried out on a Buchler digital Vickers hardness tester.

Table 1: Details of five groups of Mg-12Zn-2Y alloy samples

Samples	Casting processes	Parameters		
		Ultrasonic power (W)	Squeeze pressure (MPa)	Pouring temperature ($^{\circ}\text{C}$)
I	Permanent mold casting	-	-	640
II	Squeeze casting	-	50	640
III	Rheo-squeeze casting	800	50	537
IV	Rheo-squeeze casting	1,600	50	538
V	Rheo-squeeze casting	2,400	50	536

3 Results and discussion

3.1 Permanent mold casting and squeeze casting

Figures 2(a) and 2(b) show the SEM images of Mg-12Zn-2Y alloy specimens fabricated by permanent mold casting and squeeze casting. It can be noted that the microstructure of the permanent mold casting alloy consists of coarse α -Mg and other phases with various morphologies (petaloid, grey and lamellar). In the sample by squeeze casting, the petaloid phase and grey phase disappear, and the content of α -Mg and lamellar eutectics increases. Meanwhile the lamellar eutectics

become more dispersive and the size of eutectic cells becomes smaller. As shown in the high magnification images [Figs. 2(c) and 2(d)], the eutectic lamellar spacing decreases in eutectic cells of the squeeze casting specimens. This spacing decrease is attributed to the higher cooling rate of squeeze casting over permanent mold casting.

The EDS analysis results of the permanent mold casting alloy [Points A, B, C, D in Fig. 2(c)] are shown in Table 2. The black part (Point A) is α -Mg matrix, in which a small amount of Zn is dissolved. In the petaloid phase (Point B), the ratio of Zn/Y is about 6:1, corresponding to the Zn/Y ratio of I-Mg₃Zn₆Y phase. The Mg/Zn ratio is close to 71.8:28.2 in

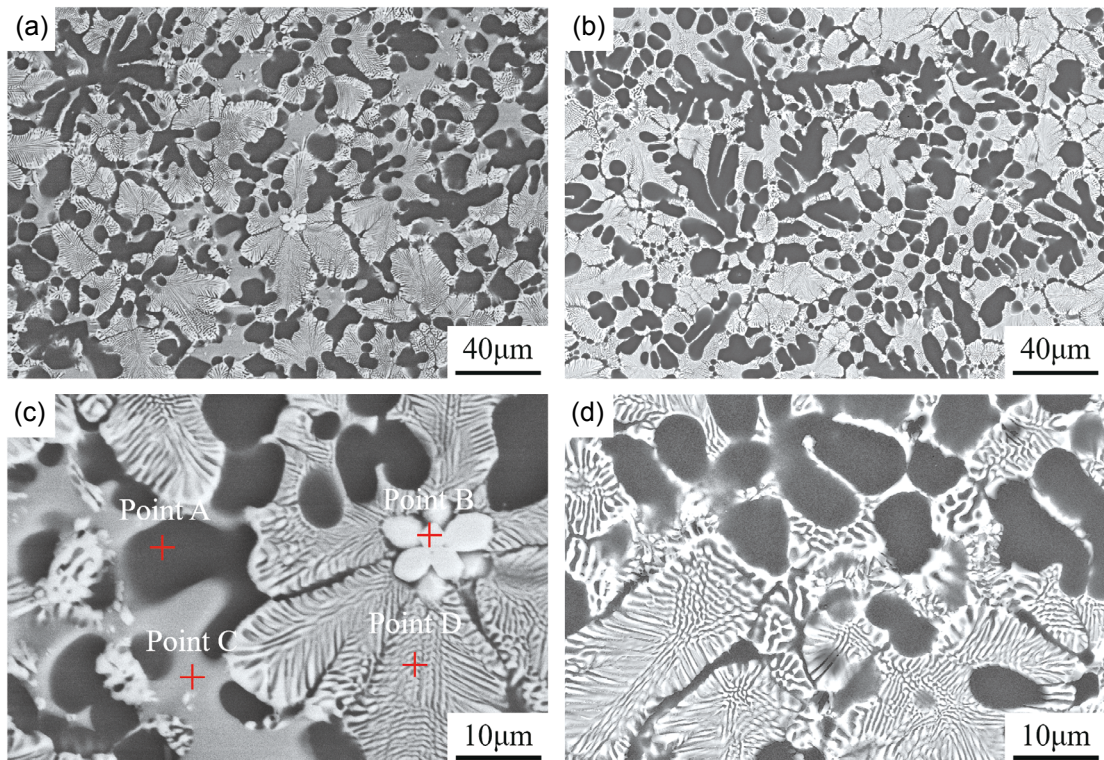


Fig. 2: SEM images of Mg-12Zn-2Y alloy fabricated by permanent mold casting (a, c) and squeeze casting (b, d)

grey phase (Point C), which is presumed to be Mg_7Zn_3 . The composition of lamellar eutectics (Point D) is Mg-26.5Zn-3.7Y , which has been identified with the α -Mg and I phase eutectic composition in another study^[18].

Table 2: EDS results of points in Fig. 2(c)

Detected points	Element composition (at.%)		
	Mg	Zn	Y
A	96.55	3.45	-
B	43.90	48.03	8.07
C	71.80	28.20	-
D	69.78	26.48	3.74

With regard to the petaloid phase, the composition is close to I-phase ($\text{Mg}_3\text{Zn}_6\text{Y}$). To further verify the petaloid phase, TEM image and diffraction analysis results are exhibited in Fig. 3. Figure 3(a) shows the diameter of petaloid phase is approximately $3.500\ \mu\text{m}$. Figure 3(b) displays the SAED pattern of the petaloid phase. It shows a typical selected area electron diffraction pattern at threefold symmetry, which verifies that the petaloid phase is an icosahedral quasicrystal structure.

The XRD patterns of the permanent mold casting and squeeze casting specimens are shown in Fig. 4, verifying that the gray phase is Mg_7Zn_3 , and demonstrating that Mg-12Zn-2Y alloy is mainly composed of α -Mg, Mg_7Zn_3 and I-phase. Meanwhile, the characteristic peaks of Mg_7Zn_3 phase decrease in squeeze casting, and the intensity of α -Mg and quasicrystal phase (I-phase) peaks increase, which accords with the results of the SEM microstructure images.

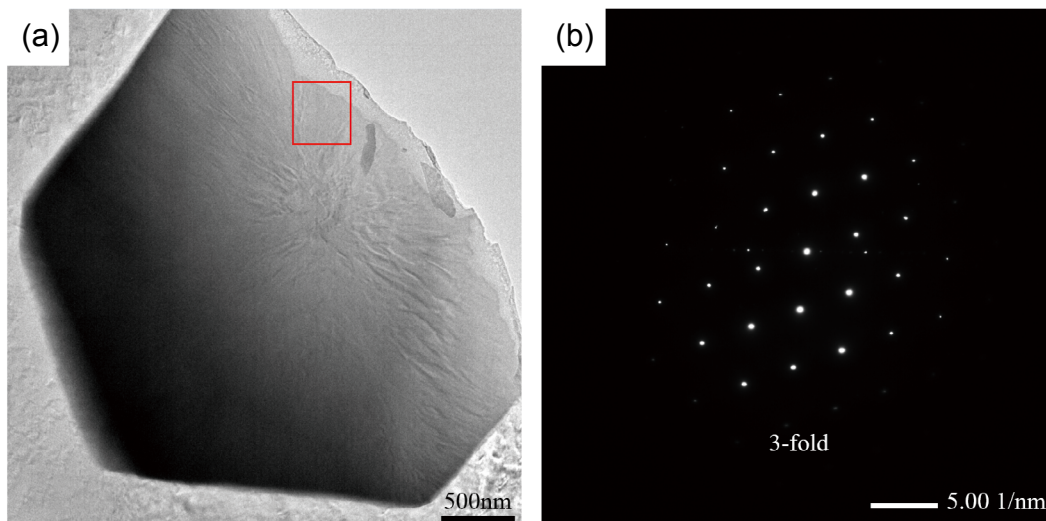


Fig. 3: Transmission electron microscope image of petaloid phase of Mg-12Zn-2Y alloy fabricated by permanent mold casting: (a) bright-field image of petaloid phase; (b) corresponding SAED pattern

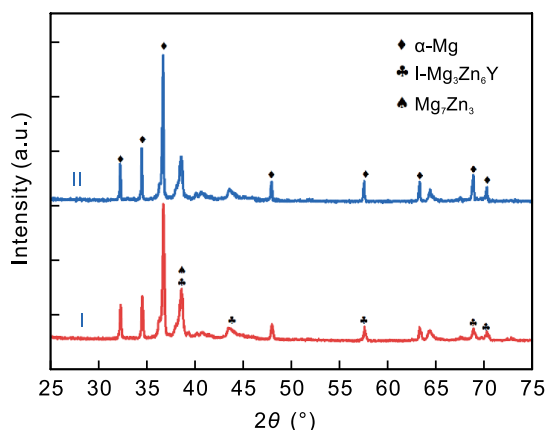


Fig. 4: X-Ray diffraction patterns of Mg-12Zn-2Y alloy fabricated by permanent mold casting (I) and squeeze casting (II)

3.2 Rheo-squeeze casting

Figure 5 is the SEM images of the microstructure for the alloys formed under squeeze pressure of 50 MPa after different

ultrasonic powers. When the ultrasound is applied, the α -Mg changes to nearly spherical grain. With the increase of ultrasonic power to 1,600 W, the size of α -Mg grain decreases gradually, and the lamellar eutectics distribute more uniformly along the boundaries of grain. Meanwhile, the spacing of lamellar eutectic diminishes, and some tiny particles appear in the α -Mg matrix. When the ultrasonic power is further increased to 2,400 W, the uniform distribution of eutectic structure at grain boundary becomes deteriorated.

For a clear comprehension of the tiny particles in the α -Mg matrix, TEM images and diffraction analysis were performed. Figure 6(a) shows the bright-field image of Mg-12Zn-2Y alloy treated by ultrasonic vibration with a power of 1,600 W. From the corresponding SAED pattern in Fig. 6(b), a representative diffraction pattern of 3-fold symmetry confirmed that the tiny particles are quasicrystal I-phase.

According to the mechanism of ultrasonic vibration, when ultrasound is introduced into the melt, the affiliated cavitation and acoustic streaming generate immediately^[19]. During the ultrasonic vibration process, the melt is affected by a periodic

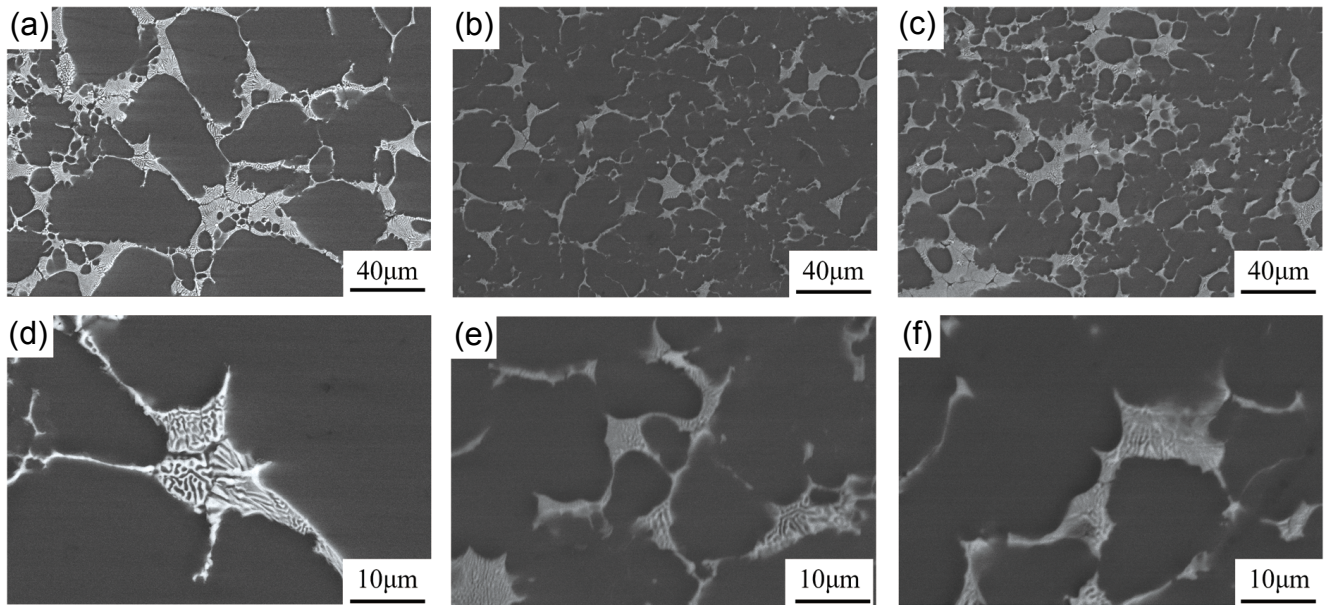


Fig. 5: SEM images of rheo-squeeze casting Mg-12Zn-2Y alloy with different ultrasonic powers: (a, d) 800 W; (b, e) 1,600 W; (c, f) 2,400 W

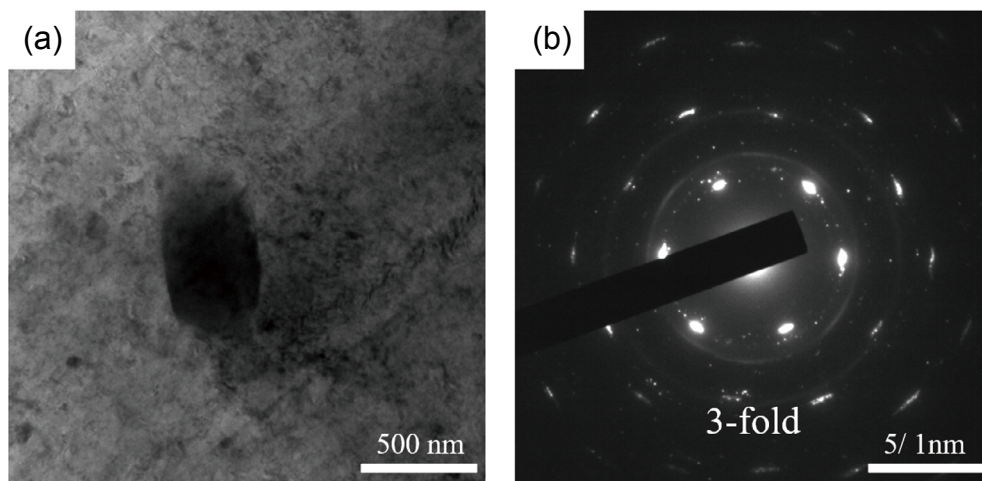


Fig. 6: TEM images of tiny particle in Mg-12Zn-2Y alloy treated by ultrasonic vibration with a power of 1,600 W: (a) bright-field TEM image; (b) SAED pattern

alternating ultrasonic field. During the stage of negative pressure, a large number of cavitation bubbles form and absorb a lot of heat, leading to the high undercooling in the area around the bubbles, which benefits the formation of crystal nucleus^[20]. Gao et al.^[21] found that the number of bubbles increased gradually with the intensified ultrasonic power. Furthermore, under compressive stress, the cavitation bubbles burst acutely and generate a high pressure, which effectively decreases the solidifying point of melt and results in the formation of numerous crystal nucleus^[22, 23]. In addition, the growing dendritic arms are broken by the high pressure, which become another source of nuclei^[24]. During the introduction of ultrasonic vibration, a certain gradient of amplitude attenuation is generated in the whole melt, which leads to an acoustic flow from the source in the direction of attenuation. Under the acoustic flow, the melt has a violent stirring and vibration effect, and the temperature of solid and liquid phases is more evenly distributed in the melt. Therefore, the growth

rate of grains in all directions is similar, which promotes the near-spherical growth of grains. Meanwhile, the acoustic streaming alleviates the enrichment of Zn and Y along the grain boundaries. In addition, since the average size of primary α -Mg is reduced, the number of α -Mg grains in the identical sectional area is increased, which contributes to the refinement of the compounds along the grain boundaries. Hence, it can be concluded that ultrasonic vibration is beneficial to the dispersive distribution of the secondary phase. However, when the ultrasonic power is increased to 2,400 W, part of the mechanical energy generated by ultrasonic vibration is converted into heat energy, and the undercooling degree of the melt is decreased, thus weakening the refinement of α -Mg grains, which is adverse to the dispersive distribution of the secondary phases. Hence, the appropriate ultrasonic power can promote the formation of crystal nucleus, leading to grain refinement.

The XRD patterns of Mg-12Zn-2Y alloy fabricated by rheo-

squeeze casting with different ultrasonic powers are shown in Fig. 7. The results show that the change of ultrasonic power has no conspicuous impact on the phase constitutions. All the alloys are mainly composed of α -Mg, I-Mg₃Zn₆Y phase, and a small quantity of Mg₇Zn₃. In addition, the magnified diffraction peak corresponding to α -Mg shifts to the right, which indicates the lattice constant of α -Mg phase is decreased with the increase of ultrasonic power. It can be deduced that the change of lattice constant is caused by the increase of solid solubility of Zn element in the matrix [25].

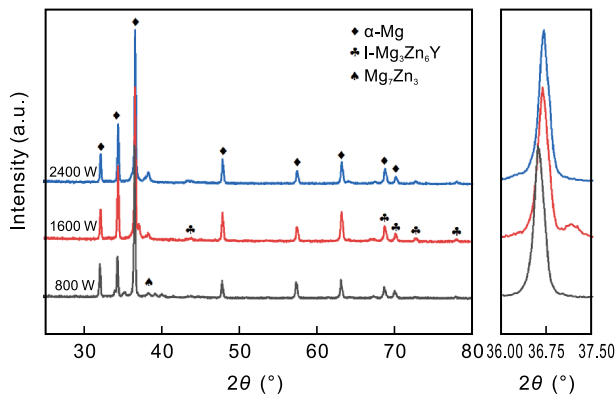


Fig. 7: XRD patterns of Mg-12Zn-2Y alloy fabricated by rheo-squeeze casting with different ultrasonic powers

3.3 Mechanical properties and fracture analysis

The microhardness of Mg-12Zn-2Y alloys treated by permanent mold casting, squeeze casting and rheo-squeeze casting under different ultrasonic powers is shown in Fig. 8. Compared with permanent mold casting, the microhardness of the squeeze casting alloy is increased by 11.6%, reaching 56.6 HV. With the increase of ultrasonic power, the microhardness of the alloys increases to a maximum of 79.9 HV at 1,600 W, which is 41% higher than the 56.6 HV of the sample with squeeze casting. However, when the ultrasonic power is further increased to 2,400 W, the microhardness of the alloy decreases, but it is still higher than that of 800 W. It can be seen that

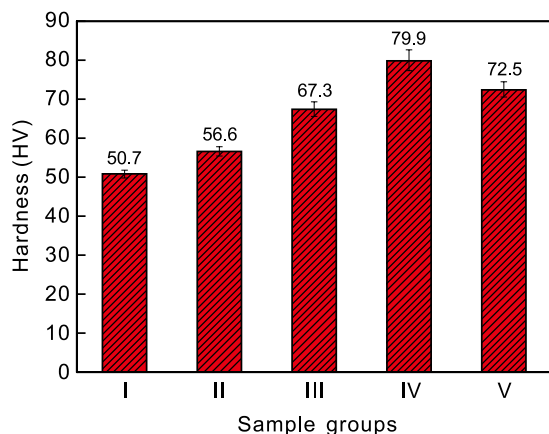


Fig. 8: Microhardness of alloys treated by different casting processes shown in Table 1

within a certain range of ultrasonic power, the introduction of ultrasonic vibration improves the microhardness of the alloy. However, when the ultrasonic power increases to 2,400 W, excessive mechanical energy is converted into heat energy, which leads to the decrease in cooling rate of subsequent squeeze casting. In this case, the uniform distribution of eutectic structure at the grain boundary is deteriorated [as shown in Fig. 5(c)], thus leading to the decrease of alloy hardness.

Figure 9 shows the tensile properties of Mg-12Zn-2Y alloys fabricated by different casting processes at room temperature. The yield strength, ultimate tensile strength and elongation of the squeeze casting samples are 111 MPa, 188 MPa and 1.41%, respectively. Compared with the permanent mold casting sample, the yield strength, ultimate tensile strength and elongation are improved by 14.4%, 35.3% and 52.2%, respectively. In specimens fabricated by rheo-squeeze casting, when the ultrasonic powers are 800 W and 1,600 W, the tensile properties improve obviously. And when the ultrasonic power is 1,600 W, the yield strength, ultimate tensile strength and elongation values are 140 MPa, 236 MPa and 3.25%, which are increased by 26.1%, 25.5% and 132.1%, respectively, compared with the samples of squeeze casting. However, when the ultrasonic power increases to 2,400 W, the yield strength, ultimate tensile strength and elongation decline slightly. Therefore, it can be inferred that the optimal ultrasonic power is 1,600 W for rheo-squeeze casting Mg-12Zn-2Y alloy.

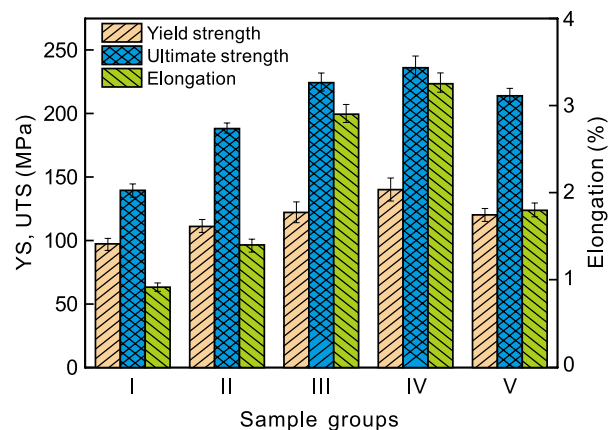


Fig. 9: Tensile properties of Mg-12Zn-2Y alloy fabricated by different processes

To research the fracture mechanism, the fracture surfaces SEM images of the squeeze casting and rheo-squeeze casting Mg-12Zn-2Y alloy are shown in Fig. 10. Without ultrasonic vibration, the whole fracture surface is dominated by bulky cleavage planes, which indicates the characteristic of cleavage fracture. With the ultrasonic vibration power increases to 1,600 W, an increasing number of cleavage steps are gradually replaced by small-sized dimples. However, the number of small dimples decreases significantly with the power increases to 2,400 W. Therefore, the fracture type of rheo-squeeze casting Mg-12Zn-2Y alloy is a mixture of cleavage fracture and ductile fracture.

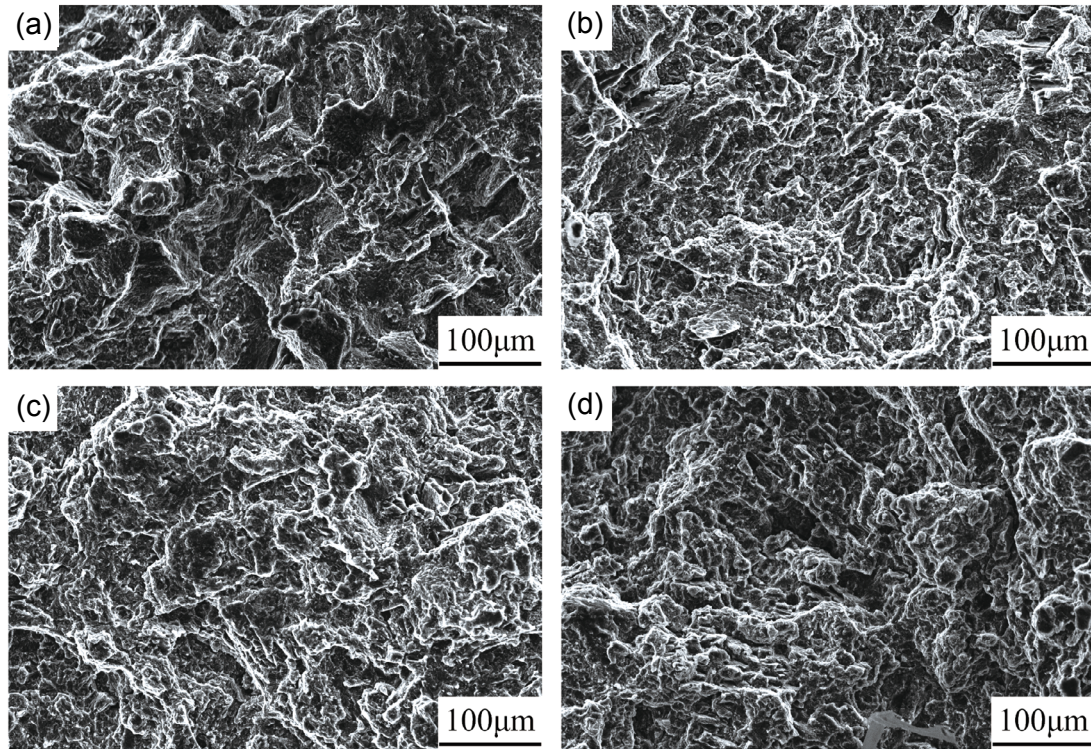


Fig. 10: SEM images of microstructure of tensile fractures for Mg-12Zn-2Y alloy fabricated by squeeze casting (a) and rheo-squeeze casting with different ultrasonic powers: (b) 800 W; (c) 1,600 W; (d) 2,400 W

Similar to the other investigations, the adoption of the rheo-squeeze casting process contributes to the simultaneous improvements in the tensile strength and elongation^[26, 27]. There are three main reasons that account for the improvements. Firstly, the samples fabricated by squeeze casting and rheo-squeeze casting have higher compactness than permanent casting, which results in simultaneous improvement in strength and elongation^[14]. Secondly, as depicted above, the introduction of ultrasonic vibration leads to the reduction in the size of α -Mg grains, and the refinement of microstructure is widely confirmed as an effective way to improve the strength and elongation at the same time^[26]. Thirdly, the morphology, size, and distribution of intermetallic compounds, especially the quasicrystal I-phase, also play a significant role in the mechanical performances. For the permanent cast Mg-12Zn-2Y alloy, the concentration of massive coarse icosahedral Mg-Zn-Y phases leads to stress concentration, offering sources of micro-cracks and causing the decline in the ductility^[13]. As to the rheo-squeeze casting sample, the coarse icosahedral quasicrystal disappears and lamellar eutectic structure is refined markedly. Meanwhile, some of the I-phase transform to tiny and dispersive particles in the matrix. The interface layer between α -Mg and I-phase particles maintains the coherency relationship because of the periodical steps and ledges along the interface^[7]. In addition, there is rigid atomic bonding between I-phase particles and hexagonal structure, which can effectively hinder the movement of dislocation, thus contributing to the improvement of tensile properties^[18]. Therefore, the elongation of Mg-12Zn-2Y alloy increases remarkably.

4 Conclusions

The microstructure and mechanical properties of Mg-12Zn-2Y alloy fabricated by permanent mold casting, squeeze casting and rheo-squeeze casting were systematically investigated and compared. The main results can be summarized as follows:

(1) The microstructure of the permanent mold cast sample consists of coarse α -Mg, Mg_7Zn_3 , petaloid icosahedral quasicrystal phase (I-phase) and lamellar eutectics (α -Mg and I-phase). For squeeze-cast alloy, the petaloid I-phase disappears and the amount of Mg_7Zn_3 phase reduces.

(2) In rheo-squeeze casting microstructure, the α -Mg changes from large dendritic crystal to nearly spherical grains. With the increase of ultrasonic power from 800 W to 1,600 W, the size of α -Mg grain decreases gradually, and the lamellar eutectics distribute more uniformly along the boundaries of grain. Meanwhile, the spacing of lamellar eutectic diminishes, and some tiny quasicrystal I-phase particles appear in the α -Mg matrix. However, when the ultrasonic power increases persistently to 2,400 W, the uniform distribution of eutectic structure at grain boundary is deteriorated.

(3) The microhardness, yield strength, ultimate tensile strength and elongation of squeeze casting alloy are 56.6 HV, 111 MPa, 188 MPa and 1.41%, which are 11.6%, 14.4%, 35.5% and 52.2% higher than the corresponding properties of permanent mold cast alloy. For rheo-squeeze casting alloy, when the ultrasonic power is 1,600 W, the microhardness, yield strength, ultimate tensile strength and elongation are the best, reaching 79.9 HV, 140 MPa, 236 MPa and 3.25%, respectively.

Acknowledgements

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