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ARTICLE

Effect of Yb and Zr Addition on Microstructure and Mechanical Properties of Al7Si0.3Mg Alloy

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Abstract: The effects of adding Yb and Zr elements on the microstructure and mechanical properties of Al7Si0.3Mg alloy and the mechanism of refinement and modification were investigated. The results show that the addition of Yb and Zr elements can significantly refine the α-Al matrix, transforming it from coarse dendritic crystals to fine petal-like crystals with a significant reduction in grain size, and the eutectic silicon metamorphoses from coarse needle-like to short rod-like. The modification mechanism of eutectic silicon is that the adsorption of Yb and Zr at the twin grooves (TPRE) changes the eutectic silicon growth mode and finally changes the eutectic silicon morphology. After heat treatment (T6), a large number of Al_3(Yb, Zr) particles are precipitated to strengthen and refine the grain, and the addition of Yb and Zr can significantly improve the mechanical properties of the alloy. The tensile strength and elongation of the alloy (T6) are 296.3 MPa and 9.2% when the Yb content is 0.3wt% and the Zr content is 0.25wt%, which are 17% and 1.1 times higher than those of the unrefined and unmodified alloy, respectively.

Key words: Al7Si0.3Mg; Yb; Zr; microstructure; mechanical properties; mechanism of refinement and modification

Aluminum-silicon casting alloys are very versatile, and eutectic alloys containing silicon are the most fluid alloys of the casting aluminum alloys which can improve strength and wear resistance. This alloy has low density, low casting shrinkage rate, excellent weldability and corrosion resistance, and adequate mechanical properties[1-3]. Thus, after heat treatment, it is used as a modern structural material for the aircraft and automotive industries, and is suitable for the manufacture of complex shaped castings with low density requirements[4]. However, the presence of coarse crystalline structures such as coarse α-Al dendrites, lamellar eutectic silicon and needle-like iron-rich intermetallic compounds in cast Al-Si alloys cut the matrix and reduce the ductility and strength of the alloy[5,6]. Therefore, the mechanical properties of Al-Si alloys are improved by refining coarse α-Al dendrites, improving the eutectic silicon morphology by adding modifiers, and eliminating Fe-rich intermetallic compounds by chemical modification[7-11].

In the past decades, the mechanical strength of alloys has been improved by adjusting their chemical composition[12], including nanoprecipitates with significant strengthening effects[13], and mitigation of crystal dislocation slippage at room or high temperatures through multiple effects of precipitating second phases and improving eutectic silicon morphology[14,15].

It has been shown that addition of appropriate amounts of rare earth elements and transition metal elements has significant strengthening effects and improves their mechanical properties, mainly due to the formation of additional precipitates in the intra-dendrite region[16-19]. Current research in this field mainly focuses on the effect of combined addition of Er(Sc) and Zr on aluminum alloys. These elements can lead to the formation of Al_3(Er/Sc, Zr) precipitates with a fine and densely distributed $L1_2$ structure of mass points[20-23],

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which have a good eutectic relationship with the matrix and resist coarsening, inhibit grain growth, and can effectively play a pegging role on subgrain boundaries and dislocations, thus increasing the recrystallization temperature of aluminum alloys. While the addition of Er and Sc in aluminum-silicon alloys can obviously change the eutectic silicon from a coarse needle-like structure to a fibrous morphology, and the researchers found that the addition of Er and Sc can induce twinning. The mechanism of modified eutectic silicon is thus considered to be impurity-induced twinning (ITT)[24-27].

Rare earth Yb has similar chemical properties with Sc or Er, Al-Zr-Yb alloy also precipitates $L1_2$ structure $Al_3(Zr, Yb)$ precipitation phase during aging process[28], and can change the eutectic silicon morphology, but the mechanism of modified eutectic silicon is different from that of Er or Sc and impurity-induced twinning[29]. Although some studies have been carried out on Yb and Zr addition alone to sub-eutectic Al-Si alloys, the effects of composite addition of Yb and Zr on sub-eutectic Al-Si alloys and the refinement and modification mechanism have been rarely reported. Therefore, this research investigated the changes of microstructure, mechanical properties and refinement and modification mechanism by Yb addition alone and composite addition of Yb and Zr in A356 alloy, which provides guidance for Yb and Zr refinement metamorphic sub-eutectic aluminum-silicon alloy in actual industrial production.

1 Experiment

In this experiment, the chemical composition of A356 alloy is presented in Table 1, and intermediate alloys of Al-10Yb and Al-10Zr were used as raw materials. In order to compensate for the reduced content of Si and Mg elements due to the addition of Yb and Zr elements, Al-26Si intermediate alloy as well as pure magnesium were added in calculated amounts. The raw material was first dried in a vacuum drying oven, and then A356 was melted in a resistance crucible furnace at 750~800 °C to form an aluminum liquid, which was de-gassed and de-hybridized using high-purity inert gas and sodium-free refining agent, and then Al-10Yb and Al-10Zr intermediate alloys were added separately and held for 15 min, and then de-gassed and de-hybridized again, and finally the alloy melt was finally poured into the preheated steel mold at 720 °C and solidified at 250 °C.

The above-mentioned casting bar was cut from 20 mm to the bottom and used to prepare a sample of Φ15mm. The metallographic samples were ground with 400#~2000# sand-paper, then polished with 0.5 micron polishing paste, and etched with 0.5% HF solution and rinsed with alcohol, while the deep etching process was polishing and then etching with 36% hydrochloric acid solution for 20~25 s followed by rinsing with alcohol. The microstructure was characterized by optical microscope, scanning electron microscope, energy spectrometer and transmission electron microscope. The average grain size and eutectic width-to-diameter ratio of the samples were counted using Image Pro Plus software. The heat treatment process was solid solution at 545 °C for 4 h+aging at 170 °C for 6 h. The mechanical properties were tested by processing each cast test bar into a standard tensile specimen with a specification length of 100 mm, and then tested on an electronic universal testing machine at a tensile rate of 1.00 mm/min.

Table 1 Chemical composition of A356 alloy (wt%)

Si	Mg	Ti	Fe	Al
6.92	0.31	0.12	0.1	Bal.

2 Results and Discussion

2.1 Effect of refinement modification on alloy microstructure

2.1.1 Effect of Yb on alloy microstructure

Fig.1 shows the microstructure of A356 alloy with different contents of Yb. The morphology of the alloy without the addition of Yb and Zr elements is shown in Fig. 1a, where primary α-Al consists of a large number of coarse dendritic crystals, and the distribution is not uniform, with a large number of eutectic microstructures between the dendrites and obvious bias. Eutectic silicon morphology is coarse needle and plate, which not only cuts the continuity of the matrix, but also increases the brittleness of the alloy, and is very easy to cause stress concentration. While the Yb is added, the α-Al changes from coarse dendritic crystals to fine dendritic crystals, and the eutectic silicon changes from coarse needles to short rods. When 0.3wt% Yb is added, the average grain size is about 250 μm, and the width-to-diameter ratio of eutectic silicon is about 1.7, exhibiting an obvious refinement effect (Fig.1c). However, when 0.5wt% Yb is further added, it can be seen from Fig. 1d that the refinement effect does not change significantly, and the eutectic size becomes larger compared to that with 0.3wt% Yb. This is due to the formation of excessive Yb-containing intermetallic compounds that reduce the effective content of Yb elements in the alloy and ultimately weaken the modification effect of Yb element.

2.1.2 Effect of composite addition of Yb and Zr on alloy microstructure

In order to avoid the formation of excessive Yb-rich intermetallic compounds, the alloy with 0.3wt% Yb addition was used to study the effect of different Zr contents on the alloy morphology, as shown in Fig.2. With the addition of Zr, α-Al shows a change from the previous dendritic crystals to petal-like fine equiaxed crystals. When 0.25wt% and 0.35wt% Zr are added, as shown in Fig. 2b and 2c, the average grain size decreases to about 130 μm and the eutectic aspect ratio is 1.6. However, Fig. 2d displays that when the Zr content is further increased, the average grain size does not change much, but the eutectic silicon starts to become larger, and its aspect ratio is 2. The eutectic silicon aspect ratio also becomes larger because of the formation of too many intermetallic compounds in the alloy due to excessive refinement and modification elements, which weakens the modification effect.

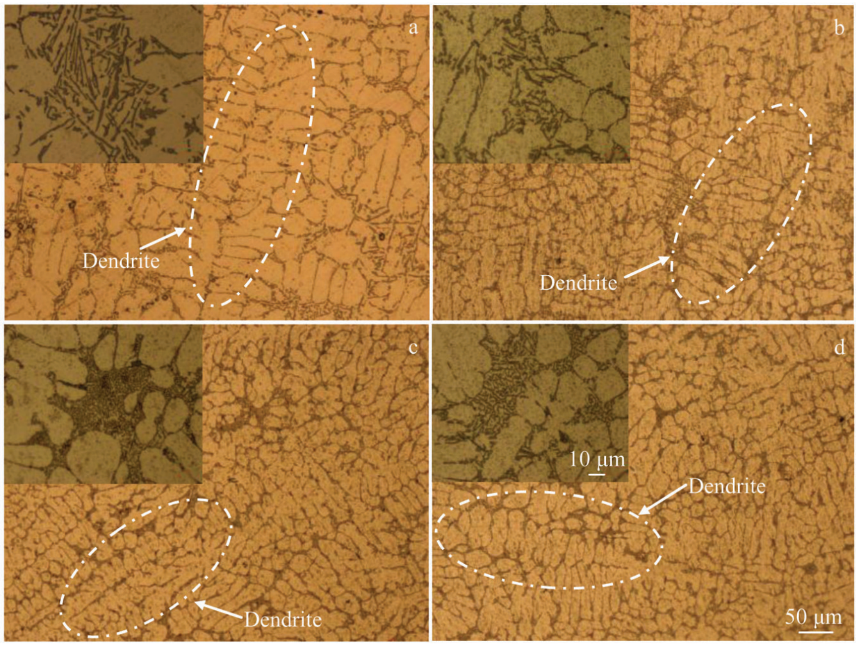

Fig.1 As-cast microstructures of A356 alloy with different contents of Yb: (a) 0.0wt%, (b) 0.1wt%, (c) 0.3wt%, and (d) 0.5wt%

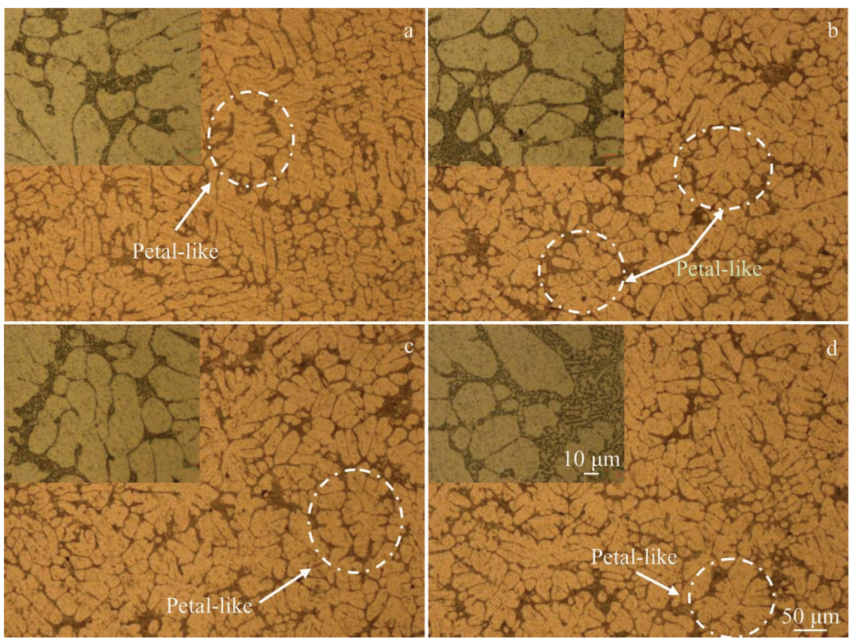

Fig.2 As-cast microstructure of A356-0.3Yb alloy with different contents of Zr: (a) 0.15wt%, (b) 0.25wt%, (c) 0.35wt%, and (d) 0.45wt%

2.1.3 Effect of addition of Yb and Zr on the average grain size and the aspect ratio of eutectic silicon of the alloy

In order to investigate the effect of Yb and Zr composite refinement and modification, Fig.3 presents the microstructure of A356, A356-0.3Yb and A356-0.3Yb-0.25Zr alloys after deep corrosion. Referring to Fig.3a, 3c and 3e, it can be seen that the α-Al structure has been refined from coarse dendrites to fine petal-like equiaxed crystals. Fig. 3b, 3d, and 3f show that the eutectic silicon morphology has been transformed from needle-like to coral-like. With the addition of Yb and Zr, eutectic silicon will produce more branches and grow in more different directions.

Image Pro Plus software was used to calculate the average grain size and eutectic silicon aspect ratio of all samples; 20 groups were counted for each sample and averaged. The average grain size adopted the cut-line method, that is, the grains were intercepted by a straight line with a certain length. It is possible to calculate how long the average straight line can cut a grain, which is the average grain size. The aspect ratio of eutectic silicon is the ratio of its length to its width. And the results are shown in Fig. 4 and Fig. 5. The average grain size of A356 alloy without Yb and Zr elements is 521

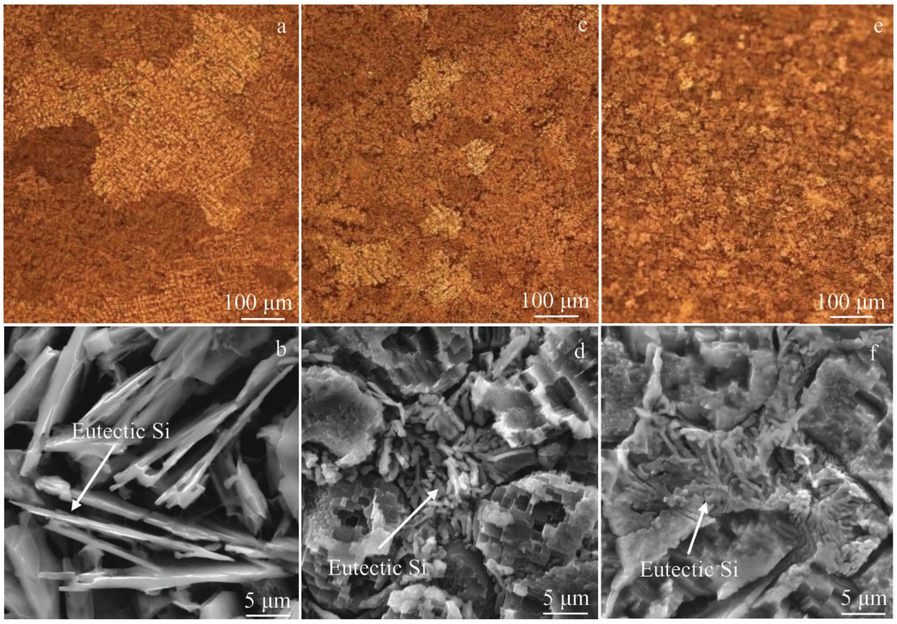

Fig.3 Microstructures of alloys after deep corrosion: (a, b) A356, (c, d) A356-0.3Yb, and (e, f) A356-0.3Yb-0.25Zr

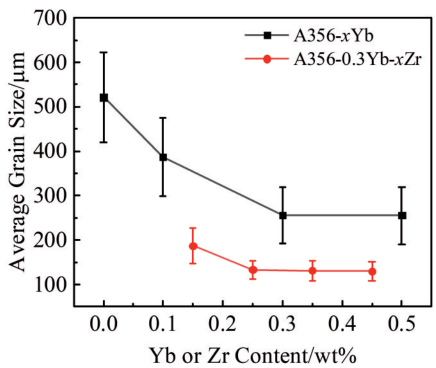

Fig.4 Effect of Yb and Zr content on average grain size of alloy

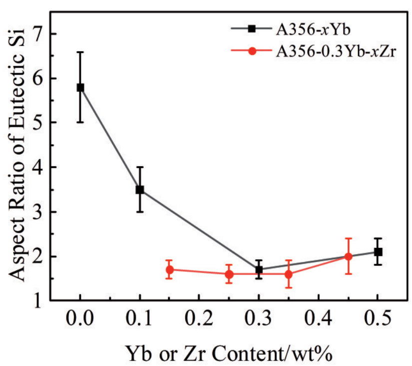

Fig.5 Influence of Yb and Zr content on aspect ratio of eutectic Si

μm, and the aspect ratio of eutectic silicon is 5.8. The average grain size and aspect ratio of eutectic silicon after modification and refinement by adding Yb are relatively reduced. When Yb is 0.3wt%, the average grain size and aspect ratio of eutectic silicon are 256 μm and 1.7, respectively. When 0.25wt% Zr is added on this basis, the average grain size is further reduced to 133 μm, but the aspect ratio of eutectic silicon is 1.6, the reduction is not large, so it shows that both Yb and Zr have a significant effect on grain refinement, but Zr does not have a significant effect on the morphology of eutectic silicon, only further refinement. However, when too much Zr is added, the aspect ratio of the eutectic silicon will increase.

2.2 Effect of Yb and Zr addition on the second phase of the alloy

SEM images of the morphology and distribution of intermetallic compounds in A356, A356-0.3Yb and A356-0.3Yb-0.25Zr alloys are shown in Fig. 6, and the chemical components are listed in Table 2. Combined with the analysis of the morphology and composition of the intermetallic compound, the bright white intermetallic compound with a large contrast with the matrix is the iron-rich phase and the rare-earth phase. Iron is an inevitable harmful element in the A356 casting alloy. During the solidification process, iron can react with silicon to form a thick iron-rich phase densely distributed in the alloy, as shown by points A and B in Fig.6a. This acicular iron-rich intermetallic compound and the matrix interface lattice mismatch, resulting in a decrease in the mechanical properties of the alloy [30]. The morphology of the iron-rich intermetallic compound is improved after refinement and modification, as shown by point C in Fig.6b and point E in Fig. 6c, which are fishbone or needle-like with smaller sizes. In addition, a long rod-shaped Zr-Ti-Al compound is also found, as shown by point H in Fig.6c.

2.3 Effect of Yb and Zr addition on the mechanical properties of alloys

Table 3 shows the mechanical properties of the A356 alloy with different Yb and Zr contents after heat treatment (T6). The results show that the addition of Yb and Zr can significantly improve the mechanical properties of the alloy.

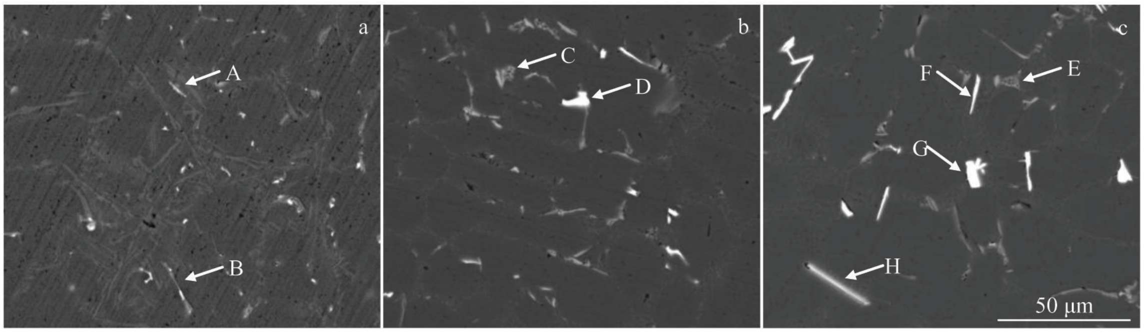

Fig.6 SEM images of alloys with different refinement and modification treatments: (a) A356, (b) A356-0.3Yb, and (c) A356-0.3Yb-0.25Zr

Table 2 Chemical composition of the second phase marked in Fig.6 (wt%)

Point	Al	Si	Mg	Fe	Yb	Zr	Ti
A	78.50	14.11	04.30	02.84	-	-	-
B	74.76	18.27	04.15	02.82	-	-	-
C	74.79	18.60	04.74	01.25	00.62	-	-
D	55.41	29.71	04.08	00.22	10.58	-	-
E	81.08	11.90	05.01	01.49	00.35	00.18	-
F	71.95	21.44	02.90	00.21	03.34	00.15	-
G	65.32	25.82	-	-	08.86	-	-
H	74.81	12.48	-	-	-	06.66	06.05

Table 3 Influence of Yb and Zr content on the mechanical properties of alloy after T6 treatment

Ingredient	UTS/MPa	YS/MPa	EL/%
0%	253.6	176.4	4.3
0.1%Yb	261.3	185.1	5.7
0.3%Yb	273.8	193.6	7.2
0.5%Yb	270.1	190.9	6.6
0.3%Yb+0.15%Zr	281.4	204.5	8.1
0.3%Yb+0.25%Zr	296.3	214.7	9.2
0.3%Yb+0.35%Zr	291.5	205.2	8.9
0.3%Yb+0.45%Zr	285.8	202.3	8.3

The tensile strength, yield strength and elongation of the alloy without the addition of Yb and Zr elements are 253.6 MPa, 176.4 MPa and 4.3%, respectively; when 0.3wt% Yb is added singly, the tensile strength, yield strength and elongation could reach 273.8 MPa, 193.6 MPa and 7.2%, and the mechanical properties are further improved when Zr is added on the basis of 0.3wt% Yb. The tensile strength, yield strength and elongation are 296.3 MPa, 214.7 MPa and 9.2%, respectively, when the Zr content is 0.25wt%, but the mechanical properties start to decrease when Zr is further added.

To further explore the alloy fracture mechanism, the fracture morphology of the studied alloy was studied, as shown in Fig. 7. The fracture surface of the alloy without the addition of Yb and Zr elements has a large number of cleavage planes and is completely brittle fracture (Fig. 7a). With the addition of Yb and Zr elements, the cleavage planes decrease, the number density of tough nests increases and it is ductile fracture, so its elongation is improved, which is consistent with the mechanical property results mentioned before.

2.4 Mechanism of alloy refinement and modification by adding Yb and Zr

In order to determine the refinement and modification mechanism of Yb and Zr in the alloy, the A356-0.3Yb-0.25Zr alloy was firstly subjected to a local surface scan to analyze the distribution of the elements in the alloy, and the results are shown in Fig. 8. From the same field of view, it can be seen that Yb and Zr are more uniformly distributed inside the alloy, and they are not only solidly soluble in the α-Al matrix, but also exist in the Al-Si eutectic eutectic structure.

According to the above experiments, the strengthening mechanism of A356 alloy by Yb and Zr composite addition was investigated, and the results show that the main strengthening mechanisms are fine grain strengthening, modified eutectic silicon strengthening and precipitation strengthening.

According to a large number of experiments and the strengthening mechanism, the mechanical strength and grain size of metallic materials satisfy the Hall-Petch equation:

$$\sigma = \sigma_0 + \frac{k}{\sqrt{d}} \quad (1)$$

where σ represents the yield limit of the material, σ_0 indicates the lattice frictional resistance when moving a single dislocation, k is a constant related to the material and the grain size, and d is the average grain size. It can be seen that the finer the grain size, the higher the mechanical strength.

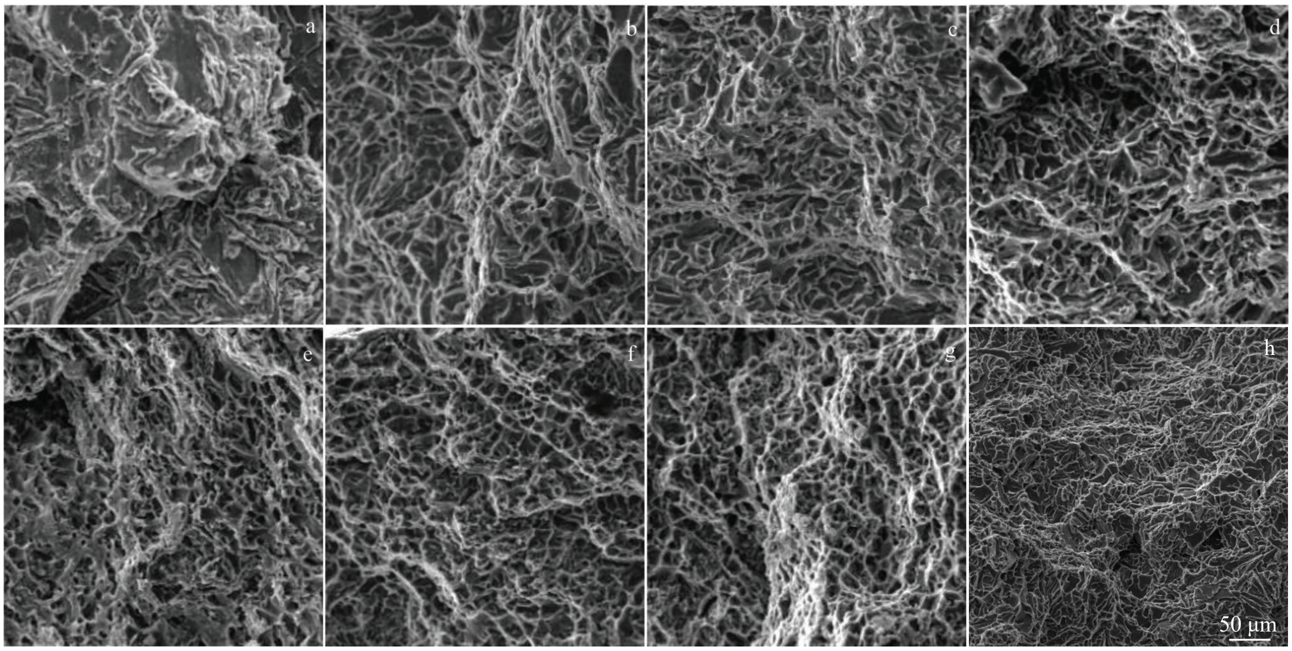

Fig.7 Tensile fracture morphologies of alloys with different refinement and modification treatments (T6): (a) A356, (b) A356-0.1Yb, (c) A356-0.3Yb, (d) A356-0.5Yb, (e) A356-0.3Yb-0.15Zr, (f) A356-0.3Yb-0.25Zr, (g) A356-0.3Yb-0.35Zr, and (h) A356-0.3Yb-0.45Zr

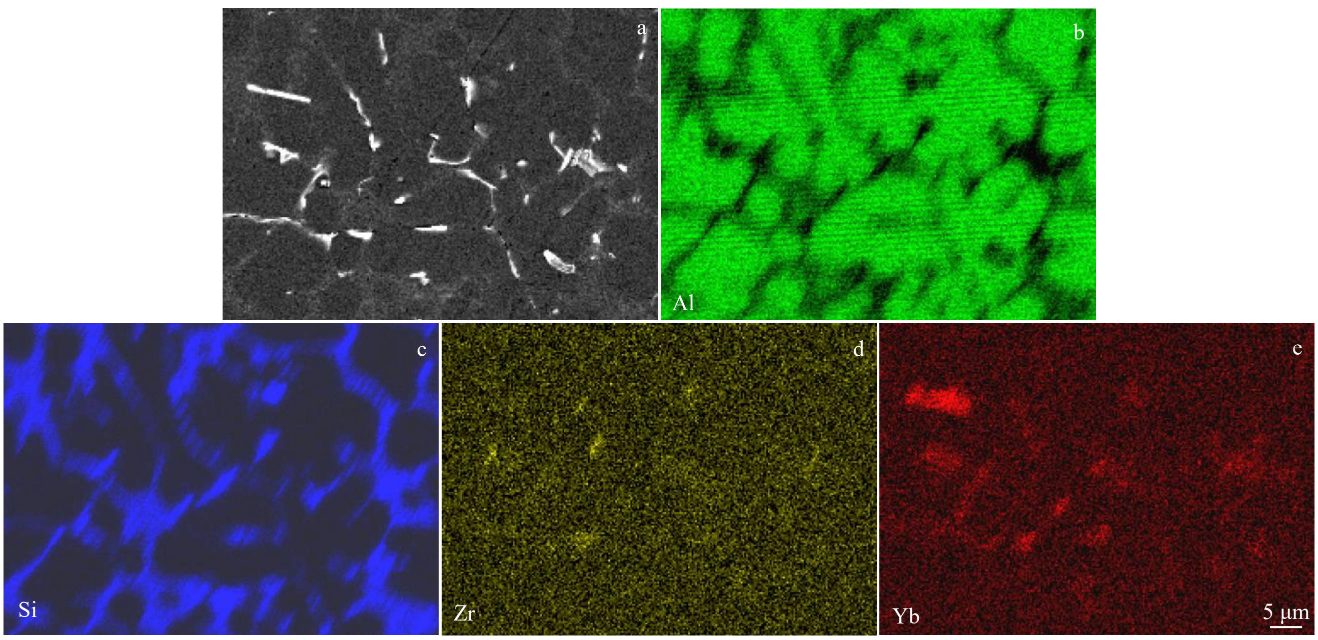

Fig.8 SEM image (a) and EDS element mappings of Al (b), Si (c), Zr (d) and Yb (e) for A356-0.3Yb-0.25Zr

Therefore, the grain size of the alloy with composite addition of Yb and Zr is reduced to 133 μm and its mechanical properties reach the highest value compared to those of the alloy.

Modified eutectic silicon changes its growth mode and thus its morphology changes, which is beneficial to the strengthening of the alloy. In order to further study this strengthening mechanism, the results of TEM analysis are shown in Fig.9. No twinning is found on the eutectic silicon of the unmodified alloy in Fig. 9a, while a few twin crystals appear on the silicon particles after refinement and modification in Fig. 9b, but their density is not high. The research shows that the radius ratios of modified atom to silicon atom close to 1.54~1.85 can induce high density twinning (the atomic radius ratio of Yb to Si is 1.65)[31], thus proving that the mechanism of Yb-modified eutectic silicon is not consistent with impurity-induced twinning (ITT), and it is speculated from the experimental results that it may belong to the twinning notch mechanism (TPRE), which suggests that the modified elements are enriched and selectively adsorbed at the twinning notch. These modified elements inhibit the

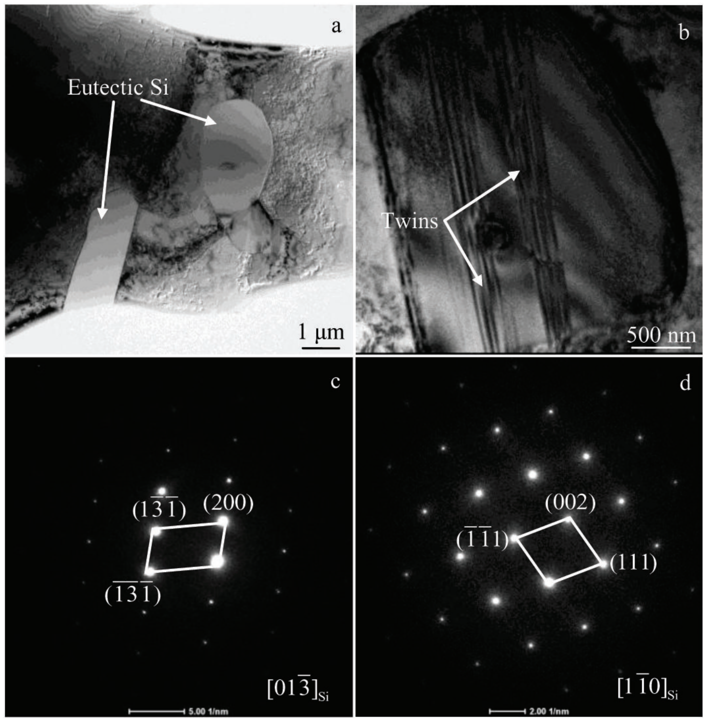

Fig.9 Bright-field TEM images of silicon phase taken in A356 (a) and A356-0.3Yb-0.25Zr (b) alloys; SAED patterns of eutectic silicon in A356-0.3Yb-0.25Zr alloys along $[01\bar{3}]_{Si}$ (c) and $[1\bar{1}0]_{Si}$ (d)

growth of eutectic silicon, causing a change in its growth pattern, leading to the branching of eutectic silicon and eventually changing the morphology of eutectic silicon. From Fig.9c and 9d, the selective area electron diffraction (SAED) patterns obtained for different crystalline band axes of eutectic silicon confirm the mechanism of growth of eutectic silicon along different directions after refinement and modification by Yb and Zr.

The precipitation strengthening can be explained by the Orowan mechanism, and the intensity change ($\Delta\sigma_o$) relationship is given by:

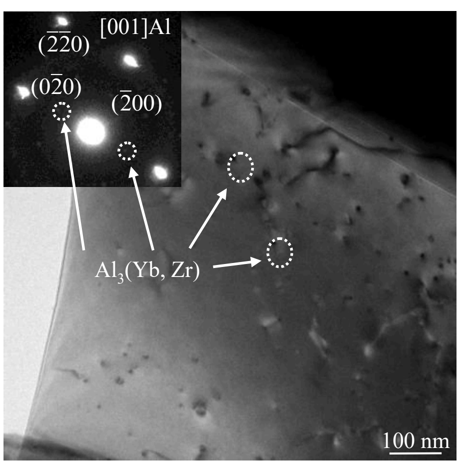

Fig.10 TEM image of precipitates in A356-0.3Yb-0.25Zr alloy after heat treatment (T6)

$$\Delta\sigma_o = M\frac{0.4Gb}{\pi\lambda}\ln\frac{2\bar{r}b^{-1}}{\sqrt{1-v}} \tag{2}$$

where M is Taylor's average orientation factor (3.06), G is the shear modulus of the aluminum matrix at 24 °C (25.4 GPa), and b is the Burgers vector length of aluminum (0.286 nm), v is Poisson's ratio of aluminum (0.345), $\bar{r}$ is the average radius of the precipitation phase particles, λ is the spacing between particles in the precipitation phase. It is shown that the smaller the particle radius of the precipitated phase and the smaller the spacing between the particles, the higher the strength of the alloy. With the composite addition of Yb and Zr, not only the precipitation of Mg_2Si (β'') phase but also the nano-precipitation phase of Al_3(Yb, Zr) with LI_2 structure appear during the aging process, as shown in Fig.10, so the mechanical properties of the alloy are improved.

3 Conclusions

1) The addition of Yb element can significantly refine the dendritic α-Al matrix and improve the morphology of eutectic silicon, reducing the aspect ratio of eutectic silicon and changing it from coarse needle-like to fibrous and short rod-like. With the addition of Zr, the α-Al matrix is further refined and transformed from dendritic to petal-like crystals, and the modified refinement effect is most obvious for 0.3wt% Yb+ 0.25wt% Zr addition.

2) The mechanism of Yb and Zr refinement is mainly the formation of second-phase particles Al_3(Yb, Zr), which plays a dual role in nucleation and refinement during the solidifi-

cation of aluminum, i. e. increasing the nucleation rate and playing a role in precipitation strengthening and thus refinement of grains. The mechanism of eutectic modification is the adsorption of Yb and Zr in the twin grooves (TPRE), thus changing the eutectic growth pattern and resulting in changes in the eutectic morphology.

3) The addition of Yb and Zr can significantly improve the mechanical properties of the alloy. When the Yb content is 0.3wt% and the Zr content is 0.25wt%, the tensile strength, yield strength and elongation of the alloy (T6) are 296.3 MPa, 214.7 MPa and 9.2%, respectively, which are 17%, 21.7% and 1.1 times higher than those of the unrefined and unmodified alloy.

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添加Yb和Zr对Al7Si0.3Mg合金组织及力学性能的影响

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摘　要：研究了添加Yb和Zr元素对Al7Si0.3Mg合金组织及力学性能的影响，以及细化变质机理。结果表明，Yb、Zr元素的添加明显细化了*α*-Al基体，使其从粗大的树枝状晶转变为细小的花瓣状晶，晶粒尺寸明显减小，并且使共晶硅由粗大的针状变质为短棒状，这是由于Yb、Zr吸附在孪晶凹槽处（TPRE）改变共晶硅生长方式，最终改变共晶硅形貌；经热处理（T6）后，大量析出的$Al_3(Yb, Zr)$粒子起到了沉淀强化进而细化晶粒的作用。当Yb质量分数为0.3%、Zr质量分数为0.25%时，合金（T6）的抗拉强度和延伸率为296.3 MPa和9.2%，较未细化变质的合金分别提高了17%和1.1倍。

关键词：Al7Si0.3Mg；Yb；Zr；微观组织；力学性能；细化变质机理

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