doi:10.20944/preprints202007.0115.v1

Article

Effect of Al-Ti-C-La Composite Alloy on Microstructure and Mechanical Properties of 6063 Aluminum Alloy

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Abstract: In this paper, 6063 aluminum alloy for common building profiles is used as the research object. By adding a new Al-Ti-C-La composite alloy, the effect of 6063 aluminum alloy on the microstructure and properties of 6063 aluminum alloy is studied. The results show that Al-Ti-C-La composite alloy has obvious effect on grain refinement of 6063 aluminum alloy. With the addition of Al-Ti-C-La composite alloy, the grain size decreased significantly. When the addition amount of Al-Ti-C-La composite alloy is 1%, the grain size is reduced from 482 μm to 121 μm. Rare earth La is mainly distributed near the Mg2Si phase and β-AlFeSi, and complex compounds such as AlFeSiMgLa are formed. After aging for 270 days on the basis of T6 heat treatment, the tensile strength and elongation of 6063 aluminum alloy augmented with the addition of Al-Ti-C-La composite alloy, and the Vickers hardness gradually diminished. When the addition amount of Al-Ti-C-La composite alloy is 1%, the tensile strength, elongation and Vickers hardness of 6063 aluminum alloy reach 177.2 MPa, 17.8% and 60.9 HV, respectively, and the tensile strength is increased by 16.3%. The elongation is increased by 50.8%, the Vickers hardness is reduced by 15.4%, and the fracture of the alloy is mainly ductile fracture.

Keywords: 6063 aluminum alloy; Al-Ti-C-La composite alloy; refinement effect; precipitated phase; mechanical properties

1. Introduction

6063 aluminum alloy is a typical Al-Mg-Si series alloy that can be strengthened by heat treatment. Due to its light weight, high specific strength, excellent corrosion resistance, easy oxidation coloring and hot workability, it is widely used in construction and decoration alloy [1-3]. So far, people have continuously strengthened their research on the production requirements and applications of higher standards. Therefore, they have also found that there are still some deficiencies in certain aspects, such as extrusion process ability, profile structural steel strength, profile surface color and decoration, etc [4,5]. Hence, in order to better improve the actual performance of its production, optimization of its organization and performance is an urgent problem to be solved [6-9].

The exploration of improving the structure and properties of aluminum alloys at home and abroad has been uninterrupted. Among them, the most widely used is the chemical modification method, which is to add modifiers or refiners to the alloy [10,11]. Cibula, a British scientist, put forward the carbide boride theory for the first time that it can significantly improve the refining effect of aluminum alloy [12], causing subsequent scholars to conduct in-depth research on the grain refining of Al-Ti-B and Al-Ti-C. At present, the application of Al-Ti-B in actual industrial production is relatively mature [13-15], but compared with Al-Ti-C, TiC particles have a smaller tendency to aggregate than TiB_2 particles and can avoid Zr, Cr, or Mn and other elements "poisoning" advantages

[16-18]. However, there are many problems in the preparation process of Al-Ti-C, such as poor wettability between C and aluminum melt, and difficulty in generating TiC by reaction [19,20].

In view of this, people use the strong surface active properties of rare earth and its own refining ability to add an appropriate amount of rare earth in the Al-Ti-C to achieve the purpose of grain refinement. Xu Chunxiang [21] et al. studied the influence of La on the structure and refinement performance of Al-Ti-C. The results showed that the addition of La would make the distribution of Al_3Ti and TiC particles in the matrix more dispersed and obtained excellent grain refinement performance for commercially available pure aluminum. Zhang Jianxin [22] et al. found that proper amount of Ce can enhance the heat treatment effect of Al-Ti-C on Al-Mg-Si alloy, and improve the strength and elongation of the alloy. The research team prepared Al-Ti-C-La composite alloy in the early stage and applied it to pure aluminum [23]. The result show that the addition of RE elements can boost the wettability between C and aluminum melt, promote the synthesis of TiC, and inhibit the aggregation and deposition of the second phase particles in the intermediate alloy in the refining process, which is conducive to improving the performance of the alloy.

Therefore, this paper attempts to add a self-made new Al-Ti-C-La composite alloy to 6063 alloy, research the refinement effect of Al-Ti-C-La composite alloy on 6063 alloy, and discuss the RE La in 6063 alloy existence form, distribution state and its effect and mechanism of action on the second phase particles, analysis of its impact on the hardness, tensile properties and fracture morphology of 6063 alloy, so as to provide theoretical and technical guidance for the practical application of Al-Ti-C-La composite alloy in 6063 alloy.

2. Experimental materials and methods

In order to ensure the accuracy of the experiment, the composition content of each material has been tested. The main experimental material is 6063 aluminum alloy and the self-made Al-Ti-C-La composite alloy made by the research group on the basis of previous research. The chemical composition is shown in Table 1. The standard experimental process is one of the key factors to ensure the success of the experiment. Firstly, 6063 alloy is put into the graphite crucible, and then it is heated to 730℃ in the silicon carbide furnace, then 0.5wt.% refining agent and 0.3wt.% covering agent are added, and the graphite rod is used for mechanical stirring and heat preservation for 10 min, and then the refining slag is carried out to achieve the purpose of degassing and slag removal. When the melt temperature is kept at 730℃, the alloy sample can be prepared by adding the self-made Al-Ti-C-La composite alloy and mechanical mixing, and casting it into the preheated mold after holding for 15 min. The sample number of 6063 alloy refined with different content of Al-Ti-C-La composite alloy designed in the experiment is shown in Table 2.

Table 1. Chemical compositions of Al-Ti-C-La composite alloy (wt.%).

Chemical element	Ti	C	La	Al
Content	5.00	0.62	1.09	Balance

The obtained alloy sample is processed into a small cylinder to facilitate the tissue analysis. Rough grinding, fine grinding and mechanical polishing are used, and then mixed acid (45 ml HCl, 15 ml HNO3, 15 ml HF and 25 ml H2O) is used for corrosion to expose the macrostructure of the sample, and photos are taken to analyze the degree of refinement. The preparation of metallographic samples is basically the same as that of macro refined samples. The polished samples are subject to electrolytic corrosion. The electrolyte (10 ml $HClO_4$, 90 ml C_2H_6O), voltage is 20V, electrolysis time is 90s, and electrolysis temperature is controlled below 35℃. Zeiss optical microscope and field emission scanning electron microscope were used to observe the microstructure and precipitate morphology of the alloy, and Image Pro Plus (IPP) software was used to measure the grain size of the alloy. At the same time, electron probe microanalyzer (EPMA) was used to analyze the mechanism of Al-Ti-C-La as grain refiner in the alloy. In order to further analyze the effect of Al-Ti-C-La master alloy on the precipitates of 6063 alloy, the sample is machined and ground to below 70 μm , and then punched. The samples with obvious thin areas were made by ion thinning instrument and observed by transmission scanning electron microscope (TEM). The samples were homogenized

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at 568℃ for 4h and air-cooled, and then treated with solution treatment and aging treatment. The comprehensive performance of 6063 alloy, such as hardness, strength and elongation, were analyzed on the basis of solution treatment temperature of 525℃, heat preservation for 12 h, water quenching, artificial aging temperature of 200℃, air cooling for 2 h, and the morphology of tensile port was analyzed and characterized.

Table 2. The sample number of Al-Ti-C-La composite alloy refined 6063 alloy.

Alloy number	Alloy composition
A1	6063
A2	6063 + 0.2% Al-Ti-C-La
A3	6063 + 0.5% Al-Ti-C-La
A4	6063 + 1.0% Al-Ti-C-La

3. Results and discussion

3.1. Microstructure of Al-Ti-C-La composite alloy

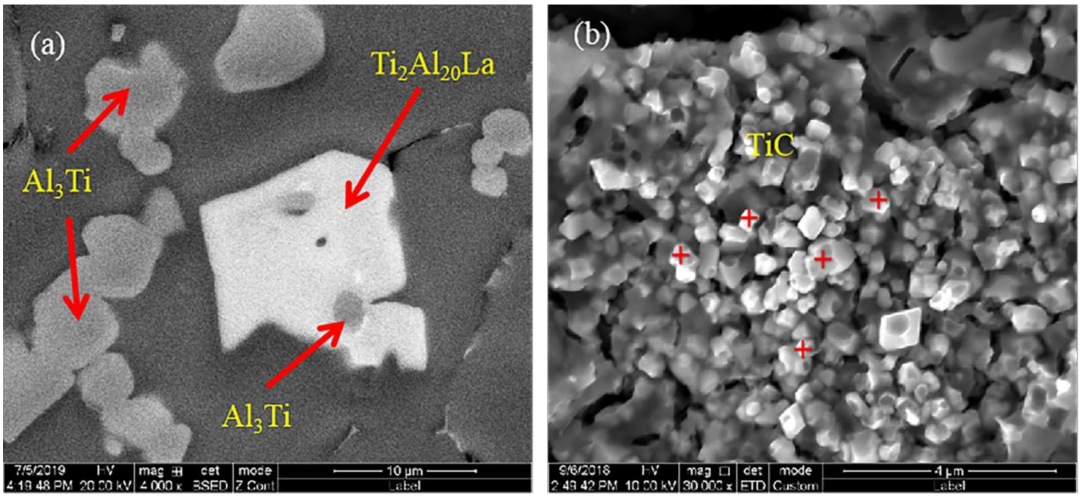

Figure 1. Micrograph of investigated of Al-Ti-C-La composite alloy: (a) SEM image of Al-Ti-C-La composite alloy; (b) micro-structure images of TiC.

The microstructure characteristics of Al-Ti-C-La composite alloy determine its refining effect. As shown in Fig. 1(a), the phase in the Al-Ti-C-La composite alloy is mainly composed of the rare earth phases $Ti_2Al_{20}La$, Al_3Ti phase and TiC particles except for the α-Al matrix. The rare earth phase $Ti_2Al_{20}La$ is a bright white polygonal block and is wrapped with Al_3Ti. As shown in Fig. 1 (b), TiC is mainly distributed in the form of granular agglomerations, with sizes ranging from 0.1 μm to 1.2 μm.

It is necessary to study the existing mode and microstructure of TiC particles for analyzing the action mechanism of Al-Ti-C-La composite alloy. The TiC particles in the Al-Ti-C-La composite alloy were analyzed by TEM. Fig. 2 shows the open field phase of TiC particles, and the particle size is about 0.2 μm, with uniform distribution and no segregation, followed by the face centered cubic structure of TiC, which has a small lattice constant and can be combined with the aluminum matrix, so a large number of TiC particles can exist stably in the aluminum melt [24], which is conducive to the grain refinement of 6063 alloy.

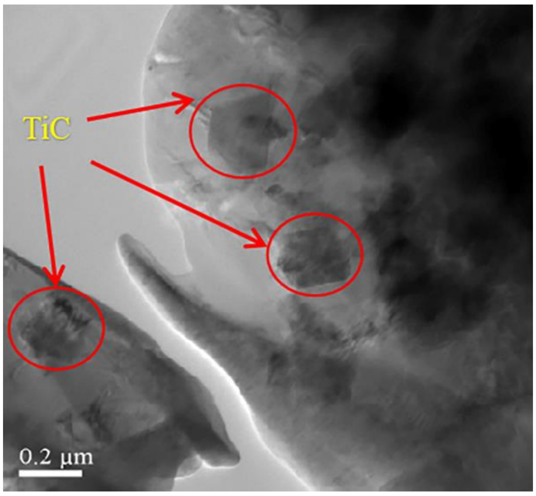

Figure 2. Bright field phase of TiC particles in Al-Ti-C-La composite alloy.

3.2. Effect of Al-Ti-C-La composite alloy on the microstructure of 6063 alloy

Fig. 3 shows the macro morphology of Al-Ti-C-La composite alloy added to 6063 alloy. It is obvious from Fig. 3(a) that the macrostructure grain of A1 alloy is coarse, the center is composed of equiaxed crystal and the edge is composed of columnar crystal. Compared with Fig. 3(a-d), it is found that Al-Ti-C-La composite alloy has obvious improvement effect on the structure of 6063 alloy. It can be looked from Fig. 3(b) that the size of coarse equiaxed crystal and edge columnar crystal in the center of A2 decreased obviously, and some fine equiaxed crystal replaced the edge columnar crystal. The grain refinement effect of A3 is very obvious. The coarse and uneven equiaxed crystal in the center is refined into fine equiaxed crystal, and the columnar crystal with long strip at the edge has vanished and substituted by fine and equiaxed crystal. When the addition amount of Al-Ti-C-La composite alloy continues to increase, the grain size is further reduced and the structure distribution is more uniform, as shown in Fig. 3 (d).

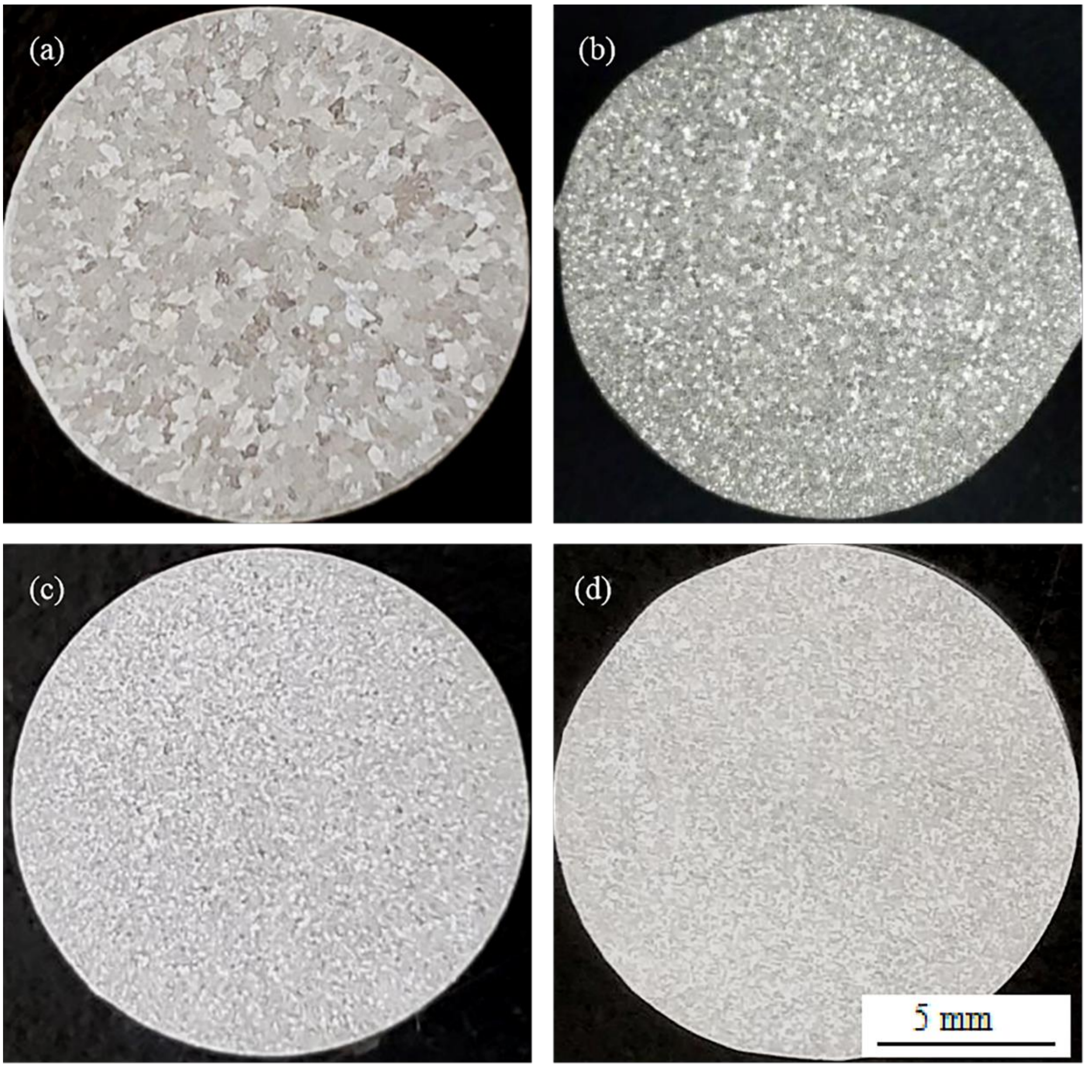

Figure 3. Macrostructures of 6063 alloy using the Al-Ti-C-La composite alloy: (a) A1; (b) A2; (c) A3; (d) A4.

Fig. 4 shows the microstructure of 6063 alloy with different content of Al-Ti-C-La composite alloy. It can be concluded that the addition of Al-Ti-C-La composite alloy to 6063 alloy has better grain refinement effect. A1 is composed of α-Al grains and eutectic structure continuously distributed at the grain boundary α-Al grains show a rough and uneven distribution of dendrites and petals, as shown in Fig. 4(a). The grain size of A2 is obviously finer than that of A1, and the precipitation amount at the grain boundary is increased, as shown in Fig. 4(b). The grains of A3 and A4 are significantly refined, and coarse dendrite grains are refined into equiaxed grains. As the number of grain boundaries increases, part of the second phase precipitates in the grains. With the increase of the content of Al-Ti-C-La composite alloy, the number of the second phase also increases gradually, as shown in Fig. 4(c) and Fig. 4(d).

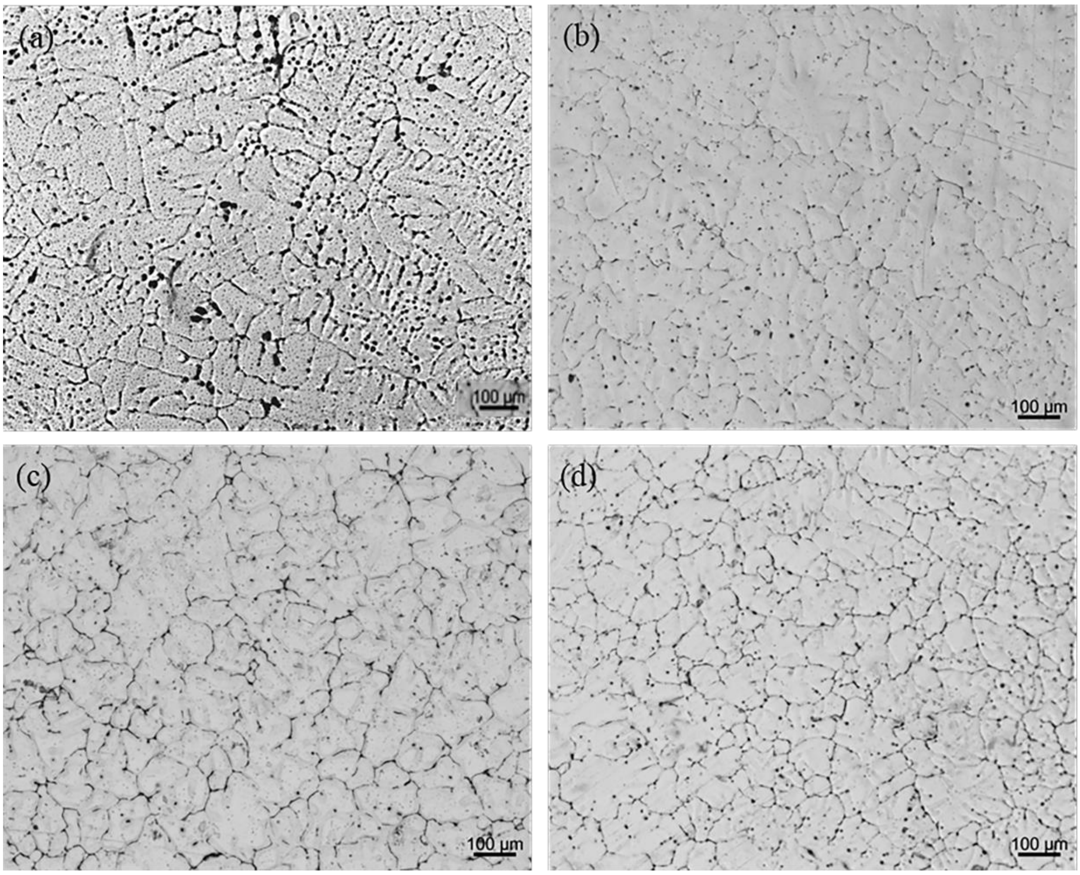

Figure 4. Optical micrograph images of 6063 alloy using the different content Al-Ti-C-La composite alloy: (a) A1; (b) A2; (c) A3; (d) A4.

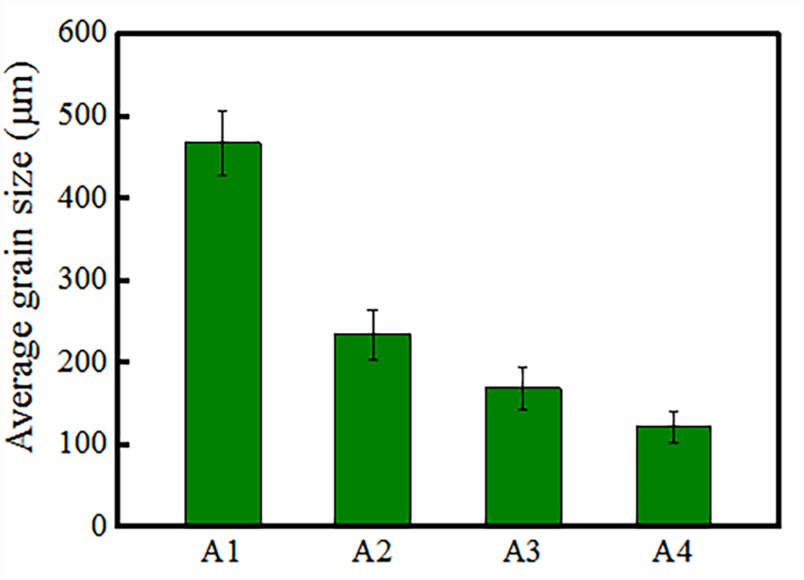

Figure 5. Histograms of the average grain size of 6063 alloy using the different content Al-Ti-C-La composite alloy.

Fig. 5 is the histogram of the average grain size of 6063 alloy with different content of Al-Ti-C-La composite alloy. It can be seen that the average grain size of α-Al gradually decreases with the increase of the addition amount of Al-Ti-C-La composite alloy. When 0%, 0.2%, 0.5% Al-Ti-C-La composite alloy is added to 6063 alloy, the average grain size of α-Al is 482 μm, 234 μm and 168 μm respectively. When the addition of Al-Ti-C-La composite alloy is 1%, the average grain size of 6063 alloy is refined to 121 μm, which is 74.8% higher than that of 0%.

Generally, there are two types of precipitates in 6063 alloy, one is β-AlFeSi, which is coarse and irregular, and the other is Mg_2Si, which is short rod or granular. Most of them are in the grain boundary, and there are a few in the grain boundary. Compared with the coarse and irregular dendrite or long strip β-AlFeSi phase at the grain boundary, the short rod or granular Mg_2Si phase disperses and distributes in the aluminum matrix, playing a role of dispersion strengthening, reducing the stress concentration, and weakening the cleavage effect on the matrix. Further study on the morphology and distribution of the second phase in 6063 alloy after adding Al-Ti-C-La composite alloy can provide experimental evidence for analyzing the refinement mechanism and performance strengthening mechanism of Al-Ti-C-La composite alloy on 6063 alloy.

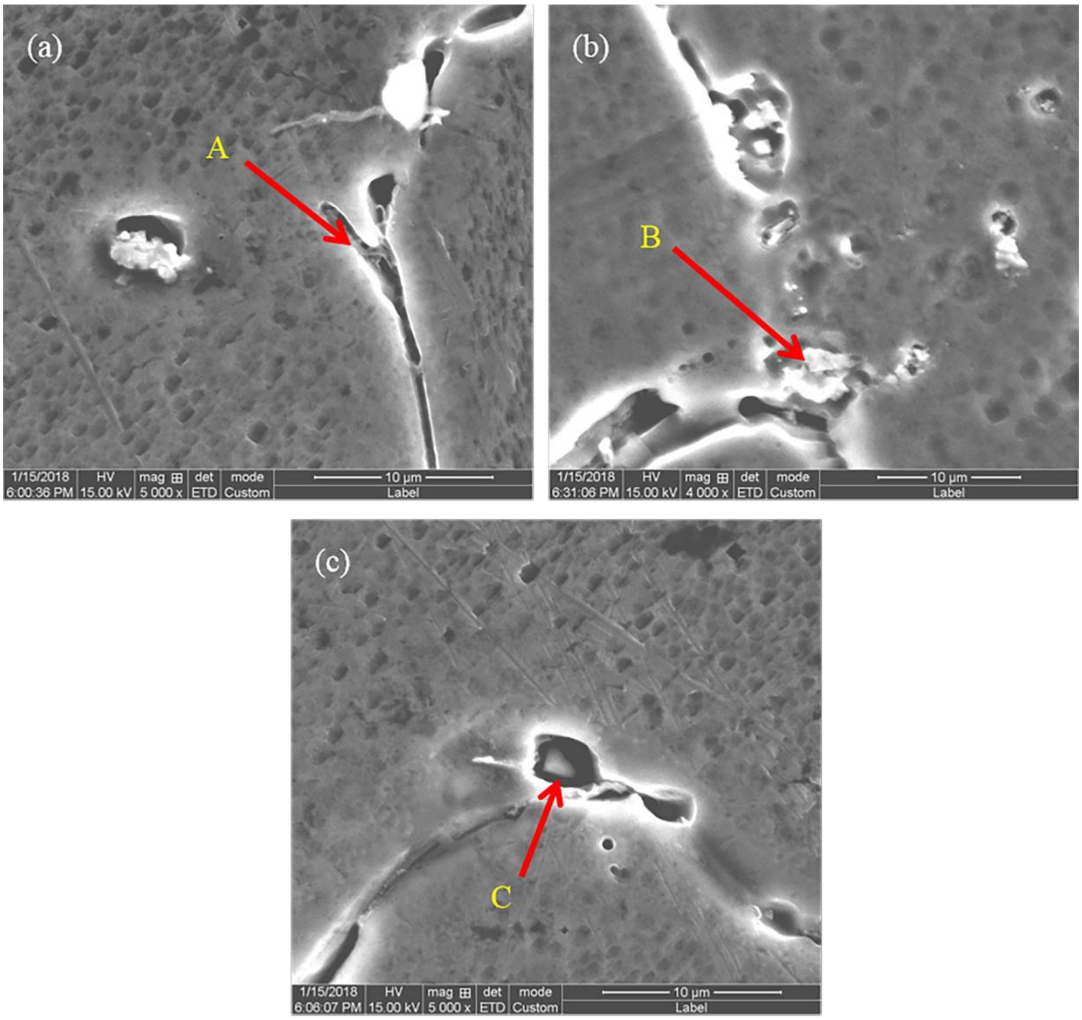

Figure 6. Precipitates at the crystal boundary of A4 : (a) long strip of A; (b) large particles of B; (c) small particles of C.

Fig. 6 shows the morphology of precipitates in A4. It can be observed that the precipitates in the alloy have three different morphologies: long strip, agglomerated large particle and dispersed small particle. It can be seen from Table 3 of the energy spectrum analysis results of the precipitates that the precipitates in the long strip shape of the crystal boundary in Fig. 6 (a) are rich in Al, Fe and Si elements, which are considered as β-AlFeSi phase by analysis; From Fig. 6 (b), it can be seen that some large particle precipitates contain Mg and La elements besides Al, Fe, Si and other elements, which are considered as AlFeSiMgLa compounds by analysis; As can be seen from Fig. 6(c), some small particulate precipitates contain Al, Si, and Mg, and the ratio of Mg and Si is close to 2:1, which is considered to be the Mg_2Si phase.

Table 3. Elemental composition of different precipitated particles (wt.%).

Precipitated phase	Element				
	Al	Fe	Si	Mg	La
A	86.1	6.5	7.4	—	—
B	76.5	17.0	5.9	0.4	0.2
C	98.9	—	0.5	0.9	—

Fig. 7(a) shows the particles inside the grains of A4 and their EDS analysis diagrams. It can be seen that the grains of α-Al contain small white particles with a size of about 3 μm. The composition detection by EDS energy spectrum shows that the main composition of the particles is divided into Al, C and Ti, which can be judged as TiC. As the TiC particles in Al-Ti-C-La composite alloy are relatively small, the results show that there is Al element in the particles.

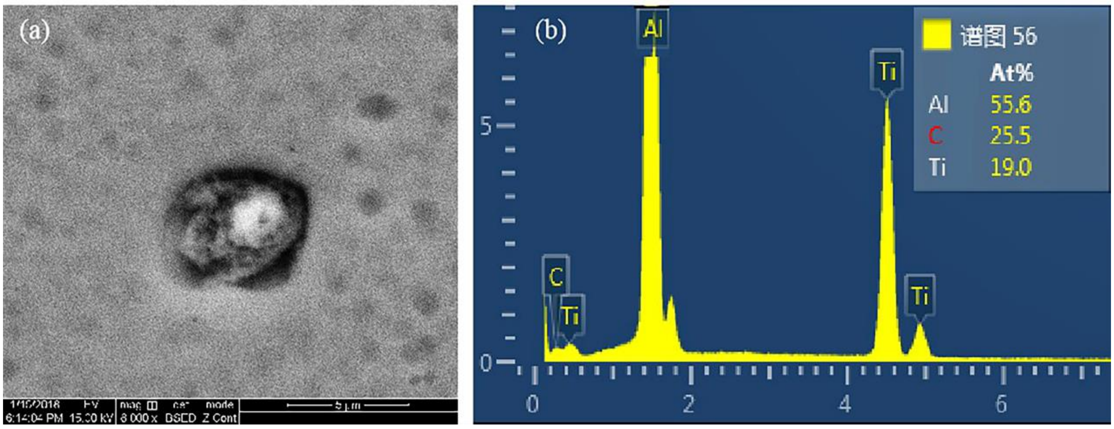

Figure 7. SEM image and EDS spectrum of A4: (a) SEM image of intragranular particles; (b) corresponding EDS spectrum.

In order to further explore the mechanism of RE La in the refining process of 6063 alloy, the distribution of RE La in 6063 alloy was analyzed by EPMA surface scanning spectrogram. Fig.8 is the EPMA surface scanning spectrum of A1. It can be seen that Fe elements are in long strips and dendrites, concentrated in the grain boundary of α-Al. The distribution of Si elements is very similar to that of Fe elements, which are concentrated in α-Al grain boundary in long strips and dendrites, and a small amount of Si elements are also present in the crystal.

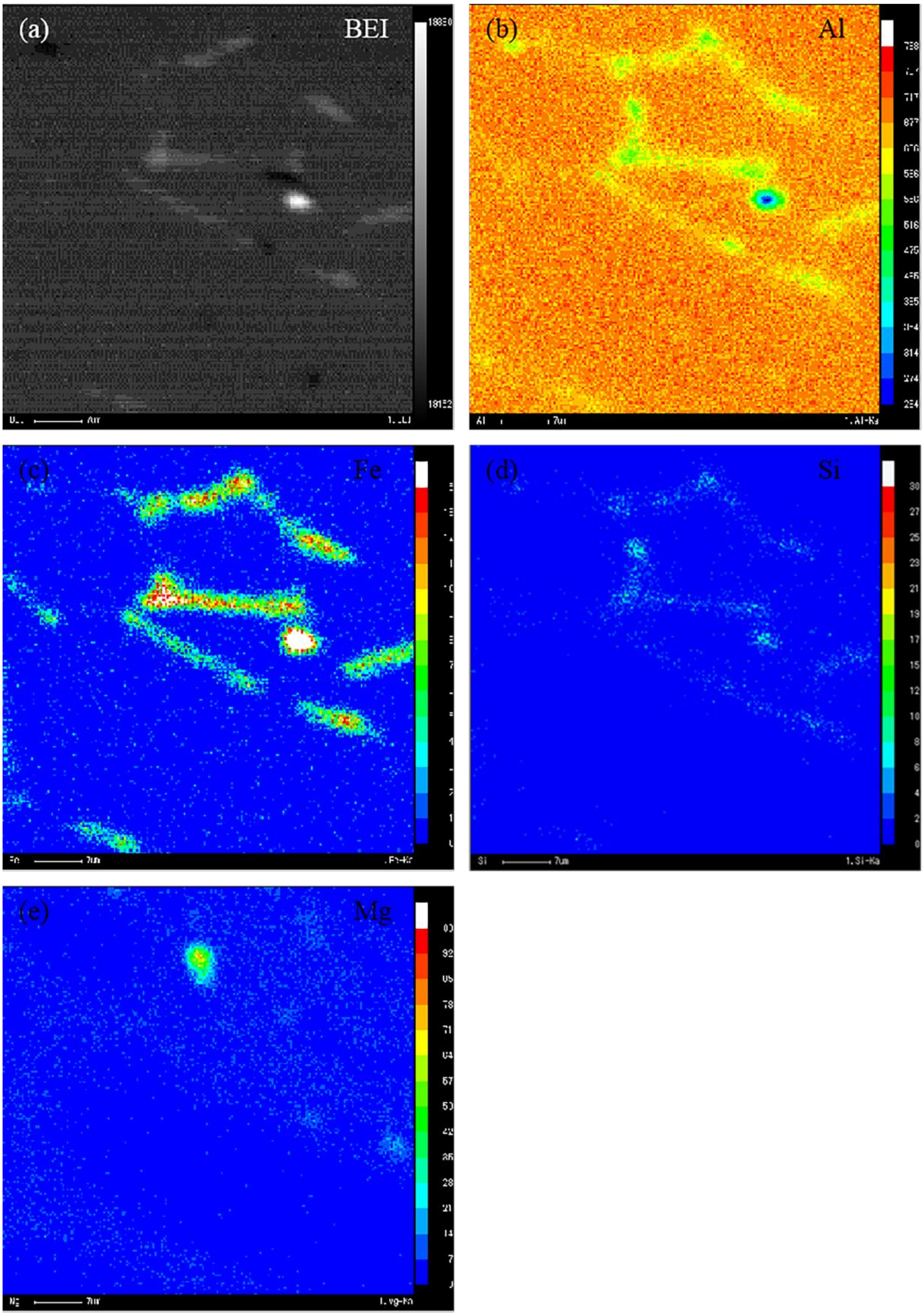

Figure 8. EPMA analysis of alloy A1: (a) back-scattered electron image; (b-e) X ray image of elements Al, Fe, Si and Mg.

Fig. 9 is a surface scanning spectrogram of EPMA of A4 . It can be seen that Fe and Si are distributed in the α-Al grain boundary in spherical or short rod shape, while Mg is more dispersed in the aluminum matrix than in Fig. 8 (e). It can be seen from Fig. 9 (f) that the rare earth La elements are mainly distributed in the areas where Si and Fe elements are enriched. In 6063 alloy, on the one hand, Mg element plays the role of solution strengthening when it is dissolved in α-A1, on the other hand, Mg and Si react in aluminum solution to form the strengthening phase Mg_2Si. Therefore, it can be concluded that La is mainly distributed near Mg_2Si phase and β-AlFeSi in 6063 alloy.

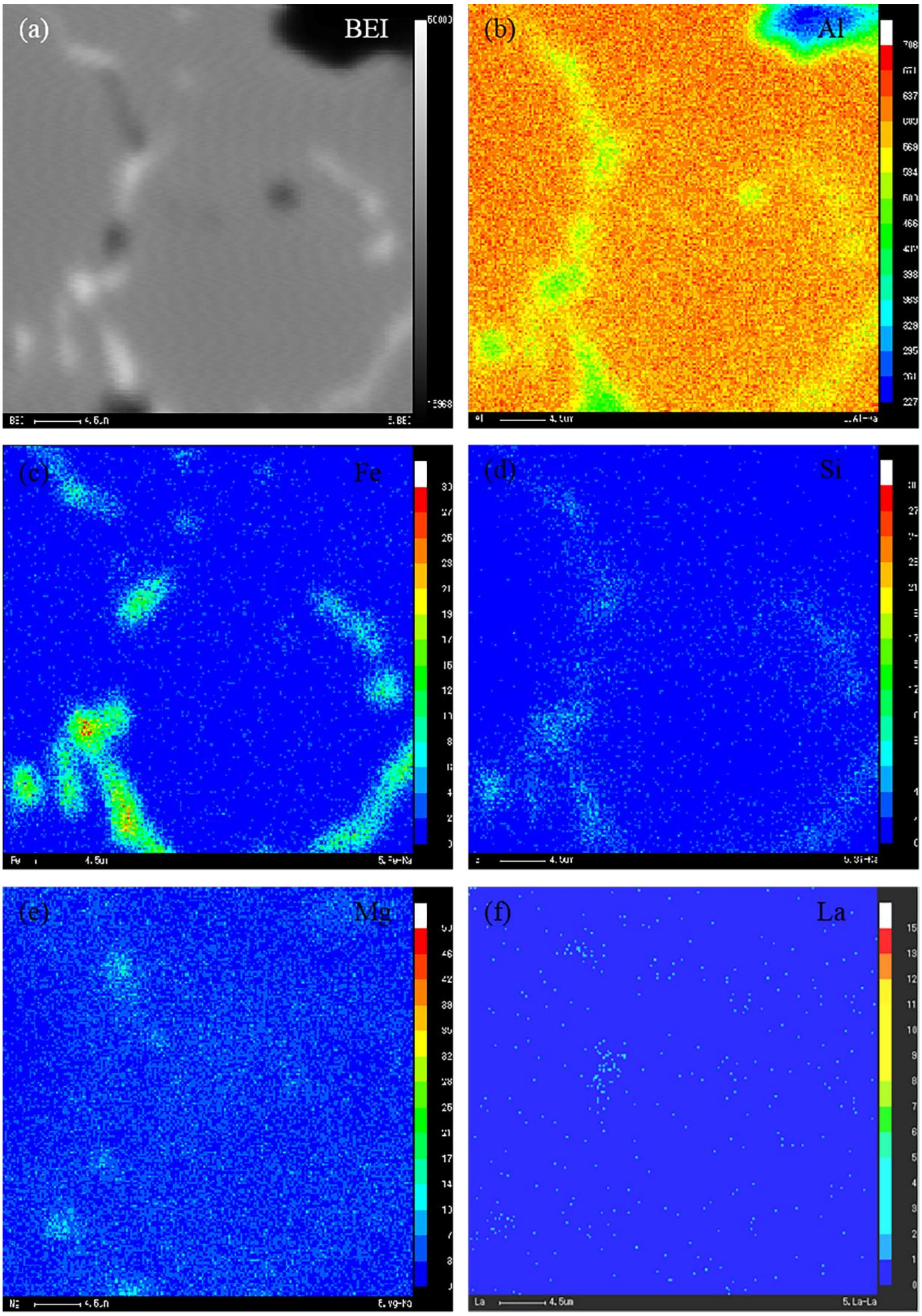

Figure 9. EPMA analysis of A4: (a) Backscattered electron images; (b-f) X-ray images of Al, Fe, Si, Mg, La elements.

Based on the above analysis, the influence of Al-Ti-C-La composite alloy on the grain refinement and microstructure of 6063 alloy is mainly due to the fact that both α-Al and TiC are face centered cubic structures, in which the lattice constant of α-Al(a_{Al}) is 0.404 nm and that of TiC(a_{TiC}) is 0.432 nm. Therefore, the lattice mismatch δ between α-Al and TiC is 6.9%, less than 25% and more than 5%, that is, there is a partially coherent interface between the TiC substrate and α-Al matrix, which makes the TiC particles contained in the Al-Ti-C-La composite alloy can be used as the heterogeneous nucleation core of α-Al [18,25]. Theoretically, the more the amount of intermediate alloy is added, the more TiC particles it contains, the better the refining effect; Secondly, the RE phases $Ti_2Al_{20}La$ and Al_3Ti included in Al-Ti-C-La composite alloy can not exist stably in the aluminum melt, and they will decompose, and the Ti and La atoms produced by decomposition will be enriched on the surface of TiC particles, forming the enrichment layer of Ti and La atoms. The enrichment layer can not only prevent TiC from poisoning due to the formation of Al_4C_3, but also cause component super cooling, thus hindering grain growth in the solidification process, playing a role of refining [23,26].

Finally, some rare earth La produced by decomposition will be adsorbed on the surface of Mg_2Si phase and β-AlFeSi phase. Because La is a surfactant, it can inhibit the growth of Mg_2Si phase and β-AlFeSi phase, improve their morphology and distribution. In addition, some scholars [27] believe that the temperature range of some alloy phases is changed because the RE La is easily accumulated in the front edge of solid-liquid interface when 6063 alloy is added with a small amount of RE La. For one thing, it causes the decrease of solidification temperature, the increase of growth speed and the decrease of dendrite spacing. For another thing, it causes the redistribution of solute at the front of solid-liquid interface during dendrite growth, which results in the increase of super-cooled zone of composition, the increase of crystal core and the decrease of dendrite spacing.

3.3. Effect of Al-Ti-C-La composite alloy on mechanical properties and fracture morphology of 6063 alloy

3.3.1. Effect on mechanical properties

After 270 days of natural aging on the basis of T6 treatment, the comparison of tensile strength, elongation and hardness of 6063 alloy is shown in Fig. 10. From Fig. 10 (a), it can be observed that the tensile strength of 6063 alloy of A1 is 152.4 MPa and the elongation is 11.8%. With the increase of Al-Ti-C-La composite alloy content, the tensile strength and elongation of 6063 alloy are significantly enhanced. Among them, A4 has the most obvious improvement, its tensile strength has increased by 16.3%, and its elongation has increased by 50.8%. It can be seen from Fig. 10 (b) that with the increase of the content of Al-Ti-C-La composite alloy, the Vickers hardness of A1, A2, A3 and A4 is 72.0 HV, 67.9 HV, 67.6 HV and 60.9 HV respectively, and the Vickers hardness of A4 is 15.4% lower than that of A1.

From the data analysis, it can be concluded that adding different content of Al-Ti-C-La composite alloy to 6063 alloy can enhance the mechanical properties of the alloy, mainly because adding Al-Ti-C-La composite alloy can effectively refine the α-Al grains. The smaller the grain size, the more the grain boundary, the higher the metal strength. The effect of grain size on the mechanical properties of metals can generally be described by Hall-Petch [28], as shown in formula (1):

$$\sigma_s = \sigma_0 + \mathrm{k}d^{-1/2} \tag{1}$$

σs is the yield strength of the material, d is the grain size, and σ_0 and k are constants related to the material. From the formula, it can be seen that smaller grain size will have higher yield strength. The essence of fine grain strengthening lies in the barrier effect of grain boundary on dislocation. The smaller the grain, the more the grain boundary, the greater the barrier effect and the better the strengthening effect. Grain boundary is also the obstacle of crack growth, so grain refinement can improve the toughness of alloy. For another, after the addition of Al-Ti-C-La composite alloy, the original coarse β-AlFeSi phase in the alloy changes into small particle and short rod, which reduces the cleavage effect on the alloy matrix and enhances the performance of the alloy. In addition, the solubility of La in 6063 alloy is relatively low, and the cooling speed is fast during solidification, which leads to the supersaturation of La at the front of solid-liquid interface, so La can also play a role of solution strengthening.

However, RE La will produce complex compounds with Fe, Si and other elements. The formation of these complex compounds consumes Si and Mg elements, resulting in a reduction in the number of strengthening phase Mg_2Si , which may lead to a decrease in the hardness of the alloy. At the same time, the addition of excessive Al-Ti-C-La composite alloy will obviously affect the amount of strengthening phase Mg2Si, which will result in a significant decrease in Vickers hardness of the alloy. Therefore, when the addition amount of Al-Ti-C-La composite alloy is 0.2%, the tensile strength and elongation of 6063 alloy can be significantly increased, while the Vickers hardness reduces slightly.

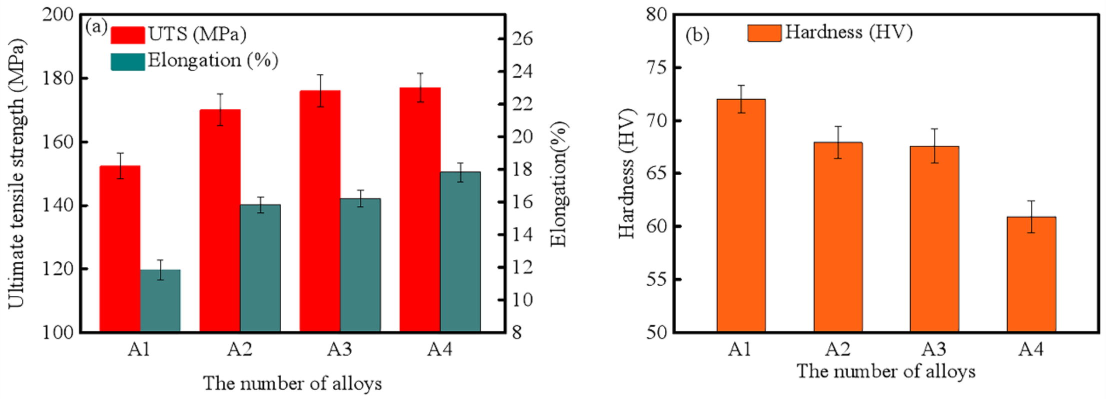

Figure 10. mechanical properties of 6063 alloy with different content of Al-Ti-C-La after heat treatment + 270 days natural aging: (a) tensile strength and elongation; (b) Vickers hardness.

3.3.2. Effect on fracture morphology

Fig. 11 shows the morphology of the tensile fracture of the alloy at room temperature. It can be seen from Fig. 11 that the necking phenomenon occurs during the fracture of the alloy. The macro fracture of the sample is cup-shaped and the crack source is generated on the surface of the sample. The alloy is mainly ductile fracture, and there are also ductile fracture and brittle mixed fracture. Fig. 11 (a) is an A1 without intermediate alloy. It can be seen that there are a large number of β-AlFeSi phases and a certain amount of dimples at the crack source. This shows that due to the segregation of large-size β-AlFeSi and other complex compounds at the grain boundary, cracks are produced and intergranular fracture occurs in the A1. It can be seen from Fig. 11 (b) that there are also a large number of β-AlFeSi phases and dimples at the crack source of A2 , but compared with A1, both the AlFeSi phase and dimples are reduced, indicating that the plasticity of the alloy is better than that of A1. From Fig. 11 (c), it can be seen that there are fewer dimples and shallow β-AlFeSi on the fracture surface of A3, indicating that the plasticity of A3 is better than that of A1 and A2. Compared with other alloys, A4 has more and finer dimples on the fracture surface, with the highest plasticity.

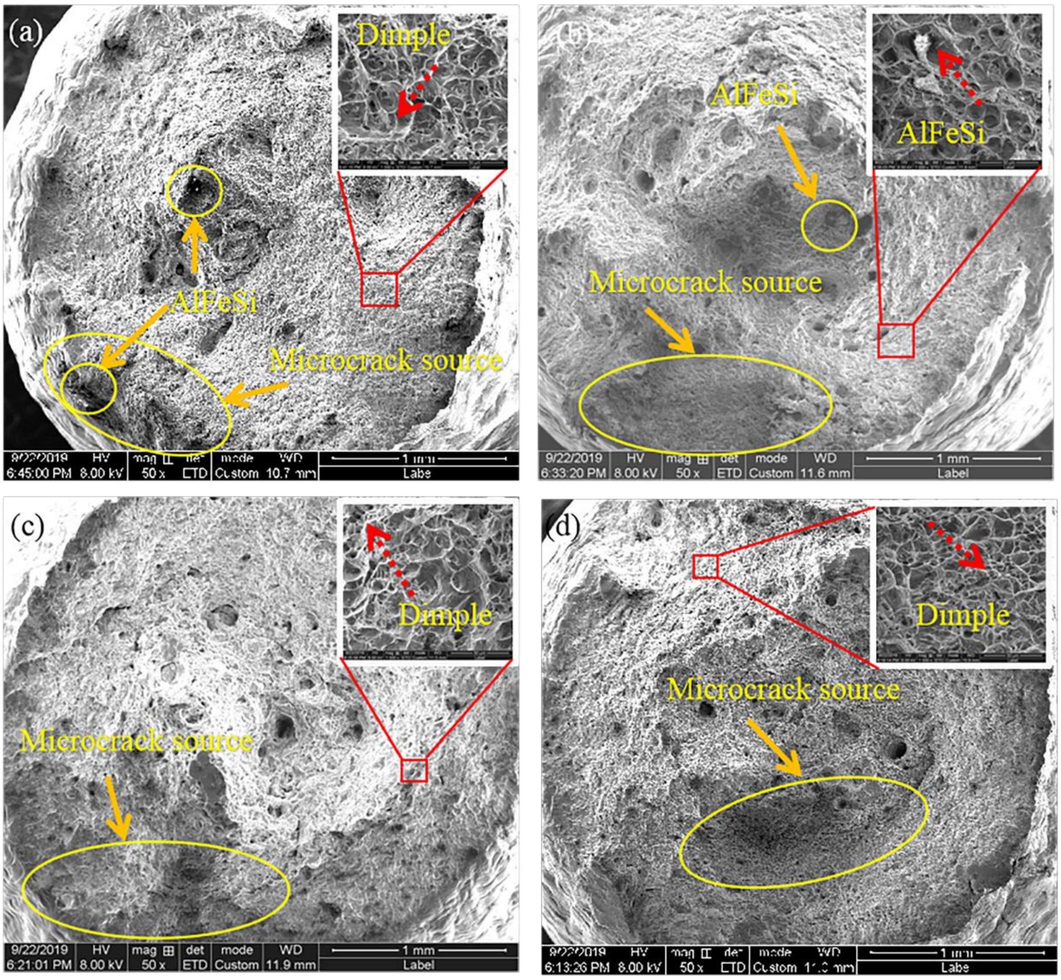

Figure 11. Tensile fracture morphology of 6063 alloy T6 with different content of Al-Ti-C-La composite alloy after heat treatment + natural aging for 270 days: (a) A1; (b) A2; (c) A3; (d) A4.

4. Conclusion

(1) Al-Ti-C-La composite alloy has better grain refinement effect on 6063 aluminum alloy. With the increase of Al-Ti-C-La composite alloy, the microstructure is mainly composed of fine and uniform equiaxed grains, and the grain size decreases significantly.When the addition amount of Al-Ti-C-La composite alloy is 1%, the average grain size is reduced from 482 μm to 121 μm.

(2) Based on the addition of 1% Al-Ti-C-La composite alloy, it is found that there are not only long β-AlFeSi phase and granular Mg_2Si phase, but also some large granular AlFeSiMgLa compounds. At the same time, there are small white particles TiC in the grains, which can be used as the heterogeneous nucleation core of α-Al. In addition, there will be enrichment layers of Ti and La atoms on the surface, which play a role of refining.

(3) Rare earth La will be adsorbed on the surface of Mg_2Si phase and β-AlFeSi phase, which can inhibit the growth of Mg_2Si phase and β-AlFeSi phase, improve their morphology and distribution, and improve the structure and performance.

(4) After 270 days of natural aging on the basis of T6 heat treatment, the tensile strength and elongation of 6063 aluminum alloy increase with the increase of the amount of intermediate alloy, and the hardness decreases gradually. With the addition of 1% Al-Ti-C-La composite alloy, the tensile strength and elongation of 6063 aluminum alloy increase the most, and there are more small dimples on the fracture surface. The fracture mode of the alloy is mainly ductile fracture.

Author Contributions: Wanwu Ding and Xiaoxiong Liu conceived and designed the experiments; Xiaoxiong Liu, Xiaoyan Zhao and Taili Chen carried out the experiments and data collection; Wanwu Ding, Xiaoxiong Liu, Haixia Zhang, Wenjun Zhao, and Changfeng Li analyzed the data; Wanwu Ding contributed

reagents/materials/analysis tools; Xiaoxiong Liu wrote the paper. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the National Natural Science Foundation of China (No.51661021) and the Gansu Key Research and Development Program (18YF1GA061) and China Postdoctoral Science Foundation (2019M653896XB). The authors would like to acknowledge the National Natural Science Foundation of Gansu (1606RJZA16).

Conflicts of Interest: The authors declare no conflict of interest.

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