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Effect of Nd on microstructure and properties of 6063 aluminum alloy

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Abstract

The microstructure of 6063 aluminum alloy has an important influence on its properties. In this paper, the effect of the composite addition of Nd (0, 0.1 wt%, 0.2 wt%, 0.4 wt%, 0.6 wt%) and Al–Ti–B master alloy on the microstructure and mechanical properties of 6063 aluminum alloy was studied. The results show that the composite modification of rare earth Nd and Al–Ti–B has great refinement effect on 6063 aluminum alloy. With the amount of rare earth Nd is 0.1 wt%, the grain boundary is mainly composed of a short rod or discontinuous granular AlFeSiMgNd complex compound. With the content of rare earth reaches 0.6 wt%, the enrichment of rare earth Nd occurs at the grain boundaries, and a grid-like AlSiNd phase is formed. After 180 days of natural aging on the basis of T6 heat treatment, with the increase in the amount of rare earth Nd added, the elongation of the 6063 aluminum alloy increased significantly, but the tensile strength and vickers hardness decreased. The alloy undergoes ductile fracture, and AlSiNd and AlFeSiMgNd particles are present on the fracture surface. This may also be the main reason for the effect of Nd on the mechanical properties of 6063 alloy.

1. Introduction

6063 aluminum alloy has the advantages of medium strength, great conductivity, good plasticity, excellent corrosion resistance, long service life and easy processing. It is widely used in some components of automobiles, ships and aluminum alloy tubular buses [1, 2]. Nowadays, Al–Ti–C and Al–Ti–B master alloy were often used to refine aluminum and aluminum alloy [3–5]. However, Al–Ti–C and Al–Ti–B master alloys show lots of disadvantages in refining aluminum alloy [6–8]. Rare earth elements have many excellent properties, such as active chemical properties, forming stable oxides and sulfides with almost all non-metallic elements. The strength, hardness, elongation, fracture toughness, wear and corrosion resistance and other comprehensive mechanical properties of aluminum alloy can be improved by adding rare earth [9–12]. It has been found that the addition of some rare earth elements on the basis of Al–Ti–C/B master alloy can not only inhibit the aggregation and precipitation of the second phase particles in the master alloy, improve its refinement performance, but also improve the thermal deformation ability, corrosion resistance and surface finish [13–15]. Li et al [16] added Y and Al–Ti–B to hypereutectic Al-Si alloy, and found that it had significant refining effect on hypereutectic Al-Si alloy, and the mechanical properties of the alloy were significantly improved. Liu et al [17] added Al–Ti–B and Al–RE master alloys into Al–16Si–4Cu–0.5Mg–0.2Mn. The results show that the microstructure, tensile strength and elongation of the alloy were improved. In recent years, some researchers have added Nd to Al-Mg-Si, which improves the tensile strength and elongation of the alloy. Therefore, this method has been widely used in the fields of overhead conductor, automobile and architecture [18, 19]. Wang Di et al [20] found that the addition of Nd to Al-Si–Cu alloy and Al-Si alloy can effectively refine the grain of alloy, around the Si improve the tensile strength and cyclic deformation resistance of the alloy. Cheng Xiaomin et al [21] added Ce to 6063 aluminum alloy, and found that the grains were obviously refined with the increase of Ce.
content, the secondary dendrite spacing decreased at the same time; In addition the electrical conductivity of the alloy slightly decreased, while the tensile strength significantly increased at first and then decreased. In this paper, the refining effect on 6063 alloy by different Nd concentrations is discussed through a Nd and Al–Ti–B intermediate alloy composite addition method. For added elements, the existence form, distribution state and the influence mechanism of second phase particles were studied. Besides, the hardness and tensile properties of 6063 alloy after composite modification by Nd and Al–Ti–B intermediate alloy are analyzed.

2. Experimental materials and methods

6063 aluminum alloy (purchased from Dongguan zhengdu metal materials Co., Ltd), Al–10Nd master alloy (purchased from Suzhou top hot runner technology Co., Ltd) and domestic Al–Ti–B master alloy are used as raw materials.

First, 6063 aluminum alloy treated in a silicon carbide furnace at 730 °C, with 0.5 wt% refining agent and 0.3 wt% covering agent, keeping the temperature for 10 min. Then 0.2% Al–Ti–B and X% Nd (X = 0, 0.1, 0.2, 0.4, 0.6) rare earth elements were added respectively. After holding for 15 min, the sample is poured into the metal mold preheated at 200 °C, and then cooled to form a rod sample with a diameter of 13 × 130 mm. The sample number is shown in table 1.

The samples of 6063 aluminum alloy cast bars with different refinement and modification were made with same size, The Φ14 × 10 mm macro refinement samples and metallographic samples were made separately. The macro grain structure was observed and the refining effect was compared. The metallographic samples were subjected to electrolytic corrosion. The average grain size was measured by the Image-Pro Plus software. The microstructure of the electrolyte (10 ml HClO4: 90 ml c2h6o) was characterized by optical microscope (OM), field emission scanning electron microscopy (SEM), and electron probe microanalysis (EPMA). The morphology and distribution of nd-in-6063 alloy were discussed.

In order to explore the effect of Nd addition on the mechanical properties of 6063 aluminum alloy, the hardness test and room temperature tensile test were carried out in this paper. The heat treatment process of 6063 aluminum alloy is as follows: homogenization heat treatment (568 ± 5 °C, 4 h, air cooling) and T6 heat treatment (solution treatment temperature 525 ± 5 °C, 12 h, quenching medium is water; aging treatment temperature 200 ± 5 °C, 2 h, air cooling). Natural aging for 180 days. The hardness of 6063 aluminum alloy was measured by using the HBRVU–187.5. The pressure load was 100 N and the loading time was 30 s. The WILSON VH1102 microhardness tester was utilized to measure the micro HV of the grain boundary and the place inside the grain. The samples of 6063 aluminum alloy after refinement and modification were processed with the national standard, and the mechanical properties were tested under the AGS-X Shimadzu electronic universal tensile testing machine. The tensile rate is 1 mm min⁻¹, and the tensile strength and elongation are obtained (three tensile performance tests are conducted for each group of tensile samples).

3. Results and discussion

3.1. Microstructure of Al–10Nd master alloy and Al–Ti–B

Figure 1 shows the microstructure and energy spectrum of Al–10Nb and Al–Ti–B alloys. It can be seen from figure 1(a) that the microstructure of Al–10Nb master alloy is mainly the composite of Al matrix and Al₃Nb. Al₃Nb is evenly distributed on the matrix in strip and block shape. Figure 1(b) shows that the content of Al and Nd in the alloy is 94.3 wt% and 5.7 wt% respectively. It can be seen from figure 1(c) that the main phases of Al–Ti–B master alloy are 3–10 μm flake Al₃Ti and 1 μm granular TiB₂, which are evenly dispersed in the aluminum matrix. Figure 1(d) shows that the contents of Al, Ti and B in the alloy are 67.52 wt%, 27.09 wt% and 5.39 wt%, respectively.

| Table 1. The sample number of Al–Ti–B + x% Nd. |
|-----------------------------|-----------------------------|
| Alloy number | Alloy composition |
| #1 | 6063 |
| #2 | 6063 + 0.2% Al–STi–B |
| #3 | 6063 + 0.2% Al–STi–B + 0.1% Nd |
| #4 | 6063 + 0.2% Al–STi–B + 0.2% Nd |
| #5 | 6063 + 0.2% Al–STi–B + 0.4% Nd |
| #6 | 6063 + 0.2% Al–STi–B + 0.6% Nd |
3.2. Effect of Nd and Al–Ti–B on the microstructure of 6063 aluminum alloy

Figure 2 shows the refining effect of different contents of master alloy on 6063 aluminum alloy. The average grain size statistics of the alloy are shown in figure 3. As can be seen from figure 2(a), #1 alloy is composite of α-Al grains and eutectic structures continuously distributed at the grain boundaries. The α-Al grains exhibit dendritic and petal-like shapes with coarse and uneven distribution, such as figure 2(a). The average grain size of the alloy is about 482 μm. #2 alloy without Nd is mainly composed of cellular grain with different size and a large number of coarse second phases. Compared with #1 alloy, when the addition amount of Nd is 0.1 wt% and 0.2 wt% respectively, the coarse cellular grain α-Al in #3 and #4 alloy are refined into fine cellular grain, which shows that the addition of rare earth Nd process great refine on the grain refinement of 6063 aluminum alloy, as shown in figures 2(c), (d). When the Nd content continues to increase, the grain of the alloy is further refined. When the Nd content reaches 0.6 wt%, as shown in figure 2(f), the grain size of α-Al decreases, and many finer and rounder grains are distributed on the grain boundary. The second phase is mainly distributed in the grain boundary, and the precipitates are also increasing. The average grain size of the alloy is about 98 μm.

Existing studies believe that [6] the TiB2 particles in the Al–Ti–B master alloy can stably exist in the Al melt, so it can be determined that part of particles in the grains and at the grain boundaries are TiB2, and these TiB2 particles effectively act as heterogeneous α-Al crystal nuclei. Some studies have shown that the eutectic reaction takes place at the aluminum rich angle: L → Al + Al3Nd [22], This means that the intermetallic compound Al3Nd will precipitates from the melt with the addition of Nd during the solidification of the alloy. Due to the high melting point and chemical stability of Al3Nd phase, it has a small amount of mismatch with α-Al [23], and is completely coherent with Al matrix, it can be used as the core of heterogeneous nucleation of primary α-Al phase in 6063 aluminum alloy, increasing the amount of heterogeneous nucleation and refining α-Al grain.

In order to study the effect of addition Nd on the microstructure and second phase of 6063 aluminum alloy, the microstructure of 6063 aluminum alloy with two different Nd contents was compared, in figures 4(a), (d), without the addition of master alloy, there are a lot of coarse precipitates about 25 μm and granular precipitates about 1 μm at the grain boundary of #1 alloy, which exist independently. When the addition amount of Nd is 0.1 wt%, as shown in figures 4(b), (e), the precipitated phase in the #3 alloy is short rod or granular, mostly precipitated along the grain boundary, and with a size of about 3–8 μm. When the addition amount of Nd
increases to 0.6 wt%, as shown in figures 4(c), (f), the grain size of the #6 alloy decreases, and a grid like compound with a size of about 5–12 μm is observed at the grain boundary.

Figure 5(a) is the SEM microstructure of #3 alloy, and figure 5(b) is EDS energy spectrum analysis of point A. As shown in figure 6(b), the white particles in figure 6(a) contain Al, Si, Mg, Nd and Fe, indicating the formation of AlFeSiMgNd compounds at the grain boundary. Figure 6 is the SEM micrograph of #6 alloy and the corresponding surface scanning measurement results. It can be seen that when the content of Nd is 0.6 wt%, the bright white grid like compound at the grain boundary is rich in Al, Nd and Si elements, which can be identified as AlSiNd compound.

Figure 7 shows the EPMA surface scanning spectrum of #6 alloy. It can be seen that the α-Al phase boundary of #6 alloy is rich in Si, Fe, Mg and Nd elements. Besides, Fe and Si coexist and distribute in the α-Al phase grain boundary in the form of discontinuous ball or short rod. There is a small amount of Nd in the coexistence of Al and Si, which is basically in the form of granular, and there is also a small amount of Nd exist in the grain. In addition, Mg and Si also coexist in a small amount. In conclusion, the main chemical phases at the grain boundary are AlFeSi, AlSiNd and Mg2Si.
From the above analysis, the grain boundary is mainly composite of short rod-shaped or discontinuous granular AlFeSiMgNd complex compounds with a particle of Nd. However, when the content of Nd is too much, the Nd will enriched at the grain boundary, and formed the grid compound AlSiNd with Si and Nd elements. Generally speaking, the existence form of rare earth in aluminum alloy is closely related to its solid solubility in aluminum matrix. When the content of Nd is lower than the solid solubility, it mainly exists in the form of solid solution. On the contrary, it mainly exists in the form of supersaturated solid solution or compound. The solid solubility is closely related to electronegativity and atomic radius. According to Hume-Rother criterion [24], it is difficult to form solid solution in aluminum alloy when the atom radius difference between rare earth and aluminum exceeds 15%. It is easier to form compounds when the electronegativity difference between Nd and aluminum is more than 0.4–0.5. The difference between the radius of rare earth atom and aluminum atom is 84.6%. The electronegativity is 1.14 and 1.61 respectively, and the difference between them is 0.47. In the process of alloy solidification, rare earth Nd is easily pushed to the grain boundary. In
addition, the electronegativity between elements is large, and the binding ability between atoms is strong, so it is easier to form compounds. The electronegativity of Si, Mg and Nd are respectively 1.8, 1.2 and 1.14. The difference of electronegativity between Mg and Si is only 0.14, which is far less than that between Si and Nd. Therefore rare earth Nd and Si elements are easier to form AlSiNd compounds in aluminum matrix. It should be noted that the Nd can also form supersaturated solid solution in $\alpha$-Al during non-equilibrium solidification. We can be sure of the fact that rare earth Nd in 6063 aluminum alloy exists not only in the form of compound, but also in the form of solid solution in $\alpha$-Al.

**Figure 6.** SEM image and EDS spectra of #6 alloy: (a) SEM image; (b)-(f) elemental mapping of Al, Fe, Si, Mg and Nd.

**Figure 7.** EPMA analysis of #6 alloy: (a) back-scattered electron image; (b)-(f) x-ray image of Al, Fe, Si, Mg, Nd elements.
3.3. Effect of composite modification of Nd and Al–Ti–B on mechanical properties and fracture morphology of 6063 aluminum alloy

Figure 8 shows the change of tensile strength and elongation of 6063 aluminum alloy after T6 treatment and natural aging for 180 days. It can be seen from the figure 8 that the tensile strength of 6063 aluminum alloy is 152.4 MPa, and the elongation is 11.8%. Different contents of Nd and Al–Ti–B master alloy were added in 6063 aluminum alloy and greatly changed its properties. The tensile strength is 213.0 MPa, 204.1 MPa, 187.6 MPa, 155.1 MPa and 139.4 MPa, and the elongation is 14.3%, 15.9%, 16.4%, 19.9% and 24.4% respectively. The tensile strength of 6063 aluminum alloy with 0.2% Al–Ti–B + x% Nd (x = 0, 0.1, 0.2, 0.4, 0.6) decreases with the increase of rare earth Nd content, but the elongation rate increases with the Nd content increase. Compared with #6 alloy, the tensile strength of #1 alloy decreased from 152.4 MPa to 139.4 MPa, decreased by 8.5%, and the corresponding elongation increased from 11.8% to 24.4%.

Figure 9 shows the stress-strain curve of 6063 aluminum alloy T6 heat treated and naturally aged for 180 days with Al–Ti–B + x% Nd (x = 0, 0.1, 0.2, 0.4, 0.6). It is further confirmed that the tensile strength of the alloy decreases with the increase of rare earth Nd content, but the elongation increases with the increase of rare earth Nd content.

Figure 10 shows the HV of 6063 aluminum alloy after T6 heat treatment and natural aging for 180 days with 0.2% Al–Ti–B + x% Nd (x = 0, 0.1, 0.2, 0.4, 0.6) intermediate alloy added. It can be seen that on the basis of Al–Ti–B master alloy, adding 0.1 wt%, 0.2 wt%, 0.4 wt% and 0.6 wt% Nd, the HV value of Al–Ti–B master alloy...
is lower than that of 6063 aluminum alloy only adding Al–Ti–B master alloy, and the HV of #5 is the most obvious one, the HV is reduced from 78.9 HV to 49.2 HV, 37.6% lower.

In order to analyze the effect of addition Nd on the hardness of 6063 aluminum alloy, the microvickers hardness of #2 and #6 alloy was measured, and the microhardness indentations are shown in figure 11. We obtain that the average hardness in the crystal of #2 alloy is 27.3 HV, and the average hardness of grain boundary is 39.2 HV. The average hardness of alloy #6 is 22.0 HV, the average hardness of grain boundary is 34.0 HV. The hardness in the interior and the grain boundary of #6 alloy is lower than #2 alloy. It can be concluded that the addition of Nd will indeed lose part of the hardness of 6063 aluminum alloy.
The analysis shows that after adding Nd, on the one hand, the interaction results between Nd and Si form AlSiNd compound phase at first, which makes the distribution of compound precipitated at the grain boundary of 6063 aluminum alloy more continuous, forming a long strip or grid shape, and the homogeneity of the structure decreases. Moreover, the splitting effect of this kind of grid or long strip compound on the matrix increases, and it is easy to cause stress concentration at the interface. Finally, the strength of the alloy is reduced. On the other hand, the amount of Mg adsorbed in Si is decreased and the formation of Mg2Si in the alloy structure is inhibited. As the main strengthening phase of Al-Mg-Si, the decrease of Mg2Si will undoubtedly reduce the strength and hardness of the alloy. Therefore, the strength and hardness of the alloy will be reduced after the addition of rare earth Nd, although its elongation is increased.

In order to exhibit the effect of Nd on the mechanical properties of 6063 aluminum alloy more clearly, especially the effect on the plasticity. The fracture surface of 6063 aluminum alloy and 6063 aluminum alloy T6 with 0.2% Al–Ti–B + x% Nd (x = 0, 0.1, 0.2, 0.4, 0.6) after 180 days of heat treatment and natural aging were observed by SEM. It can be seen from figures 12(c)–(f), with the increase of Nd content in the fracture surface, the number of dimples in the fracture surface of #3 - #6 alloy increases. The macro fracture of the alloy presents more pits with a large number of second phase particles at the bottom. Besides, with the increase of rare earth Nd, the second phase appears the number of phase particles also increases. Generally speaking, the mechanical properties of the second phase particles and the matrix are different, the size and morphology of the second phase particles are also different, and the binding energy between the second phase particles and the matrix is also different. Therefore, these second phase particles are easy to form the core of microporous cracks with the increase of stress in the plastic deformation process. When the stress increases further, the crack begins to propagate to the periphery of the core, and the alloy finally breaks.

Figure 13 is a further enlarged morphology of the macro fracture. From figure 13(a), it can be seen that the fracture of #1 alloy is generated on the surface of the alloy sample, and there are a large number of AlFeSi phases at the crack source, which indicates that due to the segregation of large-size AlFeSi and other complex compounds at the grain boundary, the cracks are generated and the intergranular fracture occurs in the sample. The fracture surface is not smooth, there is obvious plastic deformation before fracture, and the fracture surface is cup-shaped. The micro morphology is composite of dimples with great difference in size. The matrix alloy has a mixed fracture of toughness and embrittlement, which is mainly ductile fracture. The fracture surface of #2 alloy is composite of tearing edge and a small amount of dimples, which are distributed unevenly, and obvious intergranular fracture occurs in some areas, which indicates that the matrix alloy has a mixed fracture of toughness and embrittlement. The fracture surface of #3 alloy is similar to that of alloy 13 (b). The fracture surface is composite of tearing edge and dimple. Some of the tearing edges become shorter. There are more cleavage surfaces between the tearing edges, which indicates that the matrix alloy has a mixed fracture of
toughness and embrittlement. With the increase of Nd, the dimples become smaller and shallower. As shown in figure 13(d), the dimples on the fracture surface of alloy #4 are obviously dense, fine, evenly distributed, and their plasticity is significantly improved. #5 and #6 alloys have more second phase particles, it is worth noting that the dimple around the second phase particles is smaller, as shown in figures 13(e), (f). The fracture mode is typical ductile fracture, and the alloy has higher elongation, which is consistent with the mechanical property test results. It has been reported [25] that the grain refinement increases the work hardening effect and reduces the elongation of the material. However, in this study, the elongation increases. This is because the macro mechanical properties and deformation behavior of aluminum alloy not only obey Hall-Petch formula, but also are affected by other factors. From the experimental results, it can be seen that rare earth Nd is easy to combine with Si to form a coarse network of eutectic compound AlSiNd. These coarse network of eutectic compounds make the amount of Mg2Si, the strengthening phase of 6063 alloy, decrease, and easily accumulate at the grain boundary. During the plastic deformation of the alloy, the stress tends to concentrate around these particles, generate cracks and expand, so that the alloy breaks.

In order to study the mechanism of the effect of the second phase particles on the tensile properties of 6063 aluminum alloy, energy spectrum analysis and microstructure fracture analysis were completed. Figure 14 shows the EDS point analysis of the fracture surface of alloy #6. It is clear that a large number of fine particles are distributed on the fracture surface. Combined with phase analysis at the previous grain boundary, these particles may be AlFeSi, AlSiNd and AliFeSiMgNd compounds. This also confirms the previous analysis that the fracture close to the particles AlFeSi, AlSiNd and AliFeSiMgNd.

4. Conclusions

(1) The composite modification of Nd and Al–Ti–B master alloy has a great refining effect on 6063 aluminum alloy. With the increase of Nd addition, the size of α-Al decreases gradually, some coarse cellular crystals are refined into fine cellular crystals.

(2) The addition of Nd has an effect on the microstructure of 6063 aluminum alloy. When the addition amount of Nd is 0.1 wt%, the grain boundary is mainly composite of short rod-shaped and discontinuous granular AlFeSiMgNd complex compounds. When the content of Nd reaches 0.6 wt%, the rare earth Nd is enriched at the grain boundary and the grid with AlSiNd phase is formed.

(3) After 180 days of natural aging on the basis of T6 heat treatment, the elongation increases obviously with the increase of Nd addition, but the tensile strength and Vickers hardness decreased. After 0.2% Al–Ti–
B + 0.6% Nd composite modification, the tensile strength, elongation and HV of 6063 aluminum alloy reached 139.4 MPa, 24.4% and 49.2 HV respectively, the elongation increased by 70.6% while the tensile strength decreased by 34.6% and the HV decreased by 37.6%. The alloy fracture is ductile, and there are AlSiNd and AlFeSiMgNd particles on the fracture surface.

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Author contributions

Wanwu Ding and Yan Cheng conceived and designed the experiments; Wanwu Ding, Yan Cheng and Xiaoyan Zhao carried out the experiments and data collection; Wanwu Ding, Yan Cheng, Xiaoyan Zhao, Taili Chen, Haixia Zhang, Wenjun Zhao analyzed the data; Wanwu Ding contributed reagents/materials/analysis tools; Yan Cheng and Xiaoyan Zhao wrote the paper.

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