Effect of Al–Nb–B_{2}O_{3}–CuO system reaction products on microstructure and wear resistance of A356 alloy

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Abstract

The aim of present work is study the effect of Al–Nb–B_{2}O_{3}–CuO system reaction products on microstructure and properties of A356 alloy. B_{2}O_{3} powder, Al powder, CuO powder and Nb powder were used as raw materials to form Al–Nb–B_{2}O_{3}–CuO reaction system, and A356 alloy was refined by in situ reaction and near-liquidus casting. The size, phase, morphology and distribution of in situ reaction products were observed and analysed by optical microscope, x-ray diffractometer, scanning electron microscope and transmission electron microscope. The products of the Al–Nb–B_{2}O_{3}–CuO reaction system are mainly Al\textsubscript{3}Nb, NbB\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3}. The ratio of the prefabricated block is 8:1:1:2. When the prefabricated block was added at 2.5 wt\%, the particle size of NbB\textsubscript{2}, Al\textsubscript{3}Nb and Al\textsubscript{2}O\textsubscript{3} is 0.5–1 \(\mu\)m. The microstructure of A356 alloy is fine and uniform. The average grain size is refined to less than 20 \(\mu\)m. Compared with untreated A356 alloy, the wear rate is reduced from 2.091% to 0.882%, and the average hardness of the alloy is increased from 41.3HRC to 58.2HRC.

1. Introduction

A356 alloy is the most widely used material in cast aluminium alloy and is commonly used in fields, such as automobiles, aerospace, defence and industrial engineering [1]. However, A356 alloy produces coarse grains, such as dendrites and columnar crystals, during casting production, and these grains, in turn, reduce the comprehensive properties of A356 alloy [2–4]. Aluminium matrix composite material reinforced by ceramic particles exhibits excellent mechanical properties because of the plastic toughness of the matrix material and the high hardness of the ceramic particles [5]. Near-melting casting can obtain a composite material with a small microstructure by improving the supercooling degree of the melt [6, 7].

Bolzoni L and Nowak M [8, 9] found that Al\textsubscript{2}O\textsubscript{3} and NbB\textsubscript{2} intermetallic compound particles formed in situ are heterogeneous nuclei for grain refinement of cast aluminium alloys. S. Mandal and Srirangam et al [10] prepared Al–5Nb–1B master alloy as a refiner and observed that the intermetallic compound particles of the master alloy are uniformly distributed in the aluminium alloy matrix; hence, the alloy can be used as a potential grain refiner. Tijun Chen et al prepared A356 alloy by in situ reaction and reported overheated melting, short reaction time, uncontrolled reaction process, unevenly distributed reaction product particles, difficulty in combining the interface between particles and melt and serious particle agglomeration, which will cause abrasive wear and sharp decrease in the wear properties of the composites [11, 12].

Although Al–Nb–B refiner solves the problem of ‘refinement poisoning’ [13–15], Nb powder easily agglomerates in the alloy melt due to its high density and insufficient reaction power; as such, stirring must be carried out during the test to make it fully react. Therefore, a more efficient process using Al–Nb–B refining agent should be developed to obtain a simple synthesis method and an ideal refining effect. In this study, CuO was added to the Al–Nb–B system through in situ reaction and near-melting point casting to evaluate the effect of the Al–Nb–B\textsubscript{2}O\textsubscript{3}–CuO system reaction products on the microstructure and properties of A356 alloy.
2. Experimental

2.1. Preparation of sample materials

The main chemical composition of A356 alloy is shown in Table 1.

The preparation proceeded as follows: B2O3 powder (purity > 99.9%, 300 mesh), Al powder (purity > 99.9%, 300 mesh), CuO powder (purity > 99.9%, 300 mesh) and Nb powder (purity > 99.9%, 300 mesh) were mixed in a mixing machine for 60 min in an appropriate proportion, in which the molar ratio of Al:Nb:B2O3:CuO was 8:1:1:2 in the Al–Nb–B2O3–CuO system. The mixed powders were pressed into φ 20 mm × 10 mm of precast blocks and placed into a furnace for drying. After the substrate was completely dissolved, the solution was washed with distilled water and alcohol several times and dried to prepare powder samples. The obtained powders were examined by x-ray diffraction (XRD, D/Max 2500PC, Rigaku, Japan) and scanning electron microscopy (SEM, Model S-3400N, Hitachi, Japan) equipped with energy-dispersive x-ray spectroscopy (EDS). Transmission electron microscopy (TEM, Model JEM-2010, Hitachi, Japan) was used to observe the morphology and microstructure of the samples.

2.2. Phase analysis

The composite samples were placed into hydrochloric acid to corrode the substrate. After the substrate was completely dissolved, the solution was washed with distilled water and alcohol several times and dried to prepare powder samples. The obtained powders were examined by x-ray diffraction (XRD, D/Max 2500PC, Rigaku, Japan) and scanning electron microscopy (SEM, Model S-3400N, Hitachi, Japan) equipped with energy-dispersive x-ray spectroscopy (EDS). Transmission electron microscopy (TEM, Model JEM-2010, Hitachi, Japan) was used to observe the morphology and microstructure of the samples.

2.3. Test

A Rockwell hardness tester was used to calculate the hardness of each sample with a load of 150 g and holding for 30 s. Each sample of aluminium matrix composites with reinforcement particles was tested five times to reduce error, and the average was determined.

Friction and wear performance test was conducted on MM-W1 vertical universal friction and wear tester under the following experimental conditions: load of 100 N, rotating speed of 100 r min⁻¹ and wear time of 120 s. After the rotation was terminated, the wear amount of the sample was measured using an analytical balance. The wear resistance of the material was characterised according to wear amount.

3. Results and discussion

Figure 1 shows the XRD results of the sample after acid etching. Three reaction products of Al3Nb, NbB2 and Al2O3 particles were obtained. The reaction products of the Al–Nb–B2O3–CuO system were analysed. After element B was added to the A356 alloy melt, a NbB2 metal compound with Nb was formed. NbB2 particles can be used as α-Al phase heterogeneous cores, and the growth of Al grains provides an effective nucleation substrate [16]. The Al–Nb–B2O3–CuO system not only provides active [B] atoms but also reaction power for the reaction system given that the reaction between B2O3 and Al is exothermic. This phenomenon is similar to the addition of CuO, where the reaction proceeds fully and generates more Al3Nb, NbB2 and Al2O3 reaction particles. The Al–Nb–B2O3–CuO reaction system exhibits better refinement performance compared with refiner Al–Nb–B series master alloy. A356 alloy was refined by the master alloy; if the reaction product has large Al3Nb, it cannot be used as the heterogeneous core of Al, which will affect the refining effect of the alloy. The Al–Nb–B2O3–CuO system generates more heat by in situ reaction. The reaction is rapid and full and the reaction product Al3Nb has small morphology, leading to better refinement effect. The reaction products NbB2 and Al2O3 particles are high-melting, stable metal compounds, and Al3Nb and NbB2 play the role of heterogeneous nuclei.

After etching the aluminium matrix composites prepared by the system, the residual powders were studied by SEM to observe the morphology of the reaction products. Figure 2 shows the residual powder after etching the aluminium matrix composite prepared by the Al–Nb–B2O3–CuO system. Al3Nb phase, NbB2 phase and Al2O3 phase are evenly distributed. Figure 2(b) shows that the Al2O3 particles are located at the bottom. The NbB2 phase is formed on the Al2O3 phase, and the Al3Nb phase is formed on the NbB2 phase. B2O3 reacts before Al to form Al2O3 and the active element B, which then reacts with Nb to form NbB2. Al3Nb grows on NbB2.

Table 1. The components of A356 Al alloy (mass%).

| Alloy | Si   | Fe  | Cu  | Mn  | Mg  | Zn  | Ti  | Al  |
|-------|------|-----|-----|-----|-----|-----|-----|-----|
| A356  | 6.5  | 7.5 | 0.1 | 0.05| 0.30| 0.45| 0.05| 0.20| 91.53~92.68 |
Al\textsubscript{3}Nb has a hexagonal prism shape with a size of about 1 μm. The Al\textsubscript{3}Nb phase formed on the NbB\textsubscript{2} particles mainly plays a role in heterogeneous nucleation in the α-Al phase, thereby increasing the nucleation rate of the α-Al phase. Given that NbB\textsubscript{2} and Al\textsubscript{3}Nb in the melt have a small wetting angle, they have better wettability. According to double nucleation theory, the surface of NbB\textsubscript{2} is enriched with numerous Nb atoms, and Al\textsubscript{3}Nb particles are precipitated with NbB\textsubscript{2} as the matrix. NbB\textsubscript{2} and Al\textsubscript{3}Nb are agglomerated in figure 2(b). In subsequent solidification, a peritectic reaction occurs to refine α-Al phase crystal grains, and the reaction generates excess Al\textsubscript{3}Nb, which dissolves quickly; meanwhile, NbB\textsubscript{2} has a high melting point and still exists in the solid melt. The plane formed by the B–B bond in the NbB\textsubscript{2} crystal structure provides the possibility for Al\textsubscript{3}Nb to nucleate on its surface. Therefore, Nb will preferentially form a Nb-rich layer around NbB\textsubscript{2} particles, which will be the centre, and Al\textsubscript{3}Nb will form around NbB\textsubscript{2}.

TEM was employed to further investigate the distribution of NbB\textsubscript{2} particles in the aluminium-based composites prepared by the Al–Nb–B\textsubscript{2}O\textsubscript{3}–CuO system. Figure 3 shows the TEM bright-field image and energy spectrum of the NbB\textsubscript{2} particles in the reaction product of the Al–Nb–B\textsubscript{2}O\textsubscript{3}–CuO system and the electron diffraction pattern. DM software was used to calibrate the diffraction pattern spots, measure the spacing of each crystal plane and compare it with the NbB\textsubscript{2} PDF card. The four points in the calibration diagram are (101), (111), (010) and (300) crystal planes. This particle is identified as NbB\textsubscript{2} particle. In the TEM brightfield image, NbB\textsubscript{2} particles are polygonal and have a size of less than 1 μm, belonging to the sub-micron level. The interface between the NbB\textsubscript{2} particles and the matrix is clean, pollution free and well-bonded and is distributed inside the crystal grains. The interface acts as a nucleating agent during alloy crystallization and promotes grain refinement.

Figure 4 shows the same process parameters and the formulation of the molar ratio of each component of the in situ reaction product. Al/CuO is unchanged at 4:1, and the Nb/B\textsubscript{2}O\textsubscript{3} ratio is adjusted. The samples are as follows: (a) Al:Nb:B\textsubscript{2}O\textsubscript{3}:CuO = 8:1:0.1:2; (b) Al:Nb:B\textsubscript{2}O\textsubscript{3}:CuO = 8:1:0.3:2; (c) Al:Nb:B\textsubscript{2}O\textsubscript{3}:CuO = 8:1:0.5:2; (d) Al:Nb:B\textsubscript{2}O\textsubscript{3}:CuO = 8:1:0.8:2; (e) Al:Nb:B\textsubscript{2}O\textsubscript{3}:CuO = 8:1:1:2; and (f) Al:Nb:B\textsubscript{2}O\textsubscript{3}:CuO = 8:1:1.2:2. As the ratio of Nb/B\textsubscript{2}O\textsubscript{3} changes from 1:0.1 to 1:1.2, the content of B\textsubscript{2}O\textsubscript{3} powder gradually increases, the reaction intensifies and more active [B] atoms are produced, resulting in complete reaction, more reaction products and finer crystal grains. Figure 4(a) shows the phenomenon of grain refinement, but the refinement is not uniform, the particle size is different and columnar crystals are obvious. The addition of a low amount of B\textsubscript{2}O\textsubscript{3} powder and the insufficient NbB\textsubscript{2} content generated is insufficient lead to the refinement effect on Al\textsubscript{3}Nb and Al\textsubscript{2}O\textsubscript{3} particles. In figure 4(b), when the ratio of Nb/B\textsubscript{2}O\textsubscript{3} is changed from 1:0.1 to 1:0.3, the crystal grains are refined and the size is uniform, but columnar crystals still exist. Upon adding increased amount of B\textsubscript{2}O\textsubscript{3} powder and thermite, the number of reactive [B] atoms produced by the reaction \(\text{Al} + \text{B}_2\text{O}_3 \rightarrow \text{Al}_2\text{O}_3 + \text{B}\) increases, so the amount of the in situ reaction product NbB\textsubscript{2} begins to increase. Al\textsubscript{2}O\textsubscript{3}, Al\textsubscript{3}Nb and NbB\textsubscript{2} particles work together as the heterogeneous core of the α-Al phase.

In figure 4(c), with further increase in the amount of B\textsubscript{2}O\textsubscript{3} powder added, the Nb/B\textsubscript{2}O\textsubscript{3} ratio reaches 1:0.5, resulting in the formation of finer grains, with an average size of less than 50 μm, increase in the number of equiaxed crystals and decrease in the number of columnar crystals. Figures 4(d) to (e) shows that the crystal grains are further refined with increasing Nb/B\textsubscript{2}O\textsubscript{3} ratio. The reaction product contains more NbB\textsubscript{2}, leading to better fineness to the microstructure of the alloy. The increase in the amount of B\textsubscript{2}O\textsubscript{3} powder can promote the large-scale production of NbB\textsubscript{2}. The number of NbB\textsubscript{2} particles gradually increases, and the non-uniform
nucleation effect in the melt is enhanced, resulting in a finer grain structure. However, figure 4(f) shows that the crystal grains are finer due to the addition of excessive B2O3 powder and more Al2O3 and active [B] atoms are generated. Excessive active [B] atoms will react with Al in the matrix to form AlB2 (Al + 2B → AlB2). AlB2 also has the effect of refining the α-Al phase, so four particles of Al2O3, Al3Nb, NbB2 and AlB2 will appear to refine the α-Al phase grains. During solidification, it will be enriched and segregated, and the subcooling of the components will be increased for modification and refinement to obtain better refinement effect and smaller grains, some of which are less than 20 μm. Excessive B2O3 powder will agglomerate the reaction products, resulting in the splashing out of the Al– Nb– B2O3– CuO system products from the alloy metal liquid matrix. In this regard, the actual amount of the in situ reaction products added is difficult to determine. Therefore, the molar ratio of Al:Nb:B2O3:CuO = 8:1:1:2 was selected to obtain ideal refining effect not only to ensure that the thermite reaction between CuO and Al increases the heat of reaction and is fully completed but also to control
the experiment within a safe range. After adding appropriate amount of CuO powder and B₂O₃ powder to the Al–Nb–B₂O₃–CuO system, the thermit reaction can provide more heat to the system, promote the progression of the system reaction and ensure thorough in situ reaction in the system. The reaction product is small but produced in large quantity, and the A356 alloy has uniform microstructure and fine distribution.

The Al–Nb–B₂O₃–CuO system generates the highest amount of reaction products of Al₂O₃ particles after etching of the composites. The residual powders are shown in figure 5. The morphology of Al₂O₃ particles is close to round, and they are embedded in the A356 alloy matrix and have many dislocations. The small, stable and hard reinforcing particles block the slip of the dislocations through the pinning effect to strengthen the A356 alloy matrix. Based on the SEM and TEM results, the Al₂O₃ particle has a size of about 1 μm and approximately spherical shape and is embedded on the surface of the A356 alloy matrix [17–19].

Figure 6 shows the scan of the wear surface of the sample with different preform additions in the Al–Nb–B₂O₃–CuO system. The A356 alloy has high mass wear rate, poor wear resistance, deep furrow and obvious grooves on the wear surface. Few adhesive bumps and plastic deformation occur, and the shearing action mainly occurs on the joint surface. The migration of wear debris is not obvious, and the formation of adhesive wear is dominant. When the amount of the prefabricated block is 1 wt%, the A356 alloy sample still appears adhesion during the wear process due to the plastic deformation caused by the extrusion and scraping of the sandpaper. The buried depth of the hard spots on the surface is reduced, and the surface is more obvious and has many scars but shallow depth. In addition to the more obvious wear marks on the worn surface, spalling occurs due to the adhesion effect, and the alloy has a slight adhesion wear pattern. When the in situ reinforcement content is small, the amount of particles is insufficient to support friction with a pressure of 100 N. Therefore, increasing the particle content can enhance the wear resistance of the alloy. Figure 6(c) shows the Al–Nb–B₂O₃–CuO system’s prefabricated block with 1.5 wt% A356 alloy. The metal is peeled off or transferred, and surface adhesion area is formed due to the adhesion effect. The adhesive wear of the alloy and the wear of the peeling layer occur. However, the peeling phenomenon caused by the shear fracture formed by the adhesion point is reduced because the refined and modified alloy has higher plasticity. The adhesion effect caused by the alloy is mainly due to the increase in plasticity, and toughness can reduce shear failure. Figures 6(d) and (e) show that the refined A356 alloy has good wear resistance when the preform addition amounts are 2 wt% and 2.5 wt%. Given that the structure is more uniform, the hardness increases, the surface becomes smoother and the grinding marks are further reduced. The more obvious rough wear marks basically disappear because during wear, the relatively soft alloy matrix is first contacted with sandpaper and worn away. The generated NbB₂ and Al₂O₃ particles are exposed to the surface to bear most of the load (figure 6(f)). After the
prefabricated block is added in 3 wt%, the surface of the alloy is obviously cracked mainly due to the addition of excessive in situ reaction products during the friction and wear process. The generated Al2O3, NbB2 and hard Al3Nb particles are prone to agglomeration when the content is large, thereby increasing the defects of the material and losing the organisation. As such, the particles are easily peeled off, the secondary wear of abrasive particles to the matrix increases, the wear resistance of the composite material is reduced and micro-cutting occurs. The worn surface produces wear or groove marks along the friction direction, so the surface has grooves. When more wrinkles are present, the alloy has two forms of abrasive wear and adhesive wear, and the contact surface produces a higher friction temperature within shorter time. The grains of the A356 alloy are refined, and the strength and toughness are improved. The surface of the newly ground alloy undergoes slight plastic deformation, and the surface is continuously exposed and oxidised. A friction layer that differs from the structure and chemical composition of the matrix is formed on the abrasive surface. The layer is gradually distributed with the load of the medium evenly and densely, thereby improving the bonding strength with the matrix. When the thickness of the oxide friction layer reaches a certain size, it can effectively prevent the direct contact between the sandpaper and the substrate and elicits good protective properties [20]. Under these

Figure 4. Microstructure of samples using Al–Nb–B2O3–CuO system (different Nb/B2O3 ratio). (a) Al:Nb:B2O3:CuO = 8:1:0.1:2; (b) Al:Nb:B2O3:CuO = 8:1:0.3:2; (c) Al:Nb:B2O3:CuO = 8:1:0.5:2; (d) Al:Nb:B2O3:CuO = 8:1:0.8:2; (e) Al:Nb:B2O3:CuO = 8:1:1:2; (f) Al:Nb:B2O3:CuO = 8:1:1.2:2.
conditions, the wear surface is characterised by abrasive particles and slight oxidative wear. The wear mechanism is the coexistence of abrasive wear, adhesive wear and slight oxidative wear.

The wear resistance of the alloy has a positive relationship to the hardness of the matrix. The higher the hardness is, the better the wear resistance will be. The reaction products affect the hardness of the matrix through the Orowan mechanism and influence the A356 alloy. The strengthening effect of the Orowan mechanism is no longer obvious when the size of the reaction product of the system is greater than 5 μm, while the strengthening effect is very obvious when the size of the dispersed in situ reaction product is at the sub-micron level. Based on analysis of the in situ reaction products, the Al–Nb–B2O3–CuO system reaction products have Al2O3, Al3Nb and NbB2 particles with sizes less than 2 μm.

Dislocations pass through the dispersed particles. The dislocations bend due to the blocking of strong particles. Under the action of shear stress $\tau_i$, the curvature radius of the dislocation is:

$$ R = \frac{G_m b}{2\tau_i} $$

$$ \tau_c = \frac{G_m b}{D_p} $$

where $b$ is the Burgers vector and $G_m$ is the shear modulus of the matrix. When the shear stress is so large that the radius of curvature of the dislocation is $1/2D_p$, dislocation movement occurs in the matrix and A356 alloy undergoes plastic deformation. The shear stress is the yield strength $\tau_c$ of A356 alloy, and $\tau_c$ is the critical stress required for the dislocation to bypass the particle. Therefore, the smaller the particle size and the higher the volume fraction are, the stronger the ability of the dispersed particles to hinder the movement of dislocations and the better the enhancement effect will be.

In figure 7, the Al–Nb–B2O3–CuO system is added in the prefabricated block from 0% to 3%. The hardness of the Al–Nb–B2O3–CuO system reaches a large value when the preformed block is added at 2.5% and the average hardness is increased from 41.3HRC to 58.2HRC. After refinement, the hardness value of A356 alloy basically increases first and then decreases due to two main reasons. From the microscopic point of view, when the preform amount of the Al–Nb–B2O3–CuO system added to the reaction products Al2O3 and Al3Nb gradually increases and the content of NbB2 particles increases, the grains of A356 alloy gradually become
smaller and more equiaxed are refined. According to the grain refinement strengthening mechanism, the reaction product of the system becomes the core of non-uniform nucleation during condensation. A low degree of undercooling promotes the nucleation and growth of A356 alloy melt, thereby obtaining a higher nucleation rate due to the increase in number of grain boundaries. The enthalpy of the free energy of the liquid-solid phase becomes worse, and the interface of the particle formation is distributed along the grain boundary, which hinders the lateral growth of the crystal grains and promotes the rounding of the crystal grains during formation. These phenomena effectively improve the hardness of A356 alloy. From the macro point of view, Al2O3, AlN, and NbB2 particles have high hardness, round shape and small size, which directly increase the hardness of the alloy. However, when the Al–Nb–B2O3–CuO system preformed block is added at more than 2.5 wt%, the Rockwell hardness of A356 alloy shows a downward trend. The excessive particle content leads to easy segregation on the matrix during casting, thereby increasing the hardness. The uneven distribution of particles affects its strengthening effect. In addition, the agglomeration of the reaction products leads to weakened refining effect. The obtained crystal grains mostly present columnar crystals and dendrites, and the grain boundaries are reduced, so no strengthening effect on the matrix occurs.

Figure 6. Abrasion surface morphology of the samples prepared with Al–Nb–B2O3–CuO system (under different conditions of addition) (a) 0.5%; (b) 1.0%; (c) 1.5%; (d) 2.0%; (e) 2.5%; (f) 3%.
4. Conclusions

(1) The in situ reaction products of the Al–Nb–B2O3–CuO system are Al₃Nb, NbB₂ and Al₂O₃, which have particle size of about 0.5–2 μm and oval shape. Al₃Nb and NbB₂ have similar crystal structure to the α-Al phase, have a low mismatch value and play a role in heterogeneous nucleation and refining α-Al phase.

(2) The suitable reaction process parameters of the Al–Nb–B2O3–CuO system are preform moulding pressure of 10 MPa, powder mixing time of 60 min, preheating temperature of 950 °C and reactant ratio of 8:1:1:2 Al:Nb:B₂O₃:CuO. The Al–Nb–B₂O₃–CuO system prefabricated block was added at 2.5 wt%. The in situ A356 alloy prepared at a casting temperature of 650 °C has uniform microstructure and small grains, with average micro grain size refined to 20 μm, and its hardness increased from 41.3 HRC to 58.2 HRC.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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