Microstructures and mechanical properties of 4 wt%TiB₂/Al-Si-Cu-Zn (T6) composite thin-walled shell housing fabricated by high pressure die casting

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Abstract
The application demand of lightweight high-quality aluminum alloy parts in automotive and aerospace fields is increasingly. In aluminum matrix composites, reinforcing particles can significantly improve the performance of the matrix. In this paper, the microstructures and mechanical properties of die-cast 4 wt%TiB₂/Al-9Si-3Cu-0.8Zn composite were systematically analyzed by x-ray diffraction, optical microscope, scanning electron microscopy, energy dispersive spectrometer, transmission electron microscopy and tensile testing. The composite was successfully fabricated from an Al-K₂TiF₆-KBF₄ system by in situ melting technique. The research results show that the average grain sizes of the α-Al phase gradually decreased with the increase of filling distance. And the TiB₂ particles were distributed around eutectic Si in irregular polyhedral morphology or nearly circular shape. Meanwhile, the crystal structures of Ti-B compound and long needle shaped nano-sized precipitated were identified and analyzed, and they were found to be TiB₂ and Al₂Cu phase, respectively. Tensile testing results show that the mechanical properties of die-cast composite clearly increase after T6 heat treatment. The yield strength, ultimate tensile strength and elongation could reach 311 MPa, 379 MPa and 2.8% respectively, with the best injection velocity (1.8 m s⁻¹). The significantly enhancement of mechanical properties of composite after T6 heat treatment was mainly due to the introduction of TiB₂ reinforcing phase and the precipitation of Al₂Cu precipitate in the aging stage. The results implied that the introduction of TiB₂ reinforced particles could improve the mechanical properties of die castings, which has an important guiding role for its practical application.

1. Introduction

High pressure die casting (HPDC) is a near-net casting process in which molten metal is poured into the injection chamber to fill the metal cavity at high speed and high pressure and solidify quickly [1, 2]. HPDC has been widely used to produce high-precision, mass and low-cost lightweight automotive aluminum and magnesium alloy castings [3–5]. In recent years, the automobile lightweight has become the development trend of automobile industry with the development of industry, which requires die-cast aluminum alloy to have higher strength, so that thinner wall components basing on higher strength alloy can be used to achieve the purpose of reducing structural weight [6, 7]. Therefore, in order to improve the mechanical properties of materials, particle reinforced aluminum matrix composites are paid more and more attention.
The particle reinforced aluminum matrix composites can improve the mechanical properties of aluminum alloy while maintaining its excellent performance [8–12]. Many kinds of particles such as TiB₂, SiC, ZrB₂, Al₂O₃ and TiC can be used as reinforcing particles of aluminum matrix composites [13–18]. Among them, TiB₂ ceramic particles not only have high strength, high modulus, and good wear resistance and grain refinement effect, but also it can be synthesized in situ in aluminum melt and have no reaction with aluminum alloy melt [19–23]. At present, many researchers have reported that TiB₂ particles are used to enhance the mechanical properties of Al alloys. For example, S.-L. Zhang et al [24] investigated the influence of TiB₂ and ZrB₂ on microstructures and mechanical properties of AlSi9Cu3 alloy under gravity casting. It reveals that the grain size decreases and the secondary dendritic arm spacing increases slightly with the addition of TiB₂ and ZrB₂. But the ultimate tensile strength of the composite was in the medium level of 265 MPa. Xiaoli Wen et al [25] demonstrated the microstructures and mechanical properties of TiB₂/2024Al composite fabricated via laser solid forming additive manufacturing. They found that the 2024Al alloy has formed large columnar grains and appears apparent preferential growth orientation, but the yield strength of the composite was not ideal, only 163 MPa. The preparation of aluminum matrix composites by high pressure die casting is a possible solution to obtain high strength as-cast Al alloys. In addition, the current research findings about the particle strengthening of die-cast Al alloys are still rather limited [26].

Therefore, the research focuses on the microstructure and mechanical properties of die-casting 4 wt%TiB₂/Al-Si-Cu-Zn composite under HDPC. The microstructure and phase structure of composite and the morphologies, distribution and sizes of the TiB₂ ceramic particles were investigated in detail through XRD, OM, SEM, EDS and TEM. The mechanical properties of the composite after T6 heat treatment were evaluated by tensile testing. Furthermore, the effects of the TiB₂ particles and Al₂Cu precipitated phase on the mechanical properties were discussed.
2. Experimental

2.1. Preparation of the in situ 4 wt%TiB₂/Al-9Si-3Cu-0.8Zn composites

In this experiment, the 4 wt%TiB₂/Al-9Si-3Cu-0.8Zn composite was fabricated with 11 wt%TiB₂/Al-6Si as master alloy. The 11 wt%TiB₂/Al-6Si master alloy was prepared by mixed salt reaction method that uses K₂TiF₆ and KBF₄ powder, industrial pure Al and Si as the main reactants. Details of the reaction are given in previous research [27]. Then, a certain weight of pure Al (99.5%), pure Zn (99.99%), Al-50 wt%Si and electrolytic copper were added to the melt at 630 °C to ensure that the material reaches the designed composition. After melting, C₂Cl₆ was added into the melt for slag removal treatment, and finally the prepared metal melt was poured into the injection chamber for HPDC. The preparation of 4 wt%TiB₂/Al-9Si-3Cu-0.8Zn composites die castings is shown in figure 1. And the experimental processing parameters of HPDC are listed in table 1. In addition, the materials used in this experiment are all from Beijing Cuibolin Non-ferrous Metal Technology Development Center.

2.2. Heat treatment and mechanical tests

T6 heat treatment of the castings were carried out by solution treatment at 520 °C for 5 h, then quenching immediately in water and artificial ageing treatment at 170 °C for 7 h. The temperature fluctuation during heat treatment was controlled within ±2 °C. The tensile tests of the as-cast HPDC samples were pulled on a AG-X plus100KN tester at room temperature with a tensile speed of 1 mm min⁻¹, and the strain during tensile tests were monitored by extensometer. The samples were machined according to the ASTM E8 standard, and three tensile specimens were tested for each position. Figure 2 shows the dimensions of the samples for tensile testing.

2.3. Materials characterization

The samples were cut from the castings, then mechanically polished and corroded with 0.5%HF solution for 3–5 s in order to observe microstructural morphologies. X-ray diffraction (XRD, D8 Advance, Bruker AXS) using a Cu target was used to analyze and identify the phase component of the samples. Optical microscopy (OM, Eclipse MA200, Nikon) and field emission scanning electron microscopy (FESEM, Nova Nano SEM-450, FEI) with an energy-dispersive spectroscopy (EDS) detector were used to observe the microstructure of the composite and morphologies and sizes of the TiB₂ ceramic particles. Transmission electron microscope (TEM, Tecnai G2 TF30, FEI) was used to observe and identify the Al₂Cu precipitated phase and TiB₂ reinforcing phase. Scanning electron microscope (SEM, EVO18, ZEISS) was used to observe the fracture morphology of the samples after tensile testing.

Table 1. Experimental processing parameters of HPDC.

| Serial No | Pouring temperature (°C) | Mold temperature (°C) | Pressurization pressure (MPa) | Injection velocity (m s⁻¹) |
|----------|---------------------------|------------------------|------------------------------|---------------------------|
| 1        | 630                       | 150 ~ 170              | 130                          | 0.8                       |
| 2        |                           |                        |                              | 1.16                      |
| 3        |                           |                        |                              | 1.8                       |
| 4        |                           |                        |                              | 2.5                       |
3. Results and discussion

3.1. Phase of the die-cast composite

Figure 3 shows the XRD spectra of die-cast 4 wt% TiB$_2$/Al-Si-Cu-Zn composite. The diffraction peaks of TiB$_2$, Si, Al$_2$Cu and α-Al phases are clearly observed, respectively. In addition, there is not any Al$_3$Ti or Al$_2$O$_3$ diffraction peak found. The results indicate that the in situ synthesis of 4 wt% TiB$_2$/Al-Si-Cu-Zn composite from Al-K$_2$TiF$_6$-KBF$_4$ and system in our experimental scheme is described.

The intermetallic compound rich in Al and Cu was Al$_2$Cu phase, and the intermetallic compound containing Ti and B elements was TiB$_2$ reinforced phase, according to the results of XRD analysis and EDS analysis of SEM. However, the chemical formula structure of intermetallic phase may be deviated because the measurement accuracy of the element content under EDS and XRD analysis was not high enough. Therefore, the TEM analysis of Al$_2$Cu phase and TiB$_2$ phase in the composite was introduced to determine the accuracy of the measurement results. Figure 4 shows the TEM micrographs of the die-casting 4 wt% TiB$_2$/Al-Si-Cu-Zn composite. As shown in figure 4(a), the TiB$_2$ particles presents an irregular polygonal plate-like shapes with a length of 200–500 nm. The selected area electron diffraction (SAED) pattern along zone axis [0 -1 1 1] was shown in figure 4(b). And the lattice constants of the phase were calculated as $a = 0.302$ nm, $c = 0.336$ nm from the ($-1101$) and (01–12) crystal plane spacing of 0.207 and 0.142 nm measured in figure 4(b), which represented a difference of about 0.33% and 4.3% (<5%) from the standard lattice constant of the TiB$_2$ phase ($a = 0.303$ nm, $c = 0.336$ nm).
The result shows that SAED pattern of the particle matched well with the lattice structure of TiB$_2$ reinforcing phase. Therefore, the phase is determined as TiB$_2$ phase with the hexagonal close-packed structure.

In order to accurately determine the chemical formula structure of Al-Cu phase, the TEM images of nano-sized precipitated phase in the $\alpha$-Al matrix after T6 heat treatment of the die-casting composite was introduced in figure 5. Figure 5(a) presents the bright-field TEM image of the composite, and the precipitated phase uniformly distributed in the $\alpha$-Al matrix. The scanning transmission electron micrographs (STEM) of the...
precipitates was shown in figure 5(b). And the nano-sized precipitated phases present a long needle shaped with a length of 30–80 nm. Figure 5(c) presents the HRTEM morphology between the precipitated phase and \( \alpha \)-Al matrix. It is obvious that the interface of the composite is well bonded. The fast Fourier transform (FFT) of the region A in figure 5(c) was conducted as shown in figure 5(d). The lattice constants of the Al-Cu phase were \( a = 0.635 \text{ nm}, c = 0.480 \text{ nm} \), and there are only 1.6% and 4.6% (<5%) errors with the standard lattice constants of \( \text{Al}_2\text{Cu} \) phase \( (a = 0.607 \text{ nm}, c = 0.488 \text{ nm}) \), which verified that the nano-sized precipitate was \( \text{Al}_2\text{Cu} \) phase.

3.2. Microstructure of the die-cast composite

Figure 6 presents the castings of the 4 wt%TiB\(_2\)/Al-Si-Cu-Zn composites fabricated under the HPDC. Among them, figures 6(e)–(h) are enlarged views of the filling ends of the castings in figures 6(a)–(d), respectively. Figure 6(a) shows the casting of composite with injection velocity of 0.8 m s\(^{-1}\). The molten metal could not be filled to the end of the cavity, resulting in incomplete shape of the sample obtained, which indicates that the poor mold-filling capacity at this injection rate. Figure 6(b) presents the castings of composite with injection velocity of 1.16 m s\(^{-1}\), and the cavity was well filled and the shape is complete, but there are a lot of cracks in the side walls of the casting. Figure 6(c) shows the casting of composite with injection velocity of 1.8 m s\(^{-1}\), and the molten
metal was well filled to the end of the cavity, and the casting had good integrity and no hot-tearing crack, indicating the excellent die-cast capability at this injection rate. Figure 6(d) presents the casting of composite with injection velocity of 2.5 m s$^{-1}$. Although the molten metal could fully fill the mold, there were a large number of flash and burr defects, crack phenomenon in the castings and it was not easy to demould. Because when the injection speed was too high, the slurry would exert excessive pressure on the parting surface, resulting in stress concentration on the surface of the casting, thus inducing cracks and producing defects such as flaps and burrs. To sum up, when the injection velocity reaches 1.8 m s$^{-1}$, the mold-filling capacity of the composite achieve the best.

The microstructure of different parts of die-casting 4 wt%TiB$_2$/Al-Si-Cu-Zn composite before and after T6 heat treatment is shown in the figure 7. Figures 7(a)–(d) presents the microstructure of the die-casting composite before T6 heat treatment. The composite is mainly composed of primary $\alpha$-Al, eutectic Si, Al$_2$Cu phase and TiB$_2$ reinforced particles. It can be seen from the figure that the morphology primary $\alpha$-Al phase is white rose-like or nearly spherical, and Al$_2$Cu phase is presented in a bright and white bone-shape. The eutectic Si phase are gray and uniformly distributed at the grain boundaries, while TiB$_2$ reinforced particles distributed around eutectic Si in cluster structure. And a few TiB$_2$ particles are embedded on the eutectic Si surface. The distribution of TiB$_2$ particles in the matrix is affected by the following two factors: Firstly, the density difference between the matrix and the TiB$_2$ particles is large, which leads to the TiB$_2$ particles suspending in the melt. Secondly, the aluminum liquid and TiB$_2$ particles cannot be completely wetted, which further hinders the free movement of TiB$_2$ particles and eventually leads to the agglomeration of TiB$_2$ particles. In addition, the $\alpha$-Al grains in the D part of the casting (figure 7(d)) are significantly smaller than those in the A part of the casting (figure 7(a)).

The main reason for the difference of grain size in different positions of castings is the difference of cooling rate. Because the difference of cooling speed will further lead to the change of undercooling degree, the relationship between the degree of undercooling ($\Delta T$) and the critical nucleation radius ($R^*$) is shown as follows [28]:

$$R^* = \frac{2\sigma T_m}{L_m \Delta T}$$

Where $\sigma$ is the interfacial energy, $T_m$ is the solidification temperature, $L_m$ is the latent heat of metal crystallization. These formulas showed that an increase in the degree of undercooling would lead to a decrease in the critical nucleation radius, resulting in a decrease of grain size. The A part of the casting is the closest to the injection chamber, and the influence of the temperature of the injection chamber on the liquid metal cannot cool quickly, so the grain size is large there. Besides, the cooling rate of liquid metal gradually increases with the increase of filling distance, leading to the decrease of grain size. So the grain size of in the D part of the casting is the smallest.
Figures 7(e)–(h) shows the microstructure of the die-casting composite after T6 heat treatment. It is obvious that the phases composition of the composite does not change. However, the morphology of most eutectic Si phase changed from short rod to sphere, and distributed evenly and dispersedly in the \( \alpha \)-Al matrix. Furthermore, a great deal of Cu-containing soluble phase is dissolved after the solution heat treatment at 520 °C, and precipitated nano-sized \( \text{Al}_2\text{Cu} \) phase (figure 5) after artificial aging treatment at 170 °C. The TiB\(_2\) reinforced particles are still agglomerated around eutectic Si phase. In addition, the number of pores of the die-casting composite increased along with the filling distance, and the average diameter of the pores increases from 3.48 μm to 6.54 μm. The significant increase in the number and size of pores in the D part of the casting is mainly due to the fact that the gas cannot be completely smoothly discharged from the cavity and is wrapped by the slurry during the filling process, resulting in gas entrainment. Then the slurry is solidified outside, which further causes the gas to be unable to be discharged. Finally, a large number of pores are formed at the end of the cavity, which is shown in the figures 7(d) and (h).

The SEM micrographs of 4 wt\%TiB\(_2\)/Al-Si-Cu-Zn composite is shown in figure 8. As shown in figure 8(a), most of TiB\(_2\) particles is distributed uniformly and dispersedly in the \( \alpha \)-Al matrix of the master alloy. At the highly magnified SEM images (figure 8(b)), the TiB\(_2\) particles exhibited irregular polyhedral morphology or nearly circular shape. Combining with the XRD analysis of the master alloy in figure 8(a) and the EDS analysis of the reinforced particles in figure 8(c), it can be known that the phase is TiB\(_2\). Figure 8(d) presents the size distribution maps of the TiB\(_2\) particles embedded on the eutectic Si surface. The nanometer-sized TiB\(_2\) particles (<100 nm) accounted for 15.5%, while the submicron-sized TiB\(_2\) particles accounted for only 84.5%. And the average sizes of the TiB\(_2\) particles was 209.7 nm. In general, the size of TiB\(_2\) particles fabricated by \textit{ex situ} synthesis method are in the range of 10 \( \sim \) 30 μm [29], which is much larger than the size of those particle size in this study.

### 3.3. Mechanical properties of the die-casting composite after T6 heat treatment

The mechanical properties and corresponding engineering stress-strain curves of the die-cast 4 wt\%TiB\(_2\)/Al-Si-Cu-Zn composite before and after T6 heat-treated under HPDC are shown in figure 9. The results indicated that the tensile strength, yield strength, and elongation in the composites after T6 heat treatment were higher than those before heat treatment. And the yield strength (YS), ultimate tensile strength (UTS) and elongation (EL) of the die-cast composite after T6 heat treatment under HPDC can reach up to 311 MPa, 379 MPa and 2.8%. It is very difficult to make die-cast aluminum alloys reach a high yield strength of 310 MPa and hold an elongation of 2.5% which can be applied in industry. Compared with the traditional die-
casting of aluminum alloy, the significant improvement of YS and UTS of the composite may be due to the introduction of TiB₂ reinforcing phase. The strengthening mechanisms of TiB₂ reinforcing particles includes the CTE and elastic mismatch strengthening, Orowan strengthening, load-bearing strengthening and grain refinement strengthening[30]. In addition, based on the TEM observation results (figure 5), the precipitation of nano-sized Al₂Cu phase during aging process is the key factor to strengthen the mechanical properties of composite after T6 heat treatment. Because the Al₂Cu precipitates can effectively block the slip of dislocations in the matrix, it can provide excellent precipitation strengthening for the composite. And the precipitation of Al₂Cu as a new phase in the matrix will lead to the formation of grain boundaries between the two phases. The grain boundaries can also hinder the slip of dislocations to a certain extent, thus strengthening composite. In addition, there is also a CET mismatch between Al₂Cu and α-Al matrix, which will lead to CTE mismatch strengthening[31].

As shown in figure 9, the mechanical properties of the 4 wt%TiB₂/Al-Si-Cu-Zn composite castings are different in different parts. This is because the refinement degree of α-Al grains, the size, morphology and distribution of eutectic Si, the number and distribution of Al₂Cu precipitated phase and TiB₂ reinforced phase, and casting defects in the structure will all affect the mechanical properties of castings. In this study, the main reasons for the difference in mechanical properties of castings are the refinement degree of α-Al grains and porosity defects. Theoretically, the mechanical properties of the position with the smallest grain size of α-Al are the best. However, there are a lot of porosity defects existing in this position (figure 7(h)). The stress concentration will occur in the parts with porosity defects, which may promote the crack propagation during tensile deformation and eventually lead to the decrease of strength and elongation of the D part of the casting. Therefore, the C part of the composite casting has the best mechanical properties.

Figure 10 shows the fracture morphology of the die-cast 4 wt%TiB₂/Al-Si-Cu-Zn composite after T6 heat treatment. The degree of plastic deformation is related to the growth degree of dimples, so it can be considered that the number and size of dimples determine the elongation of materials. It can be clearly seen from the figure 10 that the elongation of the composite increases with the increase of the number of dimples. In addition, it can be seen from the figure 10(a) that the TiB₂ particles are seriously agglomerated on the cleavage planes. Because the stress required to break the particles decreases with the increase of particle size[22]. And the agglomerated TiB₂ particles can be regarded as the increase of particle size, so the stress required for composite fracture will be reduced. The cracks are generated and propagated in the cleavage plane area as shown in the figures 10(b) and (d). These cracks will further promote the fracture of materials and have a negative impact on the mechanical properties of the composites.

Figure 10. Fracture morphology of the die-cast 4 wt%TiB₂/Al-Si-Cu-Zn composite after T6 heat treatment: (a) A part; (b) B part; (c) C part; (d) D part.
4. Conclusion

In this study, the 4 wt%TiB$_2$/Al-Si-Cu-Zn composite was successfully prepared under the HPDC. The following conclusions were drawn by studying their microstructures and mechanical properties.

(1) The compound containing Ti and B elements was determined as the hexagonal close-packed structured TiB$_2$ reinforced phase with the lattice constants of $a = 0.302$ nm, $c = 0.336$ nm. In addition, the long needle shaped nano-sized phase precipitated in the $\alpha$-Al matrix during aging process was identified as the Al$_2$Cu phase with the lattice constants of $a = 0.635$ nm, $c = 0.480$ nm.

(2) The injection velocity has an important influence on the formability of castings. When the injection velocity is too low, the casting is prone to insufficient mold filling and crack defects. When the injection velocity is too high, the casting will have defects such as flash and burr. In this study, when the injection speed was $1.8$ m s$^{-1}$, the filling effect of the composite was the best in this work.

(3) The average size of the $\alpha$-Al grains gradually decreased with increasing filling distance, and most of the TiB$_2$ reinforced particles distributed around eutectic Si in cluster structure, whereas a few were embedded on the surface of the eutectic Si. Furthermore, the average sizes of the TiB$_2$ particles embedded on the eutectic Si surface was 209.7 nm.

(4) The die-casting 4 wt%TiB$_2$/Al-Si-Cu-Zn composite could attain the high YS of 311 MPa and UTS of 379 MPa with an EL of 2.8%, after T6 heat treatment. The additional the significant improvement of YS and UTS of the composite should be primary contributed to the introduction of TiB$_2$ reinforcing phase and the precipitation of Al$_2$Cu precipitate in the aging stage.

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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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