

Microstructure, Tensile Properties, and Wear Resistance of In Situ TiB₂/6061 Composites Prepared by High Energy Ball Milling and Stir Casting

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In-situ synthesized TiB₂/6061 composites were prepared from $AI-K_2TiF_6-KBF_4$ by high energy ball milling and stir casting. Phase analysis and microstructure observation of the samples were characterized by XRD, SEM and EDS, respectively. The effect of TiB₂ particle content on the microstructure, tensile properties and wear resistance of the composites was studied. The results show that the average size of TiB2 particles is 1 lm, which is polygonal shape. The average grain size of the composites can be refined significantly as the TiB2 particle mass content increased from 1 to 3%; however, the grain coarsening occurs in the 5 wt.% TiB2/6061composites. The 3 wt.% TiB2/6061 composites have best tensile strength, yield strength and Young's modulus among the composites in ranges of the TiB₂ mass fraction from 1 to 5%. Strengthening mechanisms of the TiB2/6061 composites were fine grain strengthening, Orwan strengthening and CTE strengthening, in which the CTE strengthening plays an important role as increasing the $TiB₂$ content. The pin-on-disk wear test results indicated that the average friction coefficient and wear rate of the TiB2/6061 composites increased firstly and then decreased with increasing the TiB₂ content from 1 to 5 wt.%. The wear mechanism of the TiB2/6061 composites was discussed.

1. Introduction

Compared with the traditional aluminum alloy, particle reinforced aluminum matrix composites (AMCs) have the advantages of high strength, high stiffness, high toughness, high wear resistance and good fatigue resistance (Ref [1-4\)](#page-9-0). Therefore, particle reinforced AMCs have broad application prospects in aerospace, automotive and electronic fields (Ref [5-](#page-9-0) [9\)](#page-9-0).

At present, the preparation methods of particles reinforced AMCs mainly include the additive method and the in-situ method (Ref [10](#page-9-0)[-13\)](#page-10-0). Compared with the additive method, insitu particles reinforced AMCs have received extensive attention from researchers in recent years due to their simple preparation process, good bonding between the reinforcement and the matrix, and the excellent properties of the composites (Ref [14-15](#page-10-0)). 2xxx, 6xxx, and 7xxx series aluminum alloys are

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commonly used as the matrix of AMCs (Ref [2](#page-9-0), [5](#page-9-0), [7](#page-9-0), [10\)](#page-9-0). Among them, 6061 aluminum alloys have generated significant interest as their outstanding properties (Ref [16-18](#page-10-0)). The ceramic particles such as TiB_2 , TiC and Al_2O_3 are usually used as the in-situ formed phase to fabricate the AMCs (Ref $17-21$). TiB₂ particles have high hardness, high melting point, good electrical and thermal conductivity, and are regarded as a very ideal reinforcement for AMCs (Ref [22-26](#page-10-0)).

In previous literatures, $TiB₂$ particles reinforced AMCs were synthesized by the chemical reactions of Al- K_2T iF₆-KBF₄ (Ref [24-26\)](#page-10-0), Al-TiO₂-B₂O₃ (Ref [27](#page-10-0), [28\)](#page-10-0) and Al-Ti-B (Ref [17](#page-10-0), [29\)](#page-10-0). The flux-assisted synthesis, using the Al-K₂TiF₆-KBF₄ reaction system, is originated from a LSM method by P. Davies and co-workers in the 1990s (Ref [30](#page-10-0)). Owing to its low cost and easy control of the reinforcement content, the flux-assisted synthesis is the most popular method to prepare in-situ synthesized $TiB₂$ particles reinforced AMCs. Liu et al. fabricated in-situ $TiB₂/Al$ composites at 900 \degree C and the grain size of the matrix refined significantly by the TiB₂ particles (Ref 24). Mozammil et al. prepared in-situ TiB₂/Al composites at 780 °C by using the flux-assisted synthesis and the size of $TiB₂$ particles was smaller than 2 μ m (Ref [25](#page-10-0)). Chen et al. processed TiB₂/Al composites at 860 °C and the size of in-situ synthesized TiB_2 particles was in the range of $0.1 \sim 2 \mu m$ (Ref [26\)](#page-10-0). Al₃Ti phase was generated during the composites processing in those researches. Tee et al. reported that $Al₃Ti$ phase can decrease the yield of TiB₂/Al composites (Ref [29\)](#page-10-0). In order to obtain the insitu particulate reinforced AMCs with excellent properties, the formation of $Al₃Ti$ phase should be avoided during the processing. The formation of Al₃Ti in Al-TiO₂-B₂O₃ reaction system can be effectively inhibited under the starting materials ball milling condition (Ref [27](#page-10-0)).

In the present paper, in-situ synthesized $TiB₂/6061$ composites were prepared from $AI-K_2TiF_6-KBF_4$ by high energy ball milling combined with stir casting technology. The phase analysis and microstructure of the composites were investigated. The effect of TiB₂ particle content on the microstructure, tensile properties and wear resistance of the composites was studied. Strengthening mechanism and wear mechanism were discussed.

2. Experimental

2.1 Materials

6061 aluminum alloy was selected as the matrix of the composites, and its nominal chemical composition is 98 wt.% Al, 0.4-0.8 wt.% Si, 0.15-0.4 wt.% Cu, 0.15 wt.% Mn, 0.8~1.2 wt.% Mg, 0.25 wt.% Zn, 0.15 wt.% Ti and 0.7 wt.% Fe. Commercial available Al powder, K_2TiF_6 powder and KBF4 powder, both of which have a purity of 98%, are used as the starting materials for preparing the reinforcement.

2.2 Preparation of the Composites

Al powder with an average particle size 20 μ m, K₂TiF₆ with an average particle size 150 μ m and KBF₄ powder with an average particle size 60 µm were weighed separately according to the mole ratio of Ti: $B = 1:2$ and the mass ratio of Al: $(Ti + B)=1:1$. The mixed powder was mixed by a planetary ball mill machine with $ZrO₂$ ball-to-powder weight ratio of 10:1 and milled at 300 rpm/min for 10 h. The diameter of $ZrO₂$ balls is 8 mm. The SEM comparison of the powders before and after ball milling is shown in Fig. 1. For each experiment, 1 kg 6061 aluminum alloy was melted and heated to 845 $^{\circ}$ C in a graphite crucible under argon atmosphere, using a resistance furnace. The schematic illustration of set-up for preparing in-situ TiB_2 / 6061 composites is shown in Fig. [2](#page-2-0). The graphite crucible shown in Fig. [2](#page-2-0) has a capacity of melting 2 kg aluminum alloy. A number of aluminum foil wrapped preforms with a size of 20×20 mm were pressed into melt 6061 aluminum alloy according to the in-situ synthesized nominal mass fraction of 1 , 3, 5 wt.% $TiB₂$ in the composites. The mechanical stirring was applied to the melt at the speed of 150 rpm/min for 30 min. Then, the molten material was cooled to 720 °C for refining, slag stripping and degassing. As the molten material temperature decreased to $700 \degree C$, the molten material was poured into the pre-heated (300 °C) graphite mold (70 \times 130 \times 30 mm), and in-situ synthesized $TiB₂/6061$ composites can be obtained; the process flow is shown in Fig. [3](#page-2-0).

2.3 Experimental Method

The differential scanning calorimetry (DSC) analysis of the mixed powder was carried by a CRY-2P high temperature differential thermal analyzer under nitrogen atmosphere from 20 to 1150 \degree C at a heating rate of 20 \degree C/min. Phase identification of the samples was conducted using the Shimadzu XRD-6100 X-ray diffractometer (XRD) using Cu Ka radiation, and the sample tank was made by $SiO₂$. The composites samples were polished and etched with Keller's reagent, then observed by the JSM-7500F field emission scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS). The average grain size of the samples was determined in

Fig. 1 SEM comparison of the powders before and after ball milling. (a) Al (b) $KBF₄$ (c) $K₂TiF₆$ (d) ball milled mixing powder

accordance with ASTM E112-2013 Standard using the Quantimet Image analysis method.

The reinforcement extraction experiments of the composites are as follows. The composites were placed on the anode, the copper plate was placed on the cathode, and the electrolyte was 3.3 mol/L KCl aqueous solutions. MD-30 pulse power with constant current mode was used to conduct the reinforcement extraction experiments. The anode slime of electrolysis experiment was cleaned and dried with dilute HCl solution and absolute ethanol to obtain the extractions.

T6 heat treatment, $530 °C$ for 6 h then water cooled, followed by $170 °C$ for 10 h then air cooled to room temperature, was carried to treat 6061 matrix and composites (Ref [31](#page-10-0)). HBS-3000 Brinell hardness tester was used to test the hardness of the samples, and the hardness value of the samples was the average value of five measurements. The tensile test was performed by an electronic universal tensile tester (CMT-5105, SANS) at room temperature with a crosshead velocity of 2 mm/min. The plate tensile specimens have a gage length of 21 mm and a thickness of 2 mm. MMU-10G wear test machine was used to test the wear properties of the composites under oillubricated conditions. GCr15 (45-50 HRC) was used as disk with a roughness $Ra = 0.8$. The disk having diameter of 43 mm and 7 mm thickness was used in the present paper. The pin samples were prepared with the dimensions of 4 mm \times 12 mm (diameter \times length). The pin-disk wear tests were carried out with sliding speed of 200 rpm/min and 400 N constant loads

Fig. 2 Schematic illustration of the set-up for preparing in-situ TiB2/6061 composites

Fig. 3 Preparation process diagram of in-situ synthesized $TiB₂/$ 6061 composites Fig. 4 The standard Gibbs free-energy of the Al-Ti-B system

for 30 min. SEA-40 lubricating oil was added 5 drops in every 10 min. The samples were cleaned in KQ2200DE ultrasonic cleaning machine with acetone solution before and after the experiment. The initial and final weight of the samples was measured with a precision of 0.0001 g in an electronic balance weighing machine. The wear rate of the samples is determined using the following expression:

$$
A = \frac{W_1 - W_2}{S} \tag{Eq 1}
$$

where W_1 is the weight of the sample before wear in mg, W_2 is the weight of the sample after wear in mg and S is the area of the wear surface of the sample in mm².

3. Results and Discussions

3.1 Phase identification

The reaction taken place in the Al-KBF₄-K₂TiF₆ system is as follow (Ref [32,](#page-10-0) [33\)](#page-10-0):

$$
K_2TiF_6+KBF_4+A\mathbf{l} \rightarrow TiB_2+KAlF_4+KF+AlF~~(Eq~2)
$$

The main components in the reaction system are Al, Ti and B. Al is equivalent to the solvent; Ti and B are equivalent to the solutes. Therefore, the Eq 2 thermodynamic system can be regarded as Al-Ti-B system. The Eq 2 reaction can be described as follows:

$$
3\text{Al} + [\text{Ti}] \rightarrow \text{TiAl}_3 \tag{Eq 3}
$$

$$
Al + 2[B] \rightarrow AlB_2 \tag{Eq 4}
$$

$$
[\text{Ti}] + [\text{B}] \to \text{TiB} \tag{Eq 5}
$$

$$
[\text{Ti}] + 2[\text{B}] \rightarrow \text{TiB}_2 \tag{Eq 6}
$$

The standard Gibbs free-energy of the Al-Ti-B reaction system is calculated, as shown in Fig. 4. The Gibbs free-energy of Eq 6 is the lowest. As the reactions can be fully taken place, $TiB₂$ is the only stable phase, and the other products are all transformed to $TiB₂$.

Fig. 5 DSC curve of mixed powder after ball milling

Fig. 6 X-ray diffraction patterns of in-situ synthesized $TiB_2/6061$ composites

Figure 5 is the DSC curve of mixed powder after ball milling. The exothermic of Al-Ti-B system start at 678 °C, peak at 798 \degree C and finish at 845 \degree C. That is to say the reactions can be taken place completely at 845 °C. The exothermic peak temperature and finish temperature are lower than the literatures, which reported the same reaction system (Ref [24,](#page-10-0) [26\)](#page-10-0). This is because the ball milling treatment to the starting materials can significantly reduce the reaction temperature of the reaction system (Ref [27](#page-10-0), [34](#page-10-0)).

Figure 6 is the x-ray diffraction patterns of in-situ synthesized $TiB₂/6061$ composites. The phase composition of the composites mainly consists of Al and TiB₂, while TiAl₃ and AlB_2 are not found. This agrees well with the calculation results of standard Gibbs free-energy of the reaction system, as shown in Fig. [4](#page-2-0).

Figure 7 shows the x-ray diffraction patterns of the extracts taken from $TiB₂/6061$ composites. The diffraction peaks of TiB₂, Al₂O₃ and SiO₂ appear in the diffraction patterns. Al₂O₃ is the product of Al during extraction experiments, and the $SiO₂$ is the sample tank peak. Al and Mg_2Si , which dissolved in the

Fig. 7 X-ray diffraction patterns of the extracts taken from $TiB_2/$ 6061 composites

dilute HCl solution, are not found. Ti B_2 is the only stable phase in the composites.

Figure [8](#page-4-0) shows the SEM and EDS micrographs of the extracted TiB₂ taken from TiB₂/6061 composites. A large number of polygonal particles with the size distribution of $0.5 \sim 1.5$ µm can be seen from Fig. [8](#page-4-0)a. Figure $8(b) \sim (f)$ $8(b) \sim (f)$ are the results of the EDS analysis of the extracts. Ti and B elements are concentrated in the same position. Combined with Fig. [8](#page-4-0)(a), polygon particles are the main enrichment regions of Ti and B elements, and it can infer that polygonal particles in Fig. [8\(](#page-4-0)a) are $TiB₂$ particles.

3.2 Microstructure Observation

Figure [9](#page-5-0) shows SEM micrographs comparison of the matrix and $TiB₂/6061$ composites. It can be seen from Fig. $9(a)$ $9(a)$ that the microstructure of 6061 aluminum matrix is composed of α (Al) + β (Mg₂Si) two-phase eutectic, and the second phase particle Mg2Si is distributed at the grain boundary, and a small amount is distributed in the grain. The grain size of in-situ synthesized 1 wt.% TiB₂/6061 composites falls to 119.6 μ m, reduced from the 154.7 µm in average size in 6061 aluminum matrix, as shown in Fig. $9(a)$ $9(a)$ and (b). As the TiB₂ content increases to 3 wt.%, the grains are further refined with an average size of $74.8 \mu m$, as shown in Fig. $9(c)$ $9(c)$. However, compared with the 3 wt.% $TiB₂/6061$ composites, the grain size of the composites increases to 89.7 μ m when the TiB₂ particle content increases to 5 wt.%, as shown in Fig. $9(d)$ $9(d)$. This may due to the aggravated aggregation of reinforcing particles, which can reduce the grain refinement of the composites (Ref [35](#page-10-0)).

Figure [10](#page-6-0) shows the SEM and EDS analysis of reinforcing particles in TiB₂/6061 composites. From Fig. $10(a) \sim (c)$ $10(a) \sim (c)$, polygonal particles with the size distribution of $0.5 \sim 1.5$ µm and the average size of $1 \mu m$ are dispersed in the matrix. The TiB₂ particles are distributed uniformly in the 1 wt.% TiB₂/ 6061 composites, as shown in Fig. $10(a)$ $10(a)$. The TiB₂ particles agglomeration can be found in the 3 wt.% $TiB_2/6061$ composites, as shown in Fig. [10\(](#page-6-0)b). As the particle content achieves 5 wt.%, serious particles agglomeration can be found in the composites, as shown in Fig. $10(c)$ $10(c)$. Figure $10(d)$ $10(d)$ is the point scan energy spectrum analysis results of the particles in

Fig. 8 SEM and EDS micrographs of extracted TIB_2 taken from $TIB_2/6061$ composites

Fig. $10(a)$ $10(a)$ \sim (c), and it can be seen that the multilateral particles only contain Al, Ti and B elements, so the polygonal particles in Fig. 11 are TiB₂ particles.

3.3 Hardness and Tensile Properties

The effect of TiB₂ particle content on the hardness of TiB₂/ 6061 composites is shown in Fig. [11.](#page-6-0) The hardness of 6061 aluminum matrix is 101.2 HBS after T6 heat treatment. With the increasing of $TiB₂$ particle content, the hardness of the composites increases first and then decreases. As the $TiB₂$ content is 3 wt.%, the hardness of the composites achieves the highest value (125.9 HBS), which is 24.4% higher than that of 6061 aluminum matrix. The hardness of the matrix and composites is better than those of the same materials reported in literature (Ref [17](#page-10-0)).

Figure 12 shows the effect of TiB₂ content on the tensile properties of TiB₂/6061 composites. The tensile properties of TiB2/6061 composites are much higher than the matrix. With the increasing of $TiB₂$ content, the tensile strength, yield

strength and Young's modulus of the composites first increase and then decrease. As the $TiB₂$ content achieves 3 wt.%, the tensile strength, yield strength and Young's modulus of the composites are the highest (308.9 MPa, 268.1 MPa and 84 GPa, respectively), which increase 16.2%, 15.9% and 23.5% compared with 6061 aluminum matrix, and their properties are better than those of the same materials reported in literature (Ref [18](#page-10-0)). The elongation of the composites decreased with the increasing of $TiB₂$ particle content; however, the decrement was not significant.

Figure [13](#page-8-0) shows the SEM micrographs of fracture surface of the matrix and $TiB₂/6061$ composites. A number of deep equiaxed dimples were distributed in the fracture surface of 6061 aluminum matrix, as shown in Fig. $13(a)$ $13(a)$, exhibiting a typical ductile failure. It can been seen in Fig. $13(b) \sim (d)$ $13(b) \sim (d)$, with the increasing of $TiB₂$ content, the dimples with reduced size were observed and the depth of the dimples decreased. This indicates that the ductility of $TiB_2/6061$ composites is lower than the matrix, which agrees well with elongation results in

Fig. 9 SEM micrographs comparison of the matrix and TiB₂/6061 composites. (a) 6061, (b) 1 wt.% TiB₂/6061, (c) 3 wt.% TiB₂/6061 and (d) 5 wt.% $TiB_2/6061$

Fig. $13(a)$ $13(a)$. Also, TiB₂ particles seriously agglomerate observed in the 5 wt.% TiB $_2$ /6061 composites, as shown in Fig. [13\(](#page-8-0)d). Under external load, the stress concentration occurs in the particles agglomeration regions and the cracks will initiate and propagate in agglomeration regions, resulting in the plasticity decreased, as shown in Fig. [12\(](#page-7-0)a).

In general, the yield strength enhancement of particulate reinforced metal matrix composites can be estimated by two methods (Ref [36](#page-10-0)): load transferring and micromechanics strengthening mechanism. In the present paper, the $TiB₂$ volume fraction of the 1 wt.% TiB₂/6061, 3 wt.% TiB₂/6061 and 5 wt.% TiB2/6061 are 0.006%, 0.018% and 0.031%, respectively. Because of the reinforcing particle volume fraction is quite lower than 5%, load transferring strengthening contribution to the yield strength enhancement is very small (Ref [37\)](#page-10-0). The micromechanics strengthening mechanism includes grain refinement strengthening, Orwan strengthening and CTE strengthening. The quantitative contributions to the yield strength of the micromechanics mechanisms are discussed as follow.

(1) Grain refinement strengthening. Dislocation slip can be blocked by the grain boundaries to strengthen the matrix (Ref 36). The TiB₂ particles refine the grains of aluminum matrix, which providing more grain boundaries to hinder the dislocation motion. The contribution of grain refinement to the yield strength of the composites can be determined (Ref [37\)](#page-10-0):

$$
\Delta \sigma_{\rm GR} = \beta D^{-1/2} \tag{Eq 7}
$$

where β depends on a number of factors, and its typical value is 0.1 MPa $m^{1/2}$, *D* is the average grain size of the composites.

(2) Orwan strengthening. The Young's modulus of the $TiB₂$ (530 GPa) is high enough to make the dislocation to loop around the $TiB₂$, which can make the plastic deformation of the composites more difficult. The yield strength contribution of Orwan strengthening can be calculated by the following expression (Ref [38\)](#page-10-0):

$$
\Delta \sigma_{\rm OR} = \frac{2 \text{Gb}}{d \left(\frac{\pi}{6V_p}\right)^{1/3}}\tag{Eq 8}
$$

where G (26 GPa) and b (0.286 nm) are the shear modulus and Burgers vector of the matrix, d and V_p are the average diameter and volume fraction of the $TiB₂$ particles.

(3) CTE strengthening. The dislocation density can be increased owing to the difference of the coefficient of thermal expansion (CTE) between $TiB₂$ particles and the aluminum matrix. The yield strength enhancement by CTE strengthening can be calculated by the following expression (Ref [39\)](#page-10-0):

$$
\Delta \sigma_{\text{CTE}} = Gb \sqrt{\frac{12 \Delta \alpha \Delta T V_p}{bd (1 - V_p)}}
$$
(Eq 9)

Fig. 10 SEM and EDS analysis of reinforcing particles in TiB₂/6061 composites. (a) 1 wt.%, (b) 3 wt.%, (c) 5 wt.%, (d) point energy spectrum analysis in (a), (b) and (c)

Fig. 11 The effect of TiB₂ content on the hardness of TiB₂/6061 composites

where $\Delta \alpha$ is the difference of the coefficient of thermal expansion between the matrix $(23.6 \times 10^{-6} \text{ K}^{-1})$ and TiB₂ $(8.3\times10^{-6} \text{ K}^{-1})$, ΔT is the difference between the processing temperature and test temperature. In this work, the molten temperature (700 $^{\circ}$ C) was taken as the processing temperature, and the temperature of the pre-heated mold $(300 °C)$ was taken as the test temperature.

The total increment of yield strength due to the above three micromechanics mechanisms can be summarized as follows (Ref [37](#page-10-0)):

$$
\Delta \sigma_{\text{Total}} = \Delta \sigma_{\text{GR}} + \sqrt[2]{\Delta \sigma_{\text{OR}}^2 + \Delta \sigma_{\text{CTE}}^2}
$$
 (Eq 10)

The calculated yield strength enhancement from different strengthening mechanisms is given in Table [1.](#page-8-0) For the 1 wt.% TiB2/6061, the calculated yield strength enhancement of the composites is higher than the experiment values, which is possibly caused by low $TiB₂$ particles content. The calculated yield strength enhancement value for 3 wt.% $TiB_2/6061$ is 32.8 MPa, which is very close to the experiment value of 35.7 MPa. For the 5 wt.% TiB₂/6061, the calculated yield strength enhancement of the composites is much higher than the experiment values, which is probably caused by $TiB₂$ particles agglomerations and voids in the composites, as shown in Fig 10. Compared with the three mechanisms, it is evident that the CTE strengthening is the most effective one, followed by grain refinement strengthening and Orwan strengthening. Moreover, the CTE strengthening plays an important role as increasing the $TiB₂$ content.

3.4 Wear Resistance

Effect of $TiB₂$ content on the average friction coefficient and wear rate of $TiB₂/6061$ composites is shown in Fig. [14](#page-8-0). The average friction coefficient and wear rate of $TiB₂/6061$ composites are lower than the matrix alloy. The formation of $TiB₂$ particles can improve the wear resistance of matrix. The

Fig. 12 Effect of TiB₂ content on the tensile properties of TiB₂/6061 composites. (a) Typical stress–strain curves, (b) average tensile strength and elongation, (c) average Young's modulus and yield strength

average friction coefficient and wear rate of $TiB_2/6061$ composites decrease first and then increase with the increasing of $TiB₂$ particle content. As the particle content achieves 3 wt.%, the average friction coefficient and wear rate of the composites are the lowest, which are 0.052 and 0.071 mg mm-², respectively, which decrease 22.4% and 26% compared with 6061 aluminum matrix.

Figure [15](#page-9-0) shows the SEM and EDS analysis of worn surface of the matrix and $TiB_2/6061$ composites. A lot of grooves on the worn surface of 6061 and $TiB₂/6061$ composites can be seen from Fig. $15(a)$ $15(a)$ to (d), which is affected by the abrasive wear mechanism. Compared with Fig. $15(a)$ $15(a)$, the grooves on the worn surface of 1 wt.% $TiB_2/6061$ composites become thinner and shallower, as shown in Fig. [15](#page-9-0)(b). It is obvious that the grooves on the worn surface of 3 wt.% $TiB_2/6061$ composites are quite thin and shallow, which are difficultly observed, as shown in Fig. $15(c)$ $15(c)$. However, the grooves appeared again on worn surface of 5 wt.% TiB₂/6061 composites. The EDS results show that the grooves contain $TiB₂$ particles and oxygen is not found, indicating oxidation of the friction surface does not occur.

In the present paper, the wear mechanism of in-situ $TiB₂/$ 6061 composites is abrasive wear. Base on the Archard wear theory, the abrasive wear can be described in Eq 12 (Ref [40](#page-10-0)).

$$
v = \frac{k_{\text{abr}}Wx}{H}
$$
 (Eq 11)

where v is the wear volume loss, W is the applied load, x is the sliding distance, H is the hardness of the worn surface, k_{abr} is a constant. The Eq 12 indicates that the higher hardness of the materials, the smaller wear loss. The 3 wt.% $TiB_2/6061$ composites have the highest hardness, which result in the best wear resistance as shown in Fig. [14](#page-8-0). The lower wear resistance of 5 wt.% $TiB₂/6061$ composites attributes to the lower hardness compared with $3 \text{ wt.} \%$ TiB₂/6061 composites. On the other hand, serious $TiB₂$ particles agglomeration can be found in the 5 wt.% $TiB_2/6061$ composites, as shown in Fig. [10](#page-6-0)(c) can readily fall off because of higher brittleness during the wear process. The exfoliated hard particles would accelerated wear process, which also leads to the wear rate of the 5 wt.% TiB $_2$ /6061 composites higher than that of 3 wt.% $TiB₂/6061$ composites. The lowest wear resistance of 1 wt.% $TiB₂/6061$ composites is because of the biggest grain size and the lowest hardness among these composites.

4. Conclusions

(1) In-situ synthesized TiB₂/6061 composites were prepared successfully from $AI-K_2TiF_6-KBF_4$ by high energy ball milling and stir casting. The average size of in-situ synthesized $TiB₂$ particles is 1 μ m, which is polygonal shape. As the content of $TiB₂$ particles in the composites increases from 1 to 3 wt.%, the effect of grain refinement is remarkable. As the $TiB₂$ particles in the composites increase to 5 wt.%, the serious particles agglomerate happens, and reduces the grain refinement of the composites.

Fig. 13 SEM micrographs of fracture surface of the matrix and TiB₂/6061 composites. (a) 6061, (b) 1 wt.% TiB₂/6061, (c) 3 wt.% TiB₂/6061 and (d) 5 wt.% TiB2/6061

Table 1 Calculated yield strength enhancement from different strengthening mechanisms (unit, MPa)

Materials	$\Delta \sigma_{\rm GR}$	$\Delta \sigma_{\rm OR}$	$\Delta \sigma_{\rm CTE}$	$\Delta \sigma_{\rm Total}$	$\Delta \sigma_{\rm Experiment}$
1 wt.% $TiB_2/6061$	9.7	3.4	12	22.2	15.8
3 wt.% TiB ₂ /6061	11.5	4.7	20.8	32.8	35.7
5 wt.% TiB ₂ /6061	10.5	5.8	27.4	45.3	19.9

Fig. 14 Effect of TiB₂ content on the average friction coefficient and wear rate of $TiB₂/6061$ composites

- (2) With the increasing of TiB₂ particles content from 1 to 5 wt.%, the hardness of the composites first increases and then decreases. As the content of $TiB₂$ particles is 3 wt.%, the hardness of the composites can achieve the highest value of 125.9 HBS, which is 24.4% higher than that of 6061 aluminum matrix.
- (3) Tensile strength, yield strength and Young's modulus of the composites first increase and then decrease among the composites in ranges of the $TiB₂$ mass fraction from 1 to 5%. The elongation of the composites decreases with the increasing of $TiB₂$ particle content, but the decreasing is not obvious. Strengthening mechanisms of the $TiB₂/6061$ composites were fine grain strengthening, Orwan strengthening and CTE strengthening, in which the CTE strengthening plays an important role as increasing the $TiB₂$ content.
- (4) Compared with the 1 wt.% and 5 wt.% TiB₂/6061 composites, 3 wt.% $TiB_2/6061$ composites have the highest hardness and best wear resistance. The average friction coefficient and wear rate are 0.052 and 0.071 mg mm⁻²,

Fig. 15 SEM and EDS analysis of worn surface of the matrix and $TIB_2/6061$ composites. (a) 6061, (b) 1 wt.% $TIB_2/6061$, (c) 3 wt.% $TIB_2/6061$ 6061 and (d) 5 wt.% $TiB_2/6061$

respectively, which are 22.4% and 26% lower than 6061 aluminum matrix. The wear mechanism of $TiB₂/6061$ composites is abrasive wear.

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