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Identification of novel dual-scale Al_3BC particles in Al based composites

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ABSTRACT

In this work, the Al_3BC/Al (Al–3Cu) composites have been firstly in-situ fabricated by liquid–solid reaction method. The microstructures of the samples were investigated by X-ray diffraction (XRD), scanning electron microscope (SEM) and transmission electron microscope (TEM). Two typical sized Al_3BC particles of 100–300 nm and 5–20 nm have been in-situ fabricated, and the particles show the uniform distribution. Mechanical property tests reveal the remarkable coordinated enhancement effects of dual-scale Al_3BC particles. The hardness, tensile strength and abrasion resistance of 26% Al_3BC/Al–3Cu composite are improved by 106.3%, 143.5% and 27.3% respectively, compared with the unreinforced alloy. The synthesis mechanisms of Al_3BC are also discussed and two different mechanisms are proposed to explain the formation of the dual-scale particles.

1. Introduction

Aluminum boron–carbide phases (such as Al_3BC_3, $Al_8B_4C_7$, $AlB_{24}C_4$, etc.) in the Al–B–C system have become the topics of several current investigations since composites reinforced by them possess excellent properties such as high strength, high hardness and low density [1–3]. Besides the above compounds, Al_3BC is another promising phase, which was firstly described as phase X by Halverson et al. in B_4C/Al composites [4]. It is the main product of the interface reaction between B_4C and Al in B_4C/Al composites and has great influence on the mechanical properties of the composites [5–11]. By using X-ray investigations on the powder samples, Viala et al. [12] reported the crystal lattice of Al_3BC which has a hexagonal unit cell with $a = 3.491(2)$ Å and $c = 11.541(4)$ Å. Later, Meyer and Hillebrecht [13] synthesized the pure Al_3BC and defined its crystal structure as a closest packing of Al atoms (sequence ABACBC) with alternating layers of edge-sharing BAl_6 octahedra and trigonal bipyramids CAl_5, linked by common corners. After that, Solozhenko et al. [14] reported the thermal stability of Al_3BC and indicated that even under a high pressure of 1.6–4.8 GPa, the phase remains stable up to 1700 K, implying its high stability. The calculated bulk modulus (152 GPa), shear modulus (140 GPa) and the Young's modulus (326 GPa) of Al_3BC were given in Ref. [15].

However, the current applications of Al_3BC are rarely reported due to the fabrication difficulties of Al_3BC. Ma et al. [16] suggested that Al_3BC was an effective refiner of magnesium alloys. The Al–1B–0.6C master alloy which contains micron-scale Al_3BC particles with size of 2–8 μm can effectively reduce the grain size of AZ63 alloy from 710 μm to 70 μm. In addition, Al_3BC is used as the raw material to manufacture Al–5Ti–0.8B–0.2C master alloy which acts as an effective refiner for aluminum alloys and the transformation process from Al_3BC phase to doped TiB_2 or TiC particles in Al–Ti melts was reported in reference [17]. In our work, a new application of Al_3BC as reinforcement for aluminum composites has been proposed. With low density, high hardness, excellent thermal stability and remarkable stiffness, Al_3BC is regarded as a competitive candidate as the strengthening phase for aluminum composites.

Even if many reinforcements such as SiC, Al_2O_3, TiB_2, and TiC have been widely investigated at present [18–21], there remain unsolved problems such as the segregation phenomenon resulting from the distinction between reinforcements and the matrix alloys and the aggregation tendency of the reinforcement like TiB_2. Compared with them, Al_3BC has its own advantages. On one hand, it has much lower density (2.83 g/cm^3) than those of Al_2O_3 (3.98 g/cm^3) and TiB_2 (4.50 g/cm^3) etc., which is more likely to achieve the demand of lightweight. On the other hand, the density of Al_3BC is very close to that of the Al matrix (2.70 g/cm^3); as a result, the sedimentation behavior in the Al melt during the process is relatively weak, which is beneficial to getting a homogenous distribution of the reinforcement. In addition, Al_3BC has high elastic modulus and excellent thermal stability as well as fine size, which also makes it appropriate to act as the strengthening phase in aluminum alloys.

The mass fabrication and morphology control of Al_3BC particles have become very essential for real applications, however, only few papers are documented. Takeshi et al. [22] fabricated Al_3BC using aluminum powders, amorphous boron powders and graphite powders as raw materials through the self-propagating high-temperature synthesis (SHS) method. According to Kubota's work [23], Al_3BC can also be synthesized by solid-state reactions occurring during heat treatments after mechanical milling (MM) of pure aluminum with 15 or 50 at.% MgB_2 powder

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mixtures in the presence of the process control agent (PCA). However, all these papers reported few on the microstructure and morphology control of Al_3BC particles.

In this work, the novel Al_3BC reinforced Al or Al–Cu based composites have been in-situ synthesized through a liquid–solid reaction method for the first time and the Al_3BC particles fabricated in this way possess dual-scale sizes and uniform distribution in the matrix. The microstructures, mechanical properties and synthesis mechanisms have also been investigated in this work.

2. Experimental details

The raw materials used in this work contained commercial Al (99.7%, all compositions quoted in this work are in wt.% unless otherwise stated), graphite powders (99.0%, 5–10 μm) as well as boron plasmid. The Al_3BC/Al (or Al–3Cu) composites were in-situ fabricated through a liquid–solid reaction method at 750 °C and then were extruded at 500 °C to decrease the porosity.

X-ray diffraction technology was used to identify the phases contained in the composites and the microstructure of the alloys were characterized utilizing field emission scanning electron microscope (FESEM, model SU-70, Japan), equipped with an energy dispersive X-ray spectroscopy (EDS) detector. Transmission electron microscope (TEM, JEM-2100) was also used to identify the morphology and structure of the nanoscale Al_3BC particles.

To verify the strengthening effects of Al_3BC on the matrix, the hardness, tensile properties as well as the abrasion tests on Al–3Cu matrix composites with Al_3BC concentration of 13% and 26% (all the Al_3BC concentration quoted in this work are nominal unless otherwise stated) after T6 heat treatment (solution treatment at 515 °C for 15 h and aging at 165 °C for 15 h) were carried out.

The hardness test bars were machined to the cube type specimens (30 mm in length, 20 mm in width and 20 mm in height) and then tested on a HBS-3000 digital Brinell hardness tester according to the ASTM E10–14 standard. The diameter of the indenter is 5 mm, and the force is 2452 N (250 kgf) with dwell time of 60 s. Each value was an average of at least four separate measurements taken at random places of the specimens. The tensile test was conducted on 'dog-bone' type specimens shown in Fig. 1a using a CMT700 universal material test machine at ambient temperature according to the ASTM E08 standard. In each case, four specimens were tested and the average values were reported. The wear-resistance test was performed on a MM200 abrasion tester according to ASTM G77 standard, and the schematic diagrams of abrasion test are shown in Fig. 1b. The wear specimens with 10 mm × 10 mm in cross-section and 45 mm in length were used with load of 120 N and rotating speed of 400 r/min. The mass loss of the specimens was measured every 10 min to calculate the mass loss–time curves. The wear rate defined as the abrasion loss per unit time was calculated to quantitatively verify the strengthening effects of Al_3BC on the wear-resistance. Friction coefficients under a series of load pressure were also measured in this work.

3. Experimental results

3.1. In-situ synthesised Al_3BC in the Al matrix

To investigate the in-situ synthesis behaviors and the microstructures of Al_3BC particles in the aluminum melt, Al_3BC/Al composites which contain about 26% Al_3BC particles were fabricated and the XRD pattern and microstructures of the samples are shown in Fig. 2. Based on the XRD pattern in Fig. 2a, besides the Al matrix, only the Al_3BC phase was detected in the sample. Fig. 2b presents the typical microstructure of the sample and the small-sized white phases distributing uniformly in the matrix are detected to be Al_3BC particles with the reference of the XRD and EDS results. High magnification images in Fig. 2c–d reveal that Al_3BC particles with tetradecahedron morphology have size ranging from 100 to 300 nm.

Since the Al_3BC particles are too small to be characterized in detail by SEM, the TEM analysis has been carried out to identify their morphology and structure, as shown in Fig. 3. Besides the Al_3BC with size ranging from 100 nm to 300 nm as mentioned in Fig. 2, more spherical particles with size ranging from 5 nm to 20 nm appear in the Al matrix as shown in Fig. 3a, which are also identified to be Al_3BC by HRTEM analysis in Fig. 3b and the result of FFT process in Fig. 3c. The interplanar spacing of the particle in Fig. 3b is calculated to be 1.76 Å, which approximately equals to that of the {110} plane of Al_3BC. Fig. 3d displays the fast Fourier

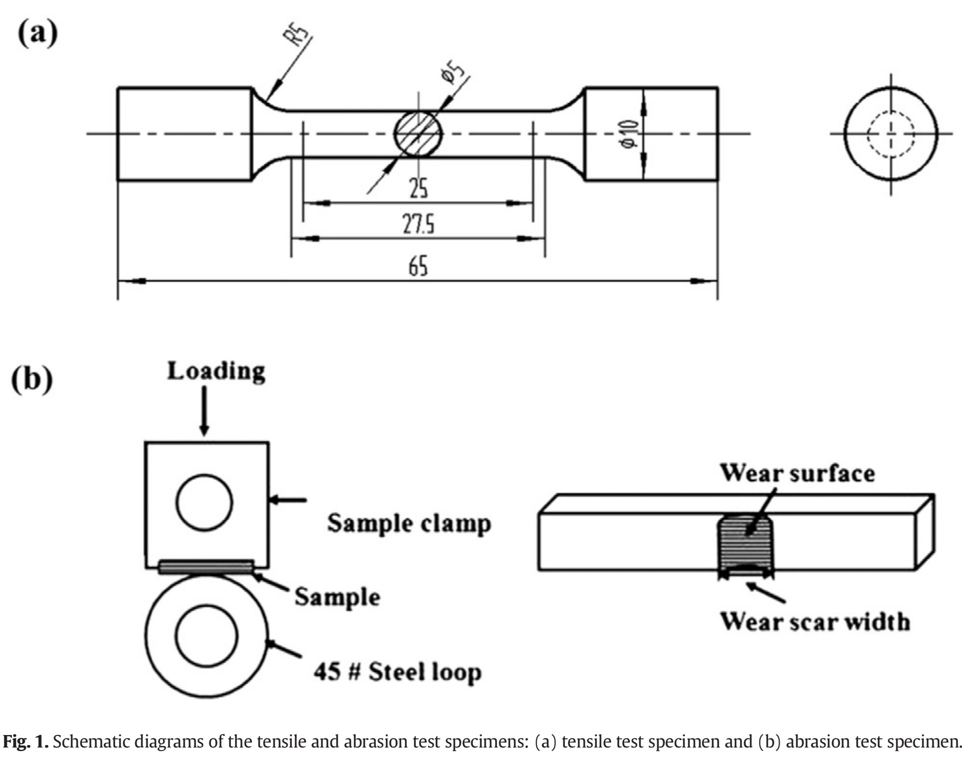

Fig. 1. Schematic diagrams of the tensile and abrasion test specimens: (a) tensile test specimen and (b) abrasion test specimen.