



Microstructural evolution of reactive-sintered aluminum matrix composites

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ABSTRACT

Aluminum matrix composites reinforced with TiB₂ and Al₂O₃ particulates were synthesized via reactive sintering of Al–B–TiO₂ three-component powder mixtures. The effects of powder particle size and reactive sintering temperature on formation of reinforcing phases and microstructural evolution have been investigated. The reduction reaction of TiO₂ by molten Al has been proved to be a stepwise process. Four oxygen deficient titanium oxides can be formed and Ti₂O₃ was a main intermediate phase. In addition to the oxygen deficient titanium oxides, there also existed several transitional phases, such as AlB₂, γ-Al₂O₃, and Al₃Ti. As the reinforcements in the composites, Al₂O₃ is formed by a series of aluminothermic reactions, while the formation of TiB₂ depends on AlB₂ transitional phase. AlB₂ was gradually transformed to a mixed diboride (Al,Ti)B₂, and subsequently to TiB₂, through displacement of Al in the metallic sublattice of AlB₂ by Ti atoms. Small-sized TiO₂ powder promoted the formation of the reinforcing phases at lower temperatures, and the resultant reinforcing particulates exhibited smaller sizes.

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1. Introduction

Aluminum-based metal-matrix composites (MMCs) reinforced with rigid ceramic particulates have become increasingly important for structural applications in aerospace, automotive and other transport industries, because of their high specific strength and modulus, good wear resistance as well as ease of processing. These MMCs have been traditionally fabricated by powder metallurgy, preform infiltration, spray deposition, and various casting techniques, including squeeze casting, rheocasting, and compocasting [1]. In these conventional fabrication techniques (*ex situ* methods), since the particulate reinforcements are directly incorporated into the matrix material either in molten or in powder form, the scale of the reinforcing phase is limited by the starting powder size, which is typically of the order of microns to tens of microns [2].

During past 20 years, various *in situ* techniques [2], such as exothermic dispersion (XD), self-propagating high temperature synthesis (SHS), and reactive gas infiltration have been developed to fabricate MMCs with ultrafine reinforcing particulates, which are formed *in situ* by chemical reactions between elements or between elements and compounds. In comparison with conventional MMCs produced by *ex situ* methods, *in situ* MMCs exhibit attractive advantages [2]: (a) thermodynamically stable reinforcements lead to less degradation in mechanical properties at elevated temperatures; (b) clean reinforcement-matrix interfaces improve interfa-

cial bonding strength; and (c) fine and homogeneously dispersed reinforcing particulates result in better mechanical properties of MMCs. In addition, *in situ* process is a cost-effective approach to fabrication of particulate-reinforced MMCs. From the viewpoint of both technical and economic aspects, therefore, the *in situ* process offers significant advantages and is a promising approach to mass fabrication of MMCs.

Thermite reactions between, typically, a metal and an oxide, are generally an exothermic process and can become self-sustaining, a feature which makes their use extremely energy efficient [3]. Thermite reactions can be used to fabricate metals and alloys, intermetallic compounds, ceramics, and composites. In recent years, Al–TiO₂ system, in which aluminothermic displacement reactions are possible, has attracted much attention in fabricating various *in situ* composites. By adjusting the ratio of Al to TiO₂, Al-based MMCs and alumina–titanium aluminide (TiAl and/or Ti₃Al) intermetallic matrix composites with interpenetrating networks can be synthesized from Al–TiO₂ powder mixtures via reactive sintering, SHS or reactive infiltration of molten Al into porous performs [4–6]. Regarding Al-based MMCs achieved by aluminothermic reactions between Al and TiO₂, in addition to Al₂O₃ reinforcing particulates, an intermetallic compound Al₃Ti with tetragonal structure and platelet morphology often appears [7]. To overcome the harmful influence of Al₃Ti on strength and ductility of the composites, boron has been tried to incorporate into Al–TiO₂ system [7–11]. In Al–B–TiO₂ ternary system, not only Al₃Ti blocks are refined and disappear gradually, but also fine TiB₂ particulates can be formed, thus further improving the mechanical properties of the composites.

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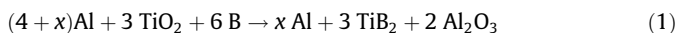
Reactive sintering of a multi-component powder mixture is one of the important routes to fabricate Al-based *in situ* MMCs. Since various chemical reactions take place during reactive sintering process, the composition and microstructure of finished MMCs are quite different from those of the starting reactants. It is believed, therefore, that the processing parameters of the reactive sintering (e.g., sintering temperature) and the characteristics of starting powders (e.g., particle size and its distribution) will dominate the formation of reinforcing phases (including intermediate phases) and, hence, the microstructural development and mechanical properties of the *in situ* MMCs. Unfortunately, the influences of processing parameters and powder characteristics on reaction mechanisms, phase evolution, and microstructural development during the reactive sintering, which are very important to optimize the *in situ* process and the mechanical properties of *in situ* processed MMCs, have not been well understood.

In the present work, Al–TiB₂–Al₂O₃ *in situ* composites were synthesized by reactive sintering of Al–B–TiO₂ three-component powder mixtures with different TiO₂ powder sizes. The objective was to clarify the effects of reactive sintering temperature and powder particle size on formation of the reinforcing phases and microstructural evolution of the Al-based *in situ* MMCs.

2. Experimental procedure

The starting powders used in this investigation were: aluminum (99.7% purity), boron (99.0%), and titania (rutile type). The average particle sizes of Al and B were 45 μm and 31 μm, respectively. Two types of TiO₂ powders with average particle sizes of 2 μm (99.0% purity) and 0.5 μm (99.9%) were used. They will be hereinafter denoted as TiO₂(L) and TiO₂(S), respectively. The particle size distributions of the TiO₂ powders are shown in Fig. 1. TiO₂(L) powder exhibited a larger average particle size and broader particle size distribution in comparison with TiO₂(S) powder.

The powders of Al, TiO₂, and B were ball-milled in ethanol for 24 h using alumina milling media. The mixing proportion of the powders was determined in accordance with the stoichiometry of the following reaction formula:



where x represents excess amount of Al, which constitutes the matrix of the synthesized composites. In this work, powder mixtures with a composition of Al-4.39 wt% B-16.23 wt% TiO₂ (i.e., $x = 39.5$ in Eq. (1)) were prepared, which corresponded to a nominal compo-

sition containing 20 vol% reinforcements (TiB₂ and Al₂O₃) after complete reaction according to Eq. (1).

The mixed powder slurries were dried and then uniaxially pressed into disc-shaped compacts, 20 mm in diameter and approximately 8 mm in thickness, with a pressure of 200 MPa. The compacts were heated in vacuum at a heating rate of 10 K min⁻¹ to a given temperature ranging from 973 K to 1273 K and held for 10 min, followed by cooling to room temperature.

Differential thermal analysis (DTA) of Al–B–TiO₂ three-component powder mixtures was conducted to obtain information on thermal characteristics of reactions during reactive sintering process. For the purpose of identifying the thermal characteristics clearly, an Al–B–TiO₂ powder mixture with a composition of $x = 6.6$ in Eq. (1), i.e., containing 60% reinforcements in Al matrix, was prepared. The samples, taken from the interior of the pressed pellets, were heated up in a purified argon atmosphere (flowing rate of 0.15 l min⁻¹) over a temperature range of 293–1400 K at a heating rate of 10 K min⁻¹ or 5 K min⁻¹. The phase identifications for green compact and reactive-sintered samples were performed by X-ray diffraction (XRD) with Cu K α radiation. Microstructural characterization was carried out by scanning electron microscopy (SEM) with an energy dispersive X-ray spectroscopy (EDS).

3. Results and discussion

3.1. Reaction mechanism

Fig. 2 shows the DTA traces of Al–B–TiO₂ three-component powder mixtures during heating in an argon atmosphere. All the curves exhibited one endothermic peak (B) and three exothermic peaks (A, C, and D). Obviously, the sharp endothermic peak at ~933 K corresponds to the melting of Al. The appearance of multiple exothermic peaks indicates the presence of intermediate reaction steps before thermodynamically stable reinforcing phases are formed. Although the DTA traces in Fig. 2 shows a similar shape, two exothermic peaks C and D were shifted to lower temperatures when small-sized TiO₂(S) powder (Fig. 2b) or slow heating rate (Fig. 2c) was used.

The X-ray diffraction patterns of Al–B–TiO₂(L) samples sintered in a temperature range of 973–1273 K are shown in Fig. 3. As a reference, the XRD pattern of a green compact from Al–B–TiO₂(L)

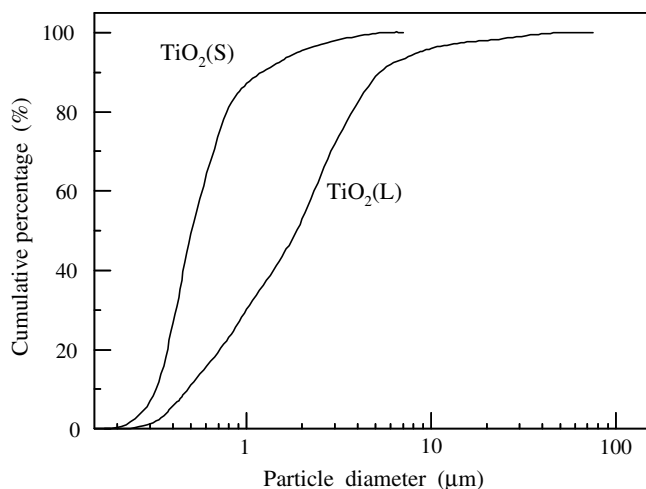


Fig. 1. Particle size distributions of titania powders used in the experiments.

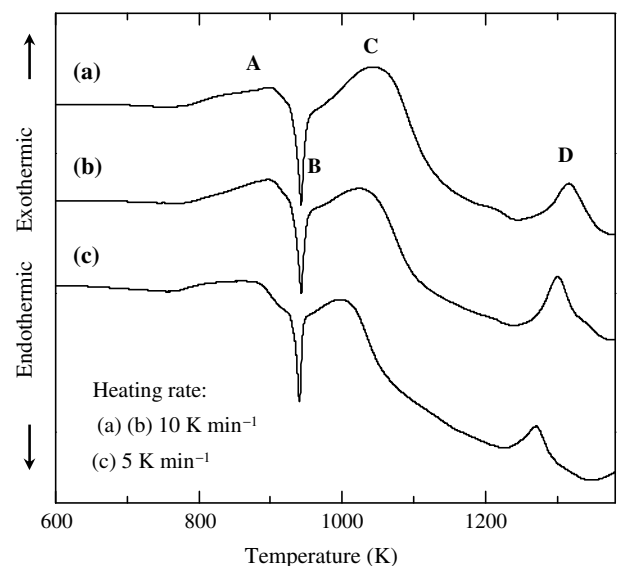


Fig. 2. DTA traces of (a) Al–B–TiO₂(L) and (b), (c) Al–B–TiO₂(S) powder mixtures during heating under an argon atmosphere.

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