Materials Science & Engineering A 625 (2015) 357–368

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

TiB_2 reinforced aluminum based in situ composites fabricated by stir casting

Fei Chen [a], Zongning Chen [b], Feng Mao [a], Tongmin Wang [a,*], Zhiqiang Cao [b,**]

[a] Key Laboratory of Materials Modification by Laser, Ion, and Electron Beams (Ministry of Education), School of Materials Science and Engineering, Dalian University of Technology, Dalian 116024, China

[b] Laboratory of Special Processing of Raw Materials, Dalian University of Technology, Dalian 116024, PR China

ARTICLE INFO

Article history:
Received 22 August 2014
Received in revised form
4 December 2014
Accepted 8 December 2014
Available online 18 December 2014

Keywords:
TiB_2 particles
In situ composites
Stirring
Tomography
Microstructure
Mechanical properties

ABSTRACT

In this study, a new technique involving mechanical stirring at the salts/aluminum interface was developed to fabricate TiB_2 particulate reinforced aluminum based in situ composites with improved particle distribution. Processing parameters in terms of stirring intensity, stirring duration and stirring start time were optimized according to the microstructure and mechanical properties evaluation. The results show that, the first and last 15 min of the entire 60 min holding are of prime importance to the particle distribution of the final composites. When applying 180 rpm (revolutions per minute) stirring at the salts/aluminum interface in these two intervals, a more uniform microstructure can be achieved and the Al-4 wt% TiB_2 composite thus produced exhibits superior mechanical performance. Synchrotron radiation X-ray computed tomography (SR-CT) was used to give a full-scale imaging of the particle distribution. From the SR-CT results, the in situ Al–$x$$TiB_2$ composites (x = 1, 4 and 7, all in wt%) fabricated by the present technique are characterized by fine and clean TiB_2 particles distributed uniformly throughout the Al matrix. These composites not only have higher yield strength ($\sigma_{0.2}$) and ultimate tensile strength (UTS), but also exhibit superior ductility, with respect to the Al–TiB_2 composites fabricated by the conventional process. The $\sigma_{0.2}$ and UTS of the Al–7TiB_2 composite in the present work, are 260% and 180% higher than those of the matrix. A combined mechanism was also presented to interpret the improvements in yield strength of the composites as influenced by their microstructures and processing history. The predicted values are in good agreement with the experimental results, strongly supporting the strengthening mechanism we proposed. Fractography reveals that the composites thus fabricated, follow ductile fracture mechanism in spite of the presence of stiff reinforcements.

1. Introduction

Al based metal matrix composites (MMCs) reinforced with ceramic particles have received extensive attention due to their high specific strength-to-weight ratio, good wear resistance, excellent dimensional stability and superior damping capacity in comparison with the matrix alloy [1,2]. The conventional practice to prepare Al based composites (ex situ composites) involves the addition of externally synthesized reinforcements, such as SiC, Al_2O_3 and TiC, to the matrix alloys [3–5]. This process, however, could lead to segregation and thermodynamic instability of the reinforcements, and poor adhesion at the interface, unless the ceramic particles have been suitably modified [6,7].

To overcome these drawbacks often occasioned in the preparation of ex situ MMCs, in situ techniques have been greatly developed in recent years. Since the formation of the reinforcements takes place within the matrix, in situ synthesized MMCs provide advantages including uniform distribution of finer particles, excellent bonding at the matrix/reinforcement interface, thermodynamical stability of the reinforcements and process economy [8,9].

A wide variety of in situ formed ceramic particulates, such as Al_2O_3, TiB_2 and TiC, have been used as reinforcements to fabricate Al based MMCs [10,11]. Among these particulates, TiB_2 is an advanced strengthening phase for Al matrix as it possesses a desirable combination of physical and mechanical properties, including high melting point (3225 °C), high elastic modulus (534 GPa), high hardness (3400 HV) and outstanding wear resistance. More importantly, it does not react with Al to form any detrimental reaction products at the matrix/reinforcement interface [7,12]. In the past two decades, numerous studies have been conducted to develop new fabrication processes of TiB_2 reinforced

* Corresponding author. Tel.: +86 411 84706790; fax: +86 41184706790.
** Corresponding author. Tel.: +86 411 84706169; fax: +86 411 8470616.
E-mail addresses: tmwang@dlut.edu.cn (T. Wang), caozq@dlut.edu.cn (Z. Cao).

http://dx.doi.org/10.1016/j.msea.2014.12.033
0921-5093/

in situ Al based MMCs with improved wear performance, mechanical properties and damping capacity [6,13,14].

Although the TiB_2 reinforced in situ Al based composites have prominent advantages, their microstructure and mechanical properties are known to be highly sensitive to the processing parameters in the production. Reinforcement segregation and residual intermediate products, such as $TiAl_3$ and AlB_2, are frequently present in the final composites [15–17]. These defects have been found to act as preferential sites for crack initiation and propagation. Therefore, elimination of these defects is regarded as a primary concern to prepare Al–TiB_2 composites with consistent properties [18,19].

In the present study, the stir casting technique, a conventional method to prepare ex situ MMCs [20,21], was elaborately modified to fabricate TiB_2 reinforced in situ Al based MMCs using the halide salt route. In this process, a well-designed device was applied to stirring at the salts/aluminum interface, based on the fact that the reactions, by which TiB_2 is formed, generally take place in the vicinity of the salts/aluminum interface. The effects of processing parameters, i.e. stirring speed, stirring duration, and stirring start time on the microstructure and mechanical properties of the final products have been studied in detail.

Furthermore, synchrotron radiation X-ray computed tomography (SR-CT) has been used to investigate the particle distribution of the experimental Al–TiB_2 composites from three dimensional (3D) view, since the third-generation synchrotron radiation (SR) source has been reported being able to offer a unique opportunity to non-destructively observe the microstructural characteristics of metallic materials with submicron spatial resolution [22,23]. However, in the conventional 2D (two dimensional) measurements, such as, optical and scanning electron microscope analysis, materials microstructure is likely to be destroyed during sample preparation (cutting, polishing and etching) [24–26]. The aim of the present work is to explore the feasibility of fabricating low cost Al based composites with improved mechanical properties, in addition, to enlarge the application of synchrotron radiation in the field of MMCs.

2. Experimental procedures

Al–TiB_2 in situ composites were synthesized by the exothermic reaction of halide salts (KBF_4–K_2TiF_6) with molten aluminum according to the following reactions:

$$3K_2TiF_6 + 13Al \rightarrow 3KAlF_4 + K_3AlF_6 + 3TiAl_3 \quad (1)$$

$$2KBF_4 + 3Al \rightarrow 2KAlF_4 + AlB_2 \quad (2)$$

$$TiAl_3 + AlB_2 \rightarrow TiB_2 + 4Al \quad (3)$$

Commercially available Al (99.8% Al: all compositions are in mass fraction unless stated otherwise), KBF_4 and K_2TiF_6 powders of analytical purity (99% purity) were used as starting materials. Fig. 1 shows the schematic diagram of the set-up for fabricating Al–TiB_2 in situ composites. For each experiment, 1.5 kg Al was melted and heated to 860 ± 5 °C in a graphite–clay crucible under ambient atmospheres, using a resistance furnace. The pre-dried (200 °C for 2 h) K_2TiF_6 and KBF_4 mixture with a stoichiometric Ti/B ratio of 1:2 was then added into the melt. After salts melted, a preheated impeller with four straight blades was employed to stirring at the salts/aluminum interface. The straight blades of the impeller, which could reduce the inclusion of post-reaction slag, were made of graphite due to the fact that graphite will not contaminate both of the molten salts and molten aluminum. Moreover, it has been demonstrated that carbon plays a positive role in the formation of TiB_2.

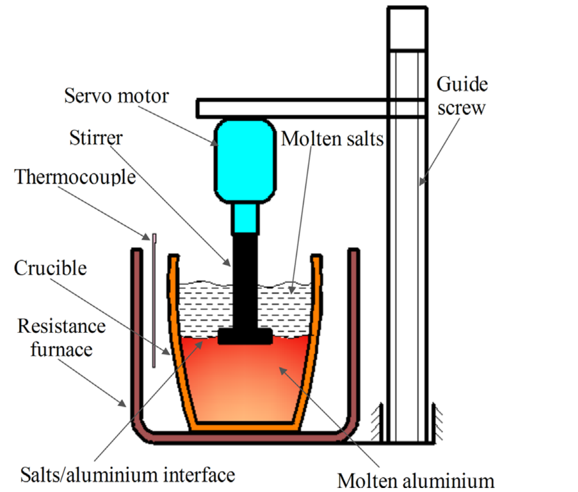

Fig. 1. Schematic diagram of the experimental set-up for fabricating Al–TiB_2 in situ composites.

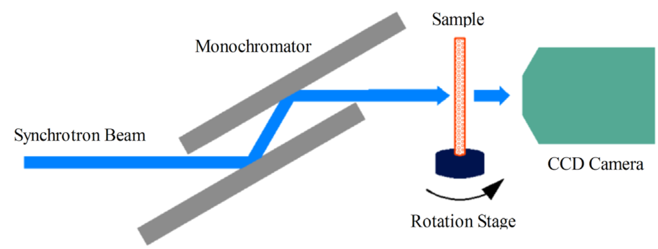

Fig. 2. Schematic diagram of experimental setup of synchrotron radiation X-ray computed tomography.

The stirring intensity (60, 180 and 300 rpm (revolutions per minute)), stirring duration (5, 15, 30 and 60 min) and stirring start time (initial, middle and later) were varied to investigate the effects of stirring parameters on the microstructures and mechanical performances of the Al–4% TiB_2 composites. The melt was held at 860 °C for 60 min to enable the reactions to achieve completion. After decanting by-product K–Al–F salts and degassing for one minute by high-purity argon, the melt of 700 °C was poured into a preheated (200 °C) permanent mold. Composites with 1% and 7% TiB_2 were also fabricated under the optimized process condition to investigate the feasibility of the technique in producing composites with different particle contents.

Phase identification of the composites was performed by an X-ray diffractometer (Empyrean) using Cu-Kα radiation operated at 40 kV and 40 mA. For microstructure analysis, the specimens were polished and etched with Keller's reagent, then examined by a field emission scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS).

The synchrotron radiation analysis was carried out at beam line BL13W1 at Shanghai Synchrotron Radiation Facility (SSRF). Experimental setup is shown in Fig. 2. Samples with 0.7 mm in diameter and 20 mm in length were cut from ingots contain 7% TiB_2 by electro discharge machining. The sample was fixed upright with its axis perpendicular to the incident monochromatic X-ray beam and a beam of 18 keV was focused on and sent through the central area of the sample. The transmitted beam was recorded by a CCD based camera with a spatial resolution of 0.37 μm placed 45 cm behind the specimens. Phase contrast (sensitive to internal structure change), and absorbing contrast (sensitive to density difference) were used to establish images. During the experiment, the sample rotated about the vertical axis and the camera collected