Materials and Design 66 (2015) 492–497

Contents lists available at ScienceDirect

Materials and Design

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

Effect of the heating rate on the microstructure of *in situ* Al_2O_3 particle-reinforced Al matrix composites prepared via displacement reactions in an Al/CuO system

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ARTICLE INFO

Article history:
Received 3 March 2014
Accepted 10 June 2014
Available online 22 June 2014

Keywords:
Aluminum matrix composites
Chemical synthesis
Microstructure
Scanning electron microscopy
Heating rate

ABSTRACT

In this study, an *in situ* Al_2O_3 particle-reinforced Al(Cu) matrix composite was successfully synthesized using a displacement reaction between Al and CuO powders. The powders were mixed at a weight ratio of 4:1 Al to CuO, cold-pressed and holding time at 900 °C for 1 h using varying heating rates. The effects of the heating rate on the microstructures of the composites were investigated using differential scanning calorimetry (DSC), X-ray diffraction (XRD), optical microscopy (MO), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS). The results indicate that all of the composites contain Al, Al_2O_3 particles and Al_2Cu phases. Although the heating rate does not significantly affect the phase compositions of the composites, it has a significant effect on their microstructures, most likely because it strongly influences the diffusion rates of the Cu and O atoms. As the heating rate is increased, the Al_2O_3 particles become more dispersed, and they have a more uniform particle size distribution. Meanwhile, the Al_2Cu structure transforms from the network (Al + Al_2Cu) eutectic to the block-like Al_2Cu phase. The ~2 μm Al_2O_3 particles and the block-like Al_2Cu phase are distributed uniformly in the Al matrix when the sample is placed directly into a 900 °C furnace. This sample has a relative higher Rockwell hardness B (HRB) value of 87.

1. Introduction

Particle-reinforced aluminum matrix composites are of interest because of their high specific strength and stiffness, good wear resistance, and low thermal expansion coefficients. Further, they can be prepared via traditional preparation processes and used for various applications in the aerospace, military, electronic device and automobile industries [1–3]. The most common particles used for reinforcing Al matrices include those composed of Al_2O_3, SiC and TiC. Among these, Al_2O_3 particles have proven their suitability for use in Al-based alloys because they have high hardness, good wettability and enhanced chemical stability when coupled with Al-based matrices. *In situ* reaction processes involving particle-reinforced composite systems eliminate interfacial compounds in favor of nucleation and growth from the parent matrix phase to form thermodynamically more stable reinforced compounds. At the same time, the composites possess contaminant-free reinforcement/matrix interfaces, and the *in situ* Al_2O_3 particles are fine and can increase the strength and ductility of the composite [4].

Al and metal oxide powders are commonly used in displacement reactions. Previous studies have shown that when the temperature is increased, the reaction accelerates. Additionally, the size of the Al_2O_3 particles increase with increasing temperature, holding time and reinforcement content [5–7]. Rapid solidification and ball milling lead to more uniformly distributed Al_2O_3 particles and produce composites with a homogeneous fine microstructure [8–10]. Fine and homogeneous *in situ* Al_2O_3 particles have been synthesized using the hot pressing process [7,11]. These studies indicate that the microstructures of the composites are dictated by the process parameters. It is well-known that small, closely spaced particles pin boundaries and dislocations. Therefore, whether the strength and ductility of the composites are influenced by *in situ* particles depends on the interparticle spacing and particle size. Furthermore, the properties of the composites will be affected by the inclusion of a coarse, continuous, network-like intermetallic compound. Thus, it is necessary to perform a systematic study of the effect of the process parameters on the microstructures of *in situ* Al_2O_{3P}/Al composites.

CuO is one of the most widely used metal oxides because fine *in situ* Al_2O_3 particles are synthesized in Al/CuO systems. The reduced Cu not only dissolves to some extent in the Al matrix but also further reacts with the Al to form an intermetallic phase

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http://dx.doi.org/10.1016/j.matdes.2014.06.023
0261-3069/

that can reinforce the matrix of the composite [12]. The effects of many synthesized process parameters on composite microstructures have already been studied; however, the effect of the heating rate on the composite microstructure in Al/CuO systems has not been clarified completely. Zhu et al. [13] and Kou et al. [14] have performed relevant studies. They reported that when the heating rate increases, the reaction temperature increases. Biswas et al. [15] and Pathak et al. [16] investigated the effect of the heating rate on combustion in the Ni–Al system and found that the heating rate strongly influences the diffusion rate, the resultant phases and their microstructures. Therefore, this study investigates the effect of the heating rate on the composite microstructure, specifically the sizes and distributions of the Al_2O_3 particles and the morphology of Al_2Cu phase. The goal is to obtain dispersed Al_2O_3 particles and avoid network-like Al_2Cu intermetallic compounds.

2. Experiments

Pure Al powder (less than 50 μm) and CuO powder (from several microns to 100 μm) were used for the preparation of the *in situ* composites. The powders were mixed at a weight ratio of 4:1 Al to CuO using a planetary ball mill (QM-BP) operating at 40 rpm for 2 h. Alcohol was selected as the grinding media. The powder mixture was then cold-pressed under 400 MPa of pressure to form discs with diameters of 10 mm. Before synthesized, differential scanning calorimetry (DSC) (Netzsch STA409 PC, heating rates between 0.1 and 50 °C/min) was conducted on small pieces of the sample (ϕ4.0 mm × 1.0 mm) from ambient temperature to 900 °C at heating rates of 5, 10, 20, 30, 40 and 50 °C/min to determine the temperatures where reactions between Al and CuO occurred.

Green samples were holding time at 900 °C for 1 h at the above heating rates in argon atmosphere, and another green sample was placed directly into a 900 °C furnace so that its microstructure could be compared with that of the other samples. All samples were furnace-cooled. X-ray diffraction (XRD, D/MAX-2500/PC, 40 kV, 20 mA, Cu Kα radiation), optical microscopy (MO), scanning electron microscopy (SEM) and energy dispersive spectrometry (EDS) were used to investigate the phase and microstructural changes that occurred when the composites were processed.

3. Results and discussion

3.1. DSC analysis

Following the relevant literature protocols [6,17], the samples were heated to 900 °C at heating rates between 5 and 50 °C/min during the DSC analysis. The results show that the peaks in the DSC curves were influenced by the heating rates and that the reaction temperatures were higher when elevated heating rates were used. Thus, only the DSC curves of the Al–20 wt%CuO samples that were prepared using heating rates of 5 °C/min and 50 °C/min are shown in Fig. 1. Negative endothermic peaks resulting from Al melting are observed in Fig. 1(a) and (b). Two positive peaks are located on the left and right sides of the endothermic peak; the first exothermic peak in Fig. 1(a) is the only one that does not partially overlap the endothermic peak. Another exothermic peak is observed at a higher temperature. The end temperature is approximately 870 °C in the two above DSC curves, so the samples were heated to 900 °C. The peaks in the DSC curves were strongly influenced by the heating rate at relatively low temperatures, while they were only slightly influenced by the heating rate at higher temperatures.

Next, the reactions corresponding to the exothermic peaks in the DSC curves were determined. Briefly, green samples possessing the same thickness as the samples used in the DSC experiments were heated to the temperatures selected according to the DSC curves in a tube furnace (argon atmosphere) and then water quenched. The temperatures were raised to between 640 and 710 °C and to between 650 and 740 °C for the samples prepared using a 5 °C/min and a 50 °C/min heating rate, respectively.

3.2. XRD analysis

The diffraction patterns of the green sample and the samples heated to varying temperatures are shown in Fig. 2. Only peaks that correspond to Al and CuO are observed in the pattern of the green sample (Fig. 2a). For the samples heated at a rate of 5 °C/min, the samples were found to contain Al, CuO, Cu_2O and Cu and Al, Cu_2O and Cu for the samples heated to 640 °C and 710 °C, respectively. The samples heated at a rate of 50 °C/min showed similar XRD results as those that were heated at a rate of 5 °C/min. Thus, the representative XRD results for the sample that was heated to 640 °C at a rate of 5 °C/min and the one that was heated to 740 °C at a rate of 50 °C/min are shown in Fig. 2b and c, respectively.

The phase components of the products that were holding time at 900 °C for 1 h at different heating rates were identified by XRD analysis. The products display similar diffraction patterns, which contain peaks that correspond to Al, Al_2O_3 and intermetallic Al_2Cu. Fig. 2(d) shows the XRD plot of the sample that was heated at a rate of 50 °C/min. As shown, an Al_2O_{3P}/Al composite can be synthesized by a reaction between Al and CuO at 900 °C.

3.3. Microstructure

3.3.1. Green sample

The microstructure of the Al–20 wt%CuO green sample is shown in Fig. 3. An SEM micrograph is shown; the insert in the top right

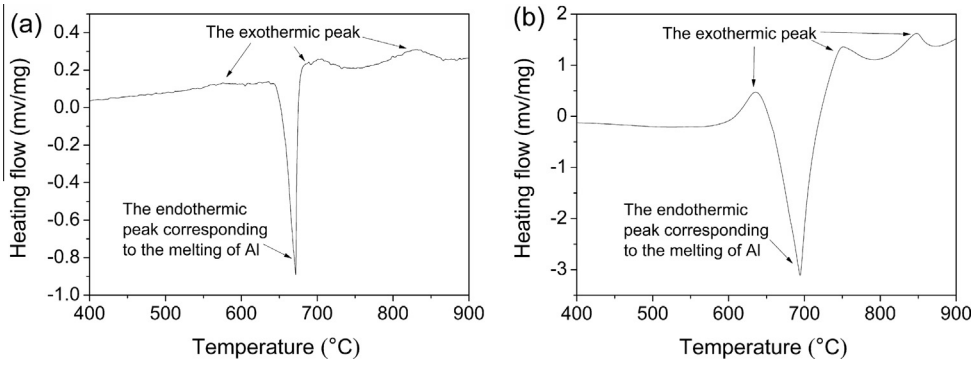

Fig. 1. DSC curves of Al–20 wt%CuO samples obtained under flowing argon using heating rates of (a) 5 °C/min and (b) 50 °C/min.