Fabrication of Al₂O₃/Cu Nanocomposites by Atmosphere-controlled Sintering and Their Properties

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Abstract

The microstructure and mechanical property of hot-pressed Al₂O₃/Cu nanocomposites with a different temperature for atmosphere changing from H₂ to Ar have been studied. When the atmosphere changed from H₂ to Ar gas at 1450°C, the hot-pressed composite was characterized by inhomogeneous microstructure and low fracture strength. On the contrary, when the atmosphere changed at a lower temperature of 1100°C, a more homogeneous microstructure and higher fracture strength was observed.

Keywords : Al₂O₃/Cu nanocomposites, sintering atmosphere, Microstructure

1. Introduction

Nanocomposite processing has received much attention due to the outstanding improvements in the mechanical properties and a high possibility for applications to functional materials [1]. The ceramic/metal nanocomposites with desired properties can be fabricated by reducing and hot-pressing the powder mixture of ceramic and metal oxide prepared by solution chemistry routes [2,3]. Considering that the mechanical and functional properties of the nanocomposites are so sensitive to the microstructure, it is apparent that some variations in the processing route can give a pronounced effect on the microstructure and thus its final properties. In this regard, a closer study of how the reducing and sintering atmosphere influences the microstructure and mechanical properties of ceramic/metal nanocomposites was performed.

In this paper, differences in the temperature of changing the atmosphere from H₂ to Ar gas and in the resulting microstructural characteristics are described for Al₂O₃/Cu nanocomposites. Moreover, the relationship between microstructure and mechanical properties is discussed.

2. Experimental and Results

High-purity -Al₂O₃ powder with a particle size of less than 0.2 m and CuO powder with an average particle size of 2 m were selected as raw materials. The starting powders were weighed with a fraction of 5 vol% Cu in the final composition. The powders were ball-milled in ethanol for 24 h and dried in an oven. The powder mixtures were kept in a graphite die and reduced by flowing H₂ gas at 350°C for 30 min. The H₂ gas was maintained at temperatures of 1000, 1100, 1200 and 1450°C, respectively. Then it was changed to Ar gas, and the sintering was carried out at 1450°C for 1 h under a pressure of 30 MPa.

Phase identification was carried out by X-ray diffraction (XRD) analysis, and the microstructure was characterized by scanning electron microscopy (SEM). More than five specimens were subjected to three-point bending tests (span: 30 mm, cross-head speed of 0.5 mm/min) to determine the fracture strength.

The XRD analysis revealed that the hot-pressed composites were composed entirely of -Al₂O₃ and Cu regardless of the gas changing temperature. However, it should be noted that the meaning of the XRD result is only restricted to the region of its resolution.

Fig. 1 shows typical SEM images of the hot-pressed composites with different gas changing temperatures. All composites showed the same relative density of 99.3%. The increase in the gas changing temperature from 1000°C (Fig. 1a) to 1450°C (Fig. 1b) induced an increased inhomogeneity in the microstructure, though the sintering was done with the same temperature. Also, the thermally etched surfaces of Al₂O₃/5 vol% Cu composites showed that a homogeneous microstructure with equiaxed Al₂O₃ grains was observed in the composite, gas-changed at 1000°C, while the microstructure of the composite with gas changing temperature of 1450°C was characterized by exaggerated grain growth. From the SEM images, the grain size of the Al₂O₃ matrix was measured. The average sizes of
Al₂O₃ grains in hot-pressed composites with various gas changing temperatures are very close to each other by about 0.68 µm. However, the Cu particle sizes increased with increasing gas changing temperature from 190 nm for 1000°C to 300 nm for 1400°C.

![Fig. 1 SEM micrographs of the polished surfaces for composites with gas changing temperature of (a) 1000°C and (b) 1450°C.](image)

The fracture strengths and their standard deviation for Al₂O₃/Cu nanocomposites are shown in Fig. 2 as a function of gas changing temperature. A maximum strength value of 819 MPa was achieved in the composite gas-changed at 1100°C, and increasing temperature to 1450°C caused the fracture strength to decrease. It is interesting to note that the standard deviation linearly increased with an increasing gas changing temperature, from 37 MPa for 1000°C to 198 MPa for 1450°C. Combined with the microstructural evolution shown in Fig. 1, also the fracture strengths and especially of their standard deviation of composites showed a strong sensitivity to gas changing temperature.

In general, it is known that the abnormally grown grains and inhomogeneous dispersion of the second phase are harmful to fracture strength due to the acting as fracture origin [4]. Thus, the deterioration of mechanical properties in the composite with high temperature gas changing can be explained by microstructural inhomogeneity, as shown in Fig. 1. In addition, the interfacial strength between Al₂O₃ and Cu can affect the fracture strength. Many researchers [5,6] reported that the fracture strength increased from about 40 MPa to 250 MPa as the CuAlO₂ thickness at the Al₂O₃/Cu interface increased from 0 µm to 8 µm.

![Fig. 2 Fracture strength and standard deviation of the hot-pressed composites.](image)

3. Summary

The influence of the reducing and sintering atmosphere on the microstructure and mechanical properties of Al₂O₃/5 vol% Cu nanocomposites was investigated. The hot-pressed composite with high temperature gas changing was characterized by inhomogeneous microstructure and low fracture strength, while that with lower temperature gas changing showed a more homogeneous microstructure, higher fracture strength and smaller deviation in strength. Mechanical properties depending on gas changing temperature were explained by the microstructural characteristics relating to the Cu wetting behavior.

4. References