*Journal of Ceramic Processing Research. Vol. 3, No. 3, pp. 192~194 (2002)*

JOURNAL OF Ceramic Processing Research

# A study on the fabrication of $Al_2O_3$/Cu nanocomposite and its mechanical properties

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**In order to investigate the effective content of second phase Cu for enhanced fracture toughness and mechanical properties of $Al_2O_3$/Cu nanocomposite, materials with variation of 1 to 5 vol% Cu were studied. To obtain a fine, homogenously dispersed Cu phase in an $Al_2O_3$ matrix, hydrogen reduction of ball milled $Al_2O_3$ and CuO powder mixtures was preformed. Pulse electric current sintering (PECS) process was used to consolidate the composite powder, which was $Al_2O_3$ embedded with Cu particles formed by reduction of CuO. Relative densities of the sintered parts were over 99% by Archimedes' principle. Sintered specimens were analyzed by X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM). Mechanical properties improved significantly with increasing Cu content. The increase of fracture toughness was explained by crack bridging and pull-out. The fracture strength was increased by the refinement of $Al_2O_3$ matrix grains through the nano-sized Cu dispersion.**

**Key words: Nanocomposite, Rapid solidification, Mechanical properties.**

## Introduction

$Al_2O_3$ possesses favorable physical and chemical properties such as high strength, hardness, high elastic modulus and excellent resistance to thermal and chemical environments. However, its application is somewhat limited because of poor toughness and inferior thermal shock resistance [1]. It has been reported that the incorporation of small amounts of nano-sized metal particles into an $Al_2O_3$ matrix, as in the case of $Al_2O_3$/Ni [2] and $Al_2O_3$/W [3] nanocomposites, can significantly improve the mechanical properties.

Pulse Electric Current Sintering (PECS) process was used to consolidate $Al_2O_3$/Cu nanocomposite powder. The advantage of using PECS lies in the fact that it can provide rapid heating of the whole sample, allowing a very short sintering time and a homogeneous sintered body [4]. Employing PECS process, one can pass quickly through the surface diffusion regime where coarsening is active and proceed to the boundary or lattice diffusion regime where densification mechanisms operate. Additionally, it has been shown that PECS makes rapid densification of the powder compact possible at relatively low temperatures under applied pressure.

An earlier study demonstrated the fabrication of $Al_2O_3$/5 vol.% Cu nanocomposite prepared from fine $Al_2O_3$ and CuO powder mixtures [5, 6]. In the present approach, the microstructure evolution and mechanical properties of $Al_2O_3$/Cu composites with various Cu contents below 5 vol.% were studied. An optimum route to fabricate nanocomposites with sound microstructures and desired mechanical properties are discussed as well as relationships of their processing and properties.

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## Experimental Procedure

Starting mixtures were prepared from the following powders; $\alpha$-$Al_2O_3$ (99.99%, 0.1-0.2 $\mu$m) and CuO powder (99.9%, 1-2 $\mu$m) [4, 6-8]. Alpha-$Al_2O_3$ and CuO powders were mixed to obtain final compositions of 1 to 5 vol.%Cu in the composites as shown in Table 1 [9]. The powder mixture was high energy ball milled at 900 rpm for 5 hr. The high energy ball milling process was performed in a horizontal rotary ball mill (Simoloyer, Zoz GmbH) using stainless steel balls with the ball-to-powder volume ratio of 15:1. The milled powders were heat treated for 30 min at 300°C in $H_2$ atmosphere to reduce CuO to Cu.

The composite powders were sintered at 1250°C for 5 min in vacuum under a pressure of 50 MPa by using PECS (Model SPS-515S, Sumitomo Coal Mining Co., Ltd., Japan). Other processing parameters were as follows; a pulse sequence time of 14 $\mu$s, 6:1 pulse on/off ratio. The heating rate was 100°/min with a 5 min soaking at 900°C during the heating up process.

Relative densities of sintered parts were evaluated by Archimedes' principle. The microstructure was observed in more detail by SEM and by TEM. The fracture toughness ($K_{1C}$) was measured by the Indentation Fracture (IF) method with a Vickers hardness tester (98 N-load applied for 15 s). The fracture strength ($\sigma_f$) was evaluated using the piston-on-3-ball test with crosshead speed of 0.13 mm/min. Figure 1 is a schematic

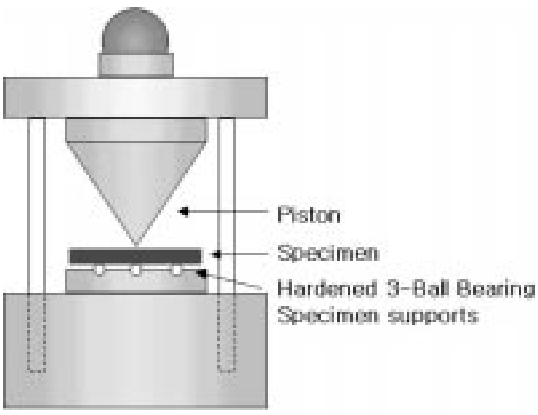


**Fig. 1.** Schematic diagram of piston-on-3-ball test apparatus.

diagram of the piston-on-3-ball test. The specimens were disks 20 mm in diameter and 2 mm thick.

## Results and Discussion

XRD analysis of the powder mixture after reduction by $H_2$ gas and after processing by PECS into composites revealed that the CuO was completely converted to metallic Cu and the composites were composed entirely of $Al_2O_3$ and Cu within the XRD resolution.

The reduced powders were sintered at 1250ºC for 5 min in a vacuum using PECS. Basic data for the $Al_2O_3$/Cu composites are summarized in Table 1 and compared with monolithic $Al_2O_3$ that was sintered under the same conditions. Table 1 shows that the relative densities of sintered parts were over 98% by Archimedes' principle. In the case of hot-pressing $Al_2O_3$/Cu powder mixtures [6], a sintering temperature above 1450ºC is typically required. It is worth noting that the consolidation process by PECS was completed at relatively low temperatures with holding time of only 30 min including heating and cooling stages. Such rapid densification has been reported in the literature [10], and is probably due to such factors as self-heat generation by electric discharge between particles, activation of the particle surface and high speed mass transfer by volume and grain boundary diffusion.

The fracture toughness of sintered specimens was measured by the Indentation Fracture Method using a Vickers hardness tester [11] and calculated by the following equation:

$$K_{IC}=0.016(E/H)^{1/2}(P/c^{3/2})$$

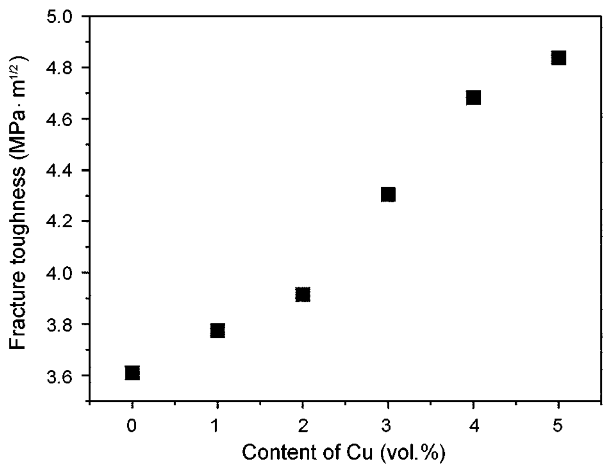


**Fig. 2.** Fracture toughness of composites consolidated by PECS.

where $E$ is the elastic modulus, $H$ the micro hardness, $P$ the applied load, and $c$ the half-length of crack introduced by the Vickers indent.

Figure 2 shows the fracture toughness of sintered composites consolidated by PECS. The fracture toughness increased with increasing Cu content. The $Al_2O_3$/5 vol.%Cu composites showed the highest fracture toughness of 4.85 $MPa \cdot m^{1/2}$, which was 1.3 times larger than that of the monolithic $Al_2O_3$.

The crack propagation behavior around the Vickers indentation is shown in Figure 3. Generally, in the case of composites that contain dispersed phases and exhibit good toughness, the increase of fracture toughness can be explained by mechanisms such as crack depletion, crack bridging, pull-out, etc. [12] It is suggested that

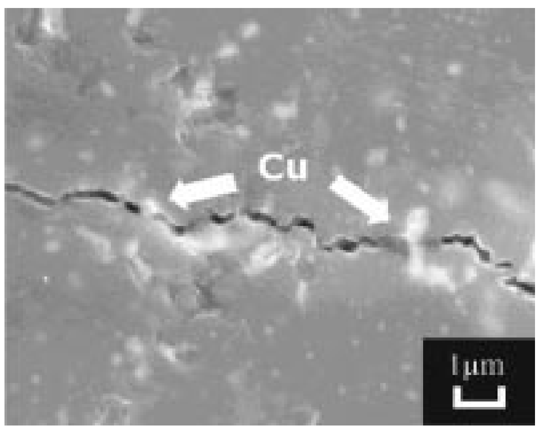


**Fig. 3.** Crack entrapped between Cu particles.

**Table 1.** Dependence of properties on Cu contents

| Cu contents | Pure $Al_2O_3$ | $Al_2O_3$/ 1 vol.%Cu | $Al_2O_3$/ 2 vol.%Cu | $Al_2O_3$/ 3 vol.%Cu | $Al_2O_3$/ 4 vol.%Cu | $Al_2O_3$/ 5 vol.%Cu |
|---|---|---|---|---|---|---|
| Relative density | 98.16 | 98.45 | 99.17 | 99.66 | 99.42 | 99.38 |
| Elastic modulus (GPa) | 375.39 | 373.106 | 371.79 | 368.88 | 365.26 | 364.98 |
| Hardness (HV) | 1862.2 | 1684.3 | 1680.7 | 1630.1 | 1623.7 | 1559.3 |

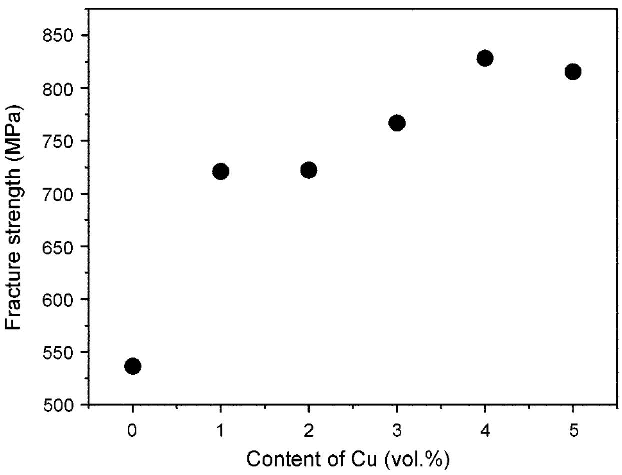


**Fig. 4.** Fracture strength of composites consolidated by PECS.

the fracture toughness of the present sample was increased by crack bridging and pull-out.

Figure 4 shows the variation of fracture strength with Cu content. On the whole, fracture strength increased with Cu content. It is well known that the strength, $\sigma_f$, of a brittle material is related to the fracture toughness $K_{1c}$ and one half of initial flaw, c, as indicated by the following equation [13]:

$$\sigma_f = \frac{1}{Y}\frac{K_{1c}}{\sqrt{c}}$$

where $Y$ is the geometrical parameter of the flaw. In general, $c$ is proportional to the grain size, $d$, in dense polycrystalline materials. Thus, the strength increased when the grain size was small because the addition of Cu second phase interrupted the grain growth of the matrix. Especially, fracture strength of the 1 vol.% Cu dispersed $Al_2O_3$ composite was significantly improved in comparison with that of the monolithic $Al_2O_3$ prepared under the same conditions. It is reasonably explained that the increased strength is attributed to the refinement of the matrix grains due to the growth restriction by fine Cu dispersion.

## Conclusions

The microstructure and mechanical properties of $Al_2O_3$ matrix composites with various Cu contents were investigated. The composites were fabricated by heat treatment of an $Al_2O_3$/CuO powder mixture in reducing atmosphere followed by PECS processing. Relative densities of sintered parts were over 99% by Archimedes' principle. The uniform distribution of Cu particles resulted in up to 1.3 times increase in fracture toughness by. The improvement in toughness resulted from crack bridging and pull-out. The $Al_2O_3$/4 vol.%Cu composites showed the maximum fracture strength of 840 MPa, which was 1.5 times higher than that of monolithic $Al_2O_3$. The increase in fracture strength was explained by the decrease in the matrix grain size, improved microstructure homogeneity and increased fracture toughness.

## Acknowledgement

This work was supported by the Korea Science and Engineering Foundation (KOSEF) through the Ceramic Processing Research Center at Hanyang University.

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