O U R N A L O F

Ceramic Processing Research

Production and characterization of Al₂O₃-Cu composite materials

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Using an intense powder mixture of Al_2O_3 with different copper contents (1, 5, 10, 20 or 30 mass %) several Al_2O_3 -Cu composite materials were fabricated. The microstructure of the composites was observed by optical and scanning electron microscopy. The observations show a microstructure formed by an Al_2O_3 ceramic matrix with fine Cu metallic networks. Due to the liquid sintering mechanism, the relative density reached by the samples was higher than 95%, this together with the fine and homogeneous microstructure present in the samples let us obtain composite materials with good values of toughness, hardness, elastic modulus and electrical properties. The incorporation of a ductile metal inside a hard ceramic matrix increments its toughness. The probable toughening mechanism is crack bridging due to the presence of a homogeneous ductile metal networks in the material's microstructure. The values of density, hardness, elastic modulus and electrical resistance of the composites are directly dependent on the copper content in the matrix, because with an increase in copper content the density of the composites is larger, whereas, the hardness, elastic modulus and electrical resistance are reduced.

Key words: Al₂O₃-Cu, Composites, Toughness, Electrical properties, Metal networks.

Introduction

Alumina (Al₂O₃) possesses favorable physical and chemical properties such as high strength, hardness, elastic modulus and excellent resistance to thermal and chemical environments. However, its applications are somewhat limited because of poor toughness and inferior thermal resistance [1]. On the other hand, most structural ceramics present poor electrical conductivity. It has been reported that the incorporation of some amounts of small-size metal particles into an Al₂O₃ matrix, as in the case of Al₂O₃/Ni [2], Al₂O₃/W [3] and Al₂O₃/Cu [4] composites, can significantly improve both the toughness and electrical properties.

In this way the requirement to produce ceramics with both high toughness and electrical properties has created the necessity to fabricate new ceramic systems, reinforced with metallic particles and produced through the development of new methods. One of these methods is mechanical alloying. Mechanical alloying has been developed since the 60's, however not until a few years ago has it started to be used significantly in the processing of new advanced materials, because with its application composite materials with unusual microstructures and properties may be obtained [5-7].

In particular, interesting studies are being made using mechanical alloying in the fabrication of Al₂O₃-based

ceramics, where the effect of copper particles dispersed in the ceramic is analyzed. The reinforcement models indicate that the size of the metallic inclusion is very important as is the adhesion of the metallic particles to the ceramic matrix, as well as the homogeneous distribution of the metallic particles in the matrix, in order to obtain a composite material with good general properties [4, 8]. Therefore, considerable effort has been made to establishment the optimal processing conditions to obtain materials with the desired characteristics.

The goal of this study is to fabricate Al₂O₃-Cu composite materials with different copper contents through combinations of techniques such as; mechanical alloying and pressureless sintering. Also, the relationships between the microstructure and mechanical properties are analyzed.

Experimental

The starting materials were Al_2O_3 powder (99.9%, 1 µm, Sigma, USA) and copper powder (99.9%, 1-2 µm, Aldrich, USA). The final copper contents in the composites produced were 1, 5, 10, 20 or 30 mass %. The powder mixture was prepared in a ball mill with ZrO₂ media, the rotation speed of the mill was of 210 minute⁻¹ for 24 h. The ball-to-powder volume ratio was of 25:1. With the milled powder mixture, cylindrical samples 2 cm diameter and 0.2 cm thickness were fabricated by uniaxial pressing using 270 MPa pressure. Then the pressed samples were sintered in an argon atmosphere following the heating cycle shown in

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Fig. 1. Heating cycle for sintering different composites.

Figure 1. The density and open porosity of green and fired specimens were determined using Archimedes' method. The microstructure was observed by optical microscopy (OM) and scanning electron microscopy (SEM). The hardness of samples was evaluated as micro hardness using Vickers indentation, toughness was estimated by the fracture indentation method [9], and the elastic modulus was determined by an ultrasonic method. Finally, the electrical resistance of the material was measured by a DC four-probe technique [10].

Results and Discussion

Density and porosity

Figure 2 shows density and porosity changes as a function of the copper content in the composites. In Fig. 2 it is observed that sample's density increases with a larger copper content in the composite, this behavior is logical because copper is denser than alumina. However, from Fig. 2 it is evident that the porosity diminishes with an increase in copper content in the sample. In this sense it can be commented that effectively the increments in the copper content enhance the final density of the composite. Li [11] has documented that liquid copper wets oxide ceramics well, so probably, the behavior observed in this study is that the liquid phase formed by the copper during sintering at 1300 °C (the melting point of copper 1083 °C) helps density the composites, because the liquid goes inside the bulk of the sample eliminating porosity. Microstructure

The microstructures of the composites as a function of copper content are shown in Fig. 3. Figure 3(a) corresponds to a sample Al_2O_3 -1 mass % Cu in which a microstructure with fine Al_2O_3 grains is observed. The copper is the light phase and it is localized in the grain boundaries. Also, some porosity is observed in the sample localized in intergranular zones. Figure 3(b) show the microstructure of a sample Al_2O_3 -5 mass % Cu, which has similar characteristics to that in Fig. 3(a). This means the gray phase corresponds to the ceramic matrix and a lighter phase that corresponds to the metallic phase. However, the increment in the content of metal in the sample is evident in the grain



Fig. 2. Density and open porosity changes as a function of the copper content in the composites. Samples sintered at 1300°C for 1 h.

boundaries. The microstructure of a sample Al₂O₃-10 mass % Cu is observed in Fig. 3(c). Here the metallic network is still partial and isolated, but the increment in the copper content is evident. Another, important observation is the grain growth in the sample. The microstructure of a sample Al₂O₃-20 mass % Cu is presented in Fig. 3(d). Notably here the grain size is very large for this sample and the pores are also larger but their amount has diminished. This behavior is due principally to the major presence of copper in the sample. Finally, the microstructure of a sample Al₂O₃-30 mass % Cu is observed in Fig. 3(e). An important characteristic of this sample is the grain growth. However, the more important observation is the metallic network formed between alumina grains. The formation of this network brings changes in both density and open porosity as before discussed and probably in the final properties of the composite. It is important to consider the following situation; sintering or densification is a diffusion process and diffusion is a mass transfer phenomena. So if there is copper in the system and this metal is a very good conductor of heat, this means by analogy that copper can transport mass easily. So mass transfer is enhanced with the presence of copper by a liquid sintering mechanism and for this reason the enhanced grain growth in the samples with increment in the copper content is also explained.

Mechanical properties

The measured hardness, fracture toughness (K_{IC}) and Young's modulus of the samples as a function of copper content are reported in Table 1. It is seen that the hardness of the samples diminish with increment in the copper content, whereas, the fracture toughness shows the opposite behavior. This behavior is logical because copper is a soft and ductile metal, so when it is incorporated in a ceramic matrix its presence diminishes the hardness of the matrix. On the other hand, the incorporation of ductile metals inside a hard ceramic matrix increases its toughness. The probably toughening mechanism is crack bridging due to the presence of a homogeneous ductile metal network in the microstructure, as observed in Fig. 3.



Fig. 3. SEM microstructures of composites as a function of the copper content. sample Al_2O_3 -1 mass %Cu, b) sample Al_2O_3 -5 mass %Cu, c) sample Al_2O_3 -10 mass %Cu, d) sample Al_2O_3 -20 mass %Cu and e) sample Al_2O_3 -30 mass %Cu. Samples sintered at 1300°C for 1 h.

Data of the Young's modulus (E) determined by an ultrasonic method are also listed in Table 1 for each sample as a function of copper content. The elastic modulus of the samples tends to diminish with the increments in the copper content. This behavior is due to the presence of a ductile metal in a stiff matrix. Also it is important not to forget that the densities in the specimens are larger with an increase in the copper content. So a composite with larger copper content is less stiff but it is denser.

Electrical properties

The results obtained in the composites for the electrical current as a function of voltage, present a

lineal behavior, allows the electrical resistance to be determined using Ohm's Law, with a lineal adjustment in the experimental data. The linear behavior was observed in all samples when different voltages were applied and the following seen;

a) The electrical current obtained rises for the same voltage in all composites.

b) This indicates that with larger copper contents in the samples, the electrical current passes easily through the composites.

c) In consequence the electrical resistance diminishes as the copper content in the sample is increased.

This behavior is presented in the Fig. 4, where it is observed that the slope of the curves diminish with an

Table 1. Hardness, fracture toughness and elastic modulus of different composites

	Al ₂ O ₃ -1 mass % Cu	Al ₂ O ₃ -5 mass % Cu	Al ₂ O ₃ -10 mass %Cu	Al ₂ O ₃ -20 mass %Cu	Al ₂ O ₃ -30 mass %Cu
Hardness/(HV)	115.2	92.0	86.4	59.3	54.9
$K_{IC}/(\mathrm{MPam}^{1/2})$	2.06	2.90	3.59	4.61	7.44
E/(GPa)	191.9	175.2	70.8	57.1	62.5



Fig. 4. Graph of voltage versus electrical current, for different composites.



Fig. 5. Zoom of figure 4 of voltage versus electrical current, for composites with 1, 5 and 10 mass % Cu.



Fig. 6. Graph of electrical resistance versus copper content in different composites.

increase in the copper content in the composite. In Fig. 4 the current-voltage behavior for the 1, 5 and 10 mass % Cu compositions cannot be observed easily because the scale used, with the same applied voltages is that appropriate for the 20 and 30 mass % Cu compositions. For this reason Fig. 5 presents this data for the compositions 1, 5 and 10% Cu.

From the linear adjustment of the current-voltage ex-

perimental data the electrical resistances were computed. The results are shown in Fig. 6 as a function of the copper content in the composites. It is observed that the electrical resistance diminishes with the increments of copper content. As is well known copper is a good conductor of electricity, so its presence in samples explains the behavior of the electrical resistance in the curve of Fig. 6.

Conclusions

• Al₂O₃-Cu composite materials with different copper contents were produced successfully by a combination of techniques such as; mechanical alloying and pressureless sintering.

• The liquid phase formed by the copper during sintering at high temperatures helps to give dense composites, because the liquid goes inside the bulk of the sample eliminating porosity.

• The microstructure of composites is made up by an Al_2O_3 ceramic matrix interconnected with a fine and homogeneous Cu metallic network.

• The incorporation of a ductile metal inside the hard ceramic matrix raises its toughness. The probably toughening mechanism is crack bridging due to the presence of a homogeneous ductile metal network in the microstructure.

• Values of density, hardness and electrical resistance of the composites are directly dependent on the copper content in the matrix, because with an increase in copper content the density of composites is larger, whereas, the hardness and electrical resistance are reduced.

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