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Fracture toughness enhancement for metal-reinforced alumina

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With the idea to determine ways of tailoring alumina (Al_2O_3) in order that one or more toughening mechanisms are activated in service, investigations about the production of Al_2O_3-based composites with different reinforcement metals and intermetallics have been carried out. The synthesis of composites materials has been made by means of both; liquid and solid pressureless sintering of an intensive mechanical mixture of powders. With the use of some metals in the chemical formulations, significant improvements in ceramic toughness have been reported. From the fracture toughness measurements and microstructural observations, it can be concluded that the toughening mechanism in Al_2O_3/metal reinforced composites is due to crack bridging and crack deflection.

Key words: Sintering, Particulate reinforced composites, fracture toughness.

Introduction

Al_2O_3 is a very useful industrial material and the most widely used ceramic. It possesses favorable mechanical properties such as: high hardness, high compressive strength, good chemical and thermal stability and a high elastic modulus [1]. However, its applications as a structural material have been limited by its low fracture toughness and low-fracture strength. Because cracks easily propagate in ceramics; thus, they fail unexpectedly in service. Several authors have reported that the incorporation of some amounts of small-size metal or intermetallic particles into Al_2O_3-ceramics can result in an improvement of its fracture toughness. As an example they have been reported the production by diverse methods and with different amount of reinforcement by metals Al_2O_3/Al [2], Al_2O_3/Cr [3], Al_2O_3/Cu [4], Al_2O_3/Ni [5], Al_2O_3/Mo [6], Al_2O_3/Ti aluminide [7] and Al_2O_3/Ni_3Al [8] with good improvements in the fracture toughness. In these systems the effective mechanism yielding that property is the crack bridging due to ductile metallic ligaments [9, 10]. On the other hand, processes that imply the use of a chemical reaction in situ in order to obtain Al_2O_3-aluminide alloys have been developed recently [11, 12]. The materials produced here also present a good improvement in the fracture toughness. In these studies the reinforcement gives an ability of the ceramic composite to activate toughening mechanisms such as: crack bridging or crack deflection.

In this article the synthesis of Al_2O_3-based composites reinforced with both: metals or intermetallics phases is analyzed as a function of their chemical formulation. On the other hand, enhancement of the fracture toughness acting in ceramic composites is discussed as a function of the final microstructure.

Experimental Procedure

The starting raw material were powders of Al_2O_3, FeO, NiO, TiO_2 and ZrO_2 (99.9%, 1 mm, Sigma, USA) and powders of aluminum, cobalt, copper, iron, molybdenum, nikel, titanium and zirconium (99.9% purity, 1-2 mm, Aldrich, USA). For the composites reinforced with pure metals the amount of powders used was one that allowed obtaining Al_2O_3-based composites with 10 vol. % of the respective metal. For the composites reinforced with intermetallics, they were used as oxides of the respective metal for the in situ synthesis of the corresponding intermetallic phase. The sum of the starting materials; was fitted to the necessary amounts to form the products indicated in reaction (1) with 10 vol.% of each intermetallic phase.

$$Me_xO_y + Al \rightarrow Me_xAl_y + Al_2O_3 \qquad (1)$$

where: Me is any of the next metals; Fe, Ni, Ti and Zr.

The processing and characterization of the composites were as follows: The weighted powders were put under a process of dry mix-milling at a speed of rotation of 300 rpm for 12 h, with the help of a horizontal mill (Cole Parmer, Labmill) using as milling elements balls of stabilized ZrO_2 (YSZ), the weight ratio of balls/powders was of 25 : 1.

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The powder mixtures were then fabricated into ten cylindrical samples of each composition with dimensions of 20 mm in diameter and 3 mm in thickness; this was done by uniaxial pressing of up to 200 MPa. The pressed samples were sintered in an electrical furnace (Carbolite, 1700) without the application of pressure at 1500 °C for 1 h in an inert atmosphere. The speeds of heating and cooling remained constant and were 10 $Kminute^{-1}$. The characterization of sintered products was carried out in the following way; the density was evaluated by the Archimedes' method, the hardness was measured with the help of Vickers indenter, the fracture toughness was determined by the method of fracture by indentation using the equation of Evans [13]. Reported values are the average of ten measurements. The microstructures of the composites were observed with the help of a scanning electron microscope (SEM). The SEM was equipped with an energy dispersive X-ray spectrometer (EDX) with which the phases present in the microstructure could be identified.

Results and Discussion

Microstructure

Fig. 1. shows typical microstructures obtained by SEM of some of the composites investigated. Here, it can be seen fine and homogeneous microstructures, with the presence of two phases, on the basis of (EDX) analysis, it is deduced that the gray phase corresponds to the alumina matrix and the small white and brighter phase corresponds to the metallic reinforcement added to the ceramic matrix. The metallic phase is localized principally at intergranular positions. The main metallic particle size is on average 1 mm. In general all microstructures are fine, however the use of Ni, Ti and the corresponding intermetrallics help to obtain the finest microstructures in the composites. Judging from the trend disclosed by the Al_2O_3/intermetallic composites, it can be noticed that the microstructures have no cracks or pores, thus suggesting that the in situ formation of the intermetallics did not just occur, but in addition helped in the diffusion process in order to obtain well consolidated bodies. Image analysis performed on all the samples studied showed that the average volume fraction of the metallic phase in the composites was approximately 9.5%.

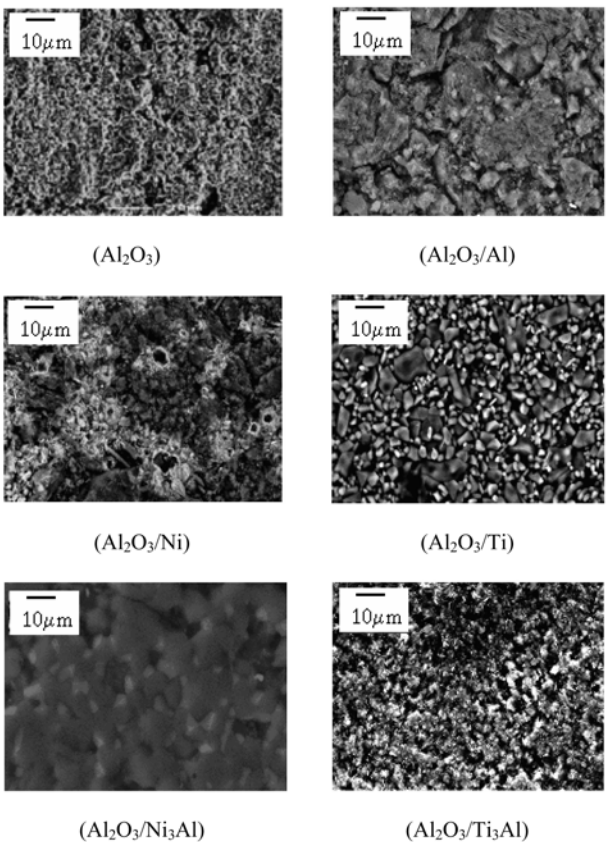


Fig. 1. Typical microstructures obtained by scanning electron microscopy of some of the composites investigated here.

The values of density, hardness and fracture toughness evaluated in the composite materials fabricated here are reported in Table 1. In this table also are reported the corresponding values for monolithic Al_2O_3 also processed here.

Density

From this table it can be observed that the composite materials reinforced with Al, Cu, Fe, Mo and Zr display a lower relative density than monolithic Al_2O_3, whereas the composite materials reinforced with the other metals (Co, Ni and Ti) including all the intermetallics used, show better densification than monolithic Al_2O_3. The worst densifications were obtained in composites with Al and Mo, this may be due to the big differences in densities and melting points between these two metals in comparison with the corresponding values of monolithic Al_2O_3. This difference provokes poor difussion during the sintering stage, leading to heterogeneous microstructures and in consequence bad densification of the products. For the cases were good densifications were obtained, as well as for the cases where in situ intermetallics were formed, in addition the reactions allowed some diffusion mechanisms

Table 1. Values of relative density, hardness and fracture toughness of the different Al_2O_3-based composites fabricated here

Reinforced Metal	ρ relative (%)	HV (GPa)	K_{IC} ($MPa{\cdot}m^{-1/2}$)
Al_2O_3	94.95 +/- 1.2	20.97 +/- 1.7	3.2 +/- 0.2
Al	89.01 +/- 0.88	18.62 +/- 1.3	4.1 +/- 0.1
Co	96.64 +/- 0.79	18.61 +/- 1.4	4.3 +/- 0.1
Cu	93.32 +/- 0.91	18.90 +/- 1.2	4.4 +/- 0.1
Fe	92.82 +/- 1.10	18.51 +/- 1.5	4.0 +/- 0.1
Mo	89.17 +/- 0.93	19.03 +/- 1.3	4.1 +/- 0.1
Ni	96.35 +/- 0.80	18.11 +/- 1.4	4.7 +/- 0.1
Ti	98.25 +/- 0.83	18.17 +/- 1.5	4.8 +/- 0.1
Zr	92.59 +/- 0.88	19.10 +/- 1.6	4.2 +/- 0.1
Fe_3Al	95.40 +/- 0.94	18.78 +/- 1.2	5.2 +/- 0.2
Ni_3Al	98.30 +/- 0.97	16.43 +/- 1.4	6.9 +/- 0.2
Ti_3Al	98.52 +/- 1.10	16.10 +/- 1.6	7.3 +/- 0.2
Zr_3Al	98.76 +/- 0.89	18.12 +/- 1.5	7.0 +/- 0.2

to be activated during the process helping the densification of the products. The densification of the reinforced sample with titanium was very good, and it was equivalent to the densification obtained with the intermetallics. This was due probably to the close relation between the densities of titanium, the intermetallics and Al_2O_3, a situation than helps atomic movement during the sintering.

Hardness

With respect to the hardness results, from Table 1 it can be seen that for all the systems monolithic Al_2O_3 is the hardest material. All the composite materials present hardness values between 18 and 19 GPa that are less than the almost 21 GPa reported for monolithic Al_2O_3. This is logical because a ceramic material has to be harder than the same ceramic material with the incorporation of ductile phases in its bulk volume.

Fracture Toughness

From Table 1 and Fig. 2 it can be observed that in all the composite cases the fracture toughness of monolithic Al_2O_3 was improved considerably, principally in composites reinforced with Ni and Ti and in all composites reinforced with intermetallic phases. The incorporation of ductile metal particles in the ceramic matrix enhances the fracture toughness due to plastic deformation of the metallic phase, which forms crack-bridging ligaments when a crack grows in the material under a tensile stress action. In other words, the energy absorbed for plastic deformation is unavailable for crack extension. Additionally, the deformed particles could bridge the faces of the crack wake, thereby exerting closure stresses, reducing the effect of the stress intensity at the crack tip [14-15].

For the case of Al_2O_3/Ni system: nickel provides a liquid phase during the sintering stage that promotes diffusion and therefore densification of the composite On the other hand, Ni helps to refine the alumina microstructure by pinning its grain boundaries and thereby restraining the grain growth of alumina.

For the case of the Al_2O_3/Ti system: because the densities of titanium and alumina are very similar, Ti is well dispersed in the alumina matrix, forming a good homogeneous composite microstructure that promotes diffusion and densification, and as a consequence good toughening of the final material.

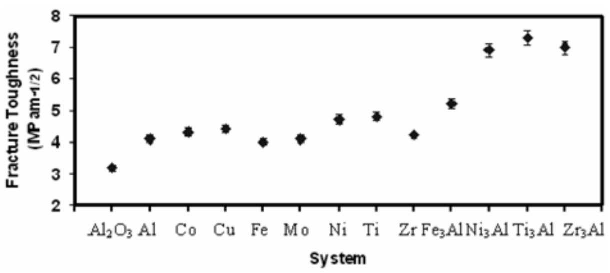
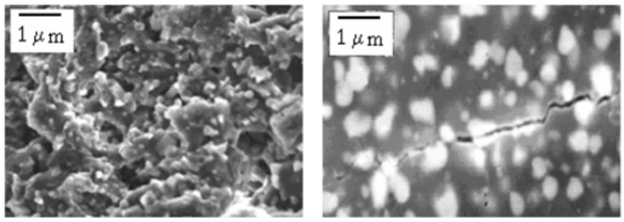


Fig. 2. Fracture toughness values measured for all the composites investigated.



(a) (b)

Fig. 3. (a) Fracture surface and (b) advanced of a crack in an Al_2O_3/Ti reinforced composite.

For the case of Al_2O_3/intermetallics systems: the use of intermetallics as reinforcement in Al_2O_3 gives an appreciable enhancement in the fracture toughness, this is due to the good ductility, low density and chemical compatibility of intermetallics with alumina. These factors help to obtain homogeneous microstructures with the formation of interfaces that allow the activation of different diffusion mechanisms thus improving the final density and then the mechanisms that improve the fracture toughness of the composites.

Figs. 3(a) and (b) show the fracture surface and the advance of a crack in an Al_2O_3/Ti reinforced composite. The fracture mode in Fig. 3(a) corresponds to microvoid coalescence as suggested by the dimple-like depressions that are typical of ceramic materials. From Fig. 3(b) it can be observed that the sample exhibits a mixed fracture mode, because metallic particles bridge the surface of the crack in the composite, but at the same time they can cause deflection of the crack. So the toughening mechanism in Al_2O_3/metal reinforced composites is due to crack bridging and crack deflection in this type of material. Steinbrech has reported that the improvement achievable in reinforced composites is governed by the mechanical properties of the ductile material, ligament diameter, volume fraction of the components, interfacial properties and the reaction products of the constituents [16]. This can explain the differences obtained in the fracture toughness of the materials investigated here.

Conclusions

Al_2O_3-based composites reinforced with different metals have been fabricated by both; liquid and solid pressureless sintering of an intensive mechanical mixture of powders. By the use of ductile particles in a hard ceramic matrix, significant improvements in fracture toughness due to plastic deformation of the metallic phase has been obtained. However, there are metals that enhance the toughness of a ceramic better than others; these are those metals that have similar densities to alumina, because they help to obtain fine and homogeneous microstructures after sintering. From the fracture toughness measurements and microstructural observations, finally it can be commented that the toughening mechanism in Al_2O_3/metal reinforced composites is due to crack bridging and crack deflection.

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