

Strengthening of Alumina-Based Ceramics with Titanium Nanoparticles

Sergio J. Esparza-Vázquez, Enrique Rocha-Rangel, José A. Rodríguez-García, Constantin A. Hernández-Bocanegra

Polytechnic University of Victory, Victory City, Mexico Email: <u>erochar@upv.edu.mx</u>

Received 15 March 2014; revised 21 April 2014; accepted 3 May 2014

Copyright © 2014 by authors and Scientific Research Publishing Inc. This work is licensed under the Creative Commons Attribution International License (CC BY). http://creativecommons.org/licenses/by/4.0/

Abstract

In this study ceramic materials with a matrix of Al_2O_3 strengthened with different amounts of Ti nanoparticles (0.0 wt%, 0.5 wt%, 1.0 wt%, 2.0 wt% and 3.0 wt%) were generated. High energy milling was used to mix the materials in a planetary mill type, in which powder particles were obtained with sizes of ~300 nm. These powders were uniaxially compacted in cylindrical samples using 350 MPa pressure. These samples were sintered at 1500°C for 1, 2 and 3 h and at 1400°C, 1500°C and 1600°C during 2 h. Microstructure observations were made with optical microscopy and scanning electron microscopy. Dense composites were identified with a homogeneous distribution of fine particles. Concerning the measurement results of fracture toughness, which were estimated by the indentation fracture method, it was shown that the composites made by mean procedure present higher values than the average of the monolithic alumina, up to 200%. Photographic evidence of arrest of crack growth by titanium particles was obtained, demonstrating that the reinforcement mechanism of these materials is due to the deflection of cracks owing to metallic bridges formed by the titanium used as alumina strengthener.

Keywords

Al₂O₃, Strengthened, Ti Nanoparticles, Fracture Toughness

1. Introduction

Ceramic materials possess favorable mechanical and physical properties; however, their applications have been limited due to their fragility or low fracture toughness [1]. Alumina is a very useful ceramic because of its wide variety of applications [2]. Its use is mainly due to its high hardness and compression strength as well as high electrical and thermal insulation properties. Furthermore, its uses range from the manufacture of cutting tools

How to cite this paper: Esparza-Vázquez, S.J., Rocha-Rangel, E., Rodríguez-García, J.A. and Hernández-Bocanegra, C.A. (2014) Strengthening of Alumina-Based Ceramics with Titanium Nanoparticles. *Materials Sciences and Applications*, **5**, 467-474. <u>http://dx.doi.org/10.4236/msa.2014.57050</u>

and medical implants to the manufacture of insulating materials both electrical and thermal. However, due to their low resistance to fracture, which is a common feature of all ceramic materials, their utility has not been expanded [3]. In order to reduce the brittleness of alumina, other studies have prepared different matrices of alumina ceramics strengthened with metallic particles, and demonstrated that the size and uniform distribution of the particles in the ceramic matrix contributed to obtain composites with good mechanical properties, and especially increased fracture toughness [4]. Some of the studied systems were: Al₂O₃-Al [5], Al₂O₃-Cr [6], Al₂O₃-Cu [7], Al₂O₃-Ni [8], Al₂O₃Ag [9] and Al₂O₃-Ti [10]. One of the main problems of the previously mentioned systems is that sophisticated processing techniques are employed for the production of composites, which involves expensive and often few productive processes. The powder synthesis method for the production of composites materials uses a high energy ball mill combined with pressureless sintering. It is a low-cost method in which a good sintered product can be obtained. The goal of this study is the synthesis of alumina-based composites reinforced with titanium nanoparticles by the combination of high energy milling and pressureless sintering in order to determine the effect of titanium on the fracture toughness of the ceramic matrix.

2. Experiment

Al₂O₃ (Mayer, 99.9% purity, 5 µm), and Ti (Sigma-Aldrich, 99.9% purity, 5 µm) powders were used in this study. Five systems were studied, being Al₂O₃ the principal component, whereas the content of Ti varied according to the following contents: (0.0 wt%, 0.5 wt%, 1.0 wt%, 2.0 wt% and 3.0 wt%). These compositions were processed by high energy milling using a planetary mill type (Retsch, PM100, Germany) with ZrO₂ grinding elements of 0.3 cm diameter and a cylindrical stainless steel vessel with a volume of 250 ml. The milling was carried out for 3 hours at 250 rpm in dry. The powder weight/ball weight ratio was 1:14, using the volume of 1/3 of the container. The granulometry of the powders resulting from the grinding is determined with the aid of granulometric equipment (Malvern Instruments, Masterziser 2000, UK). Cylindrical samples of each composition with 2 cm and 0.3 cm of diameter and thickness respectively, were compacted using 350 MPa pressure, with the aid of a uniaxial press (Montequipo, LAB-30-G, Mexico). The green samples were sintered in an electric furnace (Carbolite RHF17/3E, UK) at 1500°C for 1, 2 and 3 hours and at 1400°C, 1500°C and 1600°C for 2 hours. In all cases, a heating rate of 5°C/min and a volumetric flow of argon gas of 10 cm³/min in the furnace combustion chamber were employed. To measure the density of the sintered samples, the Archimedes' method was used. The fracture toughness was estimated by the indentation fracture method, using a Vickers microhardness tester (Wilson Instruments, S400) and applying Evans and Charles equation [11]. To observe the microstructure, an optical microscope (Nikon Eclipse, MA200, Japan), and a scanning electron microscope (SEM-JEOL, Japan 6300) were used. The SEM is equipped with an energy dispersive detector X-rays (EDX) (Hitachi, UHR FE-SEM SU9000, Japan).

3. Analysis and Results Discussion

3.1. Granulometry

Powders with very small particle sizes up to 300 nm were obtained from the high energy ball milling. The formation of agglomerates of several microns was also present. These were generated by the large electrostatic surface forces exerted between the fine powders obtained during the grinding. The particle size distribution for different samples is shown in **Figure 1**. In this figure it can be observed that in all samples over 50% of the size of the powders is less than 1 micron, which proves that nanometric sizes are mainly obtained in the powder during the milling step. This fact should favor densification of the samples due to the numerous contacts existing between particles in all compositions.

Figure 2 shows micrographs of powders obtained for samples with different amounts of titanium. In this figure it can be observed in all cases the presence of very fine powders with sizes less than $1\mu m$, the presence of agglomerates consisting of very small particles can also be seen in this figure.

3.2. Density

Results of the relative density measurement of the sintered samples are observed in Figure 3. In samples sintered at 1500° C during 2 h (Figure 3(a)) with contents of 0.5% and 1.0% the highest values were obtained, while for greater amounts of Ti there was a downward trend in the density of the composites. These results agree



Figure 1. Particle size distribution for different samples of study.



Figure 2. Micrographs of the powders after milling.

with those of granulometry, which show a greater presence of fine particles in the samples with 0.5% and 1.0% Ti. This favors compaction of the powders and therefore, a homogeneous grain growth during the sintering. In samples sintered for 2 h with a variation of the temperature (**Figure 3(b)**), the positive effect of the inclusion of Ti with respect to monolithic alumina is observed, attaining the highest value of density at 1500°C. Regarding the samples sintered at 1500°C for different times 1, 2 and 3 h (**Figure 3(c)**), the positive effect of Ti is similarly observed up to 1500°C, and later density tends to decrease. It can be said that, temperatures of 1600°C and timing of 3 h sintering are not suitable for the consolidation of the samples probably due to a significant grain growth in their microstructures, which also generates a lower elimination of porosity. Moreover, temperatures of 1400°C and timing of 1 h sintering are not favorable conditions for consolidating the samples; in this case the distribution is not large enough to achieve the densification of the samples. Therefore, the systems with 0.5 wt% and 1 wt% Ti sintered at 1500°C during 2 h are the ideal for obtaining better densified samples, since under these conditions almost 99% of the relative density of the samples is reached.





3.3. Microstructure

Figure 4 shows micrographs obtained by optical microscope (OM). The micrographs presented here correspond to samples sintered at 1500°C during 2 h, for different contents of Ti in the composite. It may be observed that the grain growth is greater with increments of titanium content. Samples with 0.5 wt% and 1.0 wt% Ti show a microstructure with good distribution and uniform grain size, due to the good thermal conductivity of Ti that adequately dissipates a portion of the thermal energy generated during sintering; thereby contributing to obtaining the good values of densification showed in the previous section. The opposite occurs with the samples with 2 wt% or 3 wt% Ti content, where very large grain sizes in the microstructures are observed. This situation is due to the significant energy absorption by titanium particles, energy which is consumed by the sample through the grain growth.

Figure 5 shows images of the microstructure of the sintered samples at 1500°C for 1, 2 and 3 hours, and sintered for 2 hours at 1400°C, 1500°C and 1600°C taken with the help of a scanning electron microscope. In this case, it is possible to see (observe figure from left to right) that the grain growth is more significant with the temperature increase than when the sintering time is increased. These results agree with those shown in the density section, where the density of the samples tends to decrease when they are sintered at 1600°C during 3 h, due to grain growth in greater proportion.

Microstructures observed using a scanning electron microscope (SEM) of samples sintered at 1500°C for 2



Figure 4. Micrographs of the sintered samples at 1500°C during 2 hours.



Figure 5. Photomicrographs corresponding to the 0.0 and 0.5% Ti sintered samples, varying time and temperature.

hours are shown in **Figure 6**. The sample without inclusions of Ti has an uncontrolled growth of grain sizes greater than 20 μ m. Regarding samples with Ti inclusions, a more uniform size and better grain distributed with 0.5 wt% and 1.0 wt% Ti is observed. As the Ti amount in the sample increases, grain size is around 10 μ m, which matches very well with the results of relative density of the sintered samples and observations with optical microscope. **Figure 7** shows typical results of microanalysis performed with X-rays energy dispersive (EDX) executed in a punctual manner in light and dark particles of the microstructure in samples with 0 wt% and 0.5 wt% Ti. For the case of the sample with 0 wt% Ti, obtained spectra indicates the presence of oxygen and aluminum in the sample, feature that corresponds to the composition of the ceramic matrix (Al₂O₃). In the case of the sample with 0.5 wt% Ti SEM photomicrographs shown in **Figure 6**, the presence of two phases is shown, in accordance with the spectra obtained by EDX. The darkest phase corresponds to the ceramic matrix, and the lighter phase belongs to Ti present in the sample. Thus, it can be seen that Ti particles are located in intergranular positions and also have a much smaller size compared to the Al₂O₃ grains.

3.4. Fracture Toughness

In Figure 8, the values of fracture toughness as a function of Ti content in the samples, as well as the temperature and time of sintering are shown. In the samples with different contents of Ti and sintered at 1500° C for 2 h (Figure 8(a)), in relation to fracture toughness positive values up to 200% are achieved compared to the monolithic alumina. This increase is even greater in samples with 0.5% and 1.0% Ti. These results are consistent with the results of the density measurements and microstructure observations, where it is shown that for the samples with 0.5% and 1.0% Ti the controlled grain growth and the homogeneous distribution of Ti accentuate the obtainment of dense bodies with homogeneous microstructures, conditions that facilitate the strengthening of alumina by the titanium. For samples with inclusions of 0.0% and 0.5% Ti sintered for 2 h as a function of temperature (Figure 8(b)), a considerable effect of Ti inclusions and of the temperature on fracture toughness is observed due to the 1600°C at which the highest value of this property is attained. The sintered samples at 1500°C as a function of sintered time (Figure 8(c)) show increases in the fracture toughness. Although, these have inferior toughness results compared with those obtained when the temperature was varied.

In Figure 9, two indentations obtained during microhardness testing of samples with 0.0% and 0.5% Ti sintered for 2 hours at 1500°C are observed. The figure on the left side has a mark obtained in the sample with 0% Ti, where the growth of a crack in a linear path which apparently spreads easily based on its size can be



Figure 6. Micrographs of the sintered samples at 1500°C.







Figure 8. Fracture toughness of the composites Al₂O₃-Ti as a function of: (a) titanium content, (b) sintering temperature and (c) sintering time.



Figure 9. Fracture toughness of the Al₂O₃-Ti composites

observed. In the sample with 0.5% Ti inclusion (right side), it can be seen that the crack generated at the apex of the mark spreads and stops when it encounters a particle of Ti, so that it may be concluded that in the Al_2O_3 -Ti system when a crack grows and hits a particle of Ti, the ductility and plastic deformation of the metal inhibits the growth of the crack or promotes its search for another propagation path, causing a higher demand of energy for the crack to grow, resulting in an increase in fracture toughness of the material, explaining the strengthening of Al_2O_3 by means of Ti.

4. Conclusions

- Through the processing methodology proposed, dense alumina-based composites strengthened with Ti nanoparticles were obtained.
- The fracture toughness of the Al₂O₃ was improved up to 200% with the reinforcement of the same by means of Ti nanoparticles homogeneously distributed in the ceramic matrix.
- The strengthening mechanism of Al₂O₃ is due to the crack deflection caused by the presence of ductile Ti particles present in intergranular zones of the composite's microstructure.

Acknowledgements

Authors would like to thank to CONACyT, for its support for the accomplishment of this research work through Project 132406.

References

- Auerkari, P. (1996) Mechanical and Physical Properties of Engineering Alumina Ceramics. Technical Research Centre of Finland, VTT Manufacturing Technology, Research Notes 1792, 3-36.
- Wessel, J.K. (2004) The Handbook of Advanced Materials. John Wiley & Sons Ltd., New York. <u>http://dx.doi.org/10.1002/0471465186</u>
- [3] Shackelford, J.F. and Doremus, R.H. (2010) Ceramic and Glass Materials: Structure, Properties and Processing. Springer, Berlin.
- [4] Ighodaro, O.L. and Okoli, O.I. (2008) Fracture Toughness Enhancement for Alumina Systems: A Review. *Internation*al Journal of Applied Ceramic Technology, **5**, 313-323. <u>http://dx.doi.org/10.1111/j.1744-7402.2008.02224.x</u>
- [5] Konopka, K. and Szafran, M. (2006) Fabrication of Al₂O₃-Al Composites by Infiltration Method and Their Characteristics. *Journal of Materials Processing Technology*, **175**, 266-270. <u>http://dx.doi.org/10.1016/j.jmatprotec.2005.04.046</u>
- [6] Marci, C. and Katarzyna, P. (2007) Processing, Microstructure and Mechanical Properties of Al₂O₃-Cr Nanocomposite. *Journal of the European Ceramic Society*, 27, 1273-1277. <u>http://dx.doi.org/10.1016/j.jeurceramsoc.2006.05.093</u>
- [7] Miranda, J.G., Soto, A.B. and Rocha, E. (2006) Production and Characterization of Al₂O₃-Cu Composite Materials. *Journal of Ceramic Processing Research*, 7, 311-314.
- [8] Lieberthal, M.I. and Kaplan, K. (2001) Processing and Properties of Al₂O₃ Nanocomposites Reinforced with Sub-Micron Ni and NiAl₂O₄. *Materials Science and Engineering*, A302, 83-91.

http://dx.doi.org/10.1016/S0921-5093(00)01358-7

- [9] Lalande, J., Scheppokat, S., Jansen, R. and Claussen, N. (2002) Toughening of Alumina/Zirconia Ceramic Composites with Silver Particles. *Journal of the European Ceramic Society*, 22, 2165-2168. <u>http://dx.doi.org/10.1016/S0955-2219(02)00031-6</u>
- [10] Travirskya, N., Gotmanb, I. and Claussen, N. (2003) Alumina-Ti Aluminide Interpenetrating Composites: Microstructure and Mechanical Properties. *Materials Letters*, 57, 3422-3426. <u>http://dx.doi.org/10.1016/S0167-577X(03)00090-9</u>
- [11] Evans, A.G. and Charles, E.A. (1976) Fracture Toughness Determination by Indentation, *Journal of the American Ceramic Society*, **59**, 371-372. <u>http://dx.doi.org/10.1111/j.1151-2916.1976.tb10991.x</u>