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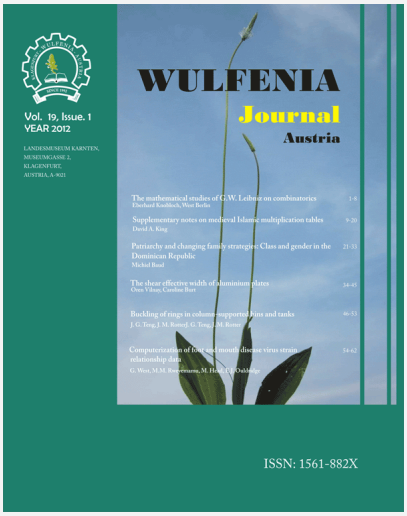


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# Ceramic Matrix Composites Reinforced with Metal Nanoparticles

**E. Refugio-García[1], J. Osorio-Ramos[1], E. Rocha-Rangel[2], S. Esparza-Vázquez[2], A. Pérez-de la Fuente[2], J. A. Rodríguez-Garcia[2] and J. G. Miranda-Hernández[3]**

[1]Departamento de Materiales, Universidad Autónoma Metropolitana, Av. San Pablo 180, Col. Reynosa-Tamaulipas, México, D. F., 02200

[2]Universidad Politécnica de Victoria, Avenida Nuevas Tecnologías 5902, Parque Científico y Tecnológico de Tamaulipas, Cd. Victoria, Tamaulipas, México, 87138

[3]Universidad Autónoma del Estado de México (UAEM-Valle de México), IIN, Blvd. Universitario S/N, Predio San Javier, 54500, Atizapán de Zaragoza, Edo. Mex., México.

Correspondence should be addressed to Enrique Rocha-Rangel; erochar@upv.edu.mx

**Abstract**

This project describes the formation of $Al_2O_3$-Ag or $Al_2O_3$-Ti nanocomposites via high-energy planetary milling combined with pressureless sintering. From the main results, it was found that after milling, the powder was constituted by very fine particles ~ 200 nm average size, and it presents a good distribution of metal and ceramic particles. Observations of microstructure of sintered samples by electron microscopy, illustrate dense composite materials with a homogeneous sharing of the metal in the alumina-matrix. The microstructure of the composites with silver is much finer than the microstructure of the composites with titanium. From fracture toughness measurements, estimated by the fracture indentation method, it has been determined that metal additions improved fracture toughness even more than 50 % with respect to the fracture toughness of monolithic-alumina. In conclusion, composites of the $Al_2O_3$-Ag or $Al_2O_3$-Ti systems are materials that have excellent mechanical properties which may result in their commercial use. Additionally, the proposed route for the manufacture of these composites can be a low-cost method.

## 1. Introduction

Alumina ($Al_2O_3$) is a ceramic material that possesses excellent mechanical properties such as: high hardness, high compressive strength, high elastic modulus and its chemically and thermally stable. However, as all ceramic materials, $Al_2O_3$ is fragile because cracks easily propagate in it; therefore they might unpredictably fail in service [Auerkari, 1996; Wessel, 2004; Shackelford et al., 2010]. The incorporation of several reinforcement materials such as ceramics, metals and intermetallics compounds into an $Al_2O_3$-matrix forming a ceramic-composite material has been proved to be an effective experimental route to improve toughness of the $Al_2O_3$ [Ighodaro, 2008]. Particularly, $Al_2O_3$ ceramics can be toughened with the incorporation of fine inclusions; for this reason, different $Al_2O_3$ systems for instance: $Al_2O_3$/TiC, $Al_2O_3$/ZrO2, $Al_2O_3$/SiC and $Al_2O_3$/AlN have been successfully fabricated through different techniques such as; spark plasma sintering, reactive sputtering and related deposition methods, hot pressing, and thermal spray processing. The main problem is that all these processes are expensive, have low productivity and they are complex in their procedures and control. Alternatively, due to the increasing developments in ceramic synthesis, new and innovative ceramic-metal composites have been introduced for many engineering applications. Ceramic-composites such as of the binary systems; $Al_2O_3$-Al [Konopka et al., 2006], $Al_2O_3$-Cu [Miranda et al., 2006], $Al_2O_3$-Cr [Marci, et al., 2007], $Al_2O_3$-Fe, $Al_2O_3$-Ni [Lieberthal, et al, 2008], have been reported to combine many of the best qualities of the ceramic and of the metal. Those materials maintain their strength

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when exposed to high temperatures, in where the best super alloys available today would deteriorate. In addition, ceramic-metal composites have enhanced strength and toughness due to plastic deformation of the metallic phase, which forms crack-bridging ligaments in the ceramic-matrix [Lalande et al., 2002; Travirskya et al., 2003]. The powder synthesis method for the production of composite materials uses a high energy planetary mill combined with pressureless sintering; it's a simple and low-cost method in which a good sintered product can be obtained. The goal of this study is the synthesis of $Al_2O_3$-based composites reinforced with titanium or silver nanoparticles by the combination of high energy milling and pressureless sintering. As well as: determine the effect of titanium and silver additions on the fracture toughness of the ceramic matrix.

## 2. Materials and Methods

In this study they were fabricated $Al_2O_3$/Ti and $Al_2O_3$/Ag composites, for that $Al_2O_3$ (Sigma-Aldrich, 99.9 % purity, 1-2 μm), Ti (Sigma-Aldrich, 99.9 % purity, 1 μm) and Ag (Sigma-Aldrich, 99.9 % purity, 1 μm) powders were used. Five systems of each composite were studied, being $Al_2O_3$ the principal component, whereas the content of Ti or Ag has varied according to the following contents: (0.0, 0.5, 1.0, 2.0 and 3.0 vol. %). These compositions were processed by high energy milling by means of a planetary mill type (Retsch, PM100, Germany) using YZT grinding elements of 0.3cm diameter and a cylindrical stainless steel vessel. The milling was carried out during 3 hours at 300 rpm in dry. The powder weight/ball weight ratio was 1:14. The granulometry of the powders resulting from the grinding was determined with the help of a granulometric apparatus (Malvern Instruments, Masterziser 2000, UK). After milling, for all compositions with the aid of a uniaxial press (Mont Equipo, LAB-30G, Mexico) and using 350 MPa pressure, they were manufactured cylindrical samples of 2 cm diameter and 0.3 cm thickness. The green samples were sintered in an electric furnace (Carbolite RHF17/3E, UK) at 1500°C during 1, 2 and 3 hours and at 1400 °C, 1500 °C and 1600 °C during 2 hours. In all cases, a heating rate of 5 °C/min and a volumetric flow of argon of 10 $cm^3$/min in the furnace chamber were employed. The Archimedes' method was used to measure density of the sintered samples. The fracture toughness was estimated by the indentation fracture method, using a Vickers microhardness tester (Wilson Instruments, S400) and applying Evans and Charles equation [Evans et al., 1976]. To observe the microstructure of sintered composites, 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. Results and Discussion

### *3.1Granulometry*

For both $Al_2O_3$/Ti and $Al_2O_3$/Ag mixtures; powders with very small particle sizes up to 200 nm were obtained from the milling stage. As a consequence the large electrostatic surface forces exerted between the fine powders obtained during the grinding they were formed agglomerates of several microns. The particle size distribution for different compositions of the two studied systems 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 lower than 1 micron, which proves that nanometric sizes are mainly obtained in the powder during the milling step. With respect to the specific surface area, the measured average was 37.48 and 38.21 $m^2$/g for the mixtures with titanium and silver respectively. This fact should favor samples' densification due to the numerous contacts existing between particles for all compositions.

In Figure 2 it is show micrographs that illustrate morphology and size of powders obtained after milling, for samples with different amounts of titanium and silver. In this figure it can be observed in all cases the presence of very fine powders with sizes less than 1μm, also the presence of agglomerates

of various sizes formed with very fine powders. The morphology of powder is round for the $Al_2O_3$/Ti systems, whereas for the $Al_2O_3$/Ag systems it has flake form.

*3.2 Density*

The Results of the relative density measurements of the sintered samples are presented in Figure 3. For the samples with contents of 0.5 vol. % and 1.0 vol. % of Ti or Ag, sintered at 1500 °C during 2 h (Figure 3a) highest values of density were obtained, while for greater amounts of the corresponding metal, there was a downward trend in the density of the composites. These results agree with those of granulometry, which show a greater presence of fine particles in the samples with 0.5 vol. % and 1.0 vol. % of Ti or Ag. 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 3b), 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 3c), 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 less elimination of porosity. For the case in which it was added Ag to the alumina matrix, the major densities were reached when samples were sintered at highest temperatures (1600 °C) and times (3 h). In these cases, high times and temperatures sintering favors considerably densification of samples. This can due to the good heat transfer of silver that stimulate atoms' migration. Moreover, in both systems ($Al_2O_3$/Ti and $Al_2O_3$/Ag) 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.

*3.3 Microstructure*

Figure 4 shows micrographs obtained by optical microscopy. The micrographs presented here correspond to samples sintered at 1500 °C during 2 h, for different contents of Ti in the composite. In these pictures it may be observed that there is grain growth with the increments of titanium content. Samples with 0.5 vol. % and 1.0 vol. % 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 in the samples with 2 or 3 vol. % 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 micrographs obtained by optical microscopy. The micrographs presented here correspond to samples sintered at 1500 °C during 2 h, for different amounts of Ag in the composite. In contrast to the situation occurring in the samples with titanium, in this case no grain growth is observed with silver increments in the composite. By contrast, in all cases extremely fine and homogeneous microstructures are observed. These observations are in good agreement with the measurements of density.

Figure 6, shows images of the microstructure of the $Al_2O_3$/0.5 vol. % Ti sintered samples at 1500 °C during 1, 2 and 3 hours, and sintered for 2 hours at 1400, 1500 and 1600 °C detected with the help of an optical microscope. In this case, it is possible to see (observe figure from left to right) that the grain growth is more significant for increments in temperature than with increments in sintering time. 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 3h, due to grain growth in greater proportion.

Figure 7, shows images of the microstructure of the $Al_2O_3$/0.5 vol. % Ag sintered samples at 1500 °C during 1, 2 and 3 hours, and sintered for 2 hours at 1400, 1500 and 1600 °C taken with the help of an optical microscope. In this case, it is possible to see (observe figure from left to right) that there is not significant changes in the microstructure characteristics, because grain growth with the variations of time or temperature does not is appreciated. So it can be concluded that silver additions on an alumina matrix controls grain growth. This is because silver helps very well to dissipate adequately heat during the sinter of the samples.

Microstructures of the $Al_2O_3$/Ti system sintered at 1500 °C during 2 hours observed with a scanning electron microscope (SEM) are shown in Figure 8. The sample without additions of Ti presents an uncontrolled grain growth, because they are observed grain sizes greater than 20 μm. Regarding samples with Ti inclusions it is observed, a more uniform size grain and a better distribution of them in the matrix, this occurs principally in composites with 0.5 vol. % and 1.0 vol. % Ti. As increases the Ti amount in the sample, grain size is around 10μm, which matches very well with the results of relative density of the sintered samples and observations with optical microscope. Figure 9 shows typical results of microanalysis performed with X-rays energy dispersive (EDX) executed in samples with 0 % and 0.5 vol. % Ti in a punctual manner in the clear and dark particles of the microstructure. For the case of the sample with 0 vol. % Ti, the obtained spectrum indicates the presence of oxygen and aluminum in the sample, feature that corresponds to the composition of the ceramic matrix ($Al_2O_3$). In the case of the sample with 0.5 vol. % Ti from SEM photomicrographs shown in figure 8, the presence of two phases is observed, in agreement with the EDX spectrum. The darkest phase corresponds to the ceramic matrix, whereas the clearest phase corresponds to the 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 with the $Al_2O_3$ grains.

Microstructures of the $Al_2O_3$/Ag samples sintered at 1500 °C during 2 hours observed with a scanning electron microscope (SEM) are shown in Figure 10. The sample without additions of Ag presents an uncontrolled grain growth, because they are observed grain sizes greater than 20 μm. Concerning samples with Ag additions, in all cases it is observed, a more uniform size grain and a better distribution of them in the sample. Grain size is very small and around 1 μm. Figure 11 shows typical results of microanalysis performed with X-rays energy dispersive (EDX) executed in samples with 0 vol. % and 0.5 vol. % Ag in a punctual manner in the clear (small dots) and dark particles of the microstructure. For the case of the sample with 0 vol. % Ag, obtained spectrum indicates the presence of oxygen and aluminum in the sample, aspect that corresponds to the composition of the ceramic matrix ($Al_2O_3$). In the case of the sample with 0.5 vol. % Ag from SEM photomicrographs shown in figure 10, the presence of two phases is observed, in accordance with the EDX spectra. The darkest phase corresponds to the ceramic matrix, whereas the white dots correspond to the Ag present in the sample. Thus, it can be conclude that grain sizes of Ag are very small compared with $Al_2O_3$ grains, and therefore they are positioned in the intergranular positions of the matrix.

*3.4 Fracture toughness*

The values of fracture toughness as a function of Ti or Ag content in the composites are shown in Figure 12. In the sintered samples at 1500 °C during 2 h for samples with Ti additions (figure 12a), they are reached fracture toughness values up to 233 % compared to the monolithic alumina. This observation is true in samples with 0.5 vol. % and 1.0 vol. % Ti. For samples with 2 and 3 vol. % Ti, fracture toughness of the composites is around 190 % bigger than fracture toughness of pure alumina. On the other hand, when alumina is reinforced with 2 or 3 vol. % Ag, they are attained fracture toughness values up to 333 % compared to the monolithic alumina. Whereas, reached fracture toughness in samples with 0.5 or 1 vol. % Ag if about 233 % larger than fracture toughness of pure

alumina. These results are totally consistent with the results of the density measurements and microstructure observations, in where the controlled grain growth and the homogeneous distribution of the metal accentuate the obtainment of dense bodies with homogeneous microstructures, conditions that facilitate the strengthening of alumina by the corresponding metal. For samples with inclusions of 0.0 vol. % and 0.5 vol. % Ti or Ag sintered during 2h as a function of temperature (figure 12b), a considerable effect of sintered temperature on fracture toughness is observed because it is 1600 °C where it reaches the maximum value of toughness. The sintered samples at 1500 °C as a function of sintered time show increases in the fracture toughness. Although, these have inferior toughness results compared with those obtained when the temperature was varied. Contrary for the sample with silver, the density decreases considerably for these conditions.

In Figure 13 they are observed two indentations obtained during microhardness testing of samples with 0.0 vol. % and 0.5 vol. % Ti and sintered during 2 hours at 1500 °C. The figure on the left side has a mark obtained in the sample with 0 vol. % Ti, where it is observed the growth of a crack in a linear path which apparently spreads easily based on its size. In the sample with 0.5 vol. % 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 in this case the strengthening of $Al_2O_3$ by means of Ti. Sometime similar has to occur for samples reinforced with silver, where major improvements are also obtained in the fracture toughness.

## 4. Conclusions

- ✓ Through the processing methodology proposed, dense alumina-based composites strengthened with Ti or Ag nanoparticles were obtained.
- ✓ Silver inhibits alumina's grain growth due to adequate dissipation of heat during sinter.
- ✓ The fracture toughness of the $Al_2O_3$ was improved up to 233% with the reinforcement of the same by means of Ti nanoparticles homogeneously distributed in the ceramic matrix.
- ✓ The fracture toughness of the $Al_2O_3$ was improved up to 333% with the reinforcement of the same by means of Ag nanoparticles homogeneously distributed in the ceramic matrix.
- ✓ The strengthening mechanism of $Al_2O_3$ is due to the crack deflection caused by the presence of ductile metal particles present in intergranular zones of the composite´s microstructure.

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**Figures**

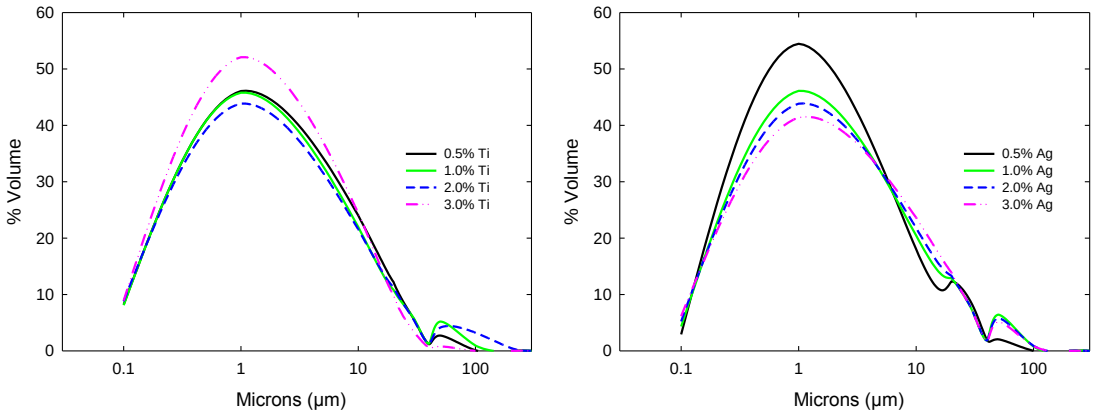


Figure 1. Graphs that show the particle size distribution for $Al_2O_3$/Ti and $Al_2O_3$/Ag powder mixtures.

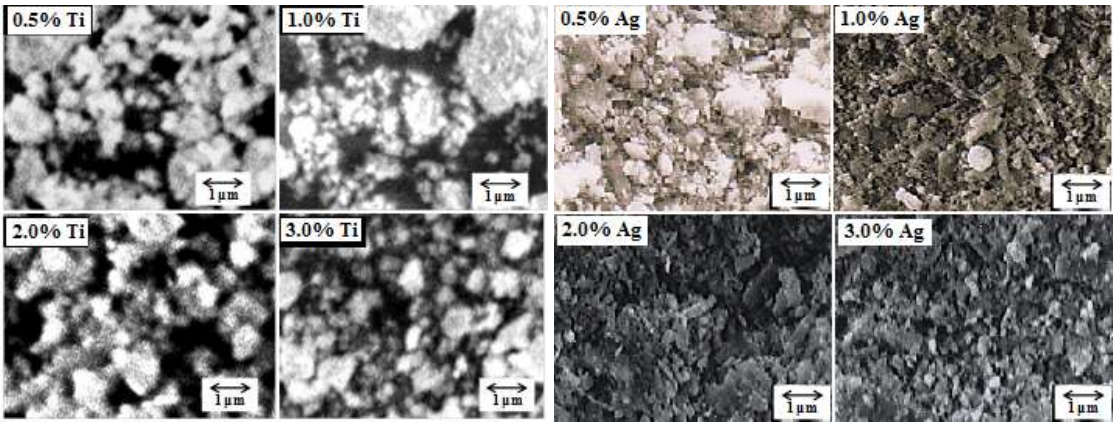


Figure 2. Micrographs of the $Al_2O_3$/Ti and $Al_2O_3$/Ag powder mixtures after milling during 3 hours at 300 rpm in a planetary mill.

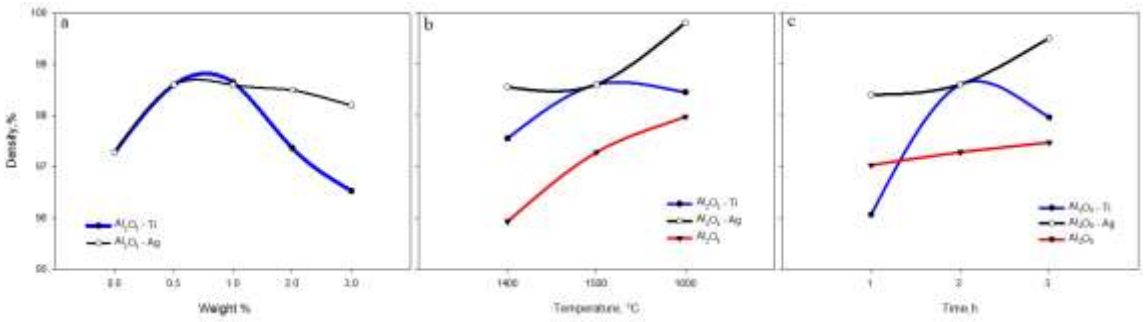


Figure 3. Relative density results for $Al_2O_3$/Ti and $Al_2O_3$/Ag studied systems after sinter at different times and temperatures.

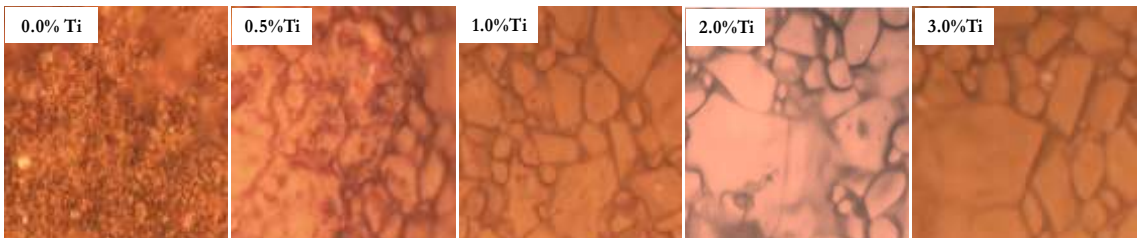


Figure 4. Optical micrographs of $Al_2O_3$/Ti samples sintered at 1500 °C during 2 h.


Figure 5. Optical micrographs of $Al_2O_3$/Ag samples sintered at 1500 °C during 2 h.

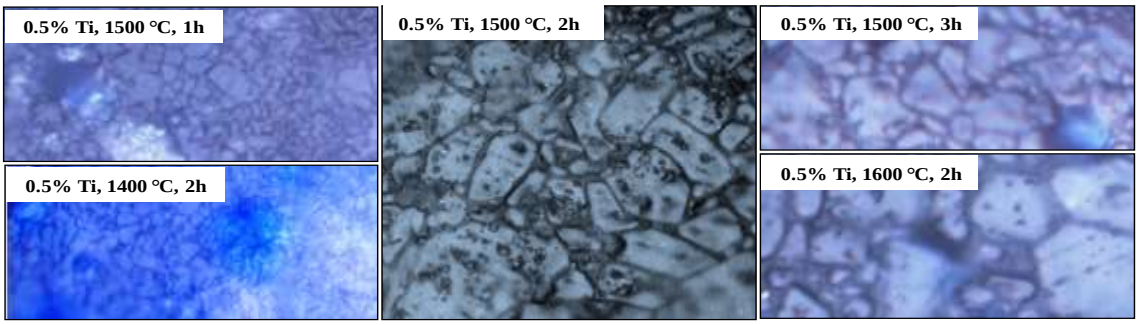


Figure 6. Optical photomicrographs of the $Al_2O_3$/0.5 vol. % Ti samples, sintered at different times and temperatures.

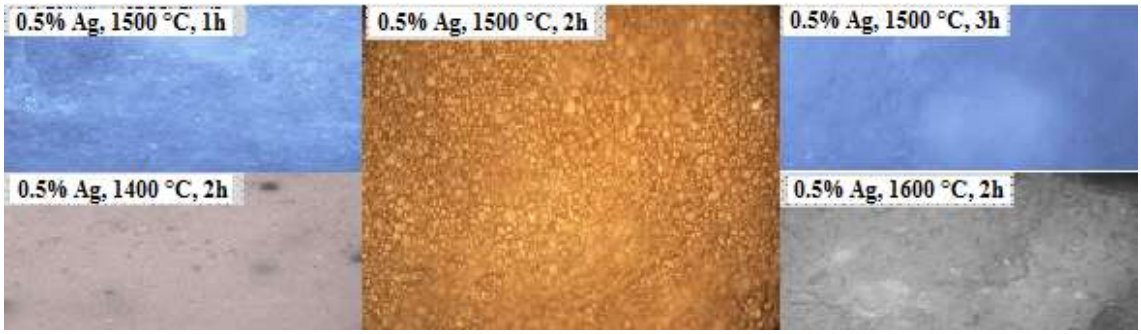


Figure 7. Optical photomicrographs of the $Al_2O_3$/0.5 vol. % Ag samples, sintered at different times and temperatures.

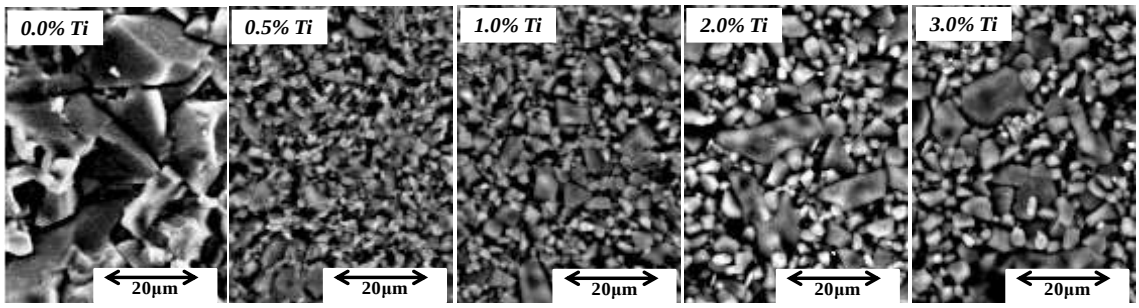


Figure 8. SEM micrographs of $Al_2O_3$/Ti system sintered at 1500 °C during 2 h.

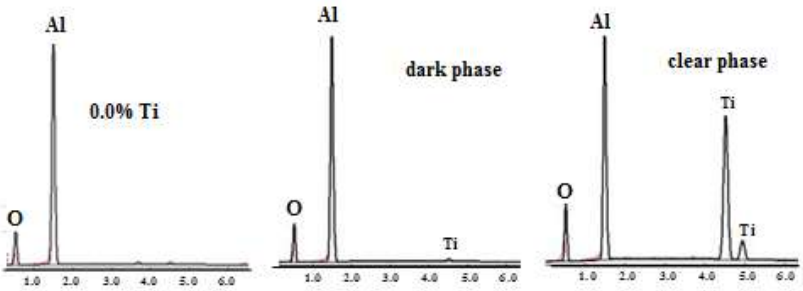


Figure 9. EDX spectra corresponding to $Al_2O_3$/0.0 vol. % Ti and $Al_2O_3$/0.5 vol. % Ti, samples, sintered at 1500 °C during 2 hours.

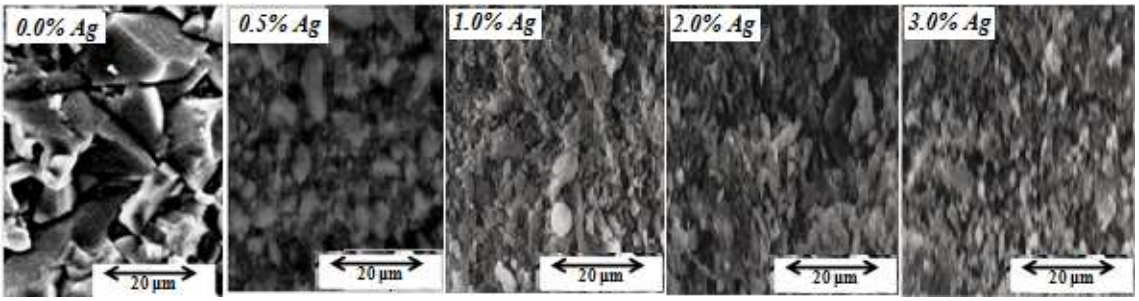


Figure 10. SEM micrographs of $Al_2O_3$/Ag samples sintered at 1500°C during 2h.

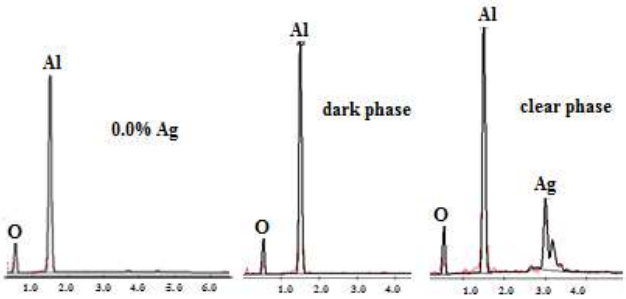


Figure 11. EDX spectra corresponding to $Al_2O_3$/0.0 vol. % Ag and $Al_2O_3$/0.5 vol. % Ag samples, sintered at 1500 °C during 2 hours.

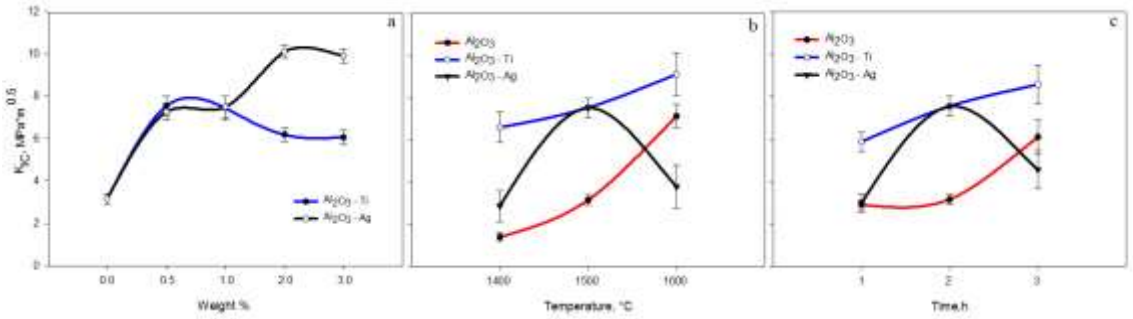


Figure 12. Fracture toughness results for $Al_2O_3$/Ti and $Al_2O_3$/Ag studied systems after sinter at different temperatures and times.

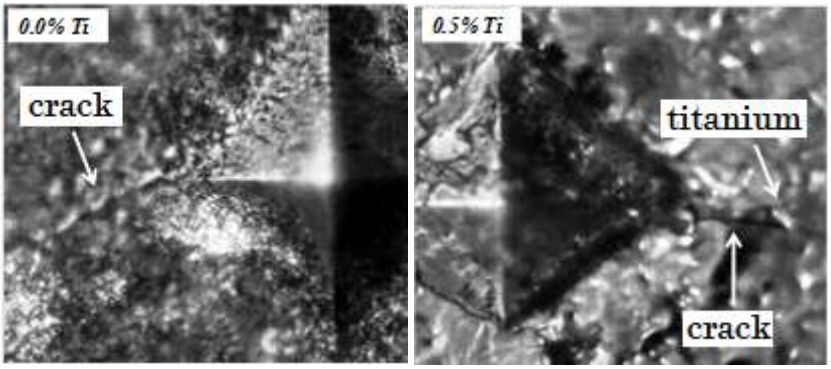


Fig. 13 - Fracture toughness of the $Al_2O_3$-Ti composites.