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Alumina-Based Composites Reinforced With Ductile Particles

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ABSTRACT: Al_2O_3/Metal composite ceramics were fabricated by the use of mechanical milling and pressureless sintering. Al_2O_3 + 10 vol.% of La, Mn, Si or Y were mixed and milled during 12 h at 300 rpm in a horizontal mill, then with the powder mixture it was conformed cylindrical samples by uniaxial pressing using 300 MPa. Pressed samples were sintered during 2 h in an electrical furnace at 1500°C. During sintered it was used an argon atmosphere inside the furnace in order to inhibit metal oxidation. XRD results indicate that alumina and metals retain its crystalline structure. Reached density by samples is small and less than 90%. Scanning electron microscopy observations show alumina's microstructure with very fine and homogeneous distributions of metal particles. Both the Mn and Si are not suitable metals to improve the mechanical properties of alumina, in particular the fracture toughness. Considered rare earth metals such as yttrium and lanthanum, have yielded favorable results in improving the fracture toughness of the alumina. However, it should be made more dense materials with them to better explore this potential.

Keywords – *Fracture toughness, Ductile particles, Reinforcement, Alumina, Composites*

I. INTRODUCTION

Alumina (Al_2O_3) ceramic presents good mechanical properties such as: high hardness, high compressive strength, good chemical and thermal stability. However, its applications as structural material have been limited principally by its low fracture toughness. Al_2O_3 ceramics can be toughened with the incorporation of fine metallic particles [1-6], for this reason it has been prepared successfully some Al_2O_3-systems by different techniques such as: pressure-assisted thermal explosion [6], slip casting [7], metal infiltration [8-10], sintering high pressing [11], Chemical deposition [12], self-propagation high temperature synthesis [13] and reaction synthesized [14]. Nevertheless, most of these processes are costly and they are very complex in their procedures and control. From these studies authors have been documented that fracture toughness of a ceramic-metal composite can be controlled by the volume fraction of metallic phase as well as the size of metal particles and its homogeneous distribution in the ceramic matrix. With these considerations in mind, a simple and cheaper process was developed for the production of ceramic-metal composites [15]. High-energy ball milling combined with pressureless sintering can be a substitute low-cost method for the production of ceramic-metal composite. The aim of this study is to synthesize Al_2O_3-based composites reinforced with 10 wt. % of La, Mn, Si or Y using powder techniques in order to determine the effect of each metal on the fracture toughness of the alumina-matrix. The contents of each section may be provided to understand easily about the paper.

II. EXPERIMENTAL

Starting materials were: Al_2O_3 powder (99.9 %, 1μm, Sigma-Aldrich, USA) and the following powder metals (La, Mn, Si and Y all of them with 99.9% purity, and sizes between 1-2μm, Sigma-Aldrich, USA). The used amount of powders is that one that allows at the end of the processing let to obtain the composite Al_2O_3-10 vol.% of the corresponding metal. The powders were milled and mixed in dry in a horizontal mill, using a rotation speed of 300 rpm, during 12 h, with the help of ceramic jars and using YSZ's balls as grinding elements, the relation; weight of powder/weight of balls was 1:25. With the powders mixture, they were made cylindrical samples by uniaxial pressing using 200 MPa with the following dimensions: 20 mm in diameter x 3 mm in thickness. Afterward, the pressed samples were pressureless sintered at 1500°C during two hours in an electrical furnace with gas argon atmosphere. The speeds of warming and of cooling were kept constant and equal to 10°C/min. The characterization of the synthesized products was of the following way: Densities were evaluated by Arquimedes' method. Crystalline phases present en each fabricated composite was analyzed by X-ray diffraction. The microstructure of the composites was observed with a scanning electron microscopy (SEM), this SEM was equipped with an energy dispersive spectroscopy detector (EDS), on the way to realize chemical analyses in the samples' microstructure. The elastic modulus was determined by the ultrasonic method.

Microhardness measurements were evaluated with the help of a Vickers indented; finally, fracture toughness was determined by the fracture indentation method, using Evan´s equation [16].

III. RESULTS

3.1 Relative density

The calculation results of the relative density achieved by each sample after sintering is shown in Fig. 1. To construct this figure it was first determined the theoretical density of each compound using the rule of mixtures, subsequently sintered density of each sample was evaluated by the method of Archimedes. With these two values, it could be calculated the density percentage attained for each sample after its sintering. Figure 1 shows that the density of the control sample was approximately 90 %, while densities reached by the samples with La, Mn, Si or Y additions, reached only values of 86, 84, 70 and 89 % respectively. Density values that are not very high, indicating that the diffusion process during the sintering step does not occurred satisfactorily. This could be due to sintering occurs in the presence of a liquid phase, formed by the fusion of the used metal in each case, and surely the liquid formed not wet the surface of the alumina, situation that difficult sintering of the composite.

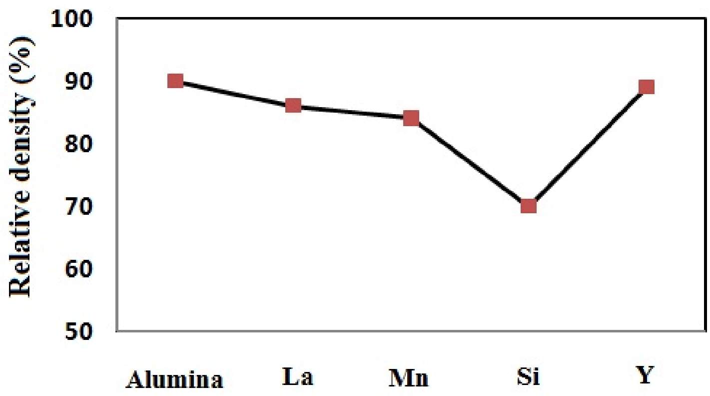

figure 1. relative density of different fabricated composites.

3.2 X-ray Diffraction

X-ray diffraction patterns of the four sintered composites are shown in Fig. 2. As seen in the corresponding pattern to each system, the diffraction spectrum shows just the presence of the alumina and the metal-added. The presence of any oxide, or other element or compound outside the initial composition of the composite is not observed, indicating that there was no contamination in any step of the process.

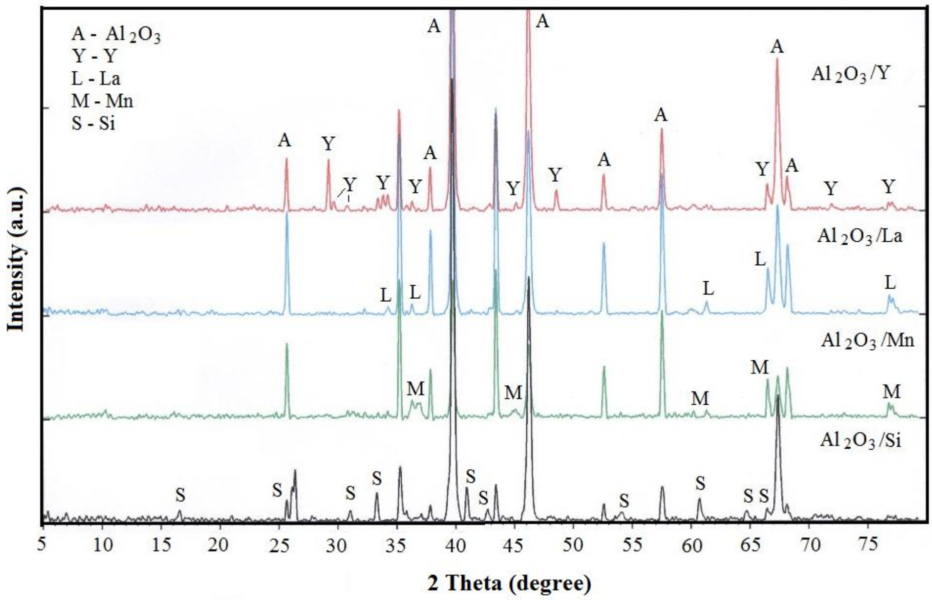

figure 2. x-ray diffraction patterns of the sintered composites.

3.3 Microstructure obsrved with optical microscope

Microstructure photographs taken with an optical microscope of each manufactured composite are shown in Fig. 3. Overall are observed fine microstructures with good dispersion of a clear and bright phase, which should be the added metal to each composite. While density measurements indicate the presence of more than 10% porosity in all samples, porosity is not evident in these micrographs. In the case of the sample with silicon, it is observed higher metal agglomeration in some parts of the matrix, which is due to the lower density of silicon (2.3 g/cm^3) in comparison to the density of the alumina (4 g/cm^3).

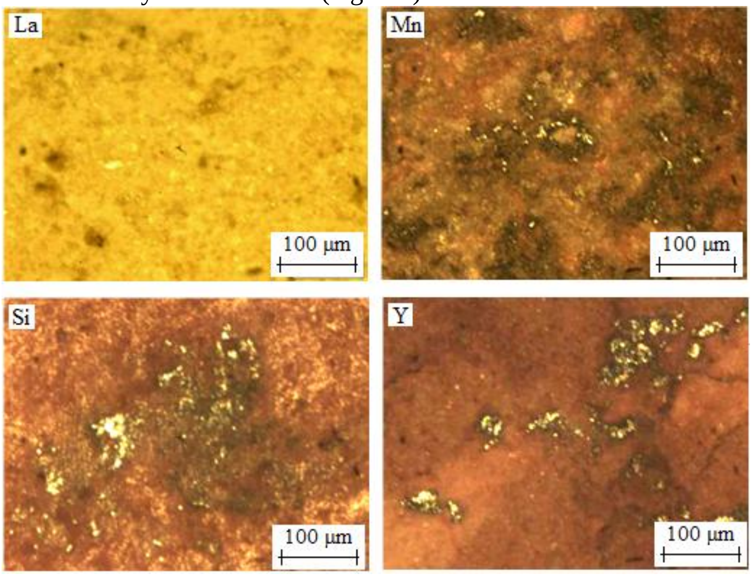

figure 3. optical microstructure of different manufactured composite.

3.4 Microstructure obsrved with scanning electron microscope

The microstructure of the sintered composite observed using a scanning electron microscope (SEM) is shown in Fig. 4. In the same figure 4, it is presented the resulting EDS spectrum realized in the bright particles of the microstructure of each sample. According to EDS patterns the metallic phase are the smaller and bright particles located mainly in intergranular positions. While the darkest phase corresponds to that of the alumina ceramic matrix. In all cases the grain size of the matrix is very similar and close to a micron, microstructure presents some homogeneity, although in these pictures the presence of porosity is clear and corresponds to the measures realized by the Archimedes' method. Although, the melting point of the metals used is below the sintering temperature, indicating that it should have a liquid phase formed during sintering, it is not observed traces of it in the microstructure.

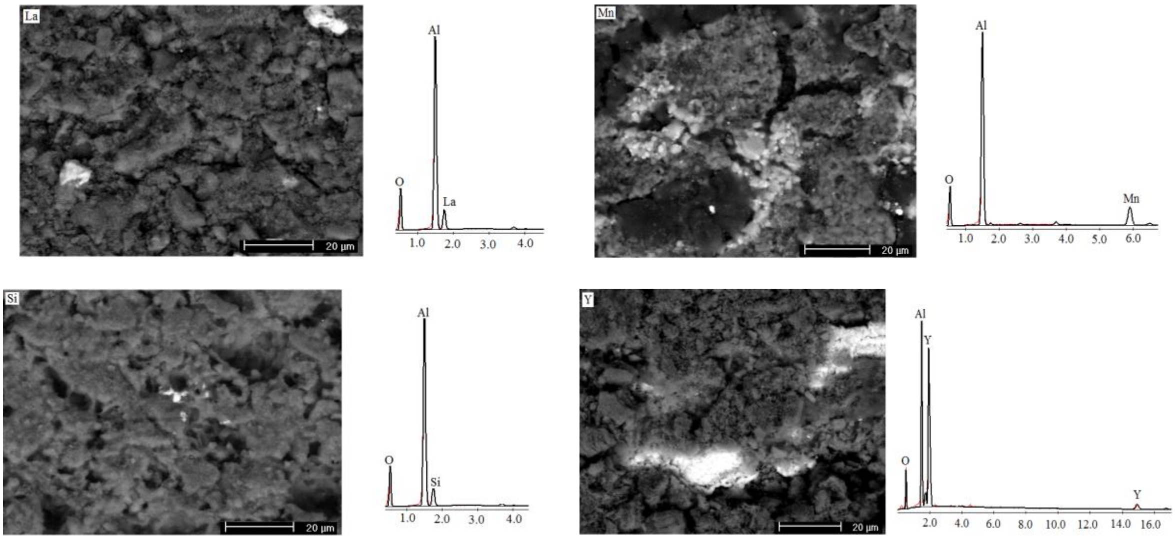

Figure 4. SEM microstructure of different manufactured composite.

3.5 Mechanical Properties

3.5.1 Young’s Modulus

The measurement results of Young's modulus for each composite are presented in Fig. 5. This figure shows that in all cases the value of Young's modulus observed for the control sample (monolithic alumina) is well above of the modules achieved by other samples. This is due to the higher density attained by the monolithic alumina. The sample with silicon has reached a very low densification; consistently it has a very small value of the elastic modulus.

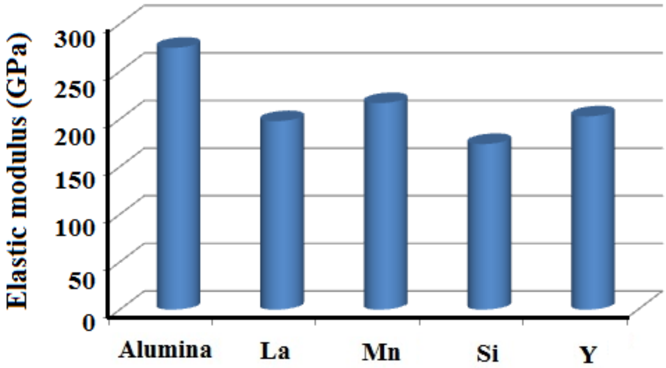

figure 5. young's modulus of sintered composites.

3.5.2 Microhardness

The results of the measurement of microhardness of each sintered sample are shown in Fig. 6. Again in this figure it is apparent that hardness of the pure alumina is well above than hardness of different composites. This result is logical and has two explanations, The first one is due to the greater degree of densification achieved by purely ceramic sample, the second is that when it is added a ductile metal, much less hard than the ceramic matrix, clearly, the final hardness of the resultant composite will be less. On the other hand, yttrium sample is the best densified, therefore it has a high value of microhardness.

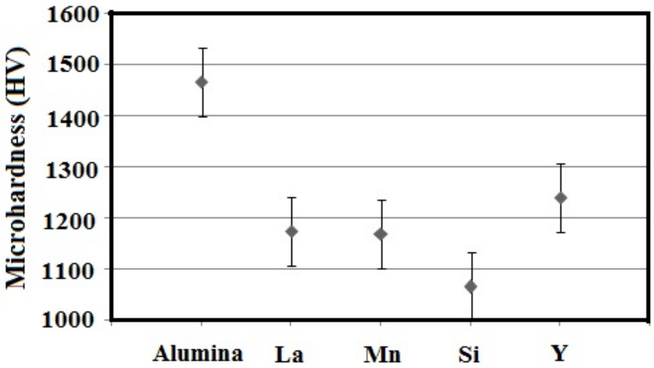

figure 6. microhardness of each sintered sample.

3.5.6 Fracture Toughness

Values of fracture toughness for each sintered composite are shown in Fig. 7. From this figure when it is considered the standard deviations for each case, is that the values of fracture toughness of the monolithic alumina and reinforced composites with silicon or manganese are very similar, so that it can be commented that nor silicon or manganese are good to improve the fracture toughness of the alumina. On the other hand, values of this property when lanthanum or yttrium is used as reinforcing there is an improvement of 13% and 16% respectively, although these values are not very significant, them support the hypothesis that this type of metals may be promising in the reinforcement of alumina. If it could be adjusted processing conditions to achieve higher densification in the fabricated composites when these metals are used, probably also the fracture toughness could increases significantly. Different authors have reported that the mechanism of enhancement of the alumina, by metals, is due to the closure of cracks through the metal tap when they tend to grow [17].

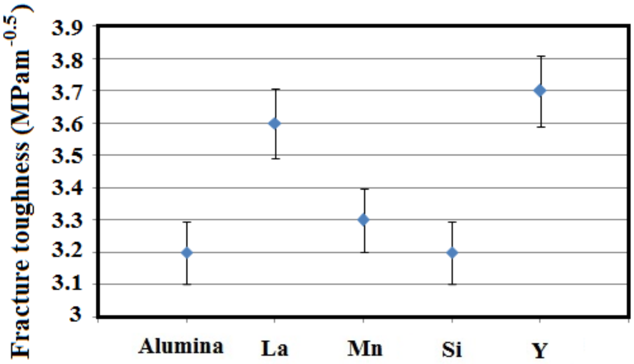

Figure 7. Fracture toughness of each fabricated composite.

IV. Conclusions

- Through the proposed methodology it could be fabricated alumina-based materials with additions of La, Mn, Si and Y with almost 90% of densification.
- Both the Mn and Si are not suitable metals to improve the mechanical properties of alumina, particularly fracture toughness.
- Rare earth metals such as yttrium and lanthanum have yielded favorable results in improving the fracture toughness of the alumina. However, it should be fabricated denser materials with them to better explore this potential.

V. Acknowledgements

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