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Improving Toughness of Alumina-Based Composites to Be Used As Bone Substitute

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Abstract:- In this study it is show the experimental results of alumina reinforced with titanium additions, forming a composite which by its components could be classified as a biomaterial. The experimental results indicate that Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3) composite material is an alternative material for its use as human bone substitute, because its density, porosity and toughness are better than of the human bone. Composites were manufactured using powders techniques. The results indicate that these composites have densities between 3.6 - 3.9 g/cm^3, values far higher than the density of the compact bone of 1.8 g/cm^3. The microstructure observed by scanning electron microscopy, shows a homogeneous distribution of titanium particles in the alumina matrix. Fracture toughness of the proposed composite present's values range between 3.3 to 8.76 MPam$^{-0.5}$, whereas fracture toughness of compact bone has a value of 4.05-4.32 MPam$^{-0.5}$.

Keywords:- Alumina/titanium, Biomaterial, Alumina reinforced, Toughness, Bone

I. INTRODUCTION

Human bones are composed of organic materials, minerals, and water; These bones are not entirely solid but have small spaces into its components, forming small channels through which the blood vessels circulating nutrient exchange charge, depending on the size of these pores can be of compact bone type (also called cortical) or spongy or trabecular bone1. Much research has focused on the characterization of different types of bones bovine, canine equine animals or human, and turn recent research has focused on finding materials that may have properties similar to human bones[1-5].

Recently, they had been significant progress in improving the ability of ceramic materials for use in structural applications. In particular their resistance has improved dramatically, to the point that today some ceramics can compete with metals in certain applications that previously would have been unimaginable their use[6].

However, regarding the mechanical properties of fracture toughness of ceramic materials still today continues making arduous research for improve it, because they are very fragile, according to recent studies ceramic materials can be toughened by incorporating fine particles of metal in its matrix, this development has been successfully used in different systems[7]. Some of these studies are focused on the strengthening mechanisms, in where it is possible to indicate that the size of the nonmetallic inclusion and homogeneous distribution thereof in the ceramic matrix is very important to ensure obtaining a composite material having good toughness[8].

Biocomposites using ceramic matrix are generally manufactured of alumina-ZrO_2, Al_2O_3 or hydroxyapatite (HAp)[9] that when are reinforced with polymers or metals it is achieved to reduce the elastic modulus of the composite material making it closer to the real bone, which lowers the stresses generated at the junctions of the implant with the bone. However, the main processes by which these composites are manufactured is extrusion, generating composites which present low values of toughness, reliability and mechanical strength, causing tribological abnormalities, originating surface wear of the biocomposites and sometimes the final break of the material. So that the fracture toughness, strength and wear are important factors which restrict their possible application as a biomaterial for use as prosthetics, therefore it must generate new composite materials whose processing routes let to improve these properties.

One such ceramic regarded as suitable candidates for making composite materials is alumina (Al_2O_3) which is a material having a high resistance to abrasion factors in both low and high temperatures and good wear resistance in corrosive environments, besides that alumina is a ceramic having good biocompatibility with the body and may be feasible to use it as a substitute for bone in the human body because of this feature.

II. EXPERIMENTAL

Composites were fabricated using two materials as raw materials classified as natural biomaterials, alumina (Sigma Aldrich, 99.9% purity, 100 nm-100 µm) and titanium (Aldrich, 99.99% purity, 5-10 µm). These materials were used in the preparation of binary mixtures of composition Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3). The mixtures were subjected to intense grinding at 300 rpm for 3h with spherical grinding media 13 mm, using ZrO2 grinding ratio 1:20. The powder mixture was then compacted by uniaxial cold pressing at 350MPa in order to form cylindrical pellets, 1 cm in diameter and 0.3 cm height. The obtained compacts were sintered in an electric furnace with MoSi2 heating elements at temperatures of 1400 °C for 1, 2 and 3 hours in a nitrogen atmosphere. Physical characterization was done by determining the density and porosity by the method of Archimedes; the mechanical properties of hardness and fracture toughness were determined using the method of indentation from measurements of the lengths of the cracks generated in the Vickers indentation with a Emco Durometer Test[10]. Finally, composites were prepared by ceramograph techniques to be observed by scanning electron microscopy. The values of density, hardness and fracture toughness will be compared with the same properties of human compact bones.

III. RESULTS AND DISCUSSION

A. Density and porosity

The density of the Al_2O_3-Ti composites was determined after performing sintering treatment at 1400 °C during 1, 2 and 3 hours, the obtained values are between 3.6-3.9 g/cm^3, which are within the theoretical density values 3.83-3.90 g/cm^3 determined by the rule of mixtures, considering 3.95 g/cm^3 as the density of the alumina and 4.51 g/cm^3 as the density of titanium; results density of alumina based composites exceed by more than 200% of the density of human compact bone (1.8 g/cm^3). The porosity of the alumina based composites is shown in Fig. 1, the graphs show the percentage of porosity in composites as a function of the titanium content and of sintering treatment time, in this way porosity is lower when the titanium content is greater, on the other hand, products with lower porosity are obtained when the sintering treatment is longer (3 hours). It is important to emphasize the effect in decreasing porosity (sample's densification) due to titanium presence, situation due to the good thermal conductivity of titanium that promotes diffusion mechanisms during sintering.

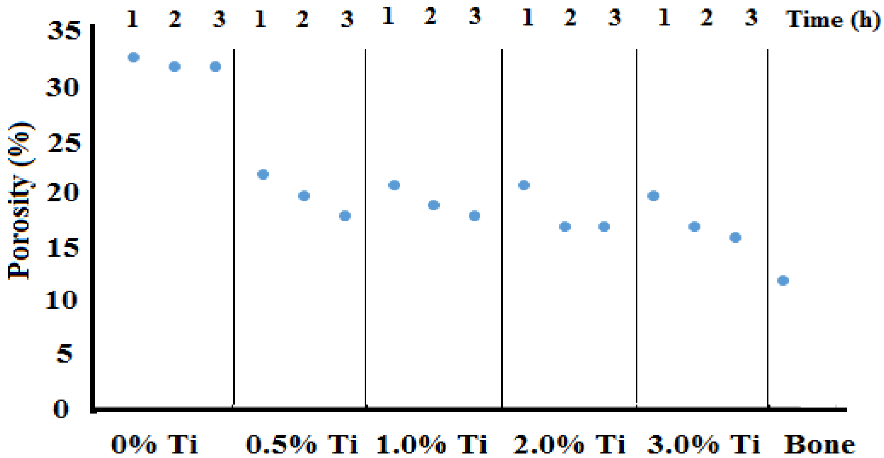

Fig. 1: Porosity of composites Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3), and porosity of human compact bone

B. Fracture toughness

The fracture toughness is shown in Fig. 2. in all cases it is observed that the presence of titanium in the alumina improves the fracture toughness of the composite, more even when titanium contents are above 2% by weight. Comparing the fracture toughness of the samples with 3% titanium sintered for 2 and 3 hours at 1400 ° C, it has which reaches 32% highest values compared with the value of the tenacity of human bone. So that the titanium effectively acts as a means of strengthening alumina, some researches[11-13] have suggested that the mechanism, by which metals reinforced alumina, is the cracks deflection due to the presence of a ductile metal in the ceramic matrix.

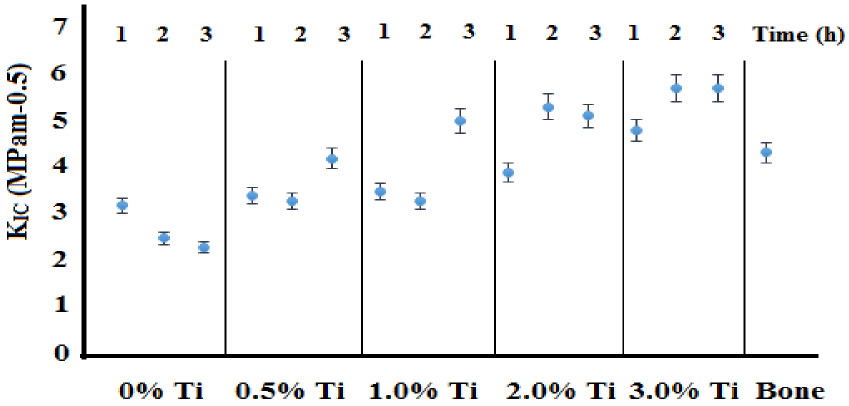

Fig. 2: Fracture toughness of composites Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3), and fracture toughness of human compact bone

C. Hardness

In Fig. 3. the hardness values are observed according to both the amount of titanium in the composite and sintering treatment time. Comparing hardness of 350 HV in compact bone with the hardness obtained in the Al_2O_3 composites, it can be seen that all manufactured composites present hardness values exceeding those of compact bone, this situation is logical because the alumina is an extremely hard material that when mixed with a more ductile metal reduces its hardness. Hence the composite hardness values tend to be more similar to those of alumina in the way as the titanium content increases by this.

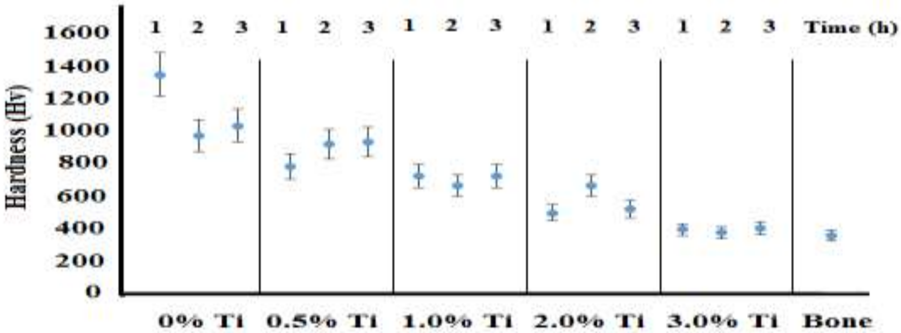

Fig. 3: Hardness of composites Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3), and hardness of human compact bone

D. MICROSTRUCTURE

Fig. 4. shows micrographs obtained by scanning electron microscopy in Al2O3-X wt. % Ti, (X = 0.5, 1, 2 and 3) composites, sintered at 1400 °C during 2 hours. In all samples are observed microstructures with irregularly grains, having average sizes ranging from 1 micron to little less than 10 microns, in some very large grains they are observed due to abnormal grain growth, this is seen more at higher sintering times, situation which is logical because large sintering times allow greater diffusion of atoms between grains. For the control sample (0% Ti) a partially sintered microstructure is observed, it means sintering conditions, principally the temperature of 1400°C, It was not high enough to achieve good densification of the ceramic, and greater activation energy is here required to consolidate the body. When the microstructures with additions of titanium in all studied percentages, it is observed a different situation, since in these samples it can be seen better consolidated bodies, situation further improved with increases in sintering times and titanium % in the matrix. This can be explained by the fact that having a metal present as titanium, which it is a good conductor of heat, the diffusion mechanism are favored between the particles that make up the material, situation that is not necessary to increase the temperature to achieve better consolidated bodies, as it would be necessary in a pure ceramic as is the case of alumina. Although, in the samples it is observed grain growth as a function of sintering time, this is minimum. It is also possible to observe that titanium is in the form of very fine particles of lighter color than the matrix, which would presumably be in this position will block the growth of cracks when they try to pass where lies this. Likewise, no significant presence of porosity it is observed in the microstructure. With respect to the microstructure of human bone, it has which is much thinner compared to the composites produced here.

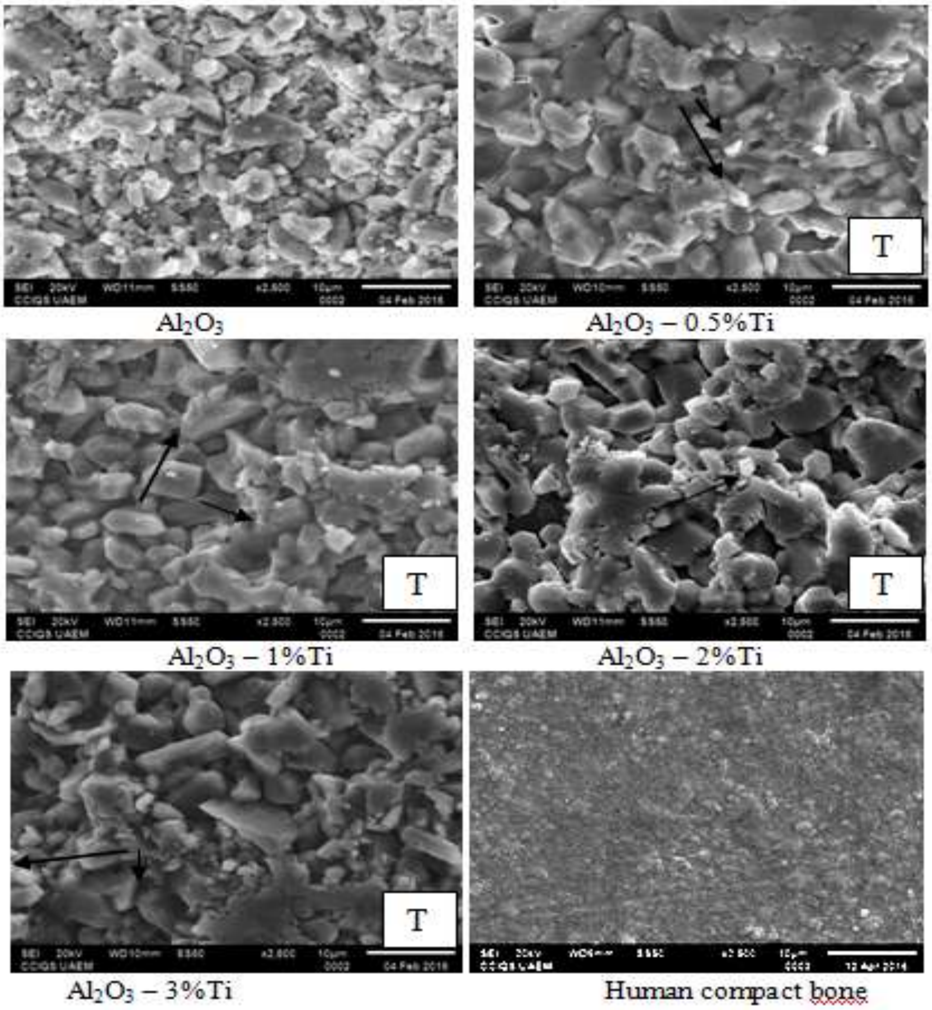

Fig. 4: Microstructure of composites Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3), sintered at 1400°C during 2h, and microstructure of human compact bone

Fig. 5. shows typical results of microanalysis performed with X-rays energy dispersive (EDX) executed in samples with 0% and 3 wt. % Ti in a punctual manner in the clear and dark particles of the microstructure. For the case of the sample with 0 wt. % Ti, obtained spectra indicate 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 3 wt. % Ti of SEM photomicrographs shown in Figure 4, the presence of two phases is observed, in accordance with the EDX spectra. 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.

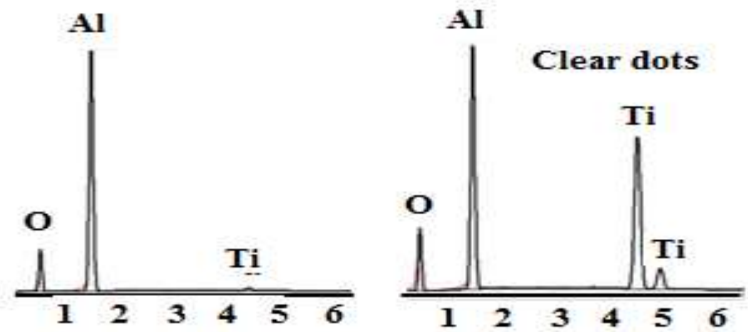

Fig. 5: EDX spectra corresponding to Al_2O_3/0.0 wt % Ti and Al_2O_3/3 wt % Ti sintered at 1400°C for 2 hours

In Fig. 6. It is observed and indentation obtained during microhardness testing of samples with 3 wt. % Ti sintered for 3 hours at 1400°C. In this figure, 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 Al2O3-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 Al2O3 by means of Ti..

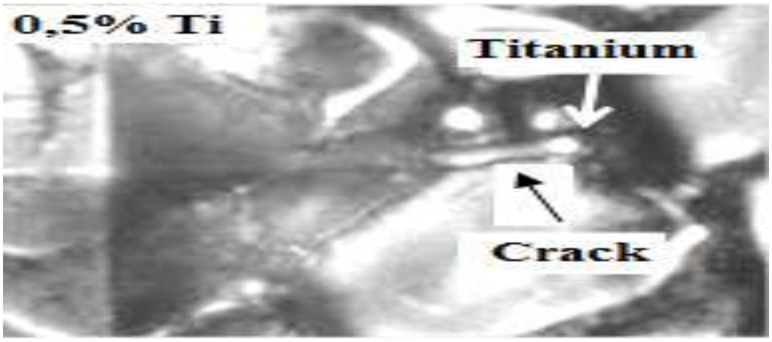

Fig. 6: Crack generated at the apex of the indentation mark during microhardness testing in sample with 3 wt. % Ti sintered for 3 hours at 1400°C

In Fig. 7: It is presented the fracture surface of samples with 3 wt. % Ti sintered for 3 hours at 1400°C. In this figure a way brittle fracture is observed, it can be seen well detachment alumina grains from each other, without any deformation in the same, also it is seen that titanium particles are located at the grain boundaries which were also torn during fracture of the material.

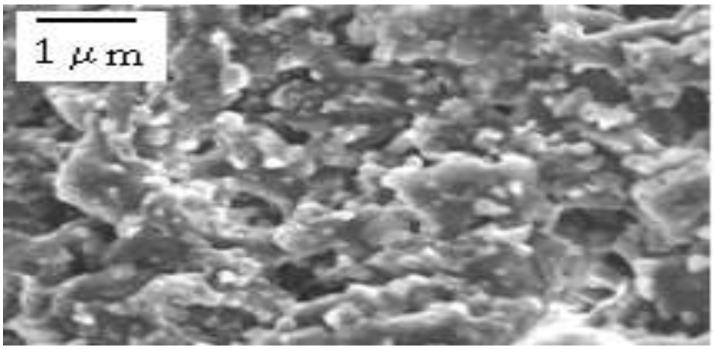

Fig. 7: Fracture surface in an Al_2O_3/Ti reinforced composite

IV. CONCLUSIONS

- Trough proposed methodology it is possible to manufactured alumina based composites with improved values of fracture toughness, making possible its use in biomedical applications.
- The composites Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3), can be classified as biomaterials, because the raw material with which these composites are manufactured are considered natural biomaterials.
- The Al_2O_3 composite containing 2 or 3 wt. % titanium and that was sintered at 1400 °C during 2 hours, exhibits values exceeding 32 % fracture toughness of a human compact bone.
- All manufactured composites of the Al_2O_3-X wt. % Ti, (X = 0.5, 1, 2 and 3) present hardness values exceeding those of compact bone.

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