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DEVELOPMENT AND PRODUCTION OF CERAMIC MATRIX BASED ON ZIRCONIA-TITANIA REINFORCED WITH RARE EARTH OXIDE FOR AEROSPACE EXHAUST SYSTEMS

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Abstract. *The reliability and availability of aerospace turbines is a constant concern since they operate at high temperatures and may fail prematurely. These are composed of alloys of nickel and cobalt, and can be coated with ceramic materials that perform well under high temperatures. Studies have been carried out on the use of zirconia (ZrO_2) incorporated with other oxides for this type of coating, because in comparison with other ceramics, it has superior mechanical properties, such as high mechanical strength, chemical stability and good fracture toughness. In this work, zirconia-titania ceramic matrix (ZrO_2 - TiO_2) reinforced with lanthanum (La_2O_3) was produced, with content of TiO_2 in 15% and varying the content of La_2O_3 in 5%, 7% and 10%. The ceramic matrix composites were characterized by X-ray diffraction, scanning electron microscopy and Vickers microhardness. The results showed that the composite with 10% La_2O_3 and 15% TiO_2 obtained a better result, indicating that this composite has good physical properties that point to possible applicability. However, it is necessary to evaluate other mechanical properties in order to ensure their use.*

Keywords: *aerospace turbines, coating, ceramic matrix composites, zirconia-titania, La_2O_3 .*

1. INTRODUCTION

The engine exhaust system of a rocket is structurally composed of nickel-based superalloys, which ensure high strength and stability at high operating temperatures. However, the need of the aerospace industry to reduce the weight on this system and to operate at increasingly higher temperatures has made the development of ceramic materials closely related to its development (Goswami *et al.*, 2004; Mechnic *et al.*, 2004).

High temperature ceramics have emerged as a promising class of materials for aerospace application (Opeka *et al.*, 2004; Fahrenholtz *et al.*, 2007). The refractory nature of this class of carbides, borides, nitrides and oxides make them attractive candidates for the greater heat flow in these areas.

Zirconium oxide (ZrO_2) is an attractive material for various engineering applications because, compared to other ceramics, it has superior mechanical properties such as high mechanical strength, chemical stability and fracture toughness combined with good wear resistance And a coefficient of thermal expansion close to that of iron and ferrous alloys, which makes it favorable for use in ceramic hardening and coating (Wu *et al.*, 2004). Zirconium oxide shows excellent mechanical properties through the adaptation of a very refined microstructure, which consists of a metastable tetragonal phase maintained at room temperature known as tetragonal-zirconia polycrystals (TZP) (Garvie *et al.*, 1975).

But the intrinsic fragility of ceramics is still a fatal factor for the use of these materials in mechanical structures and industrial applications. To reduce this brittleness, increase mechanical strength and fracture toughness, the ceramics are usually reinforced with the incorporation of one or more ceramic additives (Evans, 1990; Becher, 1991). Investigations on ZrO_2 - CeO_2 - TiO_2 systems (Pandolfelli *et al.*, 1989) and ZrO_2 - Y_2O_3 - TiO_2 (Ingel *et al.*, 1988; Pyda *et al.*, 1992) show that they are useful for the preparation of TZP materials.

In this sense, the objective of this work is to develop zirconia-titania ceramic matrix reinforced with a rare earth oxide (La_2O_3) that presents characteristics of high fracture toughness, high mechanical strength and high temperature environment resistance, for application in the system engine exhaust in aerospace applications.

2. EXPERIMENTAL PROCEDURE

In this research, high purity (99%) constituent oxides ZrO_2 , TiO_2 and La_2O_3 were used for the production of ceramic composites. We set 15% by weight of TiO_2 , and varied from 5-10 wt% La_2O_3 in ZrO_2 matrix. Table 1 presents the weight percentage of oxides in ZrO_2 - TiO_2 reinforced with La_2O_3 oxides. The oxides were weighed in an analytical balance and mixed for homogenizing, these oxides were ground in a ball mill, using high density alumina balls, for 24 hours in solid medium.

Table 1. Percentage of oxides (wt %)

Sample	ZrO_2	TiO_2	La_2O_3
1	80,0	15	5,0
2	78,0	15	7,0
3	75,0	15	10,0

The forming process was done using a hydraulic press (SCHIWING SIWA, ART6500089 model). The powders were compacted metal matrix of 10 mm diameter, using a pressure of 10 tons/cm² for 10 minutes, obtaining compact circular discs of approximately 5 mm thickness.

Then the powders were sintered in a muffle furnace (Jung 0614, maximum temperature of 1400°C). The sintering process was carried at 1385°C for 24 hours. The sintering was performed in ambient atmosphere in high purity alumina crucibles at a constant rate of temperature increase (5°C /min) and cooling to room temperature.

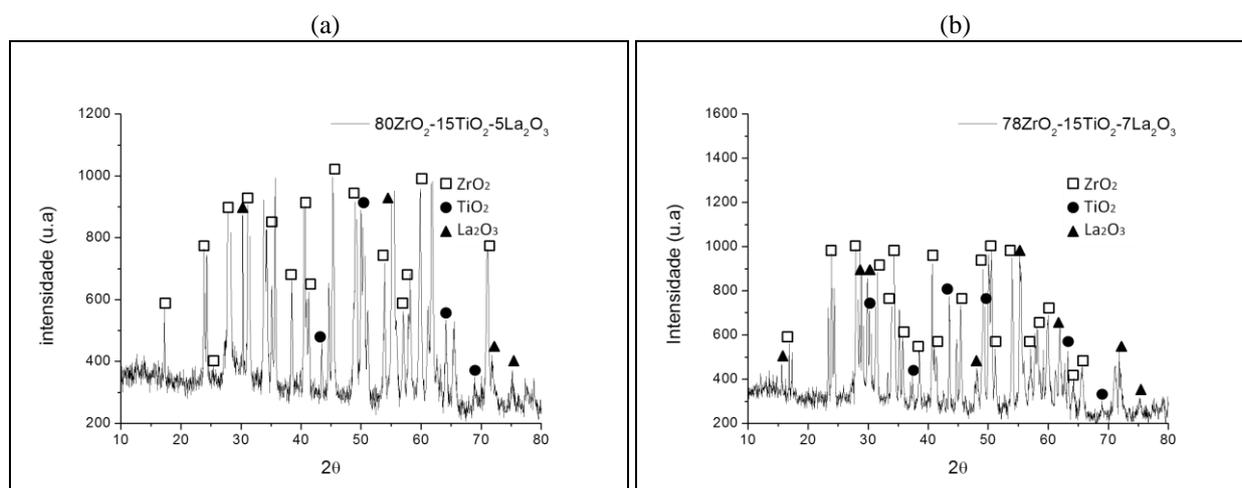
After the sintering process, the ceramic composite was subjected to structural characteristics and identification of phases were investigated by X-ray powder diffractometry (XRD) using Shimadzu X-ray Diffractometer, equipped with Cu-K α radiation ($\lambda=1.5406\text{\AA}$).

The microstructures analysis of the ceramic composites was done using a scanning electron microscope, model TM300-HITACHI.

The ceramic composite sintered pellets were subjected to Vickers microhardness test to analyze the mechanical behavior, the equipment used was the microdurometer with penetrator Model HVS-5 No. 0021.

3. RESULTS AND DISCUSSION

Through the analysis of diffraction-X obtained diffractograms presented in Fig. 1 where it is possible to identify the characteristic peaks of the raw materials used. It is possible to perceive the similarity between each composition, considering that all are formed with the same oxides, with only some variation in the content of each one of them. It is also observed a small variation in the intensity of the peaks, justified due to the percentage variation in the composites (JCPDS, 2000).



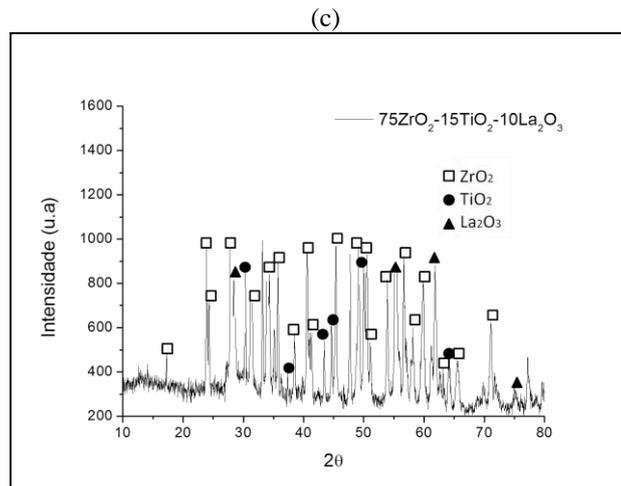


Figure 1. Diffractograms of ceramic composites. (a) Sample 1. (b) Sample 2. (c) Sample 3

Figure 2 shows the micrographs of the compositions studied with homogeneous microstructure in relation to the distribution and size of the grains.

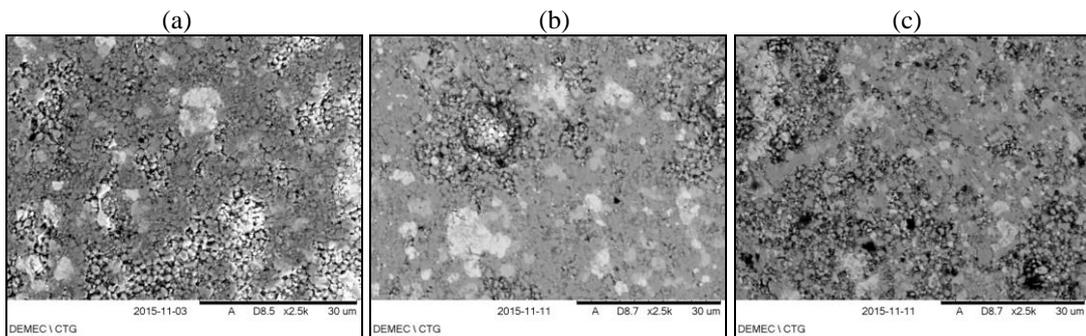


Figure 2. (a) Sample 1. (b) Sample 2. (c) Sample 3

Comparing the microhardness profiles of the compositions with 15% TiO₂ and different La₂O₃ contents, as shown in the graph of Figure 3, it is not possible to affirm that there was increase of hardness with the increase of La₂O₃, since the samples 1, 2 and 3 presented a range of similar hardness values.

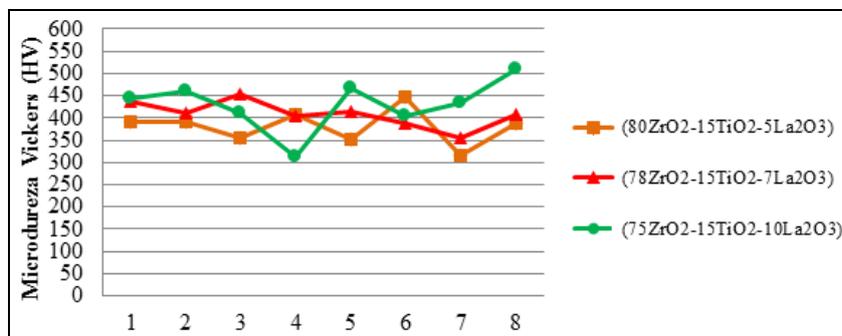


Figure 3. Vickers microhardness of samples with 15% TiO₂

4. CONCLUSIONS

Scanning electron microscopy showed good particle size distribution and homogeneity of the sintered composites.

The mean microhardness values of the samples with different La₂O₃ levels were around 380 to 430 HV, representing excellent hardness values.

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