

PRODUCTION AND SINTERING BEHAVIOR OF ZrO₂-TiO₂-La₂O₃ CERAMIC COMPOSITES FOR APPLICATIONS IN EXHAUST SYSTEMS IN AEROSPACE INDUSTRY

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Abstract. Ceramic matrix composites have emerged as a promising class for aerospace application due to its greater ability to withstand high temperatures and requiring less cooling air in order to reduce fuel consumption and improve performance. But the intrinsic fragility of the ceramic is still a crucial factor for use of these materials in mechanical and industrial applications structures. To reduce the brittleness, increase the mechanical strength and fracture toughness, the ceramic is reinforced by incorporating the addition of one or more ceramic phases. Knowing that the zirconium oxide (ZrO₂) when incorporated with other oxides can promote polycrystalline zirconia with superior properties, this work aims to develop zirconia-titania (TiO₂-ZrO₂) ceramic matrix composites reinforced by La₂O₃ an oxide of rare earth, which presents high mechanical strength and resistance to high temperature environment for application in engine exhaust system used in the aerospace industry.

Keywords: ZrO₂-TiO₂-La₂O₃, sintering behavior, aerospace industry.

1. INTRODUCTION

The rocket engine exhaust system is structurally composed of nickel base superalloys, which ensure high strength and stability at high temperatures of use. However, the need in the aerospace industry to decrease the burden on the system and operate at increasingly high temperatures have made the development of ceramic material intimately linked to their development (Goswami et al, 2004 and Mechnic et al, 2004).

Recently, engine manufacturers have become more concerned about the prospects of use of the composite ceramic matrix in the hot section of the engine, due to its greater ability to withstand high temperatures and requiring less cooling air so as to reduce the consumption of fuel and improve performance (M. Mecham, 2012, Christian et al, 2010 and Zawada et al, 2003).

In this sense, the ceramic high temperature has been emerging as a promising class of materials for aerospace application (Opeka et al, 2004 and Fahrenholtz et al, 2007). The refractory nature of this class of carbides, borides, nitrides and oxides make them attractive candidates for the higher heat flux in these areas there.

Zirconium oxide (ZrO₂) 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 a good resistance to wear and a coefficient of thermal expansion close to that of iron and ferrous alloys, which makes it favorable to use ceramic coating and hardening process (Wu, 2004). Zirconium oxide shows excellent mechanical properties by adapting a much finer microstructure, which consists of a metastable tetragonal phase maintained at room temperature, known as tetragonal zirconia polycrystals-(TZP) (Garvie, 1975).

But the intrinsic fragility of ceramics is still a fatal factor for use of these materials in mechanical and industrial applications structures. To reduce this fragility, increase the mechanical strength and fracture toughness, typically the ceramic are enhanced with the incorporation of one or more ceramic additives (Evans, 1990, Ai 1995, Becher, 1991). Investigations of CeO₂-ZrO₂-TiO₂ system (Pandolfelli, 1989) and Y₂O₃-ZrO₂-TiO₂ (Ingel, 1988 Pyda, 1992) show that they are useful for the preparation of TZP materials.

In the processing of ceramic microstructural characteristics play a vital role in the quality of the final product. The microstructural development is also dependent on the sintering behavior of these ceramics.

In this sense, this work aims to develop ceramic matrix composites reinforced zirconia-titania with a rare earth oxide (La₂O₃) to present high tenacity to fracture characteristics, high mechanical strength and resistance to high temperature environment for application in the system engine exhaust in aerospace applications.

2. MATERIAL AND METHODS

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 35% 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 $\text{ZrO}_2\text{-TiO}_2$ 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	90,0	5,0	5,0
2	88,0	5,0	7,0
3	85,0	5,0	10,0

The forming process was done using a hydraulic press (SCHIWIN SIWA, ART6500089 model). The powders were compacted metal matrix of 10 mm diameter, using a pressure of 10 tons/cm^2 for 10 minutes, obtaining compact circular discs of approximately 5 mm thickness.

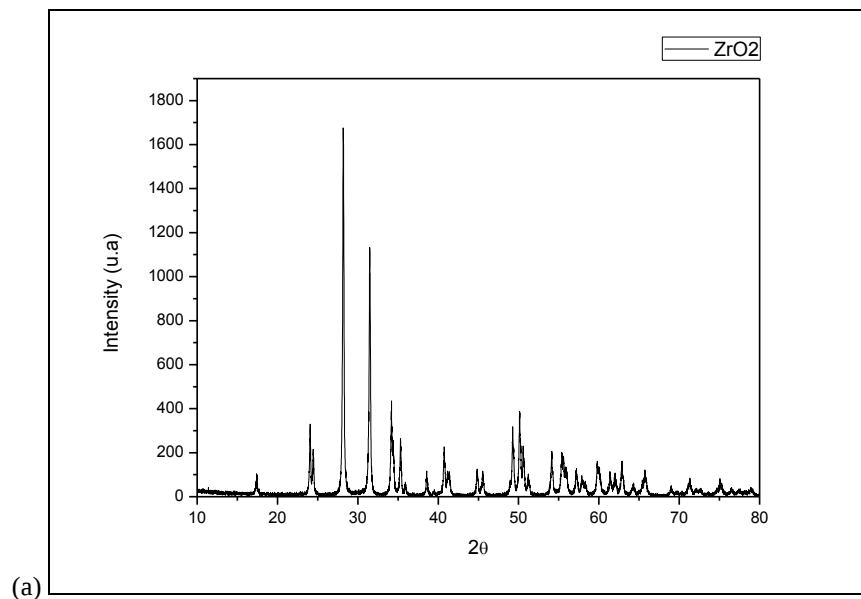
Then the powders were sintered in a muffle furnace (Jung 0614, maximum temperature of 1400°C). The sintering process was carried out between $1300^\circ\text{-}1400^\circ\text{C}$ for 24 hours. The sintering was performed in ambient atmosphere in high purity alumina crucibles at a constant rate of temperature increase (5°C/min) and cooling to room temperature.

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

The ceramic composite sintered pellets were subjected to MicrodurezaVickers test to analyze the mechanical behavior, the equipment used was the microdurometer with penetrator Model HVS-5 No. 0021. For this measurement, the sintered composites were polished with sandpaper #200, #400, #600, #1000, #1200, #1500 successively, and then was made a mechanical polishing with diamond paste with a particle size of 1 micron to submit bright appearance. Were madem10 measurements across the tablet.

3. RESULTS AND DISCUSSION

Through the analysis of diffraction-X obtained diffractograms presented in Fig. 1 (a), (b) and (c) where it is possible to identify the characteristic of each of the raw materials used in the formation of $\text{ZrO}_2\text{-TiO}_2\text{-La}_2\text{O}_3$ composite.



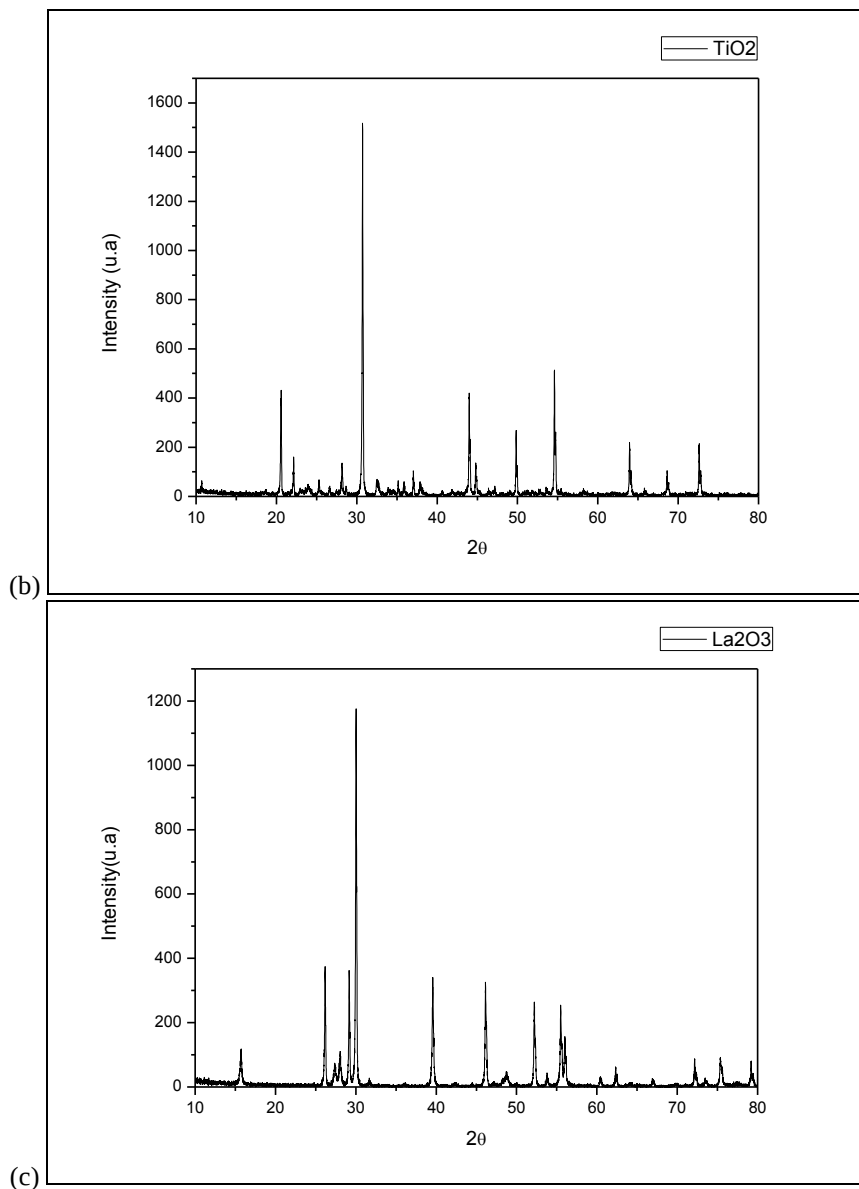


Figure 1. Ceramic powder diffractogram . (a) ZrO_2 . (b) TiO_2 . (c) La_2O_3 .

Figure 2 illustrates the diffractograms of sample 1, sample 2 and sample 3, where we can observe the characteristic peaks of the raw materials used. It is possible to realize the similarity between each composition, given that all are formed from the same raw material with only variation in the La_2O_3 content, which explains the differences in peak intensity.

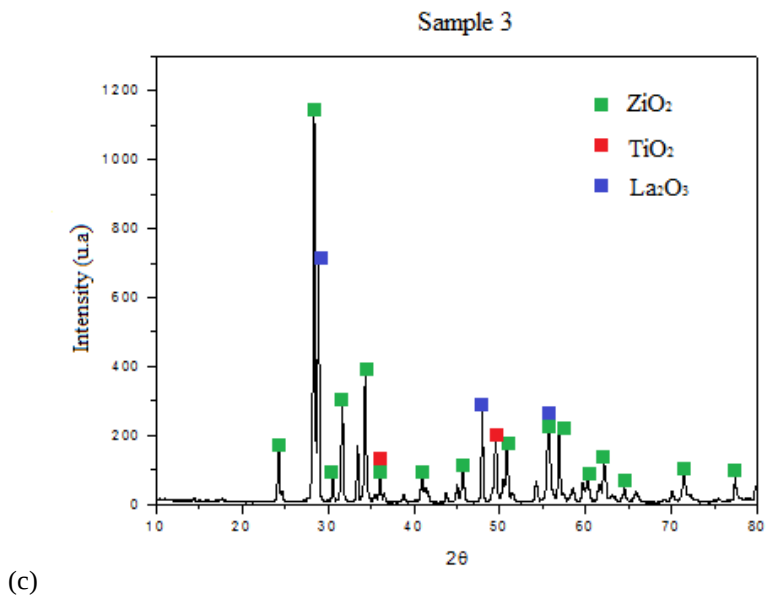
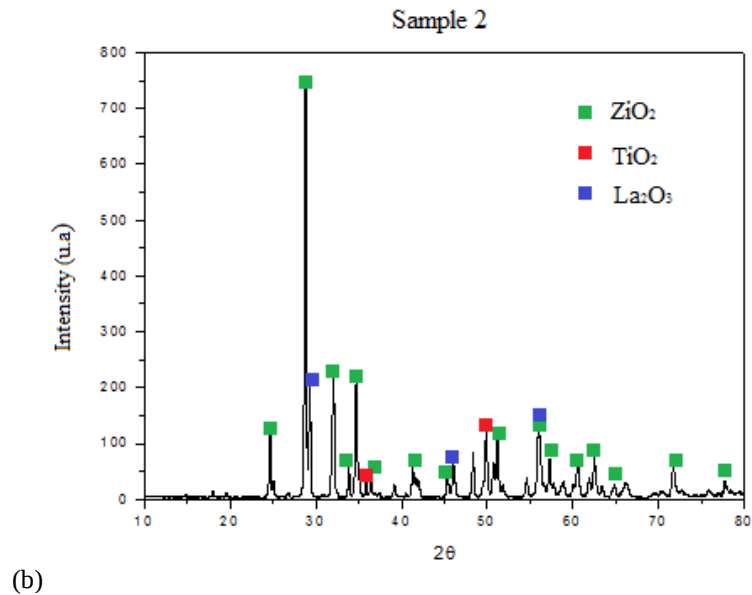
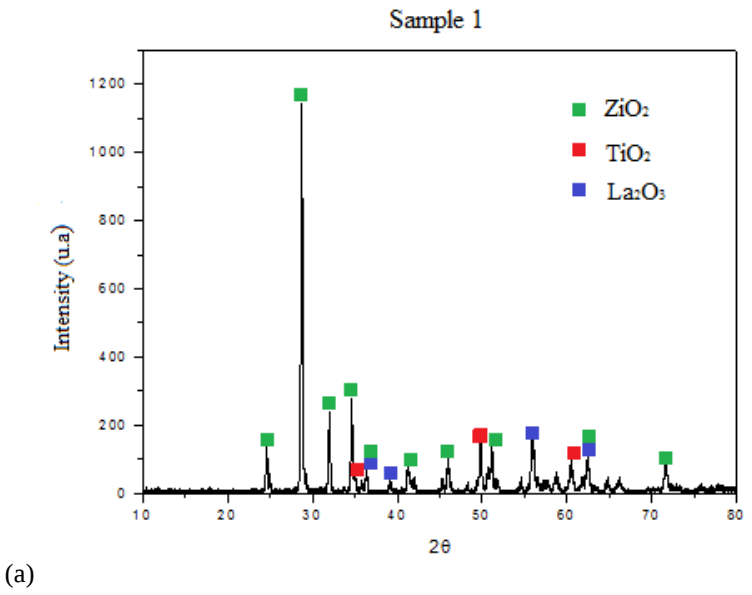


Figure 2. XRD results of samples sintered at 1385 ° C for 24 hours.
(a) Composition 1 with 5% La₂O₃. (b) Composition 2 with 7% La₂O₃.
(a) Composition 3 with 10% La₂O₃.

The hardness tests were performed on the sintered samples produced. Ten indentations were made on each sample and averaged hardness, discarding the two most disparate measures.

The best result hardness were obtained for the sample 3, with 10% La₂O₃, as illustrated in Figure 3, average hardness 170.4 HV, 115.2 HV against the sample 1 and 117.0 HV sample 2.

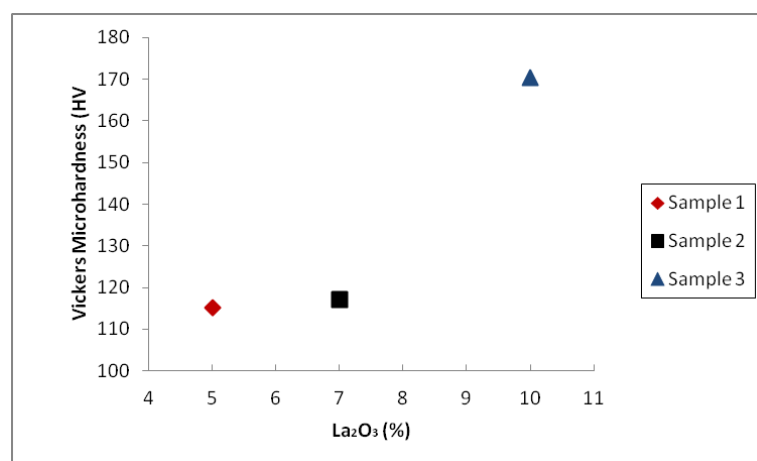


Figure 3. Average Vickers microhardness of the produced samples.

These results indicated that from 7% La₂O₃, there was an increase of about 45.64% on average microhardness of samples.

4. CONCLUSION

It was observed through the analysis results of X-ray diffraction after sintering no new phase is formed in addition to the characteristics of precursor oxides indicating formation of the composite. But there was variation in their concentration.

According to the results of the Vickers hardness test, it was observed that the sample with 10% La₂O₃ showed a better toughness compared to other samples studied.

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