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STUDY OF THE STABILITY OF THE CERAMIC COMPOSITE ZrO_2-Al_2O_3 REINFORCED WITH RARE EARTH OXIDE FOR COATING METALIC MATRICES FOR THE OIL INDUSTRY

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Abstract. *The development and characterization of the ceramic Al_2O_3 - ZrO_2 (Alumina- Zirconia) reinforced with rare earth oxide for use in oil industry will be presented in this academic work. For the tests were prepared four types of samples containing Alumina added with gradually increasing percentages of Zirconia (5%, 10%, 15% and 20% (by weight)) and 2% fixed rare earth oxide. The ceramic powders were weighed, mixed in low-energy ball mill, pressed by uniaxial pressing and sintered at high temperature furnace. For characterization of the samples were performed tests of Optical Microscopy (OM), Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD). The samples were immersed in crude oil, oils from land and from sea, coming from wells in the state of Sergipe, and their behavior was analyzed after immersion in oil environment. After that, the samples were reanalyzed by optical microscopy and were not observed cracks or defects on the material surface, indicating stability of the composite in crude oil environment.*

Keywords: *Alumina, crude oil, optical microscopy, thermal spray hypersonic*

1. INTRODUCTION

In the petroleum industry it is common that the storage and transportation tanks of crude oil are exposed to high temperatures and chemically aggressive environments. The surface degradation of these storage tanks has been an question that requires close attention, requiring reliable materials and systems for operation in these hostile conditions. An alternative to solve this problem is to use a type of inert coating to this corrosion.

At the last decades, new advanced ceramic materials were introduced in the oil industry as coating. (Evans, 1990) One of the main advantages in the use of advanced ceramics is their resistance to these hostile environments, but the intrinsic fragility (low toughness) by this kind of material, makes it susceptible to crack propagation. (Becher, 1991)

To reduce the brittleness and increase mechanical strength and improve the toughness of the material, usually ceramic are improved with the incorporation of one or more ceramic additives. The addition of rare earth oxides can greatly enhance the toughness of these ceramics. (Gong, J.; Miao, H.; Zhao, Z., 2001)

This paper wants to develop and characterize a ceramic coating Alumina reinforced with Zirconia (Al_2O_3 - ZrO_2) and rare earth oxides (La_2O_3). In the development of these materials were prepared four types of samples of Alumina containing percentage of 5%, 10%, 15% or 20% (by mass) of Zirconia and 2% (fixed) of Lantania. The ceramic powders were weighed, mixed in low-energy ball mill, pressed by uniaxial press and sintered at 1350ºC. In the characterization were performed optical microscopy tests, scanning electron microscopy and X-ray diffraction.

2. EXPERIMENTAL

The production of the ceramic Al_2O_3 - ZrO_2 - La_2O_3 consists of the following steps: weighing the powders respecting the proper proportions, grinding in a ball mill, press by uniaxial compaction and sintering in high temperature furnace.

The first step consists in mixing the compounds of aluminum oxide (Al_2O_3, Elizabeth with 99% purity), zirconium oxide (ZrO_2 Acros, 99% purity) and lanthanum oxide (La_2O_3, VETEC 99.9 % purity), which were added to Alumina in 5%, 10%, 15% and 20% Zirconia (by weight) of to 2% Lantania, according to Table 1.

Table 1. Composition of the four samples

Sample	Al_2O_3 (wt-%)	ZrO_2 (wt-%)	La_2O_3 (wt-%)
01	93	5	2
02	88	10	2
03	83	15	2
04	78	20	2

Each sample was mixed in ball mill with jar made of stainless steel containing 31 alumina balls, during 24 hours, for homogenization of grain and grain size reduction.

Then the samples were subjected to uniaxial compaction process (press SCHIWING SIWA, ART6500089 model) to give tablets in the form of circular discs with a diameter of ø30mm, under load of 12 tons, for 5 minutes and without the use of binder. The pellets were sintered at high temperature oven at 1350º C for 24 hours.

For the characterization of this material have been carried tests of X-ray Diffraction (XRD), Optical Microscopy (OM) and Scanning Electron Microscopy (SEM). The samples had the surface prepared using sanded (using abrasives of different meshes) and polished (with diamond paste).

After sintering, some tablets were broken to do the XRD. With optical microscopy, were analyzed three fields of the each sample, each field with magnifications of 50x, 100x and 200x. To obtain the SEM images, the samples were covered with a thin layer of conductive material sputtering unit (Coater BAL-TEC SCD050).

After this period, samples were removed from immersion and the excess oil removed with felt. Then they were again reanalyzed by optical microscopy.

3. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction – XRD

Seeking information about the structure of this ceramics was realized tests in the compounds individually and then in the four samples produced. Therefore were obtained the following diffractograms.

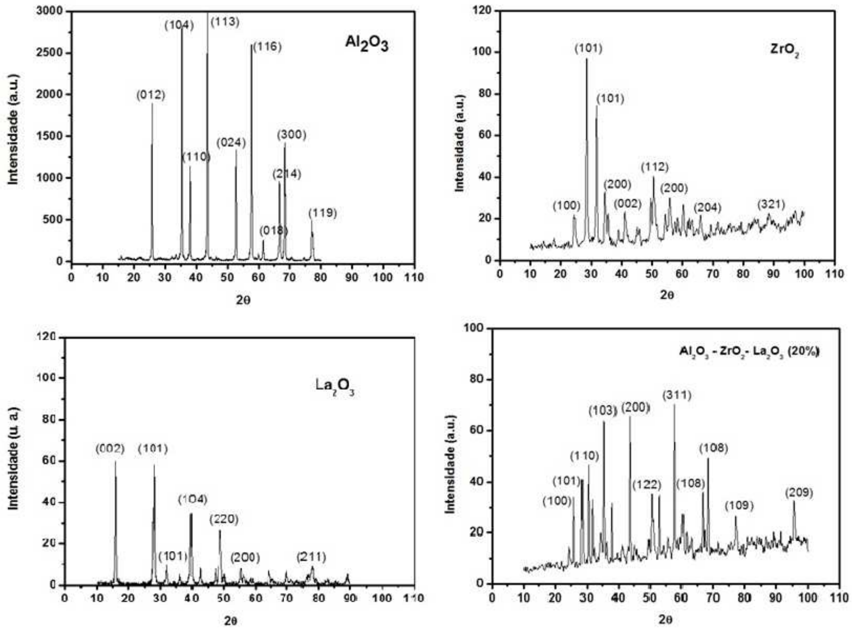

Figure 1. Diffractograms of the constituents of composite ceramic Al_2O_3-ZrO_2-La_2O_3 and of Sample 04 ($78Al_2O_3$-$20ZrO_2$-$2La_2O_3$ (wt-%))

From the XRD patterns obtained from samples that have passed through thermomechanical treatment, it was found that the constituent materials unreacted, which characterizes the composite formation.

3.2 Scanning Electron Microscopy - SEM

Part of the composite microstructure analysis was based on the images obtained from the SEM test, providing rapid morphological information of samples.

The results obtained with the SEM, done in sanded and polished surface, it can be seen that with gradual increase in the ZrO_2 content there was a microstructural modification where it was found that there is a considerable change in grain size and in distribution and homogeneity, as is stated in the following figures.

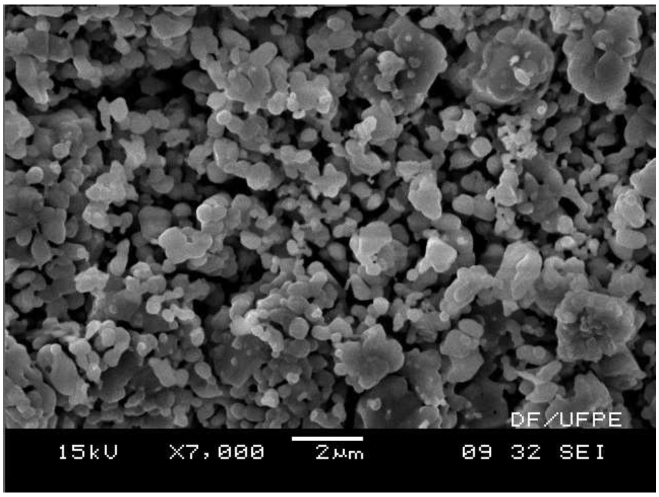

Figure 2. SEM of the Sample 01 (Composite ceramic $93Al_2O_3$-$5ZrO_2$-$2La_2O_3$ (wt-%))

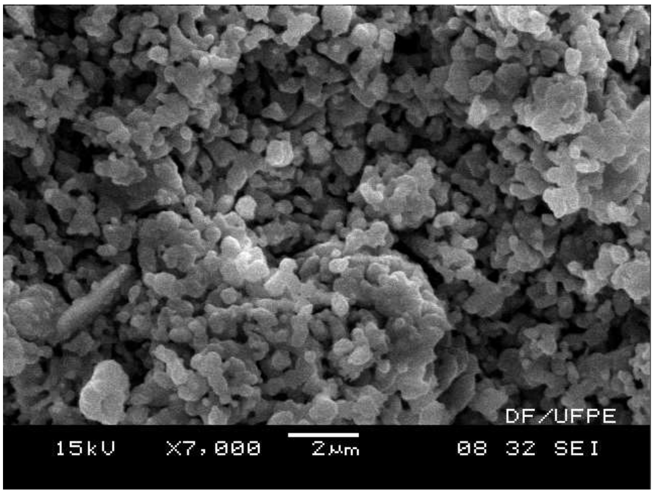

Figure 3. SEM of the Sample 02 (Composite ceramic $88Al_2O_3$-$10ZrO_2$-$2La_2O_3$ (wt-%))

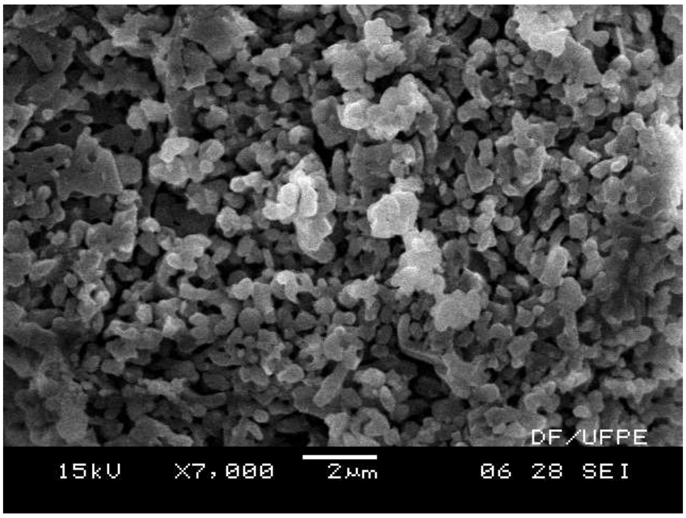

Figure 4. SEM of the Sample 03 (Composite ceramic $83Al_2O_3$-$15ZrO_2$-$2La_2O_3$ (wt-%))

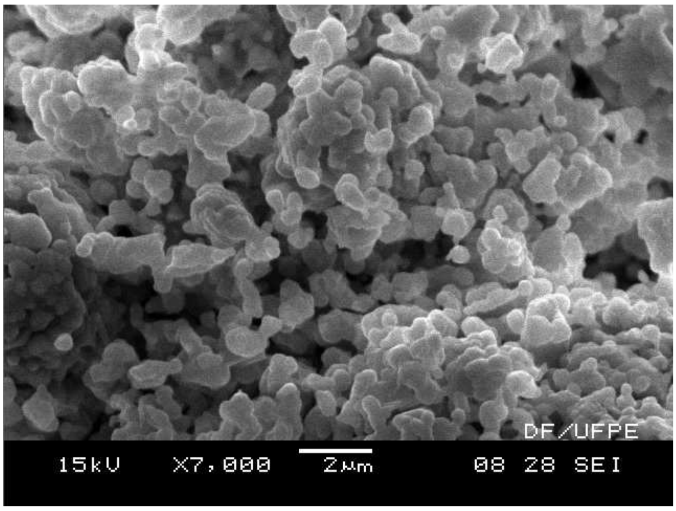

Figure 5. SEM of the Sample 04 (Composite ceramic $78Al_2O_3$-$20ZrO_2$-$2La_2O_3$ (wt-%))

3.3 Optical Microscopy - OM

Still about the analysis of the surface of the ceramic composite, OM were performed at all tablets samples before to immersion in crude oil and also after 30 and 60 days of immersion, allowing a comparison between the initial and the final state of the surface of the pellets.

The images obtained by OM exhibit good homogeneity particles for the four samples ZrO_2- Al_2O_3- La_2O_3 studied.

Analyzing the behavior and stability of the ceramic on environmental crude oil coming from Sergipe wells of land and sea, after periods of 30 and 60 days of storage, samples were reanalyzed by OM and showed no flaws or cracks on their surfaces indicating good inertia particulars at this chemically hostile environment.

Figures 6-9 show the results of tablets before immersion, after 60 days of immersion in crude oil sea and land of the samples with 5, 10, 15 and 20 (wt%) Zirconia, respectively.

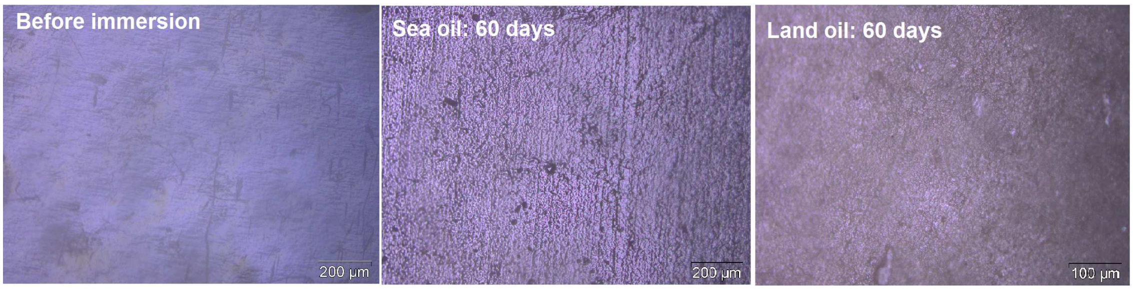

Figure 6. Optical microscopy (x100) of the Sample 01 before immersion and after 60 days of immersion in Sergipe oil from sea and from land

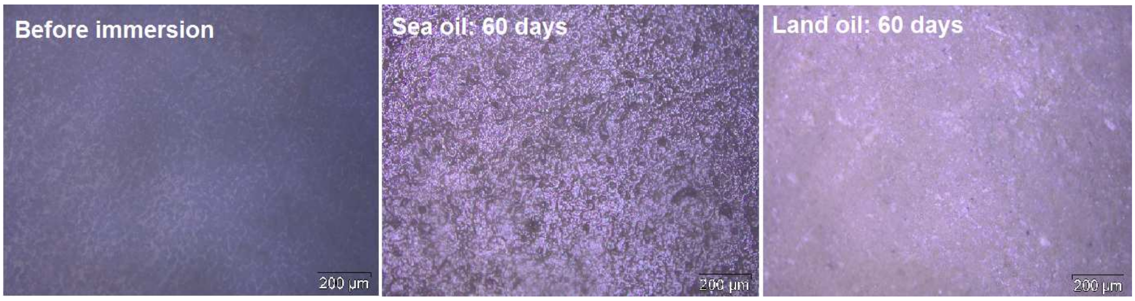

Figure 7. OM (x100) of the Sample 02 before and after 60 days of immersion in Sergipe oil from sea and from land

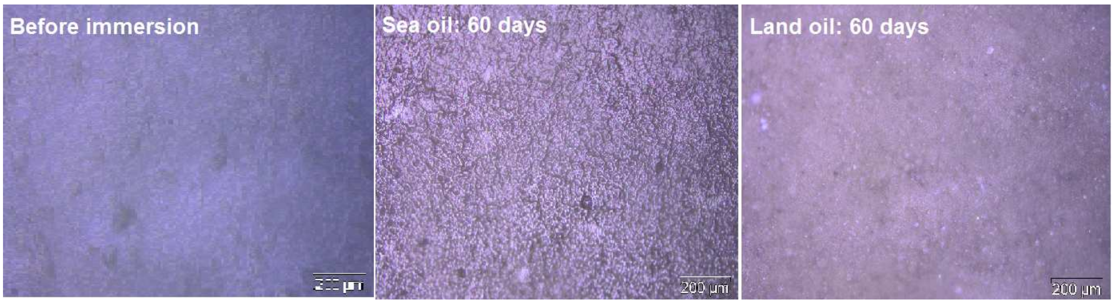

Figure 8. OM (x100) of the Sample 03 before and after 60 days of immersion in Sergipe oil from sea and from land

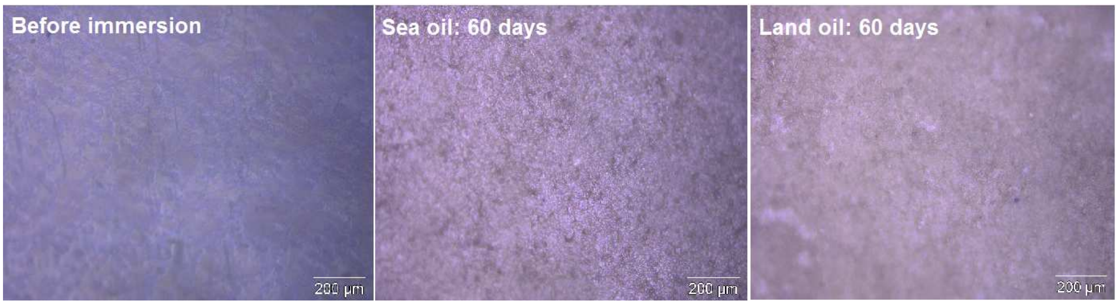

Figure 9. OM (x100) of the Sample 04 before and after 60 days of immersion in Sergipe oil from sea and from land

4. FINAL CONSIDERATIONS

The ceramic composite Al_2O_3 - ZrO_2 - La_2O_3 achieved good results micro structural and chemical stability of crude oil environment.

With the analysis microscopy seen that the advanced ceramic showed good homogeneity of particles and an improvement in its distribution, with the addition of Zirconia.

Finally, it was also observed that the composite did not react to contact with the crude oil environment during this study period, indicating that the composite ceramic in question has a good applicability for protective purposes coating metal matrix.

At the moment, remains ongoing execution of the material deposition techniques on the metal surface by thermal hypersonic spray.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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