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STUDY OF STABILITY OF AL2O3-Y2O3-TIO2 COMPOSITE CERAMICS IN CRUDE PETROLEUM ENVIRONMENT FOR INERT COATING APPLICATIONS IN PETROLEUM INDUSTRY

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Abstract. *Petroleum drilling equipment, storage tanks and transportation systems suffer from constant physical stress caused by chemical attack of crude petroleum on its structure. Ceramics have high chemical stability in hostile environment and therefore can be used as an inert coating material to solve such problems. Ceramics based on Alumina are most widely used in practice where there is demand for high strength and toughness but its intrinsic fragility is still a fatal factor. To reduce it and increase strength and toughness usually the ceramics are reinforced with ceramic additives. Mechanical properties of Alumina based ceramics improve with the addition of* TiO_2*,* ZrO_2 *etc. as reinforcement additives. We've produced* Al_2O_3*-*Y_2O_3*-*TiO_2 *ceramics composites in proportions of 5-20 wt%* TiO_2 *and 3% wt%* Y_2O_3 *with high mechanical strength, through thermo-mechanical processing and sintering techniques. To evaluate the materials developed and the possibility of using them as ceramics components for petroleum industry, we've studied the physic-chemical and mechanical stability of these materials in crude petroleum originated from onshore and offshore petroleum wells of Sergipe-Brazil. Ceramics composite* Al_2O_3*-*Y_2O_3*-*TiO_2 *were produced through thermo-mechanical processing and sintering techniques. Structural, microstructural and mechanical tests showed that composite ceramics with 15-20wt%* TiO_2 *presented better results in terms of mechanical hardness and microstructural characteristics. The study of stability of composite ceramics in crude petroleum environment showed that ceramics did not present any additional phase except the constituent phases. Microscopy tests and microstructural analysis showed that there is no visible change in these characteristics of ceramics after some days of immersion in petroleum.*

Keywords: Al_2O_3-Y_2O_3-TiO_2, *ceramic protective coating, crude petroleum*

1. INTRODUCTION

Petroleum drilling equipment's, storage tanks and transportation systems suffer from constant physical stress caused by chemical attack of crude petroleum on its structure. Ceramics have high chemical stability in hostile environment and therefore can be used as an inert coating material as an alternative to solve such problems. However, ceramics are highly fragile. Because they are mostly formed by ionic bonds and / or covalent bonds, which provide a limited number of independent slip systems necessary to achieve a homogeneous plastic deformation. Ceramics based on Alumina are most widely used in practice where there is demand for high strength and high toughness and its intrinsic fragility is still a fatal factor. To reduce vulnerability and increase strength and toughness usually the ceramics are reinforced with ceramic additives. Mechanical properties of Alumina based ceramics improve with the addition of TiO_2, TiN, etc. as reinforcement additives. In the work we've produced Al_2O_3-Y_2O_3-TiO_2 ceramics composites in proportions of 5-20 wt%

TiO_2 and 3% wt% Y_2O_3 with high mechanical strength, through thermo-mechanical processing and sintering techniques. To evaluate the quality of materials developed and the possibility of using them as inert protective coatings or ceramic components for crude petroleum extraction, storage and transportation systems, we've studied the physic-chemical and mechanical stability of these materials in crude petroleum. (Ashby, at all, 2007; Kingery, 1960; Padilha, 1985; Hosford, 2005).

2. EXPERIMENTAL PROCEDURES

The first stage of work was the preparation of the ceramic powder, where each material was weighed in predetermined proportions, thus obtaining the values of Tab. 1.

The oxides were then mixed in a mill made of stainless steel, rubber coated, containing 31 Alumina balls. Later the material was milled for 24 hours to provide a homogeneous character. Lastly, some of the material was separated and weighed on an analytical balance to obtain tablet with 8g of each sample. The powders were uniaxially pressed using a hydraulic press (Schiwing SIWA, ART6500089 model) at a pressure of 10 ton/cm² for a period of 5 minutes, in these process, circular discs were produced with 30 mm in diameter and 3-5 millimeters thickness. Ethylene glycol was used to facilitate demoulding therefore assists the work and does not modify the results to be easy evaporation. After pressing, the discs were sintered to obtain the ceramic composite. This step was performed in a muffle type furnace for 24 hours at a temperature of 1380 °C. After, the discs were sanded and polished with sandpaper between 320 and 2000 grain and then were subjected to X-ray analysis, optical microscopy and Vickers hardness tests.

Table 1: Composition of ceramic samples

Composition	Sample 1	Sample 2	Sample 3	Sample 4
Al_2O_3	92%	87%	82%	77%
TiO_2	5%	10%	15%	20%
Y_2O_3	3%	3%	3%	3%

3. RESULTS AND DISCUSSION

Structural characteristics and phase's identification were analyzed by XRD. Figures below show the results that showed no additional stage, only the constituent oxides phases, Al_2O_3, TiO_2 and Y_2O_3. The presence of Y_2O_3 phase is slightly observed in the XRD patterns because of its small amount percentage in the composite. As all the XRD figures presents the same XRD peaks, only the XRD peaks on figure 1 has been index.

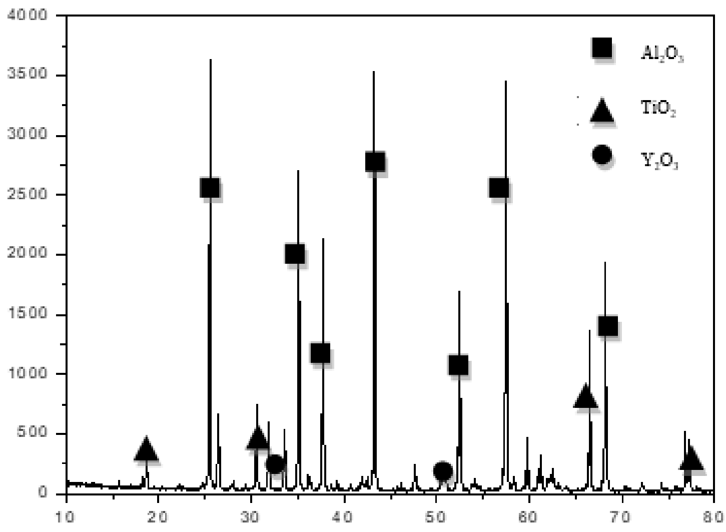


Figure 1: XRD of sample 1

(1)

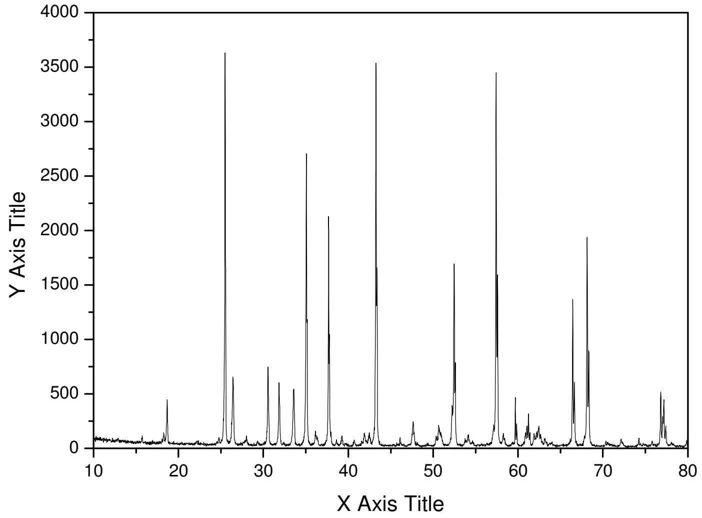


Figure 2: XRD of sample 2.

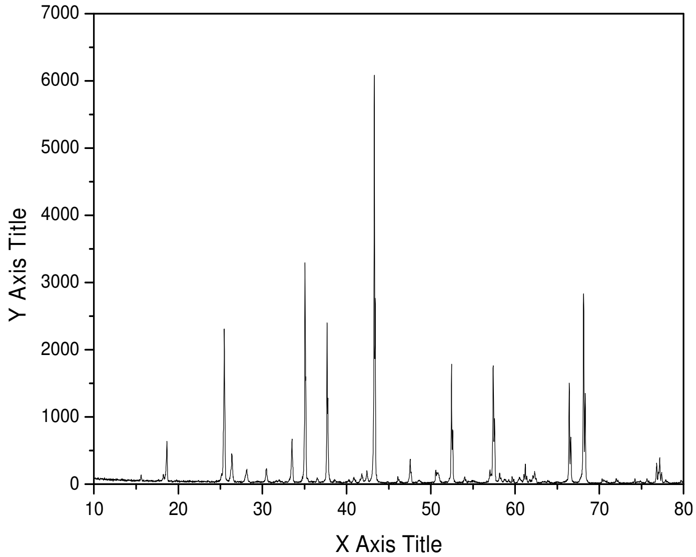


Figure 3: XRD of sample 3.

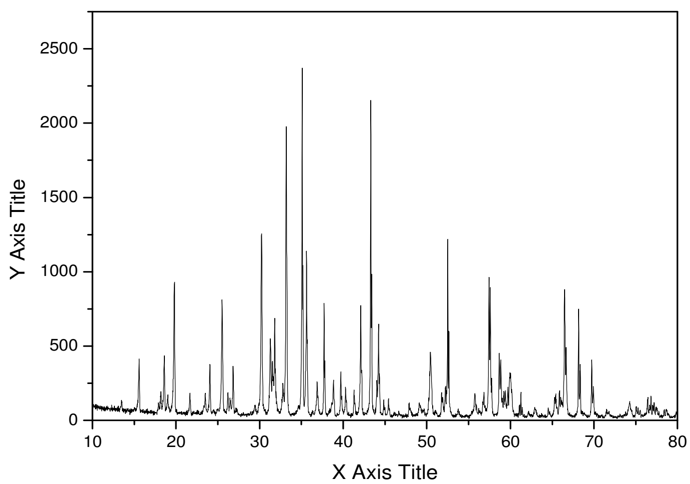


Figure 4: XRD of sample 4.

Microstructure analysis was performed by scanning electron microscopy, the results are shown in figures below. The samples had a homogenous microstructure with good grain size distribution. The homogenous and distribution in particle size was better after the gradual increase of TiO_2. The composites with 20% TiO_2, showed better.

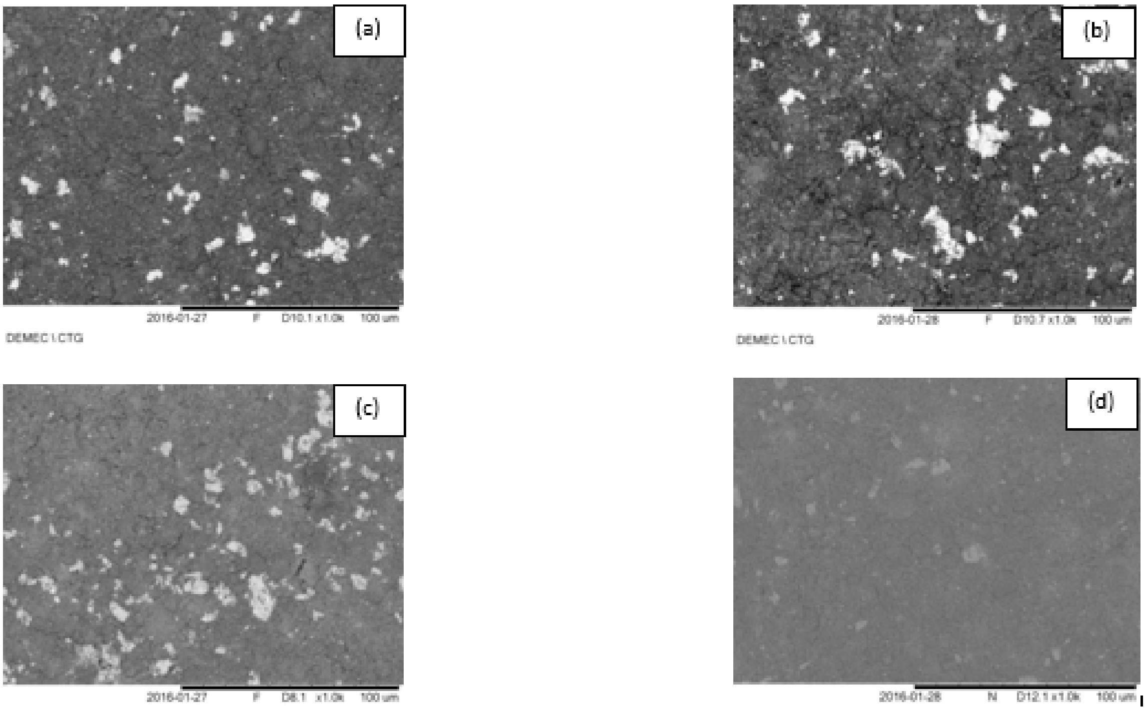
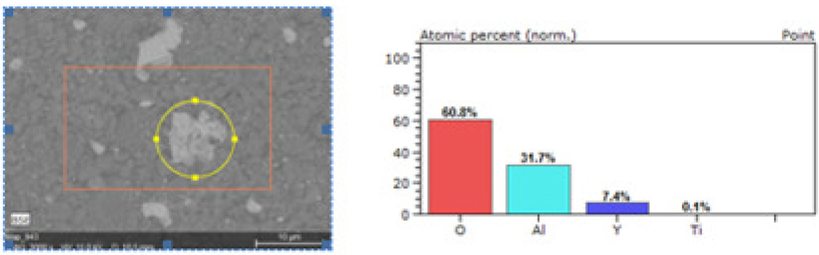
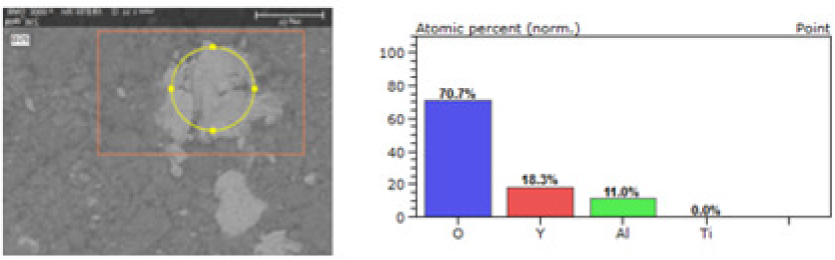


Figure 5: SEM of: (a) sample 1. (b) sample 2. (c) sample 3. (d) sample 4.

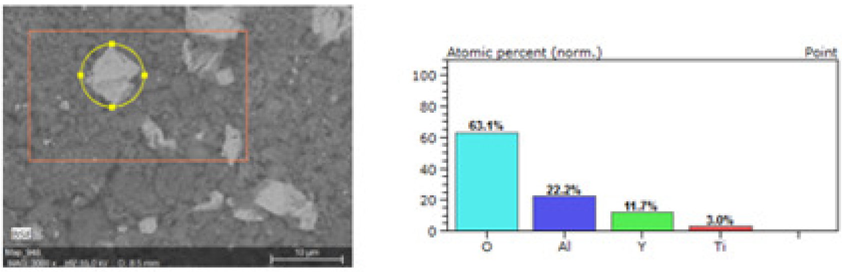
The elementary microanalysis by EDX presented in figure 6 showed that there were no contamination during the processing of the composites, since the presence of other elements, only of Al, Ti, Y and O, as expected, was not verified.



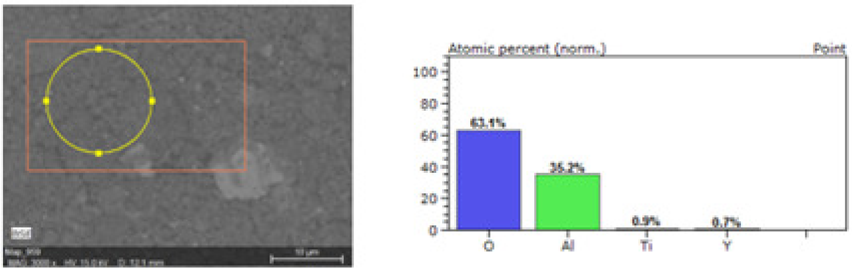
a)



b)



c)



d)

Figure 6: EDX analysis images: (a) 5% titania, (b) 10% titania, (c) 15% titania and (d) 20% titania, all with 3% of Ytria.

The Vickers micro hardness tests were carried out in the microdurometer with a Zeiss metallographic microscope, model Jenavert, figure 11. A load of 50 kgf was used during a time of 15 seconds.

Table 2: Average value of microhardness for each sample

Vickers Microhardness				
Samples with %TiO2:	5%	10%	15%	20%
Average value micro hardness:	21,68	22,96	27,2	32,47

Through optical microscopy images, performed before and after immersion of the samples in crude petroleum for 90 days, it exhibits that the surfaces of the samples showed no significant changes. We conclude that there is a good chemical stability of the composite due to lack of evidence of chemical attack.

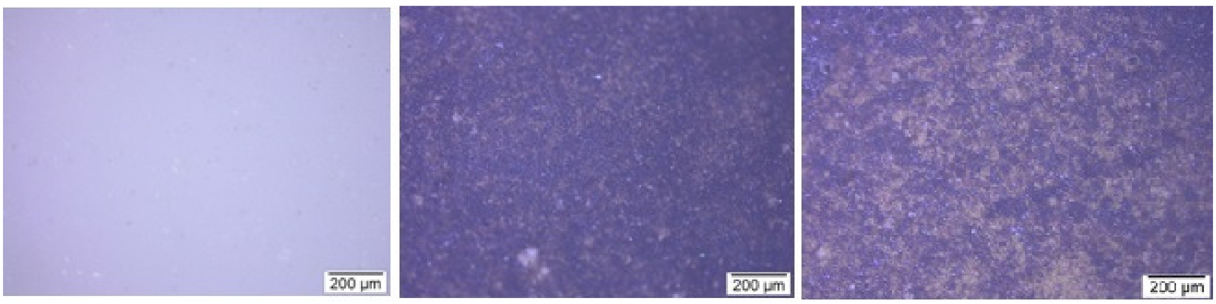


Figure 6: OM of Sample 4: before immersion in crude petroleum (a), after 90 days immersion in offshore crude petroleum (b) and after 90 days immersion in onshore crude petroleum (c).

4. CONCLUSIONS

Structural, microstructural and mechanical tests showed that composite ceramics with 20wt% TiO_2 and 3 wt% of Y_2O_3 additives presented better results in terms of mechanical hardness and microstructural characteristics. The study showed that ceramics did not present any additional phase except the constituent phases. Optical microscopy tests also showed that there is no visible change in these characteristics of ceramics after even 90 days of immersion in crude petroleum.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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