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# STUDY OF STABILITY OF AL2O3-Y2O3-ZRO2 COMPOSITE CERAMICS IN CRUDE PETROLEUM ENVIRONMENT FOR INERT COATING APPLICATIONS IN PETROLEUM INDUSTRY

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***Abstract.*** *Crude petroleum storage and transportation systems suffer from constant physical stress caused by chemical attack of crude petroleum on its structure. Ceramics are materials with high chemical stability in hostile environment and therefore can be used as an inert coating material. In the present work we have produced Al2O3-Y2O3-ZrO2 composites with high mechanical strength, through thermo-mechanical processing. To evaluate the quality of materials developed and the possibility of using them as inert protective coatings, storage and transportation systems, we have studied the physic-chemical and mechanical stability of these materials in crude petroleum originated from onshore and offshore. Structural, microstructural and mechanical tests showed that composite ceramics with 15-20wt% ZrO2 composite ceramics with 2 wt% of Y2O3 additives 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. Result of microscopy and Vickers hardness tests also showed that there is no visible change in these characteristics after even 90 days of submersion in crude petroleum. Thus we conclude that composite ceramics could be potential materials for inert coating in crude petroleum environment.*

***Keywords:*** *Al2O3-Y2O3-ZrO2, ceramic protective coating, crude petroleum, storage and transportation system*

## 1. INTRODUCTION

Rodriguez, *et al*., 2006

Discovery of Brazilian pre-salt petroleum reservoir has created an intense demand for new materials capable of withstanding direct contact with the crude petroleum. Petroleum drilling equipments, storage tanks and transportation systems suffer from constant physical stress caused by chemical attack of crude petroleum on its structure (Foster, *et al.*,1993). Ceramics are materials with high chemical stability in hostile environment and therefore can be used as an inert coating material as an alternative to resolve such problems (Moraes, *et al.*,2004). However, ceramics are highly fragile in nature. 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 (Mainier, *et al.*,1994). To date, 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 (Tafener, *et al*., 2004). To reduce vulnerability and increase strength and toughness usually the ceramics are reinforced with incorporation of one or more ceramic additives (Acchar, *et al*., 2001). Mechanical properties of alumina based ceramics improved considerably with the addition of TiO2, TiN, ZrO2 etc. as reinforcement additives. In the present work we have produced Al2O3-Y2O3-ZrO2 composites ceramics in proportions of 5-20 wt% ZrO2 and 2% wt% Y2O3 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 have studied the physic-chemical and mechanical stability of these materials in crude petroleum originated from onshore and offshore petroleum wells of Sergipe region of Brazil. Al2O3-Y2O3-ZrO2 composite ceramics were produced through thermo-mechanical processing and sintering techniques. Structural, microstructural and mechanical tests showed that composite ceramics with 15-20wt% ZrO2 composite ceramics with 2 wt% of Y2O3 additives 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. Result of microscopy and Vickers hardness tests also showed that there is no visible change in these characteristics of ceramics after even 60 days of submersion in crude petroleum. Thus we conclude that composite ceramics could be potential materials for inert coating in crude petroleum environment.

## 2. METODOLOGY

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 tablets with 8g of each sample. Each tablet was compressed using a uniaxial squeezer (Schiwing SIWA, ART6500089 model) whose cylindrical array has 3 cm in diameter were subjected to a pressure of 12 ton / cm² for a period of 5 minutes. Ethylene glycol was used to facilitate demolding therefore assists the work and does not modify the results to be easy evaporation. After pressing, the inserts were sintered to obtain the ceramic composite. This step was performed in a muffle type furnace for 24 hours at a temperature of 1380 °. After sintering, the pellets were sanded with sandpaper between 320 and 1500 grain and then were subjected to X-ray analysis, optical microscopy, Vickers micro hardness and scanning electron microscopy (SEM).

## 3. RESULTS AND DISCUSSION

### 3.1 Scanning Electron Microscopy (SEM)

After formation of the composite, microstructure analysis was performed by scanning electron microscopy, the results are shown in Fig. 1. The samples had a homogenous microstructure gradually with good grain size distribution. The composites with 15% and 20% ZrO2, showed better results in terms of homogeneity and distribution in particle size.

### 3.2 Vickers Micro Hardness

After analysis of the graph of Vickers hardness shown in Fig. 2, samples of 5% ZrO2 and 10% were discarded as they have not shown satisfactory results, because the purpose is the composite structure. However, there remain the samples with 15% and 20% ZrO2, as presented acceptable hardness values.

### 3.3 Optical Microscopy

Through optical microscopy images, shown in Fig. 3, performed before and after submersion of the tablets in crude petroleum for 60 days, it was found 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.

### 3.4 X-Ray Diffraction

#### 3.4.1 Before petroleum immersion

Structural characteristics and identification phases were analyzed by X-Ray diffraction (XRD). Figure 4 shows typical XRD Al2O3- 20%ZrO2 composite reinforced with 2% of Y2O3. The ceramic composite showed no additional stage, only the constituent phases, Al2O3, ZrO2 and Y2O3. The presence of Y2O3 phase is slightly observed in the XRD patterns because of its small amount percentage in the composite.

#### 3.4.2 After petroleum immersion

Comparing the graphs before XRD (Fig. 4) and after (Figure 5) of the composite immersion 20% ZrO2 - Al2O3 reinforced with 2% Y2O3 shore oil and sea, we concluded that there was no chemical reaction between oil and the ceramic composite since there is no presence of other phases peaks.

### 3.5 Figures and tables

| Composition | Mix 5% $ZrO_2$ | Mix 10% $ZrO_2$ | Mix 15% $ZrO_2$ | Mix 20% $ZrO_2$ |
|---|---|---|---|---|
| $Al_2O_3$ | 93 | 88 | 83 | 78 |
| $ZrO_2$ | 5 | 10 | 15 | 20 |
| Y2O3 | 2 | 2 | 2 | 2 |

Table 1: Composition of ceramic samples

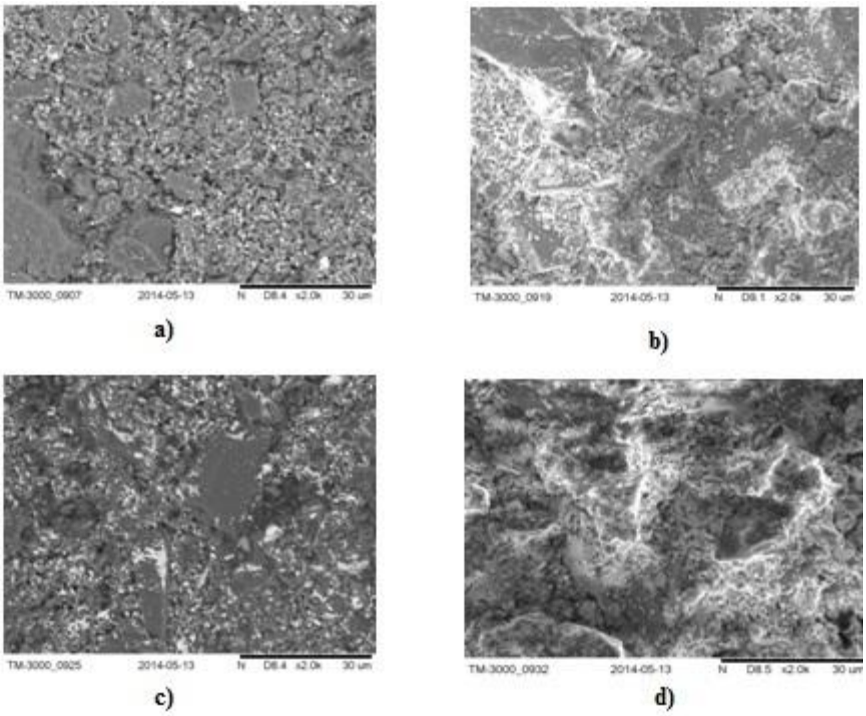


Figure 1: SEM images with a) 5% ZrO2, b) 10% ZrO2, c) 15% ZrO2 and d) 20% ZrO2.

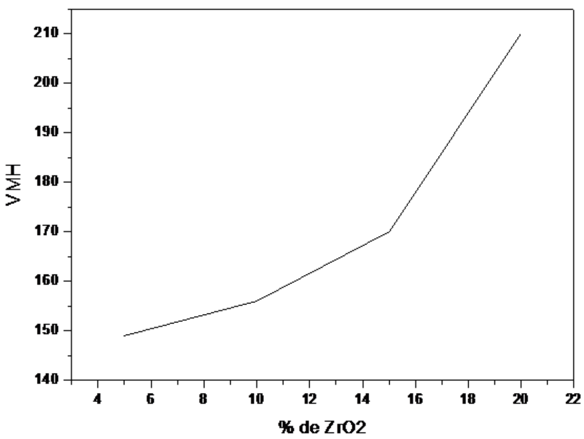


Figure 2: Vickers Micro Hardness of Al2O3- ZrO2 composite reinforced with 2% Yttria.

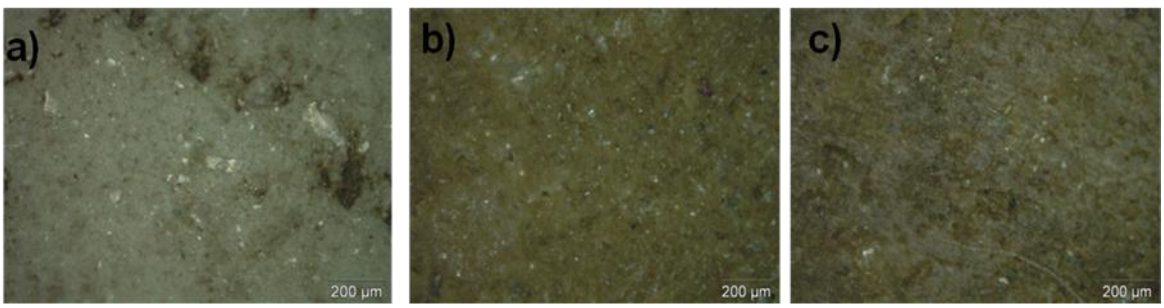


Figure 3: Optical Microscopy images  a) sample 3 before immersion; b) Sample 3 after immersion in sea oil; c) sample 3 after immersion in land oil. .

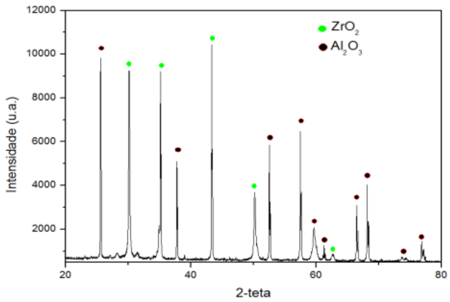


Figure 4: DRX of sample 4.

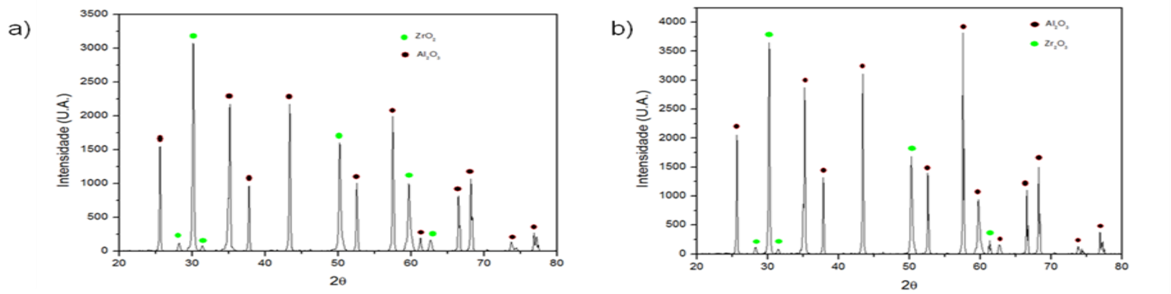


Figure 5: a) DRX of sample 4 after immersion in sea oil; b) of sample 4 after immersion in land oil.

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## 5. RESPONSIBILITY NOTICE

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