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# DEVELOPMENT AND MANUFACTURE OF INERT COATING TO CRUDE OIL BASED IN CERAMIC COMPOSITE $ZrO_2$ - $TiO_2$

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**Abstract.** In the petroleum industry it is necessary to store and transport crude oil, which is done by means of metallic materials tanks. Crude oil is a corrosive product and surface degradation of these tanks is serious problem for the oil industry. In this way, we found alternative solution to the issue is to use inert coating and corrosion that ceramic materials are in favor such as shielding materials, for support, more efficiently, the corrosion process.  $ZrO_2$  have mechanical properties superior to other ceramic oxides and refined microstructure, TZP, further elevates its properties. Studies have reported that  $TiO_2$  with earth oxides are sources of TZP materials with improved properties, such as toughness. This work aims to produce ceramic composites from zirconium oxide and titanium, the  $ZrO_2$ - $TiO_2$  type, for thermomechanical process, with variations from 5% to 20%  $TiO_2$  (by weight) of zirconia and reinforced with  $Y_2O_3$ . Then, the composites undergo milling process for 24 hours and cold pressing to be then sintered at a temperature of  $1200^{\circ}C$   $1700^{\circ}C$ . Subsequently go through microstructural analysis, structural, chemical and mechanical. They will be tested in a hostile environment of crude oil and then their properties will be reassessed to observe the behavior of ceramic composites.

**Keywords:** ceramic composite,  $ZrO_2$ - $TiO_2$ , coating, metal parts, oil, rare earth oxide.

## 1. INTRODUCTION

Crude oil is a very corrosive product, so the surface degradation of metal tanks used to transport and store crude oil is a serious problem in the oil industry.

A great material for this application would be one that presented characteristics of high mechanical strength, chemical stability and fracture toughness combined with good resistance to wear, this combination is found in yttria-stabilized zirconia-titania based composites because we ensure that the zirconia remains in its best microstructure, which consists of a metastable tetragonal phase, TZP, Wu *et al.*, 2004 and Garvie *et al.*, 1975.

From this, we aim to develop a composite zirconia-titania ceramic incorporated with yttria that exhibits the characteristics of high tenacity, high mechanical resistance and resistance to hostile environment of crude oil. Then the ceramics with certain characteristics will be used as coating in manufacturing system to store and transport crude oil.

## 2. EXPERIMENTAL PROCEDURE

Initially from the percussion powders  $ZrO_2$ ,  $TiO_2$  and  $Y_2O_3$ , in the proportion of 5 to 20% (by weight) of  $TiO_2$  and 1% of  $Y_2O_3$  with the complement of  $ZrO_2$ , we formed our composites. The ceramic powders were mixed in a high energy mill for 24 hours.

The next step was to form a hydraulic press in a 20 mm diameter die, receiving a force of 12 tons for 5 minutes to stabilize the load. After, were sintered in a muffle furnace for 24 hours at a temperature of  $1350^{\circ}C$ .

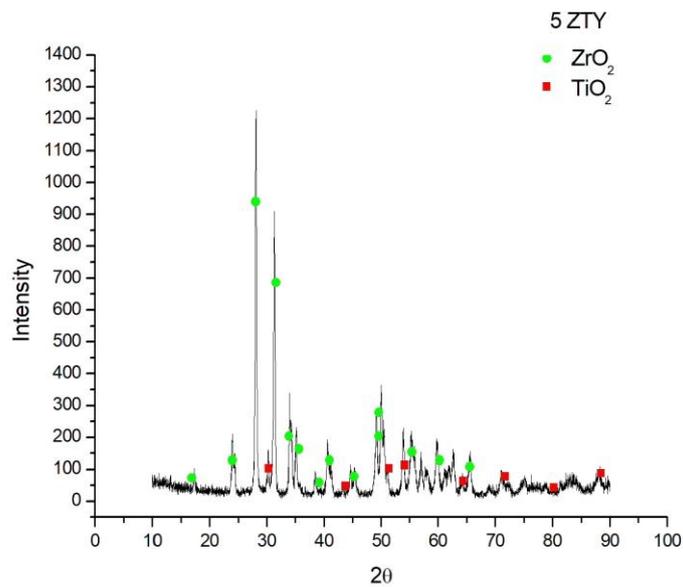
The test bodies were analyzed by X-ray diffraction, Scanning electron microscopy, Optical Microstructure and Vickers Microhardness test, and finally Stability Test in a hostile crude oil environment for 60 days.

From the results we could analyze if the composite can be used as a coating for our aforementioned application.

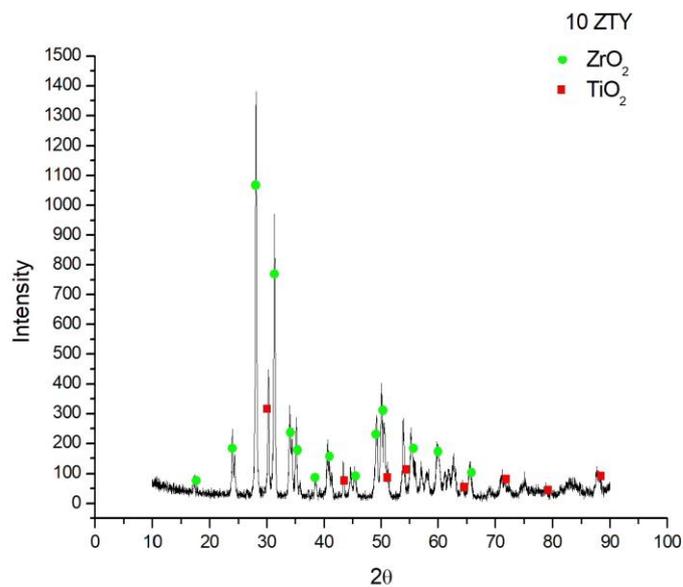
### 3. RESULTS AND DISCUSSION

#### 3.1 X-ray Diffraction

In Fig. 1 (a), (b), (c) and (d) is possible to observe the similarity between each group, considering that all are formed of the same raw material only with variations in the zirconia and titania oxide contents, which explains the differences in the intensity of the peaks.

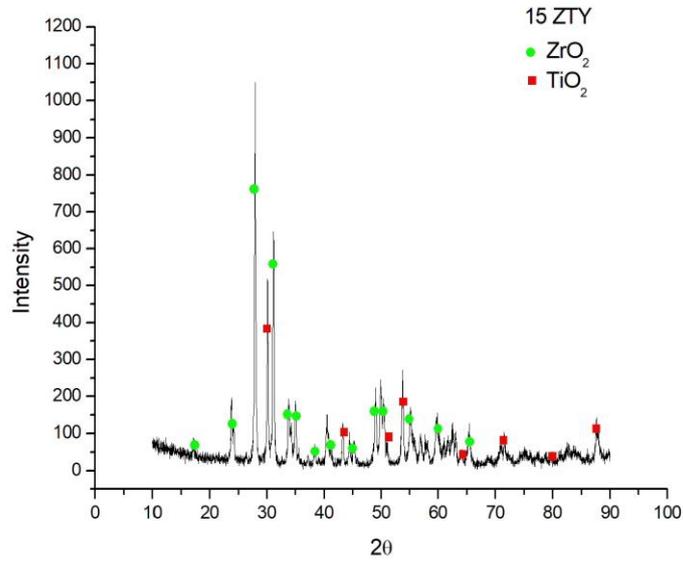


(a)

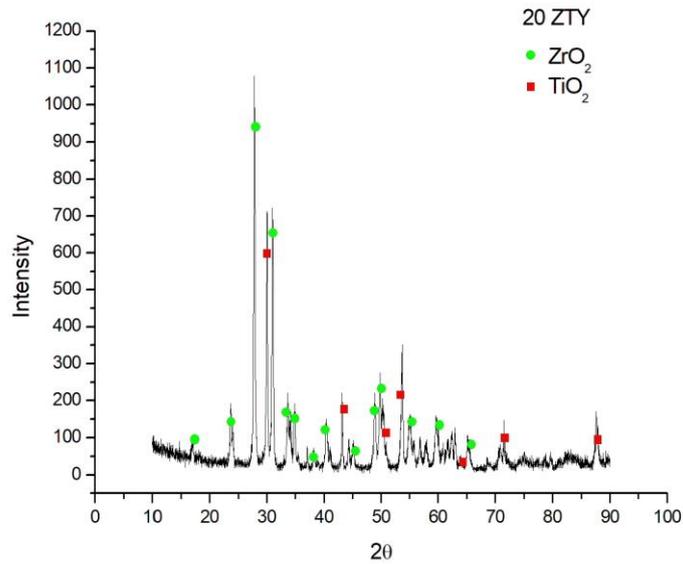


(b)

Figure 1. XRD results (a) 5%, (b) 10%



(c)



(d)

Figure 1. XRD results (c) 15%, (d) 20%

### 3.2 Vickers Microhardness Tests

The best hardness results were from the 5ZTY sample, with 5,0% TiO<sub>2</sub>, with a mean hardness of 609.86HV (standard deviation of 17,42), the other samples had satisfactory hardness also, varying between 494,47HV (deviation Standard 22,54) for ceramic 20ZTY and 541,64HV (standard deviation 38,81) for ceramic 10ZTY, Fig. 2.

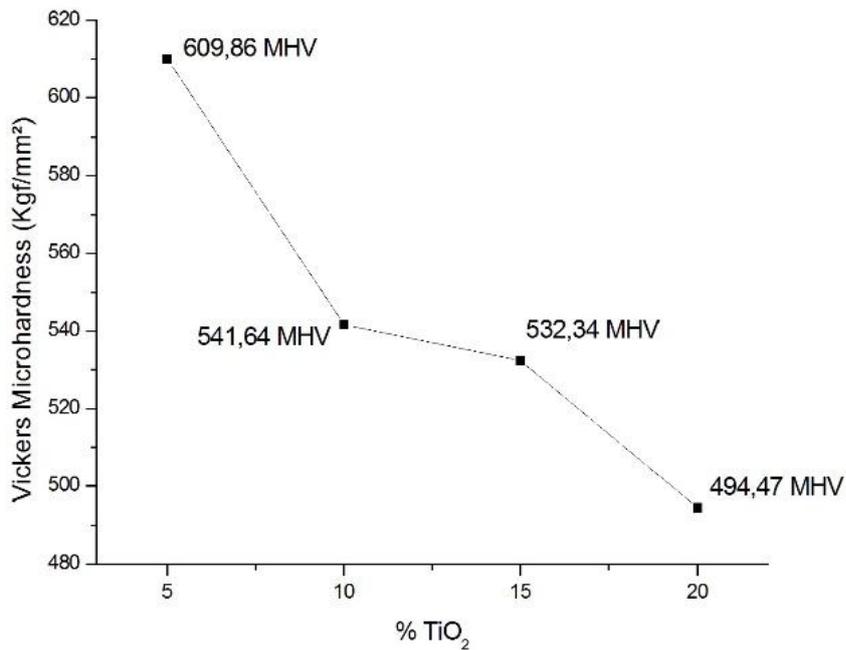


Figure 2. Comparison of Vickers Microhardness results on ZrO<sub>2</sub>-TiO<sub>2</sub> ceramic composites.

### 3.3 Scanning Electron Microscopy

The results obtained by SEM analysis, shown in Fig. 3 (a), (b), (c) and (d), illustrate good distribution and particle size homogeneity of the sintered composites at 1350°C for 24 hours. It is observed that with the increase of TiO<sub>2</sub>, the grain (Fig. 3 (d) - 20% TiO<sub>2</sub>) refinement gave better homogeneity in the distribution of the particles compared to the figure that shows the lowest amount of 5% TiO<sub>2</sub> (Fig. 3 (a) – 5% TiO<sub>2</sub>).

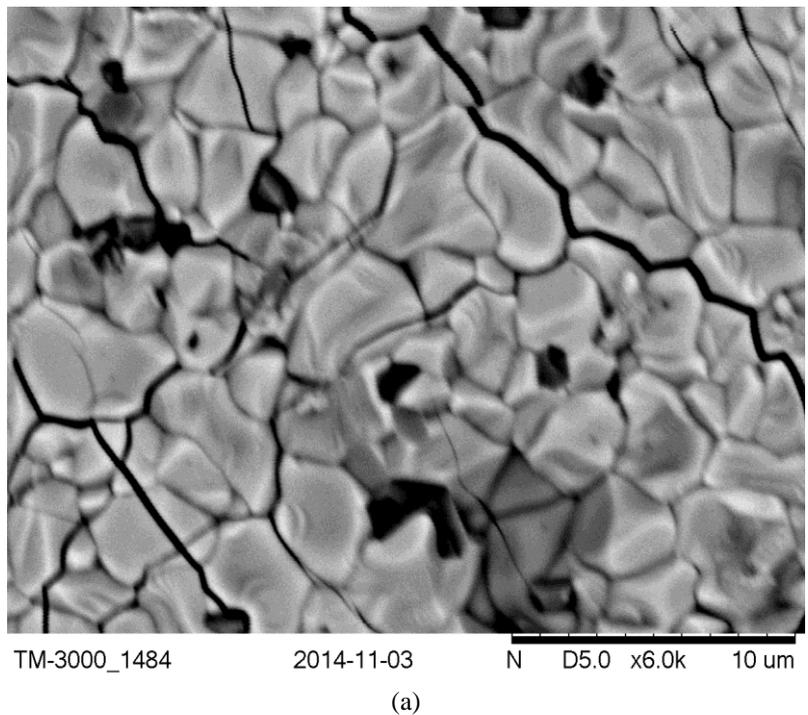
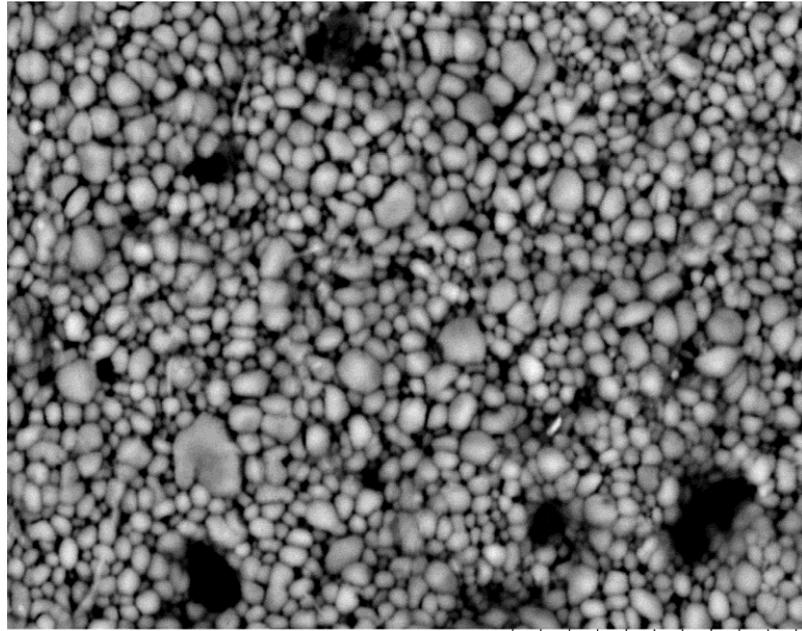
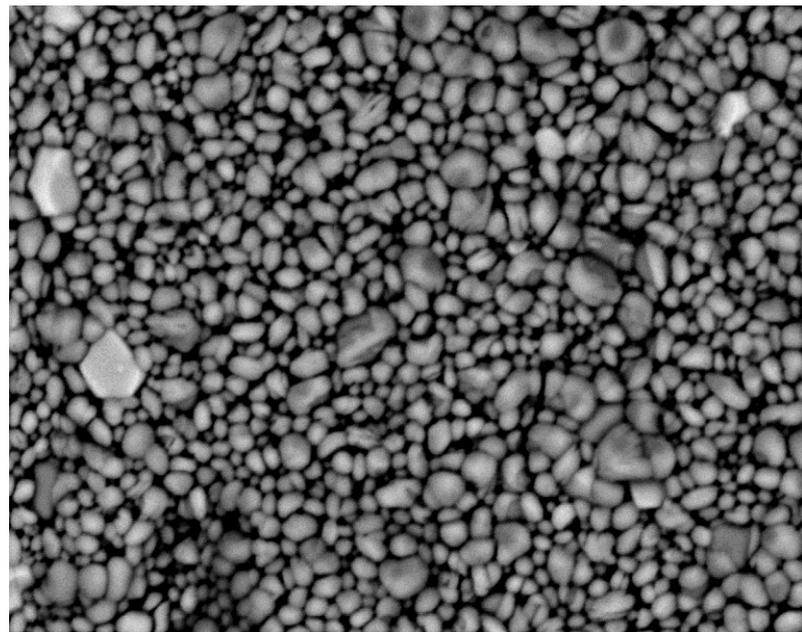


Figure 3. SEM with 1000x magnification (a) 5% TiO<sub>2</sub>

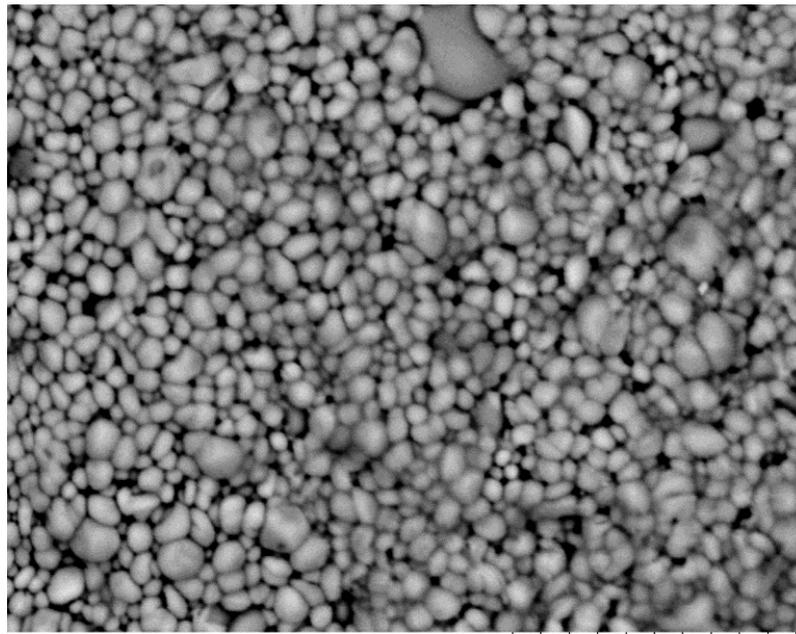


TM-3000\_1512 2014-11-03 N D4.5 x6.0k 10 um  
(b)



TM-3000\_1495 2014-11-03 N D4.4 x6.0k 10 um  
(c)

Figure 3. SEM with 1000x magnification (b) 10% TiO<sub>2</sub> , (c) 15% TiO<sub>2</sub>



TM-3000\_1503 2014-11-03 N D5.1 x6.0k 10 um

(d)

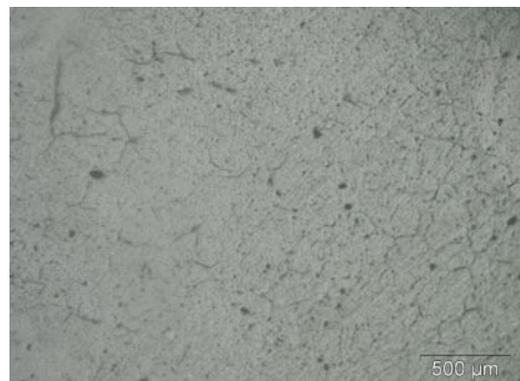
Figure 3. SEM with 1000x magnification (d) 20%  $TiO_2$

### 3.4 Optical Microstructure

Observing the images obtained by optical microscopy and exposed in Figs. 4-7, it was verified that there was no significant change in the surface of the composites in direct contact with petroleum, that is, no fissures were observed, or any evidence of chemical attack by the Crude oil, which indicates a good chemical stability for the adopted period, 60 days.



(a)



(b)



(c)

Figure 4. OM of the sample 5%  $TiO_2$  (a) before the immersion in oil, (b) after immersion in sea oil, (c) after immersion in soil oil

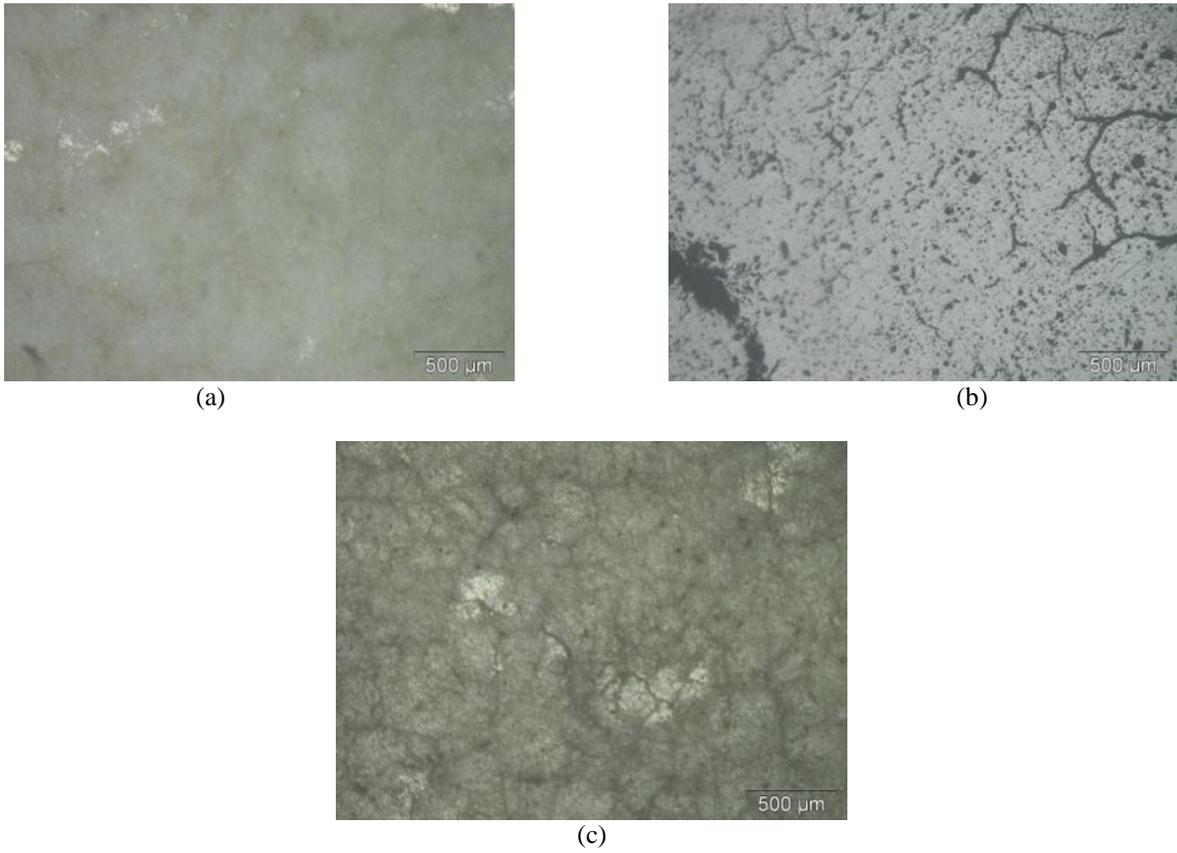


Figure 5. OM of the sample 10% TiO<sub>2</sub> (a) before the immersion in oil, (b) after immersion in sea oil, (c) after immersion in soil oil

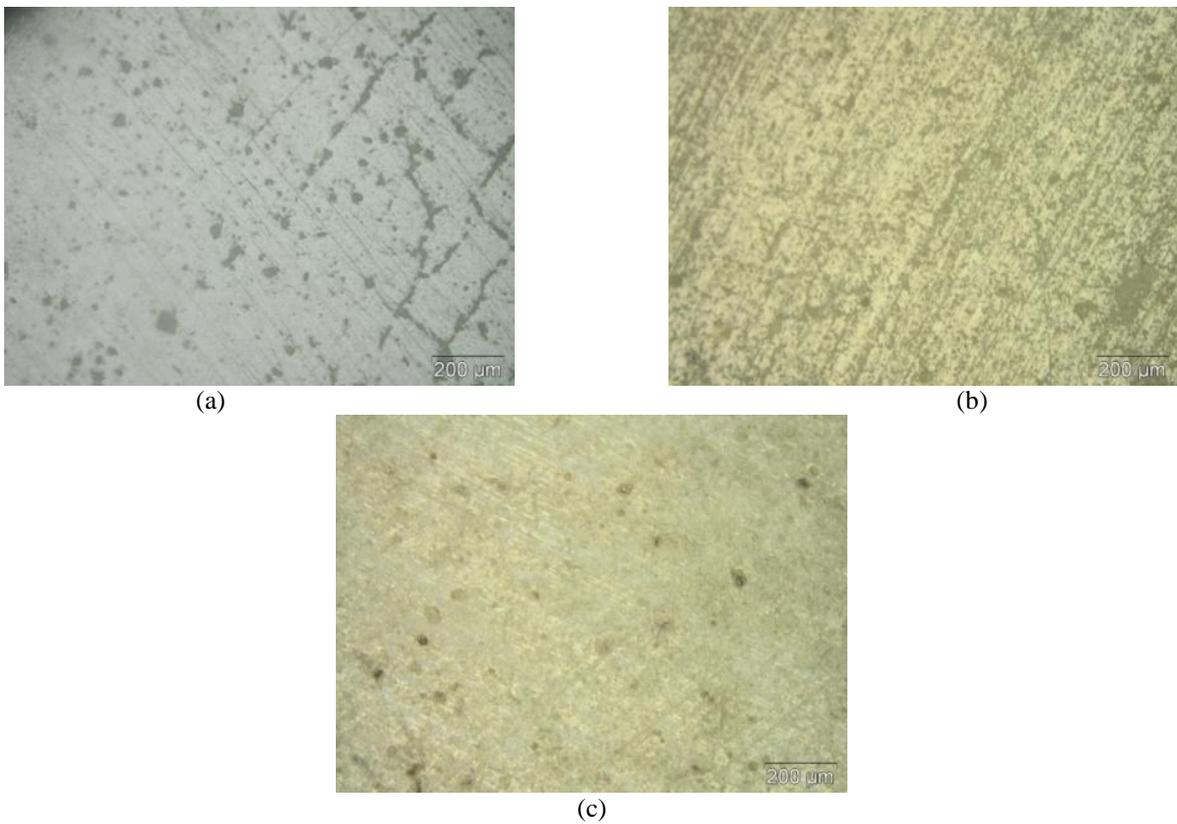


Figure 6. OM of the sample 15% TiO<sub>2</sub> (a) before the immersion in oil, (b) after immersion in sea oil, (c) after immersion in soil oil

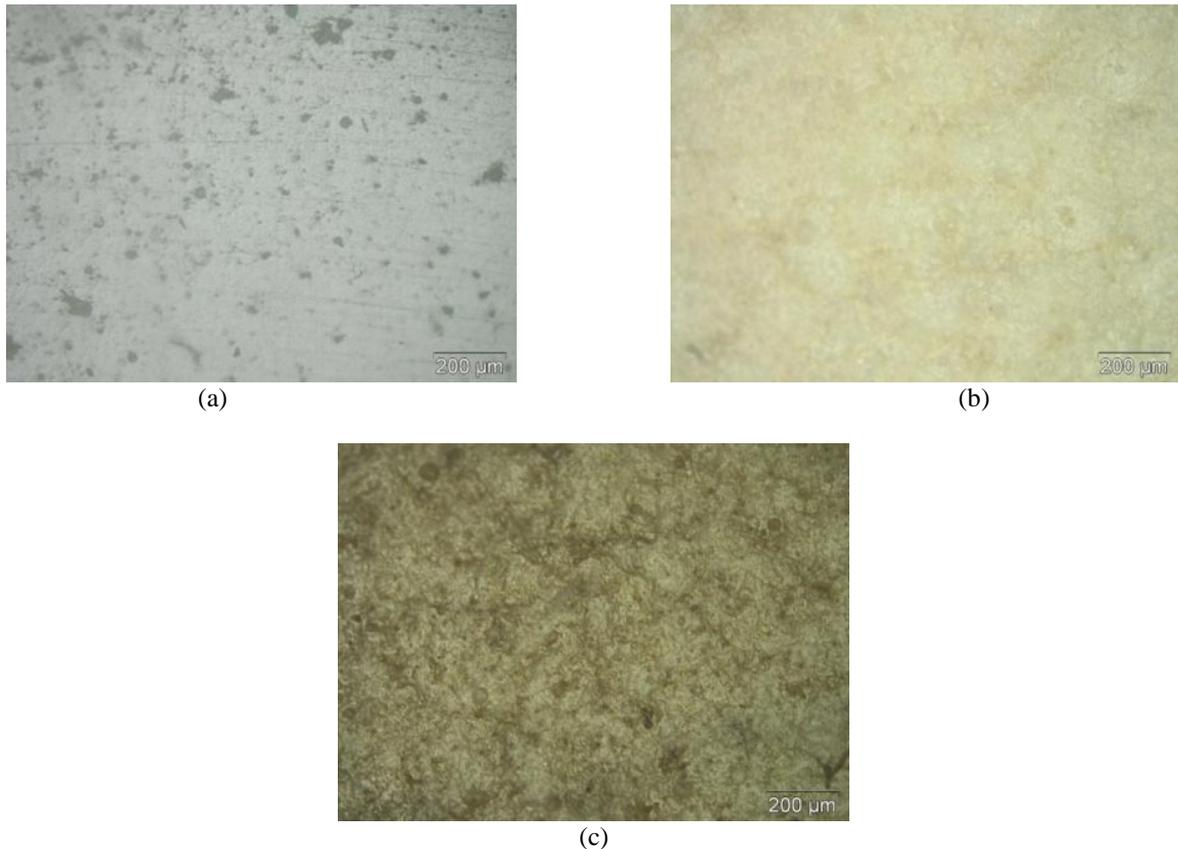


Figure 7. OM of the sample 20%  $TiO_2$  (a) before the immersion in oil, (b) after immersion in sea oil, (c) after immersion in soil oil

#### 4. CONCLUSIONS

According to the XRD results after sintering, the formation of the  $ZrO_2-TiO_2$  ceramic composite with  $Y_2O_3$  reinforcement is observed. The mechanical properties were evaluated by the micro-hardness vickers, and these are satisfactory of the four composites, obtaining hardness between 494,47 and 609,86 MHV. Through the SEM, we conclude that with the increase of  $TiO_2$  the grain refinement is obtained.

#### 5. ACKNOWLEDGEMENTS

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#### 6. REFERENCES

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#### 7. RESPONSIBILITY NOTICE

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