

# Production of $Ba_2AlNbO_6$ Ceramics and Study of their Stability in Crude Petroleum for the Conservation of Metallic Sensing Elements Used in Petroleum Extraction

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In the present work niobium based complex cubic perovskite oxide  $Ba_2AlNbO_6$  ceramics were produced, characterized and their stability in crude petroleum environment was studied for inert ceramic embedding used in petroleum extraction.  $Ba_2AlNbO_6$  ceramic powder was prepared by means of thermo-mechanical processing. Structural characteristics of calcined material were investigated by powder X-ray diffractometry, which presented a single phase complex cubic perovskite structure with lattice parameter  $a = 8.3418 \text{ \AA}$ . Compacted  $Ba_2AlNbO_6$  ceramics were sintered at  $1150^\circ\text{C}$  during 72 hours in ambient atmosphere. The microstructure of the sintered ceramics was studied by scanning electron microscopy and mechanical behavior was studied by Vicker's micro-hardness testing. The sintered ceramics were submerged in crude petroleum for 60 days. The ceramics were taken out from petroleum periodically and subjected to structural, microstructural and mechanical characterizations. The results showed that the ceramics submerged in crude petroleum did not suffer any change at any stage of submersion. These characteristics make this material suitable for inert ceramic embedding for sensors used in petroleum extraction.

**Keywords:**  $Ba_2AlNbO_6$  production, microstructure, micro-hardness testing, stability in crude petroleum.

## 1. Introduction

Nowadays, there is an increasing demand for sustainable materials and systems to operate in hostile environments such as high temperature or chemically aggressive environments of petroleum extraction industry. In petroleum extraction, different types of sensors are required to monitor temperature, pressure and other vital parameters in the petroleum wells. These sensors work in extremely hostile environment. In case of temperature sensors, normally, sensing elements are metals Pt, Nb etc., which are highly sensitive to environmental conditions. Accordingly, these sensors need to be embedded in materials highly inert to petroleum extraction environment. Ceramics are parts of a broad class of materials whose extremely vast characteristics such as, high thermal capacity, resistance to corrosion, fact that they may be insulating, conducting or superconducting, they have magnetic properties or absence of magnetism, and to be hard and resistant but fragile, make them interesting for various technological applications.<sup>1-3</sup>

Study of advanced ceramics makes it possible to develop new technologies, which could be inaccessible with the

conventional materials and also to substitute scarce materials. Their cast of production turns to be more viable than those of metals and metallic alloys, as they are lighter materials and stable at higher temperatures.<sup>4,5</sup> Earlier research works in 1950 and 1960's<sup>6,7</sup> identified a large group of ceramics, which have the basic  $ABO_3$  perovskite structure or a small distortion of that structure. These complex perovskite oxides generally have the formula  $A_2BB'O_6$  or  $A_3B_2B'O_9$  and result from the ordering of B and B' cations on the octahedral sites of the basic perovskite unit cell. Due to the increased complexity of the unit cell, a large variety of such materials are possible and hence a more continuous progression of lattice parameter could be produced.<sup>7-12</sup> Due to high stability in hostile environment, many of the new technologies incorporate perovskite structure based ceramics in their manufacture activities for such applications. We are working on fabrication of ceramic encapsulated temperature sensor for petroleum extraction industry<sup>13-15</sup> and in the present work we have developed and produced niobium based complex cubic perovskite oxide  $Ba_2AlNbO_6$  ceramics and studied their stability in crude petroleum environment for the fabrication

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of inert ceramic embedding for temperature sensors used in petroleum extraction.

## 2. Experimental Details

Ba<sub>2</sub>AlNbO<sub>6</sub> ceramic powder was prepared through thermo-mechanical solid-state processing route. Stoichiometric ratios of high purity (99.99 %) constituent oxides BaCO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, and Nb<sub>2</sub>O<sub>5</sub> were mixed and homogenized by means of a high energy ball mill using steel cylinder and alumina balls. Homogenized mixture was compacted as circular discs of 30 mm diameter with a thickness of about 4 - 5 mm. Uniaxial powder compaction was carried out in a hydraulic press and hard steel mould made of abrasion resistant AISI A2 steel (HRC 58) at a pressure of 4 ton/cm<sup>2</sup>. The compacted discs were calcined in a high temperature muffle furnace (Muffle Furnace Jung model 0614) at a temperature of 1150°C for 48 hours in ambient atmosphere. After calcination, samples were furnace cooled to room temperature. The calcined material was subjected to X-ray powder diffraction (XRD) analysis for structural characterization and phase identification studies. X-ray diffraction patterns were recorded by using a Shimadzu, X-ray powder diffractometry, equipped with Cu - K<sub>α</sub> radiation ( $\lambda = 1.5406\text{\AA}$ ). After the structural analysis and identification of Ba<sub>2</sub>AlNbO<sub>6</sub> phase formation, calcined Ba<sub>2</sub>AlNbO<sub>6</sub> material were again subjected to grinding in high energy ball for 24 hours<sup>16,17</sup> for ceramic fine powder production and homogenization of particle size distribution.

For the fabrication of ceramic components and sintering of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics, circular discs of 15 mm diameter and 3 mm thickness were produced by uniaxial pressing technique using the same above referred mould and a hydraulic press at a pressure load of 12 ton/cm<sup>2</sup>. For better compression, each fraction of pressure was applied for 5 minutes to stabilize the pressure load distribution in the compact and homogenize the necessary pressure load. The compressed ceramic discs were subjected to normal solid state sintering process at a temperature of 1150°C for 72 hours in ambient atmosphere, using the same above referred high temperature muffle furnace and finally furnace cooled to room temperature. As sintering is a thermo-dynamic process and due to furnace temperature limitation, it was necessary to increase the sintering time to achieve the sintered body.<sup>17</sup>

Microstructural analysis of the sintered Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics was carried out by scanning electron microscopy (model HITACHI TM3000), using backscattered electrons. To observe the microstructure, the samples were placed in a metal coating device to receive a 9 micronthin layer of gold, since they are not electrically conducting, to get SEM images. Mechanical properties of the sintered ceramics were studied through Vicker's micro-hardness testing. For the indentation in Vicker's micro-hardness tester, the samples were polished with # 360, # 400, # 600, # 1200, # 1500 grade sand papers, followed by mechanical polishing using diamond paste with

1 micron particle size. For this testing a Vicker's hardness indenter model HVS-5 No 0021 was used. The Vickers micro - hardness (MHV) is given by equation.<sup>18</sup>

$$MHV = 1.8544P/d^2 \quad (1)$$

Where  $P$  is the load and  $d$  is the average diagonal of the square indentation produced by the pyramidal indenter in the sample.

For the study of stability of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics in petroleum extraction environment, the sintered ceramics were submerged in crude petroleum for 60 days. Crude petroleum for this study, extracted from the earthen and offshore petroleum wells of the North-east region of Brazil, was provided by PETROBRAS, Brazil. The Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics were periodically taken out from the petroleum reservoir every 15 days and subjected to X-ray diffractometry, optical microscopy and Vicker's micro-hardness testing to observe if there were any changes in their structural and mechanical characteristics due to crude petroleum environment.

## 3. Results and Discussion

The XRD spectrum of a typical Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics produced in this work is shown in Fig. 1. It consists of strong peaks characteristics of primitive cubic perovskite structure plus few weak reflection lines arising from the superlattice reflections. No evidence for a distortion from the cubic symmetry is observed in the XRD spectrum. The basic perovskite composition is ABO<sub>3</sub>, where A is a large ion suitable to the 12-coordinated cube-octahedral sites and B is a smaller ion suitable to the 6-coordinated octahedral site. Complex perovskite with mixed species on a site (particularly the B site) may be represented by multiples of this formula unit and a larger unit cell, e.g. A<sub>2</sub>BB'O<sub>6</sub>, A<sub>3</sub>B<sub>2</sub>B'O<sub>9</sub> etc.<sup>21-23</sup> Thus, in Ba<sub>2</sub>AlNbO<sub>6</sub> composition, Ba<sup>2+</sup>, with largest ionic radius (1.35 Å) occupies position A, Al<sup>3+</sup> (ionic radius 0.55 Å) and Nb<sup>5+</sup> (ionic radius 0.70 Å) cations occupy B and B' positions on the B site due to their smaller ionic radii compared

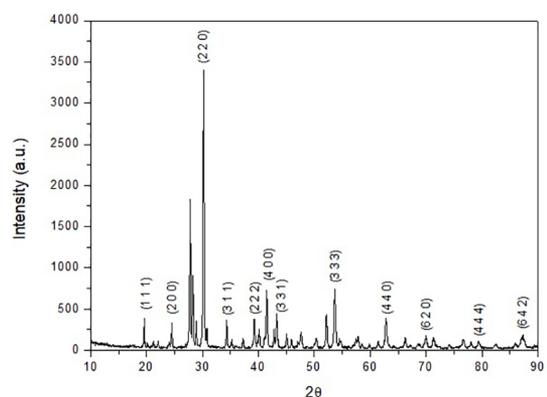


Figure 1. X-ray diffraction pattern of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics

to that of Ba<sup>2+</sup> cation. Due to the ordering of B and B' on octahedral site of the ABO<sub>3</sub> unit cell there is a doubling in the lattice parameter of the basic cubic perovskite unit cell. Thus, the whole XRD pattern of Ba<sub>2</sub>AlNbO<sub>6</sub> can be indexed in a A<sub>2</sub>BB'O<sub>6</sub> cubic cell with the cell edge  $a = 2a_p$  where  $a_p$  is the cell lattice of the cubic perovskite. The XRD spectrum of Ba<sub>2</sub>AlNbO<sub>6</sub> is similar to A<sub>2</sub>BB'O<sub>6</sub> type complex cubic perovskite oxides e.g. Ba<sub>2</sub>YNbO<sub>6</sub>, Ba<sub>2</sub>ErSbO<sub>6</sub>, Ba<sub>2</sub>DyNbO<sub>6</sub> etc. reported in the ICDD files, as judged by the similarity in d-spacings and intensity ratios. Experimental XRD data of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics is presented in Table 1.

Presence of the superstructure reflection lines (111) and (333) in the XRD spectrum of Ba<sub>2</sub>AlNbO<sub>6</sub> is the signature of an ordered complex cubic perovskite structure. In a substitutional solid solution BB', there is a random arrangement of B and B' on equivalent lattice positions in the crystal structure. Upon suitable heat treatment, the random solid solution rearranges into a structure in which B and B' occupy the same set of positions but in a regular way, such a structure is described as superstructure. In the superstructure, the positions occupied by B and B' are no longer equivalent and this feature is exhibited in the XRD spectrum of the material by the presence of superstructure reflection lines.<sup>19,20</sup>

For double cubic perovskite of the formula A<sub>2</sub>BB'O<sub>6</sub> the intensity, in particular of the (111) and/or (311) superstructure reflection, is proportional to the difference in scattering power of the B and B' atoms, when all the atoms are situated in the ideal position. A disordered arrangement of B and B' should result in zero intensity. Therefore Al<sup>3+</sup> and Nb<sup>5+</sup> cation ordering in Ba<sub>2</sub>AlNbO<sub>6</sub> in B and B' positions is clearly distinguished by the presence of the significant intensity of (111) and (333) superstructural reflection lines. Based on above discussion we have now indexed the XRD peaks of Ba<sub>2</sub>AlNbO<sub>6</sub> as an ordered complex cubic perovskite with A<sub>2</sub>BB'O<sub>6</sub> crystal structure. The lattice parameter of Ba<sub>2</sub>AlNbO<sub>6</sub>, calculated from the experimental XRD data, is  $a_{exp} = 8.3418 \text{ \AA}$ .

For the A<sub>2</sub>BB'O<sub>6</sub> type complex cubic perovskite oxides, theoretical value of the unit cell parameter can be calculated using following Shannon and Prewit mode<sup>21</sup>:

$$a_A = \frac{2(R_A + R_O)}{\sqrt{2}} \quad (1)$$

$$a_B = R_B + R_{B'} + 2R_O \quad (2)$$

$$a_{cal} = \frac{(a_A + a_B)}{2} \quad (3)$$

Where, R<sub>A</sub>, R<sub>B</sub>, R<sub>B'</sub> and R<sub>O</sub> are ionic radius of A, B, B' cations and oxygen, respectively. Using these equations theoretical value of unit cell parameter of Ba<sub>2</sub>AlNbO<sub>6</sub> is  $a_{cal} = 7.9991 \text{ \AA}$ . Experimental value of unit cell parameter of Ba<sub>2</sub>AlNbO<sub>6</sub> ( $a_{exp} = 8.3418 \text{ \AA}$ ) is 4.39% higher than its theoretical value ( $a_{cal} = 7.9991 \text{ \AA}$ ), which can be justified because Shannon and Prewit model<sup>24</sup> is based on hard-sphere model approximation.

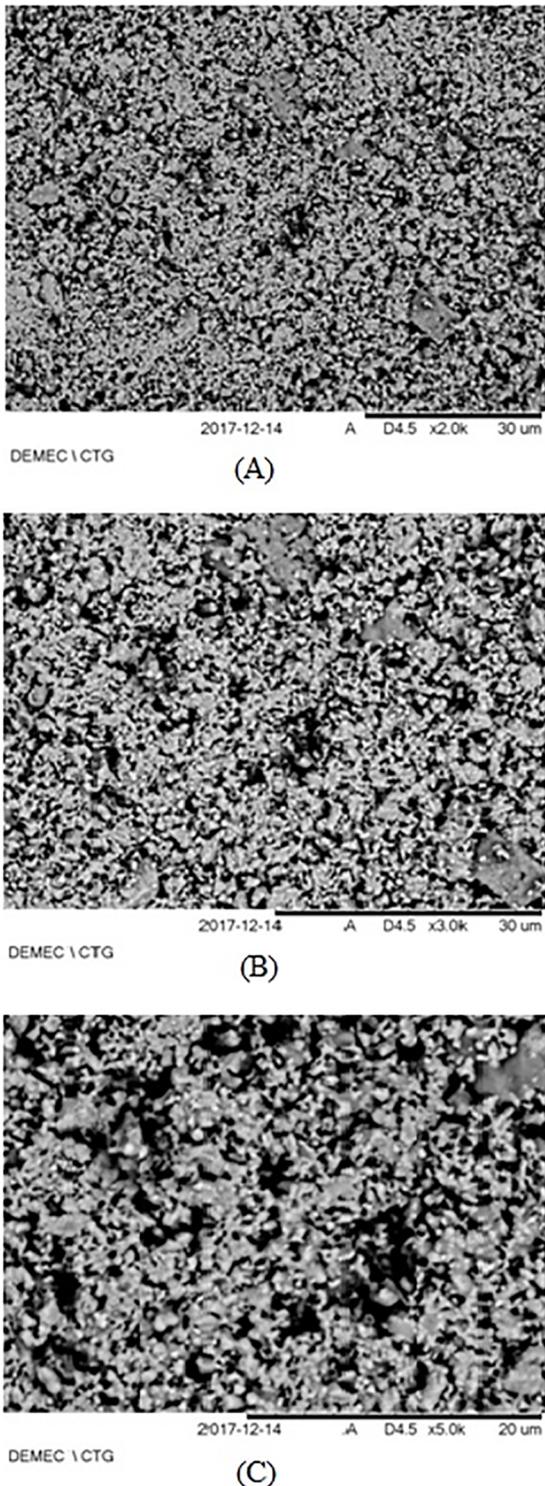
As stated earlier, the objective of this work is to produce Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics with good mechanical strength in order to guarantee the required qualities that a final ceramic product must have for structural applications. Production and functional ability of polycrystalline ceramic products are highly dependent on their microstructural features, which in turn are highly influenced by sintering kinetics. Sintering process uses the heat and mechanisms of material transport to convert after ceramic into dense polycrystalline solids. The driving force in the sintering process is obtained by the reduction of the total surface energy, which increases the contact and growth between the grains. The smaller grains are transformed into the bigger grains and, consequently, the pores substituted by the solid materials. The necessity to get uniformity in the microstructure is for preventing the creation of tensions that make to appear (or they magnify) empty spaces, for being concentrative of tensions, assist in the propagation of cracks and micro-cracks in the sintered body. Microstructural features define the final product quality of the ceramic products and their mechanical strength.

Microstructural features of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics sintered at 1150°C temperatures for 72 hours, was studied by scanning electron microscopy and Fig. 2 presents typical microstructures of these ceramics. As seen from these figures, sintering temperature has a significant role in sintering behavior of these ceramics. Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics sintered at 1150°C presented good microstructures with uniform surface morphology, grain homogeneity and particle size distribution.

Mechanical properties of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics sintered at 1150°C were evaluated by Vicker's microhardness tests. These tests were performed on 15 polished discs of Ba<sub>2</sub>AlNbO<sub>6</sub> ceramics with 10 indentation on each discs to obtain the arithmetic mean value of  $(56.43 \pm 4.45) \text{ KgF/mm}^2$ .

**Table 1.** XRD data of Ba<sub>2</sub>AlNbO<sub>6</sub>.

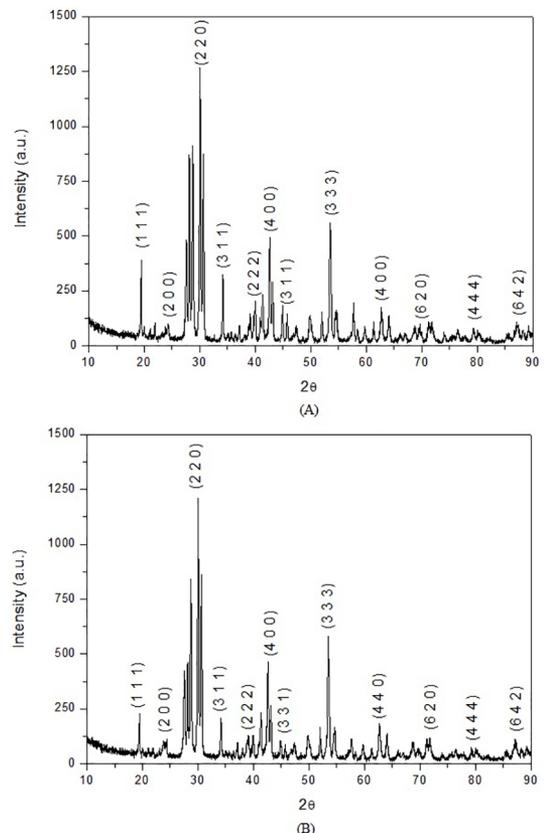
S. N.	2θ	d(Å)	h k l
1	19.62	4.5367	1 1 1
2	24.52	3.6402	2 0 0
3	30.20	2.9673	2 2 0
4	34.32	2.6200	3 1 1
5	39.28	2.3000	2 2 2
6	41.48	2.1828	4 0 0
7	43.28	2.0961	3 3 1
8	53.66	1.7127	3 3 3
9	62.80	1.4837	4 4 0
10	69.98	1.3480	6 2 0
11	79.38	1.2104	4 4 4
12	87.30	1.1199	6 4 2



**Figure 2.** SEM micrographs of  $\text{Ba}_2\text{AlNbO}_6$  ceramics sintered at  $1150^\circ\text{C}$  with magnification of: (A) 2,000X (B) 3,000X and (C) 5,000X

For the study of stability of these ceramics in petroleum extraction environment, we used  $\text{Ba}_2\text{AlNbO}_6$  discs sintered at  $1150^\circ\text{C}$  as these discs presented good microstructural features and mechanical hardness. These discs were firstly polished like mirror surface and subjected to optical microscopy. After observation in optical microscopy these discs were partitioned in two equal parts, using diamond disc cutter, to be submersed each one part in the crude petroleum extracted from earthen and offshore petroleum wells, respectively. This was carried out purposely to ensure that ceramics submersed in crude petroleum extracted from earthen and offshore petroleum wells have the same microstructure and mechanical hardness. The ceramics were submersed in crude petroleum for 60 days. The ceramics were taken out from the petroleum reservoir periodically, every 15 days and tested to observe if there are any changes in their structural, microstructural and mechanical characteristics due to crude petroleum environment.

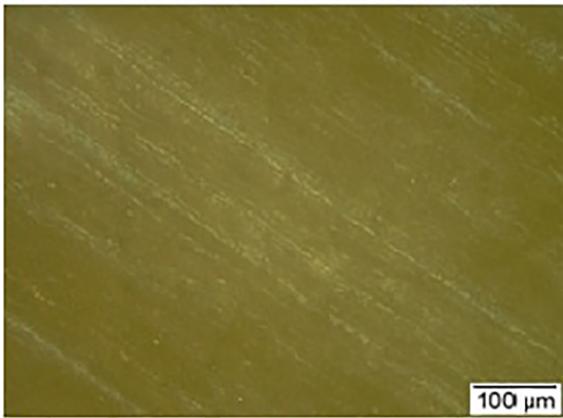
The XRD spectra of  $\text{Ba}_2\text{AlNbO}_6$  ceramics before submersion in crude petroleum, as seen from Fig. 3, along with typical results of X-ray diffractometry carried out on  $\text{Ba}_2\text{AlNbO}_6$  ceramics after 60 days of submersion in earthen and offshore crude petroleum are shown in Fig. 4 both the XRD spectrum of  $\text{Ba}_2\text{AlNbO}_6$  ceramics submersed in



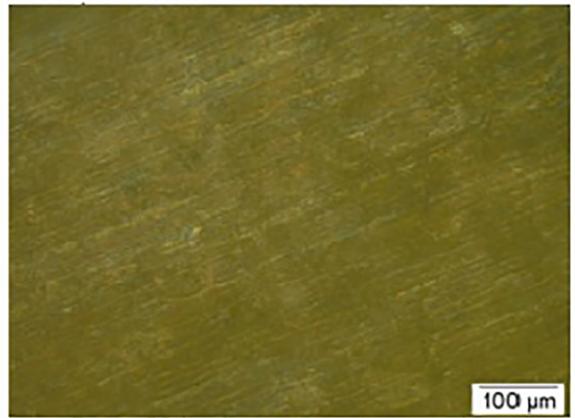
**Figure 3.** X-ray diffraction patterns of  $\text{Ba}_2\text{AlNbO}_6$  ceramics: (A) after 60 days submersion in earthen crude petroleum and (B) after 60 days submersion in offshore crude petroleum



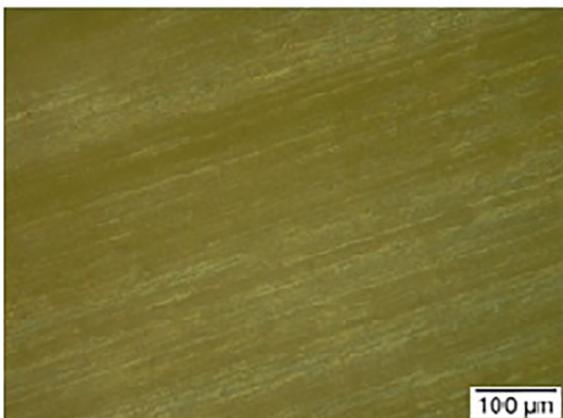
(A)



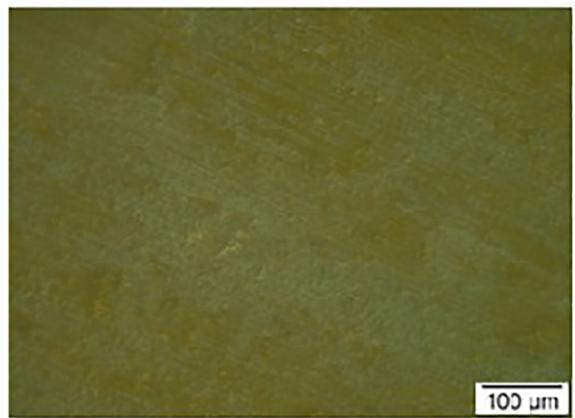
(B)



(C)



(D)



(E)

**Figure 4.** Optical micrograph (200X) of  $Ba_2AlNbO_6$  ceramics before and after submersion in crude petroleum: (A) Before submersion in crude petroleum, (B and C) after 45 and 60 days submersion, respectively, in earthen crude petroleum and (D and E) after 45 and 60 days submersion, respectively, in offshore crude petroleum

earthen and offshore petroleum present the (111) and (333) peaks characteristics of complex perovskite oxides as the XRD spectrum of  $Ba_2AlNbO_6$  ceramics before submersion in crude petroleum.

Typical Vicker's microhardness tests results carried out on  $Ba_2AlNbO_6$  ceramics before and after 60 days of submersion in crude petroleum are presented in Table 2. As we can see from these results  $Ba_2AlNbO_6$  ceramics increased their mechanical hardness due to crude petroleum environment.

Microstructural analysis of  $Ba_2AlNbO_6$  ceramics after submersion in crude petroleum was carried out using optical microscopy since after submersion in crude petroleum it was extremely difficult to use scanning electron microscopy due to the vacuum requirements. Fig. 4 presents the optical micrographs of  $Ba_2AlNbO_6$  ceramics before and after submersion in crude petroleum for 45 and 60 days respectively. As seen from Fig.4 microstructural features of  $Ba_2AlNbO_6$  ceramics remain identical before and after submersion in crude petroleum without degradation in microstructural features due to crude petroleum environment.

From these promising results we conclude that  $Ba_2AlNbO_6$  ceramics are stable in the aggressive crude petroleum environment and thus could be potential candidate ceramic encapsulation in the fabrication of temperature sensors for temperature monitoring in petroleum wells.<sup>17,22,23,24</sup>

**Table 2.** Vicker's microhardness of  $Ba_2AlNbO_6$  ceramics before and after submersion in crude petroleum for 60 days.

Ceramics	MHV (KgF/mm <sup>2</sup> )	Standard deviation
$Ba_2AlNbO_6$ ceramics before submersion in crude petroleum	56.43	4.45
$Ba_2AlNbO_6$ ceramics after 60 days submersion in earthen crude petroleum	70.49	4.93
$Ba_2AlNbO_6$ ceramics after 60 days submersion in offshore crude petroleum	69.81	4.41

## 4. Conclusions

In the present work we have produced a niobium based complex cubic perovskite oxide  $Ba_2AlNbO_6$  ceramics and studied their stability in crude petroleum environment for the manufacture of inert ceramic embedding for temperature sensors used in petroleum extraction.  $Ba_2AlNbO_6$  ceramic powder was prepared through thermo-mechanical solid-state processing route. Structural characteristics and phase identification of calcined material was investigated by powder X - ray diffractometry, which presented a single phase complex cubic perovskite structure with lattice parameter  $a = 8.3418 \text{ \AA}$ . The microstructure of the  $Ba_2AlNbO_6$  ceramics sintered at  $1150^\circ\text{C}$  presented a homogenous morphology and particle size distribution. The mechanical behaviour of the

sintered ceramics was studied by Vicker's micro-hardness testing, which gave a reasonable value of  $MHV = 56,43 \text{ KgF/mm}^2$  for ceramics sintered at  $1150^\circ\text{C}$ , which is satisfactory for encapsulation application of these ceramics. Results of X-ray diffractometry, microstructural and mechanical tests showed that  $Ba_2AlNbO_6$  ceramics submerged in crude petroleum did not suffer change at any stage of submersion. From these results we conclude that  $Ba_2AlNbO_6$  ceramics are stable in the aggressive crude petroleum environment and thus could be potential candidate ceramic encapsulation in the fabrication of temperature sensors for temperature monitoring in petroleum wells.

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