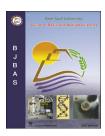


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# Morphological and electrical properties of zirconium vanadate doped with cesium



Marwa F. Elkady a,b, Mohamed Abd Alrafaa c, Noha A. El Essawy d,\*

- <sup>a</sup> Chemical and Petrochemical Engineering Department, Egypt-Japan University of Science and Technology, New Borg El-Arab City, Alexandria, Egypt
- <sup>b</sup> Fabrication Technology Researches Department, Advanced Technology and New Materials Research Institute (ATNMRI), City for Scientific Research and Technological Applications, Alexandria, Egypt
- <sup>c</sup> Electronic Materials Researches Department, Advanced Technology and New Materials Research Institute (ATNMRI), City for Scientific Research and Technological Applications, Alexandria, Egypt
- <sup>d</sup> Advanced Technology and New Materials and Research Institute (ATNMRI), City for Scientific Research and Technological Applications, Alexandria, Egypt

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#### ABSTRACT

Cesium doped zirconium vanadate  $ZrV_2O_7$  with different Cs dopant content (Cs/Zr varied from 0 to 0.5 in weight ratio) were fabricated by hydrothermal technique at 120 °C for 60 min. The synthesized materials are thermally treated using microwave technique. The structural and morphological properties of the synthesized materials and thermally treated samples were investigated using XRD and SEM respectively. It was evident that all synthesized specimens have cubic phase structural without any extra phase but after heat treatment Orthorhombic phase appear with doped samples. However, the morphological structure of the doped synthesized materials has transferred from nanoparticles into rods aspect with heat treatment for the different dopant ratio. Moreover, the electrical properties of both the synthesized and thermally treated materials are studied by AC impedance measurements. The results indicated that the ionic conductivity of Cs-doped  $ZrV_2O_7$  materials decreased by increasing the dopant ratio while that thermally treated samples the ionic conductivity increase by increasing the dopant ratio. Finally, the concentration of cesium dopants is found to play crucial role in tuning the morphology and electrical properties of nanostructures.

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### 1. Introduction

Peyronel is the first one who was reported zirconium pyrovanadate ( $ZrV_2O_7$ ) in 1942 (Peyronel, 1942; Sahoo et al., 2011).

 $ZrV_2O_7$  from  $AX_2O_7$  families of compounds such as (A=Zr, Hf and X=P, As, V) and has a framework structure with space group symmetry Pa3, Z=4. Its structure is related to the NaCl structural type (Space group Fm3m) with  $A^{4+}$  cation and  $(X_2O_7)^-$  anion. It has two first order structural phase

<sup>\*</sup> Corresponding author. Tel.: +2 01222608176. E-mail address: nony\_essawy@yahoo.com (N.A. El Essawy). Peer review under the responsibility of Beni-Suef University. http://dx.doi.org/10.1016/j.bjbas.2014.10.009

transitions at 77 °C and 102 °C (Craig and Hummel, 1972; Khosrovani et al., 1997; Sahoo et al., 2011). ZrV<sub>2</sub>O<sub>7</sub> belongs to a  $3 \times 3 \times 3$  superstructure at room temperature which vanishes above 102 °C, and a strong isotropic negative thermal expansion behavior is reported for the temperature up to 800 °C. The superstructure for this compound was established from single crystal synchrotron data, high-resolution neutron powder diffraction data, and electron diffraction studies (Evans et al., 1998; Khosrovani et al., 1996; Korthuis et al., 1995; Withers et al., 1998). There are many synthetic processes currently being used to produce Zirconium vanadate as sol--gel technique, homogeneous precipitation, solid state reaction and hydrothermal technique (Abd El-Latif and El-Kady, 2008). The reaction between ZrO<sub>2</sub> and V<sub>2</sub>O<sub>5</sub> has been described as "extraordinarily slow" and to overcome these difficulties, "hydrothermal synthesis", which is a soft-chemical method resulting in the formation of homogenized high purity products with reduced particle sizes and larger surface area, has been employed to synthesize the various members of the solid solution (Zhu et al., 2006), it also acts as one of the most attractive techniques for processing nano-hybrid and nanocomposite materials. Hydrothermal method has already been applied to synthesize nanosized ZrV2O7 with polycrystalline phase and small crystallite size (Abd El-Latif and El-Kady, 2011).

The previously stated research literatures illustrated that there were many scientists were considered with the behavior of zirconium vanadate under different conditions. Some researchers stated that ZrV<sub>2</sub>O<sub>7</sub> undergoes a reversible phase transition upon application of pressure at 1.38-1.58 GPa, where it converted from cubic ( $\alpha$  phase) to pseudo-tetragonal ( $\beta$  phase). This phase has an orthorhombic 2  $\times$  3  $\times$  3 super cell (Carlson and Andersen, 2001). Other researchers were explored that the room temperature Raman and infrared spectra of ZrV<sub>2</sub>O<sub>7</sub> have been collected at 12 and 5.7 GPa and support the phase transition behavior established by high pressure X-ray diffraction (Hemamala et al., 2007). On the other hand, ZrV2O7 electrical conductivity has also been investigated for the prepared and hot pressed samples, where they were showed good electrical conductivity suggesting semiconductor n-type behavior, which ranges from 10<sup>-5</sup> to  $10^{-6} \ \Omega^{-1} \ \text{cm}^{-1}$  (Sahoo et al., 2011). Moreover, zirconium vanadate has been successfully utilized by numerous researches as inorganic ion exchanger for the separation of <sup>134</sup>Cs and 152Eu from a synthetic mixture (Sahoo et al., 2011; Xing and Zhu, 2010). Finally, many zirconium vanadate solid solutions have been established and there thermal expansion behaviors have been considered. For example, there were various solid solutions developed from ZrV<sub>2</sub>O<sub>7</sub>-ZrP<sub>2</sub>O<sub>7</sub> (0  $\leq$  x  $\leq$  2.0) and Zr1<sub>-x</sub>Hf<sub>x</sub>V<sub>2</sub>O<sub>7</sub> (x = 0-1) series, and their isotropic thermal behaviors have been examined (Korthuis et al., 1995; Hisashige et al., 2006).

Mostly, doping is adding of impurities to the material in order to modify its properties. Zirconium vanadate doped with zirconia and vanadium (V) oxide was prepared by solid state reaction and sol—gel methods. The results show that, the doping doesn't effect on the crystal structure neither nor its thermal properties (Xing and Zhu, 2010). While, other researchers were found that the photo-catalytic activity of synthesized molybdenum doped zirconium vanadate

produced from the solution combustion method was enhanced toward the degradation of non-azoic dyes compared with its parent zirconium vanadate (Sahoo et al., 2011). However, till now there is no investigations deal with the doping effect of mano-valent cations (such as cesium ions) on the crystal, morphology and electrical properties of zirconium vanadate. Accordingly, cesium doped zirconium vanadate will be synthesized in this investigation using hydrothermal technique, where the hydrothermal doping method was hoped to create a rational dopant alkali metal distribution in ZrV2O7 with avoiding the formation of heterogeneous metal oxides phases. These synthesized materials will be thermally modified using microwave advanced heating technology, which can provide new, powerful, and significantly different tool for materials processes or to improve the performance characteristics of existing materials. In many cases, materials processing using microwave technology have numerous advantages in their properties compared to that using the traditional materials processing techniques (Clark and Sutton, 1996). This is regarded to the characteristics features of the microwave technology that include more precise and controlled volumetric heating, faster ramp-up to temperature, and lower energy consumption, that by its role enhance the quality and properties of the processed materials. Microwave heating can achieve very fast and uniform heating through a self-heating process that arises from the direct absorption of microwave energy into the materials. Furthermore, microwave heating makes it possible to synthesize the target materials at temperature lower than those required for furnace heating (Higuchi et al., 2003). Accordingly, microwave heating technology represents an economic and very fast procedure technique compared with furnace heating. Respecting to this concept, microwave technology will be utilized as a sintering process tool for the synthesized zirconium vanadate and its cesium dopants with different concentration.

This research activity will be highlighted through studying the effect of both cesium doping and thermal treatment onto the crystalline nature, morphological structure and electrical properties of prepared zirconium vanadate and their cesium-dopant produced.

#### 2. Materials and methods

#### 2.1. Materials and equipment

The main reagents used for the synthesis of Zirconium vanadate and its cesium dopant were Zirconium oxy chloride (AVONCHEM, United Kingdom), Sodium vanadate (ACROS, USA), Cesium chloride (APPLICHEM, Germany), Hydrochloric acid (ALDRICH, USA.). All chemicals have high purity. The instruments used are: Balance with shield chamber 4 digits (Sartorius, Model CP225D, Germany), Hot plate and stirrer (Stuart, Model SB 162, United Kingdum), Autoclave (Systec, Model 3850-EL), Drying oven (Nabertherm, Model TR60, Germany), Vacuume pump (Barnant, Model 400-3912, USA), Microwave Assisted Technology (MAT) furnace (CARBOLITE, Model MRF 16/22,UK).

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