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Anjaly Jose, Aneesh George, Shinju Benny, H. Hitha, and Thomas Varghese

Synthesis and Characterization of Iron Cerium Tungstate Nanoparticles and their Dielectric Studies

Anjaly Jose[1,3,a)], Aneesh George[1], Shinju Benny[1], H Hitha [3] and Thomas Varghese[2,3,b)]

[1]*Dept. of Physics, Vimala College, Thrissur, Kerala- 680009, India.*
[2]*Nanoscience Research centre, Nirmala college, Muvattupuzha-686 661, Kerala, India.*
[3]*Dept. of Physics, Newman College, Thodupuzha- 685584, Kerala, India.*

[a)]author: iamanjalyjose@gmail.com
[b)]corresponding author: nanoncm@gmail.com

Abstract. Nano iron cerium tungstate has been successfully prepared by the effective chemical precipitation method followed by calcination at 600 °C. The X-ray diffraction result verifies that the calcined sample is crystallized in the triclinic structure with space group $P\bar{1}$ and the particles are in nanometer size. The optical absorption tale of the material starts in the visible region at 660 nm. The crystalline planes grown are visible from the high resolution transmission electron microscopy images and the selected area diffraction patterns. The dielectric constant and dielectric loss values obtained at 1 MHz are 20 and 0.142, respectively.

INTRODUCTION

In recent years, the wireless communication industry has created a high demand for the development of microwave components such as filters, duplexers, resonators and antennas. [1, 2] A number of ceramic dielectric materials has been envisaged and studied to their suitability for microwave substrate applications. Such compounds include silicates, aluminates and molybdate ceramics. [3, 5] Titanate based ceramic dielectric material having a dielectric constant of 20.2 has been reported for microwave applications. [6] The microwave dielectric properties of $Ce_2(WO_4)_3$ ceramics have been reported in literature. [7]

The present paper explore the dielectric behavior $FeCe(WO_4)_3$ nanoparticles with the frequency variation at room temperature. The nanoparticles are prepared by employing the chemical precipitation method. The dielectric studies of $FeCe(WO_4)_3$ is reporting here for the first time. The dielectric properties of the prepared $FeCe(WO_4)_3$ nanoparticles are suitable for gate dielectric material for metal oxide semiconductor field effect transistor (MOSFET).

EXPERIMENTAL

Iron cerium tungstate nanoparticles were synthesized using chemical precipitation method, which produce ultrafine particles in a relatively low processing time. This method also possesses good stoichiometric control for the production of nanoparticles with improved compositional homogeneity. [8]

Cerium (III) nitrate hexahydrate, $Ce(NO_3)_3.6H_2O$ (Himedia, Minimum Assay 99.0%), Ferric nitrate, $Fe(NO_3)_3.9H_2O$ (Merck, Minimum Assay 98%) and Sodium tungstate, $Na_2WO_4.2H_2O$ (Merck, 98%) were taken as precursors and their molar concentrations ratio was maintained as 1:1:3. Initially, the solutions of all the components were prepared. The first two solutions were thoroughly mixed in a beaker using magnetic stirrer and the sodium tungstate solution was added to the mixture solution at 20 ml/min under constant stirring. The hence mixed solution was stirred well for half an hour, and brownish precipitate was obtained. The precipitate was filtered and washed with distilled water several times. The product was then dried at 70 °C, in a hot air oven. This product was powdered and calcined at 600 °C for 3 h in a muffle furnace at atmospheric pressure. Dark brown coloured iron cerium tungstate nanocrystals were formed and their structural, optical and dielectric properties were studied.

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The XRD patterns of the sample was taken by PANalytical XRD aeris X-ray diffractometer with CuKα radiation (λ=1.5406 Å, X–ray tube voltage = 40 kV and current = 15 mA). Scan was taken in the 2θ range from 5 to 60° at increments of 0.02° with a step time of 59.7s. High resolution transmission electron microscope (HRTEM) images were obtained from Jeol/JEM 2100 electron microscope. Optical absorption spectra of the samples were recorded using a Shimadzu UV-Vis-NIR spectrophotometer. The radio frequency (RF) dielectric properties were studied using Hioki-3536 LCR meter.

RESULTS AND DISCUSSION

X-Ray Diffraction Analysis

The formation of phase pure $FeCe(WO_4)_3$ nanoparticles is confirmed from the XRD pattern as shown in the Fig.1. The material crystallizes in the triclinic crystal structure with space group $P\bar{1}$ indexed with the JCPDS card number 85-0142. The broad hump in the range 20 to 30° arose due to the combination of large number of nearby broadened peaks of the nanosized crystallites. The average crystallite size calculated using the Scherrer method is found to be 20.5 nm. [9]

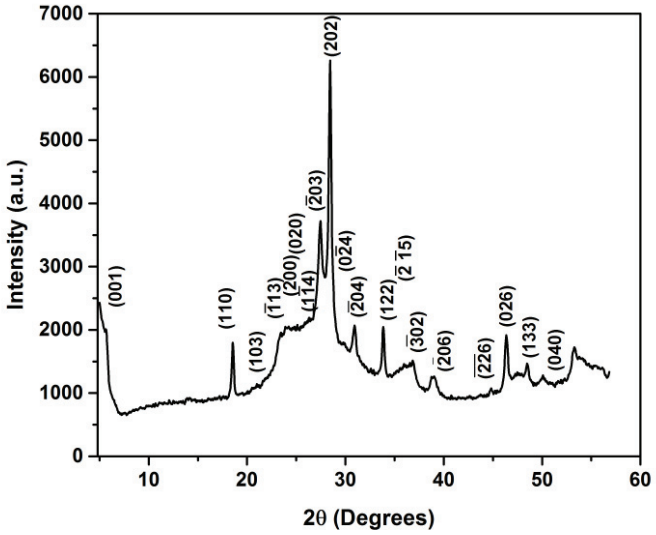
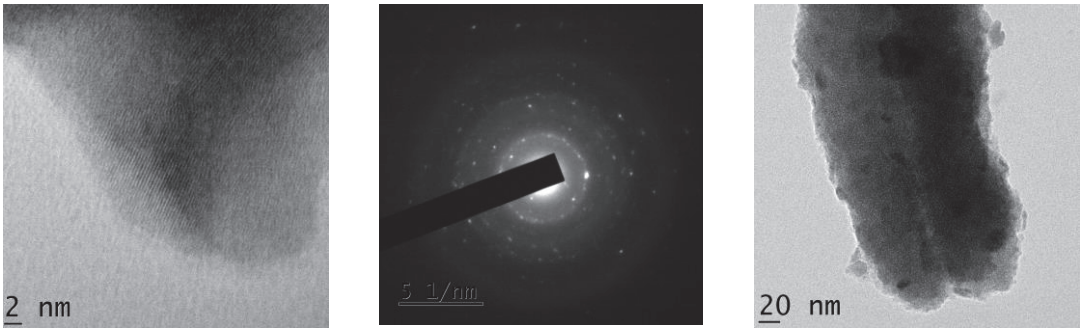


FIGURE 1. XRD pattern of $FeCe(WO_4)_3$

TEM analysis

The polycrystalline nature of the $FeCe(WO_4)_3$ nanoparticles is confirmed from the HRTEM image as well as the SAED pattern, shown in Fig. 2. The interplanar spacing d obtained from the HRTEM image is 4.68 Å, which corresponds to the angle 2θ=18.575° of (110 plane) in the XRD pattern. This plane is well observed in SAED pattern as the first bright spot. The broad hump behavior in the XRD pattern is resolved as the distinct bright spot in SAED in the region around 5 1/nm. The bright field image shows the spherical nature of the nanoparticles. The average particle size obtained from TEM is found to be 13.3 nm.

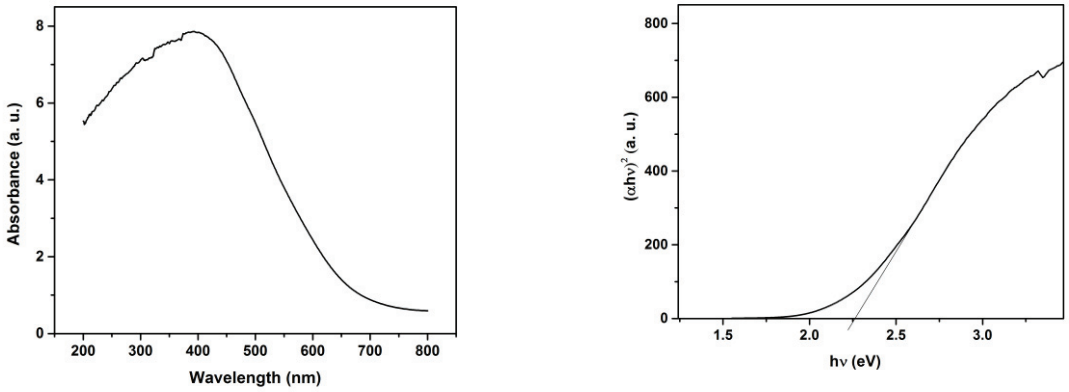


(a) (b) (c)

FIGURE 2. (a) HRTEM image, (b) SAED pattern and (c) Bright field image of $FeCe(WO_4)_3$.

UV-Vis Absorption Studies

Figure 3a shows that optical absorption starts from the farther end of the visible region at around 660 nm. The direct band gap (E_g) values of the material is determined by fitting the absorption data to direct transition equation, $\alpha hv = A(hv - E_g)^{1/2}$. The optical bandgap is determined by extrapolating the linear portion of the plot of $(\alpha hv)^2$ *vs.* energy (hv). [10] The bandgap obtained is 2.26 eV, which corresponds to the band edge emission as shown in Fig. 3b.

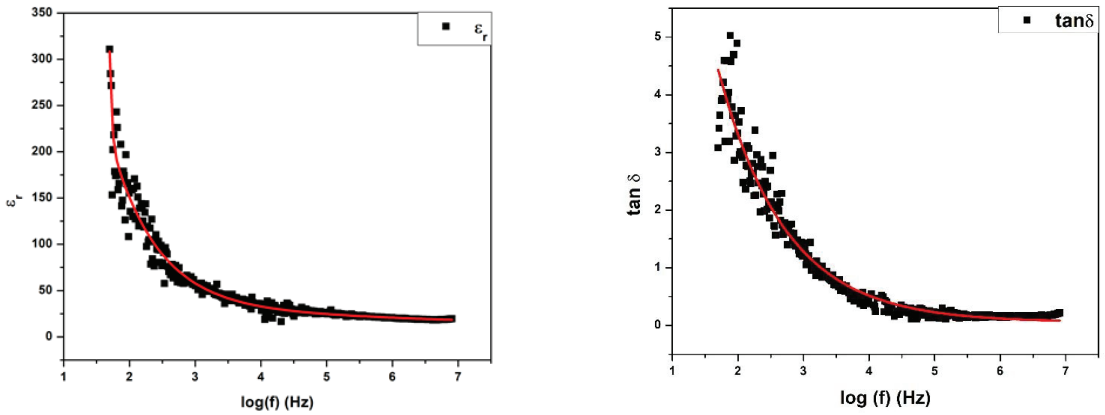


(a) (b)

FIGURE 3. (a) Absorption spectrum and (b) Tauc plot of the $FeCe(WO_4)_3$ sample.

Dielectric Studies

The dielectric properties of the material are studied using a pellet made up of the powder material. The pellet is sintered at 1000 °C at 2 h to obtain a density of 6.620 g/cm^3, which is 90% density compared to the reported value in ICDD. The dielectric properties of the sintered pellets are studied in the frequency range 50 Hz to 8 MHz at room temperature. The variation of dielectric constant (ε_r) with frequency (log f) is shown in Fig. 4. The ε_r values decrease with increase in frequency from 50 Hz to 100 kHz, which is due to the contributions of electronic, ionic, dipolar and Maxwell–Wagner interfacial polarizations. [11] When a very low frequency electric field is applied, the interfacial or space charge polarization occurs. The charges can become trapped within the interfaces of a material. Motion may also be impeded when charges cannot be freely discharged or replaced at the electrodes. The field distortion is caused by the accumulation of these charges thereby increasing the capacitance of the material known as Maxwell-Wagner effect. Therefore, at low frequencies, high dielectric constant is observed. The polarization does not occur instantaneously with the application of the electric field. The delay in response towards the electric field leads to decrease in dielectric constant. However, above 100 kHz the large relaxation time ceases respond, then the contribution from the Maxwell–Wagner interfacial polarization is minimized, so ε_r shows a saturated value. From the figure it is observed that ε_r is 20 at 1 MHz.



(a) (b)

FIGURE 4. (a) Variation of dieletric constant and (b) dielectric loss, with frequency

Figure 5b shows that dielectric loss (tanδ) decreases with increase in frequency and goes to almost saturated value. This is due to the reduction of strain and unit cell volume in the nanopowder. The compound shows a very low dielectric loss at 1 MHz and is 0.142. The suitable dielectric constant and dielectric loss values of this dielectric material can be used for gate dielectrics of MOSFET.

CONCLUSION

Chemical precipitation method is found to be the efficient method for the synthesis of iron cerium tungstate nanoparticles. The X-ray diffraction result verifies that the calcined sample is crystallized in the triclinic structure with space group $P\bar{1}$ with crystallite size about 20.5 nm. The optical absorption tale occurs at 660 nm in the visible region. The corresponding Tauc plot furnished the optical bandgap as 2.26 eV. The high resolution transmission electron microscopy reveals the crystalline planes grown images and the *d* values calculated corresponds to the (110) plane. The dielectric constant and dielectric loss values obtained at 1 MHz are 20 and 0.142, respectively. The suitable structural and dielectric properties of this material can be made useful for MOSFET construction.

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REFERENCES

1. C. X. Mao, S. Gao, Y. Wang, F. Qin and Q. X. Chu. IEEE Transactions on Microwave Theory and Techniques **64**, 2006-2013(2016).
2. Z. Fang, B. Tang, F. Si and S. Zhang, Ceramics International **43**, 1682-7 (2017).
3. S. George, P. S. Anjana, V. Deepu, P. Mohanan and M. T. Sebastian, J. Am. Ceram. Soc. **92**, 1244 (2009).
4. M. T. Sebastian, *Dielectric materials for wireless communication* (Elsevier, 2010).
5. G. Q. Zhang, J. Guo, L. He, D. Zhou, H. Wang, J. Koruza and M. Kosec, J. Am. Ceram. Soc. **97**, 241–245 (2014).
6. Y. D. Zhang, J. Han, R. Liang and D. Zhou, Materials Letters **153**, 118–120 (2015).
7. P. S. Anjana, T. Joseph and M. T. Sebastian, Ceramics International **36**, 1535–1540 (2010).
8. A. Sreedevi, K. P. Priyanka, K. K Babitha, N. A. Sabu, T. S. Anu and T. Varghese, Indian Journal of Physics **89**, 889-897 (2015).
9. B. D. Cullity, *Elements of X-Ray Diffraction* (Addison-Wesley, United States (1967).
10. S. Khademolhoseini and S. Ali Zarkar, J. Mater. Sci. Mater. Electron. **27**, 9605–9609 (2016).
11. F.Rogti, M.Ferhat, Journal of Electrostatics, **72**, 91-97 (2014).