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SYNTHESIS, STRUCTURAL AND DIELECTRIC PROPERTIES OF Nb_2O_5 NANO-PARTICLES: A PROMISING FUNCTIONAL MATERIAL

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Abstract

Chemical precipitation technique was employed to synthesize crystalline Nb_2O_5 nanoparticles. The XRD result revealed that the synthesized nanoparticles possess orthorhombic structure as confirmed by Fourier-transform infrared (FTIR) spectroscopy and FT-Raman studies. The average particle size of the as-prepared sample obtained from XRD analysis confirmed their nano crystalline nature. The prepared nanopowders were sintered at relatively low temperature of 1350 ^oC to high density. SEM images of sintered samples indicated high densification of the nanomaterials. The variations of dielectric constant (ε_r), conductance (G) and loss factor (tan δ) of the samples were studied in the radio frequency range.

Keywords: Nano particles, Chemical precipitation method, X-ray diffraction, FT- Raman studies, FT-IR studies, Dielectric studies.

1. INTRODUCTION

Niobium pentoxide (Nb_2O_5) is one of the most studied materials because of its wide range of applications include batteries, solar cells, sensors, electrolytes, semiconductors and optical materials [1-3]. Niobium oxides have been prepared by different methods such as oxidation of metallic niobium in air, by hydrolyzing alkali-metal niobates, niobium alkoxides and niobium pentachloride or by precipitation from solution in hydrofluoric acid with alkali-metal hydroxide or ammonia [4]. Sayama *et al.* [5], Kominami *et al.* [6] have reported that the properties of Nb_2O_5 prepared by various methods. This paper reports the synthesis of phase pure nano sized Nb_2O_5 by Chemical precipitation technique and their structural and dielectric characterizations.

2 EXPERIMENTAL

Niobium oxide nano particles were prepared by the chemical precipitation method. Nb_2O_5 nano particles can be obtained by a precipitation of niobate oxylate hydrate with ammonia in water/ethanol at room temperature. The entire process was carried out in deionized water for its inherent advantages of being simple and environment friendly. 20 ml of niobium oxalate was taken in a burette. 20 ml NH_4CO_3 from the stock solution taken in the conical flask along with 70 ml of de-ionized water and 10 ml of EDTA solution. The niobium oxalate solution from the burette was added drop wise at constant rate to the contents in the beaker under vigorous stirring using a magnetic stirrer. By employing a magnetic stirrer the homogeneity of the nano sized crystals formed were maintained. The precipitate was collected in

beaker. The preparation was repeated for the entire 200 ml stock solutions. The precipitate is decanted and wash repeatedly washed 5 times using de-ionized water and centrifuged to collect the precipitate. Finally the precipitate was washed using acetone to remove any trace of organic impurities. The precipitate was then dried overnight in oven at 100 ^{o}C

The structure of the as-prepared and subsequently heated powder is characterized by Powder X-ray Diffraction. The average crystallite size was estimated from Scherrer's equation. The Fourier transform infrared (FTIR) spectrum of the sample was recorded in the range 400-4000 cm^{-1} on a Thermo-Nicolet Avatar 370 FTIR Spectrometer using KBr pellet method. The FT-Raman spectrum of the as prepared sample was recorded at room temperature in the wave number range 50-1200 cm^{-1} using Bruker RFS/100S Spectrometer at a power level of 150 mW and at a resolution of 4 cm^{-1}. To study the sinterability of the nanoparticles obtained by the chemical precipitation method, the Nb_2O_5 nano particles were mixed with 5% polyvinyl alcohol and pressed in the form of cylindrical pellet of 10mm diameter and ~2mm thickness at a pressure about 350 MPa using a hydraulic press. The surface morphology of the sintered samples was examined using scanning electron microscopy (SEM, Model- JEOL JSM 6390 LV). The dielectric constant, loss tangent (tan δ) and the electrical parameters were measured in polished and silver coated pellets, using LCR meter (HIOKI, model 3532, Japan).

3 RESULTS AND DISCUSSIONS

3.1 XRD

The phase purity and crystal structure of Nb_2O_5 nanopowders are determined using XRD. Fig. 1 shows the diffraction pattern of the Nb_2O_5 powder annealed at 100°C for half an hour. The crystallinity of the sample is evidenced by sharp diffraction peaks at respective diffraction angles which can be readily indexed for its orthorhombic structure [7]. The orthorhombic phase obtained for the sample agrees with the standard data (ICDD PDF 30-0873). The calculated crystallite size from the full width at half maximum (FWHM) using Scherer formula for the major (181) plane is ~18 nm [8].

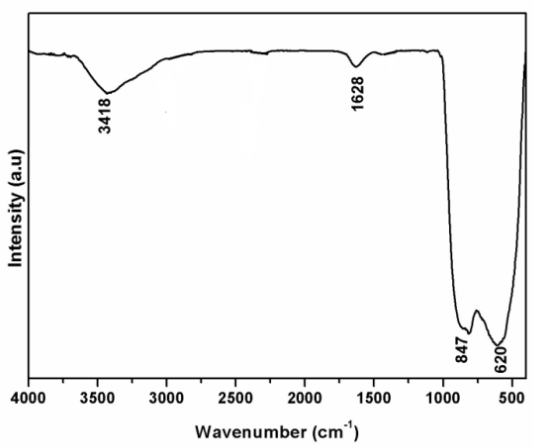

Fig. 1 XRD pattern of Nb_2O_5 nanopowder

3.2 FT-Raman and FT-IR

The structural analysis of the sample is extended to the studies of vibrational spectra of the powders using Raman and FT-IR analysis. Fig. 2 and Fig. 3 shows the FT-Raman and FT-IR spectra of nanocrystalline

Nb_2O_5. The spectrum shows well defined peaks which perfectly agree very well with the earlier [9-12]. The symmetric and asymmetric stretching vibration of Nb-O/ Ti-O bonds give rise to two Raman active modes υ_1A_{1g} and υ_2E_g modes, respectively. υ_1A_{1g} mode observed at 857 cm^{-1} and υ_2E_g at 642 cm^{-1} with a shoulder band at 666 cm^{-1}. υ_3F_{1u} is active in 528cm^{-1} as very weak band. υ_4F_{1u} due to bending vibrations of Nb-O/ Ti-O octahedral is observed as medium intense band at 476 cm^{-1}. The symmetric bending vibrations give rise to υ_5F_{2g} at 411cm^{-1}. The band at 283 cm^{-1} is the active mode υ_6F_{2u} and that below 200 cm^{-1} is due to lattice vibrations [13].

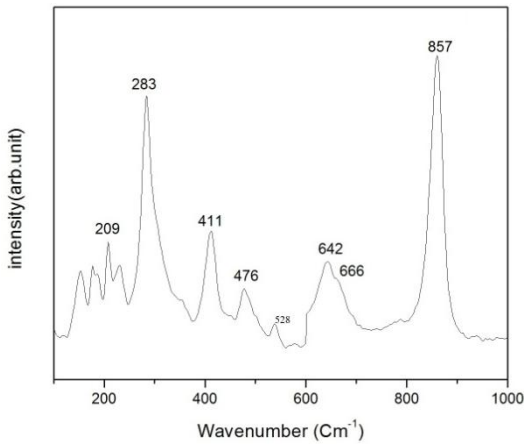

Fig. 2 FT-Raman spectrum

The IR spectrum of the sample show similar characteristics. Two sharp absorption bands are observed at 620 and 847 cm^{-1}. The highly intense band at 620 cm^{-1} is due to IR active asymmetric stretching ν_3F_{1u} mode of Nb=O bond. The weak broad bands around 1628 cm^{-1} and 3418 cm^{-1} are due to the bending and stretching vibrations of the water molecules adsorbed during pelletisation.

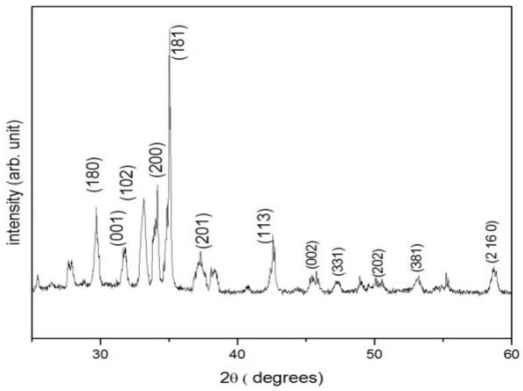

Fig. 3 FT-IR spectrum

3.3 SEM

The pellet is then sintered at 1350 ^oC for 2h. Fig. 4 shows the SEM images of the sintered pellet. The SEM image shows that the sample have achieved full densification with minimum pores and involve elongated grains of average size ~2µm. Oishi *et al*. reported that elongated grains as a characteristic of orthorhombic structure. In the present sintering study, fully dense powder compacts were obtained from ultrafine nano sized particles. The experimental density obtained is 96% of the bulk value of the same material.

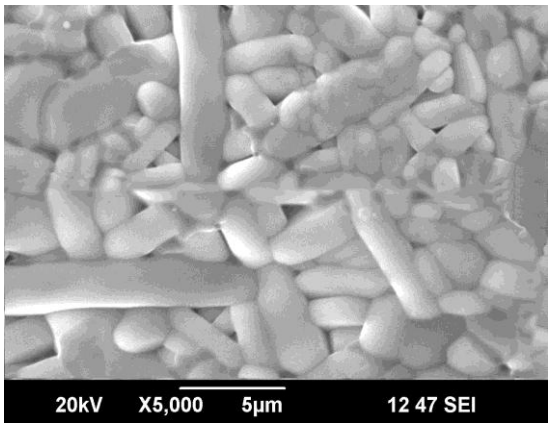

Fig. 4 SEM image of sintered of Nb_2O_5 pellet.

Figure 5, 6 and 7 shows variation of dielectric constant (ε_r), loss factor (tan δ) and conductance (G) with frequency range 100 Hz to 5 MHz at room temperature. Dielectric constant decreases initially with the increase in frequency and it reached a constant value 36 at 1MHz. The decrease in dielectric constant is due to the delay in polarization with the application of the electric field because of inertia [14].

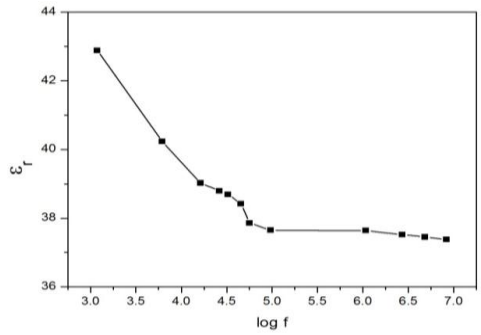

Fig. 5 variation of dielectric constant (ε_r) with frequency

Variation of dielectric loss shows that it reached a minimum value and almost constant [15]. It means that, material has low loss in radio frequency range because of the relaxation frequency of the sample is out of this frequency range. Therefore, dissipation of electrical energy is low at this range. Loss-factor can be attributed to the presence of impurities like voids in the material.

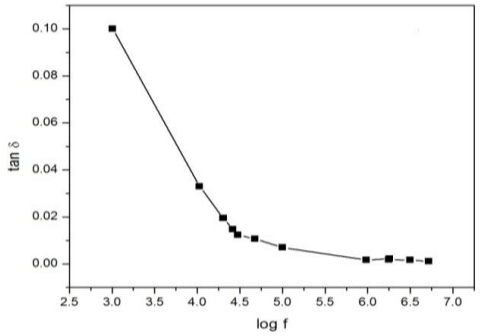

Fig. 6 variation of loss factor (tan δ) with frequency

And at low frequency range the variation of G is very less and almost constant up to 1MHz, and then increases according to the increase in frequency. Thus this material can be useful for capacitive applications in communication systems

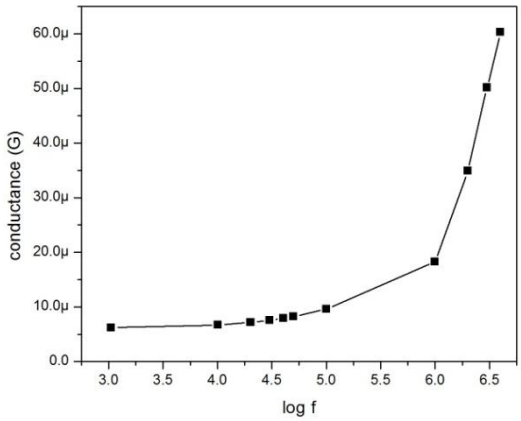

Fig. 7 variation conductance (G) with frequency

4 CONCLUSIONS

Chemical precipitation synthesis has been employed to obtain nanocrystalline phase pure Nb_2O_5 powder. XRD result reveals the orthorhombic phase with nanosized particles. The FT-IR and FT-Raman studies and SEM image analysis confirms the XRD results. The metal-oxide (NbO) band observed at the low wave number region further confirmed the phase purity of the Nb_2O_5 nanopowders. Variations of dielectric constant, loss factor and conductance with logarithm of radio frequency show that the material is suitable for communication systems.

ACKNOWLEDGMENT

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