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Original Research Article

Synthesis and electrical characterisation of partially stabilised ZrO₂ - carbon nano tube nanocomposite

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ABSTRACT

In present paper we report hydrothermal synthesis of partially stabilised cubic zirconia-CNT nanocomposites. XRD study confirms the development of CNT doped zirconia nanoparticles. Particle size of zirconia is observed to be less than 100nm. SEM study confirms the development of CNT nano networks in the parent ZrO₂ matrix. Electrical properties are analysed over a frequency range of 10 Hz < f < 8 MHz. The complex dielectric modulus study shows that relaxation dynamics are different for different samples and sample behaviour deviates from ideal Debye curve specially in low frequency regions with a presence of characteristic peak. Both ac and dc conductivities are observed to increase with CNT doping.

1. Introduction

Zirconia can be considered as unique and versatile material because of its special characteristics like redox property, chemically inactiveness blended with mechanical hardness. This material is used in different industries like dentistry and prosthetics industries as structural ceramics [1]. Also, it is used in other technical fields like fuel cells, oxygen sensors etc [2]. Now, among three different polymorphs of zirconia namely monoclinic, tetragonal and cubic zirconia, the cubic phase is more ordered and hence more industry friendly. Researchers have developed different routes to produce cubic zirconia by adding dopants to parent materials. Mu et al. have developed CNT/ZrO₂ nanocomposites by microwave technique [3]. Spark plasma route have been employed by Mazahari et al. for developing CNT-Y-ZrO₂ materials [4]. Zheng et al. developed CNT/ZrO₂ nanocomposite by hydrothermal route [5]. The electrical characteristics of prepared materials have been done by means of dielectric permittivity measurement by Li et al [6]. Enhanced ac conductivity with addition of CNT have been reported by Wisniewska et al [7].

In present work, we have developed CNT/ZrO₂ nanocomposite by hydrothermal process. Further, we have analysed XRD patterns to extract different parameters like lattice parameter, rms microstrain and particle size. The effect of CNT addition on electrical conductivity and dielectric permittivity of prepared samples are also investigated.

2. Experiment

2.1 Sample preparation

Requisite amount of zirconium oxychloride octahydrate and multiwalled CNT is dissolved in deionised water. This is kept in magnetic stirrer for 3hr. Then NH₄OH is added to it. The produced gel is collected and rinsed to wash off excess Chlorine. The gel is added to water and kept in autoclave for 10 hr with temperature fixed at 130 °C. After treatment, a ash colour precipitation is developed. This precipitation is collected and dried in a muffle furnace for 4hr at 500 °C. In this way, different CNT-ZrO₂ samples are processed. This sample is named as C1Z sample containing 1% of CNT. Through same process, pure ZrO₂ sample is produced which is termed as COZ sample.

2.2 XRD, FESEM and electrical measurements

XRD data of these samples are recorded by D8 XR diffractometer with 2 θ varying from 20⁰ to 80⁰. These data are analysed by Rietveld refinement method. FESEM images of prepared samples are done by Sigma Scanning Electron Microscope. For measuring dielectric permittivity and ac conductivity, the powder samples are pressed in a hydraulic press to produce circular pellets and Copper wires are connected to each pellets by conducting silver paste. For ac measurements, we have employed LCR meter of HIOKI. From



the measured values of capacitance and dielectric loss, ac conductivity and dielectric permittivities are obtained.

3. Results and discussion

3.1 Microstructure analysis and FESEM study

XRD patterns of C1Z and C0Z samples are analysed by Rietveld analysis method employing MAUD software [8]. Fig. 1 shows XRD patterns and their Rietveld fitting. It is found that CNT promotes development of cubic zirconia than tetragonal or monoclinic phase. In fact, weight percent of monoclinic zirconia phase is 53.5% for C0Z sample while for C1Z sample, it reduces to 45.3%. On the other hand, cubic zirconia weight percent increases from 46.4% to 54% in C1Z

sample in comparison to C0Z sample. So, CNT addition promotes cubic zirconia formation. Also, particle size as estimated is observed to be 38.82 nm for C0Z sample and 38.67 nm for C1Z sample. This confirms development of CNT-ZrO₂ nanocomposite matrix. There is a slight increase in rms microstrain in C1Z sample than C0Z sample which is attributed to the inclusion of CNT in parent zirconia matrix. Fig. 2 represents FESEM images of prepared samples. Presence of tubular MWCNT within the ZrO₂ matrix is clearly visible from the figure. Rough estimation of ZrO₂ also confirms the particle size to lie well within 100 nano meter range.

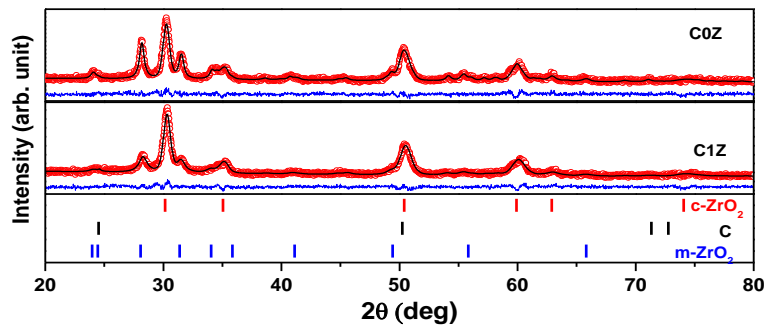


Figure 1: Figure represents XRD patterns and corresponding Rietveld fitting for the prepared samples.

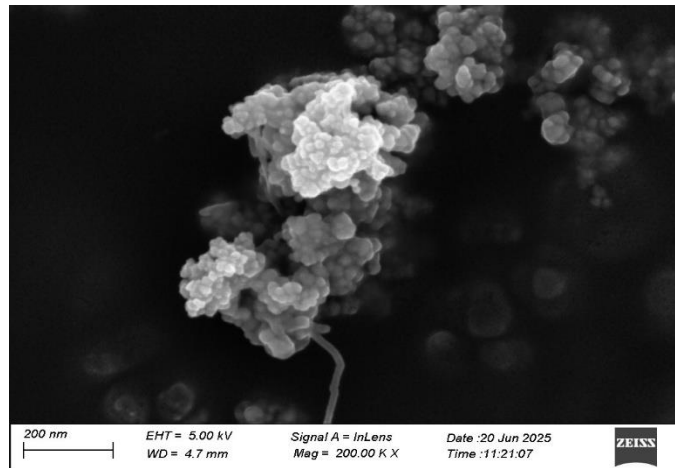


Figure 2: Figure shows SEM photo of C1Z sample. Presence of CNT is clearly visible hanging from the ZrO₂ matrix.

3.2 Study on complex dielectric modulus

During the course of movement of electron within the lattice, we need to consider this in form of polaron i.e, adjoint of charge carriers and their polarization cloud. Hopping of carrier to a new site is possible only if the associated polarization cloud also makes a successful hopping to new site. Essentially there is a time delay between the charge carrier and polarization cloud. The relaxation dynamics is different for different samples also. Thus, study of dielectric modulus formalism is crucial as it gives insight of relaxation mechanism of the concerned material. The complex dielectric modulus (M^*) can be expressed in terms of real (M') and imaginary part(M'') as:

$$M^* = M' + i M'' \quad (1)$$

Now, experimentally the quantities M' and M'' can be

calculated as:

$$M' = \frac{\epsilon'}{\epsilon'^2 + \epsilon''^2} \quad \text{and} \quad M'' = \frac{\epsilon''}{\epsilon'^2 + \epsilon''^2} \quad (2)$$

In Fig. 3, we show the frequency variation of imaginary part (M'') of dielectric modulus for the samples in a wide frequency range of 10 Hz to 8 MHz. It is observed that both the samples show a distinct peak at different frequencies. Asymmetric nature of both the curves clearly indicates deviation from ideal Debye response. To estimate the amount of deviation, we have fitted the curves by Bergman function [9] which is expressed as:

$$M''(f) = \frac{M''_{\max}}{\frac{(1-|a-b|)}{a+b} \left[b \left(\frac{f}{f_{\max}} \right)^{-a} + a \left(\frac{f}{f_{\max}} \right)^b \right] + |a-b|}$$

(3)

Here 'a' and 'b' parameters measure the amount of deviation in low frequency and high frequency regions respectively. As their values approach towards unity, deviation becomes minimum. From the fitting, it is found that 'a' values for both the samples are very close to unity, 0.96 for C1Z and 0.99 for C0Z samples. However, in high frequency region, deviation is quite high as 'b' values are 0.23 and 0.64 for C1Z and C0Z samples. So, it is quite clear that deviation mainly occurs in high frequency zone and C0Z sample has more Debye like nature than C1Z sample.

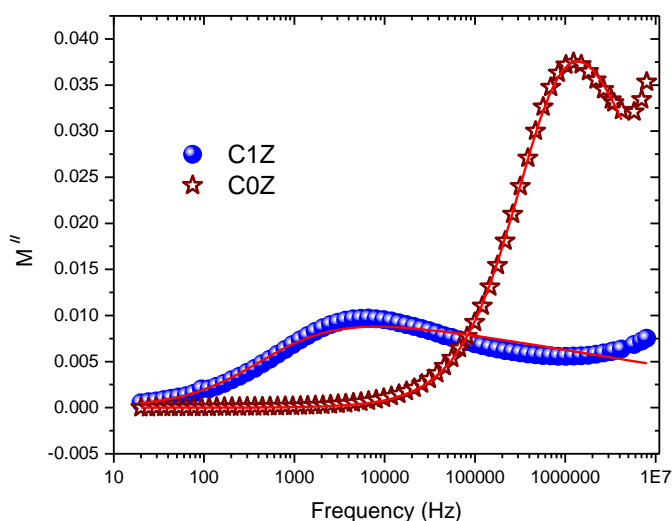


Figure 3: Figure represents frequency response of imaginary dielectric modulus and their fitting according to Bergmann function.

3.3 Study on ac and dc conductivity

We have measured ac and dc conductivities of prepared samples. It is observed that dc conductivity of CNT doped sample is higher than undoped sample and this effectively increases the ac conductivity of C1Z sample than C0Z sample. For example, ac conductivity of C0Z and C1Z samples are found to be 2.16×10^{-6} S/m and 3.87×10^{-5} S/m. So, there is more than 10 times increase in ac conductivity of the doped samples. The reasons are development of high conducting pathways by CNT within the parent matrix. Also, there are loose electrons present within the walls which eases the flow of charge carriers. This in turn increases the conductivities of doped sample.

4. Conclusions

From the work, we can conclude that addition of CNT in zirconia matrix modifies the parent character both internally and externally. More ordered cubic phases are developed due to CNT addition. On the other hand, rms microstrain increases with CNT addition. Also, doped sample shows higher amount

of deviation in high frequency region. The CNTs develop conducting pathways which enhances both ac and dc conductivity.

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Authors' contributions

The author reviewed and approved the final version of the manuscript for publication.

Conflicts of interest

The author declares no conflict of interest.

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Data availability

No new data were created.

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