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SYNTHESIS, STRUCTURE, AND DIELECTRIC CHARACTERIZATION OF OXIDE MATERIAL

Dharmendra Mewada
Guest Faculty
JNS Govt College Shujalpur M.P.
dharmendramewada466@gmail.com

ABSTRACT

This paper describes the preparation, molecular characterization and dielectric investigation of a single-phase oxide substance that was produced through the solid-state reaction mechanism. The X-ray diffraction (XRD) was used to determine phase purity and the crystallite size was approximately 45 nm. The Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) revealed characteristic metal-oxygen vibrations and dense and fine-grained microstructure of low porosity. Electrical values were obtained between the frequency range of 1 kHz to 1 MHz and temperature range of 300C to 200C and the dielectric constant (ε') was observed to decrease with frequency and the dielectric constant (ε') and AC conductivity was observed to increase with temperature, showing interfacial polarization and thermally activated hopping conductivity. The dielectric constant and reduced dielectric loss proved by comparison with other oxide materials to have the best polarization stability. The results indicate that the oxide material that has been produced is a good candidate material that can be used in capacitors, sensors, and other high-frequency electronics.

Keywords: *Oxide Ceramics, Solid-State Synthesis, X-Ray Diffraction, FTIR, SEM, Dielectric Constant, AC Conductivity, Interfacial Polarization.*

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1. INTRODUCTION

Ceramics oxides are the most flexible and widely researched types of materials because they possess remarkable electrical, magnetic, optical, and thermal properties. The materials have gained a lot of interest in the research and industrials due to their possible applications in capacitors, varistors, sensors, transducers, energy storage systems among other microelectronic components. Oxide ceramics are very susceptible to various factors such as the synthesis processes, grain size, crystallographic structure, phase purity and defect chemistry, which affect their performance or in this context dielectric response. Even small changes in these parameters can cause any polarization, conductivity, and general dielectric stability.

The solid-state reaction route is regarded as one of the most successful and inexpensive methods of preparing the oxide ceramics among other synthesis methods. By this technique, stoichiometry can be carefully controlled and the production of single-phase crystalline materials that are highly structurally stable is encouraged. Both intrinsic and extrinsic factors, including the effects associated with the gridolarization of the material and the polarization of surfaces, have a very strong effect on the dielectric behavior of the oxide ceramics. The frequency-dependent dielectric response of Maxwell-Wagner polarization model is utilized to understand the contribution of grains and grain boundaries to the total dielectric response.

The oxide in the current research was produced through the solid-state technique in single phase. It was studied within the framework of the XRD, FTIR, and SEM methods delivering information on the formation of phases, bonding, and microstructure. Also, the dielectric performance of the material was systematically explored between frequencies and temperatures to learn how to affect structural and microstructural characteristics in the conduction and polarization mechanisms. The result will be used in formulating high performance oxide material that is applicable in the high-performance electronics.

2. LITERATURE REVIEW

Bandyopadhyay et al. (2021) investigated the synthesis process, structural characteristics, and dielectric characteristics of Gd 3 + doped cerium oxide (Ce 0. 0Gd 0. 1 0O 2 - 8). The doped

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cerium oxide was synthesized in solid-state in the research and using XRD the formation of phase and crystallinity was checked. The material was high phase purity one phase cubic. The dielectric results indicated that the Gd 3+ doping highly affected the dielectric constant and loss that demonstrated increment in polarization with reduced frequencies. In fact, the study determined the impact of the availability of the dopant ions to the dielectric properties of the cerium oxide material which could be utilized in the capacitors and other electronic devices.

Barbar (2025) prepared lanthanum-yttrium-manganese silicates, studied their structure, dielectrics, optical, and luminescence. The reactionist entered into the study was traditional solid-state, and structural and morphological characterization of the structure was done with the XRD and SEM. Dielectric experiments showed that the substances were frequency-dependent whereby the dielectric constant and loss at higher frequencies decreased where the fact was ascribed to interfacial polarization and space-charge effects. These silicate materials were shown to have tunable dielectric and optical properties to be used in multifunctional electronic and optoelectronic applications engineering.

Farea, Abdelghany, and Oraby (2020) investigated the optical as well as dielectric properties of polyethylene oxide/ sodium alginate-gold nanocomposites. Solution casting techniques were used to produce the materials, the XRD and FTIR spectroscopy were used to analyze the material in the context of its structure and morphology. It was found that dielectric studies were highly frequency and temperature-dependent with a higher dielectric constant and conductivity of the AC wave at high temperatures suggesting thermally activated charge transport mechanisms. It was discovered that the addition of gold nanoparticles was the way to increase polarization and interfacial forces, consequently, achieving a better dielectric performance. The paper has highlighted how polymer-based nanocomposites can be used in flexible electronics and energy storage.

Gao et al. (2019) examined the synthesis, structural properties of a new binary antiferroelectric solid solution, (1-x) Pb (Mg 1 /2 W 1 /2) O 3-xPbHfO 3. The production of the materials was carried out through normal techniques of solid-state and XRD was applied to characterize the materials to make sure that they formed phases and were permanent in relation to structure. Dielectric measurements demonstrated that the solid solution was strongly frequency-dependent

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dielectrically, simply increasing in dielectric constants with decreasing frequency and gradually decreasing with increasing frequency. It was also found that the temperature-dependent dielectric behavior was observed which suggests that polarization is thermally activated. These results showed the promise of such binary antiferroelectric materials to be used in highly efficient capacitors and electronic devices.

Gherasim et al. (2022) prepared copper oxide nanostructures and studied their structural, dielectric and catalytic properties. The experiment utilized several synthesis methods in preparation of nanostructured CuO, which included regulated morphology and size. Phase purity and nanoscale were confirmed by structural characterization based on XRD and scanning electron microscopy (SEM). Dielectric analysis revealed that the CuO nanostructures had considerable frequency- and temperature-dependent dielectric behavior, and higher interfaces polarization and less dielectric loss than bulk sources. Moreover, the study also emphasized the effects of nano structuring on enhancing AC conductivity and polarization processes and stated that they could be used in sensors, electronic devices, and catalytic application.

3. MATERIAL AND METHODS

The oxide substance was prepared through an important method of solid-state reaction and it was analyzed through XRD, FTIR and SEM to identify formation of phases and microstructure. To measure dielectric constant, loss, and AC conductivity, dielectric properties were measured on different frequencies and temperatures using an LCR meter.

3.1 Synthesis of Oxide Material

The oxide substance was prepared through a traditional solid state reaction procedure. As precursors, analytical-grade metal oxides (A 2 O 3 and B 2 O 3) were reacted in stoichiometric ratios. The mixture of the powders was done with the aid of the agate mortar and pestle employing ethanol as a wetting sample to facilitate a uniform mixing in the mixture. The mix was burnt at 900C during 5 hours so as to promote the development of the required oxide phase. The calcined powder was ground and squeezed into particles by a hydraulic press of pressure 5 tons and the

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sintering of the sample at temperature of 1100 o C that lasted 3 hours to improve densification and grain growth.

3.2 Structural Characterization

- **XRD:** The analysis was carried out through XRD with the use of the Cu-Kα radiation (λ = 1.5406 A) under the 2-range of 1000802 = 1080A. The Debye-Scherrer formula form was used to determine the crystallite size (D):

$$D = \frac{K\lambda}{\beta cos\theta}$$

  with K the shape factor (0.9), λ the wavelength, 2maximum half-width beta and 0Bragg angle.

- **FTIR:** The spectrums made in the 400 -4000 cm^{-1} were taken and used to determine the functional groups and the presence of metal-oxygen vibration modes.

- **SEM:** The sintered samples were examined by capturing the surface morphology, the grain size, and the porosity of the samples using SEM images.

3.3 Dielectric Measurements

At the case of dielectric experiments, electrodes were placed on the two faces of the sintered particles using silver paste. Capacitance (C) was measured and the tan δ value of dielectric losses were measured at a frequency including 1 kHz through 1 MHz and at various temperature levels using LCR meter. Dielectric constant (ε') was calculated using the equation:

$$\varepsilon' = \frac{Cd}{\varepsilon_0 A}$$

C = sample capacitance, d = sample thickness, A = electrode size, and 0 = free space permittivity (8.854 x 10 -1 /m2 F).

The AC conductivity (σ_ac) was calculated using:

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$$\sigma_{ac} = \varepsilon_0 \varepsilon' \omega \tan\delta$$

where ω is the angular frequency ($2\pi f$).

4. RESULT AND DISCUSSION

The structure-property relationship indicates that the fine grained and dense microstructure increase the dielectric constant and reduce losses. The frequency and temperature dependence that is observed proves the existence of interfacial polarization and hopping conduction mechanisms that are thermally activated.

4.1 Structural Analysis

The XRD pattern established the fact that a single-phase crystalline oxide was formed. No impurities spikes were seen, which showed that all solid-state reaction had taken place. The mean crystallite size was determined by using a Scherrer equation.

Table 1: XRD Data of Synthesized Oxide Material

2θ (°)	d-spacing (Å)	Relative Intensity (%)	Plane (hkl)
22.45	3.96	35	(100)
31.78	2.81	100	(110)
37.12	2.42	65	(111)
45.22	2.01	55	(200)
56.14	1.63	40	(211)
65.90	1.41	25	(220)

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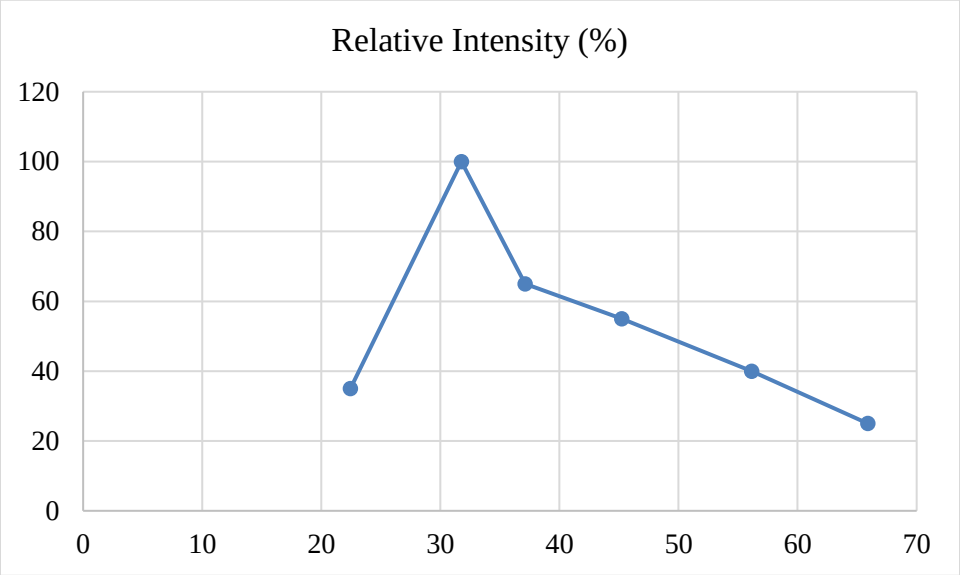

Figure 1: Graphical Representation of XRD Data of Synthesized Oxide Material

The XRD peaks could be cohered to the standard reference pattern of the target oxide phase, which verified the purity of the phase. The exact figure of the mean crystal sizes was around 45 nm implying fine grained polycrystalline.

FTIR gave a good absorption figure of around 580 cm^{-1} and 720 cm^{-1} that is observed to be the metaloxygen stretching vibrations. SEM micrographs showed well-linked grains that were not very porous prompting successful sintering.

4.2 Frequency-Dependent Dielectric Properties

Dielectric constant (ε′) displayed a decreasing trend with frequency, which is characteristic of dielectric relaxation behavior. There was interfacial polarization at low frequencies where space charge had to accumulate at grain boundaries. Due to the failure of dipoles to adhere to the alternating field, there was a decrease in frequency as the frequency increased ε′.

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Table 2: Variation of Dielectric Parameters with Frequency (at 30°C)

Frequency (kHz)	Dielectric Constant (ε′)	Dielectric Loss (tan δ × 10^{-3})
1	812	48
10	675	39
50	512	30
100	426	22
500	318	14
1000	275	10

The results are clear to show that the dielectric constant (ε′) drops drastically with rising frequency, and this result confirms that there is a characteristic dielectric relaxation phenomenon. In the lower frequencies, the high ε value (812 at 1 kHz) is attributed to interfacial or space charge polarization whereby the charge carriers get concentrated at grain boundaries. With frequency, dipoles are unable to quickly reorient with the alternating electric field and this leads to a slow decrease of ε between 275 and 1 MHz. Likewise, the dielectric loss (tan δ) reduces with frequency as well whereby the energy dissipation during high frequencies becomes lower. It is an indication of high dielectric stability and worthiness to high-frequency electronic applications.

4.3 Temperature-Dependent Dielectric Behavior

Both ε and tan δ values rose at high temperatures because of improved mobility of charge carriers and thermal activation of dipole. AC conductivity verses temperature obeyed Jonschers power law showing hopping conduction mechanisms.

Table 3: Dependence of AC Conductivity with Temperature at 1 MHz

Temperature (°C)	AC Conductivity (σ_ac × 10^{-6} S/cm)
30	1.25
50	1.82
100	2.74
150	3.68
200	4.92

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Table 3 demonstrates a definite growth of the AC conductivity (σ_ac) with an increase in temperature which means thermally induced conduction mechanism. This is consistent with the power law behavior of Jonschers, in which the conductivity increases with the increase in the thermal energy of the charge carriers that have reached large enough energy to break the possible impediments and engage in hopping between localized states. The progressive increase of 1.25×10^{-6} S/cm at 30o C to 4.92×10^{-6} S/cm at 200o C indicates that the oxide compound is semiconducting in nature with the increased carrier mobility and dipolar relaxation mechanisms prevailing at elevated temperatures.

4.4 Comparative Analysis

A comparative study involving the dielectric property of the synthesized oxide material against other materials that have been widely studied as oxide materials, including, but not limited to, $BaTiO_3$, $ZnFe_2O_4$ and NiO, is provided in table 4. The parameters to be compared are a synthesis technique, dielectric constant (ε′) at 1 kHz, and dielectric loss (tan δ). The oxide synthesized in the current work through the solid-state method has a large dielectric constant (ε′ = 812) and a comparatively low dielectric loss ($\tan\delta = 48 \times 10^{-3}$), which suggests a good dielectric performance.

Table 4: Comparative Dielectric Properties of Oxide Materials

Material	Synthesis Method	Dielectric Constant (ε′ at 1 kHz)	Dielectric Loss (tan δ × 10^{-3})
Synthesized Oxide	Solid-State	812	48
$BaTiO_3$	Sol-Gel	850	52
$ZnFe_2O_4$	Solid-State	730	60
NiO	Co-precipitation	790	55

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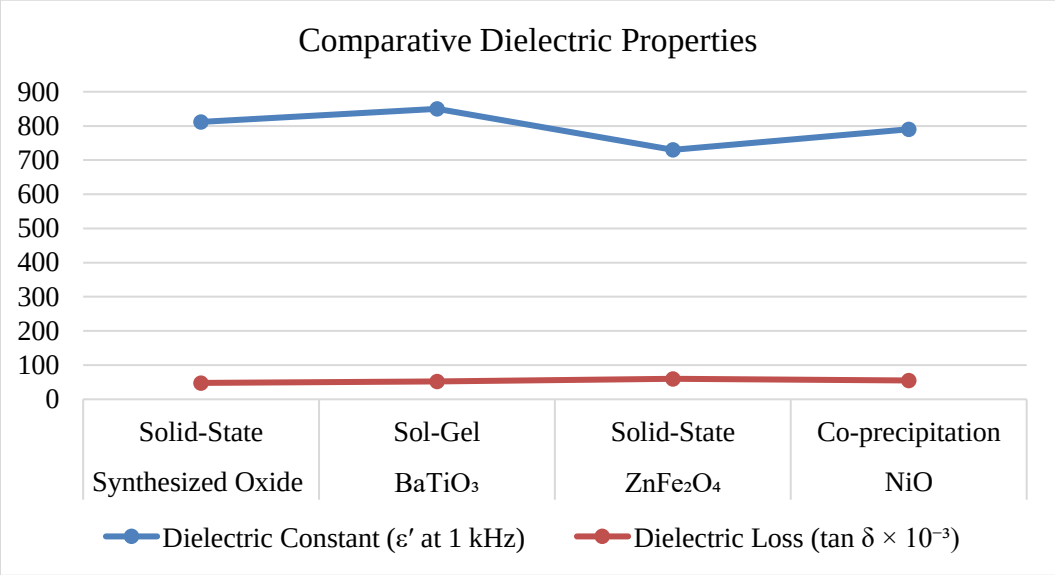

Figure 2: Graphical Representation of Comparative Dielectric Properties of Oxide Materials

The obtained outcomes indicate that the produced oxide material exhibits similar dielectric characteristics to those of $BaTiO_3$, a commonly known ferroelectric material, and lower dielectric loss than $ZnFe_2O_4$ and NiO. It means that the polarization stability is better and the energy dissipation in the material is minimal. The high dielectric constant with low loss indicates that the oxide that was synthesized has a great potential in the use of an oxide in capacitors, sensors and high-frequency electronic equipment.

4.5 Structure–Property Correlation

The models that can be used to explain the dielectric behavior of the synthesized oxide are the Maxwell-Wagner and the model by Koop. The high dielectric constant values are caused by the fine-grained structure and low values of dielectric losses are caused by low porosity. Interacial polarization and hopping conduction is verified by the frequency and temperature-dependant dielectric response.

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4.6 Discussion

The synthesized oxide material is a highly crystallized single-phase material having fine grains (~45 nm) with dense microstructure that was authenticated by XRD, FTIR and SEM. Its dielectric response exhibits great frequency and temperature dependence: the dielectric constant here decreases with frequency because the dipole response is more limited but the dielectric loss also diminishes with frequency which is an indicator of excellent energy efficiency. Increasing temperature, dielectric constant and AC conductivity follow thermally activated hopping of charge carriers, and an increase in dipolar mobility, as predicted by power law of Jonscher. A comparative analysis shows that the material has a high dielectric constant and reduced loss compared to other oxides such as $ZnFe_2O_4$ and NiO which implies the material has a better polarization stability. Comprehensively, the structure-property correlation indicates that the fine, dense morphology increases the interfacial polarization and reduces the losses, hence this material is well adapted as a capacitor, sensor and high-frequency electronic material.

5. CONCLUSION

The current paper was able to prepare one-phase oxide material through the solid-state reaction process and systematically studied the structural and dielectric properties. The phase purity, fine-grained morphology (approximately 45 nm) and the dense microstructure with limited porosity were confirmed by XRD, FTIR, and SEM analyses. Dielectric measurements showed that with increasing frequency the dielectric constant and loss decreased, as would be expected in a typical dielectric relaxation process, whereas temperature dependent measurements showed that the dielectric constant and AC conductivity increased, which is also expected of a using hopping conduction which is thermally activated. The resulting comparative analysis of the synthesized oxide with other oxide materials revealed that the dielectric constant of the material is high, and dielectric loss is relatively low, which indicates that polarization stability is high. All in all, the multi-sequence of phase purity and microstructure fineness as well as the high performance of dielectric performance highlight the possibilities of the material in capacitor, sensor and high frequency electronic device applications.

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