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SYNTHESIS AND CHARACTERIZATION OF $CaAl_2O_4:Dy^{3+}$ CERAMIC

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Abstract

Spinel oxides have potential application as industrial refractory material in chemical catalysis, in electronic industry, in integrated circuit technology. Dy^{3+} doped calcium aluminate ($CaAl_2O_4:Dy^{3+}$) has been prepared by the solid state reaction method. The phase composition of the sample is analysed using X ray diffraction (XRD) technique. The dielectric loss, dielectric constant, AC conductivity and impedance are studied at room temperature as a function of frequency (500 Hz- 5MHz). The UV visible spectrum of the $CaAl_2O_4:Dy^{3+}$ is studied and band gap of the material is found to be 3.8 eV.

Keywords: XRD, UV-visible spectrum, Dielectric response

1. Introduction

Materials having high dielectric constant, better electric properties, good mechanical strength and easiness of processing are very much essential for the development of electronic devices capable of working at high operating frequencies. In the fabrication of embedded capacitors for integrated electronic devices, high dielectric constant materials are needed[1,2].

Spinel oxides having general formula AB_2O_4($A^{2+}B_2^{3+}O_4^{2-}$) are considered due to their structural features which enables customisation of various properties. They have potential application as industrial refractory material in chemical catalysis, in electronic industry, in integrated circuit technology [3]. Due to the possession of exceptional physical properties such as high mechanical strength at elevated temperature, high melting point, low permittivity, low loss tangent and resistance to radiation damage, aluminium based compounds were considered for study, among various spinel oxides [4].

Mono calcium aluminate ($CaAl_2O_4$) is a member in the family of stuffed tridymite tetrahedral framework structure. Due to its special characteristics such as prolonged afterglow time, better chemical stability and low toxicity, $CaAl_2O_4$ is having potential applications in many fields [5].

In the present work, Dy^{3+} doped $CaAl_2O_4$ is synthesised by solid state reaction method. The structural characterisation is carried out on the basis of XRD pattern and the band gap of the material was calculated using UV-visible spectrum. The dielectric constant, dielectric loss and AC conductivity properties of the material is studied.

2. Experimental

The $CaAl_2O_4$: Dy^{3+} was prepared via solid state reaction technique. The starting reagents were high purity (99.99%) $CaCO_3$, Al_2O_3 and Dy_2O_3. The raw materials were weighed stoichiometrically and mixed thoroughly for 2h in an agate mortar with distilled water as the medium, dried and then fired at 1150^{o}Cfor 4h. Then the sample is finely ground. For dielectric studies the sample is pelletized having diameter of 8mm using a hydraulic press.

The structural analysis of the material is investigated by XRD technique.(XRD model D8 Advance Bruker). The UV-Visible absorption spectrum is carried out using Perkin Elmer Lambda 365 UV Vis spectrometer. The dielectric study of $CaAl_2O_4$: Dy was carried out using the instrument HIOKI 3532 LCR HITESTER in the frequency range of 500 Hz to 5 MHz

3. Results and Discussion

3.1. XRD Analysis

The XRD pattern of $CaAl_2O_4$: Dy^{3+} is shown in Fig 1.The diffraction peaks of the sample are in good agreement with ICDD card no.88-2477. The crystal structure of the material is monoclinic and it belongs to $P2_1/c$ space group.

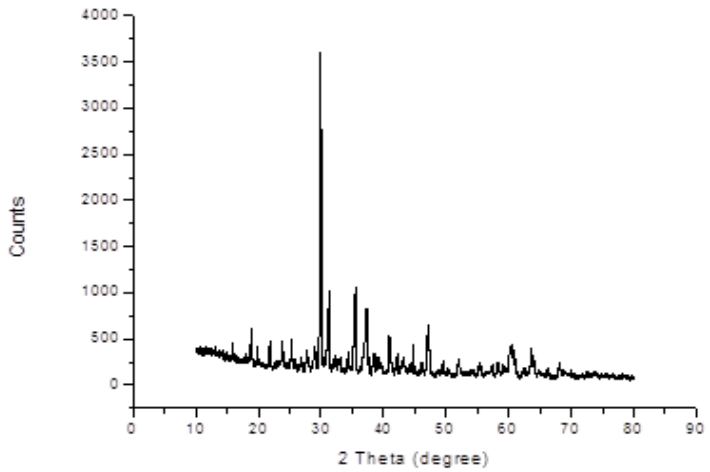

Fig 1. XRD pattern of $CaAl_2O_4$: Dy^{3+}

3.2. UV Visible Spectral Studies

The UV–visible absorbance spectrum is carried out in the range 200-700 nm. The absorbance spectrum of $CaAl_2O_4$: Dy^{3+} is shown in Fig 2(a). It shows that the maximum absorption occurs at 255nm, near UV region. It is evident that no absorption occurs in the visible region, thereby making it appropriate for optoelectronic applications.

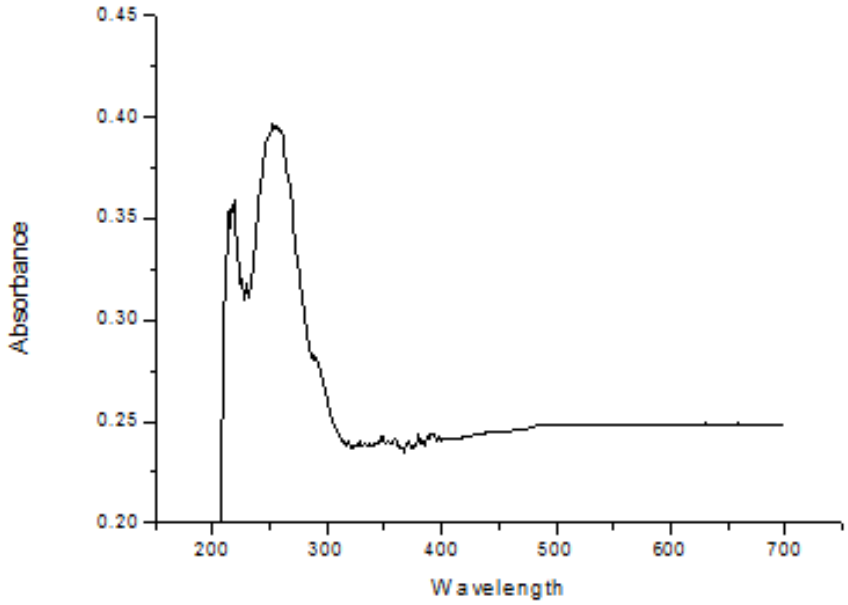

Fig 2(a). Absorption spectrum of $CaAl_2O_4$: Dy^{3+}

As per Tauc's relation, the relation between absorption coefficient (α) of a material with optical band gap (E_g) and photon energy (hν) is as follows.

$$\alpha h\nu = A(h\nu - E_g)^n \quad (1)$$

Where A is a constant for different transitions and n = 1/r, where r is an index which takes values 1/2, 3/2, 2 and 3 depending on the nature of electronic transitions, such as direct allowed, indirect allowed, direct forbidden or indirect forbidden responsible for absorption [6]. A linear portion is obtained in the graph of photon energy (hν) versus $(\alpha h\nu)^2$, where α is the absorption coefficient. Extrapolating the linear portion of the plot results in the band gap value of 3.8eV. This high band gap value of 3.8 eV shows that the material exhibits dielectric behaviour to induce polarization when strong radiation is incident on the material [7] and is shown in Fig 2(b).

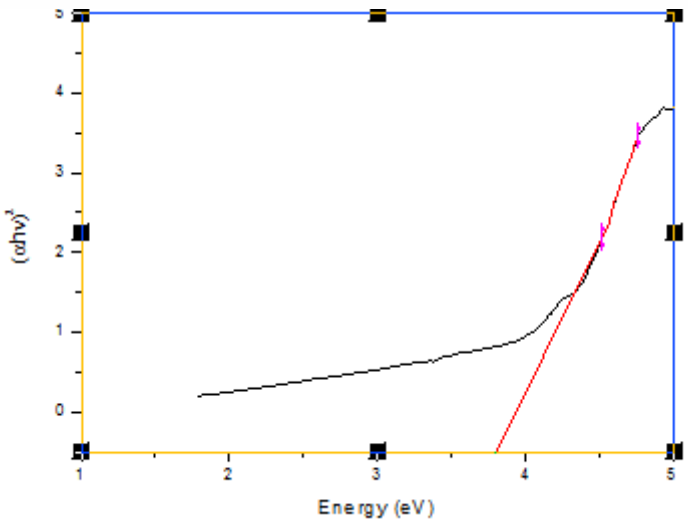

Fig 2(b) Tauc's plot

3.3 Dielectric response

The dependence of dielecric constant in the frequency range from 500Hz-5MHz is shown in Fig3(a). The dielectric constant is calculated from the relation

$$\varepsilon_r = Cd/\varepsilon_0 A \qquad (2)$$

where C is the capacitance, d is the thickness, A is the cross-sectional area of the sample and ε_0 is the free space permittivity of the sample.

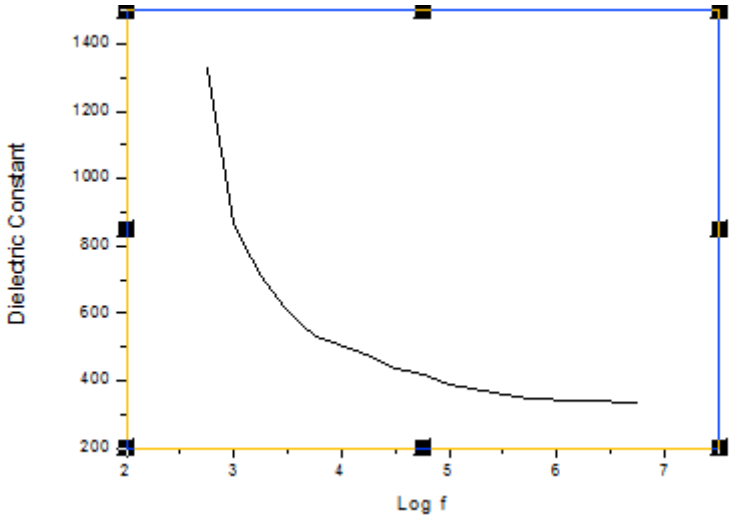

Fig3(a) Dielectric constant vs log f

The material exhibits dielectric dispersion behaviour. ie., with increase in frequency dielectric constant decreases and becomes constant at higher frequencies. After a certain critical frequency, charge carriers may not be able to trail the alternation of the ac electric field and hence dielectric constant declines with elevation in frequency and remains constant. In low

frequencies, surface polarisation also plays a vital role in determining the dielectric constant [8]. The variation of dielectric loss ($\tan\delta$) with frequency is shown inFig 3 (b)

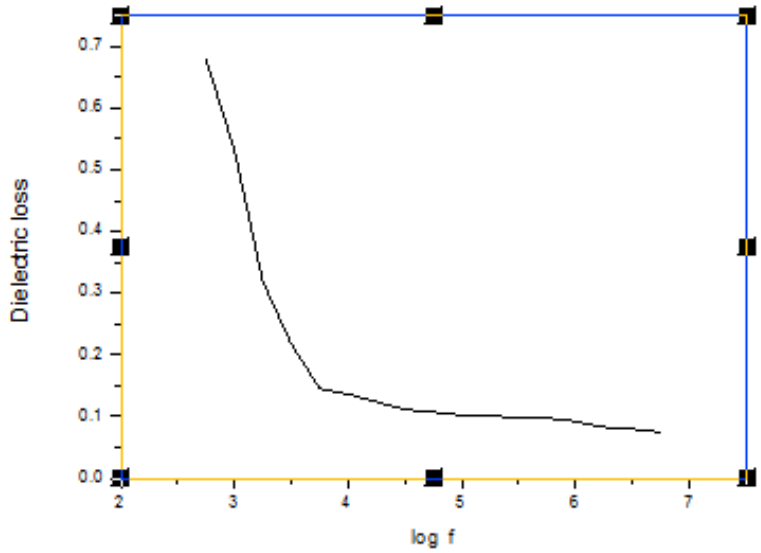

Fig 3(b) Dielectric loss vs log f

With increase in frequency, $\tan\delta$ decreases continuously which indicates the presence of space charge polarisation. Very low dielectric loss at high frequency satisfies one of the required criteria of the material to be used in high frequency device applications [8].

Using the relation

$$\sigma_{ac} = 2\pi f \varepsilon_0 \varepsilon_r \tan\delta \qquad (3)$$

where ε_0 is the permittivity of free space ,ε_r is the dielectric constant of the material and f is the frequency of the applied field, ac conductivity can be calculated.

The variation of ac conductivity(σ_{ac}) as a function of frequency is represented in Fig 3(c).

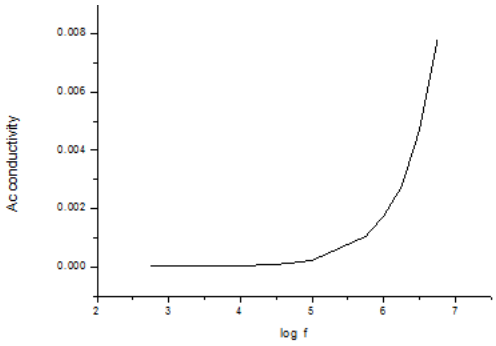

Fig 3(c) ac conductivity vs log f

AC conductivity shows an increasing trend with increase in frequency. With the increase in applied frequency, the charge carrier transfer rate also increases, which in turn assists the charge carrier to be liberalised from different trapping centres. The increase in ac conductivity at higher frequency also confirms small polaron hopping in the samples.[9]

4. Conclusion

The $CaAl_2O_4:Dy^{3+}$ was successfully synthesized by solid reaction method. The XRD pattern of synthesized $CaAl_2O_4:Dy^{3+}$ matches well with standard data. The band gap of the material was found to be 3.8eV. The studies of frequency dependant dielectric parameters such as dielectric constant, dielectric loss and ac conductivity disclose conventional ceramic behaviour. The results obtained can aid the usage of this material in electronics industry.

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