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Synthesis and Characterization of Rare Earth doped Nanoparticles for electrical and Dielectric properties

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ABSTRACT: This paper deals with the measurement of dc conductivity and the dielectric constant of the LaF_3: Pr^{3+},Ho^{3+} nanoparticles using sophisticated Hioki 3532 LCR Meter. For this, doped LaF_3: Pr^{3+}, Ho^{3+} nanocrystals have been successfully synthesized by microwave assisted technique using deionised water as solvent. The X-ray diffraction, SEM, TEM and selected area electron diffraction SAED pattern have been used for identification of crystal structure. Synthesized nanocrystals are found to belong to hexagonal crystal system with space group P3c1 and lattice parameters a = b = 7.080 A^0 and c = 7.238 A^0. Using Scherrer equation average particle size has been estimated which is found to be 11.17nm. SEM pattern shows dispersed, hexagonal, discs like, assorted particles with traces of some aggregates. SAED pattern indicates formation of strong diffraction rings corresponding to the (110), (111), (300), and (221) reflections which is in agreement with the hexagonal LaF_3 structure. Nanocrystals of hexagonal geometry with particle size 15nm have been traced by TEM analysis which is in agreement with the average crystalline size obtained from XRD studies (11.17nm). The dielectric studies shows that the ε' and ε" falls rapidly with applied frequency. Both ε'and ε"exhibit normal dielectric behaviour. Graph of log ε" vs log frequency shows near linear nature and graph of Tanδ vs log frequency shows that there is decrease in Tanδ with increasing value of frequency. This nature is attributed to the interface charge relaxation at the grain boundaries. The conductivity of the synthesised LaF_3: Pr^{3+}, Ho^{3+} nanocrystals at room temperature is found to be 1.565 x 10^{-3}/ Ωcm. Thus by doping LaF_3 the ionic conductivity of the sample is observed to be enhanced as compared to the conductivity of pure LaF_3 having conductivity of the order of 10^{-6} / Ωcm. Resistivity at room temperature is found to be 638.977Ωcm.

Keywords: Lanthanum fluoride, conductivity, dielectric constant, dielectric loss.

INTRODUCTION: Rare earth elements find wide applications in the field of lasers glasses, magnets and in many other applications. Lanthanum based fluorides are important materials for their luminescence (optical) properties. In the form of thin films [1-3] or as nanoparticles [4-6], rare earth doped lanthanum fluorides show interesting up-conversion effect from near IR to visible light. Also X ray luminescence has been observed on doped lanthanum fluorides [7].

RESISTIVITY AND CONDUTIVITY MEASUREMENT: Many solids show high ionic conductivity (> $10^{-4}Scm^{-1}$) and find immense use in diverse technological applications. Many of these solids which may be good electronic conductors are often referred to as "mixed conductors". The term "superionic conductor" or "fast ion conductor" are good ionic conductors with negligible electronic conductivity [8].

Ionic conductivity exists in LaF_3 samples due to fluoride ions [9]. Fluoride is one of the smallest anion with high mobility, exhibiting high ionic conductivity in solid state. With the development of nanotechnology, it was possible to increase the ionic conductivity of fluorides by fabricating the corresponding nanocrystalline bulk materials. Fluorides have been widely used as components of sensors, batteries and actuators due to their excellent electrolytes [8]. They also form important components of toothpaste to prevent the tooth decay and as additive of wear and crack resistance materials [10] as well as of welding materials [9].

The Conductivity of the sample has been measured using sophisticated Hioki 3532 LCR Meter, with a conventional two terminal sample holder. The powder sample of the synthesized nanocrystals were put in the form of pellets and coated with silver on both the sides and kept in the sample holder for Conductivity measurement. Proper care has been taken to ensure that the silver paste does not spread to the sides of the cross sectional face of pellet. Figure 1 shows a typical Hioki 3532-50 LCR Meter. The sample is placed in the Hioki 9262 test fixture connector and programmable software is chosen to read the resistance of synthesized sample at room temperature.

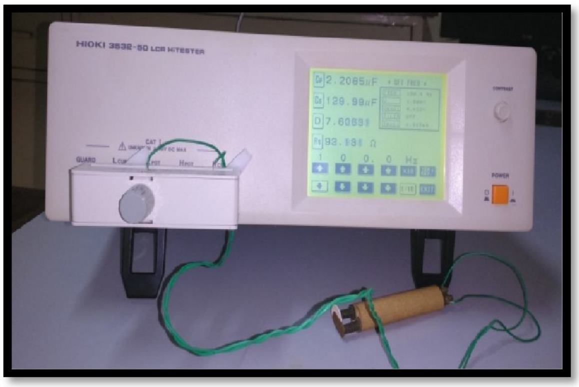

Figure 1: Hioki 3532-50 LCR Meter.

The dc electrical conductivity (σ_{dc}) of the pellet was calculated using the relation.

$$\sigma_{dc} = t / RA \qquad \textit{Equation 1}$$

Where, R is the resistance measured from LCR meter
t is the thickness of the sample
A is the area of the face of pellet in contact with the electrode

$$\text{Resistivity } \rho = 1 / \sigma_{dc} \qquad \textit{Equation 2}$$

DIELECTRIC MEASUREMENTS: The capacitance **(Co)** of the sample taken in the form of pellets is obtained by using the formula Co = $A\varepsilon_0/d$ where **A** is the cross-section area of the pellet, $\boldsymbol{\varepsilon_0}$ is the permittivity of free space and **d** is the thickness of the samples. The HIOKI 3532-50 LCR HITESTER meter has been used to record the capacitance (C_s) of the samples in the frequency range 100Hz to 5MHz. The capacitance values thus obtained was used for calculation of dielectric constant and dielectric loss values gives by $\varepsilon' = C_s/C_0$ and $\varepsilon'' = \varepsilon' \text{x } D$ where D is the dissipation factor obtained from the LCR meter, and

$$\tan\delta = \varepsilon''/\varepsilon' \qquad \textit{Equation 3}$$

OBSERVATIONS:

Sample	Thickness t (cm)	Area A(cm^2)	Conductivity σ (/Ωcm)	Resistivity ρ (Ωcm)
$LaF_3:Pr^{3+}, Ho^{3+}$	0.110	1.326	1.565×10^{-3}	638.97

Table 1: Resistivity and Conductivity of $LaF_3:Pr^{3+}$, Ho^{3+} at room temperature.

f Hz	C_s	Dielectric constant $\varepsilon' = C_s/C_0$	Dielectric Loss $\varepsilon'' = \varepsilon'$x D	Tanδ= ε''/ ε'
100	502μF	$4.703 \text{x} 10^6$	$46.982 \text{x} 10^6$	9.989
1k	55μF	$0.515 \text{x} 10^6$	$5.144 \text{ x} 10^6$	9.988
10k	5.2 μF	$0.048 \text{x} 10^6$	$0.479 \text{x} 10^6$	9.979
100k	305nF	$2.857 \text{x} 10^3$	$28.541 \text{x} 10^3$	9.989
1M	23.96nF	$0.224 \text{x } 10^3$	$1.930 \text{x} 10^3$	8.616
5M	2.62nF	$0.0245 \text{x} 10^3$	$0.077 \text{ x} 10^3$	3.142

RESULTS AND DISCUSSION: The dielectric properties of the synthesized samples were studied by plotting the graphs(i) dielectric constant versus log of frequency(Figure2) (ii) dielectric loss versus log of frequency (Figure 3) (iii) log of dielectric loss versus log of frequency(Figure 4) and iv) Tanδ versus log of frequency (Figure 5).

It has been observed from the graphs of dielectric constant versus log of frequency and dielectric loss versus log of frequency that there is an exponential decrease in both the parameters with the increase in the frequency which is the normal behaviour of dielectric materials as the dipoles lag behind in orientations proposed by Debye [11-12].

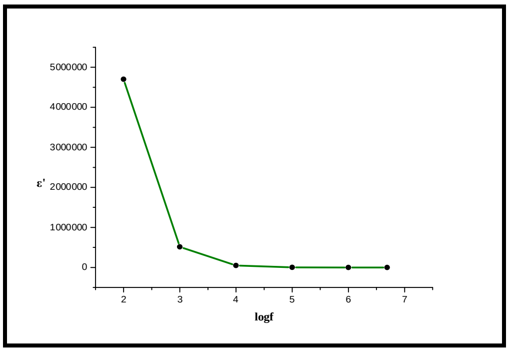

Figure 2: Variation of dielectric constant (ε') with log frequency of $LaF_3:Pr^{3+}$, Ho^{3+}.

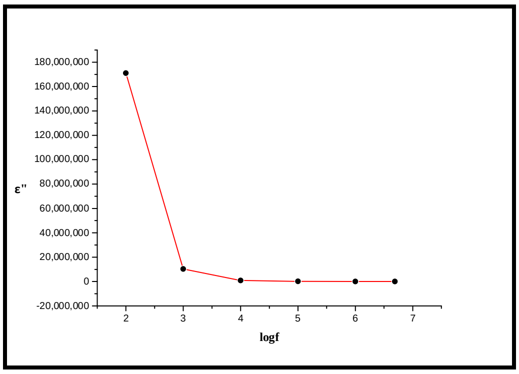

Figure 3: Variation of dielectric loss (ε'') with log frequency of $LaF_3:Pr^{3+}$, Ho^{3+}.

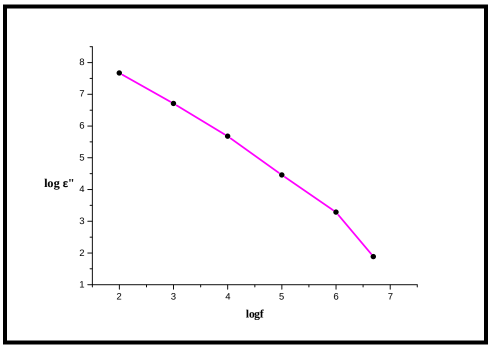

Figure 4: Variation of log dielectric loss (ε") with log frequency of LaF_3:Pr^{3+}, Ho^{3+}.

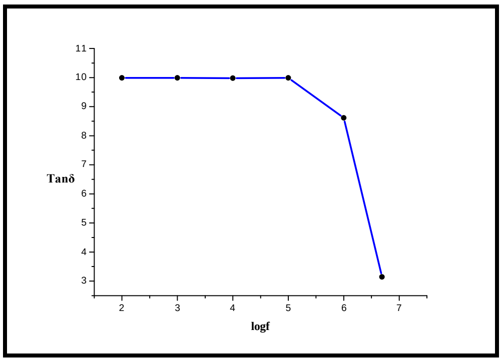

Figure 5: Variation of Tanδ versus log of frequency of LaF_3:Pr^{3+}, Ho^{3+}.

CONCLUSION: The dielectric studies shows that the ε' and ε" falls rapidly with applied frequency. Both ε' and ε" exhibit normal dielectric behaviour. Graph of log ε" vs log frequency shows near linear nature and graph of Tanδ vs log frequency shows that there is decrease in Tanδ with increasing value of frequency. This nature is attributed to the interface charge relaxation at the grain boundaries. The conductivity of the synthesised LaF_3: Pr^{3+}, Ho^{3+} nanocrystals at room temperature is found to be 1.565 x 10^{-3}/ Ωcm. Thus by doping LaF_3 the ionic conductivity of the sample is observed to be enhanced as compared to the conductivity of pure LaF_3 having conductivity of the order of 10^{-6} / Ωcm. Resistivity at room temperature is found to be 638.977Ωcm.

FUTURE SCOPE: The dielectric studies of the synthesised nanocrystals can be studied by varying the temperature and the corresponding conductivity and resistivity. Doped LaF_3 nanocrystals can be use as dielectric medium. Also there are reports on the use of LaF_3 as thin film oxygen detectors [13]. This part can be extended for development of LaF_3 sensors in future.

REFERENCES:

1. S. Sivakumar, J. C. Boyer, E. Bovero, F. van Veggel, *J. Mater. Chem.*, 19 (2009) 2392.
2. R. Sivakumar, F. van Veggel, M. Raudsepp, *J. Am. Chem. Soc.*, 127 (2005) 12464.
3. S. Fujihara, K. Tokumo, J. Fluorine Chem. 130 (2009) 1106.
4. J. R. DiMaio, B. Kokuoz, J. Ballato, Opt. Express 14 (2006) 11412.
5. D. B. Pi, F. Wang, X. P. Fan, M. Q. Wang, Y. Zhang, Spectrochim, *Acta Part A*, 61 (2005) 2455.
6. H. X. Shen, F. Wang, X. P. Fan, M. Q. Wang, J. Exp. Nanosci. 2 (2007) 303.
7. Y. F. Liu, W. Chen, S. P. Wang, A. G. Joly, S. Westcott, B. K. Woo, *J. Appl. Phys.*, 103 (2008).
8. W. Jeferey, (1997) Sensor and Actuators B: Chemical, Australia.
9. L. Feng, Y. Chung, L. S. Bao (2003) Transactions of Nonferrous Materials Society, China.
10. W. X. Hong, S. L. Song, Z. Z. Da (2004) Transactions of Nonferrous. Materials Society, China.
11. D. K. Pradhan, R. N. P. Choudhary and B. K. Samantaray (2008) Studies of Dielectric relaxation and AC Conductivity behavior of Plasticized Polymer Nanocomposite Electrolytes, India.
12. M. Marzantowicz, J. R. Dygas, F. Krok, Z. Florjanczyk and E. Zygadlo-Monikowska, (2006) Crystalline phases, morphology and conductivity of PEO: LiTFSI electrolytes in the eutectic region, Japan.
13. D. A. Kumar, S. Selvasekarapandian, H. Nithya, A. Sakunthala and M. Hema, (2010) Dielectric, modulus and impedance analysis of LaF_3 nanoparticles, India..