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Synthesis and characterization of CuS nanocomposites by using alumina channel-technique

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Abstract: CuS nanocomposites synthesized using Alumina crystallite as the medium. A core shell nanostructure developed by suitable oxidation of the composite. X ray diffraction (XRD) of synthesized CuS nanocomposites revealed the presence of two crystal structures: one is tetragonal and the other is monoclinic. All salient diffraction peaks can be closely matched to the monoclinic and tetragonal structures. The PL spectrum of the CuS nanopartic;les shows a peak centered at 410nm and 460 nm which is shorter than submicron CuS , indicating a CuS nanocrystals obtain in this work in quantum size. Materials exhibited good electrical and optical properties. Due to high dielectric constant and photo luminescence property this CuS nanocomposites can be used in different electronic and optical devices.

Keywords: nanocomposites, alumina, spectrum

INTRODUCTION

There are various techniques for synthesis of Nanocomposites. Nanocomposites materials having a phase with nanoscale dimensions dispersed within a matrix. Nanocomposites matrix acquired significance due to interesting physical properties. We have synthesized CuS nanocomposites by using alumina as medium and suitable oxidation of the composite a core shell nanostructure. Synthesized CuS nanocomposites exhibited good electrical and optical properties.

EXPERIMENTAL

The channels within a high charge density sodium flurophlogopite Mica channels having composition, Na_4 Mg_6 Al_4 Si_4 O_{26} F_4, xH_2O [Kodama and Komarneni 1999)] were used to grow CuS nanowires of diameter (2.5nm).

Alumina of the above mentioned composition is commonly referred to as Na-4 Mica and prepared by a sol gel method. The chemicals $Al(NO_3).9H_2O$,$Mg(NO_3).6H_2O$ and $Si(OC_2H_5)_4$ dissolved in C_2H_5OH and stirred for 3hr. after three hours the final composition reaches $MgO.Al_2O_3.2SiO_2$ .The gel was dried at 373K and then calcilation done at 748K for 10hr.The gel powder was mixed thoroughly with an equal amount of NaF powder. The treatment at 1163K for 16 hr in a platinum crucible. After washing the reaction products with deionized water and then with saturated boric acid .final material washed with 1M NaCl to saturate all exchange sites with Na^+. After subsequent washing with deionized water. Solid was dried at 333K for 3 days. 2 gm of Alumina powder was immersed into 25cc of 0.4(M) $Cu(SO_4)$ and added 0.5 (M) NaCl solution. An ion exchange reaction started which continued for a period of 4 weeks at 303K. By centrifuge after separating the ion exchanged powder from the solution. It was subjected to a heat treatment in H_2S at 673K for 1hr.This produced CuS wires within the Alumina channels.

RESULT AND DISCUSSION

Synthesized powder identified by diffraction (XRD). The synthesized material revealed the presence of two crystal structures: one is tetragonal and the other is monoclinic. The vertical lines indicate the positions and intensities of XRD peaks of powder CuS.The insets show the corresponding XRD patterns at higher resolution. All salient diffraction peaks closely matched to the monoclinic and tetragonal structures. Among these two structural phases, the monoclinic phase appears to be more abundant based on the XRD data. **(Fig 1).Fig 2** shows the PL spectrum from the as synthesized CuS nanoparticles (excitation at 360 nm)at room temperature .

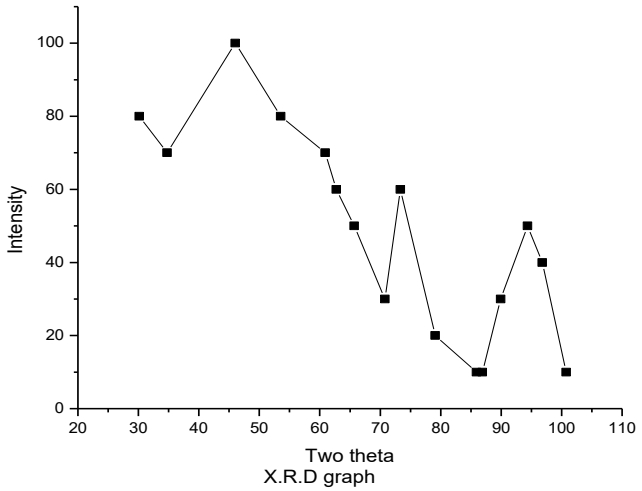

Fig.1: X.R.D graph

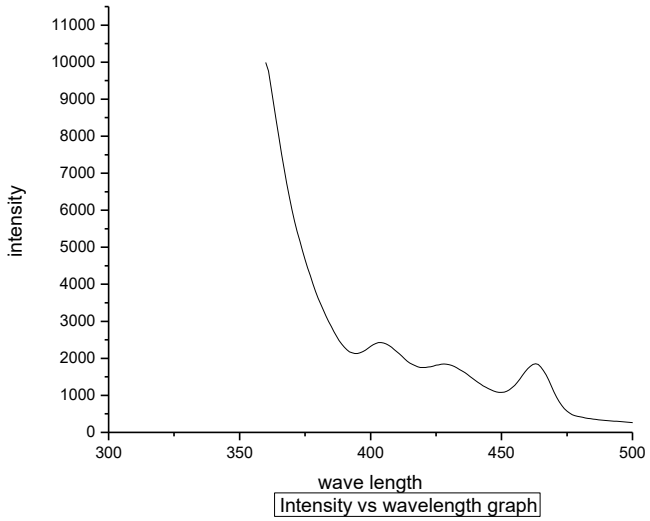

Fig. 2: Intensity vs wavelength graph

The PL spectrum of the CuS nanoparticles shows a peak at 410nm and 460 nm which is shorter than submicron CuS , indicating a CuS nanocrystals obtain in this work in quantum size. **Figure 3** gives the variation of surface resistivity of a specimen (subjected to a 2hr oxidation treatment) as a function of inverse temperature.

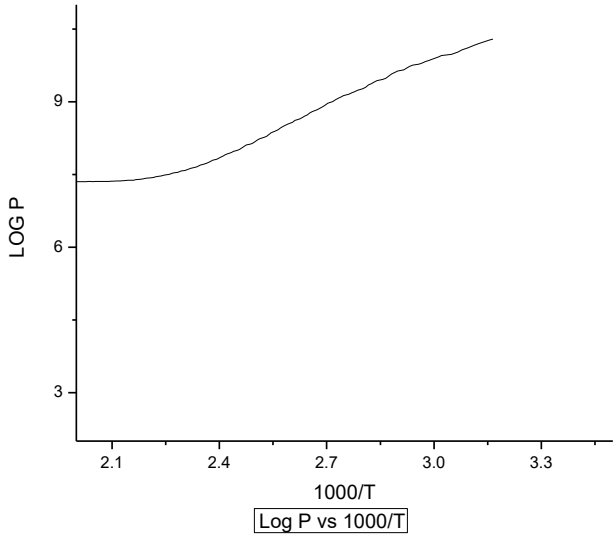

Fig. 3: Log p vs 1000/T graph

The electrical conduction evidently is controlled by Al channel shell and Cu^{+} ion. Hence a small polaron conduction model (Mott1968) was found to be operative in this system. In this model resistivity ϱ is given by:

$$\varrho = \frac{KTR}{\vartheta e^2 c(1-c)} e^{(2\alpha R)} e^{(\frac{W}{KT})}$$

where K is the Boltzman constant, T the temperature, R the intersite separation, ϑ the optical phonon frequency, e is the electronic charge, c is the ratio of $[Cu^+]/[Cu]_{total}$, α the localization length of the localized state at the Cu ion site and W is the activation energy for hopping condition. The experimental data were least square fitted to above equation. From the **Fig 4** it is shown that dielectric properties (permittivity, loss tangent, and resistivity) of CuS Nanocomposites at different sintering temperature, were measured within 50 Hz to 1 MHz frequency range. As shown in diagram tan δ (log F).

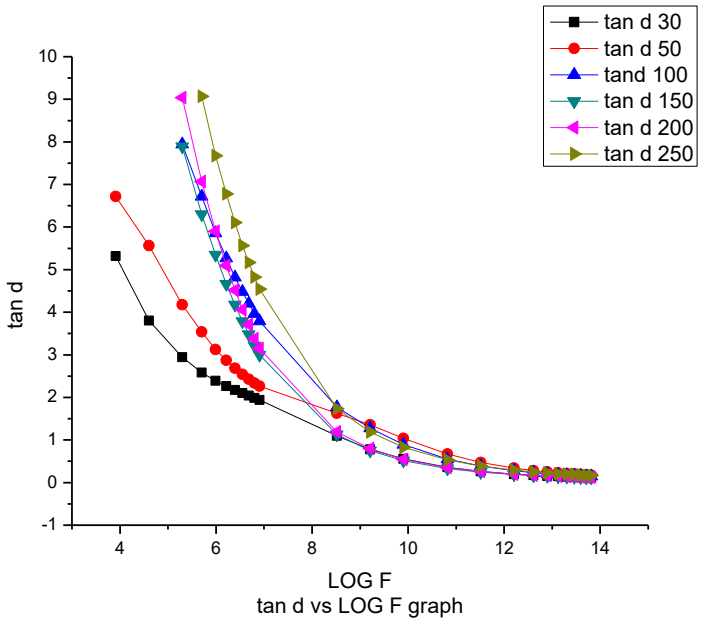

Fig. 4: tan δ vs Log F graph

Relative Dielectric values for tan δ do not exceed 6.74776, therefore they are within interval .1193 to 6.74776. In the investigated frequency range, especially after curve inflection point on frequencies higher than 50 KHz, values of tan δ are approximately constant, about tan δ =1.094. Meanwhile, in measuring range, obtained values for εr are considerably lower in relation to well-known results, accomplished for nonlinear active dielectrics. From the **Fig 4** it is shown that dielectric properties (permittivity, loss tangent, and resistivity) of CuS Nanocomposites at different sintering temperature, were measured within 50 Hz to 1 MHz frequency range. As shown in diagram tan δ (Log F) (relative Dielectric), values for tan δ do not exceed 6.74776, therefore they are within interval .1193 to 6.74776. In the investigated frequency range, especially after curve inflection point on frequencies higher than 50 KHz, values of tan δ are approximately constant, about tan δ =1.094. Meanwhile, in measuring range, obtained values for εr are considerably lower in relation to well-known results, accomplished for nonlinear active dielectrics. **Fig.5** shows frequency dependence of dielectric loss.

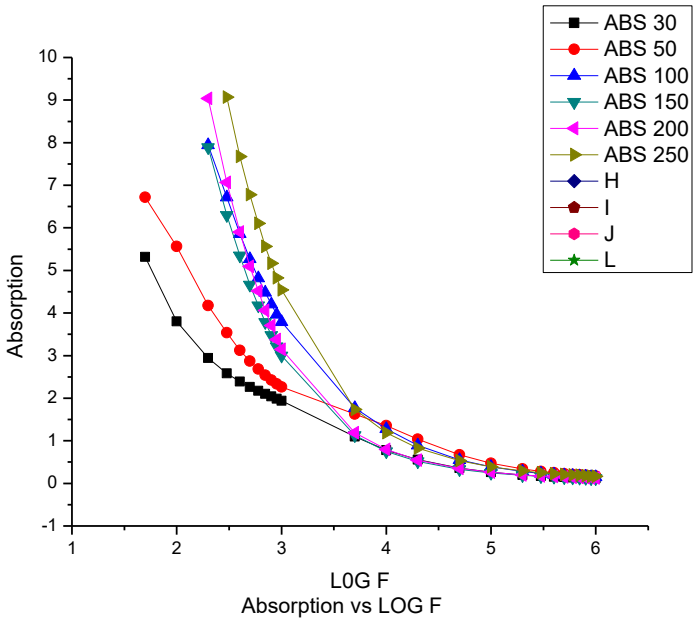

Fig. 5: Absorption vs Log F

Values of dielectric loss between 1193 to 8.973 have been found. The distinctly differences associated with sintering temperature can be observed in the vicinity of the inflection points. Given these absorption values, we can explicitly conclude that resonant method is not appropriate for investigation of active dielectric with such high dielectric losses. Resonant method is convenient for cases when absorption is lower than 0.05 and high frequencies up to several hundreds of MHz. For high loss values (loss → 1), it is difficult to accomplish resonance and therefore attenuation occurs, which can be clearly seen in **Fig.6.** It is clear that for a fixed temperature firstly conductivity increases then it shows a sharp fall and becomes saturate for a certain frequency range. The nature of curve is similar for all temperature, but values are shifted upward as the temperature is raised. The sample exhibits conductivity with highly localized carriers bound to lattice with accompanying lattice strain i.e. with polaron conduction[1,2].

The a.c conductivity σ can be related to imaginary part of dielectric constant (ε") $\sigma = \varepsilon_0 \omega \varepsilon$, Where ε_0 is the permittivity of free space and ω is the angular frequency[3]. The activation energy for conduction (*Ea*) in the entire region as shown in Fig. for various frequencies was calculated by fitting different regions with the equationis $\sigma_{ac} = \sigma_0 e^{\frac{-Ea}{kBT}}$ .The ionic conductivity can be related to diffusion constant (D) using Einstein relation $\sigma/D = N/e^2 k_B T$. This relation can be extended further as $\ln(k_B T\ \varepsilon") = \ln(Ne^2 D_0/\ \varepsilon_0) - \ln(\ \omega\ ) - E_t/k_B T$

Where, *Do*, E_t is the maximum diffusion coefficient and total activation energy due to bulk and surface conduction, respectively. Fig shows the variation of ac conductivity with frequency sharp decrease in upto 1MHz observed by Jonscher, the conductivity σ is analyzed using Jonscher power square law equation $\sigma = A\ \omega^s$. Where ω is angular frequency,'*A*' is a constant and the exponent ,'*s*' is a frequency-dependent parameter having values less than unity . The value of exponent's' at different temperature is calculated by fitting the curve. Above graph shows a relationship between capacitance and LOG F .When we increase the temperature with increase of frequency capacitance decreases (at 30 °c and 50 °c capacitance shows a sharp fall i.e at higher frequency capacitance shows low value within a temperature range 150 °c to 250 °c capacitance also decreases with frequency but within a frequency range 1KHz to 50 KHz capacitance shows a constant value i.e it's

independent with frequency[4,5]According to the theory capacitance of nanostructure material is primarily due to different types of polarizations present in material. Nanostructured material possesses enormous number of interfaces, and the large number of defects present in these interfaces and can cause a positive and negative space charge distribution resulting in space charge polarization. The high value of capacitance at low frequencies is mainly due to space charge polarization and rotational polarization. The values are shifted upward as temperature increases(from 30 °c to 50 °c and also from 150 °c to 250 °c).As the temperature increases more and more dipole will be oriented resulting in increased value of dipole moment and also dielectric constant and capacitance of sample also[6,7].

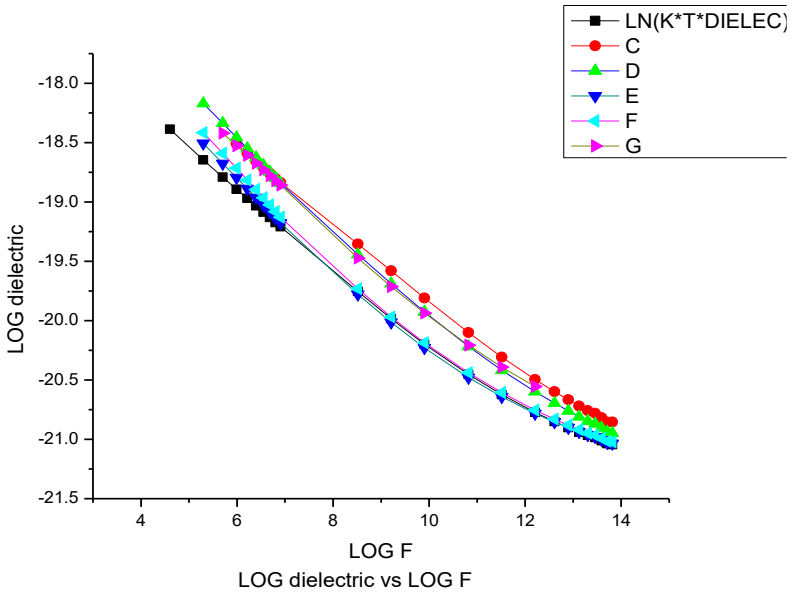

Fig. 6: Log dielectric constant vs Log F

CONCLUSION

Different nanocomposites were synthesized using Alumina crystallite as the medium .In some cases by sulfidation treatment a core shell nanostructure could be generated. Cupper sulfide (CuS) core shell nano rod exhibited a number of optical absorption peaks which arose because of their structural characteristics. Cupper sulfide nanowires of diameter, average size 25nm, were grown within nano aluminium channels. Due to high dielectric constant and photo luminescence property this CuS nanocomposites can be used in different electronic and optical devices.

REFERENCES

1. J. Maji and K. Maji ; Synthesis and Characterization of CUS Nanocomposites by Using Na-4 Mica Template; international journal for research in emerging science and technology, 2016,3, 4, 77-83
2. Nisha J Thayrali, R Raveendaran, Alexandar Varghese, P. G. Vaidyan Chithra, Indian J. Eng & Mater. Sci., 2008, 15, 489.

3. Jelena Purenović, Vojislav V. Mitić, Marjan Randjelović, Branko Matović, Milovan Purenović; Electrophysical Properties of Microalloyed Alumo-Silicate Ceramics as Active Dielectric, Serbian Journal of Electrical Engineering, 2013,10, 1, 175-184
4. Purenović Jelenaa, Mitić Vojislav V.b, Ranđelović Marjanc, Matović Brankod, Purenović Milovanc; Electrophysical properties of microalloyed alumo-silicate ceramics as active dielectric(naslov ne postoji na srpskom) Serbian Journal of Electrical Engineering, 2013, 10, 1, 175-184; doi:10.2298/SJEE1301175P
5. A. Despic, D. Drazic, M. Purenovic, and N. Cikovic; Electrochemical Properties of AluminiumAlloys Ccontaining Indium and Thallium, Journal of Applied Electrochemistry, 1976, 6, 6, 527 – 542.
6. M. Randjelovic, M. Purenovic, A. Zarubica, J. Purenovic, I. Mladenovic, G. Nikolic; Alumosilicate Ceramics based Composite Microalloyed by Sn: An Interaction with Ionicand Colloidal Forms of Mn in Synthetic Water, Desalination, 2011, 279, 1-3, 353 – 358.
7. L.M. Peng, Z. Li, H. Li: Effects of Microalloying and Ceramic Particulates on MechanicalProperties of TiAl-based Alloys, Journal of Material Science, 2006, 41, 22, 7524 – 7529.

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