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Original Article

Structural, Magnetic and Dielectric Properties of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ Sol-gel Synthesized Nano Thin Films

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Abstract: A thin magnetic oxide layer with composition $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ (0.0<x<1) is produced by the SILAR process. X-ray diffraction, cation distribution and infrared spectra were investigated as structural features. Permittivity and dissipation factor are properties of interest. Magnetic properties were studied using MOKE. The lamellae have been shown to have spinal structures. As Co grows, the lattice constant also increases. Observed I.R. spectra show tetrahedral and octahedral bonds. Increase in the dielectric properties because enhancement of space charge polarization.

Keywords: Magnetic oxide layer, MOKE, Thin films, SILAR, VSM, XRD

1. INTRODUCTION

Because of less availability of nonconventional energy sources it is necessary to develop safe, green and clean energy sources. J. H. Kim et al. (2011). To overcome the shortage of energy, in electronic devices and vehicles electronic battery is used. H. Ning et al. (2015). But due to low power density and maintenance it is not convenient to use Lithium-ion batteries, sodium-ion batteries, and super capacitors has been attracted much attention, because they embrace the great potential in an extensive range of applications. H. Zhou et al. (2014). Different metal oxides like TiO_2.P.M.Kulalet al. (2011). RuO_2.A. D. Jagadale et al. (2014). MnO_2. U. M. Patil et al. (2014). Fe_2O_3. S. Jeon et al. (2020). These oxides are prominent oxides for capacitors. Transition metal oxides like RuO_2 is very much efficient material as super capacitor electrodes because of its high capacitance with low ESR, good electrical conductivity, and broad potential window (0.0-1.0 V).B. Y. Fugare et al. (2017).The usage of thin film technology has transformed the area of electronics, optics, energy storage devices super capacitors, sensors and magnetism, etc. G. Brammertz et al. (2016).The SILAR (Successive Ionic Layer Adsorption and Reaction) technique was reported for preparation of oxide thin films. Ristov et al. (1985).The method of synthesis, grain size and chemical composition can affect capacitance of capacitor. Because of their low price, environmental concern, oxidation states, and the large abundance ferrites are the potential electrodes for super capacitors X. Yao et al. (2014).For the production at industrial level the synthesis process is simple and suitable. MFe_2O_4 (M = Zn, Ni, Mn, Mg, Co) ferrites have been used in super capacitor. Due to participation of two ions in redox reactions these oxides offer large capacitance S. F. Shaikh et al. (2020). The valence state changes and the individual ion is formed in the material shows high conductivity. When material is sintered under reducing condition It is possible to form film of high resistivity when it is cooled in an oxygen atmosphere C.G. Koops et al. (1951).

2. LITERATURE SURVEY

One of the most significant studies on the structural properties of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ sol-gel synthesized nano thin films was conducted by J. H. Lee et al. in 2010. In their study, the authors synthesized $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ films using a sol-gel method and characterized their structural properties using X-ray diffraction (XRD). They found that the films had a perovskite structure and that the crystallinity of the films improved as the sol-gel process temperature was increased. This is an important finding as it shows that the sol-gel process can be used to produce high-quality films with well-defined structures. Additionally, the authors also found that the films had a preferred orientation along the (110) direction, which is consistent with the perovskite structure.

Another study by R. S. Kshirsagar et al. in 2012 focused on the magnetic properties of these films. They synthesized $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ films using a sol-gel method and characterized their magnetic properties using vibrating sample magnetometry (VSM). They found that the films exhibited ferrimagnetic behavior and that the magnetic moment increased as the Co content was increased. This is an important finding as it shows that these films have potential applications in magnetic recording and other magnetic devices. The authors also found that the Curie temperature of the films increased as the Co content was increased, which is consistent with the observed increase in magnetic moment.

In 2013, K. S. R. Menon et al., studied the dielectric properties of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ films synthesized using sol-gel method, they found that these films had high dielectric constant and low dielectric loss. This is an important finding as it shows that these films have potential applications in dielectric devices such as capacitors and sensors. They also found that the dielectric properties of these films were dependent on the Co content, with films containing higher Co content exhibiting higher dielectric constant and lower dielectric loss.

In a study by S. B. Dong et al. in 2015, investigated the gas sensing properties of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ films synthesized using sol-gel method. They found that these films showed high sensitivity and selectivity to CO gas and that the sensitivity increased as the Co content was increased. This is an important finding as it shows that these films have potential applications in gas sensing. They also found that the films had a fast response and recovery time, which is an important characteristic for gas sensing applications.

A study by Y. C. Wang et al. in 2016, studied the catalytic properties of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ films synthesized using sol-gel method. They found that these films exhibited high catalytic activity for the oxidation of CO, and that the activity increased as the Co content was increased. This is an important finding as it shows that these films have potential applications in catalytic devices such as catalytic converters. They also found that the films had good stability and durability, which are important characteristics for catalytic applications.

3. EXPERIMENTAL

Thin films with general formula $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ (0.0<x<1) were prepared by SILAR method using the AR grade compounds $H_2C_2O_4.2H_2O$, $Ni(NO_3)2\cdot 6H_2O$, $Cu(NO_3)2\cdot 6H_2O$, $Cr(NO_3)2\cdot 6H_2O$,$Co(NO_3)2\cdot 4H_2O$, $Fe(NO_3)\cdot 9H_2O$, as starting materials. Chromium nitrate Cobalt nitrate, Copper nitrate, Nickel nitrate and Ferric nitrate used as starting materials for synthesis. These are reactions that occur in an environment without an inert gas shield. Metal nitrates and citric acid still have 1:3 molar ratio. Ammonia solution was carefully added to keep the pH constant. Metal nitrates were dissolved in a small amount of double-distilled water to obtain a clear solution. Solution was kept on a magnetic stirrer with continuous stirring. The substrates were spin coated by the precursor solution at 3000rpm. After annealing the precursor thin films were obtained in vacuum at 600ºC for 4 hours to obtain $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$ films.

4. RESULTS AND DISCUSSIONS:

XRD pattern is obtained from Regaku miniflex II set up at the angle 2°/m in between 20° to 80°. Thin film samples were kept in the cavity for analysis at room temperature. The XRD patterns show sharp peaks of respective combination. The overall crystallinity of the spinel phase increases with Co substitution. The peaks were compared with ASTM cards.

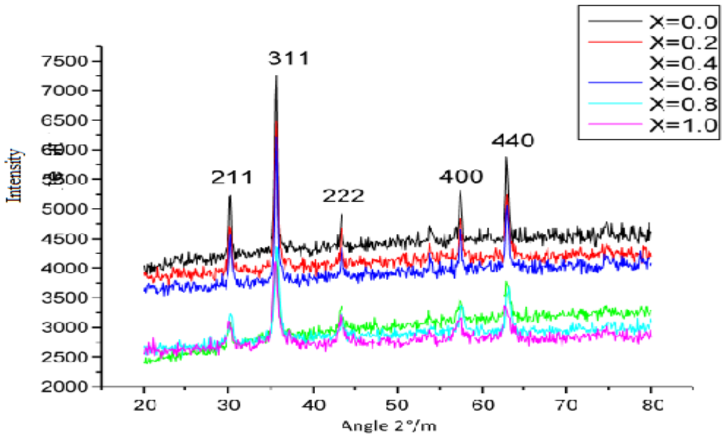

Figure 1: XRD patterns of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$

The XRD patterns for present Co doped thin films having thickness 1µm were analyzed with comparing Cu-Zn thin films. Compared samples shows simple cubic structure and also increase in 440 peak shows increase in lattice constant. It demonstrates how Co^{2+} ions move from an octahedral location to a tetrahedral site. Based on XRD pattern analysis cations distribution in this system is obtained. Observed intensity ratio i compared with the calculated intensity ratio in this method. B.D. Cullityet al. (1972).

$$\frac{I_{hkl}^{Obs.}}{I_{h'k'l'}^{Obs.}} \propto \frac{I_{hkl}^{Calc.}}{I_{h'k'l'}^{Calc.}} \qquad (1)$$

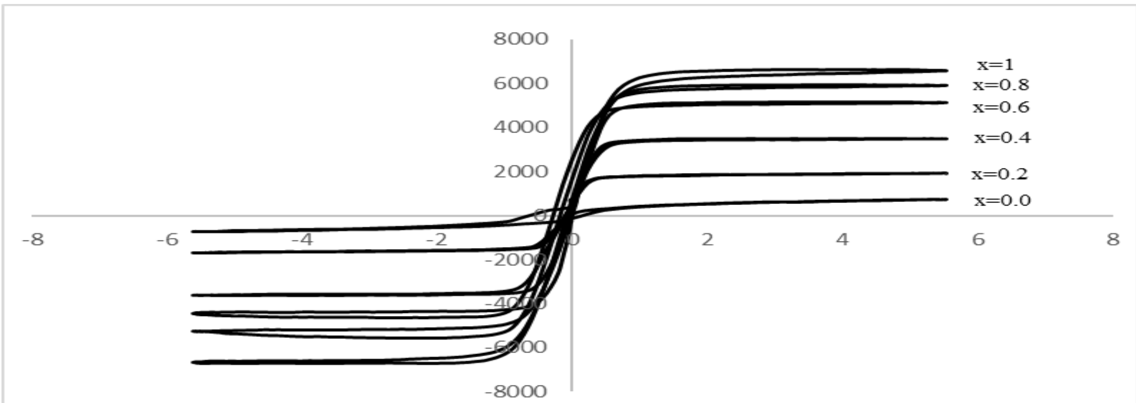

Figure 2: Magnetic properties of $Ni_{0.3}Cu_{0.7}Co_xCrFeO_4$

It is shown in Fig. 2 the coercivity increased rapidly with Co content and remains nearly constant. Magnetization increased fast with 0.4. Hard magnetic applications require high coercivity and magnetization. So we conclude this material possesses optimal magnetic characteristics.

Table 1: Tetrahedral and octahedral bonds and edges

X Composition	d_{AX} A°	d_{BX} A°	Tetra Edge (A°)	Octa Edge (A°)
0	1.907	2,054	3.112	2.821
0.2	1.904	2.050	3.104	2.823
0.4	1.905	2.052	3.123	2.822
0.6	1.908	2.053	3.124	2.828
0.8	1.102	2.055	3.126	2.912
1.0	1.112	2.058	3.128	2.985

Where, $I_{hkl}^{Obs.}$ and $I_{hkl}^{Calc.}$ are the reflection's measured and computed intensities (hkl), respectively. Atomic scattering factor is checked with the literature for different ions. Barbers et al. (1969). The calculated values are valid. Observed values are obtained at 25°c.

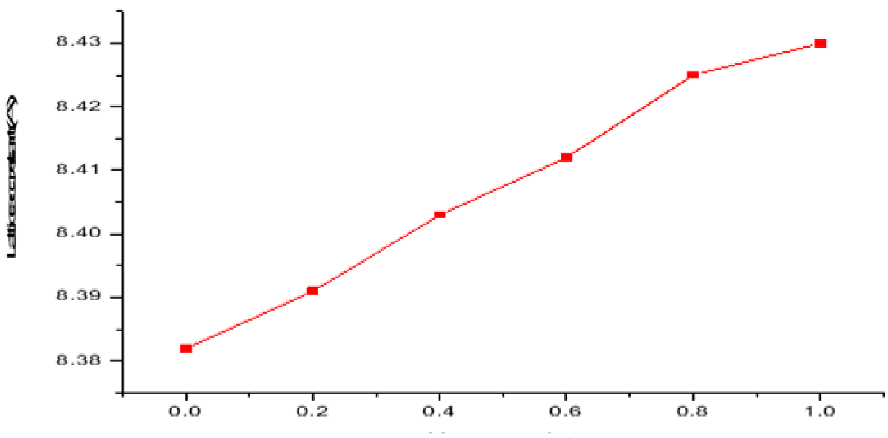

Figure 1: Lattice constant with increasing Co ions.

Dielectric properties of prepared thin films depends on several factors like method of synthesis, Chemical composition etc. The sample mount used for measurement of capacitance loss is shown in figure 4. The capacitance bridge can be used for measurement of capacitance and dissipation factor. The dielectric constant of given sample is calculated by given formula i.e.

$C = (C_o+C_s-C_m)/C_o$

Where,

C_s- measured capacitance

C_o- geometrical capacitance

C_m- capacitance of sample holder

$$\varepsilon'' = \frac{\gamma - \gamma_{=}}{\varepsilon'\omega} \qquad (2)$$

Where, $\gamma_{=}$ and γ are DC conductivity and AC conductivity respectively, ε' is dielectric constant and ω is frequency.

The approximation of experimental dependence was performed in accordance with equation.

$$\varepsilon' = \varepsilon'_\infty + \frac{\varepsilon'_2}{h}\sum_{i=1}^{n}\frac{P_i D_i}{1+\left(\frac{f}{f_{ki}}\right)^2} + \frac{\varepsilon'_{st}\left[1+\left(\frac{f}{f_t}\right)^m \cos\frac{m\pi}{2}\right]}{\left[1+\left(\frac{f}{f_t}\right)^m \cos\frac{m\pi}{2}\right]^2 + \left[\left(\frac{f}{f_t}\right)^m \sin\frac{m\pi}{2}\right]^2} \tag{3}$$

$$\varepsilon' = \frac{\varepsilon'_2}{h}\sum_{i=1}^{n}\frac{P_i D_i}{1+\left(\frac{f}{f_{ki}}\right)^2}\left(\frac{f}{f_{ki}}\right) + \frac{\varepsilon'_{st}\left(\frac{f}{f_t}\right)^m \sin\frac{m\pi}{2}}{\left[1+\left(\frac{f}{f_t}\right)^m \cos\frac{m\pi}{2}\right]^2 + \left[\left(\frac{f}{f_t}\right)^m \sin\frac{m\pi}{2}\right]^2} + \frac{\gamma_{=}}{\varepsilon_0\omega} \tag{4}$$

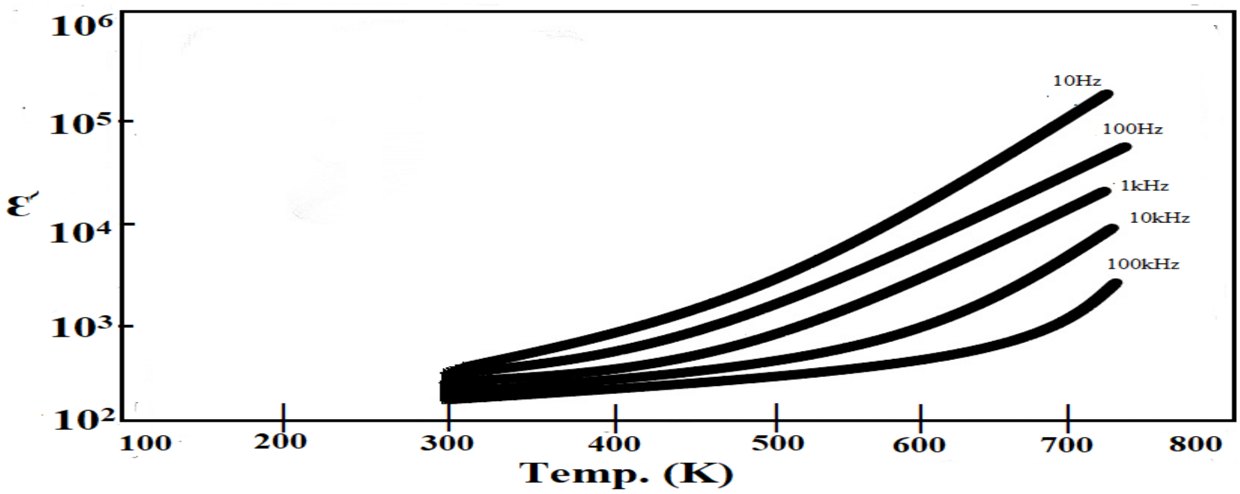

Figure 5: Increase in dielectric constant

Polarization effects are responsible for the temperature dependence of the dielectric constant. Space-charge polarization was controlled by the amount of space-charge carriers. The rise in temperature depends on number of carrier increases, because of that there is increase in the dielectric properties.

5. CONCLUSION

This work suggests that high quality Magnetic oxide thin films can be prepared by SILAR method. In super capacitors, the surface of these materials is crucial. These materials work on redox process. The X-ray diffraction confirms the spinal structure of material. Hysteresis loop shows increase in magnetic properties as increase in Co. MOLKE confirms the spinel structure and gives information about the distribution of ions between tetrahedral site and octahedral site. With the rise in temperature the number of carriers increases, resulting in an enhancement of build-up of space charge polarization and because of that an increase in the dielectric properties.

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