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Effect of annealing temperature on Magnetic Properties of Nanocrystalline $CuFe_{1.9}La_{0.1}O_4$

Swaati Saandhya[1], ManishiPuja[1] and Dolly Sinha[1]
[1]Department of Physics, Magadh Mahila College, Patna 800001, India.
E-mail: dsinha96@gmail.com

ABSTRACT

Nanocrystalline $CuFe_{1.9}La_{0.1}O_4$ powder was prepared by the citrate precursor method. The magnetic properties of the sample were measured by using a vibration sample magnetometer (VSM), which showed that the sample exhibited typical ferrimagnetic behaviour at room temperature. The lattice constants decrease with the increase in annealing temperature. The crystallite size decreases with the increase in the annealing temperature. The value of M_s is found to be maximum for the sample annealed at 550 °C and other two samples annealed at 450 and 650 °C having the similar less magnetization value because of tetragonal crystal symmetry.

Keywords: XRD, VSM, Nanocrystalline, Size Effect.

INTRODUCTION

Nanocrystalline ferrites with general formula $AB_2O_4$are very important magnetic materials because of their interesting magnetic and electrical properties with chemical and thermal stabilities [1]. Copper ferrite ($CuFe_2O_4$) is one of the most important ferrites because of the prominent magnetic and semiconducting properties. These are widely used in the electronics industry [2]. $CuFe_2O_4$ is known to exist in tetragonal and cubic structures. The tetragonal structure is stable at room temperature and transforms to cubic phase only at a temperature of 623 K and above due to Jahn–Teller distortion. The distortion is directly related to the magnetic properties. The cubic structure possesses a larger magnetic moment than that of the tetragonal one, because there are more cupric ions (Cu^{2+}) at tetrahedral sites in cubic structure as compared to that in the case of tetragonal structure [3]. However, its structure can be controlled by annealing condition during sample preparation. Various methods such as conventional ceramic method [2, 4], nitrate precursor techniques [5], combustion synthesis [6], and oxalate precursor method [7] have been developed to synthesize it. Recently, nanocrystalline magnetic materials have received more and more attention due to their novel material properties that are significantly different from those of their bulk counterparts [8]. The magnetic materials are of great interest due to their large surface-to-volume ratio, and this expands their technological application in many areas including nanocomposites, nanocatalyts, nanosensors, nano-electronics and photonics.

Experimental

Nanocrystalline $CuFe_{1.9}La_{0.1}O_4$were prepared by the standard citrate precursor method (Peddis et al. 2011). Stoichiometric ratio of copper nitrate ($Cu(NO_3)_2.6H_2O$), iron nitrate ($Fe(NO_3)_3.9H_2O$) and Lanthanum nitrate ($La(NO_3)_3.9H_2O$) were dissolved in deionized water to produce a clear solution with Cu:Fe/La molar ratio of 1:2. An aqueous solution of citric acid ($C_6H_8O_7.H_2O$) was mixed with metal nitrate solutions. Generally, the molar ratio of citric acid to metal nitrates is maintained enough high so that a sufficient amount of citric acid (acting as fuel) to be present during the nucleation and to expedite the reaction. Also, citric acid acts as a ligand to stabilize metal ions. In the synthesis of present samples, the molar ratio of metal nitrate solution to citric acid has been standardized to ratio 1:3. The resulting materials were heat treated in air atmosphere at 150 °C. The samples were further annealed in air atmosphere at 450, 550 and 650°C, respectively, for 2 hours, to obtain different particle sizes. The phase purity and the crystalline nature of the samples were examined by powder X-ray diffractometry (TTRX-III, Rigaku). Magnetic hysteresis loops were measured at room temperature by using a LakeShore (Model No. 7410) Vibrating Sample Magnetometer (VSM) over a field range of ±1.5 T.

Result and Discussions

Fig. 1 shows the X-ray diffraction pattern of all the samples. The XRD patterns show a broad and well resolved diffraction peaks for the annealed samples, suggesting the formation of $CuFe_{1.9}La_{0.1}O_4$ crystals with nanometer crystallite size. Also, one could observe closely from the above XRD patterns that the diffraction peaks become sharper with increasing the annealing temperature. The lattice parameters decrease with the increase in annealing temperature. The diffraction peaks shift to a higher 2θ position due to the shrinkage of unit cell dimension and the lattice constant decreases.

The analysis of the crystallite size has been carried out using the broadening of the XRD peaks. Peak broadening comes from several sources such as instrumental effect, finite crystallite size and strain effect within the crystallite lattice [33]. Crystallite size has been calculated by using Scherrer's formulae [34] is defined as,

$$D = \frac{k\lambda}{\beta Cos\theta} \quad \text{----------------------------------} \quad (1)$$

Where constant k depends upon the shape of the crystallite size (=0.89, assuming the circular grain), β = Full width at Half Maximum (FWHM) of Intensity (a.u.) vs. 2θ profile, λ is the wavelength of the $Cu\ K_\alpha$ radiation (=0.1542nm), θ is the Bragg's diffraction angle and D is the crystallite size. In Scherrer's formula FWHM has been calculated using Gaussian fit to the peaks in XRD pattern. The crystallite size decreases with the increase in the annealing temperature.

The room temperature hysteresis loops are shown in Fig.3 for the samples $CuFe_{1.9}La_{0.1}O_4$annealed at 450, 550and 650 °C. The Ferrimagnetic behaviour is evidenced for all samples. In addition, the coercive field is lower for the samples annealed at 650°C than for samples annealed at 450 and 550 °C due to larger crystallite size.

The highest magnetizations (as measured in the highest field), remnant magnetization and coercive field for all $CuFe_{1.9}La_{0.1}O_4$samples annealed at 450, 550 and 650 °C are mentioned in table 1. The value of M_s is found to be maximum for the sample annealed at 550 °C which is attributed due to the cation distribution and dominant cubic crystal symmetry of the sample. The other two samples are having the similar less magnetization value even if they have different crystallite size because of tetragonal crystal symmetry.

The observed decrease in highest magnetization could be explained using Neel's two sublattice model. According to Neel's two sublattice model of collinear ferrimagnetism, the magnetic moment per formula is expressed as $M=M_B-M_A$ where M_B and M_A are the magnetic moment at the

B-site and A-site respectively. The unexpected variation of the magnetization with a maximum value for annealing temperature of 550°C proves clearly a dependence of the cation distribution of present samples which is a function of the annealing temperature.

Conclusion

Nanocrystalline $CuFe_{1.9}La_{0.1}O_4$ powder was successfully prepared by the citrate precursor method and phase purity has been checked by the X-Ray diffractometer. The magnetic properties of the sample were measured by using a vibration sample magnetometer (VSM), which showed that the sample exhibited typical ferrimagnetic behaviour at room temperature. The lattice constants decrease with the increase in annealing temperature. The crystallite size decreases with the increase in the annealing temperature. The value of M_s is found to be maximum for the sample annealed at 550 °C which is attributed due to the cation distribution and dominant cubic crystal symmetry of the sample. The other two samples are having the similar less magnetization value even if they have different crystallite size because of tetragonal crystal symmetry.

Acknowledgement

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Table 1: Magnetic parameters of $CuFe_{1.9}La_{0.1}O_4$ ceramics annealed at three different temperatures. Where, M_S = magnetization at maximum applied field, H_C = Coercive field, emu/g = emu/gram at applied magnetic field of 12kOe.

Annealing Temperature	M_S (emu/g)	H_C(kOe)	M_r(emu/g)	Crystallite Size (nm)
450 °C	26.51	0.87	11.31	20.9
550 °C	118.13	0.99	56.44	11.5
650 °C	26.01	0.58	11.16	11.3

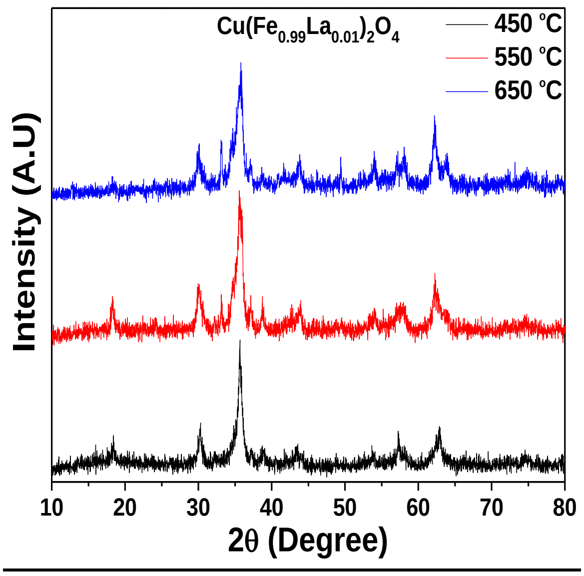

Fig. 1 XRD patterns of the sample $CuFe_{1.9}La_{0.1}O_4$.

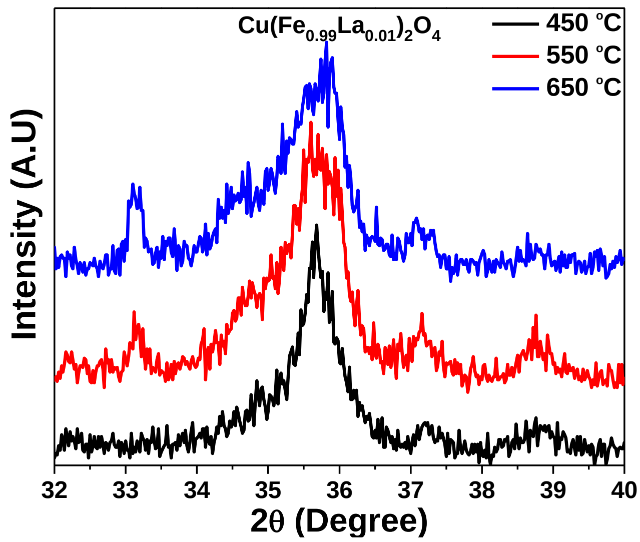

Fig. 2 Shift in XRD patterns of the sample $CuFe_{1.9}La_{0.1}O_4$.

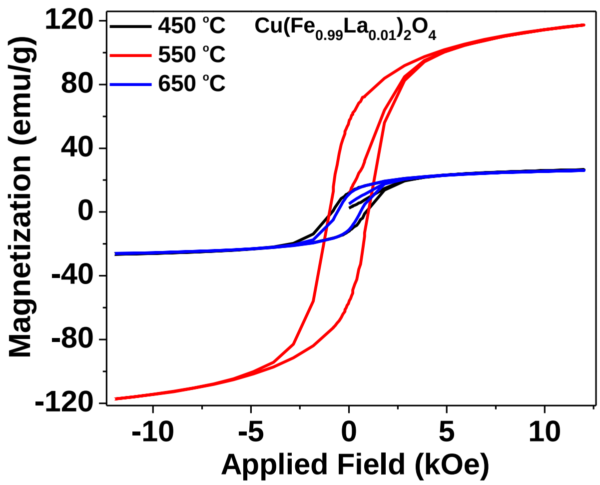

Fig. 3 Magnetization versus applied field plot for $CuFe_{1.9}La_{0.1}O_4$.

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