

# **Study of the Structural and Magnetic Properties of CuFe<sub>2</sub>O<sub>4</sub> Nano Particle Prepared by Chemical Co-precipitation Method**

**Sakia Shabnam Kader<sup>1</sup>, Shaikh Manjura Hoque<sup>2</sup>, Deba Prasad Paul<sup>3</sup>  
A. K. M Fazlul Hoque<sup>1</sup> and Faizus Salehin<sup>4</sup>**

<sup>1</sup> Daffodil International University, <sup>2</sup> Atomic energy centre, Dhaka, <sup>3</sup> University of Chittagong  
<sup>4</sup> University of Dhaka.

## **Abstract**

The properties of the CuFe<sub>2</sub>O<sub>4</sub> nano particle prepared by chemical co-precipitation have been studied. The XRD result of the as dried and samples calcined at different sintering temperature has shown that the single phase has been observed in the as dried condition and there is no observation of extra peak. The grain size has been obtained from Scherrer's formula and found as 4nm to 56nm for the CuFe<sub>2</sub>O<sub>4</sub> nano particle sample with the systematic variation of sintering temperature. The variation of lattice parameter with sintering temperature has been presented. The general increase in lattice parameter throughout the entire range of sintering temperature is due to the deviation in cation distribution in the spinel structure of CuFe<sub>2</sub>O<sub>4</sub>. Nano particle has been characterized to understand the magnetic properties from the observation of hysteresis loops. The initial permeability varies with sintering temperature due to the variation in kinetics of diffusion which depends on the particle size of the starting material. The Curie temperature has been measured from the result of temperature dependent initial permeability.

## **1. INTRODUCTION**

Magnetic nanoparticles show remarkable new phenomena such as super paramagnetism, high field irreversibility, high saturation field, extra anisotropy contributions. These phenomena arise from finite size and surface effects that dominate the magnetic behaviour of individual nanoparticles[1]. Ferrite nanoparticles are most explored nano particle which can be manipulated using magnetic field. The outstanding property of ferrites which make them suitable for many applications is their high electrical resistivity compared to those metals.[2] Copper ferrite (CuFe<sub>2</sub>O<sub>4</sub>) is basically an inverse spinel ferrite in which the tetrahedral (A) sites are occupied by ferric ions, and octahedral (B) sites by ferric and copper ions. The magnetic properties and the cation distribution are found to be different in nanocrystalline spinel ferrites, when compared to the bulk counterpart [3]. The saturation magnetization for the nanocrystalline ferrites in general is found to be lower compared to their bulk value, which is attributed to surface spin effects. In some cases an enhancement in the saturation magnetization is observed due to the change in cation distributions which depends on the crystal field stabilization energy of the cations. Apart from grain size, the cation distribution, which depends on the synthesis condition, is found to play a major role in the observe changes in their magnetic properties [4]. Nanotechnology was also expanded extensively to other fields of interest due to the novel properties of nanomaterials discovered and to be discovered. For example, nanowires can be potentially used in nanophotonics, laser, nanoelectronics, solar cells, resonators and high sensitivity sensors. Nanoparticles can be potentially used in catalysts, functional coatings, nanoelectronics, energy storage, drug delivery and biomedicines. [5-7]

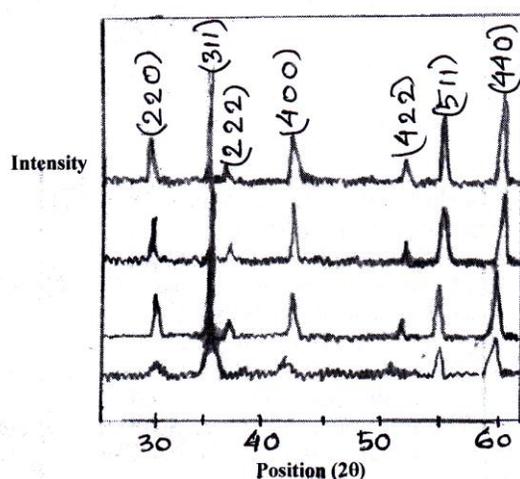
## **2. EXPERIMENTAL DETAILS**

The fine particles of CuFe<sub>2</sub>O<sub>4</sub> have produced by a standard co-precipitation technique. The starting materials were Cu (NO<sub>3</sub>)<sub>2</sub>.3H<sub>2</sub>O [copper nitrate try hydrate], Fe (NO<sub>3</sub>)<sub>2</sub>.9H<sub>2</sub>O [Iron nitrate nano hydrate] and sodium hydroxide (NaOH). Two separate solutions ,one a solution of copper nitrate in deionized water and the other contains a solution of Iron nitrate in deionized water were prepared. The former solution was added drop wise into the later. Then the two solutions

were mixed the solution of sodium hydroxide. Then the mixed solution was stirred by magnetic stirring apparatus (300 rpm) at temperature 200°C. When the reaction was complete, the resultant deep blue solution was filtered and washed with deionized water for 10 times and was dried at the temperature above 100°C for 36 hours, then calcined at various temperatures. Following this process, six batch samples were prepared by varying different parameters such as stirring temperature, pH and drying temperature. The X-Ray diffraction (XRD) of CuFe<sub>2</sub>O<sub>4</sub> nanoparticles has shown that the grain size is 4nm to about 56nm according to the systematic variation of the calcinations temperature

### 3. RESULTS AND DISCUSSION

X-ray diffraction pattern of CuFe<sub>2</sub>O<sub>4</sub> samples calcined at different temperature have been presented in **Fig 1**. In the as-cast condition, single phase CuFe<sub>2</sub>O<sub>4</sub> has formed with no extra peak. At the calcinations temperature of 600°C, slight change in the XRD pattern has found maintaining the same structure. At 800°C fully crystallized CuFe<sub>2</sub>O<sub>4</sub> has formed with all peaks matches with the standard patterns. The peak broadening shows that, the CuFe<sub>2</sub>O<sub>4</sub> nano particle has formed. The peak width decreases with the increase in calcinations temperature which reflects the coarsening of particles. The size of the nano particles have been determined by using Scherrer's formula from the FWHM of 311 peak. The grain size has obtained from 4nm to 56 nm for the CuFe<sub>2</sub>O<sub>4</sub> nano particle sample with the systematic variation of sintering temperature which is shown in **Fig 2**. The grain size increases very rapidly from 200°C-800°C with increase in the sintering temperature but from 800°C-900°C, the grain size suddenly drops and then for the sintering temperature 900°-1200°C, the size again increases very sharply.



**Fig 1: X-Ray diffraction pattern**

In order to find the experimental lattice parameter, Nelson-Riley function was used. The graph of apparent lattice parameter vs N-R function  $F(\theta)$  gives the value of the exact lattice parameter. The variation of lattice parameter with sintering temperature has been presented in **Fig 3**, which increases non linearly with sintering temperature. The increase in lattice parameter is due to the deviation in cation distribution from the spinel structure of CuFe<sub>2</sub>O<sub>4</sub>.

By using vibrating sample magnetometer, the CuFe<sub>2</sub>O<sub>4</sub> nano particle has been characterized to understand the magnetic properties. In **Fig 4**, the hysteresis loop for the sintering temperatures 900°C, 1000°C, 1100° and 1200°C have been presented. The hysteresis loop contains the soft ferrimagnetic nature of synthesized CuFe<sub>2</sub>O<sub>4</sub>. The difference in the value of the saturation magnetization can be explained in the light of cation distribution. Any change in the concentration and nature of the ions in A-site and B-sites should cause resultant magnetization to be different. The decrease in  $M_s$  value implies that outside a core of ordered moments, those

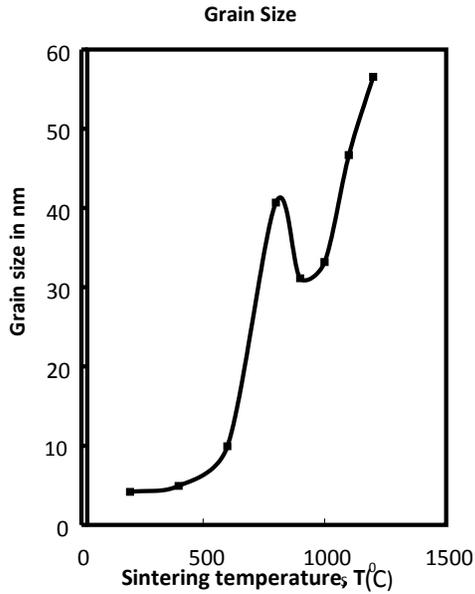


Fig 2: Variation of Grain Size with Sintering Temperature  $T_s$

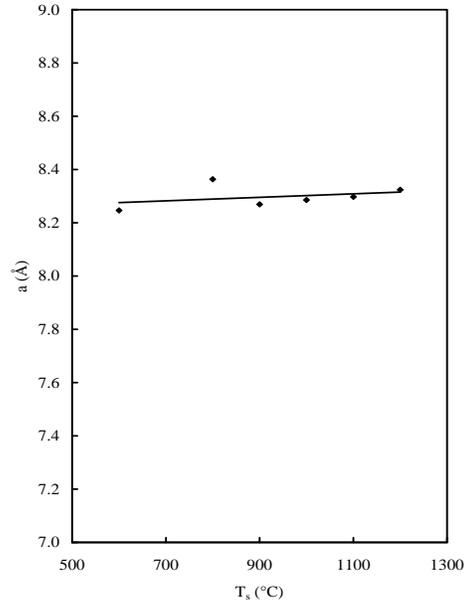


Fig 3: Variation of Lattice parameter with sintering temperature

in the surface layer are in a state of a frozen disorder. With the reduction of particle size, more atoms retains on the surface which are not exchange coupled. As a result, the value of magnetization decreases. The effect is more prominent when the particle sizes are very small. On the other hand, the saturation magnetization increases with the increase of particle size since core of the ordered moment's predominant.

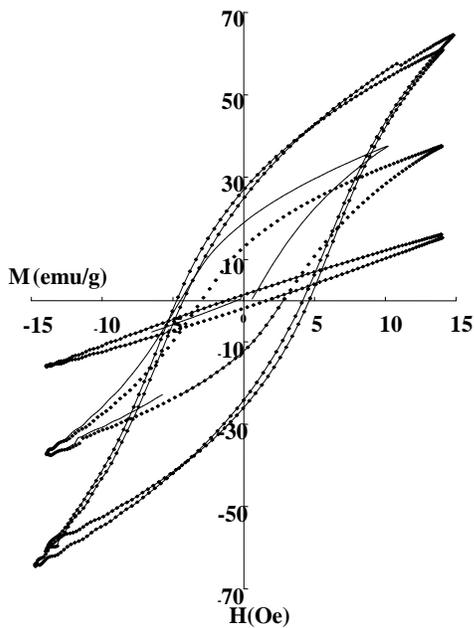


Fig 4: Hysteresis loop for  $\text{CuFe}_2\text{O}_4$  at sintering temperatures  $900^\circ\text{C}$ ,  $1000^\circ\text{C}$ ,  $1100^\circ\text{C}$ ,  $1200^\circ\text{C}$

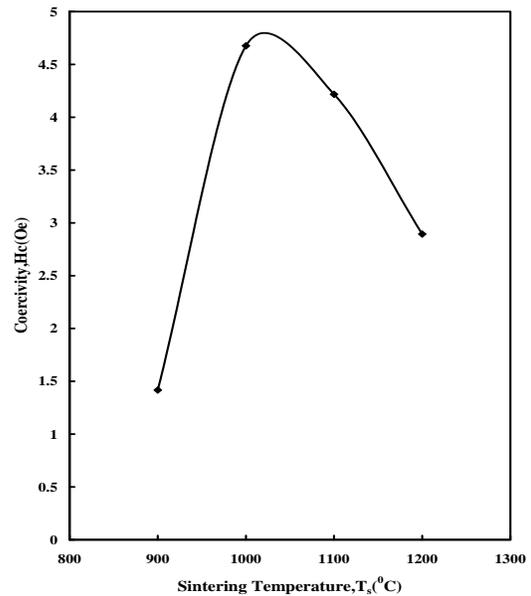


Fig 5: Variation of Coercivity ( $H_c$ ) with Sintering Temperature ( $T_s$ )

**Fig 5** shows the sintering temperature dependence coercivity in which the coercivity rises from 900°C-1000°C. But from 1000°C to 1200°C it drops rapidly. The smaller coercive field is due to a changeover from multi domain nature to single domain nature. The higher value of  $H_c$  is attributed due to the demagnetization caused by domain rotation.

**Fig 6** represents the real part of initial permeability as a function of frequency for the  $\text{CuFe}_2\text{O}_4$  nano particle sample at various sintering temperature (800°C, 900°C, 1000 °C, 1100 °C, 1200°). From the fig, it is observed that,  $\mu'$  is fairly constant up to a certain high frequency and drops rapidly to a very low value at higher frequency. The real part of  $\mu'$  describes the stored energy expressing the component of magnetic induction in phase with the alternating magnetic field. The Permeability is directly proportional to the micro structural features. i.e, grain size. The multi domain grains are obtained by increasing the grain size, induces in higher permeability due to the domain wall contribution. The Fig also shows that, due to the act of interfering of small grain size with wall motion, the permeability decreases with increasing the stability region of  $\mu'$ . That is, the stability is maximum at the value of lower permeability.

**Fig 7** represents the imaginary part of the initial permeability ( $\mu''$ ) over the frequency range from 1 kHz to 10 kHz for the  $\text{CuFe}_2\text{O}_4$  sample. The fig shows the curve with a large tail towards the high frequency at which the loss components are lowered. It occurs due to the inhibition of the domain wall motion and the variation of magnetization with the alternating magnetic field. As a result, the value of  $\mu''$  decreases rapidly at the low frequency region and becomes almost constant.

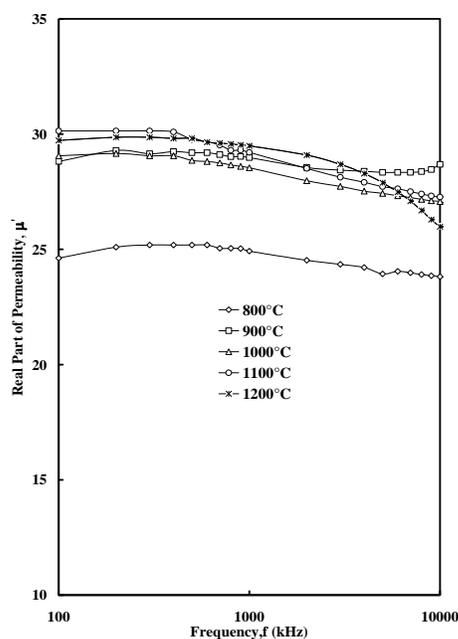


Fig 6 : Frequency(f) dependence of real part of the initial permeability ( $\mu'$ )

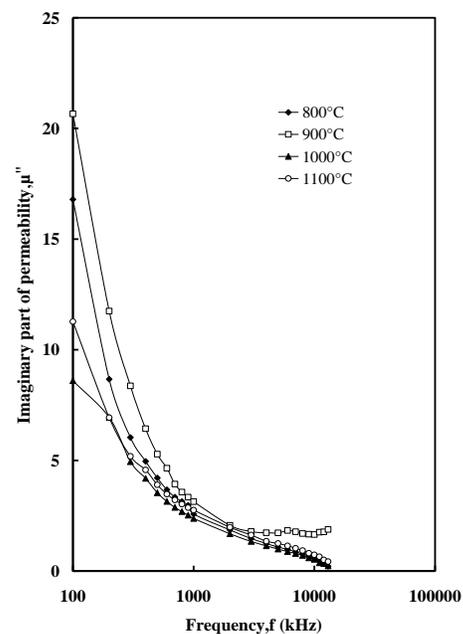


Fig 7 : Frequency (f) dependence of the imaginary part of the initial permeability ( $\mu''$ )

In **Fig 8**, the frequency dependence of the real part of the initial permeability has been presented for the  $\text{CuFe}_2\text{O}_4$  nano particle sample for different sintering temperatures. The magnitude of the initial permeability is proportional to the square of the saturation magnetization and inversely proportional to the magnetic anisotropy energy. According to the Hopkinson effect, permeability increases with temperature and shows a maximum just below the Curie point. Near the Curie point, the value of the anisotropy energy almost negligible and the complete disorder take place. At  $T_c$ , the sharp falling of  $\mu$ . **Fig 9** shows the temperature dependence Curie temperature. In order to find the result,  $d\mu'/dT$  vs.  $T$  has been plotted for different sintering temperatures. The point at which the highest slope change has observed is known as the Curie point and from this point, at low sintering temperature the grain size are very

small and the magnetic coupling will be weaker because of the lower coordination of the particles. The magnetic coupling will be stronger with the increase in the sintering temperature. This phenomenon has been reflected in the increment of the Curie temperature. Further increase in the sintering temperature, Curie temperature decreases which have occurred due to the predominance of the effect of the change in cation distribution over the finite size effect on Curie temperature.

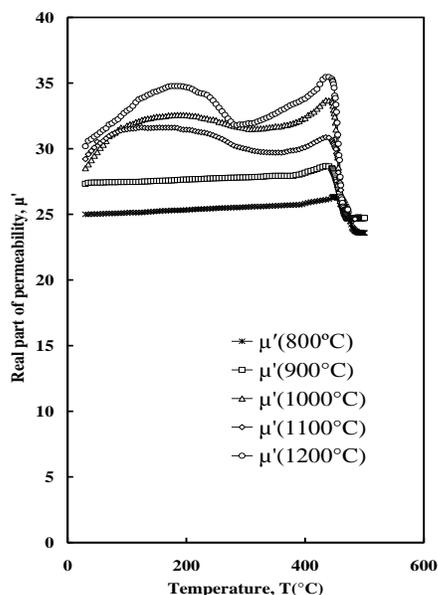


Fig 8: Temperature dependence of the real part initial permeability ( $\mu'$ )

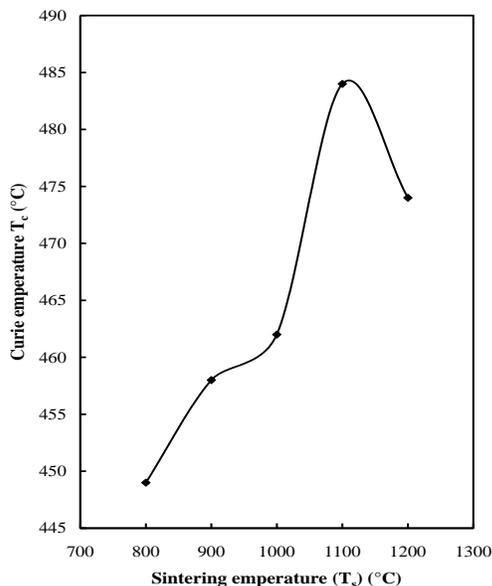


Fig 9 : Sintering temperature ( $T_s$ ) dependence Curie temperature ( $T_c$ )

## CONCLUSION

The nano particle samples which are sintered at different temperature bring the variation in the magnetic properties of the sample. According to the hysteresis loop, the difference in the value of the saturation magnetization can be explained in the light of cation distribution. The initial permeability varies with sintering temperature due to the variation in kinetics of diffusion which depends on the particle size of the starting material. Curie temperature decreases which have occurred due to the predominance of the effect of the change in cation distribution over the finite size effect on Curie temperature. The result indicates that, the properties of materials with nanometer dimensions are significantly different from those of atoms and bulks materials.

## REFERENCES

- [1] Batlle X and Labarta A 2002 *J. Phys. D: Apply. Phys.* **35** R15
- [2] Charles Kittel, Introduction to Solid State Physics, 7<sup>th</sup> edition, John Wiley and Sons, NY. 417-462.
- [3] B. D. Cullity, C. D. Graham, *Introduction to Magnetic Materials* Addison –Wesley Publishing Com181 (1972)
- [4] C. Doroftei, N Rezlescu, P.D. Popa, F. *Microstructure and humidity properties of MgFe<sub>2</sub>O<sub>4</sub> Ferrite with Sn and Mo substitutions prepared by selfcombustion method.* J.Optoelectron. Adv. Mater. 8(3),1012-1015(2006)
- [5] Andrievski R A 2003 *Journal of Nanoparticle Research* **5** 415
- [6] Cavalcanti A, Shirinzadeh B, Freitas RA Jr, Hogg T. "Nanorobot architecture for medical target identification". *Nanotechnology* 19, 1 (2008).
- [7] L Zhang, FX Gu, JM Chan, AZ Wang, RS Langer and OC Farokhzad, "Nanoparticles in Medicine: Therapeutic Applications and Developments". *Clinical Pharmacology & Therapeutics* 83, 761-769 (2008).