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Quenching Effect on Structural and Magnetic Properties of Copper Ferrite Prepared by Ceramic Technique

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A B S T R A C T

Copper ferrite particles have been synthesized by the standard ceramic technique. The prepared copper ferrite particles were sintered at 800^{o}C for 12h. The pre-sintered sample is again reground in fine powder and palletized using hydraulic press. These pallets were finally sintered at 1050ºC for 12h and then suddenly quenched to 77 K. The effect of quenching on their structure and magnetic properties of copper ferrite were investigated and reported in this work. The X-ray diffraction analysis revealed that synthesized copper ferrite particles possess single phase cubic spinel structure. A keen observation of the X-ray diffraction pattern showed the shifting of Bragg's angle after quenching. The average crystallite size of the copper ferrite obtained from Sherrer's formula is in few micro-meters range which decreases after quenching. The lattice constant and other structural parameters are influenced by quenching effect. The magnetic properties were investigated before and after quenching with the help of pulse field hysteresis loop technique at room temperature. The saturation magnetization M_S and magneton number (n_B) obtained from M-H plots is found to increases after quenching whereas remanence ratio (M_r/M_s) and coercivity decreases after quenching.

Keywords : *Copper Ferrite; Ceramic Technique; X-ray diffraction; Pulse Field Hysteresis loop technique.*

1. Introduction:

Ferrites are a group of technologically important magnetic materials of current interest. They have been widely studied from last five to six decades because of their unique combined electrical and magnetic properties. They are used in magnetic ink and magnetic fluids and for the fabrication of magnetic core of read and write heads of high-speed digital tapes or for disc recording [1-4]. The important structural, electrical and magnetic properties of ferrites arise from the distribution of cation over the available sites. The method of preparation, preparative parameters and preparative condition also plays an important role in governing the properties of ferrites [5-7]. Ferrites are the ferromagnetic oxides as proposed by L. Neel [8]. It is composed of iron oxide and metal oxide as their main components. They possess three kinds of crystal structures and on the basis of their crystal structure ferrites are classified into three groups namely (1) spinel ferrite (2) garnet and (3) hexa ferrites.

Among the three types of ferrite, spinel ferrites are the important class of magnetic materials and are widely used in many technological aspects. They have been widely studied by many researchers for their basic electrical and magnetic properties. The crystal structure of the spinel ferrite belongs to cubic spinel with space group Fd3m. The crystal structure possesses two sub lattices namely tetrahedral (A) and octahedral [B] in which cation of different ionic radii can accommodate [9]. The variation in the physical properties of spinel ferrite can be brought by either change in method of preparation or selecting appropriate cation. The substitution of divalent, trivalent and tetravalent ions in spinel ferrites has been successfully carried out and variations in the properties are studied. The ceramic method was used for the synthesis of spinel ferrite on large scale. Various physical and chemical methods have been employed to obtain -crystalline spinel ferrite. Magnetic particles are of great interest because of their unique magnetic properties dominated by single domain magnetism and super-paramagnetism [10]. However, ceramic technology has its own advantage over the other methods. Due to high sintering temperature the density of the sample increases which is useful in some application. The stoichiometric and homogeneity of the samples is maintained in the ceramic method. Many efforts have been made to improve the basic properties of ferrite by substituting or adding various ions with different valence states depending on the application of interest. The synthesis conditions (quenching, slow cooling etc.) also important in governing the properties of ferrites and are rarely studied. Quenching from high temperature to low temperature can cause modifications in the properties of ferrites.

1.2 Ceramic method

The most popular and commonly used method of preparation is the solid-state reaction/ceramic method [11, 12]. In ceramic method, very pure and fine grain constituents in oxide forms are taken as raw material for the synthesis. Then they are thoroughly, uniformly mixed and ground for 3 to 4 hours using agate mortar pestle. This mixture is sintered at specific temperature so as to facilitate solid-state chemical reaction among the oxides. Pre-sintering of the samples can be done at about lower temperature (~90^0C) and final sintering of the sample can be done at much higher temperature (>110^0C) depending on the constituents of the compound and then slowly cooled to room temperature. The ceramic method consists of the following steps.

1. Calcinations of the constituent oxides / carbonates to remove the water content.
2. Weighing and thorough mixing of constituents in stoichiometric proportions.
3. Grinding of the mixed powder for 3 to 4 hours continuously using agate mortar and pestal
4. Pre-sintering at the temperature slightly less than the solid-state chemical reaction temperature.
5. Powdering and pressing into desired shape using hydraulic press under suitable pressure.
6. Final sintering at elevated temperature (>1100^0C) and followed by slow cooling.

2. Experimental Procedure

The polycrystalline samples of copper ferrite ($CuFe_2O_4$) prepared by using the standard ceramic method. The analytical grade oxides (CuO and Fe_2O_3) mixed as per stoichiometric proportions. Grinding using Agate mortar was carried out for 3 to 4h. After that the powder was pressed into pellets and pre-sintered at 800^0C for 12h. The pre-sintered sample is again reground in fine powder and pelletized using hydraulic press. These pallets were finally sintered at 1050^0C for 12h. Further, some of the pallets of CuFe2O4 are quenched at liquid nitrogen temperature (LN2) and the same are used for further study. The quenched and unquenched samples of copper ferrite were characterized by X-Ray diffraction method. The XRD patterns were recorded in the 2θ range of 20 - 80˚ using Cu-kα (K=1.5418Å) radiation source.

3. Results and discussion

3.1 XRD analysis

Fig. 1 (a, b) shows the X-ray diffraction (XRD) patterns of unquenched (UQCF) and quenched (QCF) copper ferrite prepared by standard ceramic method. Both the XRD patterns show the reflections (2 2 0), (3 1 1), (2 2 2), (4 0 0), (4 2 2), (5 1 1) and (4 4 0). All these reflections belong to cubic spinel structure. No extra peak other than cubic spinel was observed in the XRD pattern. And the Fig.1 (c) shows the stacking of the graphs of unquenched and quenched XRD pattern. The marked difference in the XRD pattern is in the intensity of the Bragg's reflections. Further, the 2θvalue shows slight variation. The variation in the intensity of quenched copper ferrite has been observed. It is observed that the 2θ values shift towards lower 2θ side after quenching.

The crystallite size of the prepared samples (UQCF and QCF) was obtained by using Scherrer's formula.

$$D = \frac{0.9\lambda}{\beta Cos\theta} \quad (1)$$

(311) peak in the XRD pattern is found to be most intense amongst the other existing peaks and is used to determine the crystallite size of the samples. The crystallite size for UQCF and QCF is found to be 39.58 nm and 38.91 nmrespectively.

The values of crystallite size are presented in Table 1and it is observed that the crystallite size decreases after sudden quenching for ceramic samples.

The lattice constant 'a' for UQCF and QCF samples determined from XRD data using the relation [13].

$$a=d\sqrt{N} \quad (2)$$

Where, 'a' is lattice constant, d is inter planar spacing and N = ($h^2+k^2+l^2$); (h k l) are Miller indices.

The values of lattice constant for UQCF and QCF samples determined using above relation are summarized in Table 1. The lattice constant increases from 8.38Å to 8.44Å after sudden quenching. The increase in lattice constant may be attributed to quenching effect. The unit cell volume (V) was determined from the values of lattice constants (V = a^3) for UQCF and QCF samples. The slight change in the unit cell volume is due to the change in the lattice constant of the UQCF and QCF samples. The values of unit cell volume are given in Table 1

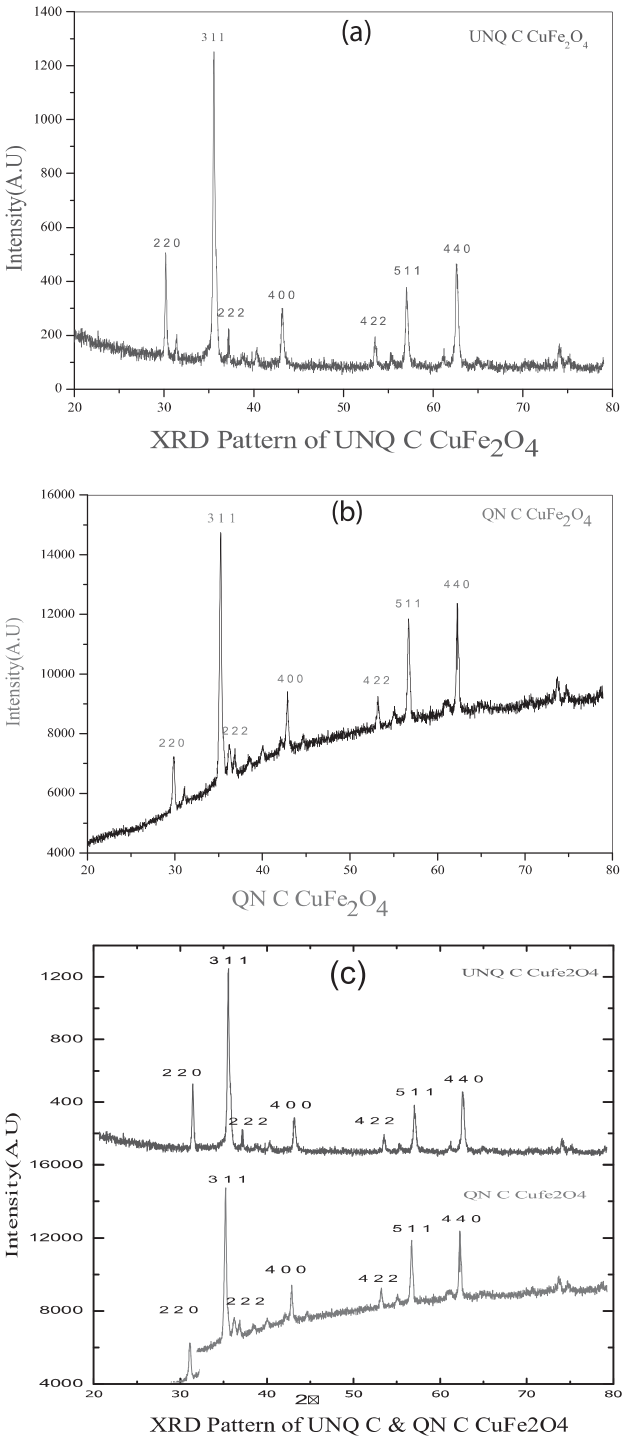

Fig. 1 (a, b, c) the X-ray diffraction (XRD) patterns of unquenched (UQCF) and quenched (QCF) copper ferrite

The X-ray density was calculated using the following relation and their values are given in Table 1

$dx = 8M / Na^3$

It is evident from Table 1 that the X-ray density decreases after quenching. The decrease in X-ray density is attributed to higher value of lattice constant of the quenched sample compared to unquenched sample. The bulk density was measured using Archimedes principle [14] and values are presented in Table 1 No noticeable change in the bulk density was found for UQCF and QCF samples.The percentage porosity (P) was calculated using the values of X-Ray density and bulk density for UQCF and QCF samples. The values of porosity are listed in Table 1 it is also decreases after quenching. The Decrease in porosity is due to quenching effect. Thus, the structural parameters lattice constant, crystallite size and porosity are influenced by quenching effect.

Table 1. Structural Parameters

Sr. No	Structural Parameters	UQ	Q
1	Lattice Constant (a)(Å)	8.38	8.44
2	Unit cell volume (v) (Å)3	588.48	601.21
3	X-ray density (dx) (gm/cm3)	5.40	5.29
4	Bulk density (dB) (gm/cm3)	3.55	3.55
5	Porosity (%P)	34.26	32.89
6	Particle (Size) (nm)	39.58	38.91

3.2 Magnetic Measurement

The magnetic properties of quenched and unquenched copper ferrite samples were investigated using pulse field hysteresis loop technique. The hysteresis curve (M-H plot) for unquenched and quenched copper ferrite is shown in Fig. 2(a and b) respectively the curve exhibits typical hysteresis nature similar to other spinel ferrites. Using the M-H plots the saturation magnetization (Ms), coercivity (Hc) and remanence magnetization (Mr) were obtained and their values are given in Table 2 for quenched and unquenched samples. It is observed that after quenching, the saturation magnetization increases whereas, coercivity and remanence magnetization (Mr) decreases. The variation in magnetic parameters is attributed to quenching effect.

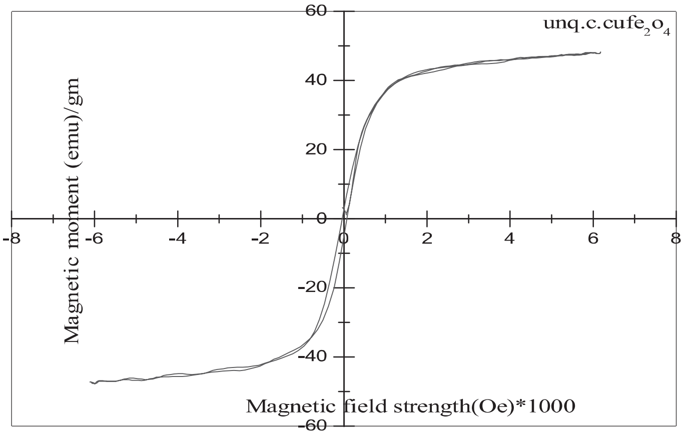

Fig. 2 a M-H plot of UNQ $CuFe_2O_4$

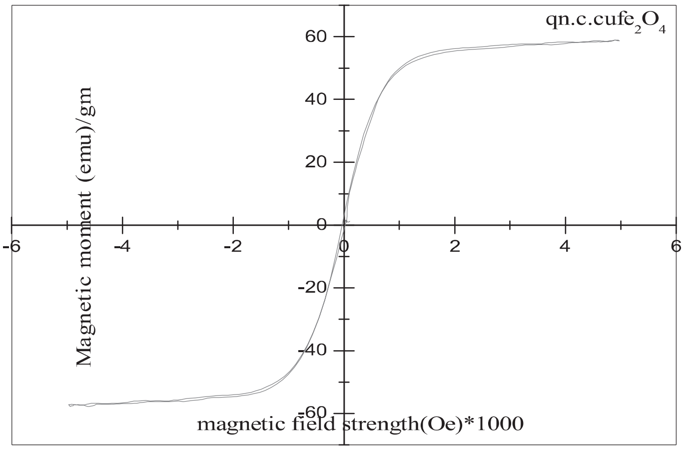

Fig. 2 b, M-H Plot of QN. $CuFe_2O_4$

Fig. 2 (a and b) respectively the curve exhibits typical hysteresis nature similar to other spinel ferrites.

Table 2. Saturation Magnetization (Ms), remanence ratio (Mr/Ms), Coercivity (Hc), Curie temperature (Tc) For Unq and Qn. CuFe2O4 ferrite prepared by ceramic method

Sr. No	Magnetic Parameters	Unquenched	Quenched
1	Ms (emu/gm)	48.07	59
2	Mr (emu/gm)	4.88	0.74
3	Mr/Ms	0.10	0.01
4	Hc (Oe)	48	15.10
5	nB	2.06	2.53
6	Curie temperature (Tc)	460	350

4. Conclusions

The experimental results on structural, electrical and magnetic properties of quenched and unquenched copper ferrite samples prepared by ceramic method led to draw the following conclusions.The sample of copper ferrite prepared by ceramic method shows single phase, cubic spinel structure. Theintensity of the Bragg's reflection found to increase in quenched samples. The lattice constant increases for quenched samples. The crystallite size decreases after quenching. The grain size also decreases for quenched samples. The saturation magnetizationincreases but coercivitydecreases after quenching. Curie temperature decreases sufficiently after quenching. Thus, it is evident from experimental results that sudden quenching from higher temperature to liquid nitrogen temperature causes change in the structural and magnetic properties of copper ferrite.

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