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X-ray diffraction and X-ray K-absorption studies of copper doped complex

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Abstract. Sample of $Ni_{1-x}Cu_xFe_2O_4$where (x=0.05, 0.10, 0.15) has been prepared by root equation method. X-ray diffraction and K-absorption fine structural measurements have been carried out. The sample structure has been found to be cubic. X-ray diffraction measurements have been used to determine particle size and lattice parameter. EXAFS spectra has been recorded at 2.5Gev Indus -2 Synchrotron radiation source at RRCAT, Indore, India. EXAFS data have been analysed by computer software Athena.

1. Introduction

Ferrites are good dielectric materials having low conductivity and have wide application of microwave device. Ferrites are technologically important material because of their unique electric, dielectric, magnetic and an optical property which makes them suitable for many technological application like microwave device, transformer, electric generators storage device[1-6] etc. Extended Absorption fine structure refers to the oscillatory variation of the x-ray absorption as the as a function of photon energy beyond the absorption edge . Such fine structure may have amplitude of up to a few tenth of the edge jump. Synchrotron based X-ray Absorption Fine Structure (XAFS) spectroscopy becomes a powerful technique providing analytical as well as structural information with applications in a wide range of scientific field because of the rapid development of the treatment technique [7-9]. EXAFS has become the technique of choice for local structural investigations in a diverse range of material systems. This technique has also proved very useful, while studying the chemical reaction of matter under extreme condition of temperature and pressure .It is used to determine thebond distance coordination no.and species of the neighbour of the absorbing atom [9].The EXAFS oscillations in the absorption spectrum are caused by interference in the final state from backscattering of the outgoing photoelectron wave by neighbour of the exited .Hence ,it is extremely sensitive to the nearest neighbour bond length.

2. Experimental

Particles of the $Ni_{1-x}Cu_xFe_2O_4$ with x (gm) varying from x=0.05 to 0.15 were prepared by solid root method at room temperature. After weighing grind 8 to 9 hours the sample and powders were sintered at 2-2:30 hours at 550^0c. The XRD measurements were carried out using Bruker D8 Advance X-ray

diffractometer. The X-ray were produced using a sealed tube and the wavelength of X-ray was 0.154nm (Cu K-alpha).The X-ray was detected using a fast counting detector based on silicon strip technology. Powder X-ray diffraction is perhaps the most widely used X-ray diffraction technique for characterizing materials. As the name suggest, the sample is usually in a powdery form, consisting of fine grains of single crystalline material to be studied. The technique is used also widely for studying particle in liquid suspension or polycrystalline solid .The term "powder" really means that the crystalline domains are randomly oriented in the sample. Therefore when the 2-D diffraction pattern is recorded, it shows concentric rings of scattering peaks corresponding to the various d spacing in the crystal lattice. In the powder method, the crystal to be examined is reduced to a very fine powder and placed in a beam of monochromatic X-rays. Each particle of the powder is a tiny crystal oriented at random with respect to the incident beam. Just by chance, some of the particle will be correctly oriented so that their (100) planes, for example, can reflect the incident beam. Other particles will be correctly oriented for (110) reflection, and so on. The result is that every set of lattice planes will be capable of reflection. The mass of powder is equivalent, in fact, to a single crystal rotated, not about one axis, but about all possible axes.

3. Results and discussions

Figure 1 shows the x-ray diffraction pattern of the $Ni_{1-x}Cu_xFe_2O_4$(x=0.05-0.15). It shows the formation of ferrite phase in all 3 samples. The interplanar distance d (Å) are calculated using Braggs law. All the peaks in the diffraction pattern have been indexed and refinement of the lattice parameter was done using powder X software. The crystalline size for each composition are calculate from XRD line width of the (2 1 0)(2 1 1)(2 1 1) peak using Scherrer formulla [10]. The Ferrite phase $Ni_{1-x}Cu_xFe_2O_4$ (x=0.05-0.15) is prepared by solid root technique. The phase confirmed by XRD studies. The particle size (13.3,9.1,8.55,7.84,7.4) nm by XRD calculation. Crystal structure has found cubic. In XRD, particle size is decrease as same manner. In the EXAFS calculation E_A & E_K value evaluated. The bond length of CU-Ni ferrite complexes with the help of Athena software [11]. XRD and EXAFS data shown in table 1 and table 2 and graph figure 1 and figure 2.

Table 1. Structural parameters of $Ni_{(1-x)}Cu_xFe_2O_4$ (x=0.05 to 0.15)

S.No.	Sample name	2θ	h k l	Particle size (nm)	Lattice parameter (Å)
1.	$Ni_{0.95}Cu_{0.05}Fe_2O_4$	43.30	2 1 1	13.43	4.68
2.	$Ni_{0.90}Cu_{0.10}Fe_2O_4$	35.50	2 1 1	11.64	6.32
3.	$Ni_{0.85}Cu_{0.15}Fe_2O_4$	33.11	2 1 1	11.54	6.0

Table 2. XANES parameters and bond length for the K absorption edge of copper in the complexes

S.No.	Sample name	E_K-Edge (eV)	E_A (eV)	Bond length
1.	$Ni_{0.95}Cu_{0.05}Fe_2O_4$	8985	8990.0	1.29
2.	$Ni_{0.90}Cu_{0.10}Fe_2O_4$	8996	8991.4	1.57
3.	$Ni_{0.85}Cu_{0.15}Fe_2O_4$	8997	8992.1	0.73

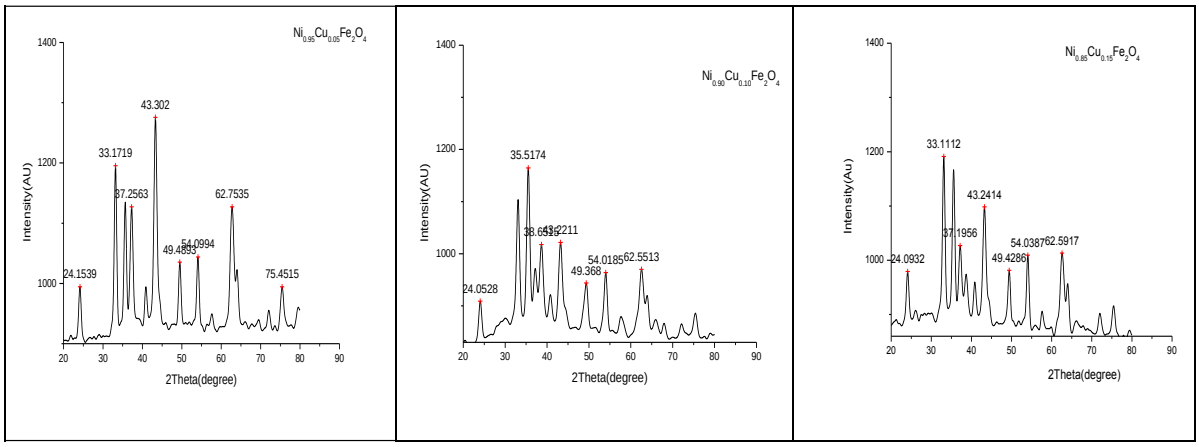

Figure 1. XRD pattern for Cu doped complexes $Ni_{(1-x)}Cu_xFe_2O_4$ (x=0.05 to 0.15)

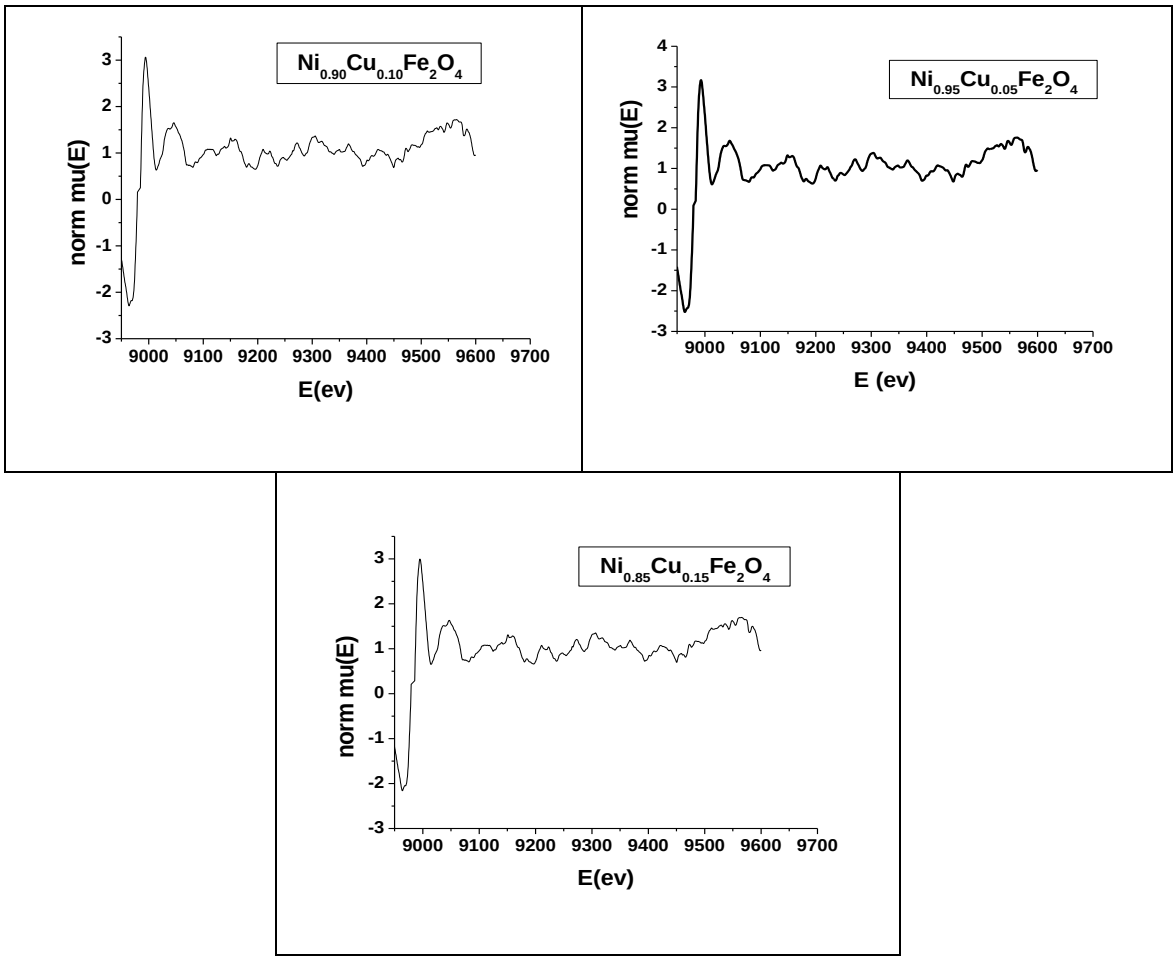

Figure 2. EXAFS spectra [μ(E) Vs E] of $Ni_{(1-x)}Cu_xFe_2O_4$ (x=0.05 to 0.15)

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