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Effect of Sintering Temperature on the Structural and Magnetic Properties of Nickel Ferrite Nanoparticles

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Abstract*—Nickel ferrite nano particles have been synthesized by the sol-gel method. The prepared sample was sintered at four different temperatures (400 ° C, 500 ° C, 600 ° C and 700 ° C) for four hours. The structural characterizations of all the prepared samples were done using SEM, TEM, XRD and FTIR. Crystallite size was found to increase with sintering temperature and this can be attributed to the grain growth of the particles. The particle size of each sample was determined using TEM. The FTIR spectra show two strong absorption bands in the range of 1000–400 cm^{-1}, characteristic of spinal ferrites. VSM analysis confirms that nickel ferrite nano particle shows superparamagnetism at 400 and 500 ° C

Keywords: **Nickel ferrite, sol gel, sintering temperature, magnetic properties.**

I. INTRODUCTION

In the recent years, so much attention has been paid to the nanomagnetic materials that show very interesting magnetic properties. In this material, different properties and applications are appeared as compared to their bulk counterparts. The magnetic properties of nanomaterials are used in medical, electronic, and recording industries that depend on the size, shape, purity and magnetic stability of these materials. In biomedical application, one can use nano magnetic materials as drug carriers inside body where the conventional drug may not work. For this purpose, the nanosize particles should be in the super paramagnetic form with a low blocking temperature. Ferrite nanomaterials are object of intense research because of their proper magnetic properties. It has been reported that when the size of particles reduced to small size or in range of nanomaterials, some of their fundamental properties are affected. nano ferrites are simultaneously good magnetic and dielectric materials. These properties of the nano ferrites are affected by the preparation conditions, chemical composition, sintering temperature and the method of preparation . Several chemical and physical methods such as spray pyrolysis, sol-gel, co-precipitation, combustion technique, high energy milling etc. have been used for the fabrication of stoichiometric and chemically pure nano ferrite materials. Among the available chemical methods, the sol-gel technique is an excellent method to synthesize nanoparticles with maximum purity. This method has the advantage of good stoichiometric control and the production of ultrafine particles with a narrow size distribution. it is observed that the magnetic properties of nickel ferrite nanoparticles are strongly dependent on their size. In spite of the development of a variety of synthesis routes, the production of nickel ferrite nanoparticles with desirable size and magnetic properties is still a challenge. This would justify any effort to produce size tuned nickel ferrite nanoparticles with size ranging from the superparamagnetic threshold of 6 nm to the critical single domain size of 60 nm. In the present paper, the structural and magnetic properties of nanocrystalline nickel ferrite in relation to sintering temperature were investigated.

II. EXPERIMENTAL

A. Synthesis

Nano particles of nickel ferrite were synthesized by the sol-gel combustion method. A stoichiometric ratio of nickel nitrate and ferric nitrate (AR grade MERCK) were dissolved in ethylene glycol using a magnetic stirrer. The solution was then heated at 60 °C for 2 hours until a wet gel of the metal nitrates was obtained. The gel was then dried at 120 ° C. This resulted in the self ignition of the gel producing a highly voluminous and fluffy product. The combustion can be considered as a thermally induced redox reaction of the gel wherein ethylene

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glycol acts as the reducing agent and the nitrate ion acts as an oxidant. The nitrate ion provides an oxidizing environment for the decomposition of the organic component. The obtained powder was ground well and divided into four portions.They were sintered for four hours in a muffle furnace at four different temperatures 400 ° C, 500 ° C, 600 ° C and 700 ° C.

B. Characterization

The nickel ferrite samples were characterized by an X-ray powder diffractometer(XRD, Bruker AXS D8 Advance) using radiation (*wavelength*= 1.5406 A°) at 40 kV and 35 mA. Lattice parameter was calculated assuming cubic symmetry. The crystal structure, crystallite size and X-ray density were determined. The particle size was determined bysubjecting the sample to Transmission Electron Microscopy (Philips, CM200). Wavelength Dispersive X-ray Fluorescence (WD-XRF) Spectrometer (Bruker S4-Pioneer) was used for elemental analysis. The Fourier transform infrared (FTIR) absorption spectra of the samples were recorded using FTIR spectrometer (Thermo Nicolet, Avatar 370) in the wave number range 4000–400 cm^{-1} with Potassium bromide (KBr) as binder. The Magnetic characterization was carried out using a vibrating sample magnetometer (VSM; Lakeshore7410) at room temperature up to a maximum field of 20kOe.

III. RESULTS AND DISCUSSION

A. SEM and TEM Analysis

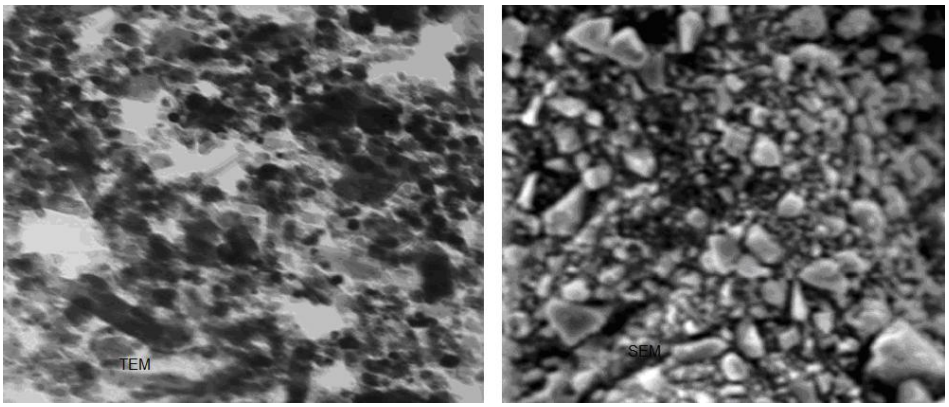

Fig.1. (a) TEM image of sample at 500 ° C (b) SEM image of sample at 500 ° C

The TEM system, which we have been used for morphology and size determination was JEOL JEM-2100 FTEM model. The SEM system that has been used for morphology of sample was CAMSCAN MV2300 model with 15 KV applied voltage. Figure 1 shows the morphology of particles. Photograph has been taken from the samples which were calcinated at 600°C. Particle size is obtained around 23.0 nm with mono disperesed nano particles as one can see from the photograph. In comparison to grain size of particles from XRD results, the sizes are matching well. The voltage range which we have used was between 160 to 200 KV. The EDX pattern also is taken from the same sample (600°C). The model of this system is CAMSCANMV 2300 and 15 KV was applied. Figure 5 shows that sample is very pure and there is no impurity in the sample.

B. Structural Analysis

The XRD patterns of $NiFe_2O_4$ nanoparticles sintered at 400 ° C, 500 ° C, 600 ° C and 700 ° C. are depicted in Fig. 2 and are typical of spinel structure. Comparing the XRD pattern with the standard data (JCPDS PDF card No. 22-1086), the formation of nickel ferrite nanoparticles was confirmed. The sample sintered at 700 ° C showed an extra peak, indicating the formation of hematite phase at this temperature.The diffraction peaks are broad because of the nanometer size of the crystallite. The crystallite size 'D' of the samples has been estimated from the broadening of XRD peaks using the Scherrer equation.

$$D = \frac{K\lambda}{\beta \cos\theta}$$

The lattice parameter 'a' is calculated for prominent peak (311) using Bragg's equatio

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$$a = d_{hkl}\sqrt{h^2 + k^2 + l^2}$$

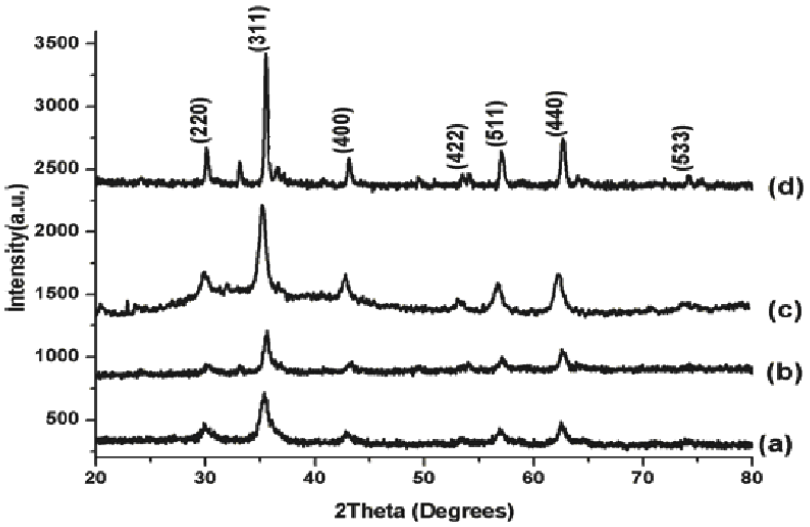

Fig.2. XRD patterns of NiFe2O4 nano particles sintered at (a) 400 ° C (b) 500 ° C (c) 600 ° C and (d) 700 ° C

The actual X-ray density was calculated using the formula

$$\rho_x = \frac{8M}{Na^3}$$

Where, 'M' is the molecular weight, 'N' is Avogadro's number and 'a' is the lattice parameter.
Calculated values of lattice parameter, crystallite size and X-ray density of all the samples are listed in Table 1.

Table 1. Lattice parameter, crystallite size, X-ray density and vibrational frequency band positions of NiFe2O4

Sintering temperature(°C)	Lattice parameter(nm)	crystallite size D (nm)	X-ray density (g/cc)
400	0.8310	10.81	5.419
500	0.8290	16.47	5.464
600	0.8301	17.72	5.442
700	0.8314	50.43	5.417

The crystallite size was observed to increase with higher sintering temperatures. It has been reported that the sintering process generally decreases lattice defects and strain, but this technique can cause the coalescence of smaller grains, resulting in an increased average grain size for the nanoparticles. Calculated values of lattice parameter of Nickel ferrite samples were in close agreement with standard data.

C. FTIR Analysis

Ferrite possesses the structure of mineral spinel $MgAl_2O_4$. It crystallizes in the cubic form with the space group Fd3m. Ferrite can be considered as a continuously bonded crystal with atoms bonded to all nearest neighbours by equivalent forces. In the wave number range 1000–400 cm^{-1}, the infrared bands of solids are usually assigned to vibration of ions in the crystal lattice. FTIR spectra of the investigated samples are shown in Fig. 3.Two main broad metal-oxygen bands are seen in the IR spectra of all spinels, and ferrites in particular. The highest one generally observed in the range 600–550 cm^{-1}, corresponds to intrinsic stretching vibrations of the metal at the tetrahedral site, whereas the lowest band, usually observed in the range 450–385 cm^{-1}, is assigned octahedralmetal stretching. The spectra showed prominent bands near 3400 and 1600 cm^{-1}, which were attributed to the stretching

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modes and H-O-H bending vibrations of the free or absorbed water. The vibrational frequencies of IR bands are listed in table 1, which are in agreement with the reported values.

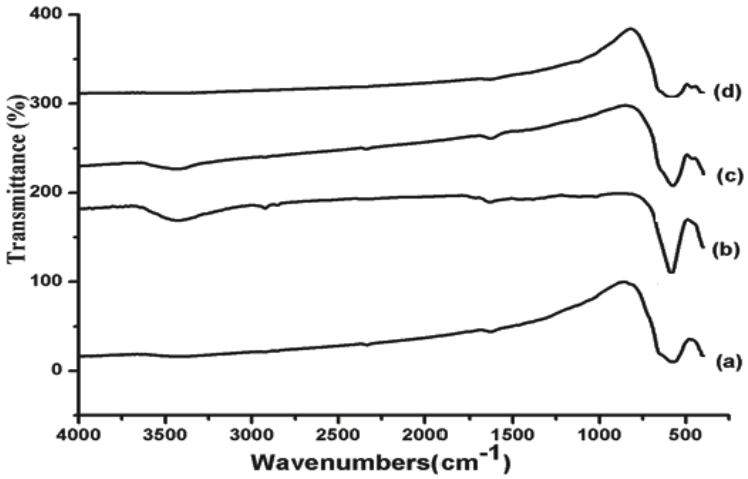

Fig. 3. FTIR spectra of NiFe2O4 sintered at (a) 400 ° C (b) 500 ° C (c) 600 ° C (d) 700° C

D. Magnetic Properties

Vibrating sample magnetometer (VSM) analysis

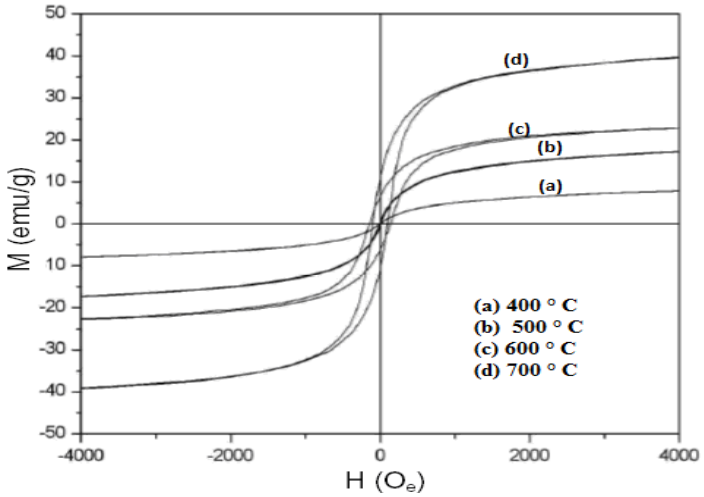

Fig.4. Hysterisis loop of $NiFe_2O_4$ at different sintering temperatures

For particles with large sizes multi-domain are there and becoming more bulk-like with increasing size. When particle size reduces, magnetic domains from multi transfer to a single domain. Thus, below a critical particle size domain walls will no longer form due to energy considerations and single domain particles are stable. Some of samples are calcined at different temperatures (400, 500, 600 and 700°C), conditions for all the samples were same except sintering temperatures. The hysteresis loops (fig.4.) show a good magnetization. Hysteresis loops according 400 and 500°C with particle size less than 8 nm that is less than critical grain size, show superparamagnetic properties that are meaning magnetic remanence (Mr) and coercive force (Hc) are zero.

IV. CONCLUSIONS

In this work pure Nickel ferrite in the ranges 6-60 nm (SEM and TEM) has been successfully synthesized by the sol-gel method. XRD results confirmed the formation of cubic spinel structure in all the samples. The crystallite size of the samples increased with the higher sintering temperatures. The saturation magnetization increased gradually with increasing sintering temperatures and at initial temperatures (400 and 500°C) sample shows super paramagnetism. These results have been explained based on the particle size and surface effects of the ultrafine materials and were in agreement with the Brown's relation.

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