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Preparation of Magnetite (Fe_3O_4) Thin Films by Sol- Gel Method and its Characterization

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Abstract : Thin films can be defined as a thin material layers ranging from fractions of nanometres to several micrometres in thickness. Thin film deposition techniques are divided into two broad categories, physical methods and chemical methods. The different deposition techniques are discussed briefly and the review of the literature related to the present study was discussed under this chapter. Materials and methods used for the preparation of Fe_3O_4 films are discussed in detail. The techniques used to characterize the thin film like UV- Visible; FTIR, XRD and SEM with EDS are discussed. The procedure for the preparation of Fe_3O_4 thin films are discussed in detail. The prepared thin films were subjected to UV-Visible, FTIR, XRD, SEM with EDS, thickness and susceptibility measurements. The obtained results from the above characterization techniques are interpreted with the available literature.

Introduction

Thin film science has grown world-wide into a major research area[40-50]. The importance of coatings and the synthesis of new materials for industry have resulted in a tremendous increase of innovative thin film processing technologies. Currently, this development goes hand in hand with the explosion of scientific and technological break thorough in micro electronics, optics and nanotechnology. Thin films are thin materials layers ranging from fractions of a nanometer to several micrometers in thickness. Thin film technology is innovative and versatile modern technology, its prime objective is minimizing the size, reducing the cost and increasing the efficiency. Thin films were first obtained in 1938. The first thin films were probably the deposits obtained in 1957 by Faraday, when exploded metal wires in an inert atmosphere [10]. Thin film deposition is the process of planning of thin layers of one substance on another. The term thin film has often been loosely used in literatures to imply not only a layer of solid material but also a liquid or a gaseous phase. A thin film is a structure whose dimensions are such that it has substantially large surface to volume ratio. For example, the structure may be macroscopically large in length and width; it may have a thickness that is only of the order of a micron or less. Thin films do not have to be planner. The properties of such thin film structures are strongly influenced by the surface properties and may be very different from that of the same material in bulk form. The thin film may consist of a pure material, or a composite or layered structure, and several of the thin films may be present in a more complex device.

Experimental

Sol-gel method is a convenient and low cost technique and also the annealing temperature is lower, which is necessary for the crystallization process, and the small grain size of the ferrite was obtained. In the present study Magnetite (Fe_3O_4) thin films have been prepared by sol gel method chemically using $FeCl_2$.

$4H_2O$, citric acid monohydrate & tartartaric acid, ascorbic acid and ethanol absolute were used as the raw material, chelating agents, antioxidant agent and solvent respectively and N-N dimethyl formamide (DMF) as a drying chemical control agent (DCCA) for reducing the crack effect.

Experimental techniques

Solution A

0.1mol $FeCl_2.4H_2O$, 0.1 mol citric acid monohydrate and 0.1mol ascorbic acid were first dissolved in 100 ml ethanol absolute, and stirred at 60ºC for 6 h, then 0.1 ml N-N-dimethyl formamide (DMF) as a drying chemical control agent (DCCA) was added in the solution. After these, the solution was stirred for half-an-hour at 60ºC. Then the solution was applied onto pre-cleaned silica-glass substrates by dip-coating technique. The thickness of the films was controllable by repeating the coating steps. The as-coated films were dried at 100ºC to remove the organic substances [33].

Solution B

The chelating agent citric acid monohydrate was used instead of tartaric acid and the same procedure is followed to prepare the required solution as given above. Then the solution was applied onto pre-cleaned silica-glass substrates by dip-coating technique. The thickness of the films was controllable by repeating the coating steps. The as-coated films were dried at 100ºC to remove the organic substances.

Coated and annealed magnetite (Fe_3O_4) thin films prepared via sol-gel method with two different chelating agents are tabulated in the following table 3.1 and all the films were chosen for the further characterization.

Preparation of magnetite thin films

In the present work, the magnetite thin films are prepared using sol-gel method. Sol-gel method is a simple and convenient method widely used in preparing ferrites thin films.

Substrate cleaning

The commercial silica glass slides were used as the substrate with the size of $(75x25x1)mm^3$. It is crucial to have smooth surfaces for producing homogeneous and crack free thin films, because the properties of the films depend on the smoothness of the film surface. The adhesion and the film growth are related to the substrate surface conditions.

For the present study, substrates were rinsed in a 5 wt.% NaOH solution with distilled water and then substrates were rinsed in a 1 wt.% HCl solution with distilled water. Between each step substrates were washed using distilled water. Substrates can be kept in ethanol absolute prior to use [8].

Results and Discussion

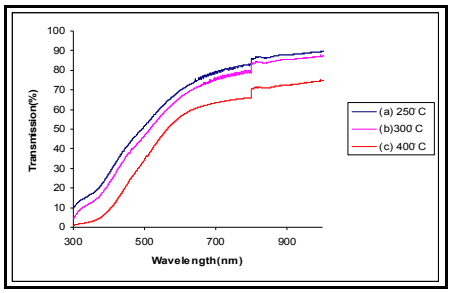
Optical property (UV- Visible study)

Optical transmittance of the films were investigated in the wave length range 300-1100nm. UV-Visible spectra of the three different films amnealed at different temperature of the solution A are shown in Fig.1.1(a). Film-1 gave a maximum of 90% transmittance, film-2 gave a maximum of 87% transmittance and film-4 gave a maximum of 75% transmittance.

UV-Visible spectra of the solution B of the three different films annealed at different temperature are shown in Fig.1.1(b). Film-5 gave a maximum of 87% transmittance, film-6 gave a maximum of 76% transmittance, and film-8 gave a maximum of 75% transmittance.

The transmittance value of the films decreases with increasing annealing temperature, similar trend was observed in the magnetite thin films prepared from both solutions A and B. The above results are in good agreement with the UV-Visible results reported by[8]. At lower wavelength region the transmittance is more in

the case of films prepared with tartaric acid as a chelating agent, the reason may be due to the decreased thickness of the films as shown in table 1.



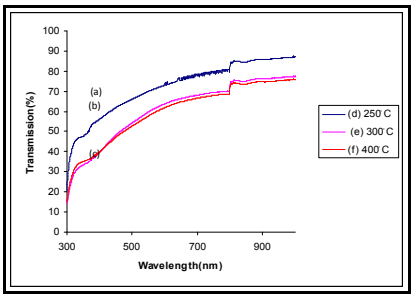


Fig. 1.1 (a) UV- Visible spectra of Fe_3O_4 thin film a) Film – 1 b) Film – 2c) Film – 4 d) Film – 5 e) Film – 6 f) Film – 8

Fig. 1.1 (b) UV- Visible spectra of Fe_3O_4 thin film

Infrared spectroscopic studies (FTIR)

FTIR spectra of the films were recorded in the range 2000-400cm^{-1}. In the present study, as our discussion are concentrated in the region 750-500cm^{-1}, the particular region is shown in Fig.1.2(a). The IR spectra of the film-1 (250ºC) shows broad band centered at 518cm^{-1}, is the characteristic band for iron oxide. At annealing temperature 300ºC (film-2), band at 518cm^{-1} becomes sharp. The bands observed at 518, 608 and 657cm^{-1} for the film-2 might have been due to iron oxide (Magnetite). [27] have reported that the band at 575cm^{-1} is the characteristic absorption band for magnetite. [39] have reported that the band at 510 and 660cm^{-1} respectively is the characteristic absorption band for magnetite. Further annealing temperature at 350ºC shows the appearance of weak band at 570cm^{-1} along with the band at 518 and 658cm^{-1} (film-3). At 400ºC (film-4), both bands at 517 and 581cm^{-1} become distinct and intense which shows that the oxidization of magnetite. Hence, the observed bands at 511, 581 and 659 cm^{-1} might have been due to iron oxide (Hematite). The band at 567cm^{-1} is the characteristic absorption band for hematite**[22].** The band at 515 and 664cm^{-1} is the characteristic absorption band for hematite**[16].** Similar observation is made in the case of films 5,6,7 and 8 from solution B as shown in Fig.1.2 (b). The above study confirms the formation of crystalline magnetite at annealing temperature 300ºC and the oxidization of magnetite above 300ºC.

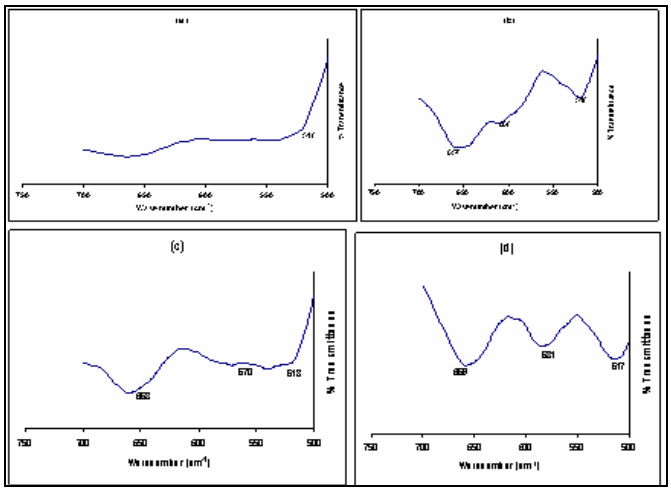


Fig. 1.2(a) FTIR spectrum of Fe_3O_4 thin film a) Film – 1 b) Film – 2 (c) Film – 3 d) Film – 4

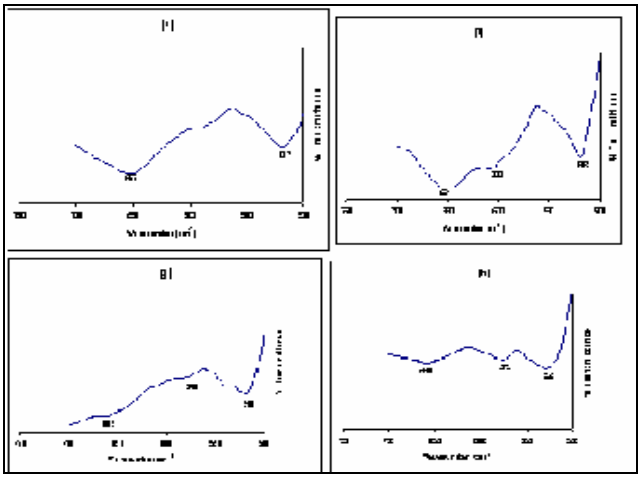


Fig. 1.2 (b) FTIR spectrum of Fe_3O_4 thin film (e) Film – 5 (f) Film – 6 (g) Film – 7 (h) Film – 8

Structural Property (XRD)

Grazing incidence X-ray diffraction was used to analyze the magnetite thin films. Fig. 1.3(a) shows the X-ray diffraction patterns of the films 1, 2 and 4. Spectrum of film-1 shown in fig.(a) is found to be amorphous in nature. At 250ºC the reactions include the decomposition of organometallic precursors and the combustion of

the ester takes place, the sample includes polycrystalline Fe_3O_4 and organic ester. XRD pattern of the film-2 shows peaks with 2θ values (2θ=35.47º,62.31º,56.89º) are attributed to magnetite [33]**.** The observed values are found to be in good agreement with the standard values (JCPDS data file 85-1436). At 300ºC the intensities of the Fe_3O_4 peaks have increased as shown in fig. (b). Clear diffraction peaks corresponding to Fe_3O_4 and no other impure peaks are detected in this film. The results also show that the film is polycrystalline with no preferred grain orientation.

The observed peaks at (2θ=33.13º, 35.57º, 54.15º, 49.39º) for film-4, are attributed to hematite [33]. The peak values are in good agreement with the standard values (JCPDS data file 24-0072). In the present study X-ray diffraction pattern of the film at 400ºC shows no peaks corresponding to magnetite, which is in good agreement with the report by **[4].**It showed that the sol-gel method is successful method for preparing Fe_3O_4 film. Fig. (C) also shows that the diffraction pattern of an oxidized Fe_3O_4 film, the diffraction peaks corresponds to the phase of α -Fe_2O_3.

The X-ray diffraction patterns of the films 5, 6 and 8 are shown in Fig.1.3 (b). At 250ºC the film-5 (fig.(d)) was found to be amorphous in nature. The increased intensity of the diffraction peak obtained in the case of film-6 (fig.(e)), corresponds to the phase of magnetite. Fig.(f) shows the diffraction pattern of film-8, the diffraction peak corresponds to the phase of α -Fe_2O_3, which shows that diffraction pattern of an oxidized magnetite. Oxidization reaction of magnetite $4Fe_3O_4 + O_2 \rightarrow 6Fe_2O_3$ will gain in weight during the reaction[4].The intense and increased no: of peaks corresponding to magnetite in film-5 (solution B) indicates that tartaric acid is the best chelating agent than citric acid monohydrate for producing magnetite thin film. Similar sharp and intense peaks are observed for film-6 attributed to hematite, shows the oxidization of magnetite. The oxidization of magnetite is being confirmed from FTIR spectra with bands at 511, 581 and 659 at 400ºC.

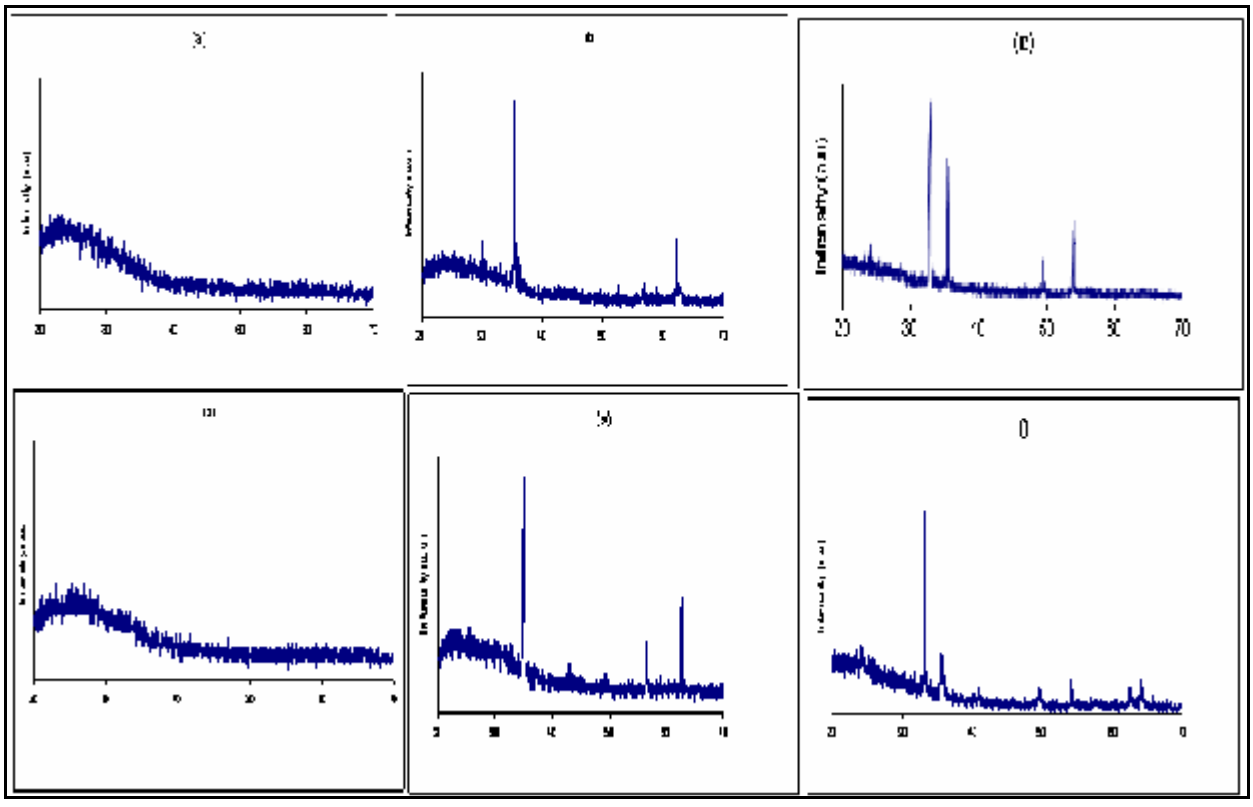


Fig. 1.3(a) XRD thin film of 1, 2 & 4

Fig. 1.3(b) XRD thin film of 5, 6 & 8

Surface Morphology Studies (SEM)

Surface morphology of magnetite thin film were examined by SEM micrographs. The SEM micrographs of coated and annealed Fe_3O_4 thin films1,2 and 6 are shown in Fig. 1.4. The surfaces of these films were found to be smooth and adherent firmly on to the substrate. No cracks and other defect (except very few

pores) can be seen in the SEM image, because of adding N-N DMF as DCCA. It shows that N-N DMF was used to reduce the pores and also shows that DCCA in the solution prevented the thin film from cracking effectively[33]. The presence of minimum number of pores can also be minimized by increasing the volume of N-N DMF.

SEM micrograph of film-1 (250ºC) shows grain size in the range 90-170nm. In the case of film-2 annealed at 300ºC grain size was found to be 120-240nm. From the above observation it is evident that, when the annealing temperature increases the grain size increases[39]. The sizes of the grain are found to be in the range 120-270 nm for the film-6. From the above discussion it is evident that the grain size of the film from solution B is found to be higher than from solution A.

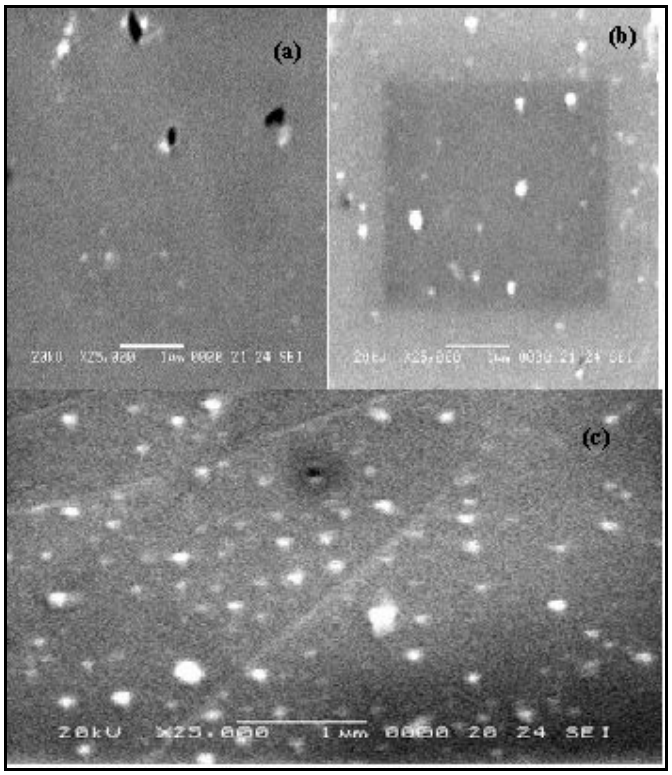


Fig.1.4. SEM micrograph of Fe_3O_4 thin film (a) Film – 1 (b) Film – 2 c) Film – 6

Elemental Composition Analysis (EDS)

Energy dispersive X-ray spectroscopy (EDS) is powerful technique used to identify the elemental composition of the specimen. The EDS for Fe_3O_4 films1, 2 and 6 are shown in Fig.1.5. The elemental composition and average ratio of atomic percentage of Fe/O for Fe_3O_4 films1, 2 and 6 are tabulated in table 1.

Table 1 Elemental composition of Fe_3O_4 thin film

Fe_3O_4 Film	Element (atomic %)		Fe/O ratio
	Fe	O	
Film -1	48.03	51.97	0.92
Film – 2	47.43	52.57	0.90
Film– 6	44.08	55.92	0.78

[40]. have reported that the atomic percentage compositions of Fe and O for Fe_3O_4 are 42% and 58% respectively and atomic ratio is 0.72. Also the author reported the theoretical value of Fe/O atomic ratio of Fe_3O_4 is 0.75. The Fe/O atomic ratio of the films 1, 2 and 6 are 0.92, 0.90 and 0.78 respectively. The atomic ratio of film-6 is found to be consistent with the theoretical and experimental Fe/O atomic ratio reported by **[40].** Deviation in atomic ratio is noticed in the case of film-1 and film-2 from solution A. XRD and EDS spectra reveals that the films are composed of Fe_3O_4 and tartaric acid is the best chelating agent.

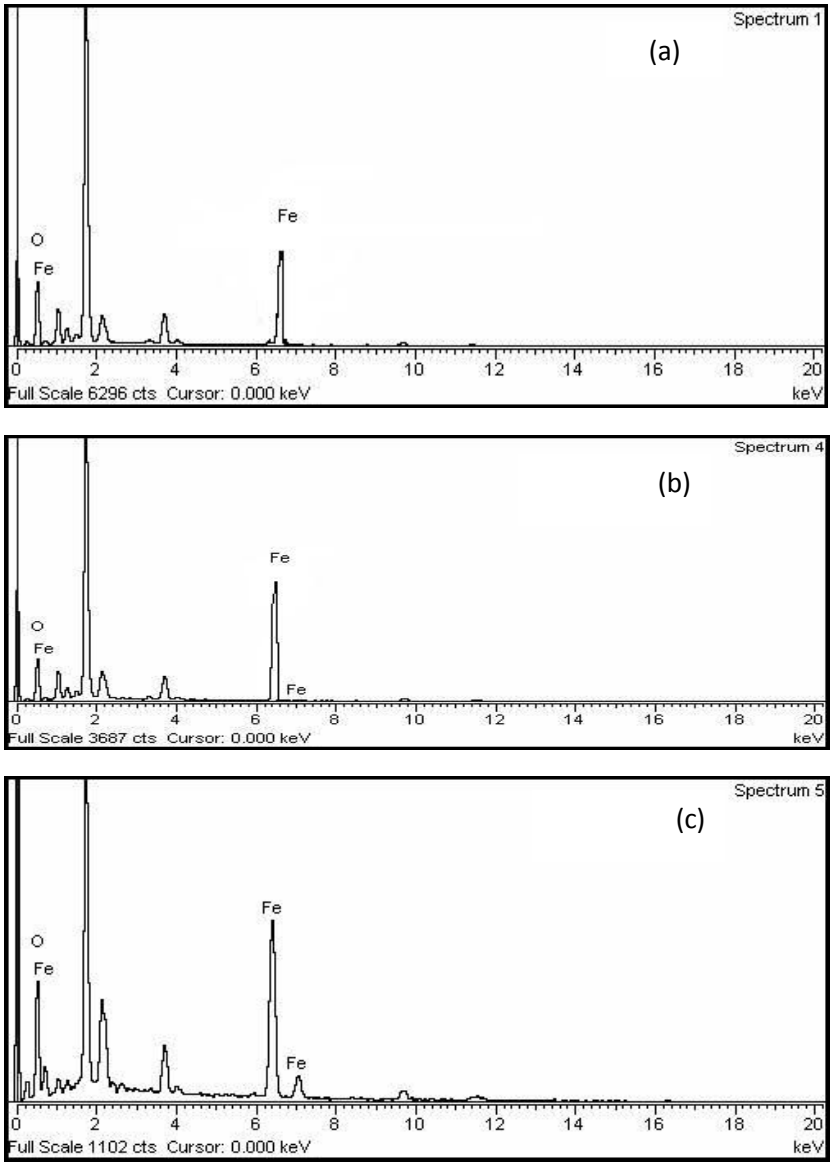


Fig. 1.5 EDS for Fe_3O_4 thin film (a) Film – 1 (b) Film – 2 (c) Film - 6

Susceptibility measurements

In this study, the dry gel was obtained after the sol was dried at 100ºC in a drying furnace for 24 h in air. The dry gel was powdered using agate mortor to measure the susceptibility values. The measured susceptibility values for the powdered samples in the as received state (100ºC) and fired to 300 and 400ºC are given in table 3.4.

Magnetic susceptibility measures the magnetizability of a material in the natural environment, which mainly tells us about Fe- bearing minerals**[35]**. Magnetic susceptibility (χ) describes the magnetic response of a sample when exposed to a (generally weak) magnetic field. χ is mainly a function of the concentration and mineralogy of the ferrimagnetic (Magnetite, Maghemite and Fe sulphides) minerals present, but can also depend on the strength of the applied field and the particle size distribution of the magnetic grains. In the absence of ferrimagnetic minerals χ can be due to antiferromagnetic (Hematite and Goethite) minerals. Magentic susceptibility is also dependent on sample size and therefore it is customary to present susceptibility as a mass normalized susceptibility in the unit of $m^3 Kg^{-1}$ [26].

Frequency-dependent susceptibility (χ_{FD}) express the difference between susceptibility (χ_{LF}) measured at low frequency (Often 0.47 KHz) and susceptibility (χ_{HF}) measured at high frequency (often 4.7 KHz), expressed as a percentage of χ_{FD} $\left(\chi_{FD}\% = \frac{\chi_{LF} - \chi_{HF}}{\chi_{LF}} \times 100 \right)$. The frequency dependent susceptibility parameter is used to detect ultrafine (<0.03 μ m) ferrimagnetic minerals lying in the superparamagnetic (SP) grain size.[6] have reported that samples with $\chi_{FD}\% > 2$ have detectable concentration of SP grains, and if $\chi_{FD}\%$ is around 6~10, samples contain significant amount of the SP grains of size 0.012 ~ 0.023 μ m.

From the table 3.4 it is seen that susceptibility value increases at 300ºC irrespective of the solution and decreases at 400ºC, due to the structural changes take place inbetween the temperature.[33] reported that the oxidization of magnetite takes place above the annealing temperature 300ºC. The lower susceptibility value of powder at 250ºC is due to the amorphous in nature as well as the smaller grain size. At 300ºC the higher susceptibility value of 27136.00 X 10^{-8} m^3 Kg^{-1} can be due to formation of ferrimagnetic mineral (Crystallized magnetite). The measured higher susceptibility value of 48211.00 X10^{-8} m^3 kg^{-1} powder at 300ºC from solution B indicates the formation of highly crystalline magnetite than from Solution A. The obtained higher susceptibility values at 300ºC are in good agreement with the reported susceptibility values 20,000 – 1,10,000 X10^{-8} m^3 kg^{-1} for magnetite[13]. All the powder samples show $\chi_{FD}\% > 2$ and most of the samples fall in between 2-4 indicating the detectable concentration of superparamagnetic (SP) magnetite grains. The sample from solution A at 100ºC with $\chi_{FD}\%$ value of 8.67 reveals that significant content of SP magnetite grains.

Conclusions

The results reveals that tartaric acid is the best chelating agent than citric acid monohydrate and also sol-gel method is an inexpensive and successful method to prepare magnetite (Fe_3O_4) thin films, which is suitable for magnetic recording media and spin valve applications.

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