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# Study of phase transition in iron oxide thin films

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**Abstract**

We here report preparation and characterization of iron oxide thin films using sol-gel method. Iron nitrate is used as precursor whereas water and ethylene glycol as solvent. Three different sols are prepared with pH 1 (Acidic), 7 (neutral) and 13 (basic). The films are spin coated on copper substrate and annealed at 300˚C for 60mins. XRD results indicate the formation of phase pure $Fe_3O_4$ at pH 1 while phase transition from $Fe_3O_4$ to $\gamma$-$Fe_2O_3$ is observed with change in pH from 1 to 7. Whereas in the basic medium (pH 13) $\alpha$-$Fe_2O_3$ phase is obtained. These transitions between different phases of iron oxide strongly affect the magnetic properties. Films with pH 1 show highest saturation magnetization while decrease in saturation magnetization is observed as pH is increased to 7, indicative of phase transition from $Fe_3O_4$ to $\gamma$-$Fe_2O_3$. Further decrease in saturation magnetization in basic medium is associated with phase transition to $\alpha$-$Fe_2O_3$ phase.


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## 1. Introduction

Study of different phases of iron oxide thin films is a motivating issue owing to their applications in spintronic and data storage devices [1]. Iron oxide exists in three important crystallographic phases that vary in stoichiometry, crystallographic structure and chemical state including magnetite ($Fe_3O_4$), maghemite ($\gamma$-$Fe_2O_3$) and hematite ($\alpha$-$Fe_2O_3$). Two oxidation states of iron $Fe^{2+}$ (ferrous) and $Fe^{3+}$ (ferric) are present in iron oxide [2].

Magnetite ($Fe_3O_4$ or $FeO.Fe_2O_3$) crystallizes in inverse spinel cubic structure. Oxygen ions are set in cubic close packed arrangement and both $Fe^{2+}$ (ferrous) and $Fe^{3+}$ (ferric) cations are present. Maghemite ($\gamma$-$Fe_2O_3$) is the transition phase between magnetite $Fe_3O_4$ and hematite $\alpha$-$Fe_2O_3$. Maghemite has cubic inverse spinel crystal structure similar to that of magnetite but with the difference that there are vacancies present in maghemite. Magnetite exhibits ferrimagnetic behavior with Curie temperature of 850K. $\gamma$-$Fe_2O_3$ is also ferrimagnetic but it is unable to have its exact Curie temperature owing to the fact that it undergoes irreversible changes in its crystalline structure at 300°C. This leads to loss of magnetization at high temperature [2, 8-10].

Hematite ($\alpha$-$Fe_2O_3$) has hexagonal corundum structure [3-5]. The oxygen anions are arranged in hexagonal close pack (hcp) framework while Fe(III) cations are arranged at octahedral sites [1]. Hematite is weak ferromagnetic with Curie temperature of 956K [2, 6, 7].

The biggest distinguishing feature of magnetite from other iron oxides is that at Fermi level only spin down electrons are present. Magnetite has a theoretically predicted spin polarization of 100%. So it acts as an insulator for one spin orientation and as a conductor for the other spin orientation [11-15]. However, synthesis of pure phase magnetite is extremely difficult as it easily gets oxidized resulting in phase transformation to maghemite. In addition there are only few reports for magnetite thin films using wet chemical synthesis especially sol-gel method [16, 17].

We here report the preparation of iron oxide thin films using sol-gel method. pH of sol was varied as 1, 7 and 13. The films were annealed at 300˚C for 60mins. It can be seen conclusively that with variation in pH of the sol different phases of iron oxide i.e. magnetite, maghemite and hematite can be obtained. These variations in phases of iron oxide strongly affect the magnetic properties.

## 2. Experimental Details

For synthesis of iron oxide thin films iron nitrate was used as precursor. Iron nitrate ($Fe(NO_3)_3.9H_2O$) was dissolved in DI water. Ethylene glycol was then added to the above solution and resultant solution was heated on hot plate at 60˚C to obtain iron oxide sol. The details of sol-gel synthesis have been reported earlier [8, 9]. The pH of sol was varied as 1, 7 and 13 using ammonium hydroxide. For deposition of iron oxide thin films copper was chosen as substrate. Before spin coating copper was etched with diluted HCl to remove the oxide. The substrates were rinsed repeatedly in DI water followed by ultrasonication in acetone and isopropyl alcohol for 20mins [18-20]. The sols were spin coated at 3000rpm for 30sec. Films were dried at room temperature and annealed at 300˚C for 60mins.

The films were characterized structurally using Bruker D8 Advance X-ray Diffractometer with CuK$\alpha$ radiations ($\lambda$= 1.5406Å) and nickel filter. Magnetic properties were studied using Lakeshore's 7407 Vibrating Sample Magnetometer.

## 3. Results and Discussion

Fig. 1(a) shows XRD pattern for iron oxide thin films prepared under pH 1 condition. The presence of diffraction peaks corresponding to (311), (222), (400), (422), (511) and (533) planes indicate the formation of pure magnetite phase (JCPDS card no. 72-2303). With increase in pH to 7 diffraction peaks emerge corresponding to planes (211) and (221) (JCPDS card no. 39-1346) (Fig. 1(b)). With increasing the pH to 13, phase conversion from maghemite to hematite phase was observed as can be seen in Fig. 1(c).