# Study Of Wet Chemical Deposited Magnetite Thin Film On Si Substrate: Synthesis And Morphology Studies

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## *Abstract*

*Chemical route has been used for the deposition of $Fe_3O_4$ thin film on the Si substrate. By varying the reaction time a variation in the size of the $Fe_3O_4$ thin films has been observed. Initially, $\alpha$-$Fe_2O_3$ thin film was formed on the surface of Si substrate which by annealing at 500$^oC$ temperature under $H_2$ gas environment converts into magnetite thin film. We have obtained uniform deposition of $Fe_3O_4$ nanoparticles on the surface of the Si substrate as confirmed by SEM characterization. The size of $Fe_3O_4$ nanoparticles was found to be in the range of 20 nm to 50 nm. The EDX have been performed for elemental analysis of the synthesized hematite and magnetite thin film.*

## 1. Introduction

Interest in magnetite nanoparticles has been increased over the past decade due to its unique magnetic properties and potential applications in various fields such as in magnetic sensors, catalysis, medical diagnostics, magnetic resonance imaging, magneto-optic devices and spintronic devices etc. [1-4]. Now a days researchers are also working on the deposition of magnetite thin films on the substrates for high performance magnetic recording applications, tunnel magnetoresistance devices, magnetic sensor technology and room temperature giant magnetoresistance applications [5-7]. Various methods used for the synthesis of magnetite thin films include pulsed laser deposition , magnetron sputtering, sol-gel method, hydrothermal processes , and molecular beam epitaxy [9-13].

In the present paper we have proposed a simple and cost-effective method for growing magnetite thin films on Si substrate based on a dip coating process. The advantage of this method used for thin film synthesis is that it gives possibility of handling large area of substrates and is time and cost effective. The magnetite nanoparticles were synthesized by wet chemical method under magnetic stirring and Si wafers were kept in solution for deposition of magnetite film on it. Furthermore it is well known that magnetite ($Fe_3O_4$) is not thermodynamically stable under oxidizing conditions and get oxidized to $\alpha$-$Fe_2O_3$. For this reason, we reduced hematite to magnetite in hydrogen gas.

## 2. Experimental details

### 2.1. Materials

Iron(III) chloride ( $FeCl_3 \cdot 6H_2O$), iron(II) chloride ($FeCl_2$), hydrogen fluoride and Ammonia. All the chemicals were of AR grade (Merck India) and were used as received. Double distilled water was used as a solvent.

### 2.2. Synthesis

Iron(III) chloride ( $FeCl_3 \cdot 6H_2O$) and iron(II) chloride ($FeCl_2$) were prepared in 2:1 molar ratio and then mixed under constant stirring with the help of a magnetic stirrer at 60 $^0$ C temperature. Silicon wafers etched with hydrogen fluoride and washed with distilled water were dipped into the solution and then ammonia solution was poured drop wise until the solution appears black. After the formation of black precipitates the solution was kept at 70 $^0$C under magnetic stirring. Silicon wafers kept in solution were removed one by one after five minutes, ten minutes, twenty minutes, thirty minutes, one hour, two hour respectively . The wafers were then washed twice with double distilled water to get thin film free from chlorine ions and other impurities. Finally the Si wafers were dried in inert gas environment at 500 $^0$C temperature for one hour to convert hematite into magnetite. Figure 1 shows the schematic representation of magnetite thin film deposition on Si substrate.

**Figure 1** Schematic diagram for deposition of magnetite thin film on Si substrate

### 2.3. Characterization

Different characterization techniques i.e. XRD, SEM and EDX have been used to study the size, shape, morphology and elemental analysis of

magnetite thin films. X-ray diffractometer (XRD) (Panalytical's X'Pert Pro) has Cu-Ka radiation (1.54 Å). Goniometer = PW3050/60 (Theta/Theta); Minimum step size 2Theta:0.001; Minimum step size Omega: 0.001 was used to study structural parameters. Scanning electron microscopy (Hitachi, S-4700, 60-10 kV operating voltage) images of material were obtained at 10 kV.

## 3. Result and Discussions

Figure 2(a-f) shows the FESEM micrographs for magnetite thin films on Si substrate synthesized at different time intervals. A variation in the morphology has been observed by variation in the substrate insertion time (5 minutes to two hrs) in aqueous solution during stirring. From the images it has been observed that the thickness of the films increases as substrate was dipped for large time with constant stirring at $70^0$C.

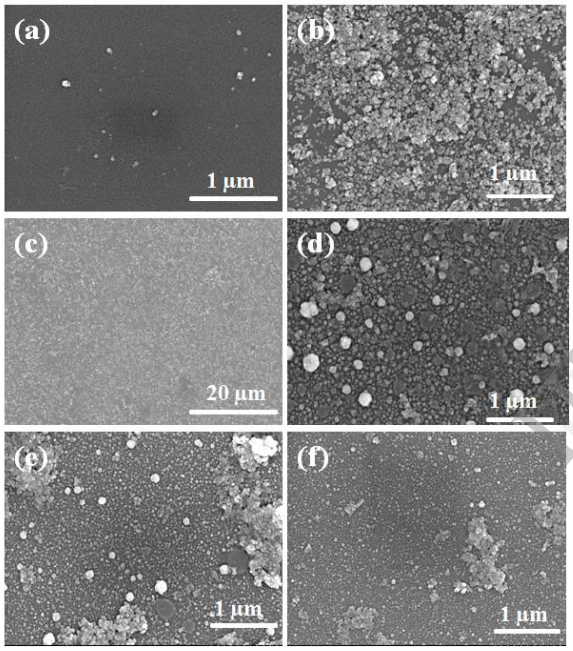



**Figure 2** SEM image of synthesized magnetite thin film on silicon substrate for different interval of dip time for (a) 5 minutes (b) 10 minutes (c) 20 minutes (d) 30 minutes (e) 1 hour (f) 2 hours.

Figure 3 shows the XRD pattern for hematite and magnetite. XRD analysis confirms the formation of hematite and then that hematite get converted into magnetite in hydrogen gas environment at 500 ºC. XRD for hematite and megneite shows the amorphous nature.

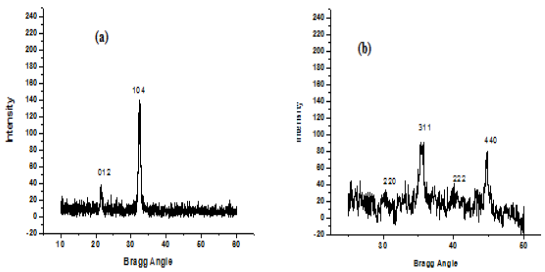
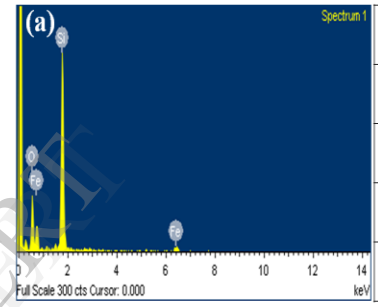
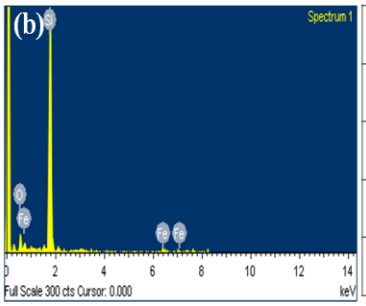



**Figure 3** (a) and (b) XRD patterns of the samples prepared with different ratios of Fe3+/Fe2+ ions for hematite and magnetite.

Figure 4(a) and (b) shows the EDX spectra for hematite and magnetite respectively. From the EDX spectra it has been observed that Fe, Oxygen and Silicon elements are present in the samples which confirms the deposition of iron oxides i.e. hematite and magnetite on Si substrate.




| Element | Wt % | Atomic % |
|---|---|---|
| O K | 24.07 | 43.03 |
| Si K | 35.74 | 36.30 |
| Fe K | 40.19 | 20.50 |
| TOTAL | 100.00 | |




| Element | Wt % | Atomic % |
|---|---|---|
| O K | 16.15 | 27.09 |
| Si K | 68.69 | 65.63 |
| Fe K | 15.16 | 7.23 |
| TOTAL | 100.00 | |

**Figure 4** EDX spectrum and table for (a) Hematite (b) Magnetite

## 4. Conclusion

Nanocrystalline magnetite thin film has been successfully deposited on Si substrate by dip coating method. Magnetite thin films were characterized by XRD, SEM and EDX techniques. We have studied the effect of reaction time on the morphology and size of magnetite thin films deposited on Si substrate by varying the reaction time from 5 min to 2 hrs. A variation in time shows a variation in size and morphology of thin films as confirmed by SEM images. The SEM images also showed that thickness of the film increase with increase in insertion time. Thus we can grow a single layer thin film of magnetite

on Si substrate by employing this simple and cost effective method for industrial applications.

**Referances**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