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CHARACTERIZATION OF SPIN COATED IRON OXIDE FILMS USING EXAFS

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Abstract

Thin films consist of Fe_2O_3 were synthesized on amorphous glass substrates at different rotating speeds up to 1000 rev/min for different durations of 1-2 min using solutions with different chemical compositions by spin coating technique. Fabricated films were subsequently annealed in air at different temperatures in the range of 200-350 ^0C for 0-2 hours in order to crystallize the phase of iron oxide. Films were characterized using EXAFS to determine the phase of Iron oxide crystallized in the sample. The EXAFS analysis reveals that the properties of the sample don't depend on the annealing conditions in this temperature range.

Keywords: *Iron oxide, spin coating method, EXAFS, annealing*

1. Introduction:

Iron oxide is a prime candidate in the applications of magnetic memory devices and microwave devices. Thin films of Iron oxide have been synthesized by both post-oxidation of pure Fe ultra-thin films and by evaporating Fe onto the MO substrates [1]. Colored iron oxide thin films have been fabricated by Sol-gel technique [2]. Iron oxide thin films have been grown over fused quartz substrate using simple metal organic deposition from Fe-(III) acetylacetonate as the organic precursor [3]. Fe_3O_4 thin films have been sputter synthesized from a target consisting of a mixture of Fe_3O_4 and Fe_2O_3 onto Si and glass substrates [4]. Also thin films of hematite have been fabricated using pulsed laser depositions [5]. But spin coating has been identified as a low cost method compared to other expensive techniques required some high tech sophisticated equipments and vacuum.

ZnO [6, 9] and CuO [7, 8, 10] thin films have been fabricated using dc sputtering by us previously. Gas sensor [8, 9, 10] and Photovoltaic [6, 7] properties of these films have been investigated. Those films characterized using X- ray diffraction method. In addition, thin films of tungsten oxide [11], titanium dioxide [12] and p-Cu_2O/n-CuO Junction [13] have been prepared and characterized by us previously. Although it is difficult to grow thin films of some materials using spin coating technique, thin films of oxide materials can be easily fabricated using some low cost techniques such as spin coating method. Therefore, the spin coating method was employed to deposit the layers of iron oxide on glass substrates. The films were synthesized using chemical compounds with different compositions at different rotating speeds. Subsequently, films were annealed at different temperatures in order to crystallize the required phase of iron oxide.

2. Experimental:

Thin films of Iron oxide were prepared using the sol gel technique. Thin films with different compositions of iron (III) nitrate and ethylene glycol were deposited on glass substrates in order to find

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the best composition. Best composition was found to be 14 g of iron (III) nitrate dissolved in 55g of ethylene glycol. All the films described in this report were synthesized using the solution with this special composition. Iron (III) nitrate was dissolved in ethylene glycol in a continuous flow of N_2 gas. Then, the solution under continuous stirring was heated to 80 ^oC for 2 h in a flow of N_2 gas. Finally this solution was applied to amorphous glass substrates. The rotational speed of spin coater was gradually increased up to 1000 rev/min with in 1-2 min. Coating time of all the samples was 1-2 min. The samples were subsequently annealed at 200-350 ^0C in air for 0-2 hours to crystallize the phase.

Films were characterized using EXAFS synchrotron with energy 6800-7800 eV located at Brookheavan national labs, USA. Data was collected at the Fe K-edge.

3. Results and discussion:

All the prepared samples were 1-2 μm thick. EXAFS of the sample prepared at speed of 900 rev/min within 90 seconds is given in figure 1. This sample was annealed at 200 ^0C in air for 2 hours. Here absorption edge at 7112 eV indicates the excitation of 1s electron of Fe. After scattering phase shift correction in Fourier transform of this pattern, the first peak in EXAFS region (not in XANES region) of the graph appears at 2.14 ^0A. This value gives the separation between Fe and O atoms in Fe-O bonding. So these data confirms the formation of the phase of Iron oxide.

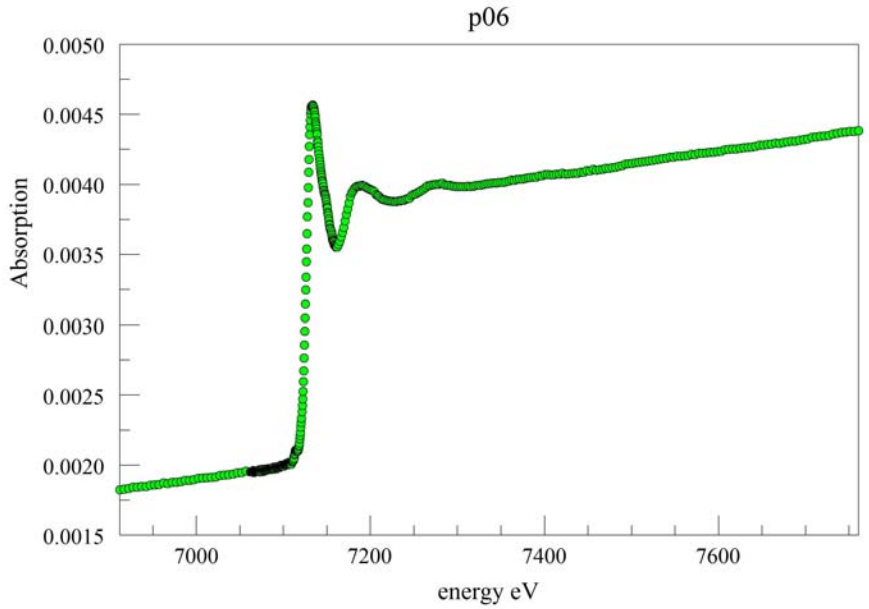

Figure 1: EXAFS of iron oxide sample annealed at 200 ^0C for 2 hours.

Figure 2 shows the XANES part of the sample coated at speed of 900 rev/min within 60 seconds on amorphous glass substrate. According to this graph, the phase crystallized in the film sample was identified to be Fe_2O_3. This sample was annealed at 250 ^0C in air for 2 hours.

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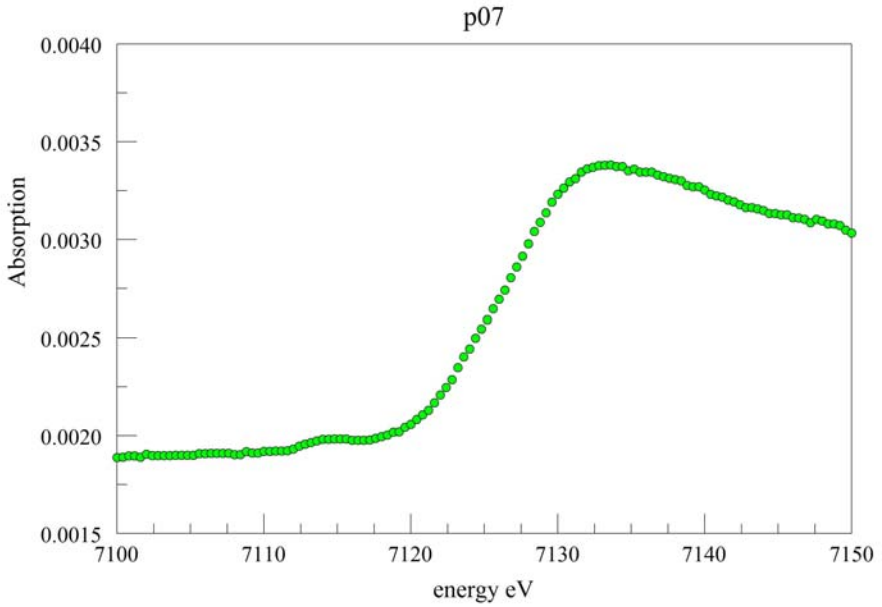

Figure 2: XANES region of sample annealed at 250 ^{0}C for 2 hours.

EXAFS of the sample synthesized at speed of 900 rev/min within 60 seconds is given in figure 3. This sample was subsequently annealed at 350 ^{0}C for 2 hours. The EXAFS and XANES parts of this sample are exactly the same as the sample given in Figure 1. This implies that the properties of the film sample don't depend on the annealing conditions in the range described in this manuscript.

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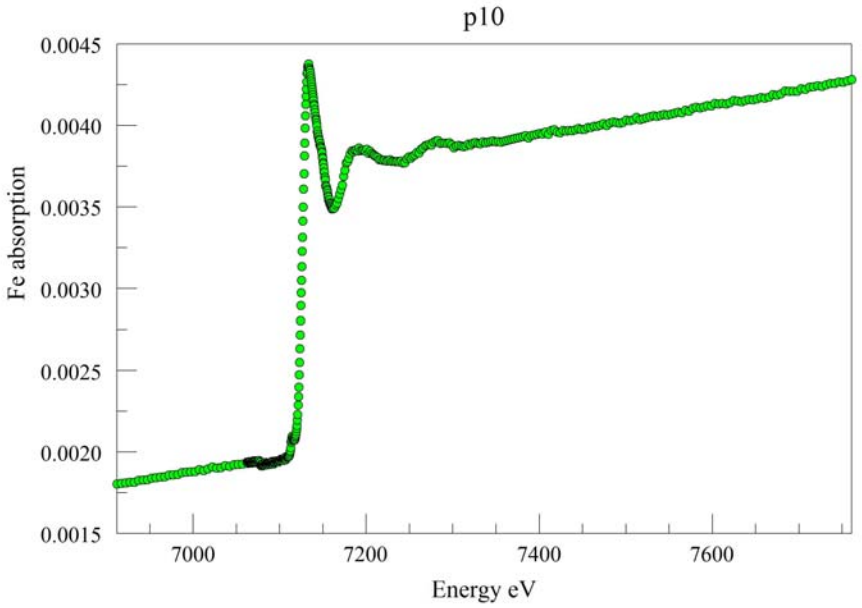

Figure 3: EXAFS of the sample annealed at 350 ^{0}C for 2 hours.

XANES region of the sample annealed at 350 ^{0}C for 1.5 hours is shown in figure 4. This sample was fabricated at speed of 900 rev/min within 60 seconds on amorphous glass substrate. This graph is exactly the same as the graph shown in figure 2 by indicating that annealing conditions in this temperature range does not affect the phase crystallized in the film sample.

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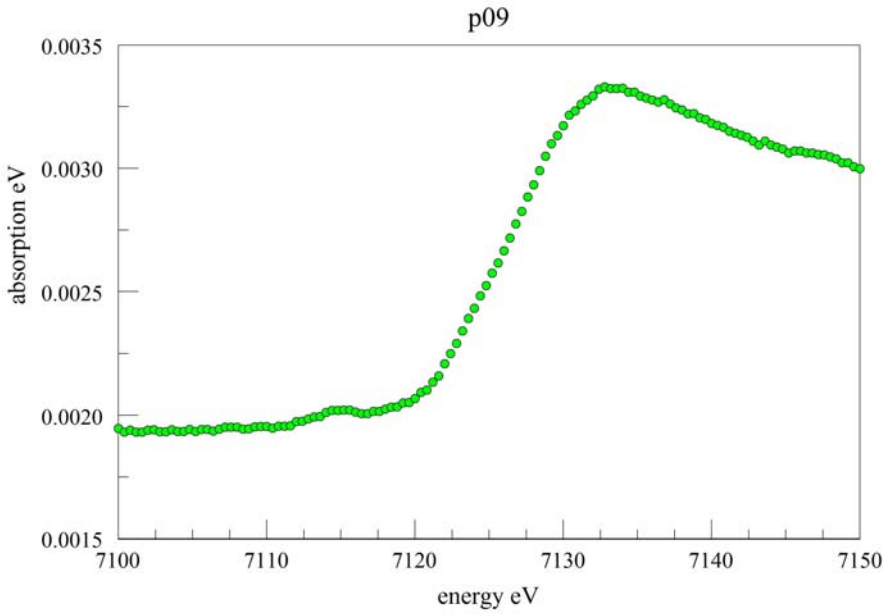

Figure 4: XANES part of sample annealed at 350 ^{0}C for 1.5 hours.

4. Conclusion:

According to the EXAFS and XANES data of our spin coated samples, the phase of Fe_2O_3 has been crystallized after annealing. All the films were subsequently annealed at 200-350 ^{0}C in air for 0-2 hours. The structural properties of the films don't depend on the annealing conditions for the temperature range given in this manuscript. Absorption edge of Fe could be observed at 7112 eV due to 1s excitation. Fe-O bond length was found to be 2.14 ^{0}A. These Fe_2O_3 films can be potentially applied in magnetic memory and microwave devices. Our results completely agree with the EXAFS and XANES patterns obtained by some other researchers for Fe_2O_3 previously [14].

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