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SPIN COATED Fe AND Ni MIXED OXIDE ($Fe_xNi_yO_z$) FILMS AND THEIR STRUCTURAL PROPERTIES

[1]P. Samarasekara, [1]Rasika Dahanayake, [2]S. Dehipawalage
[1] Department of Physics, University of Peradeniya, Peradeniya, Sri Lanka
[2] Physics Department, Queensborough Community College of CUNY, 222-05, 56th Avenue, Bayside, NY 11364, USA

Abstract
Films of $Fe_xNi_yO_z$ have been synthesized using spin coating method. Solutions consist of Fe and Ni was applied on amorphous glass substrates, and samples were fabricated at different rotation speeds of spin coating machine. Samples were subsequently annealed at different temperatures in air for different time durations. The film samples were characterized using EXAFS. Peaks of Fourier transform of EXAFS could be observed at 1.4, 2.5 and 3.0 ^{0}A without phase factor correction for Fe-O, Fe-Fe and Ni-Ni bonds, respectively for sample fabricated at 900 rev/m for 2 min and annealed at 500 ^{0}C for 3 hours. Owing to unpaired electrons in outer shell of Fe and Ni atoms, Fe and Ni mixed thin films are prime candidates of magnetic devices.

Keywords: *EXAFS, spin coating, thin films, Ni, Fe*

1. Introduction:

Nickel oxide finds potential applications in temperature sensors, frits, ferrites, porcelain glazes, electrodes in supercapacitors, electrochromic films, gas sensors, catalyst, optical filters, enamels, Nickel-Iron batteries and NiCd rechargeable batteries. Nickel oxide (NiO) nanoparticulate films have been fabricated on nickel foils by electro-deposition method [1]. Nano-structured NiO thin films have been synthesized on corning glass and single crystal silicon substrates using spray pyrolysis technique [2]. The microstructure properties of NiO films prepared using electron beam evaporation method have been investigated [3]. Optical properties of nanocrystalline NiO thin films deposited by sol-gel spin coated method have been studied [4]. Nanosized NiO_x films have been prepared using sol-gel method [5]. In addition, NiO based thin films have been grown using chemical bath and sputtering methods [6].

Iron oxide thin films are vastly applied in magnetic memory and microwave devices. Colored iron oxide thin films have been synthesized by Sol-gel technique [7]. Iron oxide thin films have been fabricated over fused quartz substrate using simple metal organic deposition from Fe-(III) acetylacetonate as the organic precursor [8]. 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 [9]. Also thin films of hematite have been fabricated using pulsed laser depositions [10]. Thin films of Iron oxide have been grown by both post-oxidation of pure Fe ultra-thin films and by evaporating Fe onto the MO substrates [11]. However, spin coating has been identified as a low cost method compared to other expensive techniques required some high tech sophisticated equipments and vacuum.

Because both Fe and Ni have magnetic properties due to their unpaired electrons, the compounds with Fe and Ni can be used in the applications required magnetic properties. One of the main compounds consists of Fe, Ni and O is nickel ferrite. Nickel ferrite films have been grown on

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polycrystalline Al_2O_3 substrates using rf sputtering [12]. In addition, nickel ferrite films have been synthesized on single crystal Al_2O_3 substrates using pulse laser deposition technique [13]. Nanostructured $NiFe_2O_4$ thin films have been fabricated on stainless steel and glass substrates using chemical deposition [14]. Bilayer thin films of $Ni_{80}Fe_{20}/Ni_xFe_{1-x}O$ have been investigated under zero field cooled conditions [15]. Polycrystalline nickel ferrite films have been synthesized on polycrystalline Al_2O_3 substrates using a pulsed laser deposition method with a higher deposition rate [16].

Previously, the EXAFS properties of spin coated iron oxide films were described by us [18]. In this manuscript, the properties of Fe/Ni oxide films characterized using EXAFS will be explained. Films were synthesized at different rpm for different spin coating times, and films were subsequently annealed in air at different temperatures for different time durations. Since any significant differences between films deposited at different conditions couldn't be found, the properties of one sample will be described in this manuscript.

2. Experimental:

11.527g of Citric acid, 1.24g of $NiCl_2$, 1.986g of $FeCl_3$,11.4ml of Di Ethylene Glycol (D.E.G) and 15ml of H_2O were used to prepare the solution. First the solution of Citric acid, $NiCl_2$, $FeCl_3$ and D.E.G was stirred in a beaker under constant temperature of 50 ^oC. After the Chemical was fully dissolved in the Solution, H_2O was added. Then the solution was stirred for 30 minutes under constant temperature of 50 ^oC. After the solution was cooled down to the room temperature, the solution was applied to an amorphous glass plate to make thin layers. 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 200-500 ^0C in air for 0-3 hours.

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:

EXAFS of the film coated at 900 rev/m for 2 min and annealed at 500 ^0C for 3 hours is given in figure 1. EXAFS measured at Ni K-edge is given here. The thickness of this film was about 2 μm. The absorption edge of Fe can be observed at 7118 eV. The XANES part of the same graph is shown in figure 2. Pre-edge is observed at 7114 eV for Fe_2O_3.

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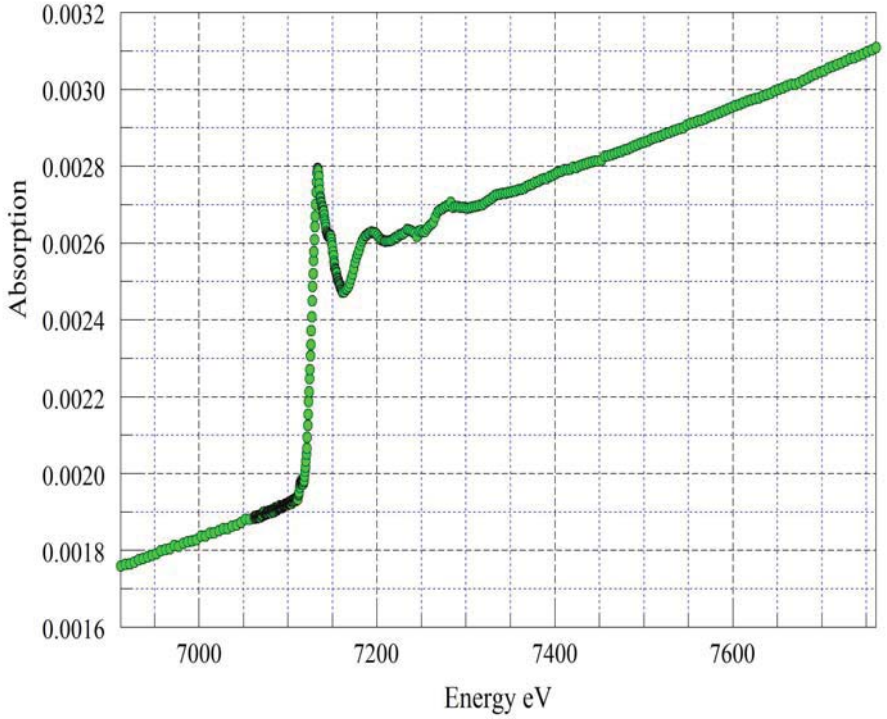

Figure 1: EXAFS of $Fe_xNi_yO_z$ film annealed at 500 ^{0}C for 3 hours.

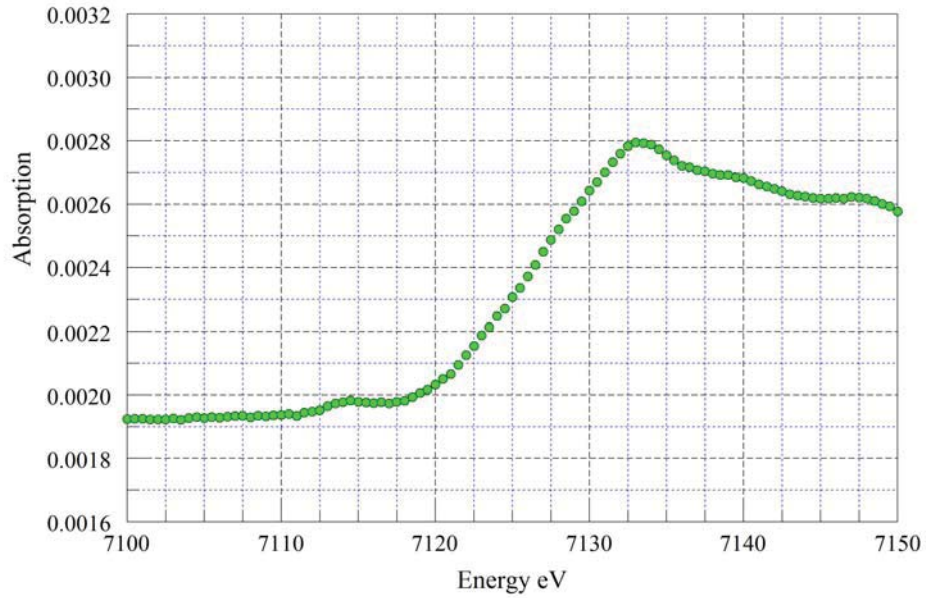

Figure 2: XANES part of the film given in figure 1.

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Figure 3 shows the Fourier transform of the same sample given in above two figures. The first peak is at 1.4 ^{0}A. This implies that the 1st shell bond length is 1.4 ^{0}A. If the phase factor is taken into account, this bond length varies in a range from 1.6 to 1.9 ^{0}A. Similarly the 2nd and 3rd peaks are at 2.5 and 3.0 ^{0}A, respectively. Here peaks at 1.4 and 2.5 ^{0}A correspond to Fe-O and Fe-Fe bonds, respectively. Peak at 3.0 ^{0}A is related to Ni-Ni bond length [17]. To determine the Fourier transform, following EXAFS equation was employed.

$$\chi(k) = \sum_j \frac{S_0^2 N_j f_j(k) e^{-2R_j / \lambda(k)} e^{-2k^2\sigma_j^2}}{kR_j^2} \sin[2kR_j + \delta_j(k)]$$

Here R_j is the variance in the absorber-scatter distance for path j, N_j is the coordination number for path j, λ is the mean-free path of the photoelectron,

f(k) is the scattering amplitude, δ(k) is the phase-shift, and σ^2 is the disorder in the neighbor distance. Furthermore, since these scattering factors depend on the Z of the neighboring atom, EXAFS is also sensitive to the atomic species of the neighboring atom. For multiple-scattering paths, the parameters are defined in analogous fashion; i.e. R_j is half of the total path length.

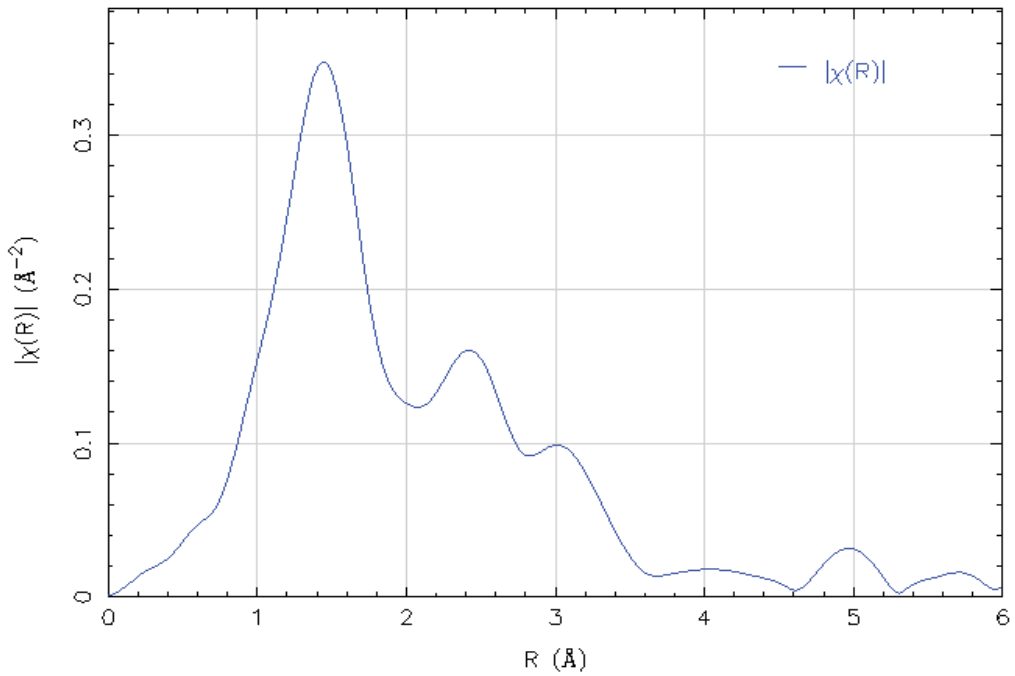

Figure 3: Fourier transform of the sample annealed at 500 ^{0}C for 3 hours.

XRD patterns didn't indicate any peaks by implying that the samples were not crystallized. All the fabricated samples were amorphous. Because EXAFS is capable to identify even uncrystallized phases, samples were characterized using EXAFS.

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4. Conclusion:

The bonds of $Fe_xNi_yO_z$ were identified using the Fourier expansion of EXAFS. All the data in this report have been given for the sample synthesized at 900 rev/m for 2 min and subsequently annealed at 500 ^{0}C for 3 hours in air. Solutions with $NiCl_2$ and $FeCl_3$ were applied on amorphous glass substrates to prepare spin coated $Fe_xNi_yO_z$ film samples. The peaks at 1.4, 2.5, 3.0 ^{0}A relate to Fe-O, Fe-Fe and Ni-Ni bonds, respectively. XANES pre-edge of Fe_2O_3 and absorption edge of Fe K-edge could be observed at 7114 and 7118 eV, respectively.

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