



Physical properties in thin films of iron oxides

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Abstract

We have grown hematite (α -Fe₂O₃) thin films on stainless steel substrates and magnetite (Fe₃O₄) thin films on (001)-Si single crystal substrates by a RF magnetron sputtering process. α -Fe₂O₃ thin films were grown in an Ar atmosphere at substrate temperatures around 400 °C, and Fe₃O₄ thin films in an Ar/O₂ reactive atmosphere at substrate temperatures around 500 °C. *Conversion electron Mössbauer (CEM)* spectra of α -Fe₂O₃ thin films exhibit values for hyperfine parameter characteristic of the hematite stoichiometric phase in the weak ferromagnetic state [R.E. Vandenberghe, in: *Mössbauer Spectroscopy and Applications in Geology*, University Gent, Belgium, 1990. [1]]. Furthermore, the relative line intensity ratio suggests that the magnetization vector of the polycrystalline film is aligned preferentially parallel to the surface. The *CEM* spectra of Fe₃O₄ thin films show the presence of only the stoichiometric phase, and the values for the hyperfine fields and isomer shifts of the A and B sites are consistent with bulk Fe₃O₄ [1]. The *X-ray diffraction (XRD)* pattern of the polycrystalline thin films also corresponds to α -Fe₂O₃ and Fe₃O₄ [JCPDS, X-ray diffraction data cards, 2001. [2]]. The samples were also analyzed by *atomic force microscopy (AFM)* and they reveal a grain morphology common for polycrystalline films. We found an average grain size of 211 nm and surface roughness of 45 nm in α -Fe₂O₃ films and an average grain size of 148 nm and surface roughness of 1.2 nm in Fe₃O₄ films.

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1. Introduction

Thin films of iron oxide are particularly appealing for experimental and theoretical investigations in view of their technological applications. Hematite (α -Fe₂O₃) is a thermodynamically stable oxide and it is a semiconducting material with an optical band gap around 2.0 eV. Between the Morin transition (250 K) and the Néel temperature (960 K) pure α -Fe₂O₃ is weakly ferromagnetic due to a slight cant in the alignment of the antiferromagnetic planes in its structure. Magnetite (Fe₃O₄) is a ferrimagnetic material and it has been considered as an important potential material for future spintronics. Fe₃O₄ forms in the cubic inverse spinel structure, in which one-third of the iron sites are tetrahedral and two-thirds are octahedral with formally Fe³⁺ and Fe²⁺ ions. Apart from the

magnetic properties, the other relevant phenomenon is the occurrence of a structural transition at around 120 K, which is called the Verwey transition. At this phase transition, a decrease of two orders of magnitude in the electrical conductivity occurs in such a way that at low temperatures magnetite is an insulator and above the Verwey transition temperature it is a metallic conductor.

We are investigating the most remarkable magnetic, structural and morphological properties of hematite and magnetite thin films produced by magnetron sputtering process to identify changes as a reference to bulk material. In order to understand which of their properties could play an important role in semiconducting and recording devices.

2. Experimental

Magnetic and structural properties of the thin films deposited by RF magnetron were studied by means of

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conversion electron Mössbauer (CEM) spectroscopy at room temperature using a home-made CEM chamber [3]. The X-ray diffraction (XRD) studies were performed using a Bruker *D-8* Advanced Diffractometer with Cu-K α monochromatic radiation and magnetic measurements were made using a Quantum Design extraction magnetometer. We carried out magnetization measurements as a function of temperature and magnetic field applied. The surface features and morphology of the films were examined using a scanning probe microscope by Park Scientific Instruments.

3. Discussion

The CEM spectrum of α -Fe₂O₃ sample was fitted using discrete distributions of hyperfine fields the one associated to α -Fe₂O₃ thin film and one singlet associated to the substrate signal corresponding to the paramagnetic fcc iron phase (γ -Fe) of stainless steel [4]. The CEM spectrum of the

Fe₃O₄ thin film was fitted with two sextets characteristic of the tetrahedral (A) and octahedral (B) sites of the magnetite in the ferrimagnetic state [5].

Fig. 1 shows the *M*-field curves of the magnetite thin films at temperatures of 300 and 110 K. We can see a considerable increase of the coercive field as soon as the temperature is decreased. The *M*-*T* curve is nearly constant above and below the Verwey transition temperature (Fig. 1 inset). Thus, there is just one phase transition without loss of ferrimagnetic behavior in the range from 5 K to room temperature, consistent with the characteristics of the magnetite system. The coercive field found in the curve performed at 300 K was 216 Oe and the remanence value was 3.9 memu. In contrast to the curve at 110 K we found a coercive field of 784 Oe, but the remanence value was 3.6 memu closely to the case above, therefore there are no sharp changes in the magnetic order on the sample, in spite of the distortion of structural phase due to the Verwey transition. Also the saturation magnetization values were similar to each other with 6.4 memu at 300 K and 5.9 memu at 110 K. The same measurements were made for a α -Fe₂O₃ thin film grown on a SiO₂ substrate at 230 K and we find a weakly ferromagnetic behavior of the sample without clear saturation and with a coercive field of 970 Oe. In the AFM image (Fig. 2) we can see a grain morphology common for a polycrystalline films, consistent with CEMS and XRD results, and we can see the cluster and granular formations.

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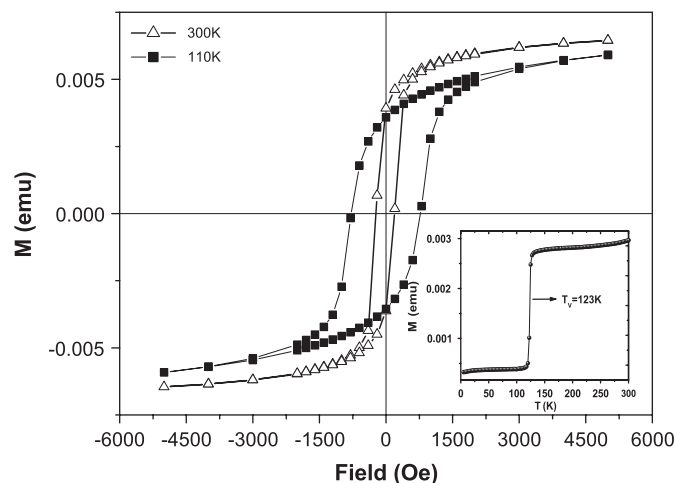


Fig. 1. Hysteresis loops of the Fe₃O₄ thin films at different temperature.

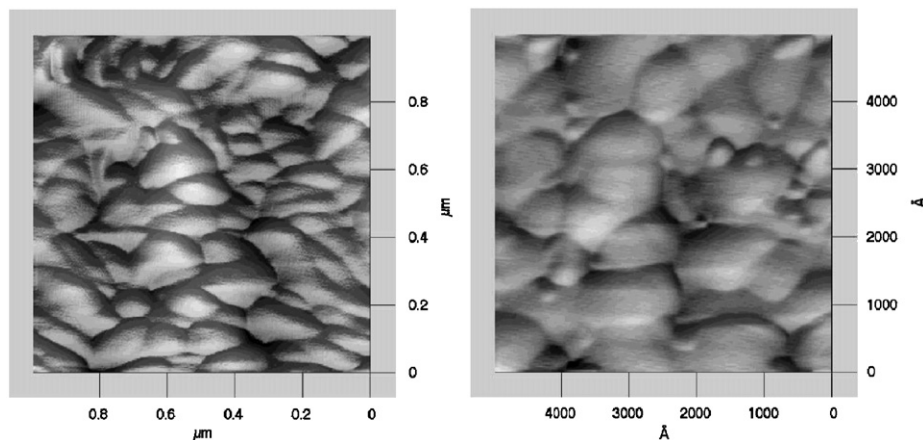


Fig. 2. AFM images of the α -Fe₂O₃ and Fe₃O₄ thin films.

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