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To cite this version:
C. Ortiz, T. Manoubi, C. Colliex. PHYSICAL PROPERTIES OF THIN FILMS OF IRON OXIDES. Journal de Physique Colloques, 1988, 49 (C8), pp.C8-2009-C8-2010. <10.1051/jphyscol:19888912>. <jpa-00229182>

HAL Id: jpa-00229182
https://hal.archives-ouvertes.fr/jpa-00229182
Submitted on 1 Jan 1988

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PHYSICAL PROPERTIES OF THIN FILMS OF IRON OXIDES

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Abstract. – This paper presents a comparative overview of the physical properties (structural, electronic, optical and magnetic) of thin films of the iron oxides: FeO, Fe3O4, α and γ – Fe2O3. Electron energy loss spectroscopy showed that the O-K edge and the L3/L2 lines are directly related to the iron valence.

This work presents a comparative study of four iron oxides in thin film configuration. The oxides FeO and Fe3O4 were deposited by reactive sputtering. γ – Fe2O3 has the same spinel structure as Fe3O4 and is obtained by further oxidation (annealing in air) of this compound. αFe2O3 is the most stable oxide and was prepared by direct oxidation of an Fe film.

The structural properties were studied with TEM. The grain size depends on the substrate temperature and oxygen flow during deposition [1]. Figures 1-3 show data for a 500 Å thick sample of Fe3O4 deposited with a substrate temperature of 300 °C and an O2/Ar flow ratio of 4.8/80 sccm. Figure 1 shows its TEM image with homogeneous distribution of grains with size ranging from 400-800 Å. The electron diffraction pattern corresponds to the well established inverse spinel structure where Fe3+ atoms sit on the tetrahedral A sites and the octahedral B sites have Fe2+ and Fe3+ atoms.

Electron energy loss spectroscopy has been performed with 1 eV energy resolution with a STEM microscope [2] on 10 nm² areas for the four oxides. The two characteristic ion edges (O-K at 532 eV and Fe L3,2 at 710 eV) contain very useful information:

1) similar to other transition metal oxides [3], the O-K edge (see Fig. 2a) shows a distinct prepeak. The latter increases its intensity and shifts its energy with respect to the threshold energy (532 eV) when going from Fe2+ to Fe3+. It reflects the local density of vacant mixed oxygen p and iron d orbitals on the oxygen

Fig. 1. – TEM image of a 500 Å thick Fe3O4 film. Its electron diffraction shows the spinel structure.

Fig. 2. – a) O-K edge, and b) Fe L3,2 white lines of same sample as in figure 1.
site. The shape of the Xanes type of oscillations is similar for \( \gamma \text{Fe}_2\text{O}_3 \) and \( \text{Fe}_3\text{O}_4 \) (same structure);

2) the \( L_3-L_2 \) white lines correspond to the excitation of \( 2p^{3/2} \) and \( 2p^{1/2} \) electrons towards vacant d states above the Fermi level. Our results show that their intensity ratio \( L_3/L_2 \) deviates noticeably from the degeneracy factor 2 and increases regularly from \( \text{FeO} \) (4.5) to \( \gamma - \text{Fe}_2\text{O}_3 \) (6.5). Figure 2b shows a characteristic \( \text{Fe} - L_{3,2} \) edge in \( \text{Fe}_3\text{O}_4 \).

The optical properties are most distinct for each oxide in the infrared (600 to 200 cm\(^{-1}\)), offering a good in situ survey method during the preparation of the films. Figure 3 shows the two infrared active modes at 565 and 350 cm\(^{-1}\) in \( \text{Fe}_3\text{O}_4 \). These peaks have proven to be very sensitive to the oxygen content of the sample.

Finally by VSM, we study the large variations in the magnetic moment for the two single magnetic spinel phases \( \text{Fe}_3\text{O}_4 \) and \( \gamma - \text{Fe}_2\text{O}_3 \). The highest magnetic moment we could obtain was 400 emu/cc, that is 80 \% of the bulk value. When we decreased the substrate temperature, the magnetic moment decreased to as low as 40 \% of the bulk value. Two possible mechanisms are discussed: the first involves A and B site mispopulation with supporting evidence from our Mössbauer spectroscopy measurements [4], and the second [5] is based on the local anisotropy detected by rotational hysteresis in torque magnetometry.

Acknowledgments

We would like to thank G. Castillo for helping with the sample preparation.