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Characterizing the phase purity of nanocrystalline Fe_3O_4 thin films using Verwey transition

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ABSTRACT

We have employed Verwey transition as a probe to check phase purity of nanocrystalline Fe_3O_4 films grown at different substrate temperatures (T_s) by means of magnetization study. The drop in magnetization at temperatures other than Verwey transition temperature T_v (120 K), in the low and high T_s films indicates the presence of antiferromagnetic (α-Fe_2O_3/FeO) impurity phases. After wet H_2 reduction treatment on these films, a vibrant appearance of Verwey transition is observed which confirms Fe_3O_4 phase at all T_s. However, high T_s films exhibit low T_v value with distribution, $T_v \pm \Delta T_v = 112 + 25$ K emanating from residual magnetic phases, which were not traced by XRD studies. Interestingly, these nanocrystalline Fe_3O_4 films exhibit anisotropic magnetic behaviors above T_v, similar to the single crystal Fe_3O_4. Below the saturation field, the easy (111) and relatively hard (110) axis of magnetizations align along their texture planes.

1. Introduction

Considering the recently demonstrated novel spintronic applications of nanosize Fe_3O_4 films [1–4], ranging from sensors based on the high magneto-resistance ratio change at Verwey transition [2] to the ultrafast switches wherein electric field driven Verwey transition is exploited [3–4], it is imperative to study Verwey transition of Fe_3O_4 films at nanoscale. The fingerprint of stoichiometric Fe_3O_4 single crystal is Verwey transition [5] at 120 K (T_v), wherein the resistivity abruptly increases by two orders of magnitude caused by structural transformation from high symmetric cubic phase to low symmetric monoclinic Fe_3O_4 phase. The consequence of this transition also influences the magnetic properties below the saturation magnetic field [5–9]. In the vicinity of Verwey transition at 130 K, the first-order magneto-crystalline anisotropy constant (K_1) of Fe_3O_4 also happens to change its sign [8–9]. However, the appearance of Verwey transition in the magnetic properties is more robust [10–12] compared to the electrical transport, wherein presence of smaller grain sizes, grain boundary volume and slight crystalline disorders/defects in nanocrystalline Fe_3O_4 could easily mask this transition [1–2,10–12]. Besides, the growth of Fe_3O_4 films by non-equilibrium processes like gas phase synthesis methods often produce many secondary insulating phases [α-Fe_2O_3 (antiferromagnetic), FeO (antiferromagnetic) and γ-Fe_2O_3 (ferrimagnetic)], drastically affecting the Verwey transition [13]. Therefore, growth of single phase Fe_3O_4 presents a significant challenge to researchers and hence alternative growth and characterization approaches need to be explored. Whilst x-ray diffraction and high resolution transmission electron microscopy have been the most powerful techniques to study bulk vs. interface properties in terms of their lattices, it is generally difficult to identify isostructural compounds (viz. Fe_3O_4, γ-Fe_2O_3 and FeO all three exhibit cubic structure) from these techniques.

In the present study, we employed Verwey transition as a novel way to check phase purity of nanocrystalline Fe_3O_4 films grown at various substrate temperature followed by wet H_2 reduction treatment through magnetic measurements.

2. Experimental

Thin films (~800 nm) were grown by pulsed laser ablation of α-Fe_2O_3 target on amorphous fused quartz substrates in vacuum of 1×10^{-5} mbar [14]. The substrate temperature (T_s) was varied from room temperature (RT) to 850 °C. After deposition, these films were reduced under wet H_2 environment in order to get single phase Fe_3O_4 as follows: the films were first introduced into

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