

Phase-Selective Deposition and Microstructure Control in Iron Oxide Films Obtained by Single-Source CVD

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Iron(III) tert-butoxide, $[\text{Fe}(\text{OtBu})_3]_2$, was used as a single source for iron and oxygen to obtain nanocrystalline hematite (Fe_2O_3) or magnetite (Fe_3O_4) films by low-pressure (LP) CVD. The decomposition profile of the molecular precursor and the crystallization temperature of iron oxide were derived from thermogravimetry/differential thermal analysis (TG/DTA). The substrate temperature was found to markedly influence the morphology and Fe/O stoichiometry in the deposited films. The morphological features and phase identification of the grown films were obtained by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. The compositional identity of the phases was determined by the X-ray photoelectron spectroscopy (XPS) of the CVD deposits. Annealing the films ex-situ under reducing or oxidizing conditions allows selective interconversion ($\text{Fe}_2\text{O}_3 \rightleftharpoons \text{Fe}_3\text{O}_4$) among the deposited phases with no particle size variation. The interplay between the rate of precursor delivery and substrate temperature controlled the mean particle size in the films. Magnetite film with a mean particle size of 10 nm was obtained on silicon at 450 °C. Formation of larger grains and grain clusters was observed at higher temperatures. High coercivity (4000 Oe) and small saturation magnetization (0.3 emu g⁻¹) of the Fe_3O_4 film confirmed superparamagnetic behavior due to small particle size. Absorption spectra of magnetite and hematite films deposited on glass show them to be transparent to the visible light. The sheet resistance of nanocrystalline Fe_3O_4 and Fe_2O_3 films was found to be 2.4 k and 2 M, respectively.