Materials Science & Engineering Doctoral Defense

Microstructure development in magnetite films via non-classical crystallization

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abstract

Polycrystalline magnetite thin films were deposited on large area polymer substrates using aqueous solution based spin-spray deposition (SSD). This technique involved the hydrolysis of precursor salt solutions at low temperatures (70-100°C). The fundamental mechanisms and pathways in crystallization and evolution of the film microstructures were studied as a function of reactant chemistry and reactor conditions (rotation rate, flow rates etc.). A key feature of this method was the ability to constantly supply fresh solutions throughout deposition. Solution flow due to substrate rotation ensured that reactant depleted solutions were spun off. This imparted a limited volume, near two-dimensional restriction on the growth process. Film microstructure was studied as a function of process parameters such as liquid flow rate, nebulizer configuration, platen rotation rate and solution chemistry. It was found that operating in the micro-droplet regime of deposition was a crucial factor in controlling the microstructure.Film porosity and substrate adhesion were linked to the deposition rate, which in-turn depended on solution chemistry. Films exhibited a wide variety of hierarchically organized microstructures often spanning length scales from tens-of-nanometers to a few microns. These included anisotropic morphologies such as nanoplates and nanoblades, that were generally unexpected from magnetite (a high symmetry cubic solid). Time resolved studies showed that the reason for complex hierarchy in microstructure was the crystallization via non-classical pathways. SSD of magnetite films involved formation of precursor phases that subsequently underwent solid-state transformations and nanoparticle self-assembly. These precursor phases were identified and possible reaction mechanisms for the formation of magnetite were proposed. A qualitative description of the driving forces for self-assembly was presented.

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