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Structural Analysis of Phase Transformation and Particle Formation in Metastable Metallic Thin Films Grown by Plasma-Enhanced Atomic Layer Deposition

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Abstract : Growth of conformal ultrathin metal films has attracted a considerable amount of attention recently. Plasma-enhanced atomic layer deposition (PEALD) is a method capable of growing conformal thin films at low temperatures, with an exemplary control over thickness. The authors have recently reported on growth of metastable epitaxial nickel thin films via PEALD, along with a comprehensive characterization of the films and a study on the relationship between the growth parameters and the film characteristics. The goal of the current study is to use the mentioned films as a case study to investigate the temperature-activated phase transformation and agglomeration in ultrathin metallic films. For this purpose, metastable hexagonal nickel thin films were annealed using a controlled heating/cooling apparatus. The transformations in the crystal structure were observed via in-situ synchrotron x-ray diffraction. The samples were annealed to various temperatures in the range of 400-1100° C. The onset and progression of particle formation were studied in-situ via laser measurements. In addition, a four-point probe measurement tool was used to record the changes in the resistivity of the films, which is affected by phase transformation, as well as roughening and agglomeration. Thin films annealed at various temperature steps were then studied via atomic force microscopy, scanning electron microscopy and high-resolution transmission electron microscopy, in order to get a better understanding of the correlated mechanisms, through which phase transformation and particle formation occur. The results indicate that the onset of hcp-to-bcc transformation is at 400°C, while particle formations commences at 590° C. If the annealed films are quenched after transformation, but prior to agglomeration, they show a noticeable drop in resistivity. This can be attributed to the fact that the hcp films are grown epitaxially, and are under severe tensile strain, and annealing leads to relaxation of the mismatch strain. In general, the results shed light on the nature of structural transformation in nickel thin films, as well as metallic thin films, in general.

Keywords : atomic layer deposition, metastable, nickel, phase transformation, thin film
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