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Thermal stability of nanoparticle thin films characterized by x-ray scattering methods

Tereza Košutová, Lukáš Horák, Jan Hanuš, Ondřej Kylián, Andrei Choukourov, Miroslav Cieslar, Hynek Biederman, Milan Dopita

Charles University, Prague, Czech Republic

kosutovat@gmail.com

Thin films composed of nanoparticles are interesting because of their inherent high porosity which is important for gas sensing, efficient batteries or hydrogen storage applications. The thermal stability of nanoparticle layers depends on chemical and phase composition, configuration of nanoparticles or the atmosphere in which they are heated up.
In this study we investigated systems of homogeneous and heterogeneous metallic nanoparticles, and also thin layers containing two different types of homogeneous nanoparticles. Nanoparticles were in all cases prepared by magnetron sputtering from pure metal targets followed by aggregation of fragments to the metallic clusters. Samples deposited on silicon substrates were characterized at the ambient air atmosphere after preparation. Size distribution of nanoparticles and the inert structure of heterogeneous nanoparticles were determined by small angle x-ray scattering (SAXS) and confirmed by transmission and scanning electron microscopies measurements (SEM, TEM). Thickness of the layers, analysed by cross-section SEM and atomic force microscopy, was used to estimate the layer porosity from absolute intensity SAXS measurements.
Microstructure of nanoparticles and its thermal evolution were studied by in-situ x-ray diffraction (XRD) experiments realized in various atmospheres (vacuum, air, argon or nitrogen atmosphere). Evolution of phase composition, lattice parameters, sizes of crystallites, and microstructural defects in present crystalline phases were described in details up to 900 ° C. In-situ SAXS measurements were done to examine the temperature dependence of the layer morphology as well as the size distribution of single nanoparticles. Electron microscopy was performed to verify the models used for fitting of the SAXS scattering curves.

Keywords
nanoparticles
SAXS
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thermal stability