HRTEM and EELS studies on the oxidation of nickel nanoparticles.

S. Stappert [(1)], H. Sauer [(2)], B. Rellinghaus [(1)] and E. F. Wassermann [(1)]

[(1)] Exp.Tieftemperaturphysik, Gerhard-Mercator-Universität, D-47048 Duisburg

[(2)] Fritz-Haber-Institut der Max-Planck-Gesellschaft, D-14195 Berlin

Nickel nanoparticles are prepared by means of DC sputtering in argon. Size and morphology of the particles are modified by means of subsequent sintering of the primary particles in the gas phase at different temperatures. This technique allows for the preparation of particularly clean particles with sizes ranging from 3 nm for primary particles to some 50 nm for agglomerate particles. After their deposition onto holey carbon films the nickel nanoparticles are oxidized in ambient air. The nature of the oxidation process is studied ex-situ utilizing high resolution transmission electron microscopy (HRTEM) combined with electron energy loss spectroscopy (EELS). Electron diffraction experiments show the occurrance of small amounts of crystalline phases of NiO in samples with primary particles of 3 nm in size. HRTEM investigations on individual particles, which have been subjected to the sintering process, reveal a core-shell type structure of metallic nickel surrounded by a nickel oxide surface layer. These surface layers are found to be predominantly amorphous. On deposited particles, which are exposed to the electron beam for a sufficient amount of time, we observe a successive re-crystallization of the previously amorphous surface layer. The formed surface crystallites are unambiguously identified as NiO. The influence of the particle morphology and thereby the influence of the type of facettes that are exposed to the air on the details of the oxidation process is discussed.