Nanocomposites and nanomaterials

Relationships Between Pore Structure and Free Carbon Content in Ni/NiO Nanopowders

I.O. Dulina, T.F. Lobunets, A.V. Ragulya

Department of Physical Chemistry and Technology of Nanostructured Ceramics and Nanocomposites, Frantsevich Institute for Problems of Materials Science, Natl. Acad. of Sci. of Ukraine. Krzhyzhanovsky St., 3, Kyiv-03142, Ukraine. E-mail: i_risha@online.ua

Nickel powders are widely used as electrode materials in multilayered ceramic capacitors. Tendency of ceramic and electrode layers thinning to 100 - 200 nm that is used for increasing of capacitor dielectric capacity leads to necessity of powders size decreasing to 10 - 20 nm. Thus, development of technology of Ni nanopowders obtaining with particle size of 20 nm and less and minimal impurities content is of great importance.

Ni/NiO nanopowders have been prepared by using thermal decomposition of aqua solutions of nickel acetate ammine complexes in air at the annealing temperature range 300 - 500 °C, time of decomposition from 30 to 180 min and ammonia content in initial complex 3.6 - 9.55 mol/mol Ni²⁺.

At 300 and 325 °C indicates that these products had amorphous nonporous structure. At temperature 300 °C nonporous structure had molecular apertures on surface and product was represented as solid flakes. Increasing of annealing temperature to 325 °C led to formation of solid nonporous amorphous particles with mean size 37 - 38 nm and flakes became distract in separate agglomerates with minimal particle size 30 - 50 nm. Process of decomposition of intermediate amorphous structure with formation of Ni and NiO crystalline particles was accompanied with appearance of pores with mean size 3 - 4 nm was observed at 350 °C. Powder at this temperature is characterized by agglomerates with mean size 30 - 40 nm and mainly monomodal particle size distribution. The linkage of isotherms adsorption-desorption legs that was observed for powders obtained at 400 and 500 °C shows formation of mixture of two phases in powder. In accordance with SEM, this process is accompanied with rising of nanoparticles agglomerates size. At 400 °C mean agglomerate size increased to 100 nm. Increasing of mean pore size at annealing temperature 500 decomposition °C was corresponded with growing of powder particles and nanoparticles sintered in solid aggregates. Decreasing of specific surface area of powders led to increasing of free carbon content because of inhibition of acetic acid evaporation because of growing of powder particle size.