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Synthesis of hollow nanostructured nickel oxide microspheres by ultrasonic spray atomization

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ABSTRACT

NiO nanostructured powders were synthesized by an ultrasonic spray pyrolysis (USP) technique. The powders were characterized using low temperature adsorption (BET), X-ray diffraction, scanning electron microscopy (SEM), and a transmission electron microscopy (TEM). The final product of the ultrasonic spray pyrolysis is hollow microspheres with 5–7 µm in diameter. The microspheres are built from nanoparticles with 40–60 nm in diameter.

1. Introduction

The study describes structural characteristics of a nanostructured NiO powder obtained by ultrasonic spray pyrolysis and the temperature influence on the structural parameters of the final product. Modern scientific methods allow obtaining nanostructured materials. Some methods provide quantity (mass, volume) of materials, whereas others provide quality (dispersion, particle size distribution, and agglomeration). Requirements for methods of obtaining nanopowders and nanostructured materials can be divided on controlling phase composition, productivity, economy, and dispersity control. The aim of the present study is to combine these requirements. Spray pyrolysis can be such a method (Manivasakan, Arumugam, & Venkatachalam, 2013). This method is based on thermal drying and thermal decomposition of an aerosol droplet of the initial salt solution (Janackovid et al., 1998; Patil et al., 2011; Sheng, Xuebin, Longying, Lijin, & Ruo, 2015). Aerosol droplet formation provides ultrasonic, pneumatic, electrostatic, hydraulic, or mechanic atomizers (Boukmouche et al., 2014; Freitas et al., 2004; Girtan, Cachet, & Rusua, 2003; Gong & Fu, 2007; Hede et al., 2008; Huang et al., 2015; Ksapabutr et al., 2015; Marchant and Green, 1982; Tratnig et al., 2009; Xie et al., 2014; Yuan, 2006). The most popular method of atomization is an ultrasonic technique that yields high productivity and high dispersity of aerosol droplets as compared to some other methods, e.g., pneumatic, electrostatic, hydraulic, and mechanic. Table 1 shows the comparative characteristics of these methods.

Table 1 shows, that electrostatic and ultrasonic atomization methods provide a lower droplet diameter as compared to mechanic, pneumatic, and hydraulic methods. The electrostatic method is useful only for obtaining films on a substrate. The ultrasonic method provides droplets with a diameter of 0.5–10 µm. The hydraulic, pneumatic, and mechanic methods make

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Table 1
Atomization characteristics of different types of atomizers and the comparison of such atomization characteristic as an aerosol droplet diameter, the rate of atomization, and aerosol flow rate for hydraulic, pneumatic, mechanic, electrostatic, and ultrasonic atomizers.

Atomization method	Diameter of the initial droplet, μm	Rate of atomization, cm^3/min	Aerosol flow rate, m/s
Hydraulic	50–350	7–10	150–300
Pneumatic	100–200	> 3	50–300
Mechanic	8–150	5–10	60–100
Electrostatic	0.1–10	30–50	1–5
Ultrasonic	0.5–10	< 2	0.2–0.4

a higher rate of atomization possible; however, due to a large diameter of the initial droplets, which have relatively large mass and are under the influence of the gravity force, the droplets settle down to the initial solution. Ultrasonic atomizers produce droplets with a comparatively sharp droplet size distribution.

Spray pyrolysis is a process of solution atomization that heats droplets to obtain solid particles. Both inorganic and organic salts are usually used as a precursor for an atomization process. A spray pyrolysis technique allows obtaining thin nanostructured films and powders with a nanostructure. The first step is atomization of the initial salt solution. Next step, the aerosol droplets move to the heated reactor by a carrier gas. As a carrier gas, we use air, nitrogen, or any rare gas. In the reactor, a solvent evaporates and forms a precursive structure. Solid particles heated in the reactor form the final structure of the material (Che et al., 1998; Cho et al., 1997). Such parameters as the temperature in the reactor, the concentration of salt in the initial solution, the chemical composition, the frequency of the ultrasonic generator, the flow rate of the carrier gas in the reactor, and the atmosphere in the reactor available, govern the structure, morphology, dispersity, and the phase composition of the final product (Che et al., 1998).

This study illustrates the influence of temperature in the reaction zone on structural characteristics of a nanostructured NiO powder. Materials with a nanostructure as well as nanopowders have unique properties, including electrical, magnetic, mechanical, physical, chemical, thermal, and optical. Nanomaterials increase the efficiency factor as well as the running time; in addition, they add specific properties to final materials due to specific surface area of nanopowders and nanostructured materials and quantum-dimensional effects (Martín et al., 2010; Rujonkov et al., 2003; Singh et al., in press). Therefore, it is important to develop a method to synthesize nanostructured materials and nanopowders that can control structural parameters of such materials.

In fact, a spray pyrolysis method can control parameters such as temperature, concentration, and combination of initial precursors, a carrier gas flow rate that influence on the structure, morphology, and chemical composition of the final product. In the paper (Nandiyanto & Okuyama, 2011), the authors show the observation of a spray pyrolysis technique. Atomization, droplet-to-particle conversion, and parameters for particle collection were described in the paper. Variation of the morphology and structure evolution clearly described in (Nandiyanto & Okuyama, 2011). In addition, the paper described the processes of influence on the morphology of a final product. The role of temperature in the reaction zone, carrier gas, gas flow rate, etc. in the particle formation were also presented. The influence of the concentration of the initial precursor solution on the morphology and the structure of the final product were observed in the paper. The most common case is the formation of hollow sphere particles due to shape of the initial aerosol droplet. The high-evaporation rate and high-mass transfer provide formation of hollow microspheres.

The spray drying method makes it possible to obtain the BN/CNT composites. Nandiyanto et al. (2009) obtained a BN/CNT nanotube with ultrasonic atomization of the suspension hBN nanoparticles and Ferrocene solvate in ethanol. The BN/CNT composites were obtained during evaporation of the solvent and catalyst activation in the reactor at 800 °C to obtain hirsute morphology of the particles in the final product.

In addition, the spray pyrolysis method can be interesting in the process porous structure synthesis (Nandiyanto et al., 2010). The formation of a silica porous structure with ultrasonic spray pyrolysis shown in the research of Asep Bayu Dani Nandiyanto, Nobuhiro Hagura and Ferry Iskandar. Both inorganic silica particles and organic polystyrene templates were used to synthesize porous microspheres. An aerosol forms due to ultrasonic cavitation. The aerosol moves to the furnace by the N_2 carrier gas. In the fixed temperature zones 200 °C aerosol droplets get dried from the solvent and at 500 °C the particles get decomposed from polystyrene. The obtained powder is collected on the filter. The final product is porous silica particles.

A spray pyrolysis method can be used not only to obtain nonorganic materials (Iskandar et al., 2009). The formation of hyaluronic acid porous particles with controllable porosity can be achieved with a spray-drying method. A combination of a hyaluronic powder and polystyrene particles was used as a precursor. An aqueous solution of hyaluronic acid and polystyrene were atomized with pneumatic atomization. A spray was dried at temperature 120 °C. Polystyrene was dissolved with an organic solvent. As a result, Iskandar, Nandiyanto and Widiyastuti obtain hyaluronic acid porous particles with a controllable porous structure.

Nanopowders are widely used in powder metallurgy owing to their specific thermal properties, such as melting temperature, which is lower than in a bulk condition. This fact is one of the reasons for using nanopowders as a precursor in powder metallurgy for various consolidation processes. In other cases, metal nanopowders can be compacted without a heat treatment. Powder metallurgy is a consolidation process of bulk materials, with a nanopowder sintering into a bulk material (Rujonkov et al., 2003).