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Synthesis of Ni/NiO Nanopowder by Thermal Decomposition of Nickel Acetate Amine

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Ni/NiO nanopowders have been synthesized using thermal decomposition of nickel acetate hexaammine in air. Obtained powders have been characterized by IR-spectroscopy, XRD and TG, DTA, DTG and HR TEM. Thermal decomposition of nickel ammine complexes occurs with forming nickel hydroxide, carbonate and hydroxocarbonate ammines precursors. Mean particle size of nickel and nickel oxide phases in powders depends on temperature. In the temperature range from 350 to 500 °C the particle size of nickel oxide has grown from 5 to 25 nm and nickel from 50 to 55 nm. Particle size of 5 nm for nickel hydroxide ammine remained unchanged with temperature.

Keywords: Nickel ammine complexes, Nanopowders, IR-spectroscopy, XRD Analysis, TG, DTA, DTG.

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1. INTRODUCTION

Nowadays, nickel powders are widely used as materials for manufacturing electrodes for multilayered ceramic capacitors instead of expensive powders of silver, palladium and platinum. The miniaturization tendency of electronic devices leads to necessity of downsizing multilayered ceramic capacitors simultaneously with increasing their capacity. The capacity of multilayered ceramic capacitors can be increased by decreasing thickness of ceramic and electrode layers and increasing number of electrodes. Owing to this, development of nickel nanopowder synthesis methods has a large importance.

2. MATHERIALS AND METHODS

Ni/NiO nanopowders have been synthesized using thermal decomposition of nickel acetate hexaammine in air in the temperature range 300-500°C.

Chemical, phase composition and decomposition completeness of products have been identified by IR-spectroscopy, XRD and TG, DTA and DTG, respectively. Mean particle size of obtained powders has been identified by HR TEM.

3. RESULTS AND DISCATION

Full decomposition of nickel ammine complexes takes place at temperature higher than 375°C and decomposition products represent mixture of Ni and NiO crystalline phases adsorbed water and carbonate (Fig. 1-3).

Temperature 300°C is insufficient for forming nickel and nickel oxide phases and decomposition product represent mixture of crystalline nickel hydroxide ammine and amorphous nickel carbonate and hydroxocarbonate ammines. At temperatures 325-350°C part of nickel-containing precursors form Ni and NiO. Due to TG, DTA and DTG, formation of metal nickel phase can occur with thermal decomposition of nickel hydroxide ammine precursor only. Thermal decomposition of nickel carbonate and hydroxocarbonate ammines leads to forming NiO. In addition, increasing decomposition temperature to 325 - 350°C leads to decreasing full carbonate content in precursors and forming of nickel carbonate ammine precursor is not occur.

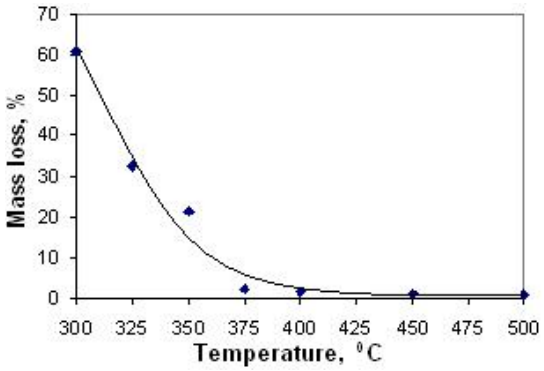

Fig. 1 – Mass loss vs. temperature for nickel acetate hexaammine decomposition products

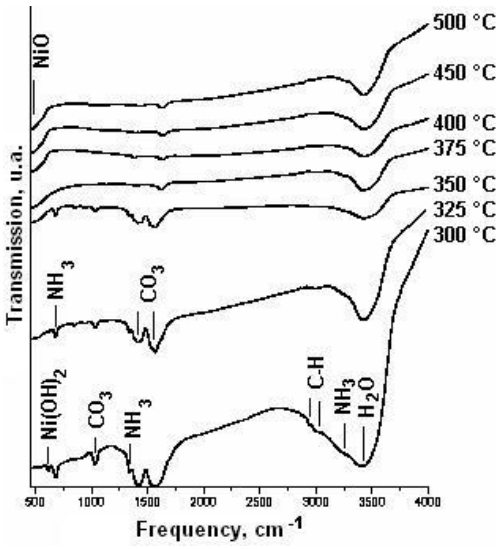

Fig. 2 – IR-spectra of nickel acetate hexaammine decomposition products obtained at different temperatures

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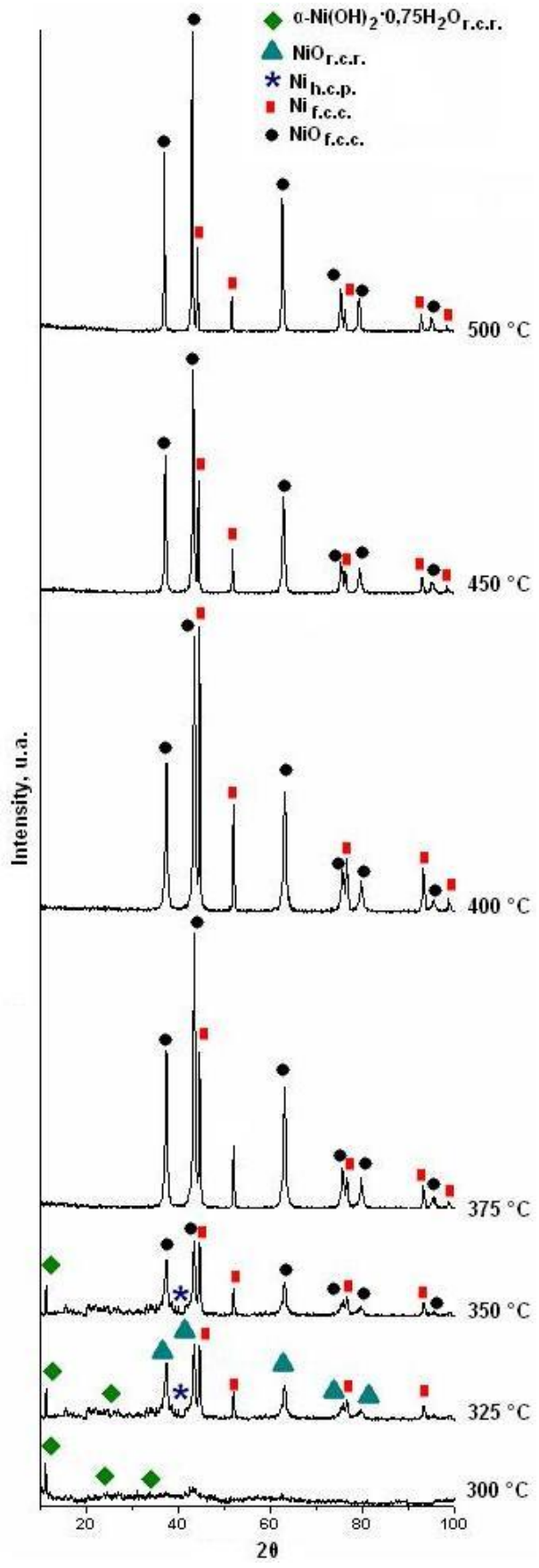
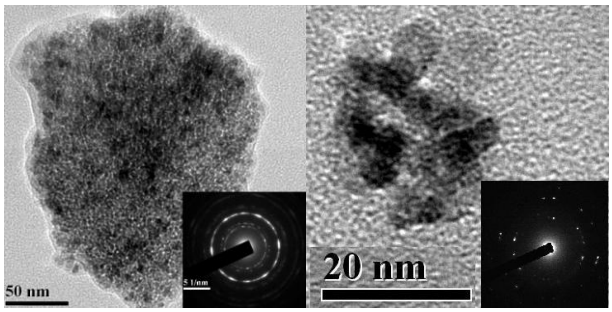


Fig. 3 – IR-spectra of nickel acetate hexaammine decomposition products obtained at different temperatures

Increasing decomposition temperature leads to increasing Ni content in powder and the largest content of nickel metal phase in powder is observed at temperature 400 °C. At temperature above 400 °C, the metal nickel content decreased in powder due to nickel oxidation in air.

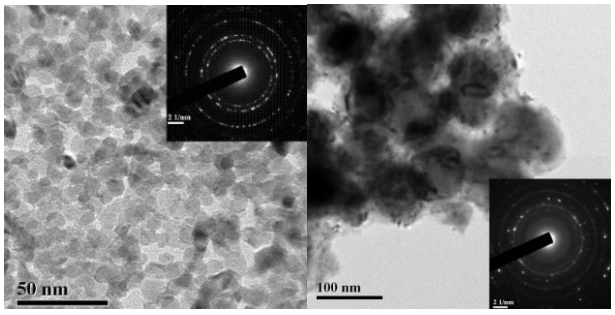
Results of HR TEM (Fig. 4-6) show that nickel hydroxide amine precursor contains in decomposition products of nickel acetate hexamine obtained at temperatures 350 and 400 °C. It is indicates that time 30 min is insufficient for full complex decomposition at temperature 400 °C. Mean particle size of nickel hydroxide ammine 5 nm remained unchanged with temperature.

Mean particle size of nickel and nickel oxide depends on temperature. In the temperature range from 350 to 500 C the particle size of nickel oxide has grown from 5 to 20-25 nm and nickel from 40-60 to 40-70 nm.



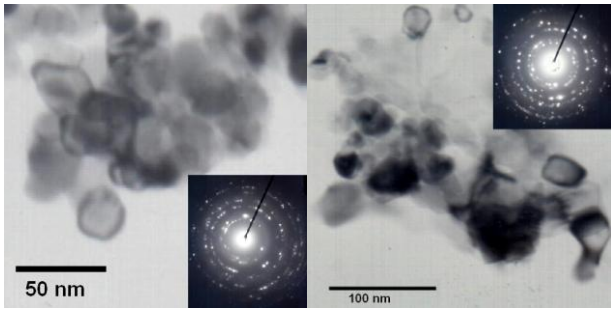
a b

Fig. 4 – HR TEM micrographs of powder obtained at 350°C: a – nickel hydroxide amine precursor; b – NiO



a b

Fig. 5 – HR TEM micrographs of powder obtained at 400 °C: a – NiO; b – Ni



a b

Fig. 6 – HR TEM micrographs of powder obtained at 500 °C: a – NiO; b – Ni

4. CONCLUSIONS

1. Thermal decomposition of nickel ammine complexes occurs with forming nickel hydroxide, carbonate and hydroxocarbonate ammines precursors. Composition of the precursors depends on temperature.

2. Formation of metal nickel phase can occur with thermal decomposition of nickel hydroxide ammine only.

3. Mean particle size of nickel and nickel oxide depends on temperature. In the temperature range from 350 to 500 °C the particle size of nickel oxide has grown from 5 to 20-25 nm and nickel from 40 - 60 to 40 - 70 nm. Particle size of 5 nm for nickel hydroxide ammine remained unchanged with temperature.

4. Temperature 400 °C is considered the optimal one for thermal decomposition of nickel acetate hexaammine to obtaining Ni/NiO nanopowder.