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# SYNTHESIS AND PROPERTIES OF Ni/C NANOCOMPOSITES BASED ON SYNTHETIC HUMIC SUBSTANCES

Metal nanoparticles encapsulated in carbon are interest in various fields of science and technology. On the one hand, it is known that nanosized metal particles are highly active in many catalytic reactions, but they are often unstable and easily aggregation and oxidation. The carbon shell of nanocomposites can maintain the size and physicochemical properties of metastable nanocrystalline materials for a long time. On the other hand, the carbon coating has unique structural, adsorption, electronic, mechanical and thermal properties [1].

The study of features of the formation, the structural characteristics and functional properties of metal-carbon nanocomposites depending on the preparation conditions is an important task, the solution of which opens up the possibility of controlling the structure of nanocomposites and their properties.

The aim of this work was to develop a technology for the synthesis of Ni/C nanocomposite using synthetic fulvic acids as a source of amorphous carbon.

The use of carbon-containing precursors on the basis of synthetic analogues of humic substances is due to the absence in their composition of the ordered structural fragments, from which the nanocrystal of carbon atoms about could by obtains in the conditions of pyrolysis. At full stochastic structure the humic substances are characterized the well-defined indicators content functional groups. Presence in the structure of synthetic fulvic acids the carboxy-groups, quinoid fragments and phenolic hydroxy-groups determined their ability to the formation salt and complexes with polyvalent cations of transition and non-transition metals.

For the synthesis of Ni/C nanocomposite, a solution of synthetic fulvic acids was obtained by the method described in ref. [2]. A certain volume of fulvic acid solution was neutralized by alkali to pH = 11 and a solution of $Ni(NO_3)_2$ was added to the resulting sodium fulvate solution. The precipitate was washed by decantation and dried at 120 ºC. The obtained nickel(II) fulvate powder was analyzed for nickel content spectrophotometrically by wet ashing with nitric acid or a mixture of nitric acid and potassium nitrate. The obtained mineralizate was adjusted with distilled water to a volume of 25 ml and the optical density of the solution was measured at 395 nm. Pyrolysis of dried nickel(II) fulvate was performed in a hydrogen atmosphere. In each of the experiments, the weight loss of the original nickel(II) fulvate was determined by weighing the crucible with the substance before and after pyrolysis with an accuracy of 0.0001 g.

Decomposition of nickel(II) fulvate in a hydrogen atmosphere begins at 240 ºC and accompanied by the release of a number of liquid and gaseous products. The dependence of the decrease in mass on temperature at a fixed exposure time of the sample (10 min) at nominal temperature is shown in Figure. 1.

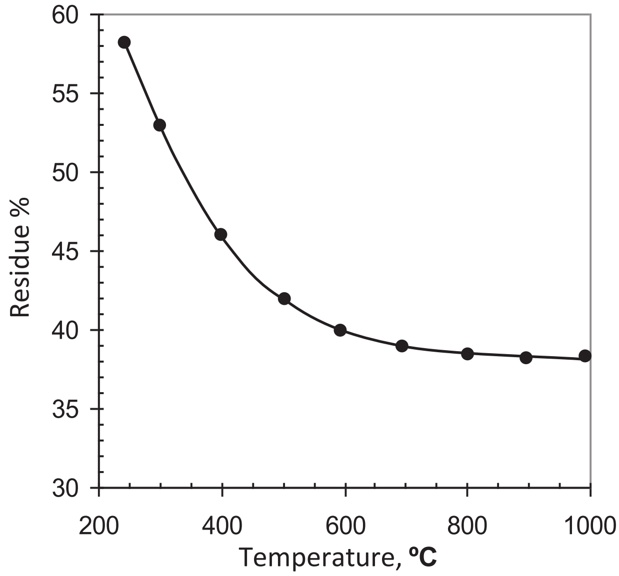


Figure 1 - Dependence of the weight loss of nickel(II) fulvate on the synthesis temperature

Analysis of the obtained curve shows that the removal of volatile decomposition products is almost complete in the temperature range of 900-1000 °C. The nickel content increases from 19% in the original nickel (II) fulvate to 41.4% in the final nickel-carbon composite.

The sizes of nickel nanoparticles were determined by the expansion of lines on X-ray diffraction patterns (Figure. 2). It has been established that the average particle size in the pyrolysis temperature range of 300 – 1000 °C varies from 9 to 52 nm.

The composite obtained at 300 ºC has no magnetic properties; becouse the size of the nanoparticle is smaller than the size of the magnetic domain in metallic nickel. Powders of nanocomposites synthesized at higher temperatures are attracted to a permanent magnet.

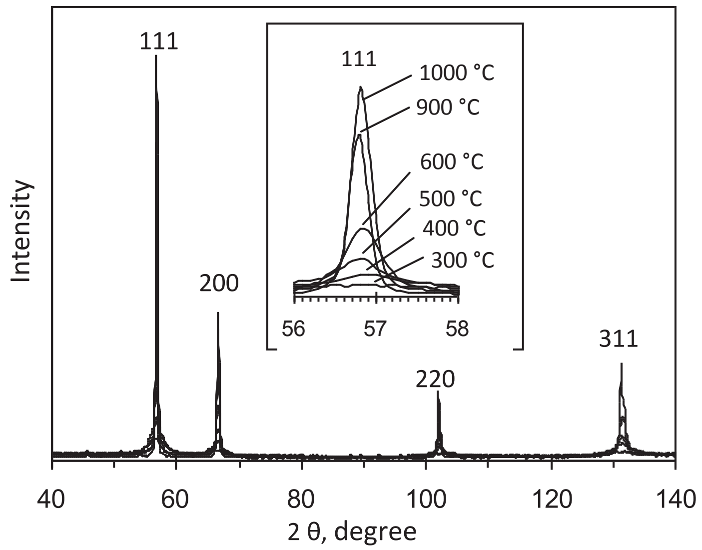


Figure 2 - X-ray diffraction of Ni/C nanocomposite for different temperatures of synthesis

On the diffractograms of all nanocomposites there is a very weak halo in the region of angles 20-30º, which characteristic of carbon materials with a certain degree of amorphization. There are also no reflexes characteristics of crystalline forms of Carbon. However, weak reflexes of the graphite-like phase are already recorded on the diffractograms for temperatures of 900 and 1000 ºC. These facts, in our opinion, indicate that the carbon matrix is amorphous.

The dependence of the electrical conductivity of composites on the pyrolysis temperature was also monitored at a qualitative level. As the temperature increased, the electrical conductivity varied from a very small value characteristic of dielectric powders to values that are characteristic of metal powders.

## REFERENCES


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