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THERMAL-CATALYTIC SYNTHESIS OF CARBON NANOTUBES IN A REACTOR WITH FLUIDIZED BED OF CATALYST

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Introduction

Currently, many research centers are working on the synthesis of carbon nanotubes from all possible carbon-containing materials using various catalysts. But despite this, the development of new methods and the search for optimal conditions for the synthesis of carbon nanotubes with low cost and good quality is the current direction of nanotechnology.

One of the most well-known and effective methods for producing carbon nanotubes is chemical vapor deposition (CVD). This method is widely used for the production of carbon nanotubes and provides high performance and the required quality of the material obtained with a comparative simplicity of the hardware design and availability of the original components.

Materials and Methods

In work, Al_2O_3 spheres with a diameter of 0.5-1 mm and having the following composition Al_2O_3 - 93.6 %, SiO_2 - 0.06 %, Fe_2O_3 - 0.18 %, Na_2O - 0.45 % were used as a matrix for the preparation of catalysts. The Ni/NiO mixture was applied to the surface of Al_2O_3 spheres by the method of solution combustion synthesis. For this, the Al_2O_3 spheres were impregnated with an aqueous solution of a mixture of nickel nitrate and citric acid ($C_6H_8O_7$). After impregnation of Al_2O_3, the spheres were subjected to a drying process until the solvent was completely removed and subjected to heat treatment at a temperature of 260 °C in a nitrogen atmosphere. The synthesis of CNTs was performed in a vertical CVD reactor. The synthesis was carried out from acetylene at a rate of 95 cm^3/min, in a nitrogen atmosphere at a rate of 948 cm^3/min. The synthesis temperature is 780 ºC, the heating rate is 25 ºC/min, the synthesis time is 30 min.

Results and Discussion

During the interaction of nickel nitrate ($Ni(NO_3)_2$) and citric acid ($C_6H_8O_7$) under the influence of high temperature, nanoparticles consisting of 85 % pure nickel (Ni) and 15 % nickel oxide (NiO) are formed on the surface of Al_2O_3 spheres. The specific surface is 12.64 m^2/g. Particle sizes are 80-300 nm. Thus, based on the results of XRD-analysis, a product consisting of 85 % Ni and 15 % NiO is formed during the interaction of nickel nitrate and citric acid (Figure 1).

Figure 2 shows the SEM image and the RAMAN spectra of carbon nanotubes synthesized on Al_2O_3 spheres with deposited Ni/NiO nanoparticles. The spectrum of this sample has the following peaks, a D-peak at a wavelength of 1432 cm^{-1}, a G-peak at a wavelength of 1595 cm^{-1}. For this sample of CNTs, the peak intensity ratio I_D/I_G is 0.88. This ratio indicates a high defectiveness of the obtained carbon nanotubes and the presence of an amorphous phase.

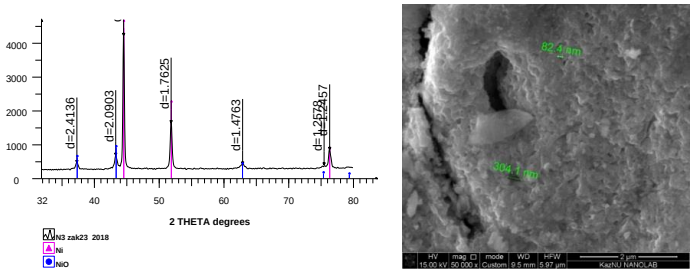

Figure 1 – XRD-pattern and SEM image of a Ni/NiO sample

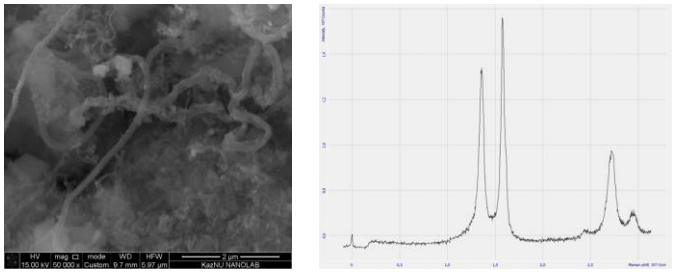

Figure 2 - SEM image and Raman spectra of carbon nanotubes at different scales synthesized on Ni/NiO@sp-Al_2O_3

Conclusions

Thus, a method for the synthesis of carbon nanotubes in an acetylene-based fluidized bed reactor is shown. New types of catalysts were synthesized, for the first time a liquid-phase combustion method was used to obtain the active phase of catalysts in the form of a mixture of Ni/NiO on the surface of Al_2O_3 spheres.

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