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Obtaining Nanofibrous Carbon from Sphagnum Moss with the Help of Pyrolysis and Mechanochemical Activation

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Abstract

Nanofibrous carbon (multi-wall nanotubes) was obtained through mechanochemical treatment of the amorphous modification of carbon, synthesized from sphagnum moss, carried out using the vario-planetary mill.

Key words: sphagnum moss, pyrolysis, mechanical activation, nanofibrous carbon

INTRODUCTION

New allotropic modifications of carbon – nanotubes – find application in many areas of priority in science and technology [1, 2]. Carbon nanotubes are stable and high-strength structures with high elasticity modulus. In addition, they are distinguished by high thermal conduction, chemical stability, while twisting or bending render them semiconductor properties.

Though many methods of obtaining carbon nanotubes are known at present, this material still remains among the most expensive ones. The technology of carbon nanotubes purification – separation of high-quality tubes from defective ones – and the method of nanotubes introduction into other systems require improvement.

EXPERIMENTAL

As a continuation of our studies and developments in the area of functional composite materials [3–5] from plant raw material, nanofibrous carbon (multiwall nanotubes) composed of the amorphous modification of carbon was formed with the help of pyrolytic and mechanochemical processes.

Brown sphagnum moss (*Sphagnum fuscum*) was used as the initial material. Sphagnum moss was preliminarily dried, sieved to remove excess humidity and foreign admixtures, and subjected to disintegration to achieve particle size of 100–150 μm.

Carbon modification with amorphous structure was obtained according to the developed energy-saving technology at a temperature of 950 °C [6, 7]. Then the carbon modification was subjected to cyclic mechanochemical treatment in a Pulverisette-4 vario-planetary mill (Fritsch, Germany). The milling bodies in the vario-planetary mill were balls made of the hard alloy VK-6, 16 mm in diameter. The mechano-reactor of the vario-planetary mill was a tight container made of corrosion-proof steel with an insert made of the hard alloy VK-6. The following regime was involved in experiments: the frequency of rotation of the main disk is equal to 400 min^{-1}, satellites – 800 min^{-1}; the ratio of initial material mass to the mass of milling bodies (intensity) 1 : 27. Argon was used for protective atmosphere.

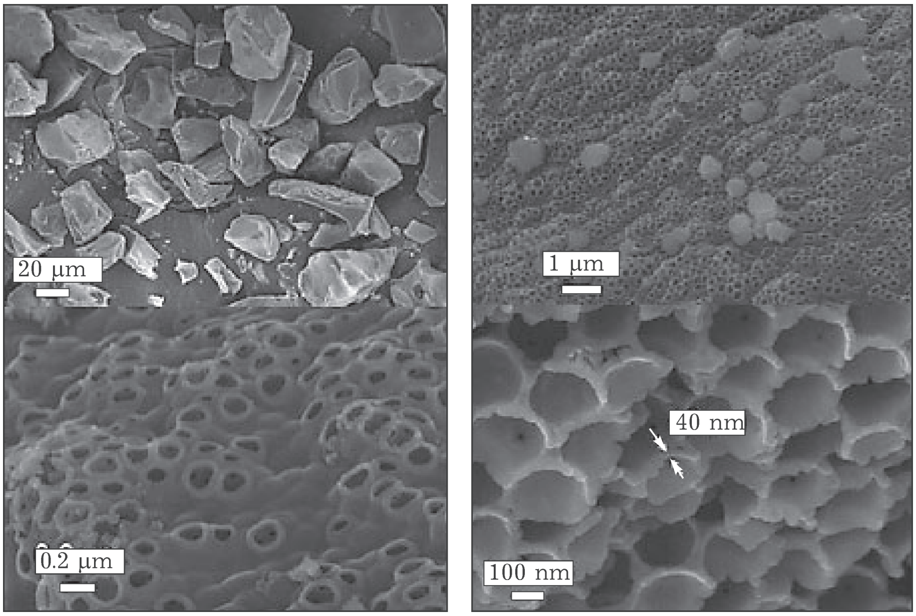

Fig. 1. Morphology of amorphous carbon modification from sphagnum moss. Pyrolysis temperature: 950 °C.

Investigation of specific surface was carried out with the Sorbtometr-M analyzer of specific surface (Katakon Co., Novosibirsk), specific surface was determined from nitrogen thermodesorption.

The structure of surface, shape and size of the particles of carbon modification obtained from brown sphagnum were studied with the help of scanning electron microscope EVO-60XVP (Carl Zeiss, Germany). The structure of nanofibrous carbon was studied with the high-resolution scanning electron microscope Hitachi S5500 with attachment for transmission microscopy.

X-ray energy dispersive microanalysis was carried out with the EVO-50XVP scanning electron microscope (Carl Zeiss, Germany) combined with the INCA Energy-350 X-ray energy dispersive spectrometer (England).

RESULTS AND DISCUSSION

At the first stage of investigation, carbon modification with amorphous structure was obtained from brown sphagnum at pyrolysis temperature of 950 °C.

The product of pyrolysis as powdered material with particle size less than 50 μm and specific surface 220 m^2/g (single-point BET method) is shown in Fig. 1.

Nanofibrous modification of carbon 10–40 nm in diameter (Fig. 2) was obtained using the amorphous carbon modification from sphagnum moss after long-term mechanical activation (grinding time ~27 h).

In the opinion of the authors, the formation of nanofibrous structure of carbon material during mechanochemical treatment was promoted by the fractal, nanoporous structure of carbon synthesized during the pyrolytic ac-

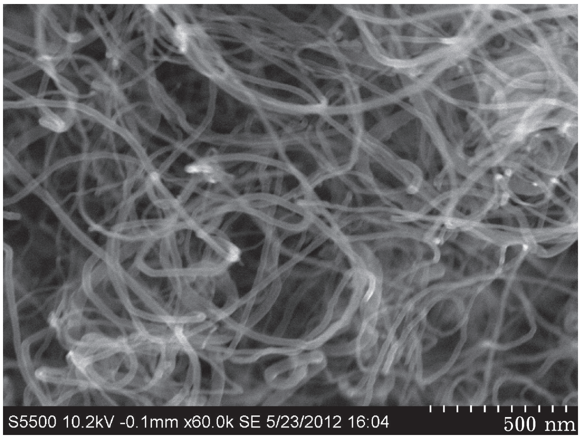

Fig. 2. SEM image of fibrous carbon from sphagnum moss.

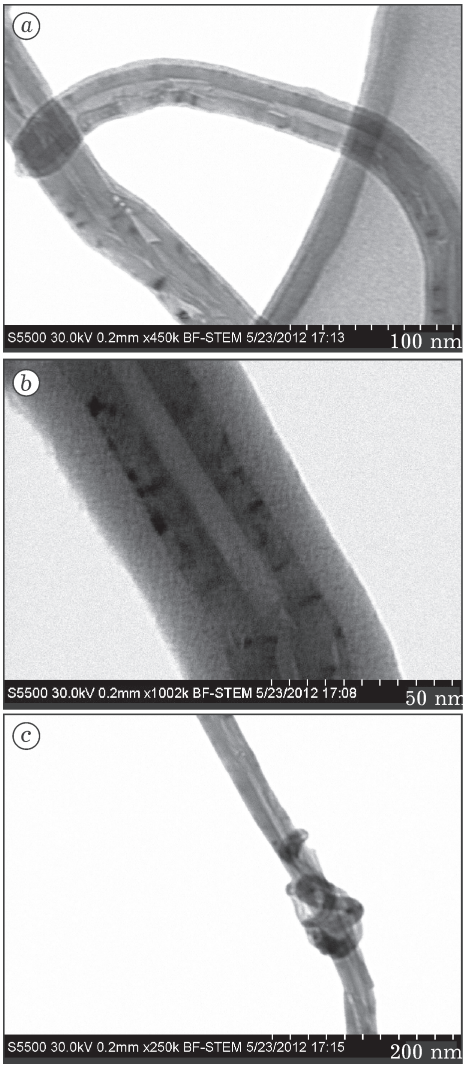

Fig. 3. SEM image of nanotubes with bamboo type structure (*a*), embedded nanocones (*b*) and the nanotubes with different diameters (*c*).

tion on sphagnum moss, specificity of grinding mechanism (the absence of shock action) in vario-planetary mill, catalytic conditions created by milling bodies made of the hard alloy VK-6 (Table 1). Increased tungsten and cobalt content is due to the wear of hard-alloy milling bodies and the walls of the mechano-reactor.

The initial hard alloy VK-6 contains Co 6 mass % and WC 94 %, but their concentrations in nanofibrous carbon are comparable, which is connected with the erosion of cobalt from the carbide matrix during the mechanochemical treatment of amorphous carbon.

TABLE 1

Concentrations of chemical elements in nanofibrous carbon, mass %

Exp. No.	O	C	W	Ni	Cu	Co	Fe	Ti
1	0.4	98.3	0.23	0.01	0.014	0.27	0.1	0.032
2	0.45	98.5	0.27	0.009	0.017	0.31	0.12	0.019
3	0.5	98.4	0.22	0.012	0.021	0.33	0.09	0.08
4	0.3	98.7	0.15	0.07	0.011	0.30	0.05	0.043

As a result of long-term mechanical activation of the pyrolytic modification of carbon from sphagnum moss, multiwall nanotubes are formed (Fig. 3).

The formed nanotubes have defect-bearing structure of bamboo and embedded nanocone type (see Fig. 3, *a*, *b*). The diameter of nanotubes is mainly about 40 nm, but nanotubes 10 and 50 nm in size also occur (see Fig. 3, *c*).

Nanofibrous carbon obtained as a result of mechanochemical treatment (nanotubes) is distinguished by rather high specific surface (S_{sp} = 550 m^2/g) and low ash value (2.7 mass %).

CONCLUSION

Thus, nanofibrous modification of carbon composed of nanotubes 10 to 60 nm in diameter was formed through mechanochemical treatment of the amorphous modification of carbon obtained by pyrolysis of sphagnum moss at a temperature of 950 °C.

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