ELECTRONIC STRUCTURE OF PRODUCTS OF POLYVINYLIDENE FLUORIDE AND TOLUENE DIISOCYANATE CARBONIZATION IN AN Al_2O_3 MATRIX

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Introduction

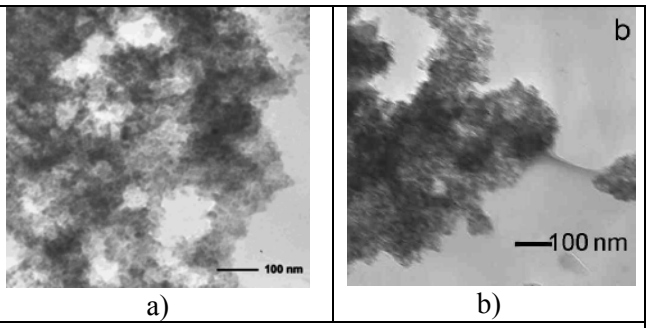
Nowadays various porous carbon materials are used in many ways. These materials are widely applied as high-performance sorption materials, catalyst carriers, membrane systems for filtration of either solutions or gases etc. At present, several methods are known to be useful for obtaining of carbon in porous materials. One of widely used methods is carbonization of polymers and organic precursors. The peculiarity of this method is a possibility to obtain carbon nanostructures with diverse morphology and anisotropy by changing a carbonization rate, precursor concentration and adding different catalysts.

In this work we present the results of a study of the electronic structure of the carbon porous materials synthesized by carbonization of polyvinylidene fluoride (PVDF) and toluene diisocyanate (TDI) in an Al_2O_3 matrix.

In both cases, fine Al_2O_3 (S_{sp}=200 m^2/g) was used as a structure-forming element for matrix obtaining. Industrial PVDF and mixture of 2.4- and 2.6-TDI (in the ratio 80:20) were precursors for obtaining carbon materials. From Al_2O_3 and mixture of PVDF in acetone in the first case and mixture of TDI in benzene in the second case, gels were obtained. These gels were dried at room temperature and were carbonized at 750 ^oC under argon atmosphere.

The method of the X-ray emission spectroscopy was used to study the electronic structure of products of carbonization. This method gives the information about the energy distribution of the electronic valence states of a matter under study. The X-ray emission spectra were registered using an X-ray spectrometer-monochromator RSM-500 with oil-free evacuation of a spectrometer chamber. The operation conditions of the X-ray tube were the following: accelerating voltage, U_a = 5 kV; anode current, I_a = 2.5 mA. The specimens under study were rubbed into a water-cooling copper anode.

Scanning electron microscopy (SEM) images of the carbon composites were derived with an electron microscope JEM100CX-II by the standard technique.



a) b)

Fig. 1. Scanning electron microscopy images of products of carbonization of PVDF (a) and TDI (b) in the Al_2O_3 matrix.

Results and discussion

Scanning electron microscopy images of carbon composite of both materials are presented in Fig. 1. As can be seen from this figure, the particles morphology of products of carbonization of PVDF and TDI in the Al_2O_3 matrix resembles each other. In the both cases, products of carbonization possess almost spherical shapes with particles sizes of about 10 nm.

We have recorded the X-ray emission C*K*α-bands for each sample under consideration. These spectra are shown in Fig. 2. The shapes of the X-ray emission C*K*α-bands of products of carbonization of PVDF and TDI were found to be different. The shape of the X-ray emission C*K*α-band of product of carbonization of TDI in the Al_2O_3 matrix is similar to that of the band of fullerene C_{60} with some graphite admixtures. The shape of the X-ray emission C*K*α-band of products of carbonization of PVDF in the Al_2O_3 matrix does not coincide with the known analogous emission C*K*α-band of pure carbon materials. This can indicate that the composite consists of a mixture of several carbons. When measuring the spectra, the shape and intensity of the X-ray

emission C*K*α-bands were rather stable. This fact confirms the existence a strong chemical bond between the matrix and the carbon composite.

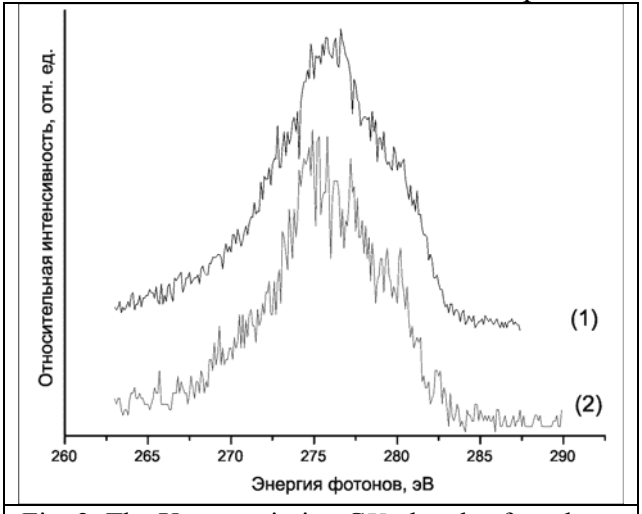


Fig. 2. The X-ray emission C*K*α-bands of products of carbonization of PVDF (1) and TDI (2) in the Al_2O_3 matrix.

The X-ray emission O*K*α-bands were obtained as well. These spectra are presented in Fig. 3. As can be seen from the figure, the shapes and the energy positions of the main fine-structure features are in close agreement with high degree of accuracy for the both samples investigated. Each spectrum is similar to the X-ray emission O*K*α-band of γ-Al_2O_3 regarding the shape and energy positions of spectra fine-structures. However, the X-ray emission O*K*α-bands of the investigated samples are broader somewhat in comparison with the X-ray emission O*K*α-band of γ-Al_2O_3. This fact indicates the existence of the chemical bonding between the matrix and the products of carbonization. During of the experiments, the shape and the intensity of the X-ray emission O*K*α-bands of the both samples under consideration were stable.

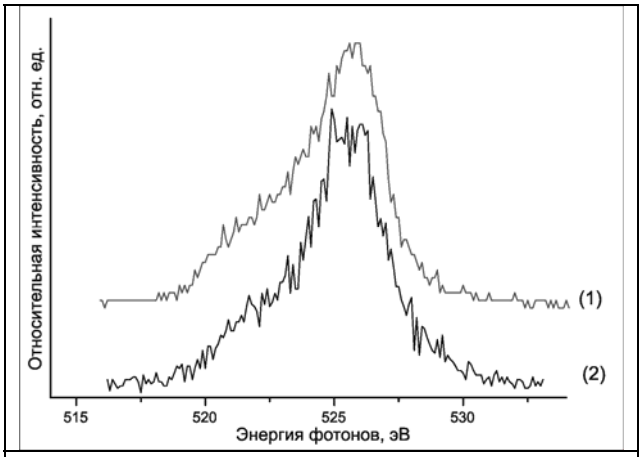


Fig. 3. The X-ray emission O*K*α-bands of products of carbonization of PVDF (1) and TDI (2) in the Al_2O_3 matrix.

Conclusions

We have synthesized monolithic nanostructured samples of products of carbonization of PVDF and TDI in an Al_2O_3 matrix.

From scanning electron microscopy images of the composites, it has been defined that products of carbonization possess almost spherical shapes with particles sizes of about 10 nm.

We have measured the X-ray emission C*K*α- and O*K*α-bands of products of carbonization of PVDF and TDI in the Al_2O_3 matrix. The analysis of results of the measurements render that, the products of carbonization consist of a mixture of different carbons. The chemical bonding between the matrix and the carbon composite was found to be rather strong.