

## INVESTIGATION OF NANODIAMONDS BY ELECTRON-POSITRON SPECTROSCOPY

GULIDOVA Lyudmila, LAPTEV Roman, MIKHAYLOV Andrey

*Tomsk Polytechnic University, Institute of Physics and Technology, General Physics Department, Tomsk, Russian Federation, [L\\_qulidova@bk.ru](mailto:L_qulidova@bk.ru)*

### Abstract

Nanodiamond samples with various densities were studied by positron annihilation spectroscopy with the aim of finding out the hydrogen saturation effect on nanodiamonds. The positron lifetime measurements were carried out on packed nanodiamonds with various densities at room temperature and atmospheric pressure. The density of compacted samples does not affect positron lifetime.

**Keywords:** nanodiamonds, positron annihilation spectroscopy, positron lifetime

### INTRODUCTION

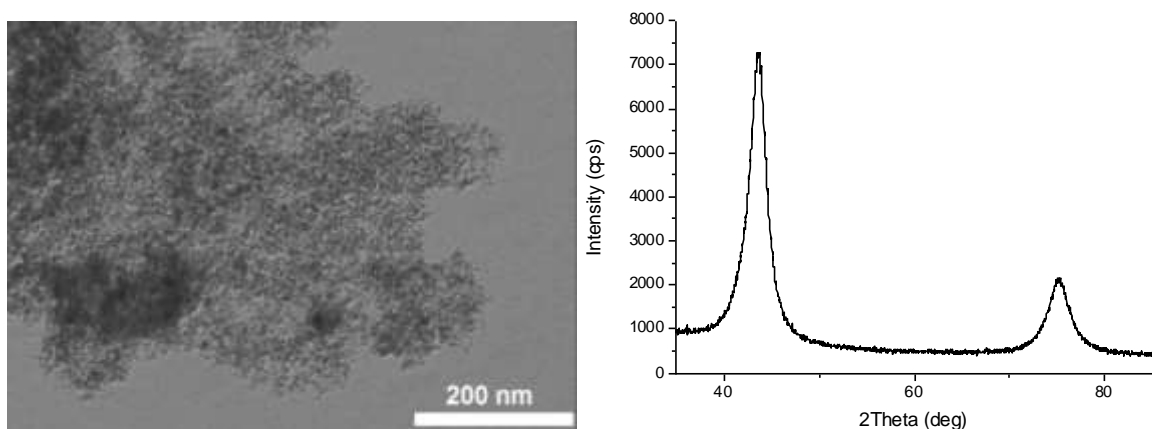
Carbon nanostructure materials have been extensively studied and found application in a wide range of science and engineering since the moment of their discovering. Carbon nanostructures are also regarded as the hydrogen storage materials. Carbon nanostructures such as nanodiamonds have unique structure. Nanodiamonds are chemically well-defined, and of high purity. Nanodiamonds are likely to share some of the unique properties of macroscopic diamond that are very attractive for a number of applications.

The positron annihilation spectroscopy (PAS) is unique method for characterization of defects, pores of carbon nanostructure materials [1, 2] and determination the hydrogen effect on nanodiamonds.

### 1. EXPERIMENTAL

The nanodiamond consists of a crystalline diamond core and amorphous or graphite shell with functional groups. The non-equilibrium conditions of the nanodiamonds synthesis influence on high density of defects and developed surface.

The samples were characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD) and positron annihilation spectroscopy. The crystallinity and purity of the samples were obtained by XRD. The carbon material consists of nanodiamonds with an average size of 8 nm.



**Fig.1** TEM image and X-ray diffractogram of nanodiamonds.

The functional groups on the nanodiamonds surfaces allow create carbon materials with desired properties. The cover around the core prevents the penetration of hydrogen into the nanodiamond as evidenced not high sorption capacity of these materials. Plots of the adsorbed hydrogen from the pressure and time are presented below.

The compacted carbon powders were used for hydrogen saturation and positron annihilation measurements. Compaction process was carried out on hydraulic press at room temperature in the pressure range 100-700 MPa without using a binder. For further studies the samples were obtained at 400, 560 and 620 MPa. At pressures below 400 MPa the material does not compact and above 620 MPa destruct of high internal stresses. Samples are tablets with 5 mm diameter and mass of 0.1 g. The densities of 1, 2 and 3 samples were 1.22, 1.51, 1.32 g/cm<sup>3</sup> and porosity were 45.8%, 32.9% and 41.3%, respectively.

Hydrogen concentration measurements were carried out by Gas Reaction Controller complex at room temperature and pressure of 8 atm. Samples were placed in a chamber, which was evacuated to the pressure of 10<sup>-6</sup> atm.

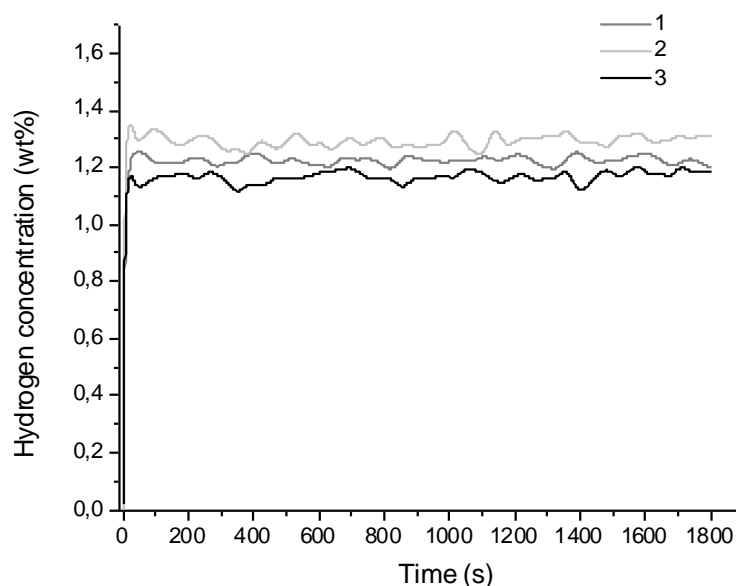
Positron methods are based on experimental measurements of the parameters of annihilation positron implanted in the test material. These methods are divided into temporary, angular distribution of annihilation photons and Doppler broadening of annihilation line. In this paper the method of positron lifetime is considered.

As a source of the positron isotope <sup>44</sup>Ti with the activity of 24.5 μCi was chosen. The <sup>44</sup>Ti was placed between two copper capsules filled the compacted carbon powder. The method based on the interaction of positrons with electrons of sample. The time resolution of the system was 260 ps. The full information about spectrometry complex was presented at the article [3].

The positron source 1 mm in thickness. The isotope <sup>44</sup>Ti was placed between two compacted carbon samples. As the results of experiment the data about positron lifetime in compacted carbon powder were taken. Positron lifetime spectra were decomposed into three components. Data are shown in Table 1.

## 2. RESULTS AND DISCUSSION

The concentration-time sorption isotherms are showed lack of density influence amount of absorbed hydrogen. The average concentration of absorbed hydrogen is 1.2 wt% (Fig. 2).



**Fig. 2** Hydrogen concentration-time diagram

**Table 1** Positron lifetime

Sample name	$\tau_1$ , ns	$I_1$	$\tau_2$ , ns	$I_2$	$\tau_3$ , ns	$I_3$
1	0.237	67.7	0.499	28.9	3.047	3.3
2	0.225	65.6	0.482	31.0	2.968	3.3
3	0.234	67.4	0.491	29.5	3.110	2.9
1+H	0.254	73.4	0.538	23.7	4.048	2.7
2+H	0.275	76.5	0.566	20.3	3.306	3.2

The short-lived component  $\tau_1$  and intensity  $I_1$  are increased after hydrogen saturation, which connected with a change in the bulk of samples. The middle-lived component  $\tau_2$  is increased, but intensity  $I_2$  is decreased. Such changes are connected with developed surface of nanodiamonds and presence of functional groups. The long-lived component  $\tau_3$  may be attributed to o-ps pick-off annihilation in small spaces between carbon nanodiamonds.

The positron lifetime in the material after exposure, for example heat treatment, introduction of impurities, such as hydrogen, significantly differs from the positron lifetime in the initial material. The positron lifetime increases because of electron density change. Hydrogen saturation of nanodiamonds increases the positron lifetime. As the internal crystalline structure of the nanodiamond presumably does not available for hydrogen, therefore surface can change the positron lifetime.

## CONCLUSION

The positron annihilation spectroscopy is unique sensory method for detection of electron density insignificant changes. It has been demonstrated that change of positron lifetime due to the hydrogen saturation effect on electron density. For more detailed analysis is necessary to combine the positron lifetime positron lifetime measurements with method of Doppler broadening of annihilation line.

## REFERENCES

- [1] BUDZIAK A., DRYZEK J., KRAWCZYK J., ZIELINSKI P.M. Calorimetric and Positron Lifetime Measurements of Hydrogenated Carbon Nanocones. Acta Physica Polonica A, Vol. 117, No. 4, 2010, pp.574-577.
- [2] CHAKRABARTI K., NAMBISSAN P.M.G., MUKHERJEE C.D., BARDHAN K.K., KIM C., YANG K.S. Positron Annihilation Spectroscopy of Polyacrylonitrile-based Carbon Fibers Embedded with multi-wall Carbon Nanotubes. Carbon, Vol.44, 2006, pp. 948-953.
- [3] LAPTEV R.S., LIDER A.M., BORDULEV Y.S., KUDIAROV V.N., GARANIN G.V., Hydrogenation-induced microstructure changes in titanium, J. Alloys Compd., Vol. 645, 2015, pp.193-195.