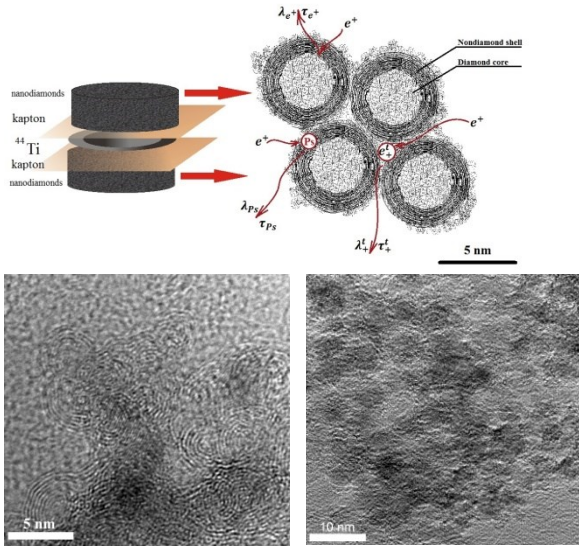
Positron spectroscopy of nanodiamonds after hydrogen treatment

L.V. Nikitina, R.S. Laptev, A.M. Lider, A.A. Mikhaylov

General Physics Department, Institute of Physics and Technology, Tomsk Polytechnic University, 634000 Tomsk, Russia

gulidova@tpu.ru, laptevrs@tpu.ru, lider@tpu.ru

The structure of nanodiamonds and their defects influence the hydrogen sorption capacity. Positronium can be used as a sensor for detecting places with the most efficient capture of hydrogen atoms. The hydrogen saturation of the carbon material is saturated from gas atmosphere. The concentration of hydrogen which is absorbed by the sample depends on the pressure and temperature. The concentration 1.2 wt% is achieved at temperature of 243 K and pressure 0.6MPa. The hydrogen saturation of nanodiamonds changes the positron lifetime. Increase of sorption cycle numbers effects on the positron lifetime and as well as the parameters of the Doppler broadening of annihilation line. Consequently, the electron-positron annihilation is a sensitive method, which allows detecting the electron density fluctuation of the carbon material after hydrogen saturation.



TEM images of the nanodiamonds material:
a) carbon onions, b) nanodiamonds

Materials

The carbon-based material was formed by detonation synthesis (FRPC "Altai", Byisk, Russia). Synthesis of nanodiamonds is carried out by detonation of solid explosives in an inert atmosphere. The release of huge energy occurs due to the breaking of chemical bonds in the front of the detonation wave. The highly disperse carbon material is condensed from the liberated carbon under conditions of high temperatures (3000-4000) K and pressures (20-30) GPa. Unpurified material contains (25.0-50.0) wt% of nanodiamonds. The purified material consists of carbon nanodiamonds and onion-like particles. Nanodiamond consists of a crystalline diamond core and nondiamond shell with functional groups on surface.

Sample preparation

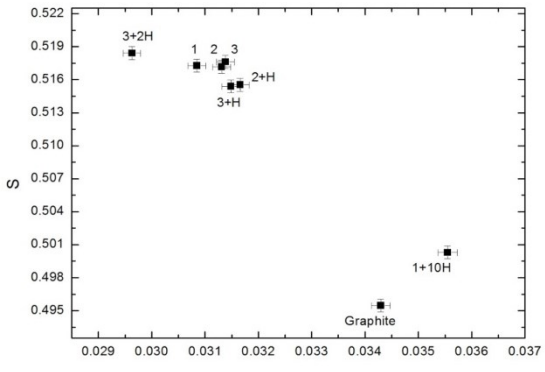
The compacted carbon powders were used for hydrogen saturation and positron annihilation measurements. Compaction process was carried out on hydraulic press at room temperature in the pressure range 100-700 MPa without using a binder.

The samples are pills with 5 mm diameter and mass of 0.1 g.

X-ray diffraction measurements were carried out using high-resolution powder diffractometer XRD-7000 with CuKα-radiation. Nanodiamonds morphology was examined by electron microscope JEM-2100F (JEOL, Japan). Hydrogen concentration was measured by Gas Reaction Controller complex (Advanced Materials Corporation, USA) at low temperature and a pressure of 0.6 MPa.

Table The parameters of positron annihilation in the nanodiamonds before and after hydrogenation

State	$\tau_{o\text{-}Ps}$, ns	$I_{o\text{-}Ps}$, %	τ_{1}, ns	I_{1}, %	μ, ns^{-1}	τ_{free}, ns	κ, ns^{-1}
1	3.05±0.04	3.35	0.51±0.01	14.8	0.60±0.02	0.30±0.03	0.18±0.01
2	3.13±0.06	2.9	0.52±0.01	13.6	0.53±0.02	0.31±0.04	0.16±0.01
3	3.18±0.06	3.28	0.53±0.02	13.2	0.51±0.02	0.31±0.04	0.17±0.01
2 (1 cycle)	4.47±0.08	2.82	0.59±0.01	10.3	0.37±0.01	0.32±0.04	0.14±0.01
3 (1 cycle)	4.1±0.1	3.19	0.69±0.09	7.37	0.24±0.07	0.34±0.04	0.14±0.01
3 (2 cycles)	3.28±0.05	3.26	0.60±0.05	9.17	0.31±0.07	0.34±0.04	0.15±0.01
1(10 cycles)	2.35±0.04	5.07	0.42±0.01	42.3	2.91±0.15	0.29±0.02	0.46±0.01
Graphite	-	-	0.42±0.01	23.10	2.7±0.1	0.28±0.02	-



Dependence of S-parameter on W-parameter for non-hydrogenated and hydrogenated nanodiamonds samples

Investigation of positron lifetime (PL) and Doppler broadening (DB) shift of annihilation line before and after hydrogenation was performed using the special complex. The samples were arranged in a so-called «sandwich» and mounted in a special sample-holder. PL and DB spectra were collected simultaneously. The positron source was represented by a ^{44}Ti isotope with an activity of 24.5 μCi.

Spectra were fitted using LT10 software. The spectral analysis was performed implementing a delayed formation of positronium (DFP) model. DB spectra were acquired by collecting 2.5×10^5 counts and analyzed using SP software package.