ISBN: 978-3-95450-240-0 ISSN: 2673-5539 doi:10.18429/JACoW-RuPAC2021-MOPSA57

EXPERIMENTAL INVESTIGATION THE SYNTHETIC CRYSTAL DIAMOND PLATES OF METHODS OF POSITRON ANNIHILATION SPECTROSCOPY

M.K. Eseev, I.V. Kuziv, Northern (Arctic) Federal University named after M.V. Lomonosov, Arkhangelsk, Russia
A.G. Kobets[1], I.N. Meshkov, O.S. Orlov, A.A. Sidorin, K. Siemek[2], Joint Institute for Nuclear Research, Dubna, Moscow Region, Russia
[1]also at Institute of Electrophysics and Radiation Technologies, NAS of Ukraine, Kharkov, Ukraine
[2]also at Institute of Nuclear Physics Polish Academy of Sciences, Krakow, Poland

Abstract

Nowadays positron annihilation spectroscopy is a powerful technique of microstructure investigations of crystalline materials. Doped diamonds were studied by Positron annihilation spectroscopy and Infrared spectroscopy. As a result of the experiments, data show the effect of nitrogen doping of diamonds on the occurrence of defects in a doped diamonds.

INTRODUCTION

Synthetic crystal diamond plates are used in scientific and technical fields and in new materials. Various defects and types of defects in diamond plates can changed its properties. Investigation the defects of diamond plated is very important for use diamond plates for fully solving applied problems in roentgen-optical systems and quantum sensorics. The investigation included three different spectroscopy types. First type is Positron annihilation spectroscopy (PAS). PAS is unique non-destructive instrument to detect open-volume defects, such as vacancies, vacancy clusters, microvoids or dislocations. PAS can define defects in the near-surface layer of materials with thickness up to 10…100 nm. It can be used in cases where other popular methods such as scanning electron microscopy (SEM) or X-ray diffraction are not applicable [1,2]. The second type of spectroscopy which used in this investigation is Infrared spectroscopy. This technique allows to define types of diamond defects such as A type and C type. The third type is Raman spectroscopy. It is valuable method because it provides readily distinguishable signatures of each of the different forms of carbon.

MATERIALS AND METHODS

Generation of the synthetic crystal diamond plates was conducted by HTHP method with addition of nitrogen in different concentrations (12,5 ppm, 75 ppm, 88 ppm). Defects in the atomic structure of diamond plates are responsible for this colour. Transparent sample contained 12 ppm, yellow - 7 ppm, pink - 88 ppm. Then diamonds were cut by laser. Several series of paired samples of the diamond plates with 0,8 mm in height and different side size were studied (Fig. 1).

One sample from each group was cut along and across and left for investigation by Infrared and PAS spectroscopy methods.

Simultaneously one sample from each group was left for investigation by Raman spectroscopy method. Pink sample contained NV^0 (575 nm) and NV^- (637 nm) defects.

Figure 1: Synthetic crystal diamond plates.

First part of experiment consisted in measurement the positron annihilation lifetime spectroscopy (PALS). PALS measurements were conducted using digital spectrometer APU-8702RU and the BaF_2-based scintillator (Fig. 2).

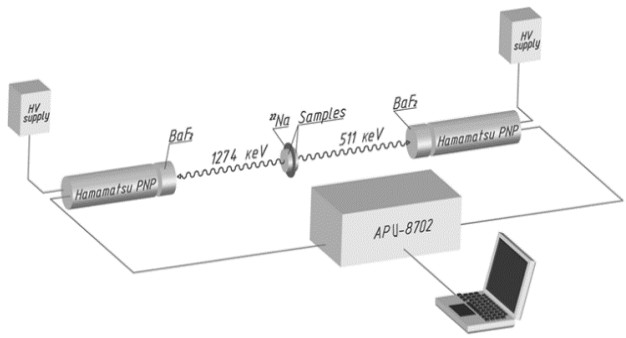

Figure 2: PALS instruments.

The timing resolution equaled 250 ps. The typical sandwich geometry was used, where the positron source located between two identical samples. The ^{22}Na isotope with an activity of 850 kBq was used as a positron source. It was placed between two identical samples. The positron source consisted of $^{22}NaCl$ salt between two titanium foils (10 μm).The measurements were carried out at room temperature.

This method shows average lifetime of the positron in diamond plates. The positron lifetime gives information

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about the electron density. Electron density inside the defect is lower in comparison with bulk area what in tern reflects in the mean positron lifetime.

Doppler broadening spectroscopy of annihilation radiation (DB) measurements were performed using ORTEC HPGe detector model GEM25P4-70 with energetic resolution FWHM 1.20 keV for energy 511 keV. Each obtained spectrum was analyzed to calculate S and W parameters by SP-16K program.

The Doppler Broadening method is based on slow monoenergetic positron beam with positrons energies ranging up to 35 keV working at JINR in Dubna, Russia (Fig. 3) [3].

The second part of the experiment consisted in the study of samples by Infrared spectroscopy. Infrared spectroscopy measurements were conducted using Bruker HYPERION 3000 Spectrometer.

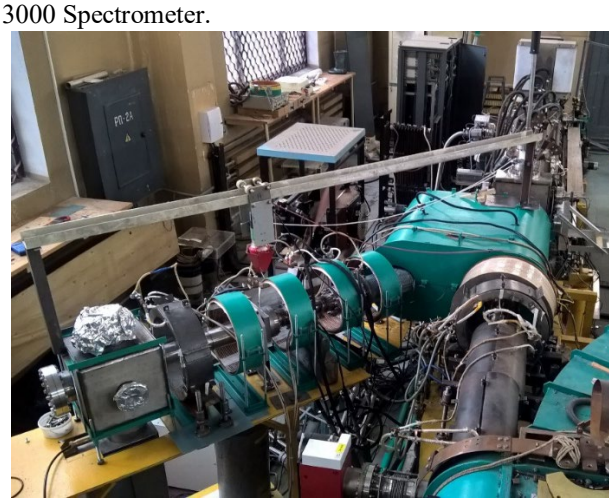

Figure 3: Doppler Broadening instruments.

RESULTS AND DESCUSSION

PALS measurements revealed two lifetime components. The total number of coincident events, accumulated in the spectrum for each pair of samples, equals to $3*10^6$. The analysis of spectra was conducted using LT 10.2 program.

The annihilation rate λ is a reciprocal value of mean positron lifetime.

$$\lambda = \frac{1}{\tau} = \pi r_0^2 c n_e \,, \qquad (1)$$

Dependence positron lifetime from nitrogen concentration of samples are shown in Table 1. τ_1 and τ_2 represent positron lifetime components and I_1 and I_2 its intensities, respectively. τ_{av} is a mean positron lifetime.

Table 1: Dependence Positron Lifetime From Nitrogen Concentration Of Samples

Nitrogen concentration, ppm	τ_1, ps	I_1, %	τ_2, ps	I_2, %	τ_{av}, ps
12,5	153 ± 1	55,9	258 ± 1	44,1	199 ±1
75	171 ± 1	68,7	332 ± 1	31,3	220 ±1
88	207 ± 1	79,4	350 ± 1	20,6	236 ±1

The positron lifetime for bulk (τ = 100-100 ps) was reported by Uedono A. [4] and for monovacancy (τ = 140-150 ps) was reported by Pu A [5].

The results of lifetime method is shown in Fig. 4.

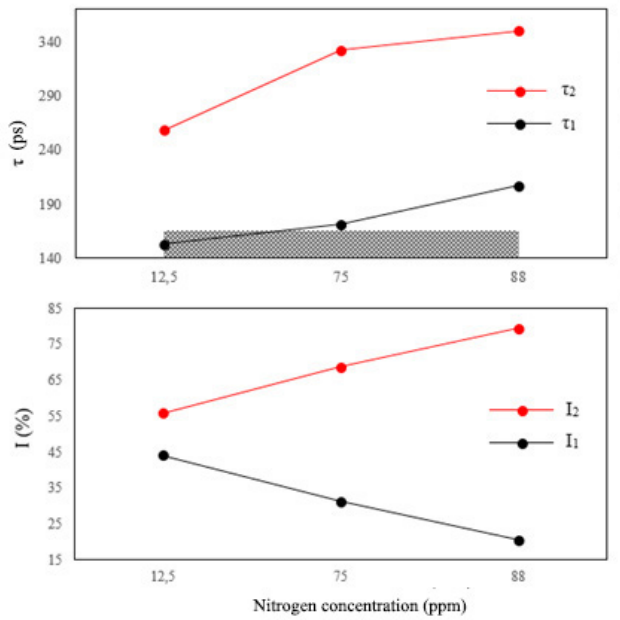

Figure 4: Dependence of the positron lifetime on nitrogen concentration.

The Doppler broadening measurement were used to study defect profiles beneath the surface.

Each obtained spectrum was analyzed to calculate S and W parameters by SP-16K program. The S parameter reflects annihilations with low momentum electrons taking place in defects. It is sensitive to open-volume defects like vacancies, vacancy cluster or jogs at dislocation lines.

Doppler broadening show that defect concentrations increase with increasing nitrogen concentration at the depth of 40-400 nm, then defect concentration decrease (Fig. 5).

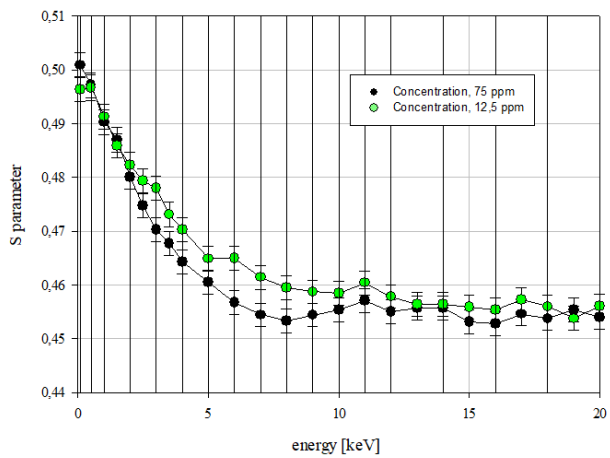

Figure 5: Dependence of the S parameter in samples (12,5 ppm and 75 ppm) on positron energy.

Infrared spectrum of transparent sample which was cut along shown in Fig. 6 and which was cut across shown in Fig. 7.

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Concentration of A defects in transparent sample which was cut along - 91,63±5,63 ppm.

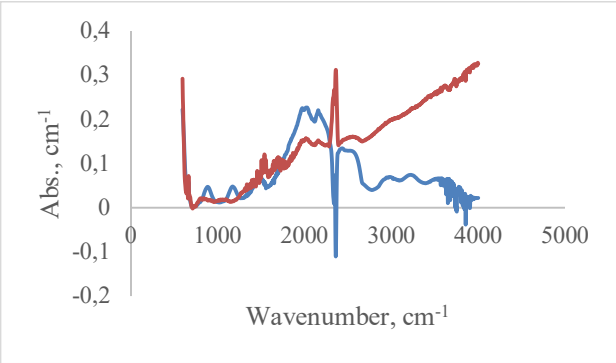

Figure 6: Infrared spectrum of transparent sample. Blue line – before cutting, red line – after cutting.

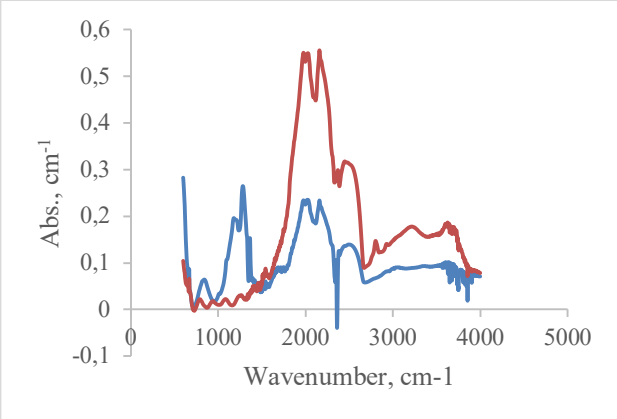

Figure 7: Infrared spectrum of transparent sample. Blue line - before cutting, red line - after cutting.

Infrared spectrum of yellow sample which was cut along shown in Fig. 8 and which was cut across shown in Fig. 9.

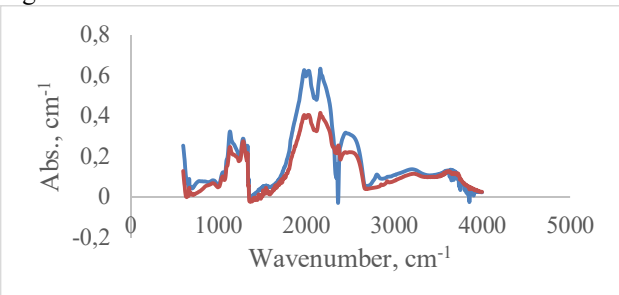

Figure 8: Infrared spectrum of yellow sample. Blue line – before cutting, red line – after cutting.

Concentration of C+ defects in yellow sample before cutting (along) - 64,40±11,69 ppm. Concentration of C+ defects in yellow sample after cutting (along) - 153,44± 27,78 ppm.

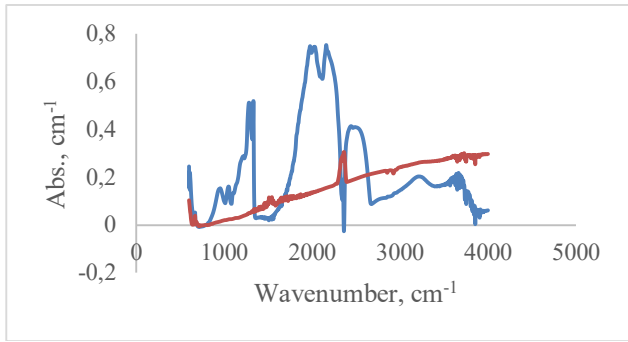

Figure 9: Infrared spectrum of yellow sample. Blue line - before cutting, red line - after cutting.

Concentration of C+ defects in yellow sample before cutting (along) - 110,31±20,06 ppm.

CONCLUSION

This PAS investigation reveals that all samples have defects. All the defects are of the vacancy-type such as monovacancy, divacancy and vacancy clusters. Dependence of the amount of such defects is also in accordance with the depth of the layer in diamond plate. Positron annihilation spectroscopy is a unique method for detection the structural defects.

Raman spectroscopy shows that pink sample contained NV defects (NV^0 (575 nm) and NV^- (637 nm) defects).

Infrared spectroscopy shows that transparent sample (Nitrogen concentration - 12,5 ppm) has just A-type defect and yellow sample (Nitrogen concentration - 75 ppm) C+-type defect.

This work was supported by the state order of the Ministry of Education and Science of the Russian Federation No. 075-00940-21-05. We would like to thank AGD Daimonos JSC for the presented samples.

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