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## BEHAVIOUR OF MUONIUM IN SYNTHETIC DIAMOND

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Мюоний в искусственном алмазе

Исследована вероятность нахождения мюона в различных состояниях в искусственном кристаллическом и поликристаллическом алмазах. В кристаллическом образце IIa-типа при 150 К наблюдаются вклады диамагнитного мюона, «нормального» и «аномального» мюония в пропорциях 1,5 %, 57 % и 8,1 % соответственно. Недостающая фракция поляризации мюона составила 33,4 %, что примерно в два раза меньше, чем в природном алмазе Ia-типа, и в два-три раза больше, чем в природном алмазе IIa- или IIb-типа. В состояниях нормального и аномального мюония скорость релаксации спина мюона в искусственных и природных образцах IIa- и IIb-типа близка.

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Behaviour of Muonium in Synthetic Diamond

The probabilities of finding the muon in various states in synthetic single-crystal and polycrystalline diamond were studied. In the IIa-type single-crystal sample at 150 K the contributions of the diamagnetic muon, «normal» and «anomalous» muonium were observed to be 1.5%, 57% and 8.1%, respectively. The missing fraction of muon polarization was 33.4%, which is approximately two times smaller than in the Ia-type natural diamond, and two or three times greater than in the IIa- and IIb-type natural diamonds. The muon spin relaxation rates at the «normal» and «anomalous» muonium states in the synthetic and natural samples of IIa- and IIb-type are similar.

The investigation has been performed at the Dzhelepov Laboratory of Nuclear Problems, JINR.

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## INTRODUCTION

Diamond with its unsurpassed mechanical strength, thermal conductivity, and radiation hardness is a promising material for radiation detectors, for electronic and optical-electronic components able to withstand high heat and radiation loads. Great advances have been made over the last years in technology of manufacturing synthetic single-crystal diamond and diamond films [1,2]. The properties of the synthetic diamond should be comprehensively studied to find out the scope of its practical application.

Man-made bulk diamond samples (single crystals and films) contain about (or more than)  $10^{17} \text{ cm}^{-3}$  dissolved hydrogen atoms. Recently, the surface conductivity was observed in a diamond sample saturated in the hydrogen atmosphere [3]. However, conventional methods do not yield sufficient information on behaviour of hydrogen in diamond.

Polarized positive muons  $\mu^+$  can be used to imitate and study the behaviour of the hydrogen atom in matter (see, for example, [4,5]). A positive muon in matter may pick up an electron and form a muonium ( $\text{Mu} = \mu^+ e^-$ ). Since the muon mass is  $\approx 9$  times less than that of proton, the muonium can be considered as the light isotope of the hydrogen atom. Theoretical calculations [6,7] show that in semiconductors with the diamond crystal structure the muonium may be localized in tetrahedral and octahedral interstitial sites or in the middle of the axis between two host atoms (bond-centre site). It was experimentally found (see [8,9]) that in diamond the muonium was localized in the tetrahedral site (commonly referred to as normal muonium and designated as  $\text{Mu}$  or  $\text{Mu}_\text{T}$ ) and the bond-centre site (commonly referred to as anomalous muonium and designated as  $\text{Mu}^*$  or  $\text{Mu}_\text{BC}$ ). One more  $\mu\text{SR}$  signal corresponding to the diamagnetic state of  $\mu^+$  was observed in experiments ( $\mu^+$  state).

In diamond  $\text{Mu}_\text{T}$  possesses isotropic symmetry with the hyperfine constant  $A_{\text{hf}}/h = 3711 \pm 21 \text{ MHz}$  [8] ( $A_{\text{hf}}/h = 3693 \pm 83 \text{ MHz}$  [10]), which is smaller than that for muonium in vacuum  $A_0/h = 4\,463\,302.765 \pm 0.053 \text{ kHz}$  [11]. In the bond-centre site the muonium is anisotropic with axial symmetry relative to [111] axes of crystal. The hyperfine interaction constants of  $\text{Mu}_\text{BC}$  are  $A_{\parallel}/h = 167.98 \pm 0.06 \text{ MHz}$  and  $A_{\perp}/h = -392.59 \pm 0.06 \text{ MHz}$  [8]. For the muonium bond-centre site the muon spin precession frequency in the magnetic

field depends on the angle between the magnetic field and the axes [111] of the crystal. However, theoretical calculations [6,7] reveal that for  $\text{Mu}_{\text{BC}}$  there is a certain «magic» field where the muon spin precession frequency is the same at any orientation of the crystal. This effect can be used to observe a  $\text{Mu}_{\text{BC}}$  signal at the «magic» field in a powder sample.

The above-mentioned values of the hyperfine constants of the muonium were obtained for two natural diamond samples: Ia-type single-crystal diamond and IIa-type diamond powder with grain size 1–6  $\mu\text{m}$ . In the present work the behaviour of the positive muon in two synthetic diamonds is studied.

## 1. MEASUREMENTS

The measurements were carried out at the GPS and Dolly spectrometers located in the  $\pi\text{M}3.2$  and  $\pi\text{E}1$  muon beams of the Paul Scherrer Institute (PSI, Switzerland). In the  $\pi\text{M}3.2$  beam line the muons were transversely polarized — the angle between the muon spin and the muon momentum was  $\sim 70^\circ$ .

The location of the apparatus in the PSI muon beams is schematically shown in Fig. 1. Muons with energy  $\sim 4.19$  MeV pass through the hole in Bw telescope counters and a thin  $M$ -counter and stop in the sample  $S$ . The positrons from the muon decay were detected by the Bw (backward), Fw (forward), Up(up) and Dw(down) telescopes located around the sample (see Fig. 1). In the case of the

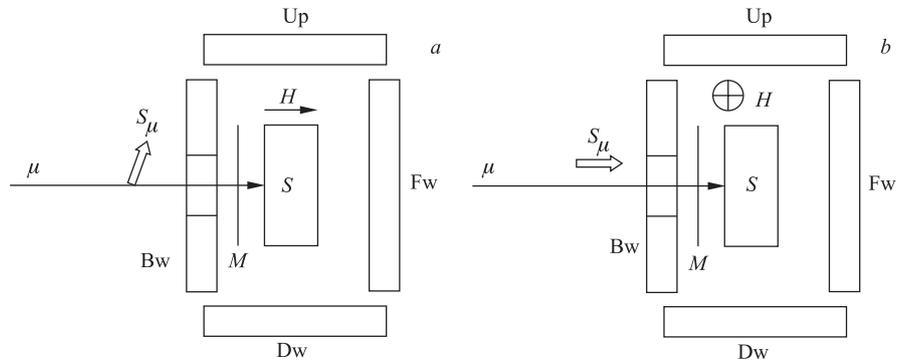


Fig. 1. Location of the apparatus in the PSI muon beams. Up, Dw, Fw and Bw are the up, down, forward and backward telescopes for detection of positrons.  $M$  is the counter for registration of the incoming muons,  $H$  is the magnetic field,  $S$  is the sample,  $S_\mu$  indicates the muon spin polarization direction. *a*) At the GPS spectrometer the muon beam is transversely polarized and the magnetic field is parallel to the incoming muon beam momentum. *b*) At the Dolly spectrometer the muon beam is longitudinally polarized and the magnetic field is perpendicular to the picture plane

Dolly spectrometer there is an additional positron telescope located behind the sample in the plane parallel to the plane of the figure. A magnetic field transverse to the muon spin polarization at the sample was created by Helmholtz coils. The long-time stability of the magnetic field was better than  $10^{-4}$ .

Measurements were carried out using a helium flow cryostat that allowed changing temperature of the sample within the range of 4.2–300 K. The temperature of the sample was stabilized with an accuracy better than 0.1 K. Measurements were carried out in «magic» magnetic field  $H_M = 0.4073$  T at the GPS spectrometer and in  $H = 7.5$  mT at the Dolly spectrometer.

In these measurements two samples (designated as D3 and D5) were used. Sample D3 with the total mass of 2 g was composed of a few Ila-type single-crystal diamonds. Orientation of the crystals in the sample was random (MSC — mosaic single crystal). The concentration of nitrogen in sample D3 was about 0.5 ppm (in diamond, 1 ppm corresponds to  $1.76 \cdot 10^{17}$  cm $^{-3}$ ). The crystals contain few wt% of Ni as inclusion grains visible by eye. The diamond crystals with dimension 1–4 mm were synthesized at the Technological Institute for Superhard and Novel Carbon Materials (Troitsk, Russia) at high pressure and high temperature with synthetic diamond as the seed material [12]. Sample D5 (CVD) of total mass 2 g was a batch of pieces of several diamond films 0.5 mm thick and about  $10 \times 5$  mm in lateral size. The diamond films were produced by a microwave plasma assisted CVD technique in CH $_4$ /H $_2$  mixtures [13]. The main impurities in sample D5 were hydrogen (65 ppm) and nitrogen (1.5 ppm). Other impurities were less than 0.1 ppm. The crystalline axis [110] perpendicular to the film plane was the predominant grain orientation.

The evolution of the muon polarization  $P(t)$  was studied by measuring the time distribution of positrons from the decay of muons  $\mu^+ \rightarrow e^+ + \nu_e + \bar{\nu}_\mu$ . In the general case the time distribution of the positrons (with respect to muon stop in sample) can be presented by the function

$$N(t) = N_0 \exp(-t/\tau_\mu)(1 + \alpha/3 \cdot G(t)) + \text{Bg}, \quad (1)$$

where  $N_0$  is proportional to the number of muons stopped in the sample,  $\tau_\mu$  is the muon lifetime,  $G(t)$  is the polarization of the muon at the decay,  $\alpha$  depends on the parameters of the  $\mu$ SR setup and is close to 1, Bg is background.

The function  $G(t)$  depends on the experimental condition: for measurements at  $H = 7.5$  mT and  $H = 0.4073$  T the respective explicit expressions for  $G(t)$  are

$$G(t) = P_\mu(0) \exp(-R_\mu t) \cos(2\pi\nu_\mu t + \varphi_0) + P_T(0) \exp(-R_T t) \cos(2\pi\Omega_1 t + \varphi_1) \cos(2\pi\Omega_2 t + \varphi_2), \quad (2)$$

$$G(t) = P_\mu(0) \exp(-R_\mu t) \cos(2\pi\nu_\mu t + \varphi_0) + P_{\text{BC}}(0) \exp(-R_{\text{BC}} t) \cos(2\pi\nu_M t + \varphi_1), \quad (3)$$

where  $\Omega_1 = (\nu_{23} + \nu_{12})/2$ ;  $\Omega_2 = (\nu_{23} - \nu_{12})/2$ ;  $\nu_{ik}$  is the frequency of the transition between levels of hyperfine structure of  $\text{Mu}_T$ ; variables with indices  $\mu$ , T and BC refer to the muon in the diamagnetic, normal muonium and anomalous muonium states, respectively;  $P_i(0)$  is the muon polarization at  $t = 0$ ;  $R_i$  is the relaxation rate of the muon spin;  $\nu_M$  is the muon spin precession frequency for the  $\text{Mu}_{\text{BC}}$  fraction in the «magic» magnetic field;  $\varphi_i$  is the initial phase of the muon spin precession.

## 2. RESULTS AND DISCUSSION

Figure 2 shows the results of Fourier transformation of the  $\mu\text{SR}$  spectra for sample D5 measured in the magnetic field 7.5 mT at the temperatures 50 and 250 K. The peaks at  $\sim 100.4$  and  $\sim 105.8$  MHz correspond to the transitions between the levels of the hyperfine structure of the muonium  $\text{Mu}_T$ . The narrowing of the lines  $\nu_{12}$  and  $\nu_{23}$  with increasing temperature was observed for samples D3 and D5.

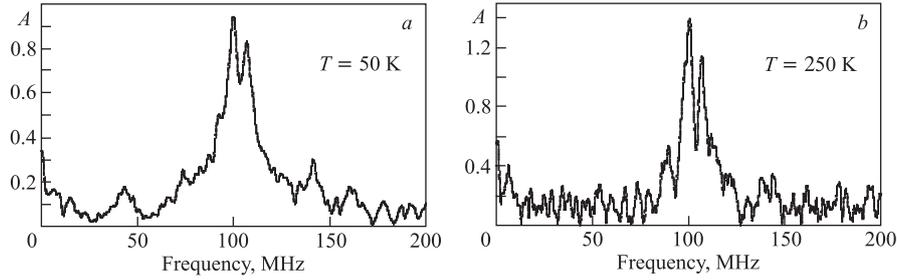


Fig. 2. The result of the Fourier analysis of the  $\mu\text{SR}$  histograms for diamond film D5 measured in the magnetic field 7.5 mT at the temperatures *a*) 50 and *b*) 250 K

The  $\mu\text{SR}$  spectra measured in the magnetic field 7.5 mT were fitted by (1) with polarization function (2) and values of the parameters  $P_\mu(0)$ ,  $P_T(0)$ ,  $R_T$ ,  $\nu_\mu$ ,  $\Omega_1$  and  $\Omega_2$  were found (see Table 1 and Table 2). It was found that  $R_\mu$  is equal to zero, and final values for other parameters were obtained by fitting the experimental data with fixed  $R_\mu = 0$ . The values  $\Omega_1$  and  $\Omega_2$  presented in Table 2 correspond to more precise results obtained at 250 K. Hyperfine constants were calculated as

$$A_{\text{hf}} = \frac{1}{2} \left[ \frac{(\nu_{23} + \nu_{12} + \nu_\mu)^2}{\nu_{23} - \nu_{12}} - (\nu_{23} - \nu_{12}) \right] = \left[ \frac{(\Omega_1 + \nu_\mu/2)^2}{\Omega_2} - \Omega_2 \right]. \quad (4)$$

**Table 1. Muon spin relaxation rate  $R_T$  (MHz) for the  $M_{uT}$  fraction in synthetic diamond at different temperatures**

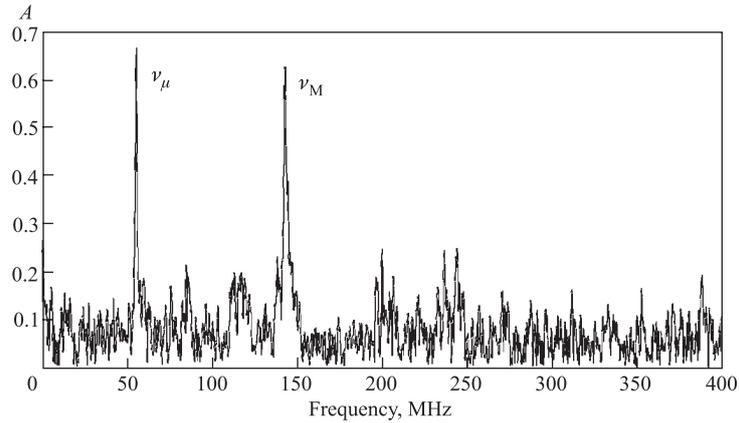
Sample	50 K	100 K	200 K	250 K
D3 (MSC)	19.1 $\pm$ 0.8			13.4 $\pm$ 0.9
D5 (CVD)	12.4 $\pm$ 0.6	17.3 $\pm$ 1.4	12.3 $\pm$ 0.6	7.7 $\pm$ 0.6

**Table 2. Muon spin precession frequency and hyperfine interaction constant  $A_{hf}$  for  $M_{uT}$**

Sample	$\nu_\mu$ , MHz	$\Omega_1$ , MHz	$\Omega_2$ , MHz	$A_{hf}$ , MHz
D3 (MSC)	0.996 $\pm$ 0.005	103.73 $\pm$ 0.12	2.95 $\pm$ 0.22	3715 $\pm$ 277
D5 (CVD)	0.992 $\pm$ 0.004	103.13 $\pm$ 0.06	2.75 $\pm$ 0.10	3940 $\pm$ 143

The values of the hyperfine constant for  $M_{uT}$  in samples D3 and D5 are smaller than for muonium in vacuum, and they are in agreement with the value  $A_{hf} = 3711 \pm 21$  MHz for natural diamond powder [8] within the accuracy of the measurements. However, substantial difference was observed in the behaviour of the muonium in synthetic diamond in comparison with the results for IIa-type natural powdered diamond [8]: 1) the muon spin relaxation rate  $R_T$  for samples D3 and D5 is approximately ten times higher than for natural sample; 2) in distinction to results [8],  $M_{uT}$  was observed at temperatures higher than 150 K, and at 250 K the relaxation rate decreases for  $M_{uT}$  fraction (see also Fig. 2). At the same time, the present results for the relaxation rate in synthetic samples D3 and D5 are close to those observed in [14] for IIa- and IIb-type natural diamonds.

Figure 3 shows the results of the Fourier analysis of the  $\mu$ SR-data measured by the Up telescope of the GPS spectrometer for mosaic single-crystal diamond



**Fig. 3. Muon spin precession frequencies observed in synthetic diamond D3 in the «magic» magnetic field 0.4073 T at 100 K**

sample D3 in the «magic» magnetic field 0.4073 T at 100 K. There are two peaks: one corresponds to the precession of the muon spin in the diamagnetic state and the other is due to the precession of the muon spin in the anomalous muonium in the «magic» magnetic field. The  $\mu$ SR histograms accumulated by means of the Up and Dw telescopes were fitted with polarization function (3) and values of the parameters  $R_{BC}$ ,  $\nu_\mu$  and  $\nu_M$  were extracted:  $R_{BC} = 2.1 \pm 0.5$  MHz,  $\nu_\mu = 55.224 \pm 0.005$  MHz and  $\nu_M = 142.8 \pm 0.1$  MHz. As for the normal muonium in synthetic diamond D3, the relaxation rate of the muon spin in anomalous muonium is an order of magnitude higher than that earlier observed in IIa-type natural powder diamond ( $0.25 \pm 0.10$  [8]).

Since in the  $\pi$ M3.2 beam line the angle between the muon spin and the muon momentum is  $\sim 70^\circ$  and the external magnetic field is parallel to the muon momentum, the  $\mu$ SR histograms accumulated by the Fw and Bw telescopes (see Fig. 1, *a*) contain information about evolution of the longitudinal (with respect to the magnetic field) component of muon polarization (LF measurement). In Fig. 4 the  $\mu$ SR histogram for sample D3 (MSC) accumulated by the Fw telescope at the magnetic field  $H = 0.4073$  T and  $T = 100$  K are presented on two different time scales. There are two components of muon polarization in the histogram: oscillating and nonoscillating. The Fourier analysis reveals only one frequency in the Fw and Bw  $\mu$ SR histograms which corresponds to muon spin precession on the  $Mu_{BC}$  in the «magic» magnetic field. Precession of the muon spin on the  $Mu_T$  in the magnetic field  $H = 0.4073$  T could not be observed because of the time resolution limit of the  $\mu$ SR setup. It is evident that the oscillating component of polarization is due to the  $Mu_{BC}$  fraction. At the same time, both  $Mu_T$  and  $Mu_{BC}$  fractions may contribute to the nonoscillating component of the Fw and Bw  $\mu$ SR histograms. These histograms were fitted by the polarization function

$$G(t) = P(0) \exp(-t/T_1) + P_{BC}(0) \exp(-t/T_1^{BC}) \cos(2\pi\nu_M t + \varphi), \quad (5)$$

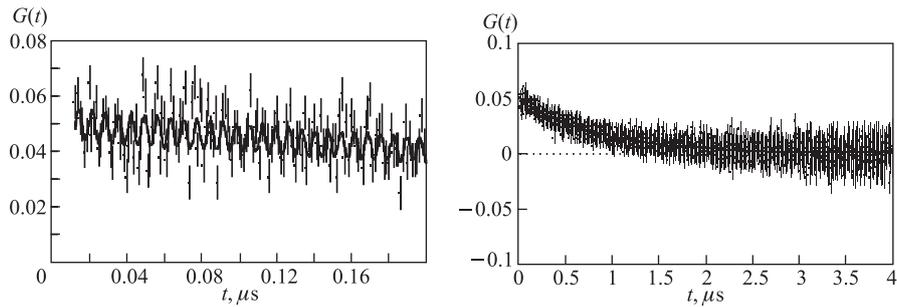


Fig. 4. The time dependence of the muon polarization in diamond D3 in the magnetic field 0.4073 T for the case of longitudinal field measurements (LF). The data are corrected for the muon lifetime  $\tau_\mu = 2197$  ns

where  $T_1$  and  $T_1^{\text{BC}}$  are the longitudinal relaxation times for the nonoscillating and oscillating components of muon spin polarization.

The following values of the muon spin polarization parameters were found:  $1/T_1 = 1.07 \pm 0.02$  MHz,  $1/T_1^{\text{BC}} = 1.14 \pm 0.14$  MHz,  $\nu_M = 142.68 \pm 0.06$  MHz. As was expected, the value of the  $\nu_M$  obtained from LF measurements is in good agreement with the results of the TF measurements (data accumulated by the Up and Dw telescopes).

To find the fraction of muon formation in the  $\mu$ ,  $\text{Mu}_T$  and  $\text{Mu}_{\text{BC}}$  states in diamond the values obtained for  $\alpha P_\mu(0)$ ,  $\alpha P_T(0)$  and  $\alpha P_{\text{BC}}(0)$  were normalized to the muon polarization for the silver sample measured under the corresponding experimental condition. It was found that for the Up, Dw and Fw telescopes of the Dolly spectrometer  $\alpha P_{\text{Ag}}(0)/3 = 0.270 \pm 0.001$  and for the Up and Dw telescopes of the GPS spectrometer  $\alpha P_{\text{Ag}}(0)/3 = 0.240 \pm 0.001$ . In the calculation it was taken into account that in the transverse magnetic field only 0.5 of the muon polarization in  $\text{Mu}_T$  and in «magic» magnetic field 11/30 of the muon polarization in  $\text{Mu}_{\text{BC}}$  [8] gives experimentally observable precession signal. The fractions of muons formed in the  $\mu^+$ ,  $\text{Mu}_T$  and  $\text{Mu}_{\text{BC}}$  states in diamond samples D3 and D5 are presented in Table 3 in comparison with the analogous data for various diamond samples studied earlier.

The data in Table 3 are divided into the three sets. The first set includes data for the Ia-type single-crystal and IIa-type powdered natural diamonds, the second set presents data for the IIa-type and IIb-type single-crystal natural di-

**Table 3. Fractions of muon states ( $\mu^+$ ,  $\text{Mu}_T$ ,  $\text{Mu}_{\text{BC}}$ ) and missing fraction (MF) observed in samples of natural (n) and synthetic (s) diamonds. SC is single-crystal diamond, and MSC is a sample composed from a few single crystals, CVD is chemical vapor deposited diamond film**

Sample		$T$	$\mu^+$ (%)	$\text{Mu}_T$ (%)	$\text{Mu}_{\text{BC}}$ (%)	MF (%)	Ref.
Powder IIa	n	4.2–90	< 10	18.5(0.9)	9.9(0.7)	> 60	[8]
Powder IIa	n	296	< 5	—	14.5(1.3)	> 80	[8]
SC Ia	n	4.2	< 10	20(4)	11.9(0.9)	> 60	[8]
SC Ia	n	5–270	4(1)	0	—	—	[15]
SC IIa	n	300	8.1(3.0)	68.9(1.0)	22.7(0.8)	0.3(3.3)	[9]
SC IIa	n	5–300	6(1)	61(4)	26(3)	7(8)	[14, 15]
SC IIb	n	5–300	14(4)	53(4)	26(3)	7(6)*	[14, 15]
CVD	s	10–300	$\simeq 6$	$\simeq 50$	$\simeq 10^*$	$\simeq 34^*$	[15]
CVD	s	4.5	19.4	50.2	6.8	23.6	[16]
SC IIa	s	5	4	54	30	12	[18]
CVD	s	50–250	0.8(0.1)	60(1)	—	—	**
MSC IIa	s	100	1.5(0.1)	57(4)	8.1(0.4)	33.4(5.0)	**

\*We estimated according to the data presented in original publication.

\*\*The present data.

amonds and the third set is for the synthetic diamond. The  $Mu_T$  fraction is minimal and the missing fraction is maximal for the first set of samples. For the second set of samples the missing fraction of muon polarization is close to zero. In the synthetic samples the  $Mu_{BC}$  fraction is 2–3 times smaller than in the IIa- and IIb-type single-crystal natural diamonds. The large missing fraction in the IIa-type powder sample (with grain size 1–6  $\mu\text{m}$ ) was explained by diffusion of the  $Mu_T$  to the crystalline boundaries [7].

It was experimentally shown that a large missing fraction of the muon polarization in the Ia-type diamond is due to one more muonium state in the nitrogen-rich diamond [16, 17]. As is well known, Ia-type diamonds contain about 0.1% (1000 ppm) nitrogen impurities and they are aggregated to large defects forming A and B centres. At A centres two nitrogen atoms occupied two nearest substitution sites. The calculation given in [17] shows that the muonium may occupy the bond centre site between the neighbouring nitrogen atoms in the case of A-type defects and the site between the nitrogen molecules in the case of the B-type defects.

The concentration of nitrogen atoms in the IIa- and IIb-type natural diamond is approximately three orders of magnitude lower than in Ia-type diamond. The fact that the missing fraction of muon polarization in the IIa- and IIb-type natural diamond is close to zero indicates that nitrogen does not play any noticeable role in formation of muonium fractions in these samples.

In the synthetic diamond the concentration of nitrogen is close to that in the IIb- and IIa-type natural diamond. Therefore it is reasonable to compare the results for the samples studied in this work with those for IIa- and IIb-type natural diamond. As is seen from Table 3, in the synthetic samples the  $Mu_T$  fraction is comparable, the  $Mu_{BC}$  fraction is 2–3 time smaller and the missing fraction is a few times larger than in the IIa- and IIb-type single-crystal diamonds. The fact that the missing fraction in the synthetic diamond is 2–3 times smaller than in the nitrogen-reach Ia-type natural sample and 2–3 times larger than in the IIa- and IIb-type samples indicates presence of some other defects in the synthetic diamond in comparison with the natural one. The observed difference may be due to the high concentration (more than 10 ppm) of incorporated hydrogen and due to a small crystalline size (a large effective surface) in the CVD films and due to the presence of Ni includes in sample D3 (MSC).

## SUMMARY

It is found that the hyperfine constant for interaction of the muon and the electron magnetic moments on  $Mu_T$  and  $Mu_{BC}$  in the synthetic diamond is close to those in the natural diamond. The longitudinal relaxation rate of the muon spin in the  $Mu_{BC}$  state is measured for the first time:  $1/T_1^{BC} = 1.07 \pm 0.02$  MHz.

The  $\mu_{\text{T}}$  fraction of the muon polarization and the relaxation rate  $R_{\text{T}}$  for the synthetic samples are comparable with those for the IIa- and IIb-type natural diamond.

The fact that: 1) the missing fraction of the muon polarization in the synthetic samples is 2–3 times smaller than in the Ia-type natural sample and 2) the  $\mu_{\text{T}}$  fraction and the relaxation rate  $R_{\text{T}}$  for the synthetic samples is comparable with those for the IIa- and IIb-type natural diamond may be considered as a sign for «good» perfectness (for low concentration of defects) of the synthetic samples.

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