Experimental report

Proposal:	**3-07-386**

Council: 10/2018

Title:	Studies of nano-diamond reflectorsfor slow neutrons at PF2, PF1B, D17 and D11 instruments
Research area:	Nuclear and Particle Physics

This proposal is a new proposal

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Samples: nano-diamonds

Instrument	Requested days	Allocated days	From	To
PF2 EDM	0	30		
D17	2	0		
PF2 UCN	30	0		
PF1B	5	5	05/02/2020	10/02/2020
D11	1	1	18/09/2019	19/09/2019

Abstract:

We propose several experiments at instruments PF2, PF1B, D17, and D11 focused on studies of nano diamonds (NDs) as reflectors for very cold neutrons (VCNs) and cold neutrons (CNs) also as probes for small heating of ultracold neutrons (UCNs). We combine them in one proposal because of their strong interplay. Information obtained in these experiments will contribute to the solution of common physical problems. The experiments include: measurement of the reflectivity of UCNs from designed NDs also small heating of UCNs at designed NDs (with BGS spectrometer at PF2), measurement of quasi-specular reflection of CNs from designed NDs and measurement of neutron transport cross-sections for designed NDs and intercalated graphite (D17), measurement of small-angle neutron scattering at designed NDs (D11), and measurement of impurities in NDs using neutron-activation and prompt-gamma analysis (PF1B). The results expected within this program are highly complementary. The principle goal of this series of measurements is further improvement of the efficiency of NDs reflectors for slow neutrons and studies of NDs mobility on NDs surface and in the powder bulk.

Note: this experiment was initially scheduled at 3 instruments: PF2, D11, PF1B. However, due to the absence of a respective seismic protection of the PF2 platform, it could not be performed there. The measurement at D11 is presented in great details in two publications [**Aleksenskii, A., *et al*,** *Clustering of diamond nanoparticles, fluorination and efficiency of slow neutron reflectors*, **Nanomaterials 11** (2021) 1945], [**Aleksenskii, A., *et al*,** *Effect of particle sizes on the efficiency of fluorinated nanodiamond neutron reflectors*, **Nanomaterials 11** (2021) 3067]; therefore, we do not repeat this information here, and just refer the readers to these publications. The rest of the report is devoted to the short test measurements of the elemental analysis of nanodiamond powders designed for the slow neutron reflectors using the neutron prompt-γ activation analysis method at PF1B.

Motivation: Slow neutrons, in particular cold and very cold neutrons (CN, VCN) are a powerful tool for fundamental and applied research. Better VCN and CN sources can improve the efficiency of these studies and broaden their scope. Therefore, new methods and technologies for building more intense CN and VCN sources are intensively developed. Some of them are based on slow neutron reflectors made of detonation nanodiamond (DND) powders.

Due to their exceptional properties, such as small absorption cross section of carbon $\sigma_{abs}^{C} = 3.5$mb (for thermal neutrons) and high optical potential of diamond $E_{lim}^{C} = 305$neV (at the density of $\rho = 3.5 g/cm^3$), DND are an excellent material for designing new-type reflectors for low energy neutrons. Because of the huge and chemically active surface of DND, there can be various extra admixtures on the surface. Such admixtures can be a reason of extra losses of slow neutrons. For instance, the presence of hydrogen atoms largely decreases the efficiency of neutron reflection because of the huge inelastic scattering cross section ($\sigma_{in.sc.}^{H} = 108 \pm 2$bn at room temperature) and significant absorption cross section ($\sigma_{abs}^{H} = 0.33$bn).

The goal of present experiment was to explore the admixture of hydrogen atoms in various DND samples using the method of neutron prompt-γ activation analysis (PGAA – Prompt Gamma Activation Analysis).

The experimental setup: The experimental setup for neutron prompt-γ activation analysis was installed in the experimental zone of PF1B instrument (Fig. 1).

Fig. 1. A scheme of the PF1B instrument.
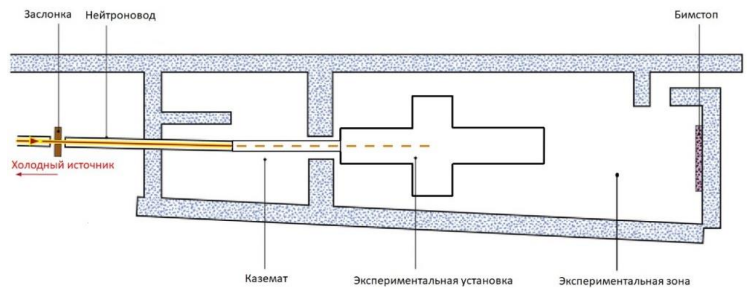
The instrument scheme is simple. It includes an experimental zone, a casemate, a valve and a ballistic super-mirror curved neutron guide. The setup scheme is shown in Fig. 2 and consists of a few elements: a collimation system, a vacuum channel, an aluminum sample holder, a high-purity-germanium (HPGe)-detector №1, a HPGe-detector №2, Compton anti-coincidence shielding of the detector, detector shielding against external backgrounds, shielding against neutrons scattered in the sample.
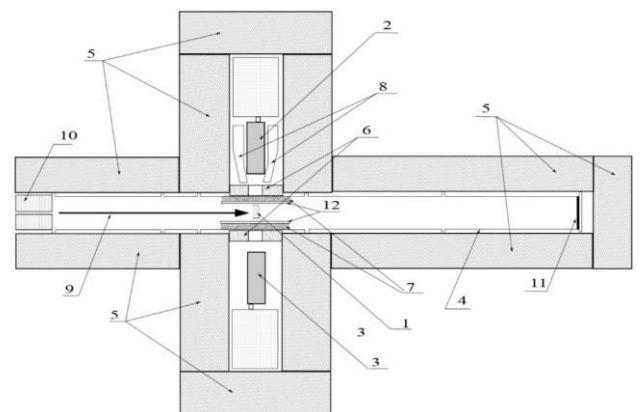
Fig. 2. A scheme of the experimental setup. 1 – a sample, 2 – HPGe detector №1, 3 – HPGe detector №2, 4 – a vacuum channel, 5 – lead

shielding, 6 – a γ-quanta collimator, 7 – LiF shielding, 8 – Compton shielding, 9 – a neutron beam, 10 – a neutron collimator, 11 – a beam stop, 12 – an aluminum sample holder.

Detector windows are placed at a distance of ~18 cm from the neutron beam axis and ~80 m from the reactor active zone. Between the sample and the detectors, there are Bi-collimators with the thickness of 5 cm and the hole diameter of 5 cm. The outer surface of the lead shielding is protected against external neutrons with boron rubber (with the thickness of 5 mm). The samples are placed in the neutron beam inside the vacuum channel and are held with the aluminum holder.

The detector and the shielding against scattered neutrons: γ-quanta are detected in the Canberra HPGe detectors with the efficiency of ~30%. The detectors are placed on the opposite sides from the sample at the distance of ~18-20 cm from the beam axis. The HPGe γ-detectors and aluminum tube are surrounded with a lot of γ- and n- shielding consisting of lead, bismuth and boron rubber. In front of the detector, there is shielding with the length of 44 cm made of baked lithium fluoride LiF. It consists of 3 sections. Each section is assembled of 4 plates. Except for one plate in the central section, all other plates are made of LiF with the natural composition. One plate in the middle section located between the sample and the detector is made of lithium fluoride with ~90% enriched Li^6. γ-quanta emitted from the sample penetrate easily through the 5-mm-thick plates in the central section and reach the detector, while the enriched Li^6 has a 13 times larger cross section for neutrons than the natural lithium. One of the detectors was surrounded with Compton shielding in order to suppress Compton γ-quanta thus improving the signal/noise ratio. It consists of germanium-bismuth (BGO) scintillators surrounding the HPGe detector with all sides except for the directions of γ-quanta from the sample and the HPGe detector mounting; this is so-called co-axial shielding.

Detonation nano-diamond samples. The samples were commercially available detonation nano-diamonds subdivided into two groups. First, using the technology of Federal state unitary enterprise "RFNC ARSIITP E.I. Zababakhina" in accordance with the technology described in TR 2-037-677-94 (DND – raw powder, F-DND – fluorinated DND, DF-DND – deagglomerated fluorinated DND). Second group consists of samples produced by different enterprises: TNT-RDX, T9, FN-01, DND-Z, F-Altay, B-HF, Microdiamond 30. The full list is given in Table 1:

Samples	
1-group	2-group
DND	F-Not DND,
F-DND	F-Altay
DF-DND	Microdiamond 30
	T-9
	B-HF
	DND_Z$^-$
	TNT-RDX
	FN_01

A sample holder is installed inside Li^6 enriched lithium fluoride shielding, which is, in its turn, is located inside the vacuum tube; it is designed to minimize its interaction with the neutron beam. It is a square aluminum frame with the sides of 55 mm (Fig. 3). Sample powders in Teflon pockets are fixed in the center of the aluminum frame using stretched thin wires.

Fig. 3. The holder with a sample in a Teflon pocket.

Sample preparation. Sample powders are inserted inside Teflon (C_2F_4) pockets in order to reduce backgrounds
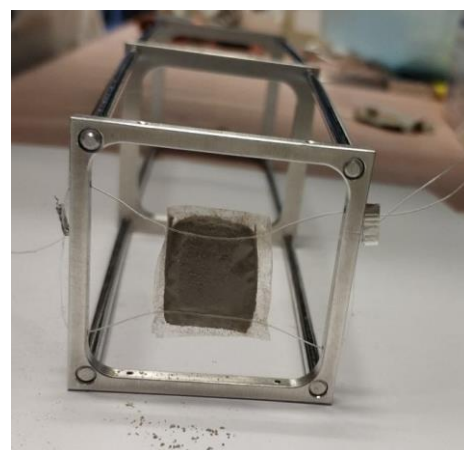
associated with hydrogen. The holder and sample positions are adjusted relative to the neutron beam and HPGe detectors using a laser (Fig. 4). To provide equal conditions, all samples were irradiated in the same positions at the same conditions. To avoid neutron scattering on the air, the samples were placed in the vacuum tube.

Fig. 4. Left: adjustment of the sample position. Right: the sample.
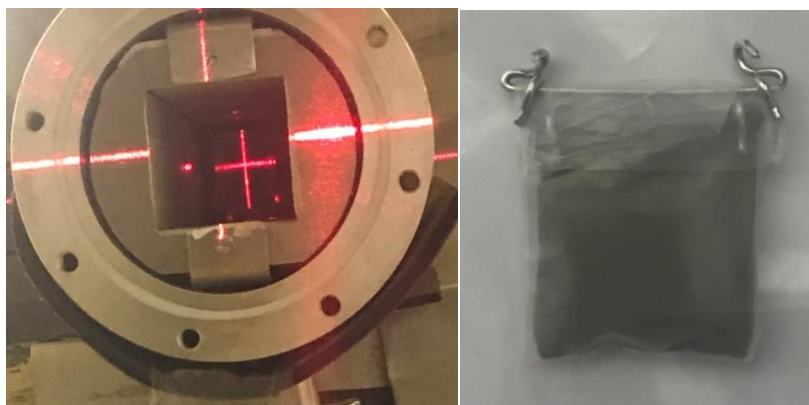
Results. The exposition durations were from 15 minutes to 5 hours; the statistics accumulation time was sufficient due to the exceptionally high neutron flux of $2 \cdot 10^{10}$ n/cm^2/s.

The results, in atomic ratios, are summarized in Table 2:

Sample	Time, s	Mass, mg	Count rate, s^{-1}, in the H peak at 2223 keV	Count rate, s^{-1}, in the C peak at 4945 keV	C/H (at.)
Полиэтилен,	1545	17.8	287±31	14.2±30	0.5
Исходный -DND	2776	20.9	8.75±20	14.02±26	16.2±58.7
F-DND	16020	62.2	10.67±20.5	16.9±26	16±52
F-Altay	2689	105	2.71±15	19.41±27.2	72.3±440
F-Not Snezh	7604	41.6	3.47±20	15.7±26.6	45.7±291
30nm-poly	17264	146.3	66.1±25.6	19.63±29.4	3±7.9
T-9	900	37	18.57±21	14.53±26.4	7.9±23.8
Batsanov	20890	74.6	13.12±21.2	19±26.7	14.6±44
DND_Z	5704	55.6	24±22	16.3±27.5	6.8±49.6
TNT-RDX	2566	19.5	10.51±20.7	14.7±26.5	14.13±48.1
FN_01	2571		19.82±21.6	16±27	8.15±23.8
DND-F_D01	3247		5.86±20.7	16.27±26.5	28.05±124

As you can see from the last three columns in the table, the accuracy of determination of the H fraction is not sufficient both when using the normalization to the carbon peak and without it. The experiment has to be improved and repeated. The optimum method of improvement of the experiment is further background reduction. Otherwise, the sample mass can be increased. However, some of the samples are available only in small amounts and therefore they have to be refabricated.