Project Activity Report

DEVELOPMENT OF PTH SAMPLE ENVIRONMENT SYSTEM FOR THE DN-12 DIFFRACTOMETER AT THE IBR-2 FACILITY

THEME: Development of Experimental Facilities for Condensed Matter Investigations with Beams of the IBR-2 Facility

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Annotation

The sample environment cryomagnetic system has been developed and designed for the DN-12 diffractometer at the IBR-2 facility with a superconducting magnet in cryostat and a cryostat-insert with a high-pressure chamber on a sample.

The magnet is produced in the Helmholtz pair configuration with a maximum magnetic field of 5 T. A 12 mm-wide and 0.1 mm-thick high-temperature superconducting (HTSC) tape with a compensation layer of copper is used as a superconductor. The magnet has a bore with a diameter of 80 mm and is arranged in the vacuum cryostat. The magnet is cooled using a closed-cycle cryocooler (a dry cryostat, without liquid helium), the second stage of the cryocooler is joined to the magnet body by aluminum flexible thermal bridges. An electric current of up to 300 A is supplied to magnet windings in the low-temperature area of HTSC by current leads, and in the area between room and nitrogen temperatures - by the use of copper bars. A sealed shaft is led in the bore of the magnet, into which a cryostat-insert with a high-pressure chamber and a test sample is inserted. The sample chamber is cooled by the second closed-cycle cryocooler. The temperature of magnet windings is defined in the range of 40 – 20 K and depends on the temperature of high-pressure chamber on the test sample which is 270 - 4.5 K. The cryomagnetic system can be arranged both vertically and horizontally. This cryostat is designed to operate on the DN-12 diffractometer at the IBR-2 facility. It will be installed horizontally inside the detector ring.

The activities have been carried out at the Frank Laboratory of Neutron Physics of JINR (Dubna, Russia) in cooperation with the National Institute for R&D in Electrical Engineering ICPE-CA (Bucharest, Romania).

Introduction

An essential task of the condensed matter physics is the research of structural devices for developing magnetic states in various types of complex magnets. Information for their establishment can be obtained in the result of simultaneous studies of the crystal and magnetic structure with variations in temperature, pressure and magnetic field.

The IBR-2 spectrometers are sufficiently equipped with the cryostats based on the closed-cycle cryocoolers. They allow to vary the temperature on samples within the range of 300-4 K. An operating procedure with high-pressure chambers up to 10 GPa has been developed on these cryostats [1]. However, up to present, on spectrometers there have been no devices of the sample environment with high-pressure chambers with possibly varying temperature and magnetic field, the development of which is an urgent task.

Magnetic studies impose restrictions on the design of the magnet using neutron scattering. It must consist of a pair of similar cylindrical coils arranged on the same axis (“Split”-type magnetic system or “Helmholtz pair” configuration). Such geometry, firstly, allows to input the sample in the area of the magnetic field without depressurization of the cryostat and its partial disassembly, and secondly, in the process of directing the magnetic field vector towards the beam, it provides scattering perpendicularly; in this geometry scattering is also provided forward at an angle of 45° towards the beam.

The overall sizes of the spectrometer, obviously, limit the sizes of the magnet and its maximum possible field. Still, as calculations have shown, the dimensions of DN-12 allow to install a cryostat with a superconducting magnet providing a field of up to 5 T with a bore of 80 mm in diameter, into which a high-pressure chamber, heat-sealed from magnet coils, can be inserted.

Also, the fundamental point in the design of the cryostat cooled by the closed-cycle cryocooler is the possibility of arranging it horizontally, which is due to the arrangement geometry of the detectors detecting scattered neutrons perpendicularly towards the axis of the beam falling on the sample. At present, such geometry at IBR-2 is possessed by the diffractometers DN-12 and DN-6 [2], [3].

At IBR-2 spectrometers, for cooling samples to temperature with an order of 4 K, closed-cycle cryocoolers in vacuum cryostats are used. Using cryocoolers is a more efficient and economic mode compared to using helium cryostats. Especially for this reason, it has been decided to design a superconducting magnet based on the HTSC tape from YBCO, cooled by a closed-cycle cryocooler. The chosen design of the cryostat with a magnet temperature of 40 – 20 K is essentially simpler compared to the design of the helium cryostat, required for the operation of the superconducting magnet made of superconducting Nb-Ti cable.

Thus, the aim of this project is to develop and design a sample environment device with a high-pressure chamber with varying temperature and a magnetic field (cryomagnetic system) for the DN-12 diffractometer. In addition to the studies of samples under high external pressure, it will be possible to provide temperature and magnetic conditions on the sample in wide ranges, which will significantly enhance the research capabilities of this facility.

Description of the cryomagnetic system

A scheme drawing of the detector ring of the DN-12 diffractometer is presented in **Fig. 1**; a layout of the cryostat positioned horizontally on the DN-12 diffractometer is shown in **Fig. 2**; a 3D model of the cryostat is shown in **Fig. 3** and a scheme drawing of the cryostat is presented in **Fig. 4**.

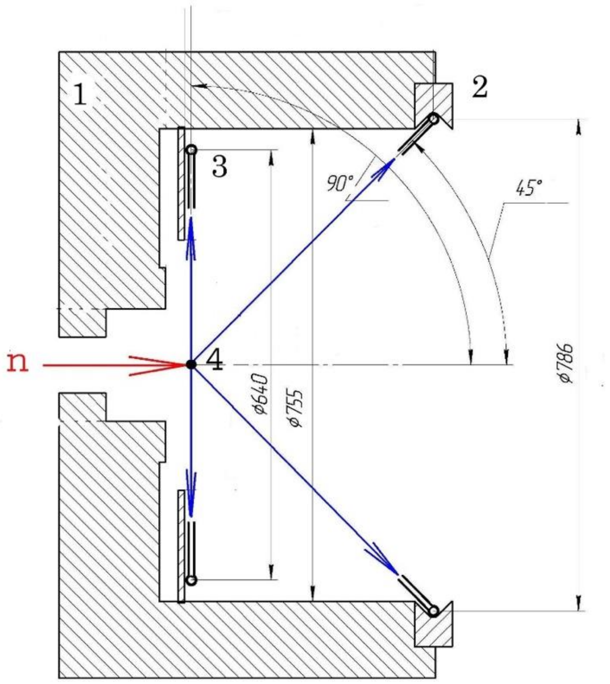

Fig.1 Scheme drawing of positioning of the detector rings on the DN-12 diffractometer.
1 - biological and background shield; 2 - detectors directed towards the scattering at an angle of 45^0; 3 - detectors directed towards the scattering at an angle of 90^0; 4 - sample.

The area, where the detector rings of the diffractometer are positioned, is bounded on all sides by concrete shield, which does not allow one to use a vertical cryostat and therefore, practically eliminates the usage of helium cryostats. An additional complexity is the fact that the detector rings are in a special access area, which has predetermined the application of closed-cycle cryocoolers to solve the problem.

The fundamental point is that the magnet coils are cooled by one cryocooler (RDK408S), and the high-pressure chamber is cooled by another cryocooler (RDK101D). The second cryocooler is the forming element of the cryostat-insert in the shaft of the magnetic cryostat, which is inserted into the axial bore of the magnet.

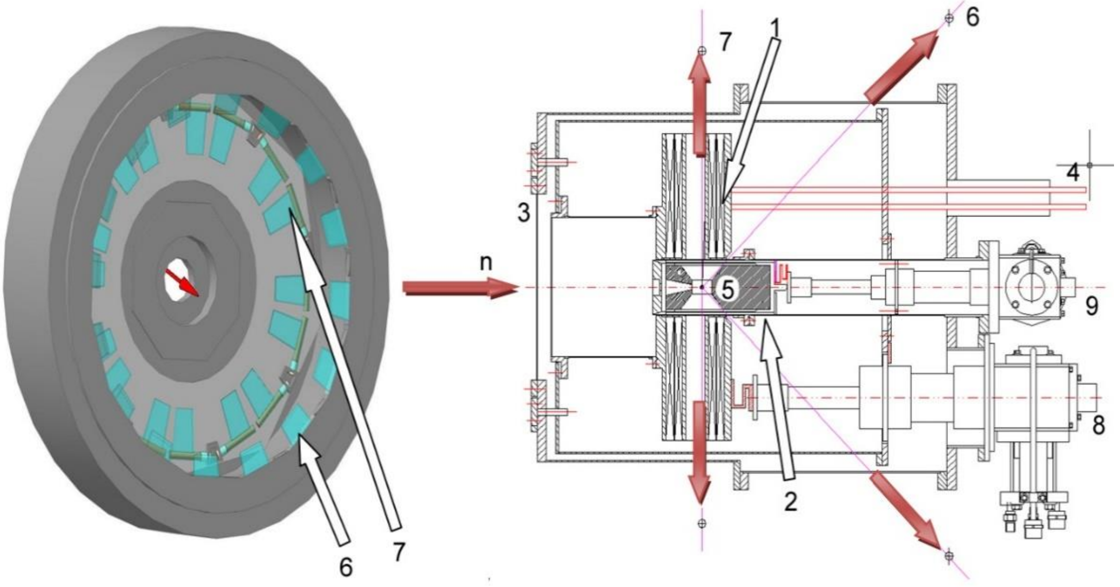

Fig. 2. Schematic of the cryomagnetic system with a superconducting magnet in the cryostat and an extra cryostat-insert, and detector rings of the DN-12 diffractometer.

1 - superconducting magnet; 2 – cryostat-insert; 3 - entrance window for neutrons; 4 - current leads for the current up to 300 A; 5 – high-pressure chamber; 6 and 7 - rings of scattered neutron detectors; 8 - cryocooler RDK408S; 9 - cryocooler RDK101D.

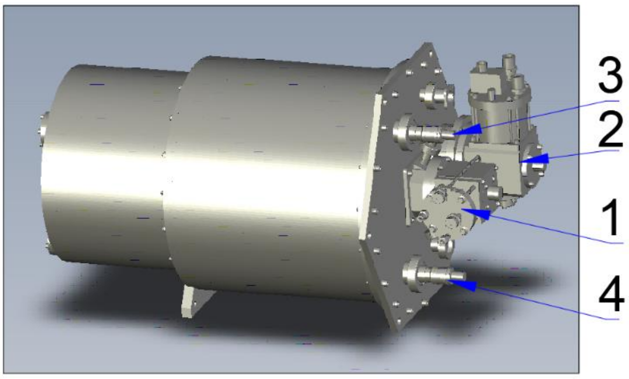

Fig. 3. 3D model of the cryomagnetic system.

Main elements: 1 - cryocooler RDK101D; 2 - cryocooler RDK408S; 3 and 4 - current leads.

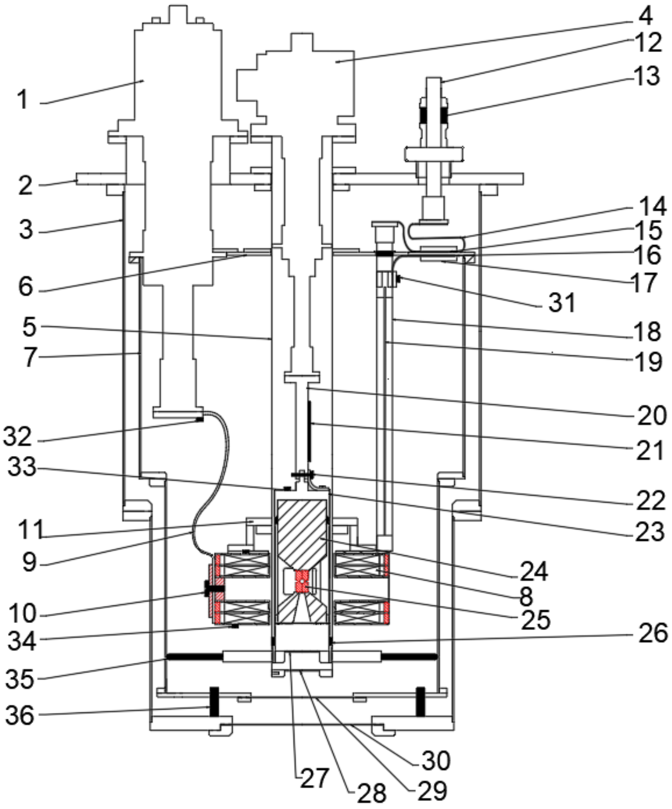

Fig. 4. Scheme drawing of the cryomagnetic system.

1 - cold head of the cryocooler SRDK408S; 2 - flange of the magnet cryostat; 3 - vacuum aluminum cryostat housing; 4 - cold head of the cryocooler SRDK101D; 5 - stainless steel shaft with a diameter of 80 mm; 6 - copper flange in thermal contact with the first stage of the cold head of the cryocooler SRDK408S; 7 - aluminum thermal shield; 8 - magnet coils; 9 - flexible aluminum heat conductor; 10 - thermal contact on a bolted connection; 11 - magnet holder; 12 – power feedthrough to the cryostat; 13 - insulator; 14 - copper current lead; 15 and 16 - insulators; 17 - thermal contact; 18 - HTSC current lead; 19 - two 12 mm-wide HTSC tapes; 20 - copper heat conductor; 21 - heater; 22 - hinge; 23 - sample chamber; 24 - presser; 25 - anvils; 26 – spacer insulators; 27, 28, 29,30 - thin-walled windows for the entrance of neutrons; 31, 32, 33, 34 - thermometers; 35, 36 - textolite stoppers.

The cryostat contains a cryocooler SRDK408S, the cold head of which is positioned on the flange of the magnet cryostat. The vacuum cryostat housing has sizes adapted to both the magnet itself and the detector ring of the diffractometer. The cold head of SRDK101D is positioned in a sealed shaft – a thin-walled pipe with a diameter of 80 mm. The magnet coils are protected by a thermal shield, which is in thermal contact with the first stage of the cold head of SRDK408S. The magnet coils are cooled by the second stage of this cryocooler via aluminum heat bridges, which are connected to the magnet coil housing by bolt joints. The magnet coils are produced from HTSC tape with a copper compensation layer; the width of the tape is12 mm, and thickness is 0.1 mm. The magnet itself is mounted on the shaft using holders. The shaft in its lower part is made of aluminum. A current of up to 300 A is carried in the cryostat via feedthroughs with an insulator, and then it passes through a copper current lead - a bar 400 mm long, 30 mm wide and 1 mm thick. This bar has a pressed thermal contact through insulators which are mounted on a copper flange that is in thermal contact with the first stage of the cold head of the cryocooler SRDK408S. Further, the current enters the magnet coils through HTSC current leads, which contain two HTSC tapes of the same type the magnet coils are made from. The sealed chamber of the sample is cooled by the cryocooler SRDK101D, to the second stage of the cold head of which a copper pipeline with a heater is attached. The high-pressure chamber with a sample, in its turn, is attached to this heat conductor using a swivel joint and a flexible thermal bridge. The sample is positioned between the anvils of the chamber, which are compressed by a presser. In our case, the presser and the anvil are produced from non-magnetic materials. Neutrons reach the sample through thin-walled windows. Temperature at several points is controlled using the CERNOX thermometers, which are insensitive to the magnetic field. For operating the cryomagnetic system in a horizontal position, the design provides textolite stoppers, which will not allow the cryostat shaft and the thermal shield to bend forward relative to the axis of the magnet and cryostat.

Photo of the internal part of the cryostat, namely the HTSC magnet is shown in **Fig. 5**.

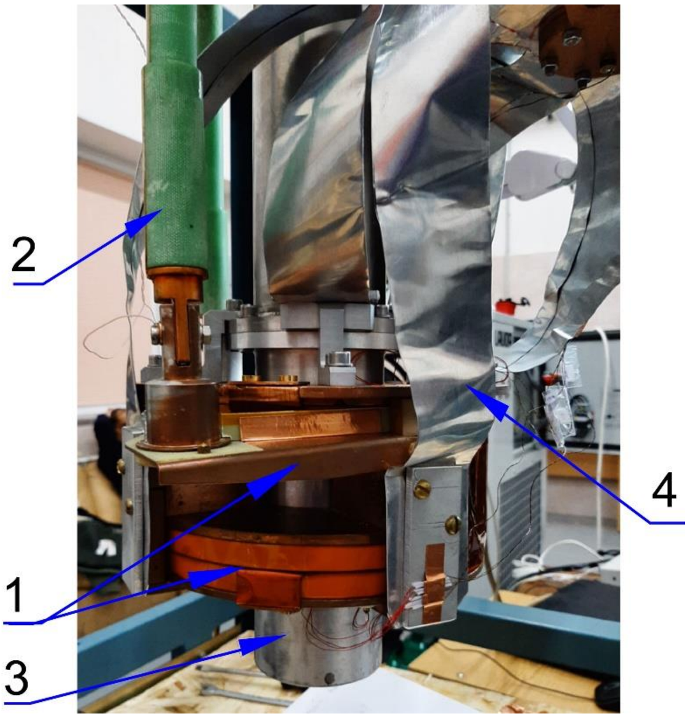

Fig. 5. Internal part of the cryostat.

1– split-system of two magnet coils, produced on the basis of HTSC tape; 2 - HTSC current leads for current injection up to 300A; 3 - shaft with a diameter of 80 mm for positioning a high-pressure chamber in it; 4 - tapes from pure aluminum, which are thermal bridges between the coil and the second stage of the cryocooler SRDK408S.

The photo of a cryostat positioned on a horizontal stand during testing in the Department of Spectrometers' complex IBR-2 is shown in **Figure 6**. It should be noted that the developed cryostat is an autonomous system depending only on the source of the electrical power. The cooling system of the cryocooler compressors and the superconducting magnet power supply is provided by the "Lauda" closed-circuit water chiller.

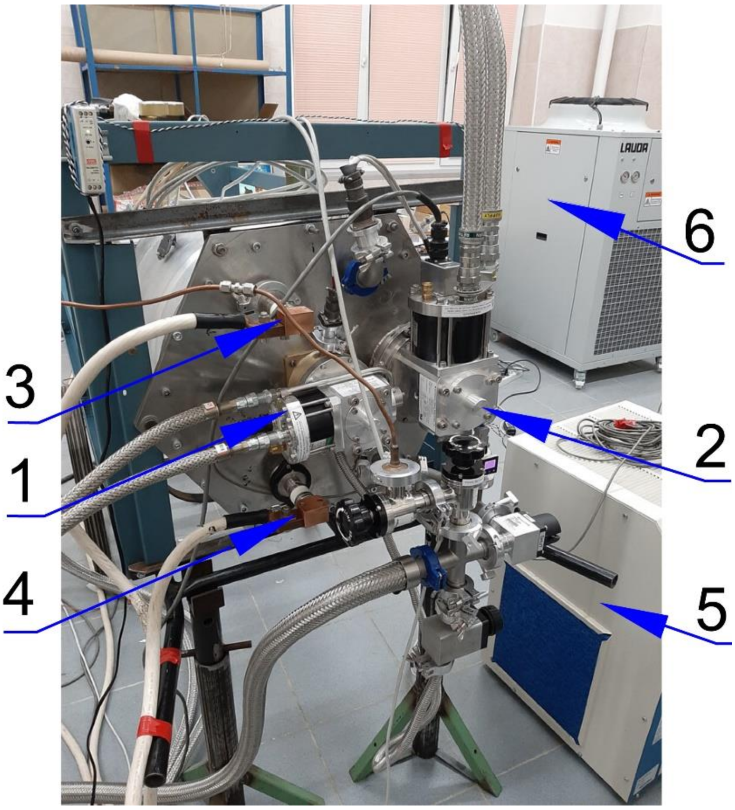

Fig. 6. General view of the cryomagnetic system in a horizontal position.
1 - cold head of the cryocooler SRDK101D – of the cryostat insert; 2 - cold head of the cryocooler SRDK408S, which cools the magnet; 3 and 4 - magnet power supply current leads; 5 - one of the cryocooler compressors; 6 - "Lauda" chiller, which cools cryocooler compressors and magnet power supply.

Test results

In the tests which were carried out on magnet, the magnetic field was measured in the center of the coils in the axial direction, depending on the value of the electric current. As a result, a characteristic of the magnet was obtained which is shown in **Fig. 7**.

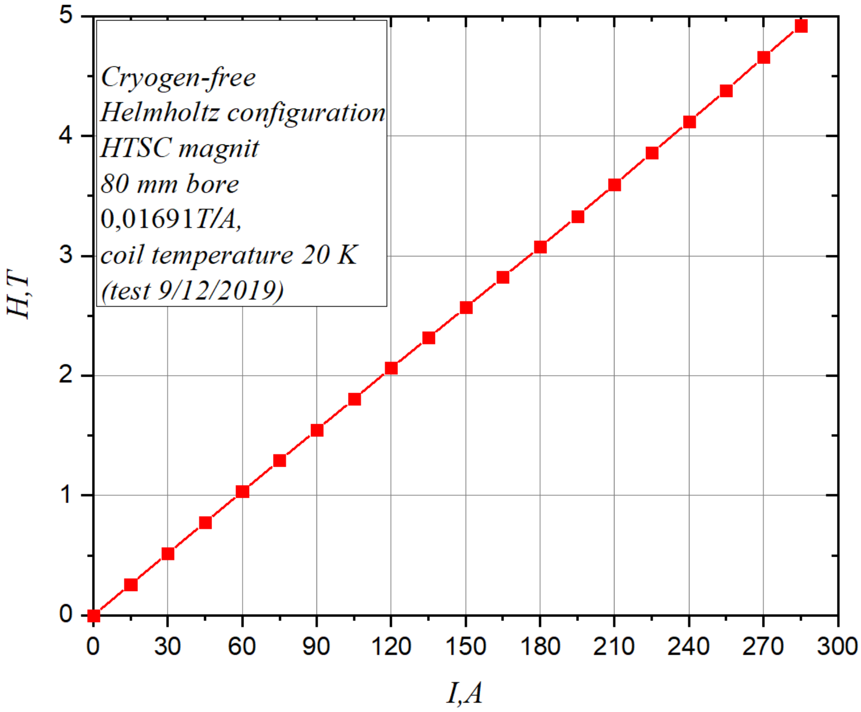

Fig. 7. Magnetic field depending on the value of the electric current inserted into the magnet coils. This insertion shows the main parameters of the magnet: liquid-free cooling; coil configuration - Helmholtz pair; a magnet bore with a diameter of 80 mm; magnet constant 0.01691T/A in the center; minimum coil temperature having reached 20 K.

An essential characteristic of the cryostat is the temperature interdependence of the cryostat-insert and magnet coils, which determines the upper temperature of the high-pressure chamber at the nominal value of the magnetic field.

Figure 8 shows the temperature dependence of the magnet coils on the temperature of the sample chamber.

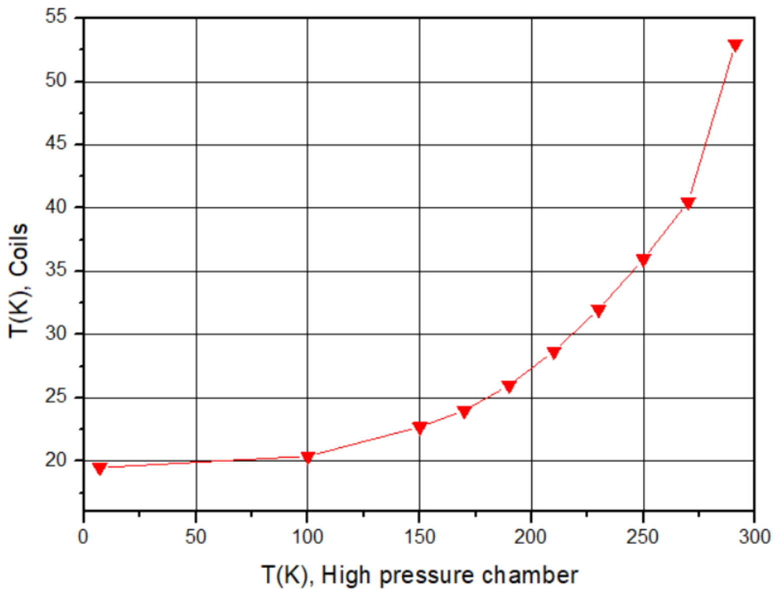

Fig. 8. Temperature of the magnet coils depending on the temperature of the sample chamber.

From the dependence on measurement of the lift-factor — the ratio of the critical field value at low temperature to the critical field value at a temperature of 77 K towards the perpendicular plane of the tape (see Appendix, **Fig. 11**), it follows that a field of 5 T is already reached at a temperature of 50 K (according to the technical design assignment for manufacturing of the magnet, the required maximum field value is 4T). Comparing the graphic chart shown in **Fig. 8** to the chart presented in the **Appendix**, it can be argued that with a magnetic field of 4 T, the temperature of the sample chamber will not be lower than 275 K, while the temperature of the magnet coils does not exceed 45 K. It also follows from **Fig. 8** that the minimum temperature of the sample chamber is 4.5 K and is limited by the residual thermal coupling of the magnet coils and the sample chamber.

An essential characteristic of the magnet intended for structural studies of a solid is the uniformity of the magnetic field, which must be at least 1% in the area in which the sample is positioned. **Figure 9** shows the magnetic field measured along the axis of the magnet (or the direction of the field vector) at 3 T. The change in the magnetic field between the peaks is no more than 0.32%, and the area of this uniformity along the axis is 30 mm. In **Fig. 9** the position of the sensitive element of the Hall sensor is shown, marked along the Y axis. The coordinate Y = 0 corresponds to the position of the Hall sensor in the upper part of the magnet (see **Fig. 4**). The coordinate Y = 80 mm corresponds to the center of the magnet.

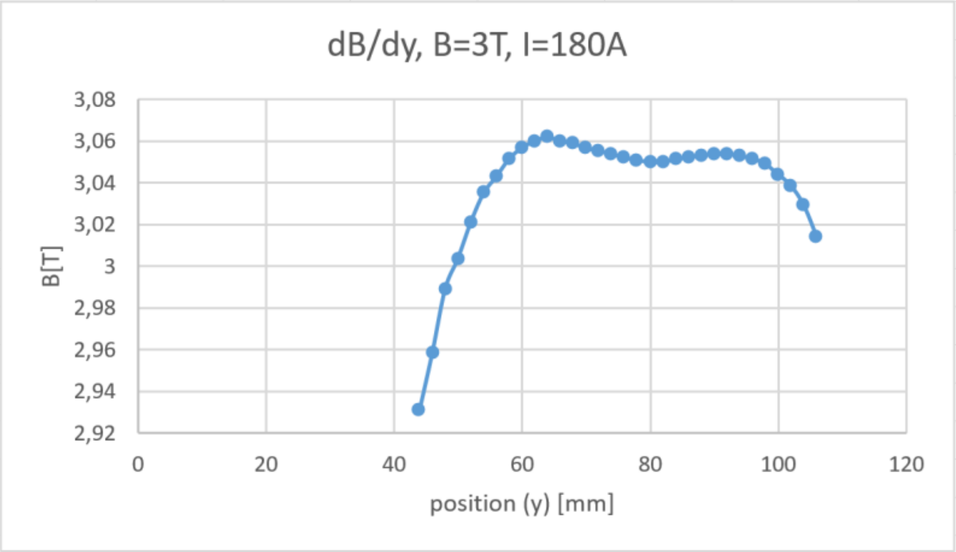

Fig. 9. Distribution of the magnetic field along the axis of the magnet.

As we can see, the sample in the high-pressure chamber has a characteristic size of 1x1x1 mm^3. However, in the case of studying larger samples, it is also necessary to know the sizes of the field uniformity in the cross section perpendicular to the axis of the magnet. **Fig. 10** shows the calculated magnetic field along the radial coordinate in a plane perpendicular to the axis of the magnet at an equal distance from the poles. As it can be seen from the chart, the field uniformity of 1% is maintained within a circumference with a diameter of 20 mm. Thus, the maximum sizes of the samples can reach 20 mm perpendicular to the beam axis and 30 mm along the beam axis.

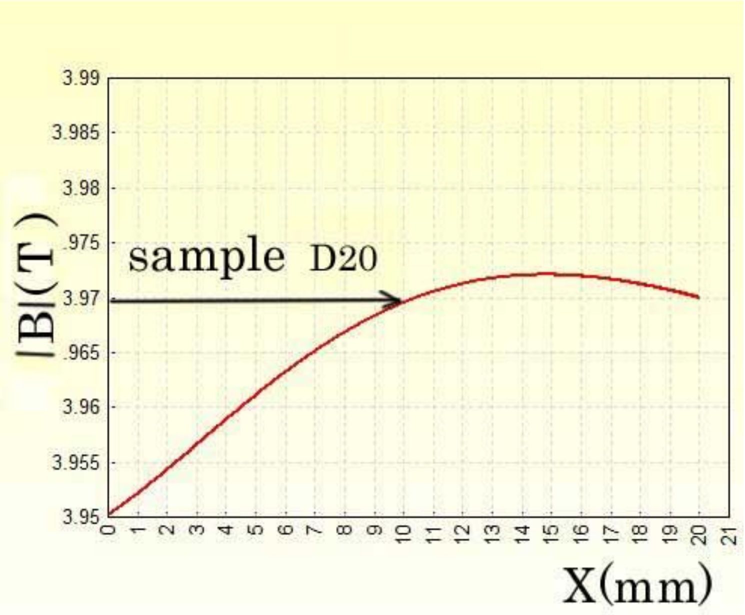

Fig. 10. The calculated magnetic field perpendicular to the axis of the magnet in a plane equidistant from the poles.

1. Work stages and project costs

Work under the project has been carried out in several general stages:

- design of a cryomagnetic system;
- calculation of magnet and cryostats;
- purchase of equipment;
- manufacturing of a cryostat and magnet;
- assembly and adjustment;
- execution of test measurements.

The work was completed to the full extent during the period 2015-2017. However, during the tests it was found out that the magnet manufactured by us passed into a normal state - underwent the so-called quench - at a current of 110 A, which was significantly lower than the calculated one - 280 A. Later, a decrease in the current was discovered at which the quench occurred up to 35 A, and it turned out that the compensating copper layer of the tape supplied by SUPEROX was peeled off from the side where there was no HTSC layer. Also, weak adhesion of the compensating layer from the side of the superconducting layer of the tape was found out. It was difficult to make claims against the supplier, since in the technology of winding magnet coils for electrical insulation epoxy resin and kapton tape were used. This is a traditional technology that is commonly applied when winding Nb-Ti superconducting magnets for work at helium temperatures. Also, the remains of the tape when tested on a supplier's run test machine, which provided a result up to 1 meter long, did not allow us to see the degradation of the HTSC tape layer.

After the extension of the project, it was decided to replace the SUPEROX supplier with the SUPERPOWER supplier.

It should be noted that the HTSC tape is supplied in the form of segments 200-250 meters long, wound into a coil. The tape was checked to the subject of compliance of the critical current in the following way. The tape wound into a coil without using insulating layer between the windings with the current leads soldered to its ends was cooled in a vacuum up to 20 K using a cryocooler. Then a current of 300 A was applied to the coil and decrease in current at the ends of the coil was measured, as well as the field value using the Hall sensor in the center of the coil. Testing showed the possibility of manufacturing magnet coils by laying winding layers without using an insulating layer.

This method of winding is a technological achievement of the project, as it allowed one to manufacture coils without the use of electrical insulation, which in its turn provided the following advantages for such coils:

- maximum possible current density;

- compactness and lower weight as compared to a coil produced using the technology with insulation;
- shorter cooling period (for a coil using insulation, to obtain a working temperature point of 20 K a three-day cooling is required, and for a coil produced without insulation only 2-day cooling is needed to reach such a working point.

The expenses within the theme for implementation of the project in 2015-2020 under scientific research items “Materials and Equipment” amounted to 730 kUSD.

Conclusions

1. In the result of implementation of the project, a cryomagnetic system with a superconducting magnet manufactured using HTSC technology has been developed, manufactured and tested, adapted to positioning conditions on the beam 12 of the IBR-2 reactor. The application of this cryomagnetic system of the sample environment with a high-pressure chamber will significantly enhance the possibilities for the study of the condensed matter at the spectrometric base of the IBR-2 reactor. This allows an independent variation of parameters on the test sample: the magnetic field, temperature, pressure. Thus, the goal of the project has been successfully achieved.
2. During the 3rd and 4th quarters of 2020, at the time of the technological break in the reactor operation, as well as in the autumn period, it is planned to install a cryomagnetic system on the DN-12 diffractometer.
3. The results of the implemented work under the project are reported at international and Russian conferences and meetings. The papers [5, 6] have been published and two more papers are being prepared for publication.

References

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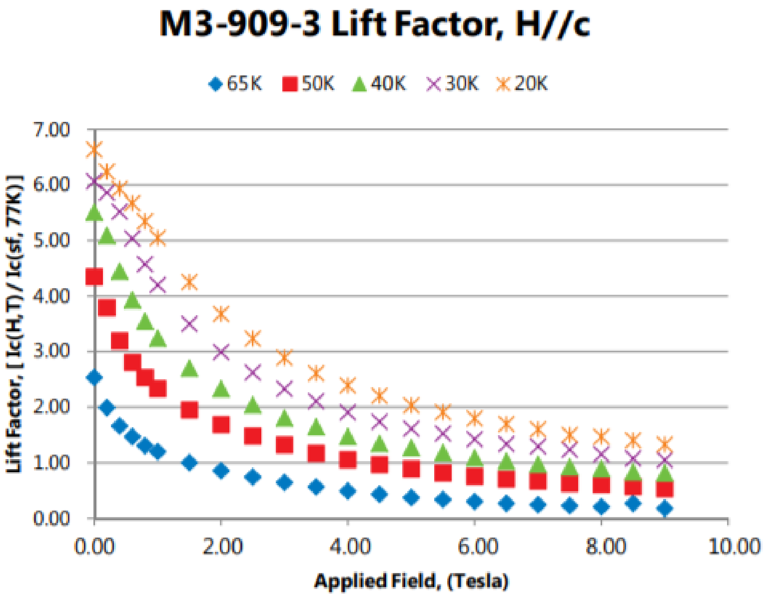

Fig. 11. Lift-factor is the ratio of the magnetic field at low temperature to the magnetic field at 77 K, depending on the obtained value of the field directed to the tape in perpendicular plane, which has been presented in the manufacturer's product data sheet for the HTSC tape to be used for producing the magnet [4].