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Development of the sample environment system for the DN-12 diffractometer on the IBR-2M pulsed reactor (pressure temperature – magnetic field). Project status.

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Abstact. The neutron diffraction experiments with microsamples use specific sample environment systems. One of such systems is under development in this project for the DN-12 diffractometer on the IBR-2M reactor. It is needed to keep of the investigated sample in the next simultaneous conditions: a high-uniformity magnetic field up to 4T, high pressure and low temperatures down to 4 K.

The arrangement of detectors of the diffractometer restricts the operations of the magnet construction and the magnetic field uniformity. The developing magnet is a superconducting split-coil type magnet. This provides the neutron scattering perpendicularly towards the vector of the magnetic field and to the beamline, as well as the scattering directed forward under the angle of 45 degrees.

The HTSC (high-temperature superconductor) magnet is made of YBCO tape with a critical current in the range of 300A@77K. The cryocooler ("cryogen-free") has been used for cooling the magnet. The cryostat also holds coaxial cylindrical channel from stainless steel provided by its own vacuuming system with an internal diameter of 79 mm for the access of the sample into the existing high pressure cell, which is cooled by the second cryocooler. Both of the cryostats are in a horizontal lay-out.

This report focuses on the description of the system and the parameters obtained at the first experiment.

1. Introduction

The investigation of structural mechanisms of the formation of magnetic states in various types of complex magnets is one of the vital problems in condensed matter physics. Important information on this subject can be obtained as a result of simultaneous research of crystalline and magnetic structures by varying structural parameters.

Neutron diffraction experiments under high pressures at various temperatures provide unique possibilities for studying mechanisms of the formation of magnetically ordered states in magnets via controlled variation of structural parameters (interatomic bond lengths and angles, atomic displacements from equilibrium positions).

On the pulsed high-flux IBR-2M reactor from JINR-Dubna, high-pressure investigations are performed on two neutron diffractometers – DN-12 and DN-6, which are equipped with horizontal cryostats for carrying out experiments in a temperature range of 4 - 300K.

2. System description

The preliminary calculations of the quantity of the required superconductor have shown that the generation of a magnetic field of 4 T requires 1480 meters of 12-mm-wide HTSC tape to form two double pancake coils. The manufacturer SuperOx [1] produces 200-m-long pieces of HTSC tape with guaranteed properties. SuperOx produces long tape by means of SnPb soldering separate pieces with 20 *10⁻⁹ ohms resistivity on junction at 77 K [2].

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Changing sample temperature requires a cryostat-insert for the magnet. This cryostat is planned to be equipped with holders and containers of high-pressure cells. The high-pressure cells of non-magnetic materials are to be used.

It is proposed to employ RDK408S cryocooler to cool the magnet. In the current design, the expected temperature of the magnet is about 15 K. At this temperature a critical field for HTSC tape is significantly higher than the estimated field of the magnet.

Carrying out temperature measurements depending on the value of a magnetic field requires the application of RDK101D cryocooler with a final temperature of ~ 3 K. Figure 1 presents the layout of a cryogenic facility with a superconducting magnet.



Figure 1 - The HTS magnet layout: 1 – superconducting magnet; 2 – cryostat-insert; 3 – entrance window for neutrons; 4 – current leads (up to 300 A); 5 – high-pressure cell; 6 and 7 – detectors of the scattered neutrons at the angles of 45° and 90°; 8 – cryorefrigerator RDK408S; 9 – cryorefrigerator RDK101D

2.1 Characteristics of the coils and the magnet

The required length of the 12mm wide tape YBCO type for each coil was achieved by joining the parts using SnPb solder. For each coil, SuperOx produced tape with two junctions. The quality of the junctions of tape pieces with SnPb solder was investigated in [2].

Next, two double pancake coils were wound, pre-coated with a layer of kapton 7 μ m thick on one side. The winding was carried out using epoxy glue EPO-TEK 301-2. The inner diameter of the coils is 84mm, the outer diameter of the coils - 320 mm. The thickness of the double pancake coil is 27 mm. The electrical resistance of each coil is ~ 25 ohms at 300 K. The distance between the coils in the magnet frame is 35 mm.

The two HTS pancake coils were assembled together into a thermally conductive support made of aluminium alloy (Figure 2).



Figure 2 - Magnet assembly

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2.2 Cryostat and magnet

The first tests of the magnet were carried out in the vertical orientation of the cryostat, which simplifies the assembly and adjustment work. The location of the magnet and HTSC current leads in the cryostat is shown in Figure 3. The magnet is mounted on the central pipe-shaft. The cryostat insert is injected into this shaft. The cryostat insert is a SRDK101D cryocooler on the second stage of which the sample chamber is mounted. Figure 6 shows the general view of the cryostat in a vertical position.



Figure 3 - The position of the magnet in the cryostat. HTSC current leads

3. Preliminary experimental results

3.1 Temperature decreasing during cooling

Since the magnet has a mass of about 20 kg, its cooling is a fairly long process. It takes three days to reach the working temperature. Figure 4 shows the dependence of temperatures of different parts of the cryostat on time in the cooling process. The DT-670A thermometers were located on the flange cooled by the first stage SRDK408S in the immediate vicinity of the HTSC current leads. The temperature of the hot ends of the HTSC current leads was also monitored. We note that the temperature of the upper part of the current leads is decreased below 60K. The upper part of the cryocooler, is a copper tape of 1 mm thick, 30 mm wide and 400 mm long. An electrically insulated thermal contact of the copper current lead was made to the flange of the first stage of the cryocooler. We expect that for a current of 300 A, the upper end of the HTSC current lead to be heated to a temperature of less than 65 K. The temperatures of the upper and lower parts of the magnet were also monitored. The achieved temperature of the magnet was 16 K. The temperature of the second stage of the cryocooler was 8.2 K.

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Figure 4 - The magnet cooling process: 1 - HTSC current leads at the first stage; 2 - the top and the bottom of the magnet, 3 - the second stage of SRDK408S

These final temperatures show a good agreement with computed ones by a Comsol Multiphusics^R numerical simulation made on the CAD model of the system [3]. The results of the cooling process show also the adequacy of the system design for the purposes of the project.

3.2 Magnetic field generation, experimental results

Figure 5 shows the results obtained with the introduction of a current into the magnet. The permanent of the magnet was measured, which was 0.0154 T / A. A current of 90 A did not lead to a quench. A current of 105 A led to a quench. A, B, C are the values of the potentials at different points of the coils as a function of time; D, E are the potentials at the HTSC current leads including connection. The dashed line indicates the temperature of the top and the bottom of the magnet as a function of time. The time from 13:28:24 to 13:29:24 is a time period with a current of 105 A. Potential C is removed from the jumper between the coils, which is a copper plate. Potential C has linear increase, indicating the increase of resistance in this section, or heating. The increase in the value of the derivative of the temperature as a function of time at 13:29:24 indicates a quench, which was also monitored by measuring the total potential of the coils. As we assume, the heating in section C led to the quench because of the absence of thermal contact to the second stage of the cryocooler. Whereas HTSC current leads in the lower part had thermal contact with the second stage of the cryocooler, which gave sufficient cooling to the incoming and outgoing ends of the HTSC tape of the magnet.



Figure 5 - Temperature and voltage evolution during charging/discharging the magnet: 1, 2 – temperature on the top and at the bottom of the magnet case; A, B, C – junctions potentials; D, E – HTSC current leads potentials. The magnet constant is 0.0154 T/A. The achieved field magnitude of 1.5 T in the first test. The reason – quench in the central connection of the coil windings. The connection will be upgraded

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4. Conclusions and further study

Further work will include: improvement of the system of thermal and electrical contacts of the magnet and current leads; testing in the required temperature ranges and magnetic field values; test in the final, horizontal position and installation work on the DN-12 diffractometer of the IBR-2M reactor.

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