Cryostat for cooling samples in the study of low-temperature structural and magnetic phase transitions by neutron diffraction

E P Popov1,2,3, A N Chernikov1, A I Beskrovny1, J Waliszewski1,4 and M N Mirzayev1,5

1Joint Institute for Nuclear Research, 6 Joliot-Curie, 141980 Dubna, Moscow Region, Russian Federation
2Institute for Nuclear Research and Nuclear Energy, Bulgarian Academy of Sciences, 72 Tzarigradsko Chaussee, 1784 Sofia, Bulgaria
3Institute of Solid State Physics, Bulgarian Academy of Sciences, 72 Tzarigradsko Chaussee, 1784 Sofia, Bulgaria
4Faculty of Physics, University of Bialystok, 14 Maria Skłodowska-Curie, 15-089 Bialystok, Poland
5Institute of Radiation Problems, ANAS, 9 B. Vahabzade Str., AZ 1143 Baku, Azerbaijan

E-mail: epetropov@abv.bg

Abstract. A vertical cryostat was developed built around a Sumitomo RDK-101D cold head. The cryostat is intended for investigating the structure and phase transformations of different materials via neutron diffraction experiments on samples under high vacuum of up to 10^{-5} mbar. A working temperature of the sample close to 2 K was reached. The drift diameter of the head is 54 mm, making it possible to cool samples with a height of 55 mm and 15 mm in diameter. Test measurements were conducted and illustrative spectra were recorded on the example of a sample of HoFeO3. The cryostat was designed and manufactured at the Frank Laboratory of Neutron Physics of the Joint Institute for Nuclear Research.

1. Introduction
We report the first test measurements. The system was cooled down to the specified low temperature and held this value for a selected period of time. The results were satisfactory, so that we went on with the first measurements with real samples, namely, HoFeO3 samples prepared at the Faculty of Physics, University of Bialystok, by means of the classic solid-state reaction method. The structure in this material is tetragonal with basic space groups Pbnm and Pbn21 [1]. Certain phase transitions of the magnetic structure have been detected by real-time neutron diffraction, as the magnetic moments of the Ho and Fe atoms reorient as the temperature is varied. These dependencies have already been addressed in other works [1-5].

6 To whom any correspondence should be addressed.
The design includes a vacuum screen and two additional heat screens (1st stage and 2nd stage). The powder samples are stored in a vanadium container of suitable dimensions. The drift diameter of the head is 54 mm, making it possible to cool samples with heights up to 55 mm and 15 mm in diameter.

We followed the experience of a previous development of a shaft cryostat of a similar purpose [6]. The need for a new instrument arose in the process of working with samples at low temperatures. The cryostat head is made of vanadium, as this material does affect the results of the neutron diffraction measurements. The cylinder in the bottom part is built of aluminium. The cryostat is based on a Sumitomo RDK 101D cryocooler with an HC-4A Zephyr compressor.

The neutron diffraction experiments were carried out on the IBR-2 reactor of JINR by a RTD DN-2 diffractometer [7, 8] using a cryostat based on a closed-cycle helium refrigerator.

2. Description of the product
Several different combinations of screen and base were made. The model described was used in the specific tests and the result is satisfactory.

Below are shown a schematic and a photograph of the device, as well as a plot of the cooling temperature time dependence.

2.1. Schematic and a photograph
Silicone washers and silicone rings were used to seal the cryostat at the screen to base connection, as well as at the base to the refrigerator housing.

2.2. Diagram of the temperature gradient
Two sensors were used, one placed at the bottom part of the sample and the other immediately above the sample to provide a complete temperature picture of the area where the object under study is located.

Figure 1 shows the schematic drawing of cryostat with the positions of the two silicon diode thermometers DT-670 and a photograph of the vanadium head.

Figure 2 shows the temperature variation with the time for the same two temperature sensors. A photograph of the measuring device (Lake Shore) with data from both sensors is also presented.
3. Test measurements and results
The test measurements were conducted on a sample of HoFeO₃, which exhibits multiple magnetic phase transitions with the temperature variation.

We used a small temperature range (2 K to 50 K) where a magnetic transition (at 34 K) and a continuous reorientation of the magnetic moments of Ho and Fe sub-lattices are observed [1, 2].

3.1. Graphic presentation of the data
The spectra registered at 2 K, 5 K, 10 K, and 50 K clearly demonstrate the phase transition and reorientation of the magnetic structure.

Figure 3 illustrates the changes in the magnetic moment vector orientation with the temperature. This is a typical spectrum registered in neutron diffraction measurements [1-5].

3.2. Phase transition illustration

| Table 1. Phase transition in HoFeO₃ sample with the temperatures [1]. |
|-----------------|-----------------|-----------------|
| Temperature     | Magnetic        |
| Space group     | 2 K             | 5 K             |
|                 | Pbn′2₁           | Pbn′2₁           |
|                 | 50 K            |
|                 | Pbn2₁           |

Illustration

4. Conclusions
- An efficient cryostat was developed for the needs of neutron diffraction measurements at low temperatures.
- Test measurements were conducted with a HoFeO₃ sample.
- Magnetic phase transitions between the Pbnm and Pbn₂₁ structural groups were recorded as the temperature was raised.

The device described would provide a user with additional capabilities in the production of low-temperature neutron diffraction screens.

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