New generation of cryogen free advanced superconducting magnets for neutron scattering experiments

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Abstract. Recent advances in superconducting technology and cryocooler refrigeration have resulted in a new generation of advanced superconducting magnets for neutron beam applications. These magnets have outstanding parameters such as high homogeneity and stability at highest magnetic fields possible, a reasonably small stray field, low neutron scattering background and larger exposure to neutron detectors. At the same time the pulse tube refrigeration technology provides a complete re-condensing regime which allows to minimise the requirements for cryogens without introducing additional noise and mechanical vibrations. The magnets can be used with dilution refrigerator insert which expands the temperature range from 20mK to 300K. Here we are going to present design, test results and the operational data of the 14T magnet for neutron diffraction and the 9T wide angle chopper magnet for neutron spectroscopy developed by Oxford Instruments in collaboration with ISIS neutron source. First scientific results obtained from the neutron scattering experiments with these magnets are also going to be discussed.

1. Introduction

Neutron scattering is an invaluable tool for solid state magnetism. In many experiments a sample needs to be exposed to high magnetic field, low temperature and neutron beam simultaneously [1]. Nowadays these conditions can be satisfied only in superconducting magnets. First generation of superconducting magnets for neutron scattering has been developed in late 1980’s of previous century and by the middle of 1990’s the design has settled down and magnets became a part of standard sample environment (SE) kit [2]. Most of them are split-pair magnets made of NbTi and Nb₃Sn filamentary superconducting wires. The magnet is usually built in a conventional liquid helium bath cryostat with thermal shield cooled by liquid nitrogen. A set of windows made of neutron transparent materials provides a neutron beam access to the sample.

However, in the last decade the rapid development in superconducting magnet technology and cryogenics has made possible to build the new generation of advanced magnets for neutron scattering SE [3, 4]. The new magnets are usually designed to satisfy specific requirements of a particular
advanced neutron scattering instrument such as magnet aperture/opening tailored to instrument detectors coverage or compatibility with special collimating systems. Then the optimal combination of instrument and magnet allows much faster experiments with much higher resolution.

In this paper we discuss the design and the operational experience of the 14T magnet for neutron diffraction and the 9T wide angle chopper magnet for neutron spectroscopy developed by Oxford Instruments in collaboration with ISIS neutron source [4]. We also present first scientific results obtained from the neutron scattering experiments, carried out on TS2 instruments at ISIS, using these magnets.

2. 14T magnet for diffraction measurements

The 14T magnet for diffraction measurements is a high field split pair magnet which consists of NbTi and Nb$_3$Sn superconducting coils. Today maximum magnetic field for state of the art split pair magnet is 15T with opening angle in vertical direction to ±3°, but by limiting the maximum field to 14T it is possible to increase the detector viewing angle to ±7.5°. In order to optimise the magnet design for using on WISH long-wavelength diffractometer we have chosen an asymmetric split -5° to +10°, which for single crystal studies, allows access to at least one extra scattering plane apart of the main horizontal plane. During factory test the magnet experienced few quenches at 14T. In order to minimise the risks associated with magnet operation it was decided to restrict the maximum magnetic field to 13.7T. Since the last planned soft quench during the magnet acceptance test in the middle of 2010 the magnet has not experienced any quenches. In future, we are planning to use dysprosium inserts [5] which should push the magnet maximum field up to ~16T.

The magnet split is supported by aluminium rings. The total thickness of Al alloy in the beam is 27 mm. The surface of aluminium rings which is not a part of neutron scattering aperture is plated with cadmium in order to reduce secondary neutron scattering. In most cases the 14T magnet is used with the radial collimator which allows to reduce the magnet background signal significantly. Main 14T magnet technical specifications are given in the Table 1.

| Table 1 Main technical specifications of 14T magnet for diffraction measurements |
|---------------------------------|-----------------|
| Maximum field                   | 13.7T           |
| Field homogeneity:              | 0.45% (over 10 mm sample diameter volume SVD) |
|                                 | 1.25% (over 10 mm x 20 mm cylinder) |
| Persistent mode stability       | <0.01%/hr       |
| Stray field (at max filed)      | ≤ 0.007T @ 2m   |
|                                 | ≤ 0.0004T @ 4m  |
| Vertical opening                | -5° to +10° (to the back of the 10 mm cylindrical sample height) |
| Horizontal opening              | 340°            |
| VTI temperature range           | 1.5K – 300K (down to 50mK with a dilution fridge insert) |
| VTI temperature stability       | ±0.1K           |
| Magnet bath temperature         | 4.2K            |

In Fig. 1 we present diffraction pattern of the ‘spin ice’ sample Ho$_2$Ti$_2$O$_7$ (courtesy of R. Aldus, UCL) previously used in neutron scattering investigation of holmium titanate's kagome-ice plateau [6]. The data have been obtained at 150 mK temperature and magnetic field 1.6T. The preliminary diffraction data demonstrate the very good angular coverage and the data quality (low noise) thanks to the collimator and the cadmium shielding.

3. 9T wide angle chopper magnet

Similar to 14T diffraction magnet the 9T wide angle chopper magnet for spectrometry is a split pair magnet which consists of NbTi and Nb$_3$Sn superconducting coils. But in this case the magnet split is supported by nonmagnetic stainless still wedges rather than aluminium rings. This design allows us to
Fig. 1 Single crystal diffraction from ‘spin ice’ sample Ho₂Ti₂O₇ at 150mK and 1.6T (by courtesy of R. Aldus)

To minimise the amount of material in the beam. The chopper instruments derive their power from being able to survey large volumes of reciprocal space in a single measurement, and so it is vital to build a magnet that can exploit this advantage. This would necessitate a trade-off between maximum field and a wide aperture. Detailed modelling allowed us to design and manufacture 9T magnet with ±15° opening in vertical plane and ±45° (two openings separated by 180°) opening in horizontal plane. In addition we also have 50mm diameter hole at 90° to allow beam in and out, which allows coverage of larger angles in the horizontal scattering plane. Such a magnet can be particularly valuable for exploring the competing phases and the microscopic interactions in the field of strongly correlated electron systems using spectrometers, such as MERLIN and LET (with the unprecedented continuous angular coverage of their detectors). The 1.5mm thick Boron Nitride tiles have been attached to the beam entry slots of the magnet former in order to reduce the background of the magnet. The 2mm thick Boron Nitride tube has been also installed in order to shield the bore of the magnet. Main 9T magnet technical specifications are given in Table 2.

Table 2 Main technical specifications of 9T wide angle chopper magnet

| Specification                  | Value               |
|-------------------------------|---------------------|
| Maximum field                 | 9T                  |
| Field homogeneity:            | 0.4% (over 10 mm SVD) |
|                               | 2.5% (over 25 mm SVD) |
| Persistent mode stability     | <0.01%/hr           |
| Stray field (at max fielded)  | ≤ 0.00045T @ 4m     |
| Vertical opening              | ±15° (to the back of the 25 mm cylindrical sample height) |
| Horizontal opening            | 90° (+45°)          |
| VTI temperature range         | 1.5K – 300K (down to 50mK with a dilution fridge insert) |
| VTI temperature stability     | ±0.1K               |
| Magnet bath temperature       | 4.2K                |

In Fig 2 we would like to show the data (by courtesy of Radu Coldea) from the first experiment on LET. The sample is a single crystal (~6g) of CoNb2O6, a quasi-one-dimensional Ising ferromagnet. Below a critical magnetic field the observed spin excitations are from pairs of ‘kinks’ in the ordered phase (see Fig. 2a). However, above a critical field the system undergoes a quantum phase transition to a quantum paramagnet and the spin excitations observed are those of spin-flips in the paramagnetic
phase (Fig. 2b). For more details on this system please see reference [7]. Both sets of data were taken with an incident energy of $E_i = 4$ meV and have a resolution at the elastic line of approximately 1% of $\Delta E/E_i$. This data are without background subtractions. The signals are clean with no spurious signals and demonstrate the low background and high energy resolutions achievable.

![Image of spin excitation spectrums of CoNb$_2$O$_6$ taken at 50 mK and with an incident energy of 4 meV (by courtesy of Radu Coldea). a) 4T field and b) 7T field](image)

**Fig.2 Spin excitation spectrums of CoNb$_2$O$_6$ taken at 50 mK and with an incident energy of 4 meV (by courtesy of Radu Coldea). a) 4T field and b) 7T field**

### 4. Magnet cryogenics

Both 14T and 9T magnets share the similar top parts of the re-condensing cryostat with Variable Temperature Inserts (VTI), but have different bottom parts accommodating split pair magnets and sample space. The design of re-condensing magnet cryostats is usually based on the design of similar bath cryostats [8]. The superconducting magnet is immersed in the liquid helium. The radiation shield is cooled by the cooler’s 1st stage and the 2nd stage re-condenses helium directly in the helium vessel. Thus the re-condensing system does not consume any liquid helium in normal operation. The main advantage of this system is that all the magnet operating modes, for example cooling, running up to field and quenching remain the same as for a standard magnet in a bath cryostat. This method also provides a homogeneous temperature distribution which is crucial for optimum magnet performance. Another significant advantage is the ability of the magnet to stay at field in the event of a power failure.

The cooling power for helium re-condensing is provided by CryoMech pulse tube refrigerator PT410. This type of cryocoolers has no cold moving parts and, therefore, has low level of mechanical vibrations [8, 9]. Additionally the pulse tube refrigerator requires little maintenance.

In a regime without VTI helium circulation and no ramping of filed the pulse tubes on both magnets performs so efficiently that it re-condenses all the helium in the cryostat and has an excess of cooling power between 600 and 650 mW [4]. This extra cooling power allows us to re-condense the helium flow passing through a VTI during the operation of a dilution refrigerator insert [10].

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