Magnetic system for small angle neutron scattering investigations of nanomaterials at YuMO-SANS instrument

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Abstract. SANS measurements using unpolarized neutron beams are able to provide quantitative information on the magnetic microstructure and the magnitude and microstructure of the magnetic anisotropy of nanomagnetic materials. A new magnetic system for SANS at the YUMO spectrometer is presented. The system includes 2.5 T electromagnet established on a two-axes goniometric table, a power supply, cooling system, PC based control equipment. Main features of magnetic system are: changeable gap for samples (up to 130 mm size), computer controlled horizontal and vertical rotation and sufficiently big space for the sample holders. First results of SANS experiments of ferrofluids and magnetic elastomers obtained at YuMO spectrometer equipped with the new magnetic system are presented.

1. Introduction

Neutron beams produced by different reactors are used for fundamental research on the structure and dynamics of the matter. Research efforts seek a better understanding of phase transitions, crystal structures, magnetic properties, superconductors, quantum liquids, the fundamental properties of new materials. Neutron scattering facilities have unique state-of-the-art capabilities for investigating structures and excitations of solid state matter for research in solid state physics, polymer science, and biology. The small angle neutron diffractometer is widely used by colloid, polymer, biology scientists, scientists involved in investigation of nano-materials and nano-composites.

The instrument YUMO (Figure 1) is located on the beamline 4 of the high pulsed IBR-2 reactor [1-3]. The useful wavelength range is $0.7 < \lambda/\text{Å} < 8$, the range of momentum transfers is $0.007 < Q/\text{Å}^{-1} < 0.5$. The time averaged neutron flux at the sample is up to $4 \times 10^7 \text{cm}^{-2}\text{s}^{-1}$.
A variety of auxiliary devices are necessary for materials studies, in special environment conditions including low and high temperature, high pressure and strong magnetic fields.

For investigation of magnetic nanostructures as nanoparticles, ferrofluids, magnetic gels and polymers, nano-carbon composites with magnetic properties there is highly necessary to apply during the experiment magnetic fields of different intensity and orientation to the samples [4-20].

A new magnetic system for small angle neutron scattering at YUMO instrument was put into operation. The system includes about 3T (factory setup) electromagnet fixed on a two-axes goniometric table, power supply, cooling system and PC based control equipment. Main features of magnetic system are: big changeable gap for the samples (up to 130 mm size), computer controlled horizontal and vertical rotation and sufficiently big space for the sample holders [21, 22].

In the present paper a magnetic system special created for the YUMO SANS instrument is presented. The first SANS measurement results on magnetic elastomers and ferrofluids in applied magnetic field performed at YUMO diffractometer are given.

2. Magnetic system at YUMO SANS instrument
The magnetic field, like the temperature or pressure, is an important parameter influencing the state of matter and properties of many materials. The applied magnetic field determine certain effects not only in ferro- and ferrimagnetic materials but also in paramagnetic and diamagnetic ones.

It is very important to control the microstructure of magnetic materials because textured or anisotropic magnetic materials usually show better performance in practical applications. Magnetic field processing is a well-proven technique to impose the desired texture and magnetic properties of magnetic materials. Macroscopic materials comprising a large number of nanostructures in crystalline alignment with one another would constitute highly anisotropic materials with wide potential
applications. It has been also found that magnetic field can make inclusions migrate in a melt and align grains in fabrication processes for the magnetic and non-magnetic materials.

The “control of materials” for materials science by means of a magnetic field, during the materials synthesis, chemical reactions or crystal growth in high magnetic fields is useful method for creating and investigating new types of materials [23].

A new dimension in advanced materials (such as magnetic materials, oxide superconductors and organics) processing, and in the microstructure research of nanometer-scaled materials, spintronics, polymer, bio-chemistry and so on, through the possibility of SANS investigation in situ of the phenomena induced by applied magnetic fields are under investigation (see e.g. [24-27]). Other aspect of using magnetic field in small angle neutron scattering experiment refers to the possibility of determination of the nuclear and magnetic elastic scattering contributions to the scattering. When investigating magnetic materials in a SANS with non-polarized neutrons experiment, measuring the scattering intensity without magnetic field and with a saturation magnetic field oriented perpendicular to the scattering momentum transfer, the nuclear and magnetic contributions can be differentiated [28-30]. Previously, this differentiation was possible to be obtained at SANS-YuMO instrument only in the case of magnetic liquids by means of the contrast variation method [31-34]. Magnetic neutron scattering plays a central role in determining and understanding the microscopic properties of a vast variety of magnetic systems, from the fundamental nature of magnetically ordered materials to elucidating the magnetic characteristics essential in applications.

For common goals the magnitude of magnetic field inducing changes in samples, (namely: anisotropy and orientation of magnetic domains in magnetic materials, as well as in the liquid crystal polymers [35]) is about few Tesla.

The new magnetic system constructed for YUMO instrument is presented (figure 2). The magnetic system components are: the electromagnet with the goniometric device, the power supply; computer for the automated command and control of the system.

Figure 2. Magnetic system for YUMO SANS instrument (electromagnet fixed on a two-axes goniometric table, power supply, cooling system)

The system was developed and constructed by the INCDIE ICPE CA Bucharest Romania in collaboration with CIPEC SRL Bucharest Romania.
The realization of the new position sensitive [1-3] permits the visualization and investigation of the magnetic field induced anisotropy in the analyzed sample.

2.1. Technical description.

The electromagnet is a rigid system, made of two coils, with distilled water cooling system and two magnetic poles with changeable caps (two types). The pole gap is variable, from 25 to 100 mm.

On the both lateral parts of the electromagnet there is one slit, 100 × 600 mm, for giving the possibility to introduce the neutron flux. The whole electromagnetic system is sitting on one rotating system. There are two rotating planes with a rotation liberty of ±40° in the vertical plane and of ±95° in the horizontal plane. The rotating devices are equipped with electrical limiters, which stop the rotation motors. The motors are step-by-step motors. The positioning accuracy for both rotation planes is 0.1°.

The assembly electromagnet and rotating system are placed and fixed on a mobile support (for translation) by means of some screws. The highest point of this support is adjustable, with ±30 mm, for having the facility to the center of electromagnet in the neutron flux. The electromagnet cooling system is a closed system, with distilled water. The cooling of the water is made by a water chiller with a capacity of 5000 W. The water flow is about 400 l/h. The electromagnet is supplied by a DC source, with a current adjustment of 10 to 60 A DC. The adjustment accuracy is 0.7% from the end of the scale. Maximum reached intensity of magnetic field is 2.5 T.

The measuring and control systems are connected with a PC. The software is an friendly one, who gives a lot of facilities:

1) Measuring and control for following parameters: value of the magnetically field; value of current in the coils; cooling water temperature; water flow; electromagnet position in vertical plane; electromagnet position in horizontal plane; turning limits in vertical plane; turning limits in horizontal plane.

2) Alarms for outrunning of measuring values set points

3) Curves, diagrams for measuring values variations.

4) History of measuring values variations, for a period of 30 days.

The technical drawings from Figure 3 present the block diagram of the whole magnetic system [21,22].

Figure 3. Block diagram of the whole magnetic system.
Standard measurements and data analysis
When investigating magnetic materials in a SANS with non-polarized neutrons experiment, measuring the scattering intensity without magnetic field and with a saturation magnetic field oriented perpendicular to the scattering momentum transfer, the nuclear and magnetic contributions can be differentiated. As example, a ferrofluid sample is examined [36,37] (Figure 4).

**Figure 4.** TEM image of a ferrofluid sample.

During standard tests the sample in closed quarts cuvette is put under external magnetic field ($B > 1$ T) so that the saturation magnetization in the sample takes place. Then, the sample is irradiated

**Figure 5.** 2-dimensional scattering patterns from magnetic sample in absent (left) and present (right) of external magnetic field. The separation of the nuclear and magnetic scattering contribution for saturated sample is demonstrated.
by the thermal neutron beam followed by registering of neutrons scattered at small angles (< 0.1 rad) by the position sensitive detector behind the sample. The 2-dimensional scattering pattern on the detector has specific features determined by two contributions: nuclear and magnetic scattering.

In the case of non-polarized neutron beam for the saturated sample the scattering intensity is

\[ I(Q, \varphi) = F_n^2(Q) + F_m^2(Q)\sin^2 \varphi \]  

where \( Q = (4\pi / \lambda) \sin(\theta / 2) \) is the module of the scattering vector with neutron wavelength, \( \lambda \), and scattering angle, \( \theta \). \( \varphi \) is the radial angle on the detector from the zero-direction corresponding to direction of the strength of the applied magnetic field; \( F_n^2(Q) \) and \( F_m^2(Q) \) are the nuclear and magnetic scattering contributions, respectively. The separation is made by averaging of the scattering pattern for given \( Q \)-value over radial angle \( \varphi \) around the vicinities of the directions parallel and perpendicular to the magnetic field (Figure 5). This procedure transforms the 2-dimensional pattern to two 1-dimensional scattering curves:

\[ I_{||} = < I(Q, \varphi) >_{\varphi=0,\pi} = F_n^2(Q) \]  

\[ I_{\perp} = < I(Q, \varphi) >_{\varphi=\pi/2,3\pi/2} = F_n^2(Q) + F_m^2(Q) \]

These equations are the base to obtain nuclear and magnetic scattering contributions.

If the applied magnetic field does not produce the saturation magnetization in the sample the scattering intensity on the detector has the general form:

\[ I(Q, \varphi) = A(Q) + B(Q)\sin^2 \varphi \]  

where \( A(q) \) and \( B(q) \) are functions corresponding to isotropic and anisotropic contributions to the scattering. The \( B(q) \) function is determined purely by magnetic scattering of the partially oriented magnetic moments in the system. The \( A(q) \) function is composed from isotropic nuclear scattering and isotropic part of magnetic scattering. The separation of these functions is made by averaging of the scattering pattern for given \( q \)-value over radial angle \( \varphi \) around the vicinities of the directions parallel and perpendicular to the magnetic field (Figure 5). This procedure transforms the 2-dimensional pattern to two 1-dimensional scattering curves:

\[ I_{||} = < I(Q, \varphi) >_{\varphi=0,\pi} = A(Q) \]  

\[ I_{\perp} = < I(Q, \varphi) >_{\varphi=\pi/2,3\pi/2} = A(Q) + B(Q) \]

These equations are the base to obtain \( A(q) \) and \( B(q) \). Analysis of the magnetic system based on changes in these functions with the strength of the applied magnetic field, is possible. The behavior of the standard sample with the change of the applied magnetic field is known.

**Conclusion**

First experiments have demonstrated that the electromagnet together with the position sensitive detector is suitable for SANS measurements at YUMO instrument.

Further, a data analysis to transform the 2-dimensional pattern to 1-dimensional scattering curves as presented above in the formulae from (2) to (5) will be performed. For this task a new SAS program with a 2D analysis is in progress.
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