RECENT TRIPLE-AXIS NEUTRON SCATTERING STUDIES OF QUANTUM MATERIALS AT THE CHALK RIVER NRU REACTOR

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The ground-breaking technique of triple-axis neutron spectroscopy was invented [1] by Bertram Brockhouse in the 1950s first at the Chalk River NRX (National Research Experimental) and further developed at the National Research Universal (NRU) reactor. A few years earlier, Clifford Shull at Oak Ridge National Laboratory (ORNL) in Tennessee had already demonstrated the concept of neutron diffraction and the type of information related to the static nuclear structure it provides. In diffraction experiments, a crystal monochromator is used to select a specific and narrow slice of the wavelength distribution from the white neutron beam produced by the fission process in the reactor. The ingenuity of Brockhouse's invention was its simplicity. He added an additional crystal monochromator (called analyzer) after the sample to determine how much energy transfer to and from the sample occurs when monochromated neutrons interact with the sample. Thereby the technique is called triple-axis spectroscopy (TAS) since it requires rotation of three crystals: monochromator, sample, and analyzer. Brockhouse's invention led to a new area in experimental condensed matter physics, allowing determination of lattice (phonon) and spin (magnon) dynamics using neutron scattering technique for the first time. Brockhouse and Shull shared the 1994 Nobel Prize in Physics for their work in neutron scattering.

In 1955, experiments by Brockhouse on an aluminum single crystal in several different orientations led to the successful determination of a phonon dispersion curve for the first time, thus demonstrating convincingly the power of the TAS method. In 1962 Brockhouse relocated to McMaster University where he continued to train young students in using this technique until his retirement in 1984. In the 1960s and the years following young scientists, such as Bill Buyers, Roger Cowley, Eric Svensson, Gerald Dolling, Dave Woods, Brian Powell, Varley Sears and Tom Holden, joined the Chalk River lab and made significant contributions to the study of excitations in solids. These include lattice dynamics and phase transitions [2], crystal field, interatomic exchange and spin-orbit coupling in $KCoF_3$ [3], and later magnetic excitations in the famous hidden order in URu_2Si_2 [4]. Investigations of this mysterious hidden order continues to present day at CNL [5]. Perhaps the most significant experiment from this era was the study of spin excitations in the quasi-1D antiferromagnetic (AF) S=1 spin chain $CsNiCl_3$ in 1986 [6]. The experiments led by Bill Buyers discovered the presence of a gap in its spin spectrum. Duncan Haldane had predicted that a gap in spin excitation spectrum occurs for integer but not half- integer spin chains. Haldane shared the 2016 Nobel Prize in Physics with David Thouless and Michael Kosterlitz for their theories on such topological materials [7].

SUMMARY

Triple-axis neutron scattering is the technique of choice for studying collective excitations in condensed matter. This technique has been instrumental in unveiling spin excitations in highly correlated electron systems such as high T_c cuprate and the more recent iron-based superconductors and quantum magnets. This article briefly reviews recent research highlights on such materials using the triple-axis facilities at the Chalk River NRU reactor.

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The condensed matter research program at CNL continues to thrive today, in particular in the area of emergent materials: novel superconductors (SC) and quantum magnets (QM). In this article, several research highlights from the past decade using the TAS C5 (polarized) and N5 spectrometers at NRU are reviewed. The selected examples reflect my personal research interests and do not intend to provide a comprehensive list of all the exciting research performed during this time. The C5 spectrometer was recently upgraded with a vertically focusing Heusler monochromator and thus provides a greater polarized neutron flux: perhaps the greatest of any steady-state neutron source in North America. Polarization analysis is a powerful tool for isolating and elucidating details of elastic and inelastic magnetic scattering cross-sections. This new monochromator together with a new five-coil assembly, to automatically change the orientation of the neutron spin parallel or perpendicular to the scattering vector at the sample position, make the polarized setup at C5 unique, allowing experiments on small samples and a precise determination of the sample magnetic moments. The N5 spectrometer was also recently upgraded with a new detector shielding drum, reducing the background to a level that TAS measurements on milligram size samples are now possible.

HIGH T_C SUPERCONDUCTING CUPRATES

Despite numerous experimental and theoretical studies since the discovery of high T_c superconductivity (HTSC) in cuprates in 1986, they still pose an immense challenge [8]. Fundamental questions, such as what is the pairing mechanism and what causes such high T_c, remain unanswered. Meanwhile, it is not hard to imagine the technological revolution that would occur by discovery of room temperature superconductivity. From early days, it became clear that magnetic rather than the electron-ion interactions play an important role in cuprates. Neutron scattering has proven to be a key experimental tool in unraveling both their static and dynamic magnetic properties.

The parent compound $YBa_2Cu_3O_6$ (YBCO6+x with x=0) undergoes a Néel transition to a long range antiferromagnetic (AF) order at T_N~450 K. The ordered phase is characterized by $\mathbf{Q}_{AF}$ = (0.5, 0.5, L) with integer L, where local spins of adjacent Cu sites in the CuO_2 building blocks of the material are antiferromagnetically coupled. The optimally doped YBCO does not show any magnetic long-range order. However, the presence of a resolution-limited excitation (the so-called resonance) peak at $\mathbf{Q}_{AF}$ and energy transfer, E_{res}, indicates magnetism is very much present. The resonance peak is correlated with superconductivity as its intensity increases below T_c and its energy scales roughly with T_c for optimally doped to slightly underdoped YBCO.

Stock *et al.* performed TAS experiments on the C5 spectrometer at NRU on underdoped detwinned YBCO6.5 single crystals with T_c = 59 K [9]. The crystals were prepared by the group of Prof. W. Hardy at University of British Columbia (UBC) who had recognized early the importance of annealing in addition to the oxygen content for the charge transfer to the CuO_2 planes [10]. The high quality of UBC crystals and careful experiments by Stock *et al.* allowed for a complete determination of the magnetic properties of YBCO6.5. They found that the resonance remains almost resolution limited and occurs at a lower energy that is consistent with the doping dependence of the resonance energy for higher doped YBCO. While the intensity of the resonance peak showed a clear increase below T_c, it was found to first appear at temperatures higher than T_c, suggesting it may be related to the pseudogap phase [9].

Li *et al.* performed TAS experiments on the C5 spectrometer at NRU to study lower doped detwinned YBCO6.45 with a lower T_c of 48 K [11]. They found the resonance peak is still correlated with T_c, but is no longer resolution limited. The position of the resonance peak was also found to be lower than the prediction of a linear dependence with T_c from the higher doped region. Based on these results they suggested that the change in the behaviour of the spin dynamics within the SC dome might be related to a fundamental change in the electronic state of the material due to a metal-to-insulator transition (MIT) inside the SC dome [11].

Stock *et al.* later used the C5 spectrometer at NRU to study further lower doped YBCO6.35 (T_c = 18 K) [12,13]. Their results showed that the only prominent feature of spin spectrum is a very broad and weak peak at low energies. They showed that this peak is overdamped and can be fit with a modified Lorentzian. They also found a quasi-elastic and commensurate peak (central mode) at the $\mathbf{Q}_{AF}$ position for this low doped YBCO6.35 which is absent for higher doped YBCO. The central mode intensity was found to gradually grow on cooling with no Néel anomaly. The results were interpreted in terms of a soft mode driving the central mode [12].

Yamani *et al.* performed TAS experiments on the C5 spectrometer at NRU to study YBCO6.33 crystals with exceedingly lower doping yet superconducting with T_c = 8.5 K, less than 1/10 from the optimally doped T_c [14-16]. Similar to YBCO6.35, they found that there is no sign of resonance and that the spin spectrum only consists of a weak and broad excitation at low energies as well as a quasi-elastic peak with much higher intensity (central mode). The lack of a resonance peak at these low doped yet superconducting materials suggest [14] that the resonance peak may not be required for the superconductivity in cuprates. The central mode peak in YBCO6.33 is also commensurate with the lattice and even though still broader than the resolution, it is narrower in YBCO6.33 than YBCO6.35. This indicates that the magnetic correlation lengths increase by reducing doping from YBCO6.35 to YBCO6.33. The fact that for both of these lightly-doped YBCO6.35 and YBCO6.33 only commensurate quasielastic peaks were observed suggests that similar to the resonance peak, the stripes may not be essential for superconductivity in general.

Yamani *et al.* found that in YBCO6.33 the quasi-static correlations also gradually grow on cooling albeit from higher temperatures than YBCO6.35 [14]. Similar experiments under an applied magnetic field revealed no effect on either the static or dynamic correlations while the applied field reduced T_c [14]. This indicates that the lack of a Néel anomaly is not due to the presence of superconductivity and that at this low doping, the spins responsible for AF correlations behave independently from the charges responsible for superconductivity. Since the correlations remain short-range and their temperature dependence does not show any Néel anomaly, a spin-glass state was suggested [14] to coexist with SC.

Although the magnetic excitations measured [14] as a function of $\mathbf{Q}$ (Fig. 1) revealed no hour-glass dispersion, Yamani *et al.* found that the momentum dependence of the width of the high energy peaks is similar to the spin-wave velocity of the insulating YBCO6.15. From these observations it was suggested [14]

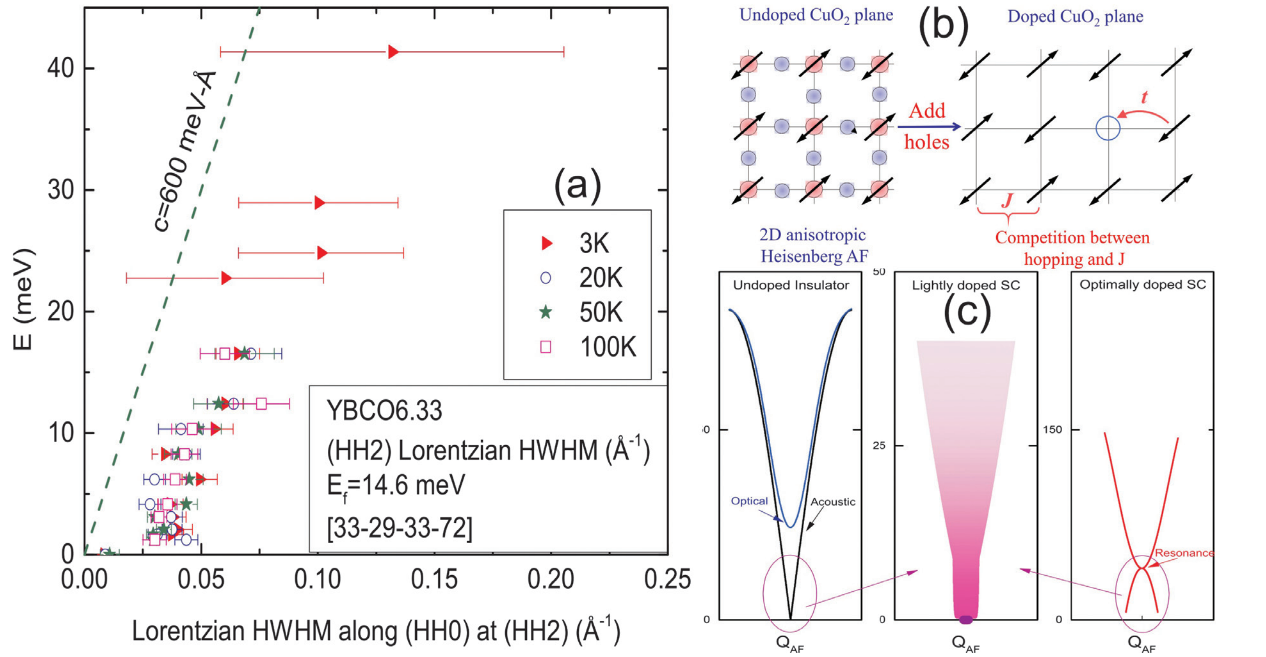

Fig. 1 (a) The resolution-corrected momentum width of the inelastic scattering. (b) The doped holes are effective in destroying long range AF order since each doped hole affects four AF neighbouring bounds. (c) Magnetic excitations in undoped insulator (left), YBCO6.33 (middle), and optimally doped YBCO (right) are schematically depicted. Adapted from Ref. [14].

that the suppression of the low energy excitations, a drastic change upon entering the SC dome, may be the key for inducing superconductivity in cuprates.

IRON-BASED SUPERCONDUCTORS

In 2008, HTSC was discovered in iron pnictides, containing iron ion with strong magnetism, sending shockwaves to the condensed matter community yet again. In conventional superconductors it is well known that addition of a small amount of magnetic impurities significantly reduces T_c. This discovery also revitalized the interest in superconductivity as other materials with T_c beyond the 30 K limit (electron-phonon coupling) than cuprates became available, generating hope that there could be other classes of SC with even higher T_c. Similar to cuprates it was soon established that magnetism plays an important role in pnictides [8]. A tetragonal-to-orthorhombic structural phase transition at T_s was found to be followed by a paramagnetic to AF phase transition at T_N with a collinear magnetic structure. For most pnictides T_s and T_N are identical or very close to one another. Wilson *et al.* used the C5 and N5 spectrometers at NRU to determine the magnetic and structural phase transitions in $BaFe_2As_2$ [17,18]. Later the effects of uniaxial strain on the structural and magnetic phase transitions in $BaFe_2As_2$ [19] and in Co-doped $Ba(Fe_{1-x}Co_x)_2As_2$ [20] were studied. Surprisingly, it was found that relatively small strain fields can change the magnetic order parameter and results in a decoupling between T_s and T_N. The evolution of the anisotropic spin excitations in $BaFe_{2-x}Ni_xAs_2$ was determined by Luo *et al.* by detailed inelastic experiments performed at NRU on the C5 spectrometer [21,22].

From the early days of studies on pnictides, two schools of thought have developed. One is based on itinerant electron physics which appears to be consistent with metallic properties of these materials. The other school of thought is based on the localized electron physics. Song *et al.* performed [23] TAS experiments on the C5 spectrometer at NRU to study the evolution of magnetism in $NaFe_{1-x}Cu_xAs$. For small Cu doping, superconductivity is induced and the static AF order is suppressed (Fig. 2). For doping well below 0.5, the magnetic transition is gradual and spin-glass-like. With increasing Cu doping, the scattering profile becomes narrower and stronger, changing from a broad peak indicative of the short-range magnetic order to an essentially instrument resolution limited peak with long-range magnetic order at x = 0.44. When doping approaches x = 0.5, the transition at T_N becomes more well defined concomitant with appearance of the insulating behaviour. This study showed for the first time that the SC phase can be continuously tuned to the Mott insulating phase, thus providing a direct evidence that the material is more correlated than itinerant [23].

When nearly 50% of Fe ions are replaced by Cu, a real space Fe and Cu ordering occurs which is a structural analogue of the magnetic order in pure NaFeAs. Thus, the use of polarized neutrons is crucial to separate magnetic from nuclear scattering [23]. By polarizing neutrons (**P**) parallel to **Q**, the spin-flip (SF) scattering is sensitive to the magnetic moment (***m***) components that are perpendicular to **Q** ($\boldsymbol{m}_\perp$) whereas the non-SF (NSF) scattering probes pure nuclear contribution. For neutron polarization perpendicular to **Q** ($\mathbf{P}\perp\mathbf{Q}$), the SF scattering is sensitive to the perpendicular component of the moment that is also perpendicular to the neutron polarization ($\boldsymbol{m}_\perp \perp \mathbf{P}$), while the NSF scattering is sensitive to the perpendicular component of the moment that is parallel to the neutron polarization ($\boldsymbol{m}_\perp \| \mathbf{P}$). The experiments shown in Fig. 2 confirmed [23] the presence of a magnetic scattering at 2 K that disappears at 240 K, in addition to the temperature-independent NSF nuclear super-lattice reflection.

NEW ELECTRONIC STATES IN STRONGLY CORRELATED ELECTRON MATTER

A plethora of new electronic phases have recently been uncovered in a new class of spin-orbit Mott insulators with both strong spin-orbit coupling (SOC) and electron-electron interactions [24]. 5d transition metal oxides, such as $Sr_3Ir_2O_7$, are considered excellent test systems for exploring carrier substitution in spin-orbit Mott materials and for hosting new spin- orbit generated states [24]. Dhital *et al.* used [25] the N5 spectrometer at NRU to study Ru doping in $Sr_3(Ir_{1-x}Ru_x)_2O_7$. The Ir^{4+} ions ($5d^5$) carry the magnetism in pure $Sr_3Ir_2O_7$ with a Néel transition at T_N = 285 K to a G-type AF structure. Dhital

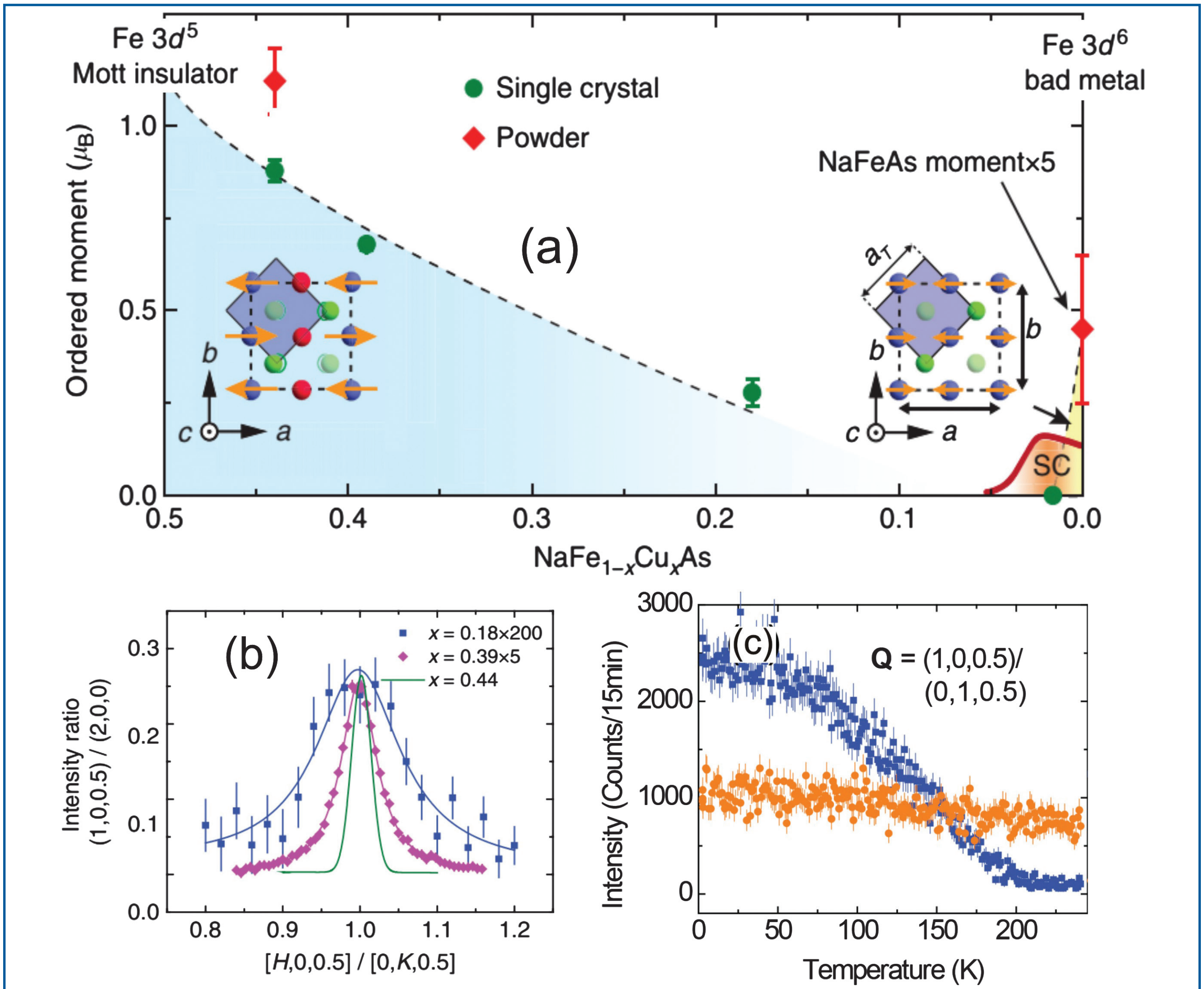

Fig. 2 (a) Magnetic phase diagram of $NaFe_{1-x}Cu_xAs$. (b) Scans along the [H, 0, 0.5]/[0, K, 0.5] direction vs x. (c) Temperature dependence of SF (blue squares) and NSF (orange circles) channels at (1,0,0.5)/(0,1,0.5). Magnetic signal at (0,1,0.5) is seen in the SF channel whereas super-lattice signal coming from (1,0,0.5) is seen in the NSF signal. Adapted from Ref. [23].

et al. showed that the addition of Ru atoms induces charge carriers and weakens the AF order [25]. Initially, the added charges remain in small metallic regions resembling metallic puddles due to the presence of strong correlations. With further increase of the Ru doping, puddles begin to percolate and eventually coalesce to induce a metallic state where charges freely flow. Eventually at a critical doping (x_{cr}), a transition to a metallic regime occurs paralleling the behaviour of Mott insulators. Dhital *et al.* also used polarized neutrons on the C5 spectrometer at NRU to demonstrate that a Bragg peak observed at 300 K ($>T_N$) in pure $Sr_3Ir_2O_7$ was non-magnetic and due to a nuclear superlattice structure.

Hogan *et al.* later studied [26] the magnetic order in La-doped $(Sr_{1-x}La_x)_3Ir_2O_7$ by means of TAS experiments on the N5 spectrometer (Fig. 3). The results indicated that upon doping electrons, the AF moment is immediately reduced. The reduction in the AF moment under light electron doping was found [26] to largely arise from electronic phase separation of the sample into AF-ordered insulating and paramagnetic metallic regions. An additional structural order parameter develops at T_S which increases with the growth of the volume fraction of the metallic phase upon La-doping. Beyond $x_{cr} = 0.04$, an abrupt, first-order phase boundary is observed where the Néel state is suppressed and a homogenous, correlated, metallic state appears and the system becomes globally metallic. It was suggested [26] that the structural distortion represents a competing instability with the parent spin-orbit Mott state.

Low dimensional spin systems exhibit many exotic magnetic properties. Spin-ladders have been the subject of intense interest recently as they are intermediate objects between 1D and 2D systems [27]. Plumb *et al.* performed [28] TAS experiments on the C5 spectrometer at NRU to study the ground state of the newly discovered quasi-2D spin-ladder $BiCu_2PO_6$. Results confirmed [28] that the $BiCu_2PO_6$ system can be described by weakly interacting two-leg ladders where frustration drives incommensurate dynamic correlations, and showed the importance of strong anisotropic interactions in addition to frustration in this material.

FUNCTIONAL MATERIALS

Functional materials consist of a large class of systems with properties important for technological applications. They are also of significant scientific interest because they possess strong electronic interactions. Multiferroics, exhibiting simultaneous ferroelectricity and magnetism because of cross-coupling between ferroelectric and magnetic order parameters, are among the most sought after functional materials [29]. Neutron scattering again has proven to be an essential tool unveiling their novel properties. Christianson *et al.* used the C5 and N5 spectrometers at NRU to perform polarized and non-polarized neutron scattering experiments under an applied field in the scattering plane of a multiferroic $LuFe_2O_4$ single crystal [30,31], a unique capability at CNL enabled by the M2 horizontal field magnet. They found a 3D Néel order below

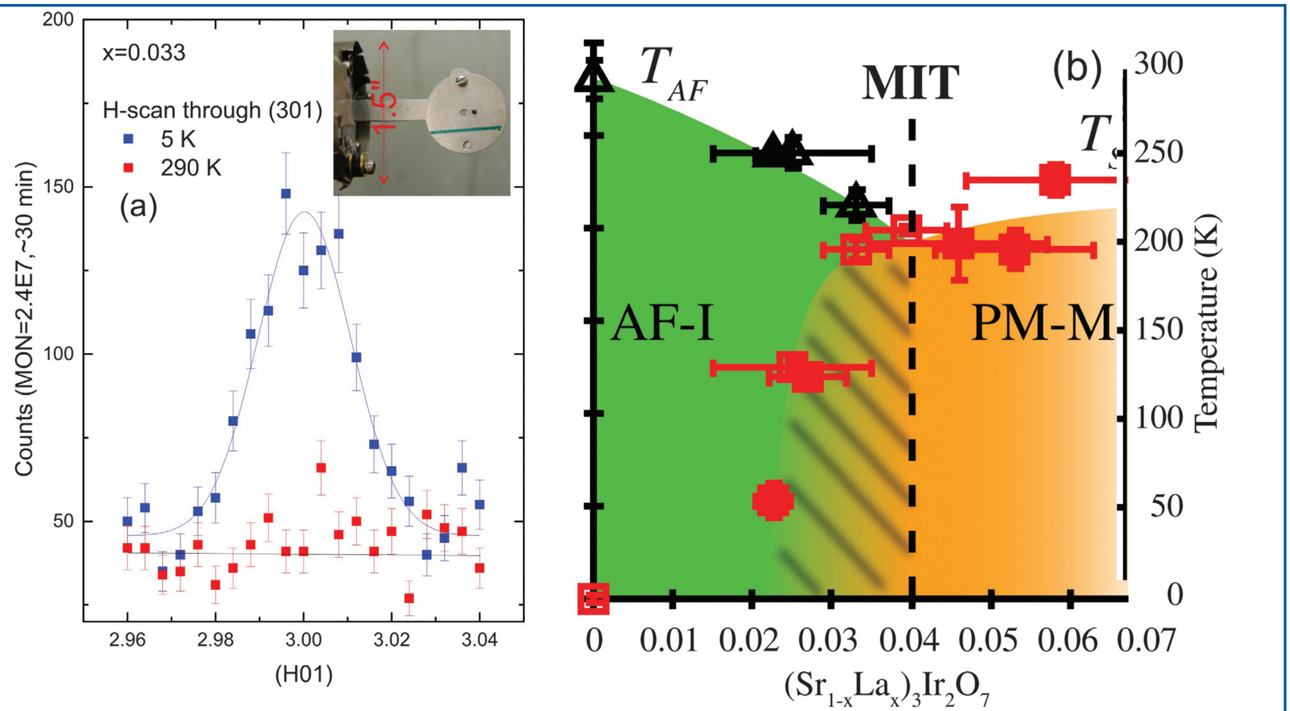

Fig. 3 (a) A representative scan through the magnetic peak (3, 0, 1) for x=0.033 in $(Sr_{1-x}La_x)_3Ir_2O_7$. The inset shows a picture of the milligram-size crystal used in the measurements. (d) Magnetic phase diagram of $(Sr_{1-x}La_x)_3Ir_2O_7$. Adapted from Ref. [26].

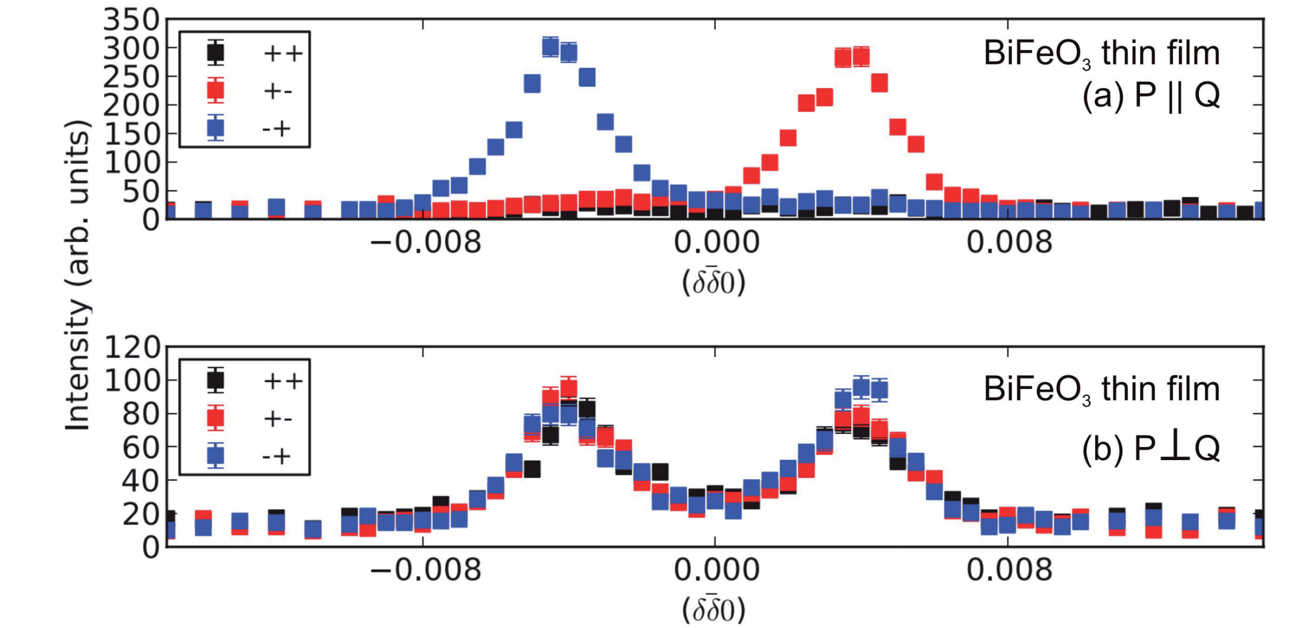

Fig. 4 SF and NSF cross-sections for **P**||**Q** (a) and for **P**⊥**Q** (b) measured close to $\mathbf{Q}_{AF}$ on a $BiFeO_3$ epitaxially grown thin film on $SrTiO_3$ substrate. Adapted from Ref. [32].

T_N = 240 K with a ferrimagnetic spin configuration arising from the charge ordering at 320 K.

$BiFeO_3$ is perhaps the only material that is both magnetic and strongly ferroelectric at room temperature [29]. $BiFeO_3$ undergoes a Néel transition to a G-type like AF order at T_N = 643 K and a ferroelectric Curie transition at T_c = 1103 K. Ratcliff *et al.* performed non-polarized and polarized experiments on the C5 spectrometer at NRU to study the magnetic structure of expitaxially grown $BiFeO_3$ films on $SrTiO_3$ substrates with ferroelectric monodomains [32,33]. The results indicate that close to $\mathbf{Q}_{AF}$=(0.5, 0.5, 0.5) a strong splitting between the I^{+-} and I^{-+} cross sections is observed for **P**||**Q**, whereas for **P**⊥**Q**, they have the same intensity (Fig. 4). The magnetic structure of this $BiFeO_3$ thin film was thus concluded [32] to be markedly different from that of a bulk single crystal since it contains a cycloid with moments spiraling in the plane normal to the polarization. The strain effects were suggested to cause the difference with the bulk [32]. The neutron scattering results provided clear evidence that the application of an electric field changes the AF domain population (concomitant to changes in the ferroelectric domains), thus suggesting a possible new direction for the realization of scalable magnetoelectric $BiFeO_3$-based thin film devices.

CONCLUSION

The thermal triple-axis instruments at the NRU reactor at CNL have been improved to increase neutron flux and to be more versatile with the addition of multi-wire detectors, extreme sample environments and sophisticated polarized setup. The TAS studies of novel superconducting and magnetic materials mentioned here demonstrate that the facilities at NRU can produce information about our world that command international recognition.

The TAS facilities at NRU have also played a tremendously important role in the training of young scientists either through performing experiments as part of graduate theses, hands-on summer schools or three-day courses [34]. Obviously none of these would have been possible without the ground-breaking work of Bertram Brockhouse.

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