Diffractometer for small angle resonant soft x-ray scattering under magnetic field

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Abstract. There has been a recent increasing interest in a topological spin texture, so-called skyrmion crystal, stimulated by small-angle neutron scattering and Lorentz-TEM studies. For the purpose of measuring the resonant soft x-ray magnetic scattering to characterize the distribution of magnetic moments with long-wavelength in range of a few tens to hundreds nm, we have developed a diffractometer for small angle soft x-ray scattering. The principle features of the diffractometer and the initial experimental results are presented.

1. Introduction

Resonant soft x-ray scattering (RSXS) is a powerful tool for observing the spatial ordering of electronic degree of freedom, such as charge, spin, and orbital, in transition metal compounds. To study the magnetic structure in thin films and in small single-crystals, the RSXS spectroscopy has been identified as one of the most powerful probes due to its direct coupling to relevant electric state and sensitivity to long-range spatial structure. The long-wavelength periodic modulations of electronic spin and orbital moment can be probed with the super-lattice reflection that can be obtained at the $L$-edge of $3d$ transition metal.

In these years, a topological spin texture, so-called skyrmion crystal, has been observed by small-angle neutron scattering (SANS) [1,2] and Lorentz-TEM technique [3] in transition metal compound with non-centrosymmetric B20 structure, such as MnSi and (Fe,Co)Si. The skyrmion crystals appear with 6-fold symmetry and via the magnetic phase transition from a helical spin structure as a function of magnetic field. An isolated skyrmion can be imaged as a vortex-like arrangement of magnetic moments, made up of downward core spins and upward peripheral spins swirling up with a unique spin chirality that is determined by the underlying chiral crystal structure. The length scale for the helical structure, depending on both ferromagnetic exchange interaction $J$ and Dzyaloshinskii-Moriya interaction $D$, generally ranges from several to tens of nano-meters. Recent Lorentz-TEM studies have revealed that the magnetic configuration of skyrmion lattices for FeGe and Fe$_{0.5}$Co$_{0.5}$Si thin sample in which the skyrmion lattice constant is $\sim$ 70 nm and for FeGe $\sim$ 90 nm for Fe$_{0.5}$Co$_{0.5}$Si [3]. In these long-wavelength magnet, the magnetic peak of RSXS at Fe $L_3$-edge ($\sim$ 707 eV) will appear in small angle region, i.e., $2\theta \sim 1.3^\circ$ for FeGe and $2\theta \sim 1.0^\circ$ for Fe$_{0.5}$Co$_{0.5}$Si.

For purpose of clarifying the magnetic and electric structure with long-wavelength magnetic structures like the skyrmion crystal by RSXS, we have developed a diffractometer for small angle soft x-ray scattering measurement under magnetic field. In this paper, we describe the principle features of the diffractometer which has been constructed at Photon Factory.
2. Diffractometer

The small-angle resonant soft x-ray diffractometer is constructed with a vacuum chamber with 6 ports and Helmholtz coil as shown in Figures 1(a) and (b). It has the following major components: (i) a sample cryostat with an XYZ manipulator, (ii) a differentially pumped rotary feed-through (DPRF) for sample omega-rotation stage, (iii) a direct beam stopper, (iv) motorized four-quadrant slits, (v) a photo-diode detector mounted on a rotary feed-through, and (vi) a CCD camera 2D detector.

The cryostat manipulator rotates sample in the horizontal plane (omega circle) by > 270 degrees, which are driven by external stepper motors through rotary feed-through. The Helmholtz coil is capable of applying a magnetic field along the incident x-ray up to ~ 0.4 T. The sample stage is isolated from the cryostat cold-head with a sapphire disk sandwiched in between to allow the total electron yield (TEY) measurement. A radiation shield is partially wrapped around it to reduce the heating on the sample. A YAG crystal and a 0.5 mm pinhole are attached on the sample stage for alignment purpose. The cryostat has built in heater for temperature control from ~ 15 K to ~ 320 K, as measured by the Si diode thermometers attached to next to the sample holder and cryostat head. Two motorized quadrant slits are set just before used to reduce parasitic scattering and the Fraunhofer diffraction. A translation of the diffractometer across the beam, and a rotation around a vertical axis is mounted on the table. These provide the necessary motions to position accurately the center of rotation of the diffractometer onto the synchrotron beam.

The photo-diodes can be used to detect the direct x-ray beam for alignment of diffractometer and to measure the fluorescence yield measurement. The current from the photodiodes and TEY current are amplified by a Keithely current amplifier. The 0 – 10 V output from this is passed through a voltage to frequency converter and into a Tsuji scaler. The motors are controlled through Tsuji motor controllers (PM16C) and the beamline is controlled through Photon Factory STARS system [5]. These controls and data acquisition are performed with Ethernet communication.

The experiments were carried out at the undulator beamline BL-16A at Photon Factory, KEK. The beamline is equipped with two APPLE-II undulators capable of providing right and left circularly polarized light, and linearly polarized light in the energy range of 180 -1500 eV with a beamsize of ~ 0.2 mm vertically and ~ 0.3 mm horizontally.
3. Initial Experimental Results

We have investigated B20-type Fe0.5Co0.5Si, which shows helical magnetic order below 40 K with a long wavelength of ~90 nm. The single crystal was grown by the floating zone technique. The soft x-ray penetration length of the transition metal compound becomes shorter than 200 nm, therefore a soft x-ray transparent thin plate with thickness of about less than a few hundreds nm was prepared by mechanical polishing and subsequent argon-ion thinning.

![Figure 2: (a) Fe L-edge soft x-ray absorption spectra (XAS) of a single crystal Fe0.5Co0.5Si, measured by fluorescence yield method with left- and right-handed circularly polarized soft x-ray (I_+ and I_-). (b) and (c) show the total XAS intensity and the x-ray magnetic circular dichroism (XMCD) with its integral, respectively. (d) The spin ordering peak of resonant soft x-ray scattering (RSXS) recorded at CCD camera. The incident photon energy was set at Fe L3 edge (707 eV). (e) Energy scan of the Fe L-edge RSXS signal at +Q magnetic reflection.](image)

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Figure 2(a)-(c) shows the results of x-ray absorption spectroscopy (XAS) and x-ray magnetic circular dichroic (XMCD) at Fe L-edge (690-740 eV) detected by the fluorescence yield (FY) measurement. The signals were measured under magnetic field of 0.4 T with left- and right-handed circularly polarized soft x-ray (I_+ and I_-). It is revealed that the XMCD signal at L3-edge (~720 eV) is much smaller than that of L2-edge (~707 eV). Based on the XMCD sum rule [6], the result indicates that the orbital magnetic moments of Fe are not totally quenched in this material. Figure 2(d) shows the resonant soft x-ray scattering (RSXS) from the helical magnetic structure recorded at CCD camera. The incident photon energy and polarization were set at Fe L3 edge (707 eV) and left-handed circularly polarized, respectively. The energy dependence of RSXS, as shown in Fig. 2(e), reveals that there is a huge enhancement of the magnetic reflection at the L3 edge, however the resonance at L2 cannot be discerned. The spectrum of RSXS is consistent with that of XMCD, because the intensity of magnetic diffraction is proportional to the square of XMCD signal. There is a possibility that the large orbital magnetic moments play a role in forming the helical magnet and skyrmion crystal.
4. Conclusion

We have developed a diffractometer for small angle resonant soft x-ray diffraction under magnetic field, and installed the instrument at Photon Factory. We have demonstrated the capability of the instrument through soft x-ray magnetic circular dichroism and resonant soft x-ray magnetic diffraction measurement from Fe$_{0.5}$Co$_{0.5}$Si. Future improvements to this system consist in a movable CCD camera stage, currently under development, for varying a sample-to-detector distance which corresponds to an observable $Q$-range.

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