| ESRF | **Experiment title:**<br>Resonant X-ray Magnetic Scattering Study of USb at Sb K-edge | **Experiment number:**<br>HE 1676 |
|---|---|---|
| **Beamline:**<br>ID15A | **Date of experiment:**<br>from: 10 March 2004 to: 16 March 2004 | **Date of report:**<br>24/11/04 |
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## Report:

The aim of the experiment was to see if, for photons tuned to the Sb K-edge (E = 30.491 keV), resonant X-ray scattering (RXS) occurred in USb associated with its antiferromagnetic (AF) order. This followed earlier experiments on other (AF) U systems, which observed RXS at K-edges of non-magnetic ions [1,2], and a theory to account for these effects [3]. The situation making USb "new" was that Sb is a 5*p* ion, while the non-magnetic ions in the previous (RXS) studies had all been 4*p* [1,2]. In addition, to our knowledge, a RXS experiment had never been attempted at such a "hard" X-ray energy (E = 30.491 keV); the previous K-edges investigated where at energies between 10 and 13 keV [1,2].

Given that the Sb K edge falls just above the low-E cut-off (~ 30 keV) of the spectrum of (dipole) radiation "viewed" by ID15A - with the maximum flux occurring at around 60 keV - and that USb has a magnetic structure commensurate (with k = 1 r.l.u.) with its (fcc) crystal structure, it was a priority in our experiment to minimize the contamination of *any* RXS (which would occur at the magnetic satellite positions, (hkl) mixed integer) by higher-order (charge) scatter. In order to maximize the $\lambda$:$\lambda$/n ratio in the scattered beam, the thinnest (Laue) crystal available at ID15A (a Ge (311) crystal) was employed as the second monochromator, and an analyser crystal was not used. A Ge solid state detector was opted for, to allow for discrimination betwen the signal of interest ($\lambda$) and unwanted, higher-order scattering ($\lambda$/n).

A relatively large (1 x 1 x 2 $mm^3$) single crystal of USb was mounted on the 4-circle diffractometer of ID15A, in a displex cryostat (borrowed from the *XMaS* beamline). The Sb-K energy was calibrated, in the standard way, by recording the fluorescence yield from the sample. Shown in Figure 1 is the Sb $K_\alpha$ "partial" fluorescence yield, obtained by "windowing" (in the MCA of the Ge detector) a range enclosing the Sb $K_{\alpha 1,2}$ photon energies. Two data-sets are shown, recorded successively, the first measurement being that with the "crude" (i.e. larger) energy step-size. One observes inconsistency between these different "runs", the two curves being offset by 8 eV. We attribute this discrepency to position irreproducibility in the stepping motors controlling the Bragg angle of the monochromator crystals - a positioning error of +/- 8 eV.

Setting the incident X-ray energy to the Sb K-edge energy (we used the value obtained from the second fluorescence run, bearing in mind a repositioning error of +/- 8 eV), a single-crystal alignment was performed, at base temperature (T = 10 K). We then searched for RXS at the magnetic position (005); we chose this position as it was the satellite of largest scattering angle available within a hard limit ($2\theta_{max}$) of (approximately) 17 °. Sample-rocking ($\Omega$) scans were performed through (005) as a function of the incident X-ray energy, tuning through the Sb K edge using various choices of energy step-sizes, from 1 to 10 eV, to combat the problem of energy setting error ($\Delta$E ~ 8 eV). We found an $\Omega$ dependence of scattering in $\lambda$ (the MCA channels covering an energy window of 30 to 31 keV were designated as the "$\lambda$" signal) to appear as the edge was approached, with a peaked-form of the dependence consistent (from its FWHM (in $\Omega$)) with it being diffraction (and, hence, possibly RXS) from our sample. However, the E dependence of the (Ω) integrated intensity of the λ signal did not follow the usual lorentzian form, leading to us to doubt that we had observed RXS. To investigate the development (with E) of this Ω-dependent signal in λ, we collected, as a function of (Ω,E), the entire MCA output (this corresponded to an E range spanning 10 to 200 keV).

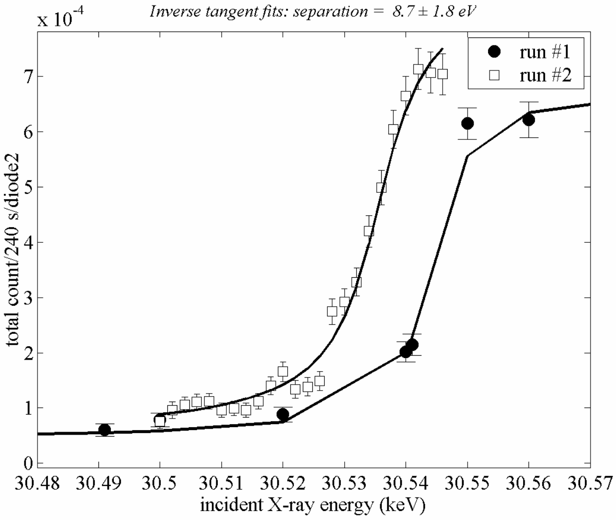


**Fig. 1.** Sb $K_\alpha$ (partial) fluorescence yield from USb, obtained by two data runs. The fitted curves are inverse tangent functions; the positions these functions differ by 8 eV.

Figure 2 shows the most dramatic situation encounted in our acquisition of MCA output, obtained with E set (nominally) at the edge (= 30.491 keV) and Ω set to the (005) position: the higher-order scattering (λ/n) is a maximum and gives rise to a broad signal at and around the λ position (see inset of figure). The intensity of this (parasitic) signal was found to scale with the intensities of the high-order (diffraction) signals; away from the diffraction condition(s), a peak due to Sb $K_\alpha$ fluorescence could be identified, but in Figure 2 (inset) this signal is lost in the noise of the (parasitic) signal. This explained the (Ω,E) dependences we had observed (see above) when looking only at λ (integrated channel) signal. Our search for RXS was thus rendered (technically) impossible due to lack of necessary sensitivity, at the energy of interest, caused by higher-order (charge) scattering.

We conclude that any future search for RXS (from such a commensurately ordered system as USb) at these energies (~ 30 keV) should strive to minimize further than was possible to do so in this study the effect of higher-order scattering. The use of a Ge solid-state detector (energy resolution around 100 eV) has been shown to be insufficient for such higher-order discrimination, even when the total sum (i.e. all photon energies) of scatter in the detector never exceeded the nominal saturation limit of 10,000 (total) count/sec.

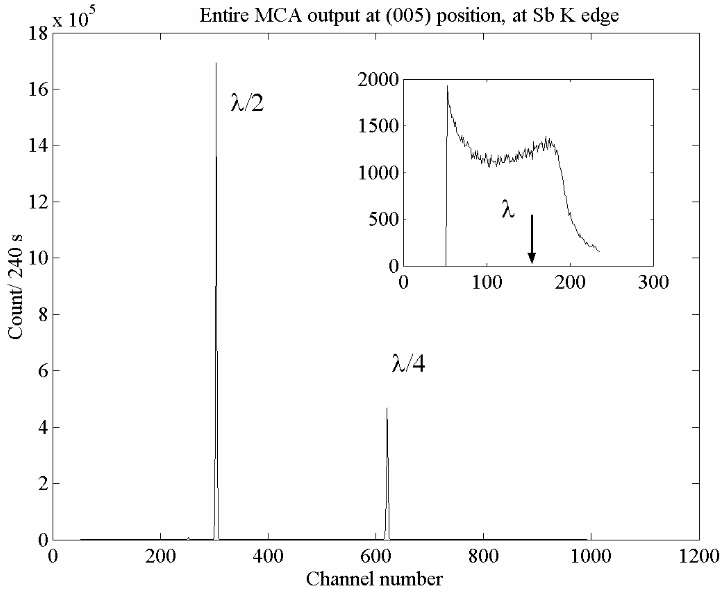


**Fig. 2.** Entire MCA output of the Ge detector, obtained at the (005) magnetic position, with the incident X-ray energy beam tuned to the Sb K-edge. The peaks labeled "λ/2" and "λ/4" denote higher order charge scattering (namely, the (0,0,10) and (0,0,20) Bragg reflections, respectively). The inset shows a close-up of the energy region around the principal harmonic "λ", i.e. where RXS would be found. Instead of observing RXS, a large "background", continuous in energy between channel numbers 50 (minimum energy detectable) and 200 is present. This background intensity was found to scale with the intensity of the higher-order scattering.

## References

[1] Mannix *et al.*, Phys. Rev. Lett. **86**, 4128 (2001)
[2] Longfield *et al.*, Phys. Rev. B **64**, 212407 (2001)
[3] van Veenendaal, Phys. Rev. B **67**, 134112 (2003)