ESRF

Experiment title:
Anisotropy of Anomalous Scattering in Cuprite, Cu_2O, and Magnetite, Fe_3O_4

Experiment number:
HS - 36

Beamline:
BM 0 1

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Report:

Using crystal spheres of 0.3 mm diameter rather than large crystals exhibiting natural or cut faces this experiment was intended to assess both quality and acquisition speed of ‘forbidden’ reflections excited at an absorption edge by Anisotropy of Anomalous Scattering (AAS).

Cuprite, Cu_2O, and Magnetite. Fe_3O_4 were chosen, since earlier experiments on large specimens had revealed well measurable effects, though considerably weaker for the latter one. In agreement with the recommendation of the scientific commitee, Magnetite being considered the more interesting sample was mounted first. However, the search for the ‘forbidden’ axial reflection 600 failed. Upon inspection of the intensities of some allowed reflections it was realized that the primary intensity was too low to observe this weak reflection. Attempts to detect the anisotropic resonant scattering from the intensity variations $I(\psi)$ of allowed reflections, e.g. 800, qualitatively showed the expected effects, however, again with inadequate signal to noise ratio.

In order to find the reason for these unexpected difficulties the primary beam intensity, reflected by the vertical scattering from a Kapton foil, was studied as a function of wavelength (Fig. 1). Tuning λ from 1.0 $\AA$ to 1.75 $\AA$, I_0 dropped by 2 orders of magnitude, an effect that cannot be explained by the energy dependence of absorption in Be and air. Taking into account the scattering from the foil an estimate shows that the effect produced by the experimental setup must be small compared to the observed one. Therefore. the intensity behaviour had to be attributed to the specific beam line construction.

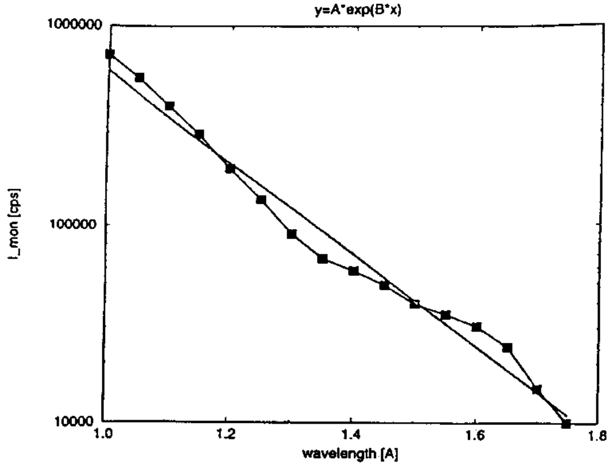

Fig. 1
Monitor counts from Kapton foil scattering vs λ.

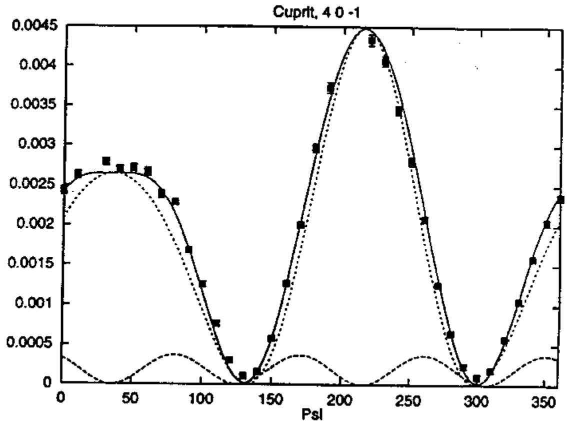

Fig. 2
$I(40\bar{1};\Psi)$: experimental points and model fit (solid line).
Dotted line: $I(\sigma\sigma)$
Dashed line: $I(\pi\sigma)$

Due to this finding the remaining experiment time was devoted to studying 'forbidden' and very weak 'quasi-forbidden' reflections (of parity eeo) of Cuprite for the well established resonance at the Cu-K absorption edge. Intensity variations $I(\Psi)$ for several (normally extinct) axial and zonal reflections as well as for some eeo-reflections were successfully recorded. $I(40\bar{1};\Psi)$ (Fig. 2) illustrates the quality of these measurements carried out on a small crystal sphere. The results are definitely superior to those earlier obtained on large crystal faces. All observations are in agreement with model calculations, and thus corroborating the scattering model. Additional tests on spheres of $K_2(Cu \cdot 6H_2O)(SO_4)_2$ and $CuSeO_3 \cdot 2H_2O$ yielded first evidence of significant AAS-effects (for the latter at both Cu- and Se-edges), which, however, could not be studied into more detail.

In summary, this experiment has shown:

i) The instrument performance is well suited for AAS-measurements on small samples. This includes the available software which could be adapted to various measuring routines without great difficulties. Consequently, valuable results can be obtained from small samples in acceptable time, provided the required radiation energy exceeds about 8 KeV.

ii) Life time and positional stability of the beam are excellent so that unwanted effects due to energy instabilities and/or shifts could hardly be detected. This being the most important prerequisite for reliable measurements within narrow resonances makes the source as attractive as hoped for.

Special thanks are due to E. Weckert for most important help with the experiment, and further support by K. Knudsen is gratefully acknowledged.