ESRF	**Experiment title:** Sequential data aquisition procedure for XAS with the energy dispersive monochromator at ID24	**Experiment number:** **MI-259**
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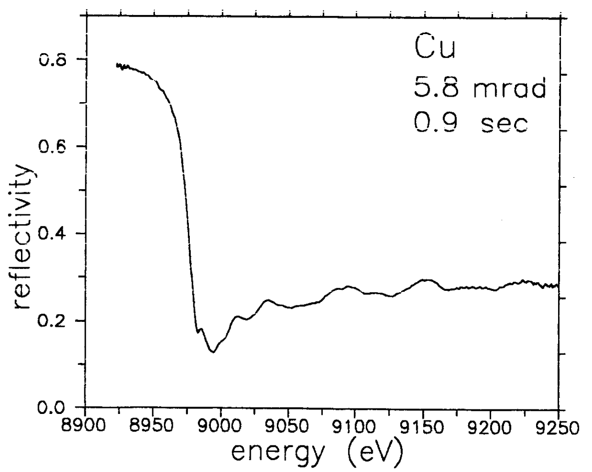
Report:

The objective of our proposal was to test in collaboration with ID24 staff members a new data recording procedure for the energy dispersive monochromator at the ESRF beamline ID24. Instead of using the existing position sensitive detector system (PSD) we planned to use conventional two ionisation chambers for the simultaneous measurment of I_0 and I,. In that way we hoped to overcome difficult problems with the I_0 normalization by the use of PSD [1]. In order to select only one narrow energy range at a time from the polychromatic X-ray fan behind the monochromator, a narrow slit (20 - 50 microns) should be moved with an adjustable speed across the fan. First tests with this arrangement were performed by the ID24 staff members at the beginning of 1998 and were very promising. In the following time the technique was further developed at ID24 and it was tested for different absorption edges using reference metal foils as well as during real experiments [2]. The output signal from the ionisation chambers is amplified by the Keithley 428 current amplifiers and is directly converted using an AD-converter. The energy resolution is defined by the opening of the moving slit is typicaly in the range of 1 eV. Using this techniqe for transmission type experiments, the recording time for a spectrum can be made shorter than 1 second. Therefore, it is well suitable for experiments than do not need very high time resolution.

So, at the the beginning of our beam time the technique was already working and we decided to concentrate on the reflection type experiments. The main drawback of the reflection mode experiments is the very small vertical size of the beam that has to be used. For example, at a glancing angle of 5 miliradians and a sample length of 10 mm the vertical beam size accepted by the sample is max. 50 microns. A further reduction of the beam height by a slit in front of the I_0-chamber is difficult due to the specie-like phenomena produced by such a narrow aperture. The other problem is the necessity for the separate partial beams of the x-ray fan to hit the sample surface nearly at the same height. This turned out to be the main problem in our attempts to use samples with lenght of about less than 4 mm necessary for the electrochemical *in situ* experiments in the energy range below 10 keV. The visible sample height at 5 mrad is then below 20 microns and every part of the fan should hit the sample surface within about 10 microns. We were not able to focus the beam verticaly within these limits. However, experiments with larger samples were possible. We performed reflection mode test measurments using a flot glass plate (3x5 cm^2) covered with a thin copper film. The measurments were performed at diffrent glancing angles below and above the critical angle. The shortest integeration time was 0.9 s for a spectrum with an energy range of about 300 eV. The sample was stored in air for about three years and as a result the copper surface was covered with a several nanometers thick copper oxide layer. Figure 1 shows as an example the energy dependent reflection mode spectrum for an glancing angle of 5.8 mrad. The x-ray penetration depth for that angle is about 6 nm and therefore the spectrum contains information from the top oxide layer as well as from the underlaying metallic copper. The total integration was 0.9 sec. In conclusion, without farther improvements of the beamline reflection mode spectroscopy is possible at ID24 for samples allowing an x-ray beam height more than about 50 microns. The time resolution achieved for such samples is better than 1 second. It seems reasonable that the further improvements of the beamline optical elements (mirrors, slit system) will result in a possiblility of using significantly smaller samples and even shorter integration times.

Fig. 1



[1] H.-H. Strehblow et *al.*, CH-369 experiment report, 1998
[2] S. Pascarelli et al., XAFS X Conference, Chicago, 1998