REPORT

Beamtime 26-01-904 DUBBLE
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Introduction and overview of experiments

A newly designed CCD microscopy instrument, X-ray excited optical microscopy 1 (XEOM1) was further developed and tested on the DUtch Belgian BeamLinE (DUBBLE) following initial tests on DUBBLE in December 2010 and XMaS in February and May 2011.

X-ray Excited Optical Microscopy (XEOM) is a surface specific imaging technique based on x-ray excited optical luminescence (XEOL). XEOM1 is so constructed that optical light in the range of 200 – 1000 nm can be collected by a 2048*506 CCD in a time span of 0.5 s upwards, consequently creating chemical maps with an ultimate lateral resolution of 1 µm. This short acquisition time provides the possibility to collect complete image stacks across the XANES edge in a period of ± 400s. During previous beamtimes at XMaS, image spectra were acquired automatically which was, up till now, not possible at DUBBLE. The effect of Ultralene® on XEOL spectra was further tested thoroughly, following earlier experiments at XMaS as well as the effect of cumulative x-ray dose on the Ultralene® itself. Various dichroic color filter were used to unravel in which particular band of the EM-spectrum, copper and its combination with Ultralene® is emitting. An attempt was made to acquire chemical maps of lead bronze and tin bronze samples with different corrosion layers.

Experiments

The hardware as well as the software of the XEOM1 was updated to acquire XEOM spectra automatically. By synchronizing the camera triggering with the monochromator motions via pulses of the Time Frame Generator (TFG), various image spectra of different bronze samples could be recorded. A new illuminator- consisting out of a LED light, shutter and diffuser - was added to the set-up of the XEOM1. The illuminator is used to expose the samples with white light and acquire flat-field images since it can be mounted both in the 45° port as well as in the sample stub.

Following the previous beamtime at XmaS in May 2011 the effect of the beam on the Ultralene® optical transmission was investigated by measuring the XEOL counts at 9.027 keV both unfiltered and filtered with various dichroic color filters. Concerns had risen about reproducibility of these measurements, especially when the Ultralene® was in contact with the copper surface. When an air gap is present between the copper surface and the Ultralene® window, created by a Viton® O-ring, XEOL-spectra appear to be reproducible and a database was created of spectra of Copper + O-ring + Ultralene® with a complete set of color filters.

An experiment was set up in order to test the properties of Ultralene® after it being irradiated with X-rays. During the beamtime the irradiated Ultralene® was placed in the filter-carrier position in

order to test the effect on the XEOL-signal, where after the beamtime the physical characteristics of the damaged Ultralene® will be investigated (e.g. with light microscopy). During this experiment, the effect of cumulative dose on Ultralene®, and therefore also the normalized XEOL-intensity, was tested.

Images of lead bronze samples with cuprite, nantokite and nantokite/paratacamite and tin bronze with cuprite were captured as well as short and full range image stacks were recorded during this beamtime. For the first time image spectra were acquired with dichroic color filters.

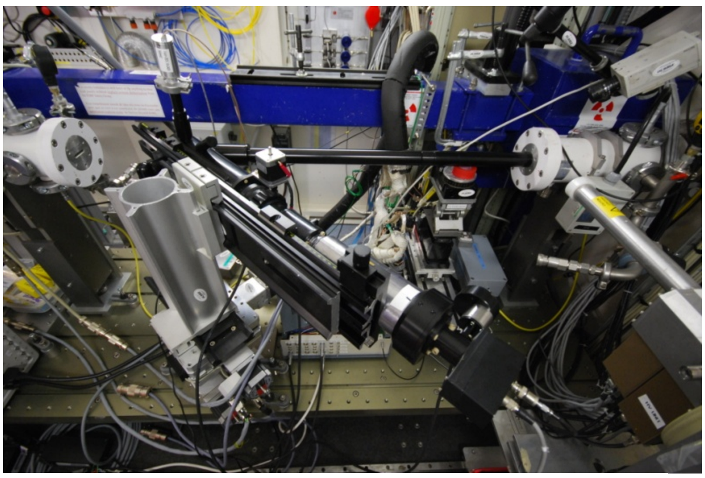
First Conclusions

The hard- and software were successfully implemented into XEOM1. The problem of missed triggers, as was seen during the previous beamtimes at XMaS, did not occur during this beamtime. The new illuminator was successfully used during this beamtime.

When Ultralene® is in close contact with the copper surface, XEOL spectra seem to have lost their reproducibility. This problem could be solved by creating an air space in between the sample surface and the Ultralene® via a Viton® O-ring. Via tests through a complete set of dichroic color filters and comparison with data acquired at DUBBLE in December 2008, we could conclude that Ultralene® does not change the waveband, in which copper is emitting. On the other hand it could be concluded that Ultralene® is the cause of a fierce drop in XEOL intensity.

Irradiated Ultralene® has no effect on the copper XEOL spectrum when the 'damaged' Ultralene® was put into the filter carrier and therefore interfering with the visible light emitted by the sample. Alterations in the physical properties of the Ultralene® will be looked further into. In contrast with what was previously mentioned, when Ultralene® is continuously struck by X-rays, it appears that the cumulative X-ray dose has a strong effect on the intensity of the XEOL signal.

Image stacks of bronze samples were recorded with success. The XEOM images will be further investigated during with the Region of Interest (ROI) - analysis using an updated version of EsaProject.



XEOM1 set-up during beamtime at DUBBLE.