REPORT

Beamtime 26-01/884 DUBBLE

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1. Background and Objective

X-ray Excited Optical Luminescence (XEOL) is an emerging technique in the field of X-ray absorption spectroscopy. During the XEOL-process, a sample is bombarded with X-rays (usually from a synchrotron) to induce core-level photo-ionization of specific atoms. The emitted photoelectron, or the events subsequent to the photo-ionization process (e.g. Auger transitions), may directly/indirectly induce electronic transitions which results in the emission of light in the visible or near-visible region. During a XEOL-experiment the energy of the incident X-ray beam is scanned across the absorption edge of the target atom. The intensity of the emitted (nearly-)visible light is, close to the absorption edge, dependend on various scatter processes and is modulated by the photoelectron escape probability at slightly higher energies above the edge. Consequently, this optical emission (therefore also the XEOL-spectra) carries site specific information related to the information provided by XANES- (X-ray Absorption Near Edge Spectroscopy) and EXAFS- (Extended X-ray Absorption Fine Structure) spectra. Simultaneously the spectrum of the emitted light itself carries multiple additional information channels, for example, emission characteristic of the electronic state in the vicinity of the ionized atom. Since optical photons can only travel short distances within an opaque sample, the resulting spectra/images are characteristic of a layer less than 200 nm thick on the sample surface.

XEOM, in full X-ray Excited Optical Microscopy, was tested for the first time during the present experiment at DUBBLE (ESRF, Grenoble). XEOM works according to the XEOL principle but uses a (1024 x 512) CCD-detector. This is different from XEOL, where a photomultiplier tube is used to record the spectra. Employing this broadband detector has – in theory – various advantages with respect to a photomultiplier tube. The rapid collection and display of the chemical state and local atomic order in µm-resolution scale next to X-ray micro-focus beam lines becoming redundant for obtaining micron-scale lateral resolution maps are only some of this advantages. The objective of the measurements executed at DUBBLE is to test if this theory can be coupled to reality.

2. Experiments

The experiments performed during this beam time were aimed to test the possibilities and restrictions of the XEOM equipment so far. After proper alignment of the XEOM equipment, attempts to image a bare copper grid (200 mesh) and a fluorescent atacamite grid remained at first unsuccessful. Adapting the software, cooling down the camera to as low as -37.25 °C, focusing the energy of the beam onto one spot and filtering out undesired light with a black curtain, a cardboard tube and aluminum foil made it possible to make the first XEOM picture of cuprite (Figure 2). In addition we were able to image the cuprite grid on top of a nantokite coupon across the Cu K edge. The total integrated intensity of the image acquired during 20 seconds has been plotted against the beam energy (8.98 keV – 9.04 keV with 0.5 eV step size) in Figure 3 and matches a typical cuprite XEOL spectrum.

We remained unsuccessful in imaging bare copper or other copper compounds. A discussion with the manufacturer of the camera after the beam time has indicated that background X-rays may be scattered into the ventilation grilles of the camera, building up charge somewhere, and causing the camera to flush at some critical level. We will check this at the next beamtime by putting an aluminium absorber or lead around the camera.

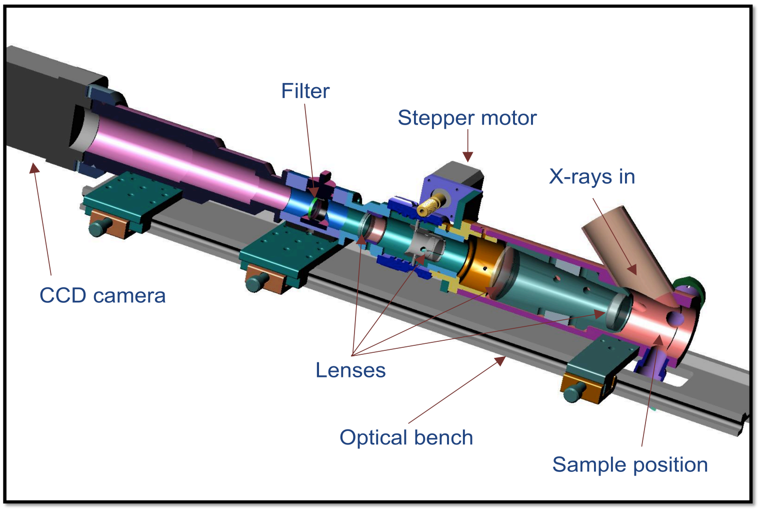

Figure 1: XEOM equipment

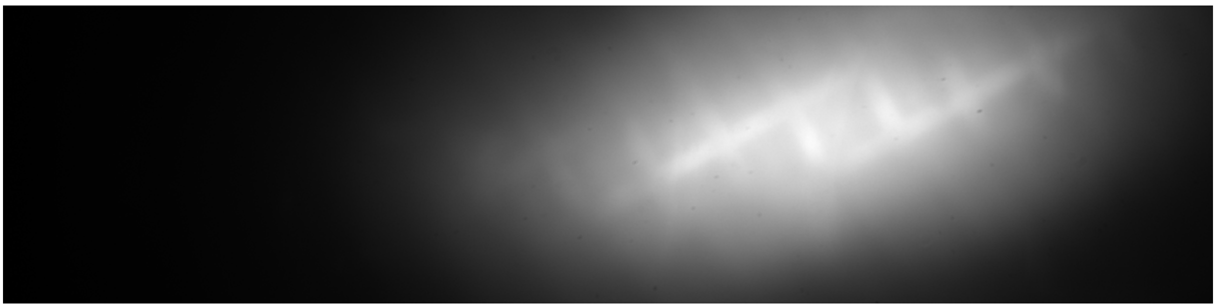

Figure 2. XEOM image of cuprite grid on nantokite substrate

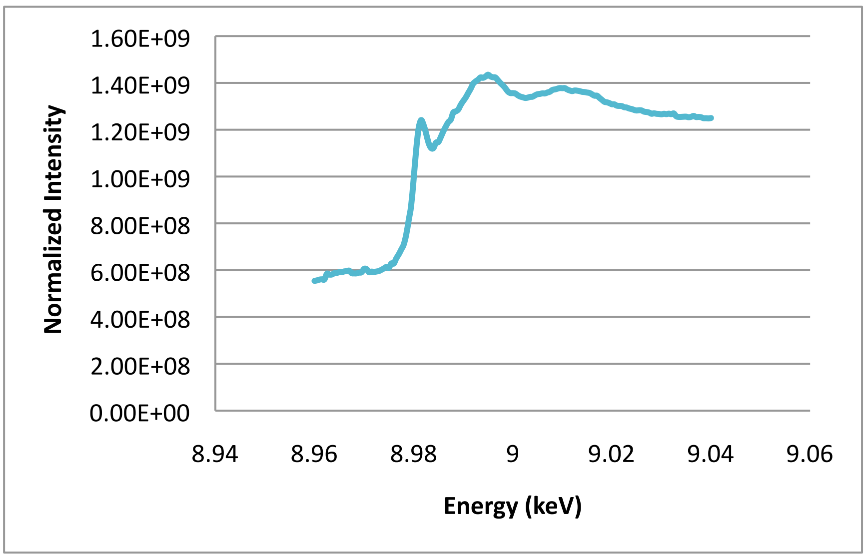

Figure 3. XEOL of cuprite mesh on nantokite substrate obtained by integrating 161 XEOM images across the Cu K-edge