

Developing An X-ray Excited Optical Luminescence Microscope

M. Hand¹, M. Dowsett¹, A. Adriaens²



Introduction

X-ray absorption spectroscopy (XAS) refers to a set of techniques that use synchrotron radiation as a probe which can be used to determine the local environment of a particular elemental species within a sample. In general, XAS spectra are an average across the sampled area, i.e. the beam footprint. Micro-focus beam lines with footprints on the micron scale allow spectroscopic maps to be produced, but obtaining a complete EXAFS spectrum for each 'pixel' in a large map is a very time-consuming process. How can we speed this up?

We are currently developing a new X-ray excited optical luminescence microscope (XEOM) which uses the visible emission caused by X-ray bombardment to map the electronic state and local atomic order at the surface of a sample. It will be possible to obtain micron-scale lateral resolution maps, but using an X-ray source with a large footprint, such as a conventional bending magnet beam line.

XEOL for XAS

The emission of photoelectrons as X-rays strike a sample gives rise to a range of secondary processes which cause light to be emitted; this is referred to as X-ray excited optical luminescence (XEOL) [1]. The visible and near-visible emission is modulated by the escape probability of the original photoelectron, as for X-ray fluorescence, so the same structural information is encoded in the light.

- XEOL has a greater surface specificity (< 200 nm) than conventional X-ray absorption techniques.
- Emission in specific wavebands gives extra local information.
- Manipulation of the visible emission is easy using conventional optics.



Figure 1: The XEOM is mounted on the Huber goniometer at beam line BM28 (XMaS) at the ESRF, Grenoble, France.

We have developed a proof of concept device which uses a simple two-lens system to maximise its light collection efficiency. A photomultiplier tube mounted at the end of the optical column is used to measure the intensity of the visible emission.

The XEOM couples directly with a novel electrochemical cell (eCell) [2] which allows electrochemical and spectroscopic measurements to be obtained in parallel.

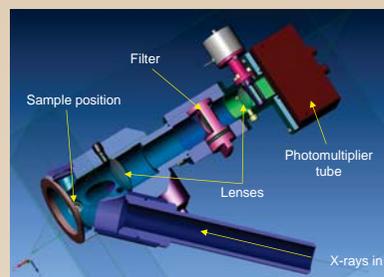
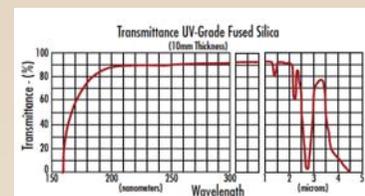


Figure 2: 3D section of the existing XEOM. A number of ports at the base provide flexibility when mounting the XEOM and allow additional detectors to be used. The XEOM and the eCell are both controlled via a laptop – data acquisition can be automated allowing time-resolved experiments to be carried out over the course of many hours.

Fused silica is used as the lens material due to its optical and physical properties:

- Flat transmission profile from near-UV to infra-red.
- >90 % transmittance in this region.
- Low X-ray fluorescence.



The body of the XEOM is constructed from black acetyl copolymer; it is easily machined and, importantly, exhibits low X-ray fluorescence.

Measuring XEOL and fluorescence in parallel

The spectra in Figure 3 are the result of parallel collection of XEOL and X-ray fluorescence signals from a bare copper coupon. They also indicate the effect of using an Ultralene® window and filling the cell with liquid (in this case, deionised water).

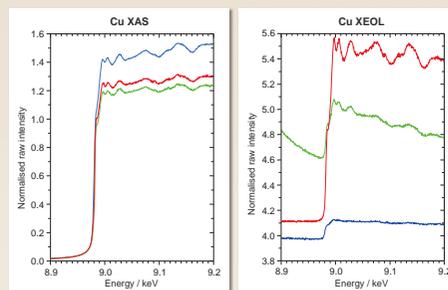


Figure 3: The Ultralene® window appears to increase the background luminescence. This is not seen when the water is introduced; it is possible that the water modifies the Ultralene®, or even permeates the window.

References

- [1] A. Rogalev and J. Goulon, in *Chemical Applications Of Synchrotron Radiation Part II: X-ray Applications*, T-K Sham (World Scientific, Singapore, 2002), pp. 707-760
- [2] M. G. Dowsett and A. Adriaens, *Analytical Chemistry* **78**, 3360-5 (2006)

Designing the XEOM

A ray-tracing package, OSLO (Optics Software for Layout and Optimization), has been used to design the lens system for the new XEOM. The software incorporates diagnostic (Figure 4) and optimization routines for assessing and improving the simulated design.

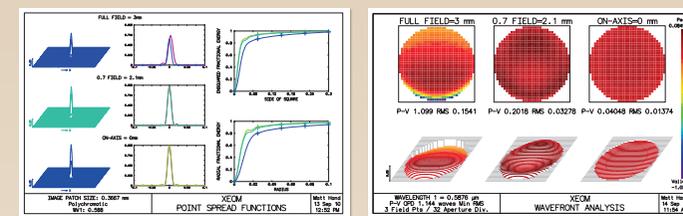


Figure 4: Graphical reports such as (a) point spread function and (b) wavefront analyses provide a useful indication of the expected performance of the optical system.

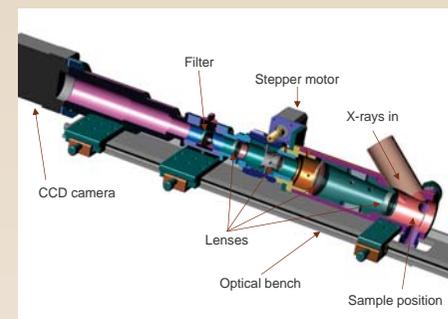


Figure 5: 3D section of the new XEOM, shown with a simple filter carrier. The focusing lens can be operated manually or controlled remotely by driving it with a stepper motor. The total length of the system is increased to accommodate the new optics which provide ~12x magnification.

- The optics system includes two aspherical lenses to minimise spherical and other aberrations.
- Focusing mechanism to allow imaging of different wavebands across the trans-visible region.
- Images captured using a 2048x506 pixel CCD detector.

Future developments include:

- Automated filter system to allow many wavebands to be imaged without the need to replace filters.
- Second optical path for spectroscopy.

Acknowledgements

The eCell and XEOL optics were developed using private funds from EVA Surface Analysis (UK) and from the Paul Instrument fund. The development was supported by ESRF Long Term Project EC188 and Fonds voor Wetenschappelijk Onderzoek (FWO) (Belgium). The author's own work is supported by the EPSRC.

Contact Info

¹ Department of Physics, University of Warwick, Coventry, CV4 7AL, UK
Matthew Hand: m.hand@warwick.ac.uk
Prof. Mark Dowsett: m.g.dowsett@warwick.ac.uk

² Department of Analytical Chemistry, Ghent University, Krijgslaan 281-S12, B-9000 Ghent, BE
Prof. Anemie Adriaens: anemie.adriaens@ugent.be