

Beam time 26-01-926 DUBBLE
30.11.2011 – 05.12.2011

Annemie Adriaens¹, Mark Dowsett², Rosie Grayburn², Matt Hand² and Pieter-Jan Sabbe¹

¹ Department of Analytical Chemistry, Ghent University, Krijgslaan 281-S12, 9000 Ghent, Belgium

² Department of Physics, University of Warwick, Coventry, CV4 7AL, UK

Introduction

This work continues the development of a portable instrument for X-ray excited optical luminescence microscopy (XEOM), a novel analytical technique which uses the visible emission caused by synchrotron X-ray bombardment - X-ray excited optical luminescence (XEOL) - to map the electronic state and local atomic order at the surface of a sample. XEOM1 is a CCD-based microscope which can be used to collect image 'stacks' while scanning the X-ray energy to obtain surface specific chemical and structural information. There is a potential advantage in imaging the surface, rather than the more conventional method of using a micro-focus beam as scanning probe, since data acquisition can be much faster, though we have found this to be very sample-dependent. The ultimate goal is to develop this instrument into a tool for surface analysis of cultural heritage artifacts.

Microscope Testing and Calibration

It has become apparent during previous use of the microscope that the inherent chromatic aberration caused by the optics is a problem for seeing chemical differentiation in the captured images. It is already expected that we need to filter the XEOL-emission to form sharp images, but there was a question mark over whether the filters we have been using are effective; their transmittance is not defined over the entire spectral band to which the camera is sensitive.

For this beam time, two new filters were brought to try and solve this problem: a visible bandpass filter to be used in combination with the existing colour dichroic filters, and a UV bandpass filter. The initial period of beam time was used to carry out diagnostic tests of the optics, namely attempting to find the best image focus for each filter combination. Unfortunately, we did not see the improvement in image quality that we had hoped for, but the focus positions provided a basis for capturing the highest quality images during the remaining experiments. Further investigation into the optics behaviour will be carried out offline.

50/50 Copper/Nantokite and Copper/Atacamite Coupon Filtered Image Stacks

A new set of corroded copper samples was prepared prior to the beamtime. Half of the surface of a polished copper coupon (12.6 mm diameter) was coated with a cupreous corrosion layer leaving the remaining half bare, creating two separate copper regions with a sharp interface. Image stacks were recorded for a 50/50 copper-nantokite (Faltermeier protocol) and a 50/50 copper-atacamite (Lamy protocol) sample. Image stacks of the interface between the adjacent copper products were taken over the copper edge from 8.960 – 9.016 keV in 0.5 eV steps (for direct comparison with previous measurements).

Specific wavebands from the XEOL emission were isolated by using a number of filter combinations. The visible bandpass filter was used in combination with red, green and blue dichroic filters to ensure that no UV or IR wavelengths were permitted. A UV filter and a red/longpass filter combination were also mounted so that the UV and IR regions respectively could be isolated separately. Region of interest (ROI) analysis was performed on all image stacks using esaProject to investigate whether the two regions could be differentiated chemically using the extracted spectra. Six rectangular ROI's were chosen and applied to each of the image stacks. The resulting spectra showed both positive and negative edges. Nantokite and atacamite spectral shapes were clear in some spectra, but others were too noisy for chemical

identification. A more detailed look into this data will hopefully provide us with an answer on how good the chemical differentiability is for the moment and how to improve this.

Cuprite Grid on Copper and Cuprite Powder Filtered Image Stacks

Image stacks of a cuprite (Cu_2O) grid on copper and cuprite powder samples were taken using an array of filter combinations to isolate red, green, blue and IR parts of the EM spectrum. A range of camera exposure times were used depending on the intensity given by the filter combination and sample – generally images taken with the red filter required a shorter exposure, as did the powder sample.

A combination of the red and longpass filters isolated the red/IR region of the spectrum. The shape of the red/IR emission from the cuprite grid alluded to the presence of tenorite (CuO) and cuprite. One tenorite feature at 8.996 keV diminishes in the red, green and blue emission whereas the other tenorite peak at 8.987 keV is present in all the spectra. The presence of tenorite on the grid could be an artefact of the cuprite grid synthesis. Tenorite was not seen in the cuprite powder sample. In both samples the intensity of red/IR emission is much greater than red and green emission and there is almost no blue emission.

After looking at a grid and powdered sample of cuprite through a range of filter combinations it was found that cuprite emits strongly in the IR region. Copper is not seen in these image stacks due to the long exposure time required to achieve intensity from copper.