

Spherical Grating Monochromator (SGM) Beamline 11ID-1

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Beamline Overview

Status	Operational – accepting proposals
Source	45 mm Planar Undulator
Energy Range	250 – 2000 eV
Resolution (E/ΔE)	>5000 @ 1500 eV >12000 @ 285 eV
Flux @ 100 mA (ph/s)	1012 at 285 eV 1011 at 1840 eV

Introduction

2009 marked the SGM beamline's third complete year in full operation and saw a total of 310 user shifts delivered to 32 different user groups. The bulk of the activity took place on the Solid State Absorption (SSA) endstation and consisted of high resolution X-ray absorption spectroscopy of environmental and material science samples. An increase in the number of shifts delivered for projects on the XPS endstation was noted as well as considerable use of the XEOL technique. There was a general increase in the complexity of experiments that was observed throughout 2009 with considerably more work being done on the chemical speciation of low concentration samples, in situ sample preparation and sample loading in an inert atmosphere. This is seen as an indication of the growth in the maturity of the SGM user community who are now attempting to push the capabilities of the beamline and endstation hardware. Another positive indicator of the health of the community was the 100th publication containing data taken at the SGM (including refereed journals (68), conference proceedings (17), theses (13) and book chapters (2)). For this the SGM users are to be commended.

Science

Research highlights in both the environmental science and material science fields were seen in 2009. A collaboration among researchers at the University of Rostock, the University of Saskatchewan and the CLS published work on rhizosphere chemistry and cultivation effects in soils [1,2]. These studies used synchrotron-based XANES spectroscopy and Pyrolysis-Field Ionization Mass Spectroscopy to perform chemical speciation of the carbon and nitrogen in whole soils.

The utility of soft X-ray absorption spectroscopy for studying the relative abundance of different organic compounds in untreated soil samples was demonstrated in these works.

The results of a study on the distribution of Y(3+) and Gd(3+) in NaYF(4)/NaGdF(4) core/shell nanocrystals was published by a University of Victoria group in 2009[3]. This investigation looked at the ratio of the Y 3d to Gd 4d core level photoemission intensities as a function of excitation energy (or the photoelectron kinetic energy) which showed that the Gd lies primarily at the surface of the nanocrystals, providing proof that the core/shell structure exists in these nanocrystals. This work was published in the Communications of the Journal of the American Chemical Society in October 2009.

New Capabilities

Significant upgrades were made to the SGM beamline endstations in 2009. The most significant was the development of a new sample holder system based on the one designed for the REIXS beamline. A photo of the transfer arm and the sample manipulator is shown in Figure 1. This upgrade improves transfer reliability and user friendliness, and accommodates helium and nitrogen cooling, with sample temperatures of 20 K demonstrated. The sample manipulator was also upgraded for motorization which facilitates more accurate sample positioning and automated multi-sample scanning. The same sample transfer system is now installed on both the SGM endstations.

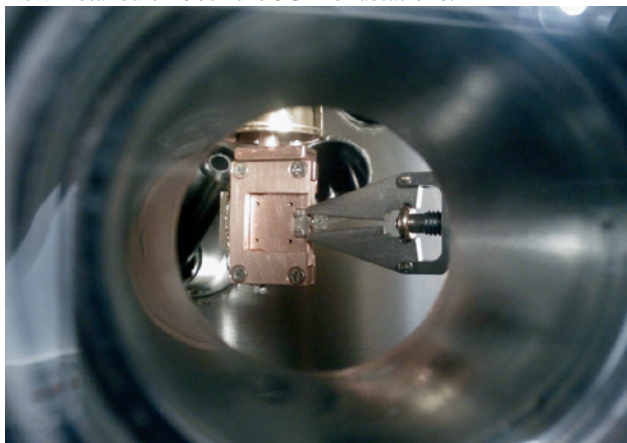


Figure 1: Photograph of the new SGM transfer system taken through a viewport on the Solid Sample Analysis (SSA) endstation. The same sample transfer system is now installed at both of the SGM endstations.

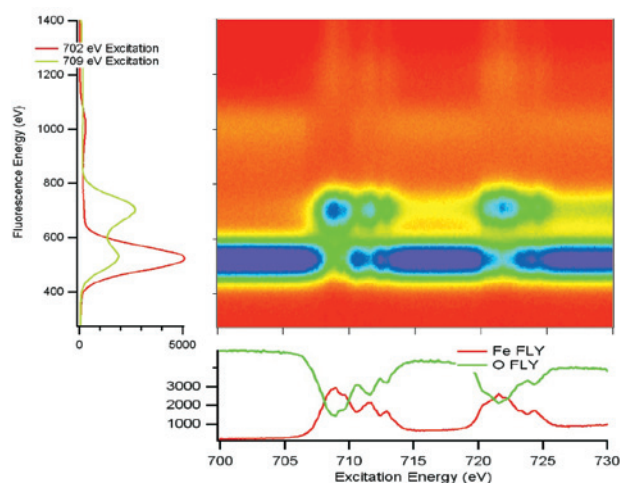


Figure 2: Fluorescence yield of FeO₂ nanoparticles measured by a silicon drift detector. The plot on the left shows the fluorescence spectrum taken below and at the Fe 2p resonance. The partial fluorescence yield of the Fe and O is shown at the bottom showing a measurement method called Inverse Partial Fluorescence Yield (IPFY)[4].

Over the past year we have also completed the integration of a Silicon Drift Detector (SDD) into our standard detector suite. With this detector it is now possible to obtain a complete X-ray fluorescence spectrum for each excitation energy in a NEXAFS scan. This is a relatively new application for this type of detector technology and allows for the measurement of the fluorescence yield from the non-resonant atoms in the sample as well as the resonant atoms, generating a fluorescence map such as the one shown in Figure 2. This capability has led to the development of a new measurement technique called Inverse Partial Fluorescence Yield (IPFY) by a collaboration of researchers from the University of Waterloo, University of British Columbia, University of Toronto and the CLS [4]. Using this method, it is shown that for certain samples, the X-ray absorption cross section can be determined from fluorescence yield, without the need for self-absorption corrections. It is believed that this new measurement method will find applications on many different beamlines around the world as it facilitates the acquisition of bulk sensitive fluorescence measurements that were previously believed to be inaccessible due to self-absorption.

Commissioning of the Time Resolved XEOL (TRXEOL) apparatus was also completed in 2009. This system uses a streak camera to measure the time evolution of the optical emission of samples excited by X-rays. Close collaboration with the Accelerator and Operations Division (AOD) has resulted in the optimization of the storage ring for producing an extremely pure single bunch with a pulse width of 20 ps (r.m.s). The time resolution of the detector was determined to be better than the pulse width allowing for the extraction of excited state lifetimes on the order of a few hundred picoseconds. General user proposals for TRXEOL experiments are now being accepted.

Ongoing upgrades include the addition of electron flood guns to both endstations as well as the development of a portable

glove box system that can be attached directly to the load lock of either endstation. The glove box will be available for users who wish to prepare samples in an inert environment with direct insertion into the endstation load locks. A liquid flow cell is also being designed and built with the first experiments scheduled for March 2010.

Major Upgrades

In June 2009, the University of Western Ontario was awarded \$4 million for upgrading the SGM and PGM beamlines through the Canadian Foundation for Innovation (CFI) Leading Edge Fund (LEF). This money will be used to replace the two linear undulators with Elliptically Polarizing Undulators (EPU) on the SGM and PGM as well as substantial upgrades to the endstation equipment. The details of the endstation upgrades are still being determined but will involve the addition of focusing optics for the beamline and the development of separate clean and 'dirty' endstations. Instrumentation to exploit the polarization capabilities will also be added as well as improved in situ sample preparation instrumentation. Users are encouraged to discuss their specific instrumentation requirements with the beamline scientist in the coming months so that they might be included in the upgrades.

References

1. Gillespie, A.W., Walley, F.L., Farrell, R.E., Leinweber, P., Schlichting, A., Eckhardt, K.U., Regier, T.Z., and Blyth, R.I.R. 2009. Profiling rhizosphere chemistry: Evidence from carbon and Nitrogen XANES and Pyrolysis-Field Ionization Mass spectroscopy. *Soil Science Society of America Journal*, Vol. 73, pp. 2002-2012.
2. Leinweber, P., Walley, F.L., Kruse, J., Jandl, G., Eckhardt, K.U., Blyth, R.I.R., Regier, T.Z. 2009. Cultivation Affects Soil Organic Nitrogen: Pyrolysis-Mass Spectrometry and Nitrogen K-edge XANES Spectroscopy Evidence, *Soil Science Society of America Journal*, Vol. 73, pp. 82-92.
3. Abel, A.A., Boyer, J.C., van Veggel, F.C.J.M. 2009. Hard Proof of the NaYF₄/NaGdF₄ Nanocrystal Core/Shell Structure, *Journal of American Chemical Society*, Vol. 131, pp. 14644-14645.
4. Achkar, A.J., Regier, T.Z., Wadati, H., Kim, Y.-J., Zhang, H., Hawthorn, D.G. 2009. Bulk Sensitive X-ray Absorption Spectroscopy Free of Self-Absorption Effects. *CLS Activity Report 2009*, pp. 154-155.