Report on CLS Spectromicroscopy Workshop

When: Sunday Nov 14, 1999 9:00 am to 5:30 pm

Where: Physics 103, University of Saskatchewan, Saskatoon, SK

Organizers: Adam Hitchcock, Stephen Urquhart

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Attendance: ~40 (attendee list available)

Brief Summary

The workshop began at 9 am with a brief presentation by *Adam Hitchcock* (BIMR, McMaster U.) of the scope of synchrotron-based X-ray spectromicroscopy along with a description of the main properties of the scanning transmission X-ray microscopy (STXM) and photoelectron emission microscopy (PEEM) techniques which were the subject of the workshop. This was followed by a clear and comprehensive exposition by *Michael Feser* (SUNY, Stony Brook) of the STXM program in the X-ray group at Stony Brook who have built and operated four STXM at NSLS over the past 18 years. After outlining the nature of the X-1 undulator beamline, he described each of the two most modern microscopes – the room temperature STXM-IV and the cryo-STXM. He illustrated the power of these microscopes with examples from environmental (oil in hydrated clays), biological studies (live cells, tomography) and astrophysics (interplanetary dust). Among the latter was an exquisite view of the innards of a single cell (dead!) achieved by cryo-tomography. This type of tomography, performed with variable photon energy and biochemical labeling, will be an incredibly powerful tool to understand the 3-dimensional structure-function relationships of cells. In addition to conveying the excitement of STXM research Feser provided much useful advice concerning how to design a STXM for reliability and performance. The next speaker, *Tony Warwick* (Advanced Light Source, LBNL) was unable to attend at the last minute due to a family emergency so his presentation on the optimization of scanning zone plate microscopes and their beamlines was not delivered (we hope parts of it will be posted on the CLS web site soon). Instead, *Adam Hitchcock* described the concept of phase space and the ways in which beamline designs can be optimized by matching the insertion device and the X-ray optics to the relatively restricted phase space of a STXM. The need for a high brightness undulator source point and careful attention to matching the soft X-ray optics to the actual phase space used by the STXM was emphasized. The relative merits of an elliptical polarized undulator (EPU) versus a more conventional linearly polarized undulator was also discussed. The higher cost and increased complexity, both of construction and operation, would be challenges to CLS. The EPU would enable X-ray magnetic circular dichroism studies of magnetic materials, and facilitate studies of orientation in polymer and other materials . *Howard Padmore* emphasized that the increase in cost was not that great (he estimated US$750K for a linear undulator and perhaps US$1250K for an EPU of the types currently installed at the ALS), and that EPUs will be well established devices in a year or two. He noted that BESSY 2 routinely installs only EPUs.

After the coffee break *Stephen Urquhart* (UoS) gave an overview of Canadian activities in, and prospects for research in soft X-ray microscopy. He stressed primarily STXM applications, and gave many examples including: studies of fungal diseases in plants, geochemical studies (wood preservation, meteorite analysis), biological studies, and many polymer studies. The large number of opportunities for industrial and other applied analysis was stressed. Following this *Pupa De Stasio* (Wisconsin-Madison) lead a discussion of the attributes and state-of-the-art in PEEM. She emphasized the versatility of PEEM. Although it is a UHV surface sensitive technique with a large extraction field and thus high sensitivity to charging for insulating samples, in many cases (at least at SRC) there is often sufficient conductivity to allow studies of organic, biological and polymer samples. She also stressed that the high sensitivity of the current electron optical systems to low energy secondary electrons means that the sampling depth is 2-10 nm, making it more of a bulk or near surface technique rather than a true surface analysis technique. In discussion, both de Stasio and Padmore stressed the benefits of a fixed installation to ensure high resolution and reproducible performance. This is particularly important if the team choses to develop a state-of-the-art aberration corrected system.

After lunch, there was a number of contributed talks dealing with potential applications of the proposed CLS spectromicroscopy capability. *Adam Hitchcock* (McMaster) gave a brief overview of the science in the spectromicroscopy proposal which currently reflects the interests of about a third of the 22 co-applicants. There is a rather even distribution of needs in the STXM and PEEM areas, and there are applications spanning a wide range of disciplines and problems. The second presentation given by *Konstantin Kaznacheyev* (SUNY-SB) described many exciting and often undemonstrated PEEM techniques. He pointed out, that both PEEM and STXM have unique potential for investigations of magnetic properties of materials. Magnetic domain formation, switching and movement of domain walls may be successfully addressed with the above techniques for both ferromagnetic and antiferromagnetic states. The ability to visualize the magnetic field distribution through the magneto-optic effect may be used for instance for investigating superconducting materials. The high spatial resolution offered by PEEM may lead to the imaging of an individual vortex. The PEEM, which use electrons for image formation, may be used at other energy range, such as the hard x-ray or ultraviolet, visible and even IR light (for appropriate low work finction materials!). PEEM is attractive in this regard since submicron resolution is difficult to achieve in many regimes due to the lack of proper focusing elements. Flexibility in implementation of both the PEEM and the STXM microscopes will be needed if some of these are to be achieved. *Farid Bensebaa* (ICPET, NRC) outlined his needs for improved techniques in semiconductor surface contamination characterization. Although PEEM has many of the attributes it was determined that a system like the micro-XPS system at ALS might be better suited for many of these needs. *T.K. Sham* (UWO) described his interest in applying PEEM to problems in the areas of diamond like carbon and organic light emitting diode thin films. These are both hot technological areas and there are a number of active Canadian groups (e.g. D'Iorio, IMS). Capabilities to study these systems will be needed. *Mike Bancroft* (CLS) then outlined his program in optimization of tribological films, particularly those made

from the ZDDP oil additive. This well funded industrial program is being carried out through a combination of spectroscopy (using the CSRF grasshopper beam line) and spectromicroscopy (using de Staio's PEEM) at SRC. The final presentation of this segment was made by *Tom Ellis* (U. de Montréal) who briefly described the IR spectromicroscopy program and emphasized that many research problems would benefit from the application of a combination of IR, soft X-ray and hard X-ray spectromicroscopy analysis. Procedures to facilitate this were proposed and discussed.

After the coffee break, *Stephen Urquhart* facilitated a discussion about the scientific needs of the CLS spectromicroscopy community and how they could be linked to the specifications for the CLS soft X-ray microscopes. A number of new applications were proposed by workshop attendees. These included:

- mineral surface analysis relating to optimization of chemical and biochemical concentration techniques (*Alan Pratt, Wayne Nesbitt*)
- visualization of amino acids on various substrates (*Katie Mitchell*)
- environmental studies of biological development (metal uptake) (*Ron Martin, Randy Mikula*)
- magnetic imaging to assist development of magnetic nano-structures (*Hong Jiang*)

There was a strong interest by several participants in linking the instrumental and scientific capabilities of the IR, soft x-ray and hard x-ray spectromicroscopes, particularly to make correlative studies easier, but also to enhance and encourage industrial analysis use.

The final part of the discussion, aimed at proposal building, was facilitated by *Adam Hitchcock*. A number of specific choices were discussed with the goal of achieving consensus about key aspects of the conceptual design.

- **source** – the group felt that there was sufficient scientific justification to adopt an EPU. Given the emittance of CLS, it is likely that only half of the length of a straight (~2 m) would give almost as good performance as the full straight section (~4 m), with much reduced cost and provision for installing a second undulator in the same straight at a later time. The latter could be a second EPU arranged so as to provide high frequency switching between left and right circular polarization (as needed for AC magnetization studies). Alternatively, if the two undulators are designed correctly a completely independent undulator design could be implemented.
- **beamline** – the desirability of a single, extended range grating was stressed. However given the desire for a very wide total energy range (100-1900 eV), we should also be prepared to live with a two grating system with one grating covering 100-600 eV and the other covering 500 – 1900 eV. Detailed optical design is required prior to any decision in this area.
- **PEEM** – notwithstanding the expert advice to opt for a fixed installation, the group felt comfortable with adopting the proposed commercial, mobile-PEEM approach. Some of the ancillary capabilities needed for specific experiments were outlined (LEED-Auger,

evaporation, gas dosing, etc in the prep chamber). In general it was felt that a conservative approach to the first PEEM was suitable, especially if the group puts this into operation at the CSRF lines at SRC as soon as possible. A state-of-the-art aberration corrected PEEM could be developed in a second stage.

- **STXM** - designing for high reliability and user-friendliness was stressed. *Michael Feser* warned of the complexities of vacuum and cryo in STXM. He also noted the need for rather extensive (and sometimes expensive) associated capabilities, such as a rapid freeze station for cryo-microcopy of cells (only needed if a biology program is initiated ~20 K), several high quality optical microscopes (~50 K), facilities for sample prep by spin casting (~10K), microtoming (~40 K), etc, and also a simple SEM/TEM ((~50 K). Sample prep capabilities adjacent to the microscope may also be needed for delicate, air sensitive samples.
- **Spectroscopy side station** - this concept received relatively little discussion, although it was noted that some means to characterize the line independent of the STXM will be required even if the side station was not used for PEEM. Some warned of the risk of compromising the dedicated spectromicroscopy aspect if too elaborate alternate capabilities were developed. At the same time it was noted our group will have a responsibility to provide access to the EPU for non-microscopy studies.
- **PRT structure** – the co-applicants were requested to make their views known on this and also to comment as to the suitability of the proposed breakdown of internal beam time allocation (listed on the draft proposal circulated prior to and at the meeting).

The workshop ended at 6:15 pm. Most of the group then adjourned to the Samurai for a very enjoyable Japanese grill table dinner. High enthusiasm for the soft X-ray spectromicroscopy project was expressed by quite a few participants