CLS beamline proposal **SUMMARY FORM** **8 Feb-00**

<table>
<tr><td colspan="3">Title: SOFT X-RAY SPECTROMICROSCOPY</td><td colspan="3">CLS ref.:</td></tr>
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S.G. Urquhart Chemistry (U Saskatchewan)</td></tr>
<tr><td>Source:</td><td colspan="5">insertion device - Elliptically Polarized Undulator (EPU)</td></tr>
<tr><td>Optics:</td><td colspan="5">grating & zone-plate (& electron microscope optics)</td></tr>
<tr><td>Photon range:</td><td colspan="2">Wavelength (Å): 6 – 60 (120)</td><td colspan="3">Energy (eV): 250 (100) to 1900</td></tr>
<tr><td>Photon flux (/100mA/0.1% band pass/sec):</td><td colspan="2">Pre-ZP: 10^{14}
Post-ZP 10^{8} in 50 nm spot
Brightness CRITICAL</td><td>Resolution (E/ΔE):</td><td colspan="2">8000</td></tr>
<tr><td>Capability:
i) STXM
ii) PEEM
iii) other</td><td colspan="5">(i) Imaging, microscopy, XAFS (NEXAFS), micro-spectroscopy (NEXAFS)
Non-UHV – high vacuum, atmospheric (He, air), liquids
(ii) Imaging, microscopy, XAFS (Nexafs, EXAFS), micro-spectroscopy (NEXAFS) photo-emission (total, partial yield, energy analyzed), UHV
(iii) gas phase (for beamline diagnostics ; calibration, possibly gas filtering)</td></tr>
<tr><td>Funds required:</td><td colspan="2">Capital: CDN $5.7 M</td><td colspan="3">Capitalized salary/other: $0.3 M</td></tr>
<tr><td>Sources of funding:</td><td colspan="4">CLS CFI = $2.2 M; CLS – other = $3.3 M {Alberta - $1.0 M, other - $2.05 M); NRCan = 0.25 M}; NSERC-MI = $0.5 M</td><td>Total:
$6.0 M</td></tr>
<tr><td>CLS Comments:</td><td colspan="3"></td><td>Commission date (tentative):</td><td>PEEM station 1-July-2002

STXM & line 1-Dec-2003</td></tr>
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Soft X-ray Spectromicroscopy at the CLS

File: CLS-SM-prop.doc (last changed: 7-Feb-00)

CONTENTS

(Extended (6-9 page) CV's for each member are also provided as a separate document)

I. Executive Summary

1.1 Technical Description for review committee

In collaboration with CLS staff, the beamline team (BT) proposes to design, to assist in construction, and to operate a soft X-ray undulator based beamline dedicated to spectromicroscopy studies of materials and biological systems. Two types of microscopes are proposed – a zone plate based scanning transmission X-ray microscope (STXM) and a photoelectron emission microscope (PEEM). The proposed science includes: soft material studies, mainly polymers (*Hitchcock, Urquhart, Dutcher, Stöver*) and biomaterials (*Hitchcock, Gardella*); environmental and natural resources issues (*Brown, Martin*); tribology (*Bancroft, Kasrai, Norton*); technologically oriented surface science (*Norton, Roy, Guay*) magnetic thin film structures (*Robertson et al*). Innovations in the instrumentation include: an elliptically polarised undulator with full polarisation control; an advanced STXM design (interferometric tracking, high level of automation; planned extension to cryogenic sample temperatures); and a mobile PEEM to provide surface analytical microscopy over a wider photon energy range than will be available at the STXM spectromicroscopy beam line. Development of novel aspects of microscope instrumentation and acquisition/analysis, including advanced detector developments, will be an important aspect of BT activities (*Hitchcock, Tyliszczak*). The research of the BT is estimated to be ~70% academic and ~30% fee-for-service or proprietary.

1.2 Functional description for the general public

Spectromicroscopy is a combination of spectroscopy – the way different wavelengths (colors) of light interact with matter – and microscopy – imaging on a scale finer than the human eye can resolve. There are many variations, including a number which use synchrotron light (**Fig. 1**). The research challenges which our beamline team deal with are ones which can best be addressed using light in the soft X-ray region (wavelengths from 6 nm to 120 nm). In one technique, called scanning transmission X-ray microscopy (STXM), we will focus the light to 50 nm and make images by scanning the sample under the fixed small spot, just like making an image in a television. The focussing element,

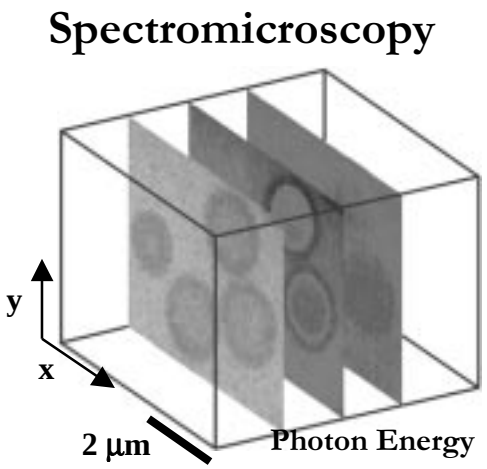


Fig. 1 *Images at three energies of four* ***polymer microspheres*** *(courtesy Stöver, McMaster) recorded with the ALS STXM. The different chemical compositions of the core and shell regions can de determined quantitatively from the changes in the image contrast at different photon energies. Studies of structured polymer spheres are helping optimize paints, adhesives and devices for chemical separation.*

called a zone plate, works with only the coherent (laser-like) part of the synchrotron light beam. For this reason, a third generation light source like CLS is optimal. With STXM we will explore many materials – polymers, cells, plants, soil, minerals, wood, etc – with the goal of understanding the chemical basis for fine scale structure which often controls the properties or determines the function. Improved understanding of the chemical basis of nano- and microstructure is a critical part of developing better materials, medical procedures, environmental problem solving etc. In a second technique, called photoelectron emission microscopy (PEEM), we use the electrons produced by X-ray ionisation, to images with a technique similar to that used in electron microscopes. Here the requirements for the synchrotron light are less demanding so the PEEM will be capable of being moved to any of the CLS beam lines. PEEM detects photo-ejected electrons which can only escape from a very thin surface layer. PEEM will be used to study surface and thin film phenomena such as: protective films formed by oil additives in car engines, magnetic structures such as those used in the computer recording industry, polymers in contact with blood, such as those used in artificial hearts. PEEM and STXM are related and complementary techniques. The mission of the spectromicroscopy group is to build high performance soft X-ray spectromicroscopy instrumentation and to develop a strong national and international user community, with participation by academic, industry and government researchers.

II. Introduction and Background to Soft X-ray Spectromicroscopy

II.1 Overview

Spectromicroscopy refers to a variety of experimental techniques that allow imaging and chemical analysis with good spatial resolution. Examples include analytical electron microscopy (with X-ray fluorescence or electron energy loss detection), fluorescence, IR and Raman microscopy and X-ray excited techniques such as X-ray microprobe, photoelectron microscopy and (scanning) transmission X-ray microscopy. The high intensity and brightness of modern synchrotron radiation sources is greatly improving the capabilities of a range of X-ray spectromicroscopy techniques leading to significant new materials science, biological and environmental applications.

This proposal deals with the development and application of soft x-ray spectromicroscopy at the Canadian Light Source (CLS). This project complements beamline proposals that are being generated for **infrared spectromicroscop**y (Tom Ellis, U.de Montréal) and for hard-ray fluorescence/absorption spectromicroscopy ('**microprobe**', Don Baker, McGill).

The field of soft X-ray spectromicroscopy is relatively new, but expanding rapidly, due to its unique capabilities to address complex problems in materials, environmental, and biological sciences.

The essential characteristics of X-ray spectromicroscopy include:

- High spatial resolution - better than 100 nm with a state-of-the-art of ~30 nm.
- Quantitative chemical analysis on a molecular and not just elemental basis, with quantitation based on high resolution X-ray absorption or photoelectron spectroscopy
- Applicable to complex materials including buried and curved interfaces, wet samples (e.g. biological and environmental) and vacuum and radiation sensitive materials.

By combining spectroscopic chemical information with high spatial resolution, X-ray spectro-microscopy provides new research opportunities. These capabilities are best achieved with the high brightness provided by third generation light sources, particularly undulators in the case of zone plate based instruments. Other synchrotron source types do not provide the necessary tunability, polarisation control and brightness. We are particularly interested in developing a beamline coupled to an advanced insertion device, one which achieves full control of polarisation so the user can chose at will circular, linear or elliptical polarised light of a specific helicity and spatial orientation. To our knowledge this capability is not yet achieved but is considered feasible with suffient flexibility for independent motion of the four quadrants of an undulator (*Padmore, private communication*).

While there are a number of techniques for spatially resolved chemical analysis, they are characterised as either having excellent chemical speciation but inadequate spatial resolution (IR and NMR microscopy), or by having high spatial resolution but inadequate capability for chemical identification (secondary ion microscopy, scanning probe microscopy, Auger, secondary or transmission electron microscopy). Even when they have analytical potential, radiation damage often precludes polymer analysis at high spatial resolution using techniques based on electron or ion impact.

Recently **scanning transmission X-ray microscopy (STXM) using synchrotron radiation, has been shown to be a powerful tool for analysing the chemical basis of polymer microstructure**. STXM spatial resolution is as good as 30 nm [AZ&92, AH93, AS&95]; it has an exquisite ability to distinguish very similar chemical species based on details of inner-shell X-ray absorption spectra (NEXAFS) [S92]; it can deduce the orientation of specific chemical bonds [SA96]; and it has much lower radiation damage than competing techniques [RH&97]. In principle, electron energy loss in a transmission electron microscope (TEM-EELS) has better spatial resolution but in practice, radiation damage reduces the resolution at which meaningful information can be recorded. It has recently been shown that x-ray spectromicroscopy has an advantage of about three orders of magnitude over the equivalent electron microscopy techniques in being able to record spectra and images from small sample areas of radiation sensitive polymers [RH&97].

Currently there are only two centres for soft x-ray STXM microscopy in the world - that at the National Synchrotron Light Source (NSLS, Brookhaven, NY) developed by Kirz, Jacobsen and co-workers [JW&91], and that at the Advanced Light Source (ALS, Berkeley, CA) developed by the spectromicroscopy group of Tonner, Warwick and co-workers [WA&98]. There are three working STXM microscopes at NSLS (beamline X1A) and one at the ALS (beamline 7.0.1), with a second beamline and microscope currently under construction (beamline 5.3.2). The PI, Hitchcock, is a 22% partner in the 5.3.2 participating research team, an experience that is providing invaluable training for this project. The unique capabilities of STXM have lead to a tremendous increase in demand on the existing x-ray microscopes for biological, polymer and environmental studies. At present, the demand exceeds supply by at least a factor of 3. Clearly there is an excellent case for a developing a new centre for STXM spectromicroscopy at the CLS. Other soft x-ray STXM projects, currently under way in Korea (Pohang Light Source) and at Bessy, Berlin, are not yet functioning to our knowledge.

X-PEEM, photoemission electron microscopy using synchrotron x-rays, has experienced a similar explosion in application and utility for spatially resolved chemical analysis of surfaces and thin films. While there are quite a few lab-based PEEMs which use work function or shadowing contrast, there are only a handful of synchrotron X-PEEMs, even though the variable photon energy NEXAFS image contrast and microspectroscopy has tremendous chemical analytical capability. In parallel with the STXM, we intend to implement an X-PEEM at CLS.

II.2 Primer on STXM and PEEM

Since these techniques may be unfamiliar to some reading this document, this section is a brief description of each instrument with a few examples of their capabilities. For those interested in a more detailed exposition, please consult Vol 84 of J. Electron Spectroscoscopy [A97], a special issue dedicated to soft X-ray spectromicroscopy.

II.2.1 Scanning Transmission X-ray Microscope (STXM)

Scanning Transmission X-ray Microscopy (STXM) is a form of chemical imaging that combines 30-50 nm spatial resolution with Near Edge X-ray Absorption spectroscopy (NEXAFS). Figure 2 presents a schematic of a STXM microscope beamline, while the details of the zone plate focussing optics are shown as **Figure 3.**

In a STXM microscope, monochromatic X-rays are focussed by a Fresnel zone plate, which is a circular, variable line density, transmission diffraction grating. A central stop in the zone plate, in conjunction with a slightly smaller order sorting aperture (OSA), is used to isolate the positive first order diffraction and to suppress unwanted diffraction orders. In most instruments the sample is mechanically raster scanned in the focal plane of the spot.

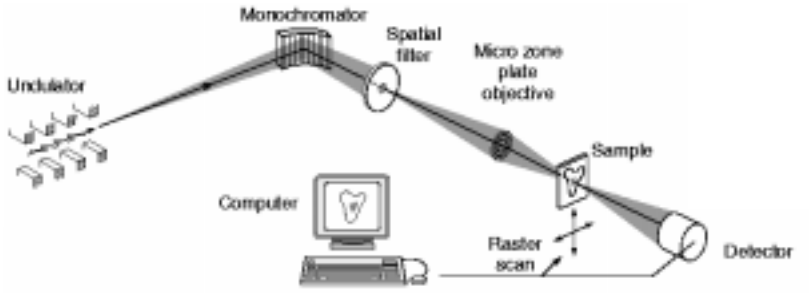


Fig. 2 Schematic of a scanning transmission X-ray microscope (STXM) beamline (CXRO - LBNL).

When a STXM microscope operates in imaging mode, the sample is raster-scanned across the focus point of the zone plate while the photon flux that passes through the sample is measured. In spectroscopic mode, the focused beam can be left on the same spot while the photon energy is scanned. Absorption spectra (optical density, OD) are then derived from the transmitted X-ray intensity as $(-\ln(I/I_0))$, where an energy scan from the sample (I) is normalised to another energy scan recorded without a sample (I_0). Quantitative analysis is provided by the Beer's law dependence of the absorbance:

$$A = OD = \mu\rho t = -\ln(I/I_0)$$

where μ is the energy dependent mass absorption coefficient, ρ is the density, and t is the sample thickness. Since x-ray absorption images contain the same spectroscopic information but at one energy, image processing can be used for quantitative chemical image analysis[SA&98]. An

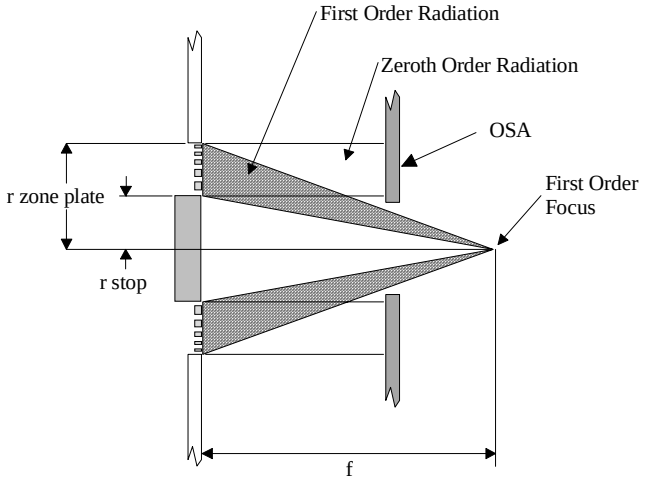


Fig. 3. Schematic of zone plate (ZP) and two of its diffraction orders[A98]. All orders other then the positive first order are stopped by the Order Selecting Aperture (OSA) placed in the shadow of a central ZP stop, and the first order focal spot (f) is used as the microprobe for our instrument. The focal length f is 0.5 - 5 mm depending on the ZP and photon energy.

example of STXM analysis by imaging and spectroscopy is shown in **Figure 4**, which shows how various components of a complex polymer can be distinguished using images at different photon energies in the C 1s, N 1s and O 1s regions.

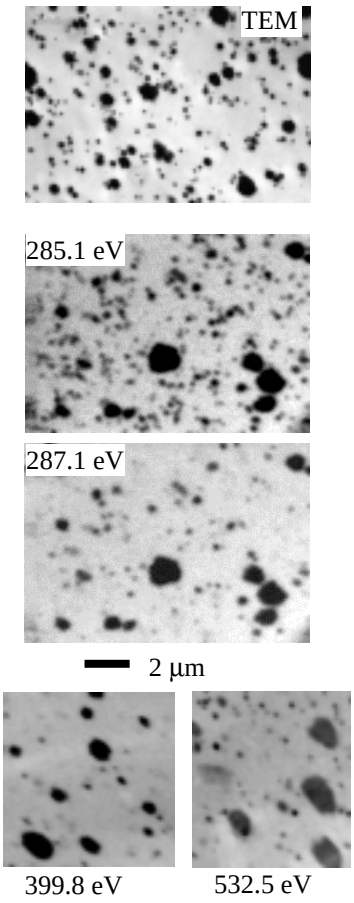


Fig. 4 Comparison of TEM, C 1s, N 1s and O 1s STXM images of a polyurethane with two different filler particles types. Two C 1s images of the same area readily distinguish the particle types [HK&00].

Spatial Resolution

The diffraction limited spot size of the zone plate determines the highest attainable spatial resolution of the microscope. This limit is 30-50 nm with the highest quality zone plates currently available [JKW92]. Recent micro-fabrication efforts have pushed the diffraction limited resolution to at least 20 nm [SR&96, SJT97]. It is expected to reach 10 nm over the next decade.

The instrumental spatial resolution of NEXAFS microscopy is much lower than that obtained with transmission electron microscopy (TEM), which is ~0.2 nm with the best commercial instruments. However, in practice, the need for a high dose to acquire useful core excitation spectra by electron energy loss spectroscopy (EELS), combined with the low critical dose for radiation damage of most polymers, means that the effective spatial resolution that can be achieved for *analytical* measurements is often comparable [RH&97]. While radiation damage rates are considerably lower in soft X-ray spectroscopy than in TEM-EELS [RH&97], radiation damage is still of some concern, particularly when high quality spectra are acquired from small sample areas or the sample is particularly radiation sensitive.

Photon characteristics of existing STXM microscopes

The Stony Brook STXM at the National Synchrotron Light Source (NSLS)[JW&91] and the BL7.0.1 STXM at the ALS[WA&97, WA&98] use undulators as the X-ray source. Undulators are several orders of magnitude brighter than bending magnet sources and about eight to ten orders of magnitude brighter than non-tunable laboratory X-ray tubes. Spectra are acquired with resolving powers of 2000-9000, corresponding to an energy resolution in the C 1s region of better than 0.1 eV. In principle, all elements with inner shell thresholds in the 150-1200 eV energy range can be accessed with NEXAFS microscopy, although most work to date has used the carbon 1s edge. At

this core edge, energy calibration is provided *in situ* by leaking CO_2 into the microscope atmosphere while the sample is in place [RH&97]. There are also higher energy zone-plate based STXM projects at ESRF (*Susini*), APS (*McNulty*) and Spring-8.

Sample Environment:

In current STXM microscopes, the sample is in air or He at atmospheric pressure and at room temperature. If the sample is enclosed between two X-ray transparent silicon nitride windows, it is possible to investigate wet samples, such as fully hydrated superabsorbent polymers, biological samples such as cells, and aqueous suspensions of environmental samples such as soil particles. A STXM capable of examining samples at cryogenic temperatures has recently been implemented at NSLS [MO&00]. Cooling the sample is known to reduce the rate of radiation damage in TEM of polymers [DA&88]. A similar beneficial effect is expected when cryo-STXM techniques are applied to polymers. The ability to heat and cool a sample while making measurements is not currently implemented but isa goal of this project.

Typically, sections for C 1s studies are ~100 nm thick, with somewhat larger thickness (up to 1 μm) being appropriate at higher energy edges or cases where the element of interest is a minority species. In polymers, samples thinner than 80 nm often suffer rapid beam damage and low signal, whereas samples thicker than 200 nm can be distorted by absorption saturation leading to distortions from higher order photon contamination and detector dark noise. For microscopy of wet samples, the hydrated sample is kept suspended between two thin membranes, typically 100 nm thick Si_3N_4. Otherwise, the samples used in STXM are very similar to those in TEM. The thin sections can be prepared by by ion thinning, spin casting, or ultramicrotomy, and are typically mounted on 3 mm TEM grids.

II.2.2 PEEM

Photoelectron emission microscopy (PEEM) uses electron optics similar to that found in a scanning electron microscope to image the electron distribution emitted by photoionization of a region (typically 10-100 μm) illuminated by monochromatic light.

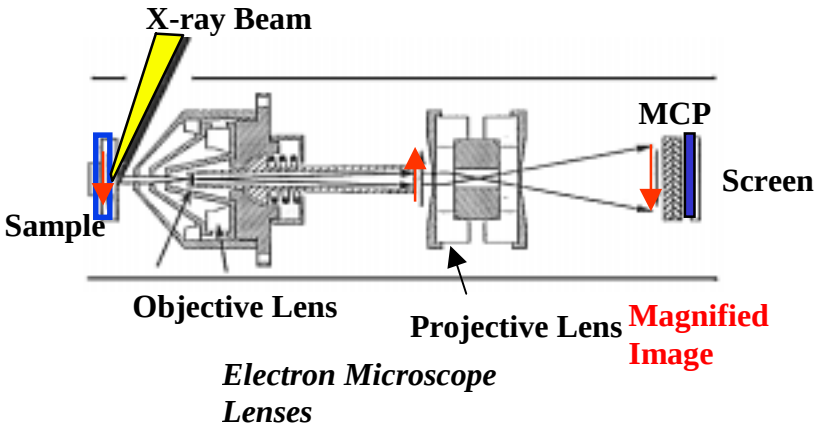


Fig. 5 Schematic of the ALS PEEM (BL 7.3.1, ALS).

Figure 5 is a schematic of a synchrotron based PEEM. PEEM was initially developed using low energy light sources such as a Hg arc lamp and relied mainly on work function variations as the source of contrast [BM&89, VR&91]. Commercial devices, primarily aimed at the non-synchrotron market, are sold by a number of vendors (Staib, Omicron, Specs). When the light used is tuned X-rays from a synchrotron, the power of X-ray absorption as an analytical tool is introduced. The PEEM x-ray spectromicroscopy method combines x-ray absorption spectroscopy and electron microscopy. The secondary photoelectron intensity (yield) from the sample is used as the signal in the imaging process. Since there is a close relationship (generally linear) between photoelectron yield and the x-ray absorption coefficient, the measured electron yield signal provides not only image contrast but spectroscopic information. Information is obtained by tuning the x-ray energy to a particular absorption edge and recording an image ("microscopy" emphasis), by selecting an area in the image and measuring its intensity as a function of photon energy ("spectroscopy" emphasis), or by recording complete image "stacks" as a function of photon energy (spectro-microscopy). The

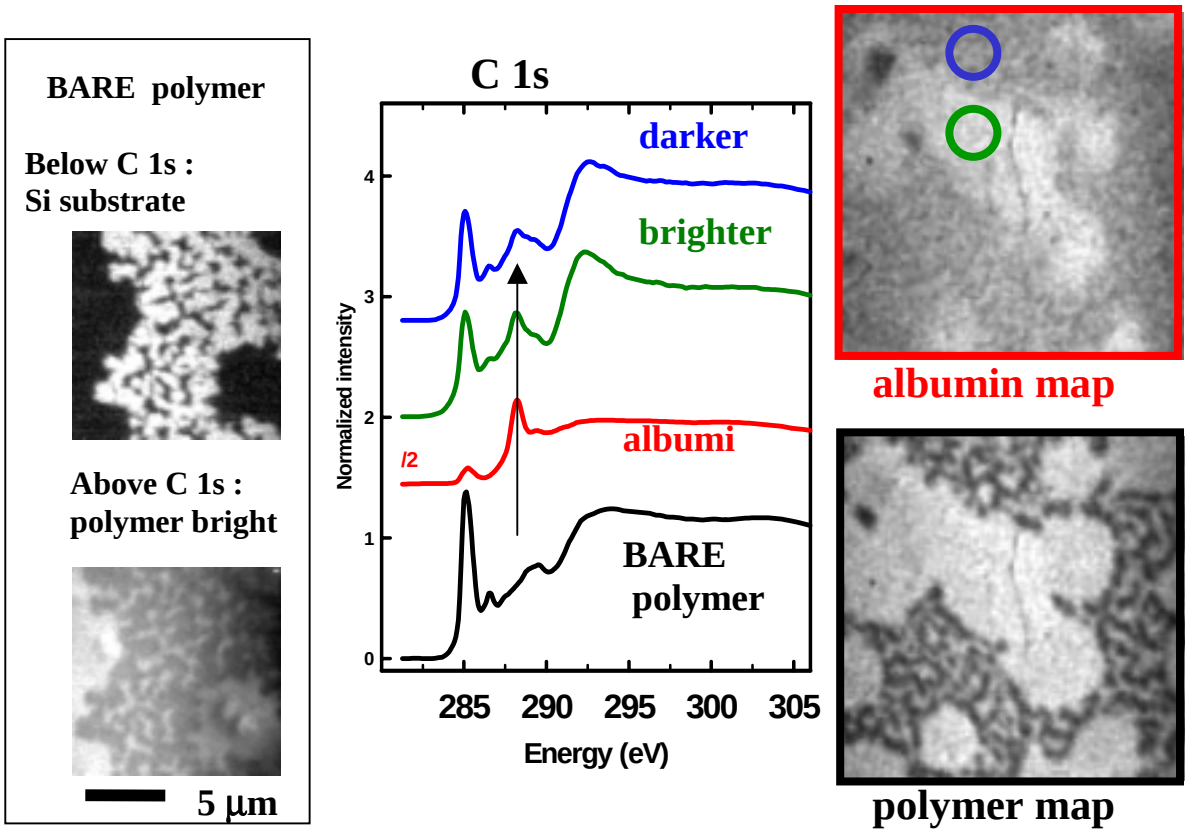


Figure 6 (left) images below (280 eV) and above (300 eV) the C 1s edge. The regions of the Si wafer with low polymer coverage are bright on account of the large photoemission of the underlying Si substrate. Above the C 1s edge the contrast is reduced due to stronger polymer absorption. (centre) spectra of bare and two regions of the protein covered polymer, compared to that of pure human serum albumin (recorded with STXM). (right) example of chemical analysis performed by fitting spectra at each pixel in an image sequence ('stack') from 280-340 eV, to a linear combination of the polymer and protein C 1s NEXAFS spectra. (Work performed by S. Anders, A. Scholl, F. Nolting and A. Hitchcock, ALS BL 7.3.2, July 1999)

method provides several different contrast mechanisms: elemental specificity is gained from tuning the x-ray energy to characteristic absorption edges; chemical specificity as well as electronic and structural information is obtained by tuning to specific features in the absorption edge fine structure; bond and charge anisotropies are determined by means of the polarisation dependence (conventional linear dichroism) of near edge absorption resonance; the orientation and size of magnetic moments are probed through magnetic linear and circular dichroism effects of near edge resonances; and finally topographical contrast is obtained by the distortion of the extraction field at surface topographical features.

High spatial resolution with each contrast mechanism is available by imaging the emitted photo-electrons by means of an electron microscope, typically using an all-electrostatic column. Because of the high extraction fields used in PEEM the technique emphasizes very low energy secondary electrons and thus has a sampling depth of 2-10 nm, much larger than other photo-emission techniques. This is usually an advantage as PEEM is able to probe near surface regions of samples covered by thin protective and/or conducting layers. An example of the results of a recent PEEM experiment carried out at the ALS PEEM 2 is presented in **Figure 6**. In this investigation of protein adsorption on a polymer, it was possible to image the polymer as deposited on a Si wafer both before and after protein adsorption. This is likely a favourable case where the residual conductivity of the aromatic polymer was sufficient to avoid charging artefacts. However similar experiments have been carried out on quite insulating polymer systems by first depositing a very thin (<0.5 nm) metal layer.

III Scientific Program

Some of the scientific problems to which this beam line and end stations will be applied are described in the sections below, which outline the research interests of the co-applicants, grouped by themes.

III.1 Polymers and Soft Matter

(a) Optimising Polymer Microstructure (*Dutcher* (Guelph); *Hitchcock, Stover, Tyliszczak,* McMaster) The development of polymers optimised fo specific tasks (automotive components, packaging, medical devices, adhesives, drug delivery, etc) is limited by inadequate understanding of the relationships among microstructure, polymer properties, and the chemical recipe. Many polymers have a complex microstructure with different chemical composition at the surface, various bulk phases, and at phase boundaries. The development of materials optimised for specific applications is often carried out on a semi-empirical basis, without an in-depth knowledge of the chemical microstructure and how it relates both to the method of preparation and the resulting properties. The microstructure may be controlled using additives, compatibilizers, elastomeric particles, fillers, etc. Additionally, the morphology or composition may differ between the surface and core of a sample. For example the shear of injection molding can alter morphology at a surface, or the thermal history of the interior could differ from that at the surface of a molded part. An ability to characterise locations of polymer components and their interactions is key to more rational design of polymeric materials. These problems incorporate a fascinating combination of **applied** aspects as well as **fundamental** polymer chemistry and physics. Among the latter are questions such as - what drives phase segregation? What determines the resistance of a fundamentally metastable material to transformation to a more thermodynamically stable form? The research that will performed in this area involves a number of members of the CLS spectromicroscopy team - *Dutcher, Hitchcock, Stöver, Urquhart* - as well as a number of

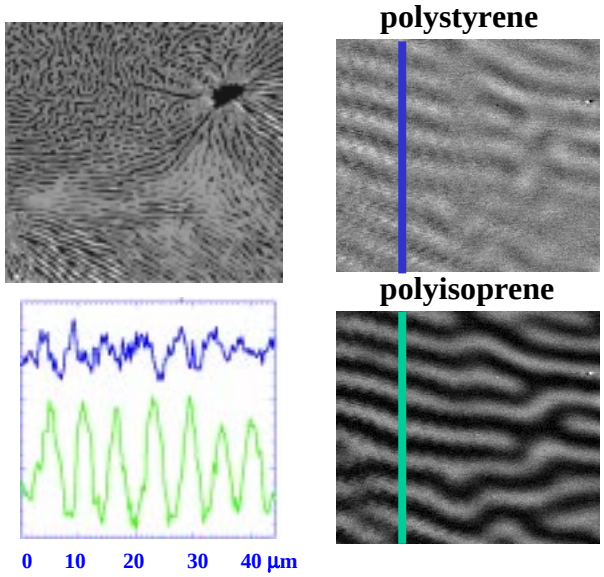


Figure 7 STXM image of PS/PI/PS film annealed near T_g of PS. Composition maps derived from images at 5 C 1s energies. Intensity profiles at indicated locations. (ALS, July 1999) [KH&00]

collaborators who are not co-applicants on this proposal, but who are participating in the current STXM/PEEM research program of Hitchcock at the ALS (*Mitchell, Rightor, Childs*). This type of study will also be of interest to a number of groups active in polymer microstructure studies (*Kumacheva*, Toronto) and various industries (Dow, 3M, Dupont, BASF, etc)

As an example of the proposed research in this area, **Figure 7** presents results of a collaborative study by the *Hitchcock* and *Dutcher* groups of self-organisation of a polymer tri-layer structure (PS/PI/PS where PS = polystyrene and PI = polyisoprene) [KH&00]. Analysis of the C 1s NEXAFS indicates that the observed patterning is not only in the PI layer (as expected from earlier studies of the annealing of confined free-standing films) but also in the polystyrene layer (unexpected since the annealing temperature was below the glass transition temperature of PS). It is expected that other collaborative projects dealing with issues in micro- and nano-structure of polymers which are currently underway in: polyurethanes (Dow); polymer microspheres and encapsulation structures (*Stöver*, 3M); and water purification membranes (*Childs*, 3M) will migrate from ALS to CLS in whatever form they are at the time the CLS STXM becomes operational.

(b) Phase Segregation in polymer blends (*Urquhart*, Univ. Saskatchewan)

Polymer blends and composites are widely used in industrial products, where different polymers are blended to combine the positive attributes of each component polymer (i.e. impact strength, rigidity, chemical resistance, cost, etc). Development of stable and controlled phase morphology is critical for the commercial application of polymer blends and therefore the thermodynamics of phase segregation is of both fundamental and applied interest [R94]. Soft x-ray microscopy can play a critical role in developing basic understandings of phase segregation in polymer blends. Such phenomena can be difficult to study since composition measurements of sub-micron domains are difficult to obtain by conventional methods. Microscopy methods (e.g. electron, confocal, optical) lack direct chemical sensitivity, chemically sensitive spectromicroscopy methods (e.g. FTIR and Raman microscopy) lack necessary spatial resolution, and scattering methods (e.g. SAXS, SANS) have a decreased sensitivity to structures larger than 50 nm. While correlative approaches combining spectroscopy, scattering and microscopy have provided important insights to these questions, direct “real space” chemical microscopy measurements by soft x-ray microscopy will add a new dimension to basic and applied polymer studies.

Kinetics and Models for Phase Segregation in Polymer Blends:

Nanostructure formation in polymer blends by phase segregation is a complex process of phase separation (by spinodal decomposition in **fig. 8a**) followed by phase coarsening (**fig 8d**). My intent is to explore the transition from early state to late stage formation of polymer nanostructure. Theoretical models [MB94] are based on a strict separation of early and late stage kinetics, although electron and optical microscopy [R94,CN95,PS&97] provide compelling indications that this separation is invalid. As soft x-ray microscopy can uniquely measure both the composition and morphology of dispersed polymer blend phases, a more illuminating investigation of the phase segregation kinetics in such polymer blends is possible. Are early and late stage phase segregation mechanisms in fact separable, or is a more complex description (and kinetic model) of the cross over between these processes required?

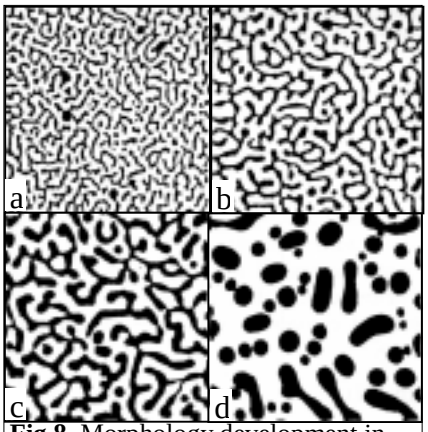


Fig 8. Morphology development in polymer blends following spinodal decomposition (a) & coarsening (d)[R94].

Fundamental understanding of the kinetics and mechanisms of phase segregation and phase stabilisation are necessary for the formation of optimised and controlled polymer nanostructure in commercial polymer blends. The CLS soft x-ray microscope, in concert with atomic force and optical microscopy will be used to evaluate these questions.

Phase Structure of Polymer Blends: **Polyolefin Blends:** While it is broadly accepted that different forms of polyethylene are compatible (e.g. HDPE, LDPE, etc), indirect measurements (principally rheology) suggested that phase separation does occur (*M.C. Williams, private communication*). Unfortunately, most common methods (i.e. electron and optical microscopy) lack suitable contrast for similar polymer materials (e.g. HDPE, LLDPE, etc). The ability to characterise the phase properties of this class of commodity polymers can have a significant impact (e.g. annual production of PE by Nova Chemical at Joffre, Alberta: 545 kilotonnes). Small differences observed in the NEXAFS spectra of different forms of PE (*Ade, Urquhart, unpublished data*) indicate that soft x-ray microscopy can resolve the potential phase structure in blends of polyethylene and related polymers. *Are the different types of polyethylene in fact compatible? If not, what effect does this have on the physical properties of the polymers?* **Crystalline Blends**: Methods to characterise the morphology and phase structure of semi-crystalline polymer blends are complicated by the difficulty of measuring the composition of nano-scale crystalline domains (10 – 1000 nm). By combining linear dichroism spectral imaging with orientation-sensitive “singular value decomposition”

chemical mapping (a variant on that used by Ade *et al.* [AW&98]), a new method to characterise the crystalline components in polymer blends is proposed. While there are a number of significant experimental challenges to this method, this ability would open a new domain for the materials science of crystalline polymers.

Cryo-microscopy – control of radiation damage in polymer microscopy: These polymer investigations only provide meaningful results if carried out below the threshold for significant radiation damage. Cryogenic cooling to reduce the temperature below that for mobility of radicals permits longer exposure times. Cryo cooling also eliminates “mass loss” or ablation which is significant for many organic materials. However, it does not alter the probability of direct changes in chemical bonding and thus changes in spectral signatures. Cryo-cooling has been implemented by Jacobsen and Kirz (SUNY-SB) for biological STXM microscopy [MO&00]. This has enabled long exposure microscopy studies of cells such as a recent 36 hour x-ray microtomography measurement [F99] We intend to implement cryo capability at the CLS STXM as it brings important advantages for both polymer and biological imaging.

The studies described in this section will be initiated using the modest general user time available at the NSLS and the ALS, but will accelerate and expand upon access to the new facilities and new modes of operation (e.g. cryo) at the CLS.

(c) Self-Organisation of Free-Standing Confined Thin Films (*Dutcher*, Guelph)

There is much interest in understanding the fundamental processes of self-assembly on the nanometer scale. When free-standing, confined homo-polymer films are annealed close to or above their glass transition temperature (T_g) highly organized patterns are formed. The driving force for this self-assembly is postulated to be long range Van der Waals or dispersion forces which lower the total energy of the system by reducing the distance between two, mutually attractive, confining layers [DND99]. Quantitative models have been proposed.

Our interest is to test and thereby refine such models by measuring in-plane chemical maps of the freely-standing trilayer films to determine the amount of the central layer which remains in the "pinched-off" regions produced in the morphology which is driven by the attractive van der Waals or dispersion forces which act across the thickness of the trilayer film.

STXM is ideal for this project because the films are reasonably thin, without a supporting substrate so that transmission is straightforward; the thickness variations are large enough to be measurable in the STXM experiment; and the in-plane periodicity is typically several microns which is large compared with the in-plane resolution for chemical mapping information. Interesting studies

include variations of the annealing history of the samples, as we have started to do, variations of the central polymer to vary the miscibility with the capping layers, and also the possibility of heating in-situ in the STXM experiment to track the onset of the instability (the dynamics of this process can be easily adjusted by adjusting the temperature).

(d) Polymer Surface Chemistry (*Gardella,*SUNY-Buffalo)

Ongoing polymer surface chemistry research in the Gardella group at SUNY Buffalois focused into three research areas. The first involves the development of new quantitative measurements of surface structure, composition and reactivity, and currently involves electron (XPS, HREELS), vibrational (FTIR, HREELS) and mass (ToF-SIMS and low energy ion scattering) spectrometries. The second area of research involves the synthesis and characterization of multicomponent polymers (block and graft copolymers, blends) with unique surface structure and composition. The third area of research involves the development of new materials for biomedical and environmental uses, again focusing on polymer structures and surface structure property relationships.

The development and availability of the CLS PEEM/STXM facilities in Saskatchewan will be a major complement to the polymer research capabilities in Buffalo, because the major issues which can be addressed by both methods overcome many aspects of work using only the existing surface science methodology in Buffalo. The development of these measurements with thin film and organic/polymeric model system structures is an obvious extension of our more basic work in measurement science. We would propose studying the orientation of tertiary polymer structures with PEEM by using helical monolayer structures of polymers prepared as Langmuir Blodgett films. Recent work using double helical crystalline isotactic poly(methyl methacrylate) (PMMA) has shown that ion formation mechanisms in SIMS are sensitive to the tertiary structure of the double helix. NEXAFS spectra of the oriented PMMA [NG&99,NG&96] could be sensitive to the different orientations of the backbone and sidechain carbons (double bonded Carbonyls are in the sidechains), and this could allow the differentiation the tertiary structures from single helical PMMA or amorphous PMMA.

More applied work with both PEEM and STXM measurements will be carried out to study phase separation and reorganization of surface structures under environmental challenge. Three polymer systems are currently under study in Gardella's group. Much work on the structure of siloxane and fluorocarbon based copolymers and blends has been accomplished over the past 16 years [G85-00], with efforts focused on determination of the surface composition and in depth

profile of siloxane copolymers and blends. Two recent developments include the development of algorithms to simulate in-depth profiles of surface excess layers in siloxane and fluorocarbon segregated copolymers [ZR&99,ZG&96,CG&95] and the development of low temperature sample handling probes for the study of polymers exposed to water. In the latter approach, we can freeze the polymer after water exposure to below the glass transition temperature,T_g, of PDMS (ca. –120^{o}C) and examine the reorganization of the surface domains of phase separated and surface segregated PDMS copolymers. We have published XPS, SIMS, TEM (cryo-ultramicrotome cross sections) and FTIR studies which relate chain length and dispersity to the surface structure and thickness of the segregated PDMS layer. Systems such as PDMS-PS, PDMS-polyureaurethanes, PDMS-nylons, PDMS-BPAC, PDMS-polyimides, PDMS-PHEMA, etc. have all been synthesized and examined. Effects of solvents casting and preparation, crystallinity, annealing, polymer structural parameters (composition, chain length, etc.) and other phenomena have been systematically examined in siloxane polymers. More recently, we have synthesized new materials which are amphiphilic copolymers of poly(hydroxyethyl methacrylate) (poly HEMA) a hydrogel widely used in soft contact lenses, and PDMS siloxanes. Further, two other systems under study are newly synthesized materials of polyperfluoroether-polycaprolactone and PDMS-caprolactone. In air, in all three systems, there is a surface segregated layer of the lower surface energy component (fluorocarbon or PDMS). However, we are interested in the role of reorganization of the surface upon exposure to water. The polycaprolactone polyester is semicrystalline and provides an interesting hard segment to study upon exposure to water. We intend to study the role of siloxane structure on its reorganization after wetting/absorption of water into the hydrogel portion of the polymer.

All of these studies would be facilitated by the PEEM and STXM. The capabilities of the STXM to visualize domain structures in common environments could be used to correlate our compositional studies from XPS and ToF-SIMS. Imaging domain structures in two dimensions by PEEM is very important to understand how partially crystalline or hydrophobic structures are resegregated near the surface, and the role of the low surface energy component and surface phase dimensions on the mediation of the reorganization.

All of these basic polymer surface structure studies are directly related to our interests in designing materials for biomedical and environmental use [GW&97,GH96,CG99]. One area of interest is the development of new biodegradable polymers for tissue engineering and drug delivery. We have developed means to estimate the reaction kinetics of the surface regions of such polymer

by ToF-SIMS. Following the degradation of partially crystalline copolymers with phase segregation should be an important application of PEEM, and possibly of STXM.

III.2 Biomaterials, Biology and their interfaces

(a) Optimization of blood contact polymers (*Hitchcock, Brash*)

Block copolymers, blends of homopolymers, and combinations of the two have considerable potential as biomaterials for medical applications, e.g. in the construction of vascular grafts and other blood contacting devices. In this area the creation of a biocompatible tissue-material interface is the greatest challenge. Key problems here and in the many other situations where blood is in contact with artificial surfaces (heart-lung bypass for open heart surgery, artificial heart valves, heart assist devices, intravascular stents, involving in total thousands of patients daily world-wide) is activation of blood coagulation, thrombosis, and the immune system. These effects are known to be initiated by interactions of blood proteins with the material (often a polymer) surface. The goal of research in this area is to develop surfaces that prevent or minimise these phenomena.

This project is a collaboration between the groups of *Adam Hitchcock* (Chemistry, McMaster) and *John Brash* (Chemical Engineering, McMaster). It has been suggested that the microphase or nanophase separation characteristics of block copolymers, projected onto the tissue material interface, may influence the interactions at this interface, which in turn determine **biocompatibility** [CK&93]. For example, protein adsorption, which strongly influences biocompatibility via its effects on cell adhesion, may be fundamentally different at an interface containing the distinct domains of two or more microphases as opposed to a homogeneous interface. Questions of interest include:

1. Do proteins distribute differentially between (among) the domains, eg in a complex protein "mixture" like blood do the different proteins adsorb preferentially to one or other of the two domains?
2. Do proteins adopt different conformations or orientations on the different phase domains?
3. What are the differences in chemical properties of domains that support such differentiation? An example might be anticipated to be hard versus various soft domains (polyether, polyester) in segmented polyurethanes.
4. How is subsequent cell adhesion affected by the spatial distribution of protein types on micro- or nano-phase domains, eg one domain type adsorbing an adhesion promoting protein and the other a cell resistant protein.

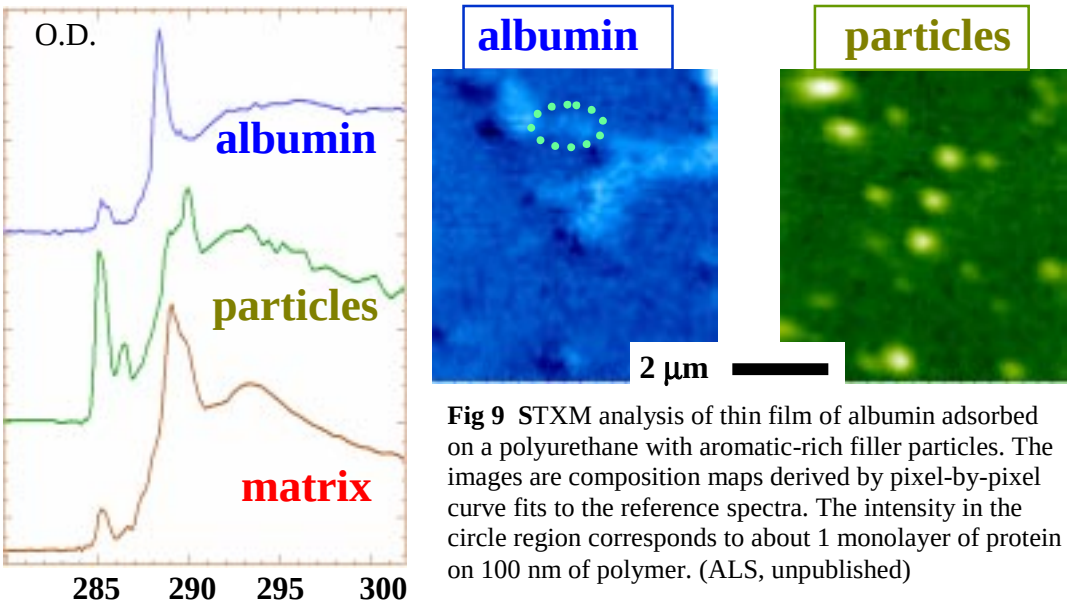


Fig 9 STXM analysis of thin film of albumin adsorbed on a polyurethane with aromatic-rich filler particles. The images are composition maps derived by pixel-by-pixel curve fits to the reference spectra. The intensity in the circle region corresponds to about 1 monolayer of protein on 100 nm of polymer. (ALS, unpublished)

Although there is considerable knowledge of protein interactions with chemically homogeneous surfaces [CK&93], and although there have been studies of the "global" interactions of proteins with some block copolymer systems (e.g. polyurethanes) there is essentially no information on interactions at the phase domain level.

The objectives of this work would be to provide answers to the types of questions listed above. The information generated is anticipated to lead to new approaches in the design of biocompatible interfaces. For example, the Brash group is developing novel polyurethane-based polymers for blood contact applications [SB91, SB97]. One strategy is to graft moieties to the polymer chains which can act as links between the polymer and specific proteins in blood which lead to desirable biological outcomes. For example if plasminogen is selectively bound to the surface (using lysine as a bridging ligand), then it will potentially have clot-dissolving properties [WWB96] since plasminogen is the zymogen precursor of plasmin, the primary enzyme in the fibrinolytic pathway. One question which arises is whether these ligands should be attached to the hard or soft segment of the polyurethanes since it is likely that one of these segment types will dominate the surface or the interface with the biological milieu. This is an excellent example where there is pressing need for more sophisticated methods of characterisation of polymer phase segregation.

The average surface properties of these materials are routinely characterized by both contact angle and X-ray photoelectron measurements. However, the surface morphology on a submicron spatial scale is unknown. We will use PEEM and/or electron yield detection in the STXM (1) to characterize the surface chemical morphology of the virgin surface of biomaterials (this may vary with solvent contact and thus comparison of the dry and wetted material would be of considerable

interest); (2) correlate this surface morphology with spatial patterns of protein and cell attachment by imaging a similar surface after exposure to an appropriate biological medium such as blood plasma. **Fig. 9** shows recent results from studies at the ALS which demonstrate that, even with transmission mode, STXM is capable of detecting proteins on polymer surfaces at the monolayer level. **Fig. 6** illustrated a parallel study by PEEM.

(b) Other applications in biology

While biological or medical research is not a major focus of any of the current beamline team members, the extensive work by TXM (Schmäl et al, Larabel, Meyer-Ilse) and STXM (Kirz, Jacobsen and collaborators) clearly shows tremendous applications of X-ray microscopy in this area. A CLS bio-imaging user community will develop as a matter of course, and use of the STXM will be a significant component. **Fig. 10** gives a recent example of the capability of STXM in the strictly biological arena. This figure shows 2 STXM images of a pancreatic tissue sample in the N 1s energy regime, whereby the sensitivity to different nitrogenous components is dramatically enhanced and DNA/protein specificity introduced by ratio-ing of images at selected N 1s near edge features to the signal before the N 1s onset. Spectra from image sequences are being used for more detailed

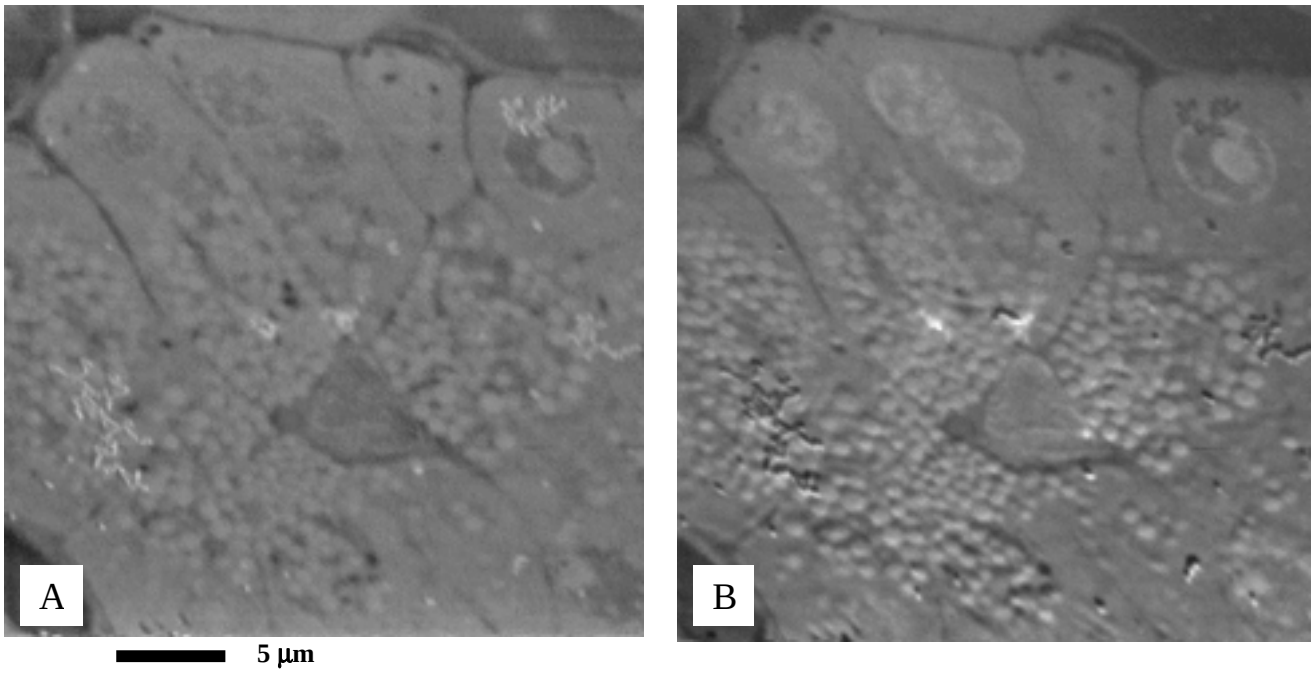


Fig. 10. STXM images at (a) 400 eV and (b) 413 eV, ratio-ed to 392 eV image of the same region to enhance contrast of nitrogen containing microstructures. The sample is rat pancreatic tissue in quiescent state (pre-stimulation of enyme release) fixed in glycomethacrylate [LS&00]. The submicron circles are the zymogen granules, which are protein rich digestive enyme factories. They cluster around an acinar cell which guides the released enzymes to the gut. Details of cell nuclei and cytoplasm structures can be identified. [*Loo, Hitchcock, unpublished, run 6-Feb, 2000 on BL 7.0 STXM with ALS operating in 2-bunch mode (10% of normal intensity)*]

identification of subcellular and intracellular components. The goal of this work is to better understand the mechanisms of release of digestive enymes from zymogen granules in the pancreas by visualizing and identifying the microstructure pre- and post-stimulation of enzyme release. Malfunction of these mechanisms may be involved in cystic fibrosis. The STXM study was carried out to complement full field transmisison X-ray microscopy studies on the identical sample [LS&00].

III.3 Environmental and Natural Resource Applications

(a) **Natural Resources Canada** (*Brown*, NRCan SL steering committee, CANMET)

Significant synchrotron radiation and spectromicroscopy applications are envisaged to several core (long-term) materials, minerals and energy related programs in CANMET, as well as in analytical services operations of NRCan. Over the past 6 months over 30 senior scientists in the Energy, Earth Science and Minerals & Mining Sectors have become familiar with the capabilities of synchrotron light research and many are initiating access via existing users. The Canadian Forestry Service (the fourth NRCan Sector) will also use CLS but their staff are only now becoming knowledgeable about synchrotron research. The forestry group may be the major user of spectromicroscopy techniques (IR, STXM and PEEM). Other important NRCan project areas include: micro-structure and nano-chemical speciation of (wet) HV battery electrodes and co-polymers (fuel cell and battery membranes), thin multi-metal films and clusters, (wet) bio-materials (forestry), solid fossil fuels (bitumen, cokes, coal, etc). These topics are of continuing interest here and to our stakeholders and clients. NRCan will provide $2 M of beamline capital investment to CLS. The proportion to be provided to the soft X-ray spectromicroscopy program is under negotiation.

(b) Micro-chemical Analysis of plant material (*Martin*, UWO)

Synchrotron radiation is valuable in the analysis of environmental aspects of plant material, especially small samples. To date the Martin group has concentrated on micro X-ray fluorescence analysis of metals in the annual growth rings of trees. Recently this work has been extended to the metal distribution in hyper-accumulating species used in bioremediation. It is planned to extend the work to the EXAFS, XANES, and STXM spectromicroscopy of plant thin sections. This research would benefit immeasurably if it could be extended to low atomic weight atoms such as C and O. In addition small area analysis would allow studies of regions of physiological interest.

III-4 Nanostructure optimization in other systems

(a) Nanocrystalline and nanocomposite electrocatalysts (*Guay*, INRS)

In recent years, there has been considerable interest in investigating electrochemical phenomena using chemical structures that are defined and measured on a nanometer length scale. This is particularly so in the field of electrocatalysis. At INRS-Énergie et Matériaux, nanocrystalline and nanocomposite materials are commonly studied for various applications, including the electrocatalysis for hydrogen evolution, the development of CO-tolerant hydrogen oxidation electrocatalysts and the preparation of new hydrogen absorbing materials.

Most of the materials we are interested in are defined on the nanometer length scale and are made of at least two different phases. The problem of defining the distribution of these phases has never been addressed. In principle, this could be done by transmission electron microscopy, but the requirement that the sample should be thin enough has severely retarded our efforts. It would thus be highly desirable to carry out **PEEM** studies on electrocatalysis samples. We have interest in a broad range of elements, spanning the whole periodic table. Since, we are interested in structures that are defined on the nanometer scale, resolution of ~ 20 nm, or even better, would be necessary. The possibility of being able to do nano-XPS would be of great interest to us.

(b) Nanoscale surface studies (*Norton, Bancroft, Kasrai*, UWO)

Bancroft and Kasrai, and Norton and his group at UWO are carrying out nanoscale surface studies in a number of areas. In all of this work the intention is to connect macroscale phenomena, such as the friction and wear properties of coated metals, or polymer surfaces, to the micro- and nanoscale properties of those surfaces. Studies at UWO involve quantitative determination of the nanoscale mechanical properties of the surfaces by modern scanning probe techniques, particularly quanitative interfacial force microscopy. These studies would be complemented by spectromicroscopy studies at CLS. A number of specific experiments are planned.

Tribology: Antiwear (AW) and extreme pressure (EP) agents are used in a wide variety of lubricants, such as engine oil, to control wear under conditions of boundary lubrication. Some of the most commonly used commercial AW and EP agents contain S, P and N and function by forming surface protective films. These protective layers result from the chemical reaction (coating) of a surface by an additive or its decomposition products. Zinc dialkyldithiophosphates (ZDDPs) are a common class of AW and EP additives and are used in engine oil in combination with detergents and dispersants. Currently, new additives are being sought which can replace the present ones, since these materials (particularly the P) deteriorate the performance of vehicle exhaust catalyst systems.

Boron containing additives are among the new additives being investigated in this area. Engine manufacturers are also concerned about legislated exhaust emission levels. As a result, tighter engines that consume less lubricant are required. Engine manufacturers are considering the lubricant as a design component for future engines, so that it can cope with the problems of decreased oil consumption and increasing oil stress. This is forcing the need for better lubricant capacity in the areas of wear control and extreme pressure performance. Thus better understanding of the mechanism of action of existing antiwear/extreme pressure additives and the design of new additives will be extremely valuable for formulating lubricants to address the more severe service that is expected.

The nanomechanical properties of antiwear films derived from an oil additive, zinc dialkyl dithiophosphate (ZDDP), are being determined by interfacial force microscopy. Astonishing mechanical properties are evident in the antiwear "pads" in effective antiwear films. These pads are microns in size, but there are lateral variations in elastic modulus and hardness over much smaller length scales. The films are believed to consist of phosphate glasses of various chain lengths. Speciation of these films is best achieved by micro-XANES. It is essential to correlate the nanoscale mechanical properties of the films with their structure and chemistry. XANES microscopy at resolutions below 50 nm will be correlated with Interfacial Force Microscopy data recorded at UWO. The overall goal is to understand the chemical and microstructural origins of the best mechanical properties of antiwear films, use this knowledge to develop other routes to the same microstructure and wear resistance, and hence help to eliminate the ZDDP's from lubricating oils.

Reduction of weight in automobiles has led to the substitution of aluminium for iron in engine blocks. The poor wear resistance of aluminium currently necessitates a number of costly design features to minimise wear problems, for example the use of iron inserts for valve guides and cylinder liners. Cost and performance benefits would be realised if the need for inserts of wear resistant materials was reduced or eliminated. Historically, lubricants and additives have been developed for use with traditional wear resistant materials, such as irons and steels, with little understanding of how these lubricants interact with aluminium and its alloys (or indeed with the ferrous alloys). Improved understanding of the behaviour of existing automotive lubricants on aluminium and development of lubricants to improve the wear behaviour of aluminium based materials in engine applications would facilitate introduction of aluminium engine blocks in automobiles.

In the past, several analytical techniques have been used to characterize antiwear and extended performance films to understand the mechanism of the film formation. Electron probe

micro-analysis and X-ray fluorescence spectroscopy have shown the presence and spatial distribution of elements, such as S, P, O and Zn. Auger electron spectroscopy has been used both for depth profiling and chemical analysis of the films. Photoelectron spectroscopy has also been widely used in this respect by several investigators in the past for chemical speciation of the films. In recent years, we have used P L-edge, S L-edge XANES, and P K-edge and S K-edge XANES/EXAFS to chemically characterise both the surface (TEY measurements), and bulk (fluorescence yield measurements) of antiwear films [YK&97]. What is lacking in all the above investigations is the spatial distribution of the chemical species in the films. Very recently, with the collaboration of Dr. De Stasio from SRC, Madison, we used her X-ray Secondary Electron-emission Microscope (MEPHISTO) to obtain micro XANES images at the P L-edge, S L-edge, B K-edge, Zn L-edge and Fe M-edge of the antiwear films [CB&99]. We have found that PEEM microscopy is very suitable for imaging antiwear films and we intend to mount a intensive PEEM research program at CLS.

Polymer surfaces, biomaterials, biological materials. The Norton group is studying the nanoscale mechanical properties (modulus, hardness, chemical reactivity, adhesion) of polymer surfaces, biomaterials and biological materials. The goal is to correlate the mechanical properties of the materials (including the origins of mechanical signal transduction in biological cells) with microchemical information at <50 nm lateral scale. XANES microscopy with PEEM, using chemical state resolution at the carbon edge, will play a key role in these studies.

Photonics. Norton is involved in the photonics network Canadian Institute for Photonic Innovations (CIPI) and is working on nanoscale clustering on Ge on Si, using STM techniques. The UWO STM studies would be nicely complemented by micro-XANES studies by PEEM.

Aluminum alloy optimization (Bancroft, Norton, Kasrai) A project of high industrial relevance is the chemical speciation of oxide films formed on the surfaces of Al alloys, particularly the 5000 and 6000 series Al-Mg alloys. The latter should find use in automobiles. At many stages in the processing of these alloys to control their *bulk mechanical properties*, oxide films of varying composition and structure are formed which can have significant effects of subsequent processing steps. The ability to identify and quantify the complex oxides (spinels, MgO, Al_2O_3 etc.) is believed to be crucial to the better understanding and control of the films. This could be accomplished by XANES at the O, Si, Mg and Al edges.

(d) Surface Modifications of Graphite by Ion Bombardment (*Roy*, Laval)

In collaboration with J.-M. Layet (*Laboratoire de physique des interactions ioniques et moléculaires* Université de Marseille, campus St-Jérôme) Roy is studying "plasma-surfaces" -

specifically the surface modification of highly oriented pyrolitic graphite (HOPG) by ion bombardment, a problem related to damage of fusion reactor walls. To date we have used vibrational energy losss spectroscopy to study samples exposed to different doses of Ar^+ and H^+. Fluences varied from $1x10^{14}$ to $4x10^{16}$ ions/cm^2 with 5 keV ion energy. At low fluences, the low energy plasmon (53 meV) shifts to lower energy for the two ion species. For higher fluences (>10^{16} ions/cm^2) the plasmon mode vanishes, and no loss feature is observed in the case of Ar^+ bombardment. With H^+, chemical reactions occur at the surface and C-H stretching modes are observed which reflect various carbon hybridizations. These phenomena are linked to the structural modifications induced by ion bombardment.

In the context of this proposal these graphite samples would be examined with PEEM. Ideally one wants depth profiles of the structural modifications in graphite, and their dependence on doses and thermal treatments. The chemical specificity of PEEM will help understand the chemical changes that occur on ion bombardment, which can then be extrapolated to the situation of fusion reactor walls.

Another interest is the alkali metal / H-Si(111) (1x1) interface for which the lateral resolution of PEEM is promising. That project is studying a H-passivated silicon surface upon which an alkali layer would be deposited by evaporation. The PEEM study (Si 2p edge) would be used to determine what kind of bonding occurs on the surface in the presence or absence of the hydrogen termination layer - i.e. a comparison of the nature of bonding to bare silicon (with dangling bonds) versus H-covered silicon where there are no dangling bonds available. Will any diffusion take place below the top layers? PEEM may help to find answers to such questions.

III-5 Magnetic materials - surfaces and interfaces

B.W. Robertson, P.A. Dowben, S. Adenwalla, and B. Doudin (Nebraska-Lincoln)

Semiconductor technology has historically ignored a fundamental property of the electron: its spin. This situation has evolved in the 90's, with the emergence of magneto-electronics or spintronics. This revolution was initially driven by the discovery of the giant magnetoresistance effect in magnetic multilayers in 1987, and spin-dependent electronic devices were subsequently designed and realised. As part of our interest in high-density data storage, magnetoelectronic and spintronic materials and devices, we are involved in various aspects of research on submicron and nanoscale magnets and magnetic multilayers - shape, size, and scaling effects on coercivity and magnetisation, and spin injection - that we fabricate by radiation-assisted organometallic chemical vapour deposition and by electrochemical methods.

Funded by the National Science Foundation, we are developing longitudinal, as well as transverse, spin polarised instrumentation for EELS (SPEELS), photoemission and inverse

photoemission spectroscopy (SPPES and SPIPES) and are combining spin polarimetery with scanning transmission Lorentz and holographic electron microscopies. Polarization values for a number of magnetic systems that might be used as spin injectors are still a source of debate. The main reason is the difficulty of characterising and understanding interface properties that are often significantly different from the bulk values. Full characterisation of buried interfaces in spin tunnelling devices and of the surfaces of nanoscale patterned magnetic materials under passivation layers is essential to understanding their behaviour. We already have considerable experience in SPPES, SPIPES and Lorentz techniques. By the second half of 2000, our additional spin and nanoscale magnetisation mapping instrumentation will add considerably to our capabilities. Access to magnetically sensitive PEEM at CLS will be extremely valuable for complementary studies on the element-specific bonding at surfaces and buried interfaces of the same samples that we analyze at the University of Nebraska and elsewhere. We are already equipped to transport such samples to CLS in a self-powered portable UHV system.

IV Description of the proposed X-ray spectromicroscopy facilities

This section describes the ID, beamline and instrumental capabilities needed to carry out the scientific program, leading from the proposed scientific program, through the spectroscopy to the microscope configuration. The focus is primarily on the scientific capabilities rather than beamline design details, which are outlined in subsequent sections.

Table 1 Required properties for sub projects

Scientific Focus	**Elements**	**Core edges,Energy Range (eV)**	**Photon Polarisation**	**Required Resolving power**
Polymers (Gardella, Hitchcock, Dutcher, Stover, Urquhart)	C, N, O	K: 270-600	Linear	5000-10,000
Environmental and Bioremediation (Martin) NRCan (Forestry)	C, N, O	K: 270-600	Linear	10,000
	Ti-Zn (3d TM)	L: 560-1200	Linear	10,000
	Contaminants As, Se, Tc, Cd, Sr	L: 1-3 keV *PEEM (CSRF DCM)*	Linear	
	Actinides	O, N and M	-	
	Lanthanides	N and M	-	
Nanomaterials and Electrochemistry (Guay)	C, N, O 3d TM	K: 270-600 L: 560-1200	Linear	
Surfaces (Bancroft, Norton, Roy)	Al, Si, P, S, Cl	L: 100-1900	Linear	10,000
Magnetic Materials (UNebraska, future Canadian content)	Ti-Zn (3d TM)	L: 560-1200	Linear (XMLD)	3,000
		L: 560-1200	Circular (XMCD)	3,000
Geochemical	3d TM	L: 560-1200	Linear &circular	10,000

IV-1 X-ray source

Table 1 presents an overview of the photon characteristics required for the proposed studies of materials science (polymer chemistry and physics, magnetic materials, thin films and surfaces), geochemical, biological and environmental studies. The ability to perform measurements at several different core edges on the same sample region is essential.

Energy range: While there are some targeted applications for 100-250 eV photon energy range – Si 2p semiconductor studies; P 2p , S 2p for surface adsorbate, thin film and environmental work, etc – most of the users would be served with a beam line with a lower energy limit of 250 eV. *Our preliminary design is predicated on full performance only to a lower photon energy limit of 250 eV as this greatly reduces the requirements for optics cooling, in order to get adequate energy resolving power.* It will be possible to do some work below 250 eV, but only with reduced photon flux to avoid distortion of the optics. As the design refines, ways in which extension to include full performance in the 100 –250 eV range will be explored.

Energy Resolution: An ideal beamline for chemical microspectroscopy should have an ultimate resolution of half the core hole lifetime width at all energies of interest (i.e. 10,000 for O 1s). Practically, a resolving power of 5000-6000 with high flux is suitable for C 1s studies and a similar resolving power is also good in the tender X-ray range.

Photon Flux, Brightness and Coherence: The following metric is used to define adequate flux: The flux should be adequate to record a 512 x 512 image with adequate per pixel statistics in one minute at 4000 resolving power (i.e. typical energy resolution for chemical spectroscopy). To detect 1000 photons in 0.2 ms transmitted through a sample with an optical density (OD) of 3, an incident photon flux of 100 MHz is required. These numbers are somewhat higher than currently achieved at ALS BL 7.0 but there is every expectation that an undulator line fully optimised for STXM can achieve this performance (*Warwick, private communication*). Note that this estimate of the flux at the sample does not include zone plate and exit window losses. For a 10% zone plate transmission and two 50% transmission windows (280 eV), the beam should have about $4x10^9$ ph/s. Since a soft x-ray undulator beam line can produce 10^{11} - 10^{12} photons/s into a 50 μm beam spot (0.1 eV bandwidth), this requirement is practical, even with the addition of a 10 μm aperture to act as a spatial filter to ensure diffraction limited zone plate performance.

High x-ray brightness and coherence is required to perform diffraction-limited scanning x-ray microscopy using Fresnel zone plates. As the coherent flux is directly proporotional to the

spectral brightness, undulators operating at low emittance 3rd generation electron storage rings provide a high coherent flux for scanning x-ray microscopy.

Photon Polarisation: Circular polarised x-rays are necessary for the spectromicroscopic characterisation of ferromagnetic and ferrimagnetic materials using x-ray magnetic circular dichroism (XMCD) as a contrast mechanism [SP&98]. Both STXM and PEEM experiments are possible. Ideally one would like to vary the helicity of the light, rather than change the magnetisation of the sample – i.e. one wants to vary the probe, not the sample. For X-PEEM magnetisation studies this is essential since it is not possible to introduce a magnet field at the sample. The helicity is readily selected on bending magnet lines by using the beam above and below the plane. For an undulator line, the only possibility (aside from polarization devices [KK&97] which have limited applicability) is to use an eliptically-polarised undulator.

The ability to control and alter the angle of the x-ray linear polarisation will greatly improve our ability to perform linear dichroism imaging of antiferromagnetic (XMLD) [SP&98] and aligned materials. With typical horizonal x-ray polarization, rotation of the sample mount is necessary for differential linear dichroism image contrast. Sample position registry is inevitably destroyed by such a crude rotation, requiring realignment and greatly preventing the "differential dichroism" imaging experiments needed to map aligned and crystalline materials. On an EPU with full control of the linear polarisation direction it would be possible to acquire two images with the E-vector along the maximum and minimum orientation directions without rotating the sample. This would make a dramatic improvement in positional and rotational accuracy of linear dichroism studies.

The Beam line

A schematic of the proposed spherical grating monochromator beamline is shown in **Fig. 11**. This is based on the ALS 7.0 and 5.3 beam lines and is considered a schematic for purposes of identifying design challenges and costing issues, rather than the final optical design. While aspects of the line are similar to the ALS 7.0 undulator line we have borrowed heavily from the 5.3 design since, although it is mounted on a bending magnet, the phase space conservation issues which drove the 5.3 design are more suited for an undulator beam line optimised for STXM than the ALS 7.0 beam lines which is mainly optimised for spectroscopy. More technical aspects of the beam line are described in section V.

IV-2. Scanning Transmission X-ray Microscope

The STXM microscope will be designed as a multimode instrument for performing a wide range of scientific studies. User-friendliness and reliability will be stressed in the design of this instrument. The field of STXM microscopy is rapidly changing from one of mainly x-ray optics and instrumentation research to mostly applications in materials, environmental, biological and geological science. Thus our focus will be on instrument reliability and high quality of the results.

STXM Microscope Energy Range: The STXM microscope (focusing stages) would be mechanically designed to work throughout the entire beamline energy range (100-1900 eV, or Si 2p – Si 1s), but optimised for highest quality operation in the 250 – 600 eV energy range (C 1s – O 1s). Si_3N_4 membranes will be used as an exit window, since the transmission of a 100 nm Si_3N_4 membrane is adequate for energies from 250 – 1900 eV except perhaps at the N 1s core edge. The use of Si thin film windows as an alternative for better N 1s performance will be explored. For operation from 100 - 250 eV, provision for operation without an exit window (and with differential pumping) is envisioned.

Focusing Elements (zone plates): The initial zone plates for the STXM microscope would be acquired from CXRO-LBNL (Appendix letter B2). An e-beam lithography group in IMS, NRC has been approached to see if they would be interested in developing expertise in this area.

STXM Microscope Environment: The proposed STXM microscope is a multi-mode instrument, designed for a range of compatible experiments. The microscope will be able to perform measurements with dry and solvated samples, in air, He flushed, or a vacuum environment. A He flushed environment is suitable for most operating conditions, and does not pose great design challenges. However, the microscope must operate in vacuum for electron and ion yield detection studies, for optimum spectromicroscopy at the N 1s and O 1s core edges and from 100-200 eV, and during cryogenic cooling of the sample. Fast, high capacity pumping is needed to enable rapid exchange of samples for cryogenic and N1s / O1s operation.

This STXM microscope will have the ability to mount a cryogenic cooling stage, which requires a vacuum of 10^{-6} torr or lower to reduce icing on the sample. A light weight, miniature Joule Thomson cooling stage [M00]) will be used to cool the sample. With suitable stage design and operation constraints, fingerprint friendly operation is compatible with these high vacuum requirements. The design of the recently implemented SUNY-SB STXM microscope (STXM IV) at

the NSLS achieves similar design requirements [FB&00]. For high-resolution imaging, vacuum pumps must be vibration free and mechanically isolated: the SUNY-SB cryo-STXM at NSLS uses an ion pump[MO&00]. Electron microscopes routinely use a large reservoir tank for turbo backing so that the vibrationally noisy, backing mechanical pump can be turned off in periods of critical high-resolution operation.

STXM Microscope Positioning Elements: Stepper and peizo stage design will achieve reproducible and high accuracy positioning of microscope elements to ensure 20 nm spatial resolution, compatible with current and prospective zone plate optic resolution. A particular design goal is the ability to acquire spot-NEXAFS spectra of samples at the highest microscope resolution using dynamic interferometry position correction to compensate for changes in lateral registry as the ZP moves to maintain focal position (e.g. on the fly position correction). This interferometric scheme will also be able to make corrections to track and compensate vibrational motion up to a few hundred Hertz.

In current STXM designs, there is misalignment between the optical axis and the mechanical stage axes which results in shifts in the position on the sample during spectral scans. Several strategies have been used to correct for this misalignment: correction tables (ALS BL 7.0 STXM) or image-sequence methods [JW&00], but these approaches can be time consuming and unreliable. While is too early to define the next generation of microscope stage and feedback methods, the CLS STXM microscope design will build on the ALS BL 5.3 STXM development, which include interferometer-based measurements of the relative (x, y , z) positions of the sample relative to the zone plate. This will provide much more precise and accurate sample positioning, stability of the field of view during photon energy scanning, feedback for piezo stability, feedback for movement along the optical axis, and z-axis feedback for optimum zone plate focus.

Such feedback will also be necessary to overcome mechanical and inertial effects from the movement of heavy sample holders and sample holders that experience drag from heating, cooling or other connections for *in situ* sample modification (e.g. tubes for solvent modification, etc.).

STXM Sample Mounting: A basic sample holder design will be established that can be indexed to an external optical microscope, with compatibility with other CLS microscope indexing systems (FTIR and microprobe beamlines). The sample holder will involve kinematic mounts, to enable the rapid and reproducible exchange of samples, through a load lock or glove bag interface.

A basic kinematic sample mount design will be established, permitting modification for specialised sample holders within the spatial constraints of the microscope (viz STXM-IV design at NSLS). Such designs shall include suitable thermal isolation, to permit cryo-cooling (using micro-miniature Joule Thomson cooling stages [M00]) or *in situ* sample heating.

STXM X-ray Detectors: No single detector will satisfy all STXM operation modes, but a "swappable detector" platform should meet most of the detector problems identified below.

A motorised detector stage will allow detectors to be changed without the need for opening the microscope for detector re-alignment, as well as fast swapping between the detector and an internal optical microscope. For example, the new SUNY-SB STXM microscope (STXM-IV) uses such a platform and can accurately and reproducibly change from the optical microscope to a gas proportional detector in < 10 seconds. The importance of quick and reliable exchange between x-ray detection and optical microscopy for fast sighting of desired sample regions can not be understated. An internal optical microscope, along with pre-indexing of the interesting sample regions on a fiducialized external optical microscope, can largely overcome the slow speed of scanning large sample areas by STXM microscopy.

Due to the range of x-ray energies and experimental needs, the provision for several different types of x-ray detectors is necessary. For analytical applications, a detector that is linear at high-count rates (>20 MHz) is essential. A combination of detectors for low and high count rate modes is envisioned (i.e. the best detector for 20 MHz may not be suitable for 30 KHz operation). In addition to enhancing throughput, fast detection (high photon flux plus fast detectors) will open up new classes of kinetic experiments. Flexibility for future developments, such as phase contrast and fluorescence detection, will be built in.

Dark field and some phase contrast can be implemented in STXM with a suitable multi-detector structure. There are detector schemes already implemented in the NSLS STXM to do some types of phase contrast imaging, and some preliminary work is now underway. These aspects will be of growing importance so we intend to equip the CLS STXM with a multi-segment detector that is able to do dark field and some phase contrast. In addition, a number of projects are envisaged in which one would like to use surface sensitive total electron yield (TEY) detection at the same time as transmission detection. With good vacuum, this is quite feasible using approaches explored at the ALS BL 7.0 [HT&98].

IV. 3. PhotoElectron Emission Microscope (PEEM)

Overview of PEEM Design

At the CLS spectromicroscopy workshop the CLS x-ray microscopy community indicated strong support for the stategy of purchasing a commercial, mobile-PEEM microscope station which could be implemented on existing beam lines prior to CLS commissioning. The PEEM microscope will not be a state of the art aberration correction PEEM, since this is unambiguously considered incompatible with mobility. Such an instrument may be considered as a future addition to the beamline.

A mobile-PEEM based on a commercial instrument will give modest resolution NEXAFS imaging (~50 nm), with a perfromance similar to Mephisto [S&98], that will satisfy the present needs of the community. This will be a UHV instrument, with a conservative implantation of characterisation tools (LEED-Auger, evaporation, gas dosing, etc in the prep chamber).

A mobile arrangement is desired in order to perform spectromicroscopy at beamlines other than the SM-EPU. This will require a stand that is suitably flexible to allow access to different beam heights, beam line angles and focus positions. The microscope, chamber, stand and electronics will be optimised for simplicity and flexibility, so that moving can be done with a minimum of set-up time. A means to rapidly adjust to an “indexed” chamber position will be used to speed chamber relocation (i.e. the rate limiting step should be pumping and baking).

PEEM Capabilities

The PEEM microscope, end station and support hardware will be designed to have the following characteristics:

- Imaging with sub-100 nm resolution. The state of art is ~20 nm in favourable cases, but the actual resolution depends on the electron emission and conductivity properties of the sample.
- NEXAFS spectroscopy / spectromicroscopy
- Surface EXAFS
- Small spot XPS (offered by Staib and Specs/Elmitech; Omicron only has an energy filter. There is performance data for the Specs (Bauer instrument) from Trieste but none for Staib. There is some concern that the intensity for all but low energy photoelectrons (<50 eV) from high cross-section core levels will limit wide elemental range studies)
- Sample load-lock with indexed sample holders.
- Ancillary preparation and characterisation capabilities. For the first CLS PEEM microscope, a conservative approach is desired. This design will focus on common surface science

preparation and analysis techniques (LEED-Auger, sputtering, evaporation and gas dosing) rather than try to develop an 'all-techniques type of surface analysis chamber.

PEEM beamline: The CLS "home" of the PEEM microscope will be a branch of the soft x-ray microscopy beamline, but the microscope stand and components will be designed to be portable, and will move to other beamlines of interest at the CLS. This arrangement will permit studies at the following energies:

- UV-vis (10-40 eV), for valence band studies (future monochromator)
- Magnetic and crystalline materials study, on the "home" soft x-ray microscopy SGM (as described above). This beamline will enable full control of linear, elliptical and circular polarisation in the energy range 100 – 1900 eV.
- CSRF SGM (60-1200 eV)
- CSRF DCM (1400 - 9000 eV)

IV.4 Experimental Support

Experimental components that are considered essential parts of the 'central' soft x-ray microscopy facility are considered here and, where necessary, included in the overall beamline budget. There are other experimental resources at the CLS and the University of Saskatchewan which will support the operation of this beamline that are not considered part of this application.

Core Support Equipment for Soft X-ray Microscopy Beamline

a.) Stand alone optical microscope with indexed stage: A good optical microscope (5x, 10x, 50x and 100x, differential interference contrast (DIC), transmission and reflectance imaging) is essential for sample previewing and evaluation *at the X-ray microscope* (not in a remote location) to enable fast decisions of experiment suitability, strategy, etc. The stage will be indexed directly to the co-ordinate system of the STXM and PEEM microscopes through specifically designed sample holders. The ability to capture, process and store optical microscope images with the x-ray microscope data will be incorporated.

b.) Laminar flow hood: A reasonable quality laminar flow clean sample preparation area is eesential for UHV sample preparation, etc. for the PEEM sample, It is a lso a desirable environment for STXM sample preparation.

Other Equipment Existing at the University of Saskatchewan

Sample Preparation:

- Room temperature ultra-microtomy (facilities at Vet. College, biology at USask)
- Cryo-ultramicrotomy (Urquhart – NSERC application pending)
- Ion milling, metal and carbon evaporators (Vet. College, USask)
- Spin caster (Urquhart – NSERC application pending)
- Vacuum oven, fume hood (available in CLS chemistry lab)

Sample Characterisation:

- Scanning electron microscopy, transmission electron microscopy (Vet. College and others at USask)
- Scanning Transmission Electron Microscopy (to be purchased, CFI funded, Ron Miller, Mechanical Engineering)

Atomic Force Microscopy and Confocal Microscopy (to be purchased, CFI funded ***Saskatchewan Structural Science Centre***)

V. Technical Details

V.1 Overview of project design, construction and commissioning issues

The undulator would be the responsibility of CLS. The design of the insertion device will be carried out by CLS (which is in the process of hiring an undulator expert) and be tendered. CLS intends to have capabilities to confirm magnetic and X-ray optical properties of critical elements and we would rely on these capabilities to ensure adherence to design specifications.

The beam line would be designed as a collaboration between CLS 'experimental systems group' and Dr. Tony Warwick (TW Consulting - see attached consulting proposal). Dr. Warwick has designed, managed construction, commissioned, and operated several X-ray microscopy beamlines, including the highly success BL 7.0 at the Advanced Light Source. His expertise will bring much to this project, particularly with regard to optimising the match between microscope, beam line and insertion device properties. Most of the beam line components would be tendered, with assembly and commissioning being carried out by a combination of CLS staff and members of this beamline team (BT).

Software for beamline control (automated slit setting, grating changes, grating rotation, etc) and for microscope control will be developed by the BT. Undulator control and permit setting would be done using standard CLS protocols.

The STXM microscope would be built in-house from commercial stages, using design and fabrication expertise developed through the involvement of *Hitchcock* and *Tyliszczak* in the ALS BL 5.3 project, and *Urquhart* in the NSLS STXM development.

Personnel salary during construction and commissioning for the BT components (see **table 2**) would be paid as part of the capitalized salaries indicated in the proposed budget. The BT members would provide the personnel and operating costs for the negotiated fraction of BT dedicated time, as indicated in the 1-page summaries of each BT member.

A time line for the development of the overall project is given in **Figure 12**. This is aggressive, but has been considered feasible by our expert consultant (Warwick). It is consistent with the goal of being one of the 6 beamlines operating at formal commissioning of CLS in Dec-03 which is mandated with the CFI agreement

The technical design of the undulator and beamline needed for these x-ray microscopes is described in greater detail below, starting from the ring and moving downstream to the target. This is very much a conceptual discussion - a rigid design will not be proposed in the 10-Feb-00 application since more design work is required before finalisation. We note that the timeline (Fig. 11) contains numerous checkpoints at which design and progress reviews will be used to enusre orderly and timely development.

V.2 Elliptically Polarized Undulator (EPU)

Configuration Summary

This beamline will be designed for maximum brightness in 250-600 eV range, but will be able to cover the energy range from 100 eV to 1900 eV (Si 2p to Si 1s). The flux must be adequate to meet the STXM photon requirements discussed above in section IV-1. Since STXM uses zone plate focussing, a high brightness source, such as an undulator is essential. We propose to use an elliptical polarised undulator (EPU) as the insertion device for this beamline. The photon flux and brightness of an undulator is extremely valuable for zone plate microscopy, but an EPU will enable a new class of experiments. The EPU design we propose will provide full control over the photon polarisation – linear in all orientations, elliptical and both circular polarisations. Two different control modes are proposed:

- Tuneable polarisation from user control (e.g. a knob) at 'reasonable' rates (10's of seconds).
- Fast (automatic) switching between left and right circular polarisation or vertical and horizontal linear polarisation for spectroscopy (difference spectra) and microscopy (difference imaging). This latter will only be implementable assuming the adopted EPU design takes up only ½

of the straight section and that funds are found to build the second half of the two undulator scheme required for fast polarisation switching.

The conceptual model for this device is the EPU on beamline 4 at the ALS [YMP99] (see **Figure 13**). It is a Sasaki-type device [SMT92, SS94] composed of 37 periods each 5 cm long (total length = 1.9 m) with 4 quadrants (16 magnets) comprising each period. The polarisation is adjusted by moving the quadrants relative to each other. For full polarisation control this requires the ability to move 3 of the 4 quadrants independently. Q2 and Q4 are moved horizontally synchronously to rotate circularly polarised light while moving Q1 andQ4 synchronously vertically rotates the linear polarisation direction. Considerable sophistication is required in meshing the motions of the undulator with compensating changes in the rest of the storage ring (*Padmore, private communication*). We would propose that CLS avail itself of the offer of ALS to assist in the training of CLS ID and accelerator staff to learn from the ALS's extensive experience in this area.

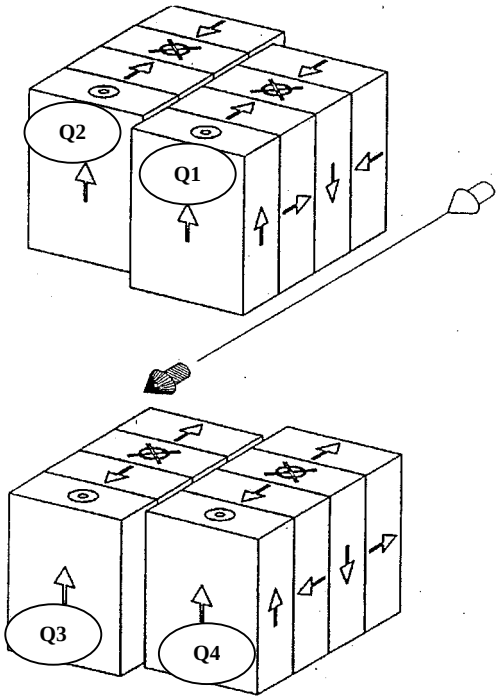
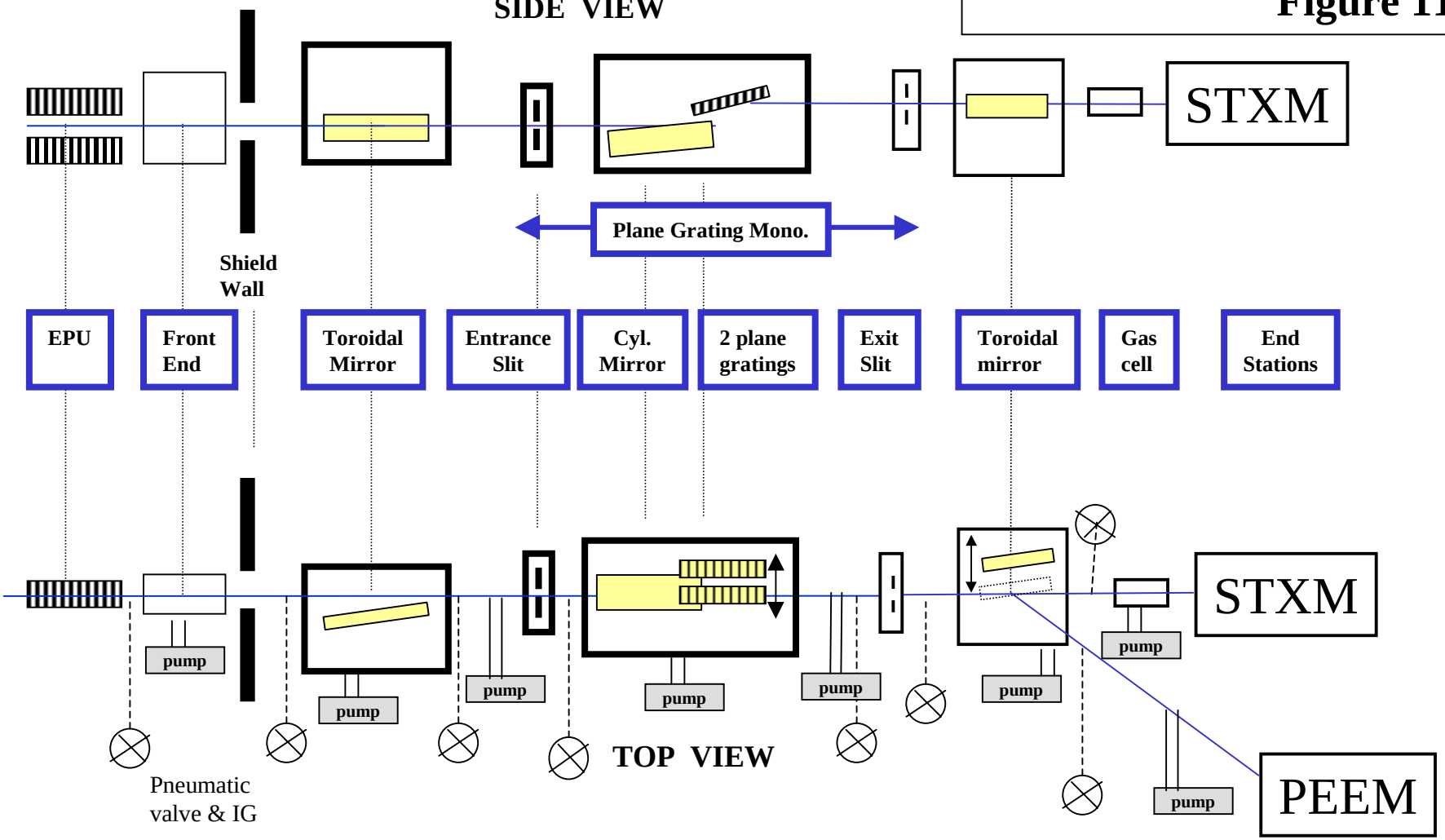


Fig. 13 Sasaki-type EPU. Polarization is varied by translating Q2, Q4 along the beam, while Q1 and Q3 are fixed. (Young 99)

The additional cost and increased complexity of an EPU, both for construction and operation will be challenging for CLS. The benefits of this device over a conventional linearly polarised undulator would be greatest for magnetic circular dichroism measurements, but would also enhance and enable studies of anisotropic and crystalline materials, such as aligned and crystalline polymer materials and other systems (e.g graphite-like materials). Recent experience from the ALS [P99] indicates that the increased cost for an EPU is not excessive (e.g. estimated US850K for a linear undulator versus US$1150K for an EPU). EPUs are being installed at BESSY 2. The increasing implementation of EPUs in 3rd generatation synchrotrons indicates that this is no longer considered to be an "unusual" technology, although it is definitely not 'off-the-shelf'. The improved performance, instrumental flexibility and capacity of experimental innovation strongly indicate the desirability of EPU technology for this beamline at the CLS.

CLS high resolution Spectro-microscopy undulator beam line optimized for STXM and PEEM from 250 eV to 1900 eV

CLS spectromicroscopy proposal
Figure 11.



SM-sketch..ppt 7-Feb-00

CLS Spectromicroscopy Beam Line Project Schedule

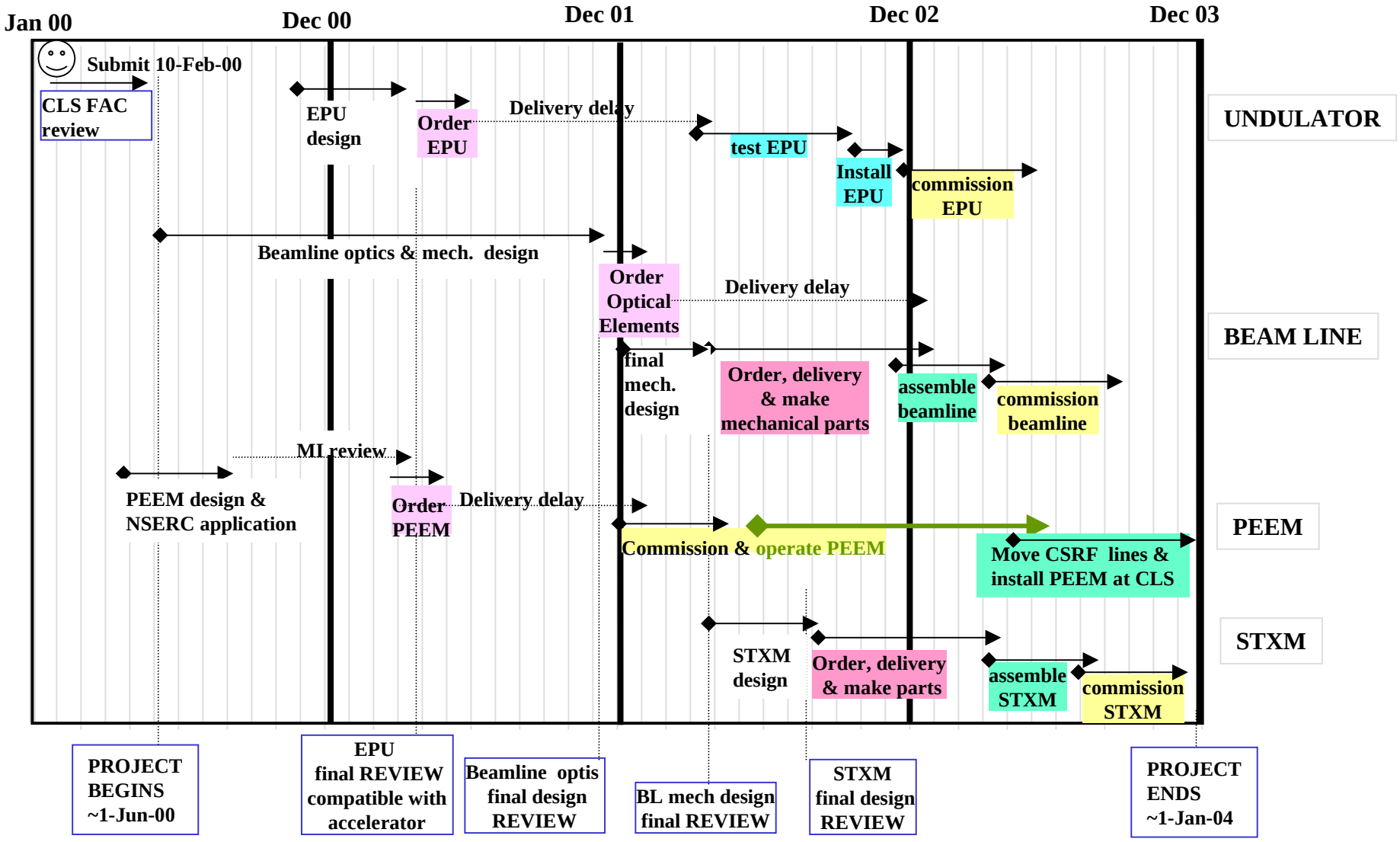


Figure 12. **Timelines and review milestones for the CLS spectromicroscopy project**

V.3 Grating monochromator beamline

Configuration Summary

The current concept for this beamline is a plane grating monochromator (PGM) which is an 'SX-700' type design, in some ways modelled on the PGM on the EPU beamline 4.0 at the ALS. Similar design principles are being used for the EPU undulator based super-STXM MES beamline now being developed at ALS (Warwick, private communication).The monchromator on ALS BL 4.0 achieves a wide spectral range with a single variable groove depth grating (20-1800 eV, with 1 x 10^{13} photons/sec/0.1%BW at 800 eVwith ALS operating at 1.9 GeV, 400 mA) and a resolving power (E/ΔE) of 5,000-10,000). This is the basis for the beamline sketch given in **Figure 11**. A serious alternative is a spehrical grating monochromator (SGM) such as those used in ALS BL 5.3 and 7.0. The BL 5.3 STXM line will use an SGM grating with small optics, and The choice of SGM or PGM will be established early in the design phase of this microscope beamline.

The desired energy range of this beamline will be 100 to 1900 eV, although, our current design restricts full power operation to above 250 eV in order to keep optics specifications within currently feasibly fabrication and cooling technology. The grating ranges will be optimised to combine energies of likely correlative interest onto a single grating, so that grating changes can be avoided as much as possible. The current proposal is:

- Grating #1, optimised for 280-600 eV (correlative C 1s, N 1s and O 1s)
- Grating #2: optimised for 500 – 1900 eV

The resolving power of this SGM will be at least 5000 over the 250-1900 eV range. Optics parameters (groove depth and blaze angle of the grating, included angle) will be chosen to provide peak performance of grating #1 in the C 1s region, and peak performance of grating #2 at 1000 eV.

Provision will be made for incorporation of a set of pinholes which can be used to spatially filter the beam to achieve diffraction limited operation. In addition attention will be paid to ensuring the light supplied by the line is spectrally pure to better than 0.5%. This will be accomplished by one or more order sorting strategies such as mirror coatings, a gas filter and/or metal foil filters, based on methods now in use at the ALS and NSLS STXM microscopes.

Sideline(s)

A **side station for PEEM and spectroscopy** at ~0.1 mm spatial resolution will be built to take advantage of the EPU for spectroscopy and XMCD/XMLD studies. However the monochromator and beam line X-ray optics should be optimised for the spectromicroscopy experiments. A bendable, variable focal-length mirror will be used to direct the beam to the side station line to allow a range

of different experimental end station placements, since the STXM, PEEM, and potential spectroscopy chambers will not be at same distance along the X-ray path. This arrangement will be designed to permit easiest optimisation of the STXM and PEEM microscopes which will be the core of the CLS spectromicroscopy facility. In order to preserve the delicate stability of the STXM alignment, this mirroring arrangement will be designed so that STXM operation can be regained without significant realignment of beamline optics (e.g. mirror for side station would simply be retracted for STXM operation). Our current preferred solution is to have the STXM take the 0^o beam, and the side station be activated by moving a deflection mirror into the beam.

Beamline Design and Construction Philosophy

So far the beamline conceptual design has been generated with input from *Adam Hitchcock, Emil Hallin, Stephen Urquhart, Tolek Tyliszczak,* and *Tony Warwick.* This group of individuals, with perhaps others from CLS as the CLS staff grows, would be the design team for the project. A beamline development co-ordinator would be hired using part of the capitalized salary component of the budget. The undulator design, tendering, and installation would be the responsibility of CLS. The monochromator and beam line will be designed by this team, relying heavily on contract design work of Tony Warwick, paid from this project budget (see Appendix B-1). Fabrication would be done on a parts-only basis by external contractors, with CLS as prime contractor and a mix of CLS and BT members acting to assemble the beamline and instrument. Commissioning would be carried out by the BT. This approach is chosen, rather than a tendered contract for the full beamline, because we believe it is essential for CLS to develop in-house expertise for design, assembly, parts testing, and commissioning of soft X-ray monochromators. CLS will build 5-10 such devices in the course of its existence. It is imperative that local skills in this arena are developed as soon as possible.

CLS Spectromicroscopy Proposal Budget Details (confidential)

component	supplier	CDN $000	Q-source	delivery (mo)	Test/install costs	shielded ?	responsible	Comments
Breakdown of costs for CLS spectromicroscopy project				capital =	**5172**	install =	**280**	
1. Behind wall components		**2250**			**50**			
Insertion device								
EPU	ALS	**1400**	(Padmore)	18	**0**	**Y**	CLS	CLS accel installs
low-beta quads	CLS	**200**	(hallin)	18	**0**	**Y**	CLS	
Front end	Jonsen UV	**650**	(Hallin)	12	**50**	**Y**	CLS	
the works	package tender							
2. BEAMLINE		**1902**			**180**			
Design / oversee	TW consult	**50**	Q (Dec-99)					
First mirror		**265**			**10**	**Y**		ALS BL 5.3 =CDN $284 installed
toroid element	JY	180	(TW estimate)	12			CLS	
cooling	CLS	30	guess	6	5		CLS	
pump&diagnostics	MDC	20	guess	6			CLS	
vacuum tank	MDC	10	guess	6	5		CLS	
FOE hutch	CLS	25	UMA	6			CLS	
Entrance slit		**75**			**15**	**Y**		TW estimate = $86
slit mechanism	Jonsen	25	guess	8			CLS	
cooling	CLS	10	guess	6			CLS	
vacuum tank	MDC	10	guess	6	5		CLS	
pump & diagnostics	MDC	20	guess	6			CLS	
ent slit hutch	CLS	10	guess	6	10		CLS	
Monochromator		**845**			**30**	**Y**		
2 gratings	Zeiss	225	TW estimate	12	10		BT / CLS	
cylindrical pre-mirror	J-Y	75	TW estimate	12			BT / CLS	
mechanism & vac tank	JenOptic	500	TW estimate	8			BT / CLS	compare Jonsen
cooling	CLS	15	guess	6	10		BT / CLS	
hutch	CLS	30	guess	6	10		BT / CLS	
Exit slit (2)		**92**			**10**	**N**		
mechanism	McPherson	92	TW estimate	8	10		BT / CLS	

CLS Spectromicroscopy Proposal Budget Details (confidential)

Switchyard		**100**			**10**	**N**				
toroid mirror	JY	50	guess	12	5		BT / CLS			
mechanism	CLS	30	guess	6			BT / CLS			
vacuum tank	MDC	10	guess	6	5		BT / CLS			
pump & diagnostics	MDC	10	guess	6			BT / CLS			
Beam tubes etc		**160**			**70**	**N**		1 valve/section		
wall-FOE	MDC	15	guess	6			BT / CLS	4 pumps		
FOE - ent slit	MDC	25	guess	6			BT / CLS	1 ion pump		
entr. - grating	MDC	15	guess	6			BT / CLS			
grating - switch	MDC	25	guess	6	70		BT / CLS	1 ion pump		
switchyard -exit slit	MDC	15	guess	6			BT / CLS			
exit slit - gas test	MDC	25	guess	6			BT / CLS	1 ion pump		
gas test - STXM	MDC	15	guess	6			BT / CLS			
exit slit - PEEM	MDC	25	guess	6			BT / CLS	1 ion pump		
Gas Test station		**20**			**5**					
valves & plumbing	MDC	10	guess	6	5	N	BT/CLS			
pump & controls	MDC	10	guess	6			BT/CLS			
Beamline controls		**120**			**30**					
valve/guage interlocks	MDC	40	guess	6	10		CLS	part of FE ?		
interfacing	various	80	guess	6	10		CLS			
software	CLS/BT	0	guess	6	10		CLS			
Services	UMA	**175**	hallin	12			CLS	35 m line @ 5K/m		

CLS Spectromicroscopy Proposal Budget Details (confidential)

3.1 STXM		**500**			**50**	**N(*)**		enviro hutch		
enviro hutch	CLS	20	guess	6			BT	for noise		
fast shutter (in vac)	Princeton Instr	10	guess	6			BT	only		
vibration table	Newport	15	guess	6			BT			
vacuum tank	MDC	25	guess	6	10		BT			
pumping & guages etc	MDC	30	guess	6			BT			
fine stage (200 μm xy)	PI	40	guess	6			BT			
coarse stage (xy)	Newport	40	guess	6			BT			
OSA stage (xy)	Newport	10	guess	6			BT			
ZP stage (z)	Newport	20	guess	6			BT			
zone plates	(CXRO)	40	(Attwood)	6			BT			
sample motion (z)	Newport	15	guess	6			BT			
detector stage	Newport	20	guess	6			BT			
laser interferometer	HP	45	quote	6			BT			
control & acquisition	as per 5.3	30	guess	6			BT			
computer, software	clones, Labview, IDL	15	guess	6			BT			
in-vac microscope	Zeiss	15	guess	6			BT			
PMT detector	Hammamatsu	10	guess	6			BT			
design, link parts	CLS	20	guess	12	40		BT			
indexing light microsope	Zeiss	50	(catalog)	6			BT			
ethane chiller (cryo)		10	guess	6			BT			
cooled load lock	MDC	20	guess	16			BT			
3.2 PEEM		**500**			**0**	**N**	BT	NSERC - see		
Staib system	Staib, Omicron	480	Q (Nov-99, Feb0	12	0		BT	application		
laminar flow lood		20	guess	6	0		BT			
TOTAL		**5172**			**280**					
Contingency	**10%**	**520**			**28**					
Grand Total				**6000**						

VI. Financial Plan

VI.1 BUDGET SUMMARY

capital

Insertion device (undulator) , front end including heat control	2.25 M
monochromator and beamline (*PGM line*)	1.90 M
End stations	
Scanning Transmission X-ray Microscope (STXM)	0.50 M
Photoelectron surface imaging system (PEEM)	0.50 M
capitalized salaries for development (5 people-years)	0.30 M
Contingency (10%)	0.55 M
Total CDN	**$6.00 M**

VI-2 Budget details

A section-by-section breakdown of costs is given in **table 2** (Excel spreadsheet). The prices for specific items were estimated from catalogues or, in a few cases, quotations. The pricing has been reviewed by Tony Warwick, and amended according to many of his suggrstions. The intent is NOT to provide a rigorous budget, but rather to build confidence that the funding requested is sufficient for the project to succeed. A 10% contingency is included.

If it is of interest, this table can be supplied electronically to allow anyone to adjust cost estimates etc, to quickly see impact on overall budget etc.

VI.3 Funding Plan

We intend to submit a NSERC Major Installation application in Oct-00 for $500 K for the PEEM station. This funding is not CFI matchable. The remaining funding requirement is CDN $5.5 M. Of this, following the CFI matching rules, $2.2 M (40%) is requested from the beamline component of the CFI grant. 3.3 M must be found outside the CFI grant to CLS. If the current funding initiatives to Alberta and Ontario are successful, we request this amount from these sources. We note that NRCan may provide 0.25 M towards this capital component but has not committed at the present time (Appendix letter A.1). Any funding from NRCan would act to reduce the funding request from the direct provincial grants. In addition, the Alberta request explicilty identifies $1 M for this project. If both these identified sources come in at the indicated levels, this would leave 2.05 M as the amount requested from the Ontario funding.

If the provincial initiatives are not fully successful, the BT would apply directly to their provincial CFI funding agencies (Innovation Trust in Ontario, FCAR in Quebec) in proportion to the relative benefit and involvement in the project. At present this would consist of perhaps $ 1 M from Quebec and $2.3 M from Ontario.

VII Beamline Team: benefits and operational responsibilites

VII.1 Internal beamtime allocation for BT members

The beamline team (BT) will negotiate with CLS for dedicated beam time on the basis of
(a) the funds generated by the BT; and
(b) the extent of participation in design, construction and commissioning.
In the limit that all funding outside of that from the CFI grant to CLS (i.e. 60% of the overall cost) is provided by BT fundraising, we will argue that this, along with the projected high level of involvement of BT members in design, construction (of the STXM microscope, and possibly some beam line components), software, commissioning and operation, entitle us to the maximum dedicated beam time, 75%. At the other limit, if all of the funding is provided from CLS, the BT would claim 30% dedicated beam time, on the basis if its projected contributions to design, construction, commissioning and operating costs. The proposed breakdown of the BT allocation in the case of an allocation of 60% of total beamtime to the BT is indicated in **table 3.**

VII.2 "BT membership" rights and responsibilities will be negotiated internally and with CLS. A starting point is a mutual recognition that providing funds, in-kind resources, and/or development/operating personnel is required for being part of the core-membership group, with 'special' privileges / responsibilities. Those who have indicated their support and commitment at the time of this proposal (as indicated in the individual 1-page member summaries, which have all been individually approved), and who 'hang-in' with on-going support of the capital and operating costs of the project, will get a suitable share of the special access 'reserved' for the BT in the first period of operation. We support the general principle that BT members must provide operational support to cover expenses and salaries of the beam line personnel during the period for which they obtain 'reserved' access.

Table 3. Proposed internal beam time allocations

Name	Affiliation	Dept.	Application(s)	Time (8 hr shifts/year)	
				STXM	PEEM
G.M. Bancroft,M. Kasrai	CLS		Tribology		20
J. Brown	NRCan	CANMET	Resource technologies	30	10
R.G. Cavell	U. Alberta	Chemistry	Alberta applications	10	20
J. Dutcher	Guelph	Physics	Self-organisation	20	
J. Gardella	SUNY-Buffalo	Chemistry	Biomaterial interfaces	10	20
D. Guay	INRS	Energie et Materiaux	Nanomaterials for Electrocatalysis		20
A.P. Hitchcock, T. Tyliszczak	McMaster	Chemistry	Polymers Bio-material interfaces	50	20
R. Martin	UWO	Chemistry	Plant materials	10	
P.R. Norton	UWO	Chemistry	Tribology, nanoproperties		20
B. Robertson, P.A. Dowben, B. Doudin, S. Adenwalla	Nebraska-Lincoln	Physics Mech. Eng	Magnetic thin films		40
D. Roy	Laval	Physique	Ion modified materials		10
H. Stöver	McMaster	Chemistry	Designer polymers	20	
S.G. Urquhart	Saskatchewan	Chemistry	Polymer microstructure	60	10

* An estimate of time the group would use per year, expressed as # of 8-hour shifts By 2006/7 CLS will provide about 5000 hrs or 625 8-hr shifts per year. In first years this will be reduced due to larger commissioning and ID installation needs. 500 shifts per year at CLS start-up would be a reasonable target. The table is based on the BT internally allocating 60% or 300 shifts/year in the first 3-year operating period (2004-2007). The times indicated sum to **210** shifts for STXM and **190** shifts for PEEM. I assume 100 of the indicated PEEM shifts per year are on other beam lines.

In constructing the time allocations outlined in Table 3, it is assumed that the BT will have at least 60% of beam time reserved for internal allocation to BT members. For this beam time the BT members would not have to go through the application process for general user time. Each BT member is expected to make an annual report of their activity that will include projections of their work in the coming year. This document would be provided to CLS management as a record of activities. Since there are a large number of BT members and thus the share for each individual is small, the BT members expect to be eligible for access to additional beam time on the line as general users, on the basis of peer reviewed competition.

VII.3 BT personnel and operating details

Based on the assumption that the beamline team will play a major role in operating and maintaining this beamline and the two microscope end stations, it is projected that the beamline team will fund at least 2 permanent staff members (a scientist and a technologist). In addition to training new users, maintenance, and assistance with the research programs of the beamline team

members, these individuals would carry out fee-for-service research, on contracts arranged either through BT members or the CLS. In order to accommodate this activity, and to facilitate generation of CLS operational funding throgh provision of timely fee-for-service, *a fixed period of each week's operating schedule would be reserved for rapid access*. For STXM where sample turn-around of 30 minutes is typical, this would be achieved by reserving 4 hours every afternoon (12-4 pm). These hours are chosen to allow 'same day service' whereby high priority samples could be studied shortly after the last FedEx arrival, and the results posted on the web later that evening. This would allow clinical studies to be carried out for example. For PEEM, where UHV load locking protocols can require several hours, it is likely a day per week would be blocked off for fee-for-service activities. In the event there was no high priority rapid access work, any outstanding lower priority fee-for-service work would be executed. In the absence of no fee-for-service demand, this time would revert to the currently shceduled BT or independent investigator as a bonus, i.e. not counted against assigned hours.

II.4 BT Operating budget (fee structure for fee-for-service and proprietary work)

- BT members are committed to paying **$50/hour**, through an annual levee to cover their share of overall BT operating costs. In-kind contributions would be allowed as partially coverage of the BT membership fees.
- Low priority fee-for-service would be charged at **$120 / hour**, of which 50% would revert to CLS and 50% to the BT budget.
- High priority (rapid access) or proprietary work carried out in fee-for-service mode would be charged **$200 / hour**, of which $80/hour would go to the BT budget and $120/hour to the CLS.
- Charges would be made only for periods that synchrotron light was available, while the fee-for-service activity was being carried out. (no charge over injections or down times).

The following operational budget is based on

total of 400 * 8 = 3200 hours of internally allocated BT time
(300 on the STXM beam line; 100 on other lines)

30% of all BT time being fee-for-service = 1000 hours, of which 800 hours are normal and 200 hours (20%) will be rapid turn access or proprietary

INCOME

BT levee (400 shifts – 125 fee shifts) = 275 shifts * 8 hours/shift* $50 /hour
= 110,000

fee-for-service (rapid turnaround) = 200 * 80 = 16,000

fee-for-service (regular service) = 800 * 60 = 48,000

Total = **174,000**

Operating budget (*continued*)
EXPENSES

Scientist (with 15% benefits)	$75,000
Technologist (with 15% benefits)	$50,000
Maintenance, operating expenses	$49,000
total	**174,000**

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VI. Acknowledgments

We wish to acknowledge the scientific leadership and inspiration of the groups of Janos Kirz and Chris Jacobsen (Stony Brook), whose 20 years of design experience is largely responsible for the current state of the art of STXM microscopy, and the pioneering work of Harald Ade (NCSU) in applying soft x-ray microscopy to soft condensed matter and polymer physics. We thank the speakers and participants of several X-ray spectromicroscopy workshops who have helped define this proposal. We especially thank Tony Warwick, Howard Padmore and others at ALS who have generously shared information and provided helpful technical and moral support for this initiative. Sample provision by various colleagues, such as Billy Loo (UCSF), is gratefully acknowledged.

X Appendix

1. Letters of Support

A.1 Jim Brown – for NRCan contribution
A.2 Ron Cavell – for Alberta (ASRA) proposal

2. Quotations

B-1 Design consulting services (TW Consulting)
B-2 Zone plate supply (David Attwood, CXRO-LBNL)
B-3 PEEM station: Omicron
B-4 PEEM station: Staib instruments

A Letters of Support

Natural Resources Canada | Ressources naturelles Canada

Minerals and Metals Sector | Secteur des minéraux et des métaux

Ottawa, Ontario K1A 0E4 | Ottawa (Ontario) K1A 0E4

January 17, 2000

Professor Adam Hitchcock
Dept. of Chemistry & BIMR
McMaster University
Hamilton, Ont.,
L8S 4M1

Dear Adam,

NRCan is negotiating with CLS on the split of the department's $2M contribution (for beamline/end-stations). This credit (capital $) will be applied to beamlines of specific application to NRCan and its clients with some preferred time/user fee schedule and a role in beamline management. I have recommended a funding scenario which includes a sizable financial allocation (>$0.25M) to the spectromicroscopy project. Note, since no operating funds have been committed to CLS or its beamlines by NRCan, a user fee arrangement for access time (payable at the project management level) would also be needed to off-set yearly beamline operations.

I wish to remain on the co-investigator list for the beamline team (BT) in order to represent NRCan and indirectly our sizable industrial client base, at least till NRCan can develop a CLS- PEEM/STXM strategy.

Thank you for incorporating my suggestions as to research areas in which soft X-ray spectromicroscopy facility would be used by NRCan.

Sincerely,

Jim Brown, PhD
Materials Technology Lab., MTB
Natural Resources Canada

Canada CANMET

Dr. A.P. Hitchcock
Co-ordinator, CLS Soft X-ray spectromicroscopy proposal
BIMR, McMaster University
Hamilton, ON L8S 4M1

21 Jan 2000

Dear Adam:

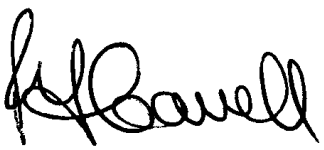
Soft X-ray spectromicroscopy is an emerging area which does not, as yet, have established practitioners in Alberta. However, as you will undoubtedly appreciate, our present ongoing staff renewal in all Universities will bring persons to us who will be interested in the capabilities of the Canadian Light Source. We recognize that a Soft X-ray spectromicroscopy beamline will offer unsurpassed capabilities in areas such as:

1. Imaging of biological materials - bacteria, cells and other structures of the appropriate size. This would be of interest to the Alberta medical research community.
2. Polymer and soft materials microscopy also has enormous potential for biomedical materials and the potentially large polymer community of Alberta. There is a large potential for industrial collaboration in this area.

In our hiring processes, we will be actively searching in these and related areas and so, on behalf of the future Alberta community of researchers and scientists in both University and Industry, I register the interest of Albertans as participatants in this beamline.

Financial support for the Soft X-ray Spectromicroscopy project is listed as an "emerging technology of interest to Alberta researchers and industry" in the CLS beamline funding proposal currently under consideration by the Alberta government. An amount of **$1M** has been allocated to the Soft X-ray Spectromicroscopy project. If this proposal is funded then Alberta would provide this level of funding but of course, the status of this proposal is pending. At this writing, funding is not secured nor is the probability of success easy to estimate.

For the present, please include my name on the list of participants to reserve space for ready future participation by Alberta researchers should it become possible for us to participate and keep me informed of all developments. I will eventually transfer the connection to suitable individuals or group once they are identified.



Ronald G. Cavell
Professor of Chemistry
Department of Chemistry
University of Alberta
Edmonton, Alberta, Canada T6G 2G2

E-mail: Ron.Cavell@Ualberta.ca
Voice: 780-492-5310
Fax: 780-492-8231

B.1 Proposal for consulting services during the design and construction of an undulator beam line for soft x-ray spectro-microscopy at the Canadian Light Source.

TW Consulting
667 Spokane Avenue
Albany, CA 94706 USA
tw_consulting@yahoo.com

Prof. Adam Hitchcock,
BIMR, McMaster University,
Hamilton, Ontario,
L8S 4M1, Canada.

Adam,

Here is a proposal for consulting services I can provide during the design and construction of an undulator beam line for soft x-ray spectro-microscopy at the Canadian Light Source.

I assume

1) project budget will be fixed and known at the outset,
2) purchasing authority will reside with the 'project team' so that work and procurement will be ordered by the project team,
3) beam line components will be built to specification for the project team by external vendors,
4) project can begin May 1st 2000 and specifications must be ready for procurement to begin November 1st 2001.

In order to assure performance, I propose:

1) to provide performance and cost estimates for alternative beam line designs to facilitate a decision by the project team between these alternatives
2) following this decision, to generate a complete beam line design consistent with the project goals and the project budget
3) to generate technical specifications for procurement of components consistent with project goals, the capabilities of the project team, the capabilities of commercial vendors and with the project budget.

Phase 1

The beam line would employ either

a) an SGM with moderate resolution and limited energy range from each of several gratings
b) a VLS grating monochromator with high resolution and limited energy range from each of several gratings
c) a VIA PGM with high resolution, extended energy range from each grating and dispersion selection.

These options are in order of increasing cost and performance. The choice of system would be made by the project team.

Deliverables:

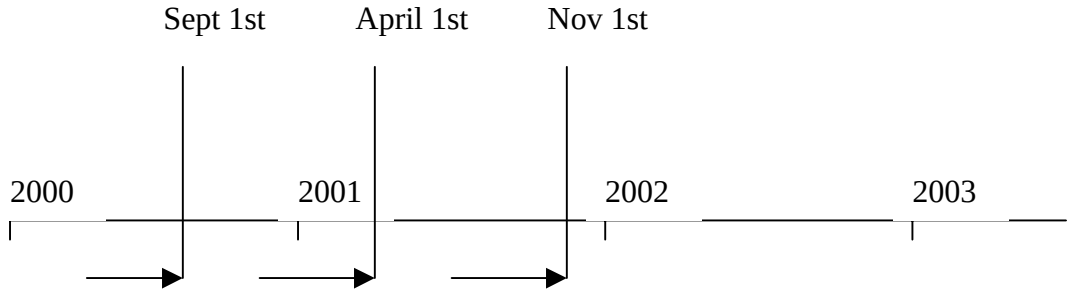
1) Description of beam line sub-systems required for each case.
   Estimates of energy range, beam line resolution and transmitted brightness for each case.
   Description of beam line operational procedures for each case.
2) Cost estimates (based on prior procurements of similar articles, where available) for each case.

Schedule:
Start May 1st 2000,
interim progress report July 1st 2000
final report September 1st 2000.

Phase 2
The detailed beam line design would be developed for one of the alternatives, based on the decision made by the project team after phase 1.
Deliverables:
The detailed beam line design would specify deflection angles, energy ranges, dispersion, resolution and transmitted brightness, based on published parameters of the CLS source. It would include estimated photon count rates in STXM and PEEM on this beam line. Account would be taken of the available floor space, based on published plans of the CLS building. Slope error tolerances for optics and groove profiles for gratings would be specified. Optical coatings would be specified. Positioning tolerances for all components would be provided. A set of diagnostic equipment would be specified to allow commissioning and operation of the beam line.
Schedule:
Start November 1st 2000
interim progress report January 1st 2001
final report April 1st 2001.

Phase 3
Detailed technical specifications would be provided so that all beam line sub-systems could be procured by the project team from external vendors. The specifications would be written such that the project goals could be accomplished within project budget.
Deliverables:
1) Complete technical specifications for each sub-system of the beam line, for inclusion in the wording of purchase orders by project team.
2) One or more price estimates for each subsystem from one or more external vendors, consistent with project goals and with project budget.
Schedule:
Start June 1st 2001
interim progress report August 1st 2001
final report November 1st 2001.

SCHEDULE



Phase 1	**Phase 2**	**Phase 3**
May	Nov	June
June	Dec	July
July	Jan	August
August	Feb	Sept
	March	Oct

Cost of consulting services:

I propose that each of the three phases described above be performed as a fixed price contract, with the deliverables described. Additional work beyond the deliverables described would be on an hourly basis at an hourly rate of US$110 per hour. Costs for any travel at the request of the project team would be reimbursed in addition. Work performed on travel at the request of the project team would be additional work on an hourly basis.

Phase 1) fixed cost = $9,680 (based on estimated 88 hours @ US$110)
Payment would be due on receipt of deliverables in final report.

Phase 2) fixed cost = $12,100 (based on estimated 110 hours @ US$110)
Payment would be due on receipt of deliverables in final report.

Phase 3) fixed cost = $12,100 (based on estimated 110 hours @ US$110)
Payment would be due on receipt of deliverables in final report.

TOTAL US $ 33,800 (= CDN $50,000 at 1.48 conversion)

Original signed by

Tony Warwick

TW Consulting

B.2 CXRO zone plates for the proposed scanning transmission X-ray microscope at the Canadian Light Source.

Materials Science Division
Centre for X-ray Optics

February 2, 20000

Dr. Adam Hitchcock
BIMR,
McMaster University
Hamilton, ON L8S 4M1
CANADA

Dear Adam:

This letter is to confirm the willingness of CXRO at LBNL to provide zone plates for the proposed scanning transmission X-ray microscope at the Canadian Light Source. I understand from you that the microscope will be installed in mid-2003 which is the time frame at which these zone plates would be provided. A small number (2-3) could be provided as a matter of collegiality. If a larger number are required, negotiations as to financial reimbursement would be undertaken at the appropriate time.

I wish you all the best with the CLS spectromicroscopy project.

Sincerely,

Orginal Signed by
David Attwood
Director, CXRO

Cls-SM-cxro.doc

Ernest Orlando Lawrence Berkeley National Laboratory
One Cyclotron Road | Berkeley, California 94720 | Tel: 510.486.4985 | Fax: 510.486.4550

McMaster University
Institute of Materials Research
1280 Main Street West
Hamilton, Ontario Canada L8S 4M1

Attn: Prof. Adam Hitchcock.

Complete PEEM Spectromicroscopy System, comprising:

Item 1: MULTIPROBE S (Order Code B000663)

Multi-technique surface analysis UHV system with a rigid bench frame suitable for high resolution SPM work and an analysis chamber for multi-technique surface analysis, such as SPM, XPS, UPS, ISS, LEED, AES, and sample sputtering, heating or thin film evaporation. (Upgrade with a separate preparation chamber as a retrofit is possible.) The analysis chamber provides a sample manipulator with a resistive sample heater and the provision for direct heating. The chamber is pumped by a combination ion getter, titanium sublimation and turbo molecular pumps. A Fast Entry Chamber with a magnetically coupled transporter facilitates fast sample/tip introduction into the analysis chamber. The UHV system is fully bakeable and provides pressure measurement, bakeout cover, heaters, programmable bakeout control and vacuum interlocks.

consisting of:

Multi-technique analysis chamber (stainless steel) (Order Code B000676)
Analysis chamber for multi-technique surface analysis made from non-magnetic stainless steel, vacuum annealed, including blank flanges, viewports and venting valve.

UHV precision manipulator (Order Code B000679)
UHV precision sample manipulator with rotary drive and a sample holder equipped with a resistive heater and provision for direct heating, including heating power supply and thermocouple cables. A thermocouple type K is fitted to the sample heater. Maximum temperature at the sample: 900 C. Rotation: 360 . X/Y-motion: ±8 mm. Z-motion: 150 mm. Base flange: NW 100 CF (6" OD)

Titanium sublimation pump with control unit (Order Code B000654)
Titanium sublimation pump head with 3 filaments, mounted to a NW 40 CF (2 ¾" OD) flange, including controller and 6 m bakeable cable.

Ion getter pump 230l/s with control unit (Order Code B000688)
Ion getter pump with a nominal pumping speed of 230 l/s and an inlet flange NW 150 CF (8" OD), including controller and 6 m bakeable cable.

Turbomolecular pumping system 240l/s with control unit (Order Code B000689)
Turbomolecular pump with a nominal pumping speed of 240 l/s and an inlet flange NW 100 CF (6" OD), including venting valve, pneumatically operated gate valve NW 100 CF (6" OD), 3 m³/h rotary pump with pumping line to turbo pump, controller and 6 m cable.

Pressure measurement consisting of ion and pirani gauges, control unit (Order Code B000653)
Pressure measurement controller with ion gauge head mounted on a NW 40 CF (2 ¾" OD) flange with a thoriated iridium twin filament, including 3 m bakeable cable, and pirani gauge on a DN 16 KF flange including 3 m cable.

Sample introduction system with magnetically coupled transporter (Order Code B000690)
Fast entry chamber (FEC) with magnetically coupled transporter and transfer head, gate valve NW 40 CF (2 3/4" OD) for isolation from the analysis chamber, pumped via a by-pass pumping line by main turbo pump.

System bench with bakeout oven and heater (Order Code B000691)
Rigid MULTIPROBE system bench for high resolution SPM work, including bakeout cover and heater.

Instrumentation rack(s) with mains, bakeout, vacuum interlock control (Order Code B000693)
19" instrumentation rack housing all control electronics including mains/bakeout/vacuum interlock control.

Complete assembly of the system, electrical wiring and testing (Order Code B000694)

Installation & acceptance test acc. to document 'acceptance criteria' (Order Code B000696)

Item 2: Upgrade analysis chamber with PEEM extension (Order Code B000665)
Replacement of B000676 and B000679.
consisting of:

Multi-technique analysis chamber w. PEEM extension (stainless steel) (Order Code B000678)
Analysis chamber for multi-technique surface analysis with extension for IS-PEEM made from non-magnetic stainless steel, vacuum annealed, including blank flanges, viewports and venting valve.

UHV precision manipulator for chamber with PEEM extension (Order Code B000683)
UHV precision sample manipulator with rotary drive and a sample holder, equipped with a resistive heater and provision for direct heating including heating power supply and thermocouple cables. A thermocouple type K is fitted to the sample heater. Maximum temperature at the sample: 900 C. Rotation: 360 . X/Y-motion: ±8 mm. Z-motion: 250 mm. Base flange: NW 100 CF (6" O.D.)

Item 3: FOCUS IS-PEEM HR, high resolution photoemission electron microscope (Order Code B000603)
Photoemission Electron Microscope with integrated sample stage, for real-time imaging of flat surfaces using various contrast mechanisms with a field of view from 10 µm to 600 µm, positionable within an area of 5 x 5 mm, guaranteed resolution better than 40 nm, including contrast aperture and stigmator, the sample stage is prepared for in situ sample heating up to 2300 K, a mu-metal chamber is recommended.
consisting of:

PEEM column with integrated sample stage (Order Code B000336)
Photoemission Electron Microscope including an electrostatic tetrode objective lens and two projectives for a field of view of 10 µm to 600 µm, a channelplate, a luminescence screen (YAG, yellow) and a mu-metal shield. The piezomotor driven sample stage is integrated into the objective lens and can be adjusted by ±2.5 mm in x and y direction. It is heatable up to 2300 K with electron bombardment heating (power supply optional). Mounting flange is NW 100 CF (6" OD). Flange to sample distance to be specified with order: 120 to 254 mm (10").

PEEM contrast apertures (Order Code B000340)
In-situ selectable apertures for adjustment of resolution, contrast and intensity, integrated into the PEEM column, selected and adjusted by means of a piezo drive.

Deflector/stigmator (Order Code B000342)
Octopole integrated into the PEEM column for deflection of a PEEM image and correction of astigmatism for resolution improvement.

PEEM control unit for high resolution with sample stage control (Order Code B000359)
High stability 15 kV power supply for PEEM column with contrast apertures and deflector/stigmator, including electronic board for operation of piezo drive and cables.

Item 4: Variable iris aperture for PEEM (Order Code B000339)
Continuously variable iris aperture with rotary drive for adjustable field of view, background reduction and micro spot analysis.

Item 5: Slow scan camera for PEEM (Order Code B000346)
Slow scan camera with adaptation facility to the screen flange including integrated frame grabber and digital image processing software DIGBI for use with Windows 3.11 or higher.

Item 6: High pressure mercury UV source for PEEM (Order Code B000347)
UV excitation source 4.9 eV, 100 W, including power supply. Viewport to sample distance to be stated on order.

Item 7: Heat filter for high pressure mercury UV source (Order Code B000349)

Item 8: Suprasil viewport NW 35 CF (Order Code B000351)
Viewport to use with deuterium or mercury UV source.

Item 9: Sample plate for IS-PEEM, molybdenum (Order Code B000355)
Molybdenum sample plate for sample diameter between 5 and 11 mm.

Item 10: HIS 13 H, high intensity, high power VUV source package (Order Code B000387)
UV source for photon flux of more than 10(16) photons/s sr with integral port aligner and ports for 2-stage differential pumping, water cooled, flange to sample distance 203 mm, including power supply for up to 300 mA discharge current and pressure measurement.
consisting of:

High intensity VUV source with port aligner (Order Code B000362)
Source mounted on a NW 35 CF (2 ¾" OD) flange.

Capillary, inner diameter 1.7 mm (Order Code B000363)
Capillary for insertion depth 138 mm, working distance 65 mm, for a flux of up to 6x 10^{12} photons/sec using He I.

NG HIS 13 power supply, 300 mA (Order Code B000369)
Power supply for up to 300 mA discharge current, including cable, pressure meter and pirani vacuum gauge.

Total Price for Items 1 – 10 = $ 298,540.00

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Options

Item 11: PEEM IEF, Imaging Energy Filter

Additional $ to be advised

Item 12: PEEM Analyzer, for microspot spectroscopy

Additional $ to be advised

Item 13: HIS 13 P2, two stage differential pumping system (Order Code B000376)
Two stage differential pumping kit with rotary pump and turbo pump.

Additional $ 18,790.00

Item 14: LEED/AES package SPEC 4 / 203 / 45 mm retractable (Order Code B001435)
consisting of:

4-grid SPECTALEED, 203 mm (Order Code B000030)
Rearview LEED optics (for observation of diffraction pattern from rear and front) with phosphor-coated glass screen, miniature integral electron gun for maximum viewing angle, and non-magnetic grids made of gold plated molybdenum. The 4-grid SPECTALEED can optionally be operated as a retarding field analyser for Auger electron spectroscopy with an energy resolution better 0.5%.

Internal linear movement facility for SPECTALEED, 45 mm travel (Order Code B000014)
Minimum flange to sample distance of 203 mm is required.

Power supply for LEED optics NG LEED with lock-in amplifier/oscillator (Order Code B000024)
The power supply has active HV modules for all elements of the electron gun for a beam energy ranging from 0 eV to 3.5 keV, emission regulated high stability filament supply for thoriated tungsten and LaB_6 filaments, beam current monitor (20 nA to 100 µA), screen high voltage supply up to 7 kV. Retard voltage and beam energy can be controlled externally for Auger electron spectroscopy (0 - 2 keV) or dynamic LEED. Monitor outputs for beam current and beam energy are provided. The lock-in amplifier/oscillator cassette is built in.

DATAuger spectrometer control and data handling kit (Order Code B000021)
Spectrometer control software for CMA and LEED/Auger, including oscilloscope mode for set-up, experiment control and data analysis software. The PCI board includes 16 bit DAC for energy sweep, analogue and fast pulse counting inputs, software control of precision oscillator and lock-in amplifier with drivers for Windows NT (requires one PCI slot).

DATAuger interface box (Order Code B000205)
Computer interface and cables to connect the DATAuger board to the LIO lock-in amplifier / oscillator cassette.

Cable for operation of screen of LEED using NG LEED (Order Code B000022)

Matching unit for AES with 4-grid SPECTALEED (Order Code B000023)
The matching unit comprises modulator and preamplifier.

Additional $ 31,350.00

<u>Item 15</u>: EFM 3-EVC 100 (Order Code B000467)
Evaporator package for sub-monolayer and multi-layer thin film growth, with shutter and flux monitor, for deposition areas from Ø 5 to Ø 20 mm.
consisting of:

EFM 3 evaporator with Mo crucible (Order Code B000459)
Evaporator for sub-monolayer and multi-layer thin film growth, for deposition areas from 5 to 20 mm diameterm

EVC 100 power supply for EFM, 100 W (Order Code B000450)
Power supply for EFM 3, EFM 4 and EFM T3, maximum power output 100 W, including flux monitor electronics.

Cable for EFM 3 and EFM 4 (Order Code B000452)
Cable for connection of EVC 100 or EVC 300with EFM 3, EFM 4 or EFM VT, 5 m.

Additional $ 15,910.00

Prices: F. O. B. Point of Entry.

Delivery: 6 - 7 months from point of complete technical clarification.

Terms: 30% with order
65% on delivery
5% on acceptance or within 60 days if acceptance delayed due to circumstances outside of OMICRON's control (site not prepared, personnel not available, etc.) All invoices are *strict* net 15 days. ALL NOTED TERMS TO BE EMBODIED IN ANY PURCHASE ORDER AND ADHERED TO FOR DISCOUNTS TO APPLY.

Validity: 60 days.

Warranty: Standard one year parts and labor, assuming any defective component is returned freight/insurance prepaid to Pittsburgh, PA. Consumable items and problems caused by operation outside of standard operating conditions or misuse are not covered. Defective parts to be repaired or replaced at OMICRON's discretion.

Notes: *a) Installation and training in the use of the quoted system and analytical instrumentation is* **included** *in the quoted price.*

OMICRON ASSOCIATES

Original signed by

FREDERIC C. HENN
Technical Sales

Staib – quotation (page 1 of 7)

Photo Emission Electron Microscope
PEEM

PEEM 350 Family

PEEM Now Available as a Modular System

The PEEM - 350 family is based on an inexpensive basic system that can be upgraded to a top PEEM system by adding specific modules. All systems are designed to fit a standard CF 100 or larger flange, and have a standard flange to sample distance of 254mm. Other distances are available on request. Please contact our office for prices.

Model	Description	Magnification	Resolution	Field of View
PEEM-350-00	Basic System consisting of: - 2 lenses (1 objective, 1 projective) - 40mm diameter detector - magnetic shielding	x200 to x600	200nm to 250nm	200µm to 70µm
PEEM-350-10	Basic system **plus**: - 1 projective lens - 40mm Chevron Channelplate detector	x200 to x2000	100nm	200µm to 20µm
PEEM-350-20	Basic System **plus**: - 1 projective lens - 1 stigmator system - variable resolution/contrast aperture - 40mm Chevron Channelplate detector	x200 to x8000	to 40nm	200µm to 20µm

ACCESSORIES and OPTIONS

PM-350-Hg	Ultra Violet Illumination system, with lens system adapted to user's vacuum chamber.
PM-350-EF	Imaging Energy Filter
PM-350-EA	Micro-spot Filter for Spectrometry
PM-350-SH	Built-in sample holder attached to the microscope. Sample size limited to 10x10mm, requires a transfer system.
PM-350-MS	Additional magnetic shielding around the sample. Openings through the shielding to be defined with the user.
PV-350	Data system to control the microscope and to acquire data.

Sample holders, camera upgrades, UHV chambers, and additional options available on request.

STAIB INSTRUMENTS, INC. Newport News, Virginia 23606 Phone: 757-873-0099
staib-us@staib-instruments.com Fax: 757-873-0130

Photo Emission Electron Microscope
PEEM

PEEM 350 - 20
System Description

Mu Metal Shield	The system is equipped with a **mu-metal** shield to minimize the influence of present AC stray fields, which could keep the instrument from achieving the resolution limit, as well as DC fields, which could cause an off-axis imaging symmetry and therefore lead to image distortions and contrast asymmetries. The shielding can be upgraded to cover the sample region. Picture 1 gives a full view of the instrument with mounted mu-metal shield.
Mounting flange and design length	The system is mounted on a 6" O.D. flange to fit most vacuum system. The standard insertion length of the device is 10". Modifications to support special chambers and retraction/ insertion devices are available.
Sample, sample holder, and manipulator	The system has proven to reach a lateral resolution of **below 80 nm with standard manipulator** systems, with lateral displacement in the X and Y direction of 25mm and in the Z direction of 50 mm. There is **no limitation of the sample size** introduced by the design of the PEEM. A variety of sample carriers can be used to support effortless sample transfer throughout the vacuum system. For a lateral resolution close to the resolution limit, a high stability UHV compatible manipulator known from high resolution SEM work should be used.
Objective lens	The objective lens is a modified Bruche/ Johannson type tetrode emission lens, designed to support imaging with aberrations well below the aberration contribution of the homogeneous accelerating field between the sample and the first lens electrode.
Objective and contrast aperture	The back focal plane **objective lens aperture** limits the acceptance angle for the released photoelectrons and, therefore, the aberration contribution of the accelerating field. The limitation of acceptance angle in particular is responsible for topography **contrast enhancement** with asymmetric emission at the site of, for example, edges, whiskers, and steps.

STAIB INSTRUMENTS, INC. Newport News, Virginia 23612 staib-us@staib-instruments.com Phone: 757-873-0099 Fax: 757-873-0130

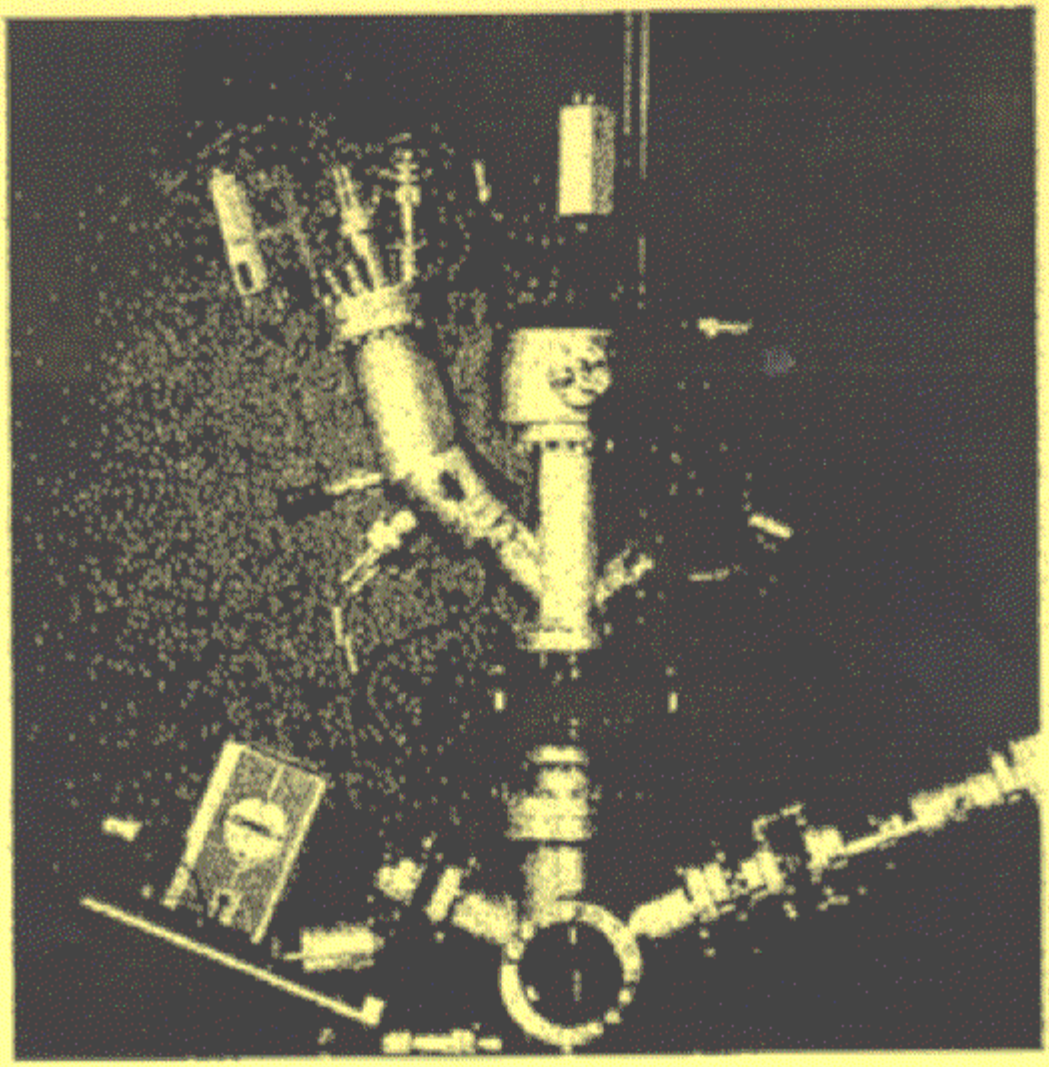
Photo Emission Electron Microscope
PEEM

PEEM 350 - 20
System Description

Stigmator stage	An octopole **stigmator** stage for higher order astigmatism correction with beam alignment capability reduces the astigmatism in the image. The astigmatism is mainly caused by lack in circular symmetry and a limited size of the sample and has to be corrected to get to the **resolution limit of 40 nm**. The beam alignment capability furthermore provides additional contrast selection features.
Zoom lens	The zoom lens accounts for the large magnification range of the instrument, between approx. **100 and 4000 times,** and a distortion free **field of view,** between **10µm and 400µm**. The high magnification range also ensures that the final resolution is not limited by the structure size of the micro channel plates and the multichannel activation of Chevron type assemblies.
Projective lenses	The projective lenses are responsible for the distortion free magnification of the intermediate images.
Exit lens	The exit lens brings the final image of **40 nm in diameter** onto the MCP assembly, forms the field of view, and reduces the contribution of stray electrons to the image background.
Chevron channelplate assembly	The Chevron type microchannelplate assembly is mounted on a CF 6" O.D. flange that gives comfortable access to the channelplates. It comes with an **active area of 40 mm** in diameter and gives a **field of view between 400 µm and 10 µm**, depending on the magnification setting, a leading standard in the industry. The fiberoptic screen collects the light from the phosphorus layer and brings it, unaffected by any artifacts of total reflection as present in standard lead glass designs, to the outside surface of the assembly for further imaging and data collection. Picture 2 gives a view of the detector assembly mounted to the instrument.

STAIB INSTRUMENTS, INC. Newport News, Virginia 23612 stalb-us@staib-instruments.com Phone: 757-873-0099 Fax: 757-873-0130

Photo Emission Electron Microscope
PEEM



PEEM 350-20 with Imaging Energy Filter and Micro-spot Filter
and UV light source

STAIB INSTRUMENTS, INC. Newport News, Virginia 23612
staib-us@staib-instruments.com
Phone: 757-873-0099
Fax: 757-873-0130

Photo Emission Electron Microscope PEEM

PEEM 350

Energy Resolved Photoelectron Microscopy

The Photoelectron Emission Microscope (PEEM) is a powerful instrument for chemical investigation of surfaces at a nanometer scale. The instrument gives magnified images obtained by direct imaging, without the need of scanning, at a very high speed. Most of the observed chemical features are not visible using a regular SEM. The spatial resolution for chemical contrast is in the range of 40 nm. The PEEM is also able to image topographical contrast, for instance due to steps. As the instrument uses an immersion objective, the sample surface should be free of topographic structures to give meaningful data at high spatial resolution.

The new PEEM 350 family developed by Staib Instruments now incorporates energy filtering in addition to the imaging capability. The photoelectrons are energy analyzed before reaching the image detector and the image is energy filtered. The energy distribution is measured by varying the analyzer energy. This corresponds to Ultraviolet Photoelectron Spectroscopy (UPS) at high spatial resolution, or UPS-PEEM. As the spectrometer resolution is excellent, all details of the photoelectron spectra are recorded. The most striking features of this new system are the exact determination of energy levels, such as Fermi and vacuum levels, and of the major lines present in the spectrum. The spectra shown in figure 1 are examples obtained from various materials (C, Au, Ag, Si, SiC) and demonstrate the elemental sensitivity of the method. The contamination layers and sorbed layers show very sharp lines, very sensitive to any chemical modification of the surface. Some lines have a FWHM as low as 200 meV. Argon ion cleaning shows drastic changes of the spectra, a proof of the ongoing modification of the chemical environment in the near surface region.

The powerful data acquisition system WinSpectro32, operating as a true 32 bit application, allows state of the art multi-tasking in image and data acquisition. In addition, data can be exported to user's favorite data processing software. Acquisition, processing and output are simultaneously performed.

Figure 2 shows an example of peak deconvolution, breaking the experimental spectrum in three single Gaussian peaks.

STAIB INSTRUMENTS, INC. Newport News, Virginia 23612 staib-us@staib-instruments.com Phone: 757-873-0099 Fax: 757-873-0130

STAIB INSTRUMENTS, Inc.
PO Box 120484
Newport News, VA 23612
Telephone: 757-873-0099 Fax: 757-873-0130
E-mail: staib-us@staib-instruments.com
www.staib-instruments.com

Dr. Adam Hitchcock
McMaster University

Quotation # 8212 Date: November 11, 1999

We are glad to offer you the following quotation for our **PhotoEmission Electron Microscope (PEEM).**

Pos.	Qty.	Description	Price (US$)
1.0		**PEEM basic system consisting of:**	
	1	Microscope	
	1	Detector	
	2	Regulated power supplies for microscope and detector with cables.	
2.0		**Upgrade 1**	
	1	Extension of microscope and detection	
	1	Regulated power supply upgrade	
3.0		**Upgrade 2**	
	1	Extension of microscope and detection	
	1	Regulated power supplies with cables	
	1	Hg UV lamp kit	
4.0		**PEEM energy analysis**	
		Imaging Energy Filter	
5.0		**PEEM control and analysis** – PC-control with image acquisition	
	1	PEEM vison, includes standard CCD camera (PMCCD-00)	
	1	PEEM computer control	
	1	Computer system	
6.0		**UHV-chamber, with**	
		• Ion pump, titanium sublimation pump	
		• sample holder + heating 600° C + cooling	
		• Ion gun for cleaning	
		• CCD camera for sample observation	
		• Vacuum gauge	
		• Bakeout system	
		• Adjustable beam line access and orientation	
7.0		**Installation and Training – on site**	
		Total for items 1.0 through 7.0	**$236,000.00**

Page 2
Quotation: #8212

PEEM 350 Work Station

Pos.	Qty.	Description		Price (US$)

Options for PEEM System:

8.0 **Optional integrated introduction chamber, with**
- Load lock and spare flanges
- Turbo molecular pump
- Gate valves
- Transfer system

39,000.00

9.0. **Micro-spot analysis filter (MAF) for spectrometry** 49,900.00
Adjustable analyzer aperture

Multichannel detection for MAF (4 channels) including software 16,000.00

10.0 **PEEM CCD-Camera Upgrades**

1	VarioCam + VCR	PMCCD-01	10,000.00
1	SensiCam	PMCCD-02	16,700.00

11.0 **Add-on LEED system**
4 grid optics LEED system, electronics, cables. 24,900.00

Notes:
- System extension with additional chamber will be possible.
- The complete system will be delivered including a table support unit.

TERMS AND CONDITIONS

1.) Assistance by phone and fax is provided.
2.) All prices F.O.B. destination.
3.) Delivery: 150 days or less after receipt of order.
4.) Warranty: 1 year for all parts, except consumable parts.
5.) Taxes are not included and are not the responsibility of Staib Instruments, Inc.
6.) Conditions of payment: 30 days after delivery.
7.) Validity of the quotation: 2 months.
8.) STAIB INSTRUMENTE GmbH and STAIB INSTRUMENTS, INC. are expressly NOT liable for consequential damages arising from the use or non-use of the equipment.

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Lillyan Dylla
Manager, US Marketing

CLS Spectromicroscopy Proposal – researcher profile

Name: **Shireen ADENWALLA**

Affiliation: University of Nebraska-Lincoln

Department: Physics and Astronomy

Position: Professor

Research interests: magnetic thin films, neutron scattering

Current operating funding:

NSERC/NSF	US 375,000 (and more)
Industrial	0
Contract	0

Current Resarch Group:

Postdoc / associates	1
PhD students	0
M.Sc. students	1

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **G. Michael BANCROFT**

Affiliation: CLS, University of Western Ontario

Department: Chemistry

Position: Professor, CLS Director

Research interests: tribology, surface and thin film science

Current operating funding:

NSERC/NSF	89,000
Industrial	293,000
Contract	262,000

Current Resarch Group:

Postdoc / associates	5
PhD students	0
M.Sc. students	2

Anticipated fraction of total research effort at this beamline: 20%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	20

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 8,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **JAMES R. BROWN**

Affiliation: Materials Technology Laboratory, (MTL)

Department: Natural Resources Canada; Minerals Technology Branch, (MTB); Minerals & Metals Sector (MMS) "CANMET"

Position: Senior Research Scientist (SE-RES-4)
(surface spectroscopist)

Research interests: Fundamental and applied research in Materials, Mining and Energy Sectors. Also representing interests of NRCan generally via NRCan Synch. Light steering Committee

Current MTL funding:

Internal	>$10M
NSERC/NSF	n/a
Industrial (shared/cost recovery)	>$3M /yr
Contract	n/a

Current MTL Analytical Research Staff: MTL (14)
Postdoc / associates: 5 PhD students: n/a M.Sc. students: n/a

Anticipated fraction of total research effort at this beamline: (n/a)

Requested research time (# 8 hour shifts/year):
STXM: 30 PEEM: 10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat. Estimated commitment based on the requested research time: $ 16,000

Notes:
NRCan has the largest R &T program of all Federal Government Ministries. NRCan is the lead Ministry coordinating CLS activities within the federal government. The Energy Sector (ES), Minerals & Metals Sector (MMS), Earth Sciences Sector (ESS) and Forestry Sector (CFS) of NRCan have extensive in-house analytical laboratories and access to external facilities via user agreements. R &T resources at NRCan exceed 500 scientific researchers and technicians with a budget >$100M annually. The Materials Technology Laboratory (MTL) has a staff of 85 and a budget >$10M annually.

CLS Spectromicroscopy Proposal – researcher profile

Name: **Ronald G. CAVELL**

Affiliation: University of Alberta

Department: Chemistry

Position: Professorr

Research interests: inorganic chemistry

Current operating funding:

NSERC/NSF	47,355
Industrial	108,000
Other (shared)	150,000

Current Resarch Group:

Postdoc / associates	2
PhD students	0
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 20%

Requested research time (# 8 hour shifts/year):

STXM:	10
PEEM:	30

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 16,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Bernard DOUBIN**

Affiliation: University of Nebraska-Lincoln

Department: Physics and Astronomy

Position: Assistant Professor

Research interests: magnetoelectronics, nanostructured magnetic materials, mesoscopic transport effects in magnetic systems

Current operating funding:

NSERC/NSF	US 55,000/year
Industrial	US 20,000
Contract	0

Current Resarch Group:

Postdoc / associates	1
PhD students	1
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Peter A. DOWBEN**

Affiliation: University of Nebraska-Lincoln

Department: Physics and Astronomy

Position: Professor

Research interests: thin film and surface electronic and magnetic structure

Current operating funding:

NSERC/NSF	US 85,000/yr
Industrial	0
Contract	US 110,000/yr

Current Resarch Group:

Postdoc / associates	1
PhD students	5
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 10% (to be taken with Adenwalla, Robertson and Doudin)

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **John R. DUTCHER**

Affiliation: University of Guelph

Department: Physics

Position: Professor

Research interests: polymer physics; morphology and dynamics of thin films; soft materials

Current operating funding:

NSERC/NSF	38,462
PREA	20,000
Industrial	10,000
Contract	----

Current Resarch Group:

Postdoc / associates	1
PhD students	1
M.Sc. students	2
Undergraduate students	2

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	20
PEEM:	

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 8,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Joseph A. GARDELLA, Jr.**

Affiliation: State University of New York at Buffalo

Department: Chemistry

Position: Professor and Associate Dean

Research interests: biomaterials, surface science, analytical/environmental chemistry, polymer science

Current operating funding:

NSERC/NSF	US	285,000
Office of Naval Res	US	200,000
Industrial	US	53,000
Contract	US	19,000

Current Resarch Group:

Postdoc / associates	1
PhD students	9
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	10
PEEM:	20

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 12,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Daniel GUAY**

Affiliation: Institut National de Recherche Scientifique

Department: Energie et Materiaux

Position: Professeur (Université de Québec)

Research interests: electrochemistry, surface science

Current operating funding:

NSERC/NSF	160,000
Industrial	25,000
Contract	-------

Current Resarch Group:

Postdoc / associates	2
PhD students	2
M.Sc. students	3

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Adam P. HITCHCOCK**

Affiliation: McMaster University

Department: Chemistry, BIMR

Position: Professor

Research interests: synchrotron instrumentation, polymer structure, biomaterials

Current operating funding

NSERC/NSF	124,000
Industrial	80,000
Contract	0

Current Resarch Group

Postdoc / associates	2
PhD students	3
M.Sc. students	1

Anticipated fraction of total research effort at this beamline: 50%

Requested research time (# 8 hour shifts/year):

STXM:	50
PEEM:	20

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $28,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Masoud KASRAI**

Affiliation: University of Western Ontario

Department: Chemistry

Position: Research Associate

Research interests: tribology, surface and thin film science

Current operating funding:

NSERC/NSF	0
Industrial	0
Contract	0

Current Resarch Group:

Postdoc / associates	0
PhD students	0
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 20%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	20

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 8,000

Note: *Dr. Kasrai will execute the PEEM studies on tribological samples*

CLS Spectromicroscopy Proposal – researcher profile

Name: **Ronald R. MARTIN**

Affiliation: University of Western Ontario

Department: Chemistry

Position: Associate Professor

Research interests: environmental and biological analysis

Current operating funding:

NSERC/NSF	35,500
Industrial	0
Contract	0

Current Resarch Group:

Postdoc / associates	0
PhD students	1
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 20%

Requested research time (# 8 hour shifts/year):

STXM:	10
PEEM:	0

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Peter R. NORTON**

Affiliation: University of Western Ontario

Department: Chemistry

Position: Professor

Research interests: tribology, interface and surface science

Current operating funding: *(approximate)*

NSERC/NSF	<100,000>
Industrial	<200,000>
Contract	<200,000>

Current Resarch Group:

Postdoc / associates	4
PhD students	10
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	20

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 8,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Brian W. ROBERTSON**

Affiliation: University of Nebraska-Lincoln

Department: Mechanical Engineering

Position: Associate Professor

Research interests: novel semiconductor and nano-magnetic materials and devices; near-atomic scale structure and property characterization

Current operating funding:

NSERC/NSF	US 134,000 /yr
Industrial	
Contract	

Current Resarch Group:

Postdoc / associates	one quarter
PhD students	3
M.Sc. students	1 full plus 2 part-time

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Denis ROY**

Affiliation: Université Laval

Department: Physique

Position: Profeseur titulaire

Research interests: electronic structure, surface science

Current operating funding:

NSERC/NSF	62,500 (NSERC & FCAR)
Industrial	0
Contract	0

Current Resarch Group:

Postdoc / associates	1
PhD students	2
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	0
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $ 4,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Harald STÖVER**

Affiliation: McMaster University

Department: Chemistry

Position: Professor (3M NSERC Industrial chair)

Research interests: polymer chemistry

Current operating funding:

NSERC/NSF	44,000
Industrial	330,000
Contract/other	400,000 (CFI, etc)

Current Resarch Group:

Postdoc / associates	2
PhD students	4
M.Sc. students	2

Anticipated fraction of total research effort at this beamline: 10%

Requested research time (# 8 hour shifts/year):

STXM:	20
PEEM:	0

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $8,000

CLS Spectromicroscopy Proposal – researcher profile

Name: **Tolek TYLISZCZAK**

Affiliation: McMaster University

Department: Chemistry, BIMR

Position: Research Associate

Research interests: synchrotron instrumentation, polymer structure, biomaterials

Current operating funding

NSERC/NSF	0
Industrial	0
Contract	0

Current Resarch Group

Postdoc / associates	0
PhD students	0
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 80%

Requested research time (# 8 hour shifts/year):

STXM:	50
PEEM:	20

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $28,000

Note: *Dr. Tyliszczak will be heavily involved with design, construction, commissioning and operation of this beamline and endstation equipment.*

CLS Spectromicroscopy Proposal – researcher profile

Name: **Stephen G. URQUHART**

Affiliation: University of Saskatchewan

Department: Chemistry

Position: Assistant professor

Research interests: polymer science, synchrotron spectromicroscopy

Current operating funding:

NSERC/NSF	65,000 (applied)
Industrial	0
Contract	0

Current Resarch Group:

Postdoc / associates	0
PhD students	0
M.Sc. students	0

Anticipated fraction of total research effort at this beamline: 50%

Requested research time (# 8 hour shifts/year):

STXM:	60
PEEM:	10

Participation in this application is a commitment to provide operating funding proportionate to the number of beamline team (BT) shifts allocated internally. At the estimated annual BT operating budget of $120,000 and an allocation of 300 BT shifts, this amounts to $400 per shift - $50 / hour. Income from fee-for-service activities carried out by the BT will reduce this somewhat.

Commitment based on the requested research time: $28,000