X-ray Spectromicroscopy Beamline 10ID-1

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Beamline Overview

Status	Operational
Source	75 mm EPU
Stations	STXM, X-PEEM
Spectral range	100-2500 eV
Flux	STXM: 10^8 ph/s/0.1%BW /0.1A in focused spot (~30 nm)
Spatial Resolution	X-PEEM:10^{12} ph/sec/0.1%BW/0.1A
Resolving Power (E/ΔE)	STXM: 30 nm; X-PEEM: 25 nm
Spot size	Nominal 3000, can reach >10 000

Introduction

2007 marks the first year of scheduled general user operation of the Spectromicroscopy (SM) beam line. Commissioning, completed in June 2007, provided reference data that show that the beamline and microscopes [1,2] are very competitive on an international scale. The research by beam team members and general users includes projects in all of the main themes of CLS research: life sciences, materials and molecular sciences and environmental science. Since July 2007 the beamline and microscopes have been operated at full capacity with 30% of time for general users, 30% for beam team research, 10% for fee-for-service applications and the remainder for staff scientist research, maintenance and further development. Table 2 lists the proposals from allocated beam time, beam team, and staff scientists. There are first publications [3,4] and there are reports on many of these projects elsewhere in this report [5-16].

The year was also marked by a continuous growth of SM technical capabilities, including success at picosecond regime magnetization dynamics with STXM and extending the photon energy range to 2600 eV, thus enabling scientific studies at the P and S *1s* edge. In the following the main achievements are summarized.

Science

Three main areas of beamline development continue to push SM technical limits and define the research frontiers. Advanced polarization capability has been explored in liquid crystal (LC) research [3, 4], where X-ray linear dichroic (XLD) measurements combined with the high STXM spatial resolution permit the LC director (a vector along which molecules are aligned) to be mapped with 5° (angle) and 5% (magnitude) precision at 100 nm spatial resolution. XLD was also used to study the orientation properties of natural fibers, both in dragline spider silk [5] and in flax [6]. Measurements were made on both dry and humidified samples of several types of biopolymers, which exemplifies a theme common to a number to research projects at CLS-SM, namely investigation of the effect of various control parameters on the properties of materials, with either external modification or *in situ* modification of the sample. Modification of a liquid cell to allow active *in situ* control of the humidity of samples is a development target for 2008. High flux and spectral purity (which has been improved in the C 1s region with installation of a Ti filter) permitted extension of XLD measurements to "dilute systems," including ions and nanoparticles. The first quantitative measurements of the dichroism of individual multi wall carbon nanotubes (MWCNT), which have a diameter of a few 10's of nanometers were carried out [7,18]. The magnitude of the dichroism of the C 1s $\rightarrow \pi^*$ peak (285 eV) was found to be highest in MWCNTs with the fewest defects, suggesting that imaging the dichroic signal along individual nanostructures such as MWCNTs has potential for quantitative mapping of structural defects.

Time resolved measurements are another area where there have been world class developments at CLS-SM in 2007. There are many phenomena where smaller spatial scale is coupled to faster and more energy efficient response, and magnetic information storage is an excellent example. The groups of Hermann Stoll (Stuttgart) and Bartel Van Waeyenberge (Gent), in collaboration with beamline (Kaznatcheev, Bertwistle) and other CLS personnel (Johannes M. Vogt), and Tolek Tyliszczak (ALS) successfully used the STXM for time resolved X-ray magnetic circular dichroism (XMCD) measurements of magnetic vortex dynamics in micron sized permalloy samples at 100 ps time resolution [8]. This required commissioning of the inclined sample stage to allow out of plane sample scanning in order to be sensitive to the in-plane magnetization. Given this is one of the most technically challenging of all synchrotron measurements (10^{-10} s in time, 10^{-8} m in space, and a signal that is a few percent of the total absorption), the ability to set up the necessary mechanical scanning, timing synchronization with ring signals, and sophisticated control and acquisition in a few days of set up is a tribute to the technical expertise of all involved.

Environmental science is a major theme [9-11]. The extended energy range of the SM beamline (250-2600 eV) allows the type of multi-element characterization of specimens that is essential to understand the functioning of complex environmental samples. Environmental research includes studies of: mechanisms of antimicrobial resistance in bacterial biofilms

[9], calcite nucleation promoted by the surface of cyanobacteria [10], and the chemistry of nitrogen in soils [11]. Measurements were made at the S *1s* edge (2470 eV) [4] even though such studies are quite challenging due to low intensity, and high sensitivity to beam motion and vibrations of optics due to the extremely grazing angles involved.

The CaPeRS PEEM was used by many groups, both on the SM line [12-14] as well as on beamlines 11ID-1 and 11ID-2 (VLS-PGM and SGM). It is functioning well and a new mode of contrast involving variations in the secondary electron emission spectrum [12] has been developed and christened with the acronym of SEEM. The STXM technical capability was extended in other aspects in 2007. A combination of full field imaging and soft X-ray coherent scattering was explored [15] which promises to further extend spatial resolution and sensitivity limits. *In situ* stages for both azimuthal and polar rotation were implemented and the first CLS-based tomographic measurements were made. While it will be complicated by timeouts for the REIXS beamline commissioning we still look forward to a very exciting and successful scientific program in 2008.

Acknowledgements

As it was the first year of user operation, KK specifically wish to acknowledge the role Chithra Karunakaran, Martin Obst and Uday Lanke played, as they indeed took a leading role in user training while keeping the beam line, STXM and PEEM working well. 2007 is a year of transformation for CLS-SM. As of January 2008, Dr. Kaznatcheev joined the NSLS II Project at Brookhaven National Laboratory, and the CLS gratefully acknowledges his significant contributions to the development of the SM beamline.

References

1. Kaznatcheev, K.V., Karunakaran, C., Lanke, U.D., Urquhart, S.G., Obst, M. and Hitchcock. A.P. 2007. Nuclear Instruments and Methods in Physics Research A. 582, 96-99.

2. Kaznacheyev, K.V., Karunakaran, C., He, F. Sigrist, M., Summers, T., Obst, M. and Hitchcock. A.P. 2007. Nuclear Instruments and Methods in Physics Research A. 582,103-106.

3. Kaznacheev, K.V. and Hegmann, T. 2007. Physical Chemistry Chemical Physics. 9, 1705–1712

4. K.V. Kaznatcheev, P. Dudin, O.D. Lavrentovich and A.P. Hitchcock. 2007.Physical Review E. 76, 061703-1 - 061703-14

5. Pézolet, M., et al. This report.

6. Kaznatcheev, K. V. et al., This report.

7. Najafi, E. et al. This report.

8. Waeyenberge, B.V. et al. This report.

9. Dynes, J. J. et al. This report.

10. Obst, M. et al. This report.

11. Lehmann, J. et al. This report.

12. Christensen, S. et al. This report.

13. Horny, P. et al. This report.

14. Kruse, P. et al. This report.

15. Chen, P. et al. This report.

16. Kaznatcheev, K.V. et al. This report.

17. Hanhan, S. et al. 2007.CLS Activity Report 2004-2006, 119-121.

Table 2: Beam team* and general user research projects at CLS-SM in 2007

PI	Proposal
A. Felten (Namur)	Scanning transmission x-ray microscopy of multiwall carbon nanotubes
A. Hitchcock (McMaster) (*)	Probing carbon nanotubes with soft X-ray absorption and linear dichroism
C. Karunakaran (CLSI)	Effect of bleaching on the composition and structure of flax fibers
P. Kennepohl (UBC)	Oxidative Mapping of Alzheimer's related amyloid plaque
P. Kruse (McMaster)	X-PEEM study of corrosion inhibition of steel by aniline trimer
J.R. Lawrence (NWRI) (*)	Mapping antimicrobial agents and their effects on bacterial biofilms
J. Lehmann (Cornell)	Physical protection or chemical recalci-trance in soil carbon sequestration
M. Obst (CLSI, McMaster) (*)	Mechanisms of calcite nucleation on the surface of cyanobacteria and within biofilms
M. Pézolet, (Laval Univ)	Mapping orientation in silk fibers by STXM
F. Rosei (INRS) (*)	Nanospectroscopy of Bioactive Titanium Surfaces
L. Tortora (Kent State)	Alignment and aggregation in chromonic liquid crystals: a study by STXM
S. Turgeon (U. Quebec)	Investigation of Plasma Deposited Fluoropolymer Coatings for Stent Applications
H. Stoll, B. van Waeyenberge	Ultra-fast vortex core switching in nano-scaled magnetic elements
H. Stöver (McMaster) (*)	Polymer Nanocomposites for cell encap-sulation and slow release
S.G. Urquhart (Saskatchewan) (*)	XPEEM, XPS, SEEM studies of phase seg-regation in Langmuir-Blodgett fiolms
J. Gardella (SUNY Buffalo) (*)	Spatial and chemical mapping of a biodegradable polymer thin films