Scanning photoemission microscopy on MAXIMUM reaches 0.1 micron resolution

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We present the first results from the upgraded version of the scanning photoemission spectromicroscope MAXIMUM, based on synchrotron undulator light and on a multilayer-coated Schwarzschild objective. The upgrade involved nearly all parts of the instrument, notably the beamline and the electron analysis system. Micro-images of Fresnel zone plates and of metal test patterns on semiconductor substrates reached a new record in lateral resolution, well beyond 0.1 micron. The first spectromicroscopy tests were also successfully performed on the new instrument, with analysis of f and d core levels in different systems.

The upgrade version of the MAXIMUM (multiple-application X-ray imaging undulator microscope) system has been recently tested in its photoemission spectromicroscopy mode of operation, and the results demonstrate that it has reached and surpassed its target performances. To the best of our knowledge, the present performances, better than 1000 Å lateral resolution and better than 400 meV energy resolution, are the best in the world for photoemission spectromicroscopy. No evidence of obstacles has been found against further improvements of the MAX-IMUM technology, except for the photon source brightness. This indicates that the performances will be significantly enhanced by the use of ultrabright synchrotron sources like ELETTRA in Trieste and the advanced light source (ALS) in Berkeley.

MAXIMUM is a collaboration program that involves several groups and institutions [1-3]. The core program is developed at the Center for X-Ray Lithography of the University of Wisconsin-Madison, using the storage ring Aladdin of the University's Synchrotron Radiation Center. Other partners are the Center for X-Ray Optics of the Lawrence Berkeley Laboratory, the Xerox Corporation, the University of Minnesota and the Ecole Polytechnique Fédérale de Lausanne. The photon source, a 30-period undulator, was originally developed by Stanford and Berkeley [4].

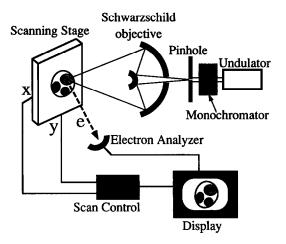
The main objective of the MAXIMUM program is to reach high lateral resolution in established synchrotron radiation spectroscopies. The tests have been primarily conducted in photoemission spectroscopy because this is a technique for which the need to achieve lateral resolution is most urgent. Photoemission has been for decades one of the leading experimental techniques in materials science, but with a very severe handicap: the need to average over sample areas of the order of 1 square millimeter, thereby losing all microscopic information [5].

In 1988, it became apparent that this severe limitations could be eliminated because of two significant technical advances [1]. First, the demonstrated reliable operation of undulators, that significantly increased the brightness of the synchrotron radiation sources. For example, after moving the Stanford–Berkeley undulator [4] to the storage ring Aladdin, its brightness exceeded by more than two orders of magnitude that of the ring's bending-magnet radiation.

Second, progress in multilayer coatings made it possible to enhance the reflectivity of surfaces – including non-plane surfaces – in the soft-X-ray spectral region. This opened, in particular, the possibility to use reflection devices like the Schwarzschild objective to focus this type of radiation [1-3].

These two elements of progress were brought together in the MAXIMUM system, whose artist's view is shown in fig. 1. MAXIMUM is a scanning instrument, that takes the radiation emitted by the Stanford-Berkeley undulator [4] on Aladdin, filters it with a monochromator, then focuses it onto the sample. This enables us [1-3] to perform different kinds of synchrotron radiation spectroscopies on a microscopic sample area; such spectroscopies include, for example, absorption, reflection and desorption techniques. In the photoemission mode, photoelectrons emitted by the small sample area are collected and analyzed by a double-pass cylindrical-mirror electron energy analyzer.

Besides taking spectra from a small sample area, we can also scan the sample position with



respect to the focused beam, and create two-dimensional micro-images. For example, we can scan while measuring the photoemission signal at a fixed photon energy, corresponding to the emission from a given core level of a given element in a given chemical status [1–3,5]. This produces micro-images of the lateral distribution of that element in that chemical status. Whereas other techniques exist that can perform microchemical analysis on the scale of MAXIMUM [6–9], no other technique can reach the energy resolution of photoelectron specromicroscopy in delivering fine information in the chemical status of elements.

The first stage of the MAXIMUM program adopted several technical compromises to fit a limited budget [1]. For example, the first monochromator was borrowed from the Synchrotron Radiation Center and not optimized for the undulator output. Severe problems were identified, for example those related to the roughness of the Schwarzschild lens surfaces. These problems notwithstanding, we were able to demonstrate good lateral resolution for total-yield photoelectron micro-images [1-3]. In the years 1989–1990, the lateral resolution of these images improved from a few microns to one-half micron [2]. Breaking the micron barrier opened the possibility to use the instrument for life-science experiments on neuron systems [3].

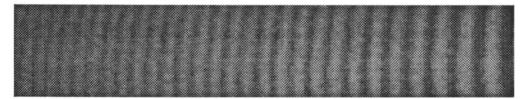


Fig. 2. Photoelectron micro-image of a portion of a Fresnel zone plate. From the smallest distinguishable zones we estimated a lateral resolution of approximately 900 Å.

In 1991–1992, the system was rebuilt and optimized. Virtually every portion of the instrumentation was improved, but the most important changes concerned the beamline. The borrowed monochromator was replaced with a sphericalgrating instrument from the Lawrence Berkeley Laboratory. After extensive computer simulation of the beamline response, the optical system was optimized to the undulator's output. By the end of 1991, most of the rebuilding work had been completed, and tests were initiated of the new instrument's performances.

Since then, we completed a long and extensive series of such tests, that demonstrated, on one

hand, a marked improvement with respect to the previous performances, and on the other hand, performance levels that are unmatched at present for this kind of instruments.

Perhaps the most important element in the new performance level is the lateral resolution. Fig. 2 shows the image of a portion of a Fresnel zone plate, used as a standard for one of the lateral resolution tests. By analyzing the features from progressively smaller zones, we observe that MAXIMUM is capable of imaging features whose size is consistent with a resolution of the order of 900 Å. Furthermore, we tested the spectroscopic capabilities of the instrument by analyzing core-

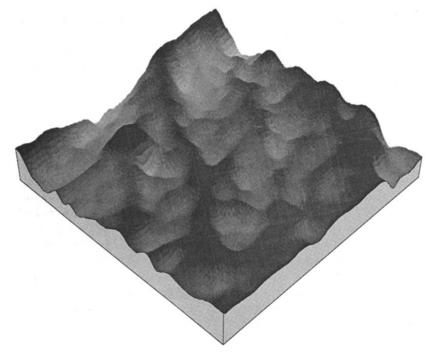


Fig. 3. Three-dimensional reconstruction of a photoelectron micro-image of a portion of a neuron specimen. The reconstruction was performed with the $^{\circ}$ Spyglass software.

level and valence-electron spectra of different systems, in particular f and d core levels of semiconductor surfaces and interfaces. These tests demonstrated a limit energy resolution better than 400 meV.

The MAXIMUM system in its new version was already tested in real experiments, both in materials science and in the life sciences. These last are quite demanding, and absolutely require a lateral resolution better than the typical size of cell components [3]. Fig. 3 shows the three-dimensional reconstruction of a photoelectron-yield image of portions of a neuron specimen, with cells and connections. This reconstruction emphasizes the already observed topographic component in the photoelectron image formation process, that has not yet been completely clarified.

From the point of view of biological photoemission spectroscopy, MAXIMUM was successfully used [10] to detect elements in the neuron cell membrane: see, for example, the potassium, sodium and calcium features from the ion channels in fig. 4. Note that the detection of these elements is made possible by the surface sensitiv-

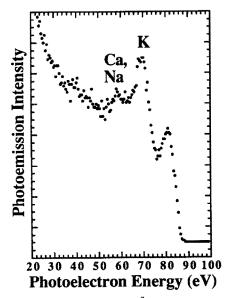


Fig. 4. Spectrum from a $1 \times 1 \ \mu m^2$ area of cell body in a neuron network [10], with features related to elements in the ion channels of the cell membrane.

ity of photoemission spectra at these energies. In a sense, results like those of fig. 4 are the first applications of surface-science techniques to biological experiments.

As to the future, we are now planning to extend MAXIMUM's technology to the new ultrabright synchrotron sources ELETTRA and ALS. The motivation is quite clear: even with the extensive improvements and fine tuning, MAXI-MUM does not reach its ultimate performances, and the limit is primarily the brightness of the present photon source. On the new sources, the brightness will further increase by 2–3 orders of magnitude, so that the ultimate (diffraction) limit of lateral resolution can be reached with additional photons available, that can be used to improve other MAXIMUM performances.

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References

- [1] F. Cerrina, B. Lai, C. Gong, A. Ray-Chaudhuri, G. Margaritondo, M.A. Green, H. Höchst, R. Cole, D. Crossley, S. Collier, J. Underwood, L.J. Brillson and A. Franciosi, Rev. Sci. Instrum. 60 (1989) 2249.
- [2] F. Cerrina, S. Crossley, D. Crossley, C. Gong, J. Guo, R. Hansen, W. Ng, A. Ray-Chaudhuri, G. Margaritondo, J.H. Underwood, R. Perera and J. Kortright, J. Vac. Sci. Technol. A 8 (1990) 2563;
 W. Ng, A.K. Ray-Chadhuri, R.K. Cole, S. Crossley, D. Crossley, C. Gong, M. Green, J. Guo, R.W.C. Hansen, F. Cerrina, G. Margaritondo, J.H. Underwood, J. Korthright and R.C.C. Perera, Phys. Scr. 41 (1990) 758;
 C. Capasso, A.K. Ray-Chaudhuri, W. Ng, S. Liang, R.K. Cole, J. Wallace, F. Cerrina, G. Margaritondo, J.H. Underwood, J.H. Underwood, J.B. Kortright and R.C.C. Perera, J. Vac. Sci. Technol. A 9 (1991) 1248.

[3] G. De Stasio, W. Ng, A.K. Ray-Chaudhuri, R.K. Cole, Z.Y. Guo, J. Wallace, G. Margaritondo, F. Cerrina, J. Underwood, R. Perera, J. Kortright, D. Mercanti and M.T. Ciotti, Nucl. Instrum. Methods A 294 (1990) 351; D. Mercanti, G. De Stasio, M.T. Ciotti, C. Capasso, W. Ng, A.K. Ray-Chaudhuri, S.H. Liang, R.K. Cole, Z.Y. Guo, J. Wallace, G. Margaritondo, F. Cerrina, J. Underwood, R. Perera and J. Kortright, J. Vac. Sci. Technol. A 9 (1991) 1320;

G. De Stasio, C. Capasso, W. Ng, A.K. Ray-Chaudhuri,
S.H. Liang, R.K. Cole, Z.Y. Guo, J. Wallace, F. Cerrina,
G. Margaritondo, J. Underwood, R. Perera, J. Kortright,
D. Mercanti, M.T. Ciotti and A. Stecchi, Europhys. Lett.
16 (1991) 411.

[4] K. Halbach, J. Chin, E. Hoyer, H. Winick, R. Cronin, J. Yang and Y. Zambre, IEEE Trans. Nucl. Sci. 28 (1981) 3136;

H. Winick, R. Boyce, G. Brown, N. Hower, Z. Hussain, T. Pate and E. Umbach, Nucl. Instrum. Methods 208 (1983) 127.

- [5] G. Margaritondo and F. Cerrina, Nucl. Instrum. Methods A 291 (1990) 26.
- [6] A. LeFurgey, S.D. Davilla, D.A. Kopf, J.R. Sommer and P. Ingram, J. Microscopy 165 (1992) 191;
 D.E. Johnson, Ann. N.Y. Acad. Sci. 483 (1986) 241;
 R. Rick, A. Dörge, F.X. Beck and K. Thurau, Ann. N.Y. Acad. Sci. 483 (1986) 245;

S.B. Andrews and T.S. Reese, Ann. N.Y. Acad. Sci. 483 (1986) 284.

- [7] F.P. Ottensmeyer, Ann. N.Y. Acad. Sci. 483 (1986) 339;
 H. Shuman, C.F. Chang, E.L. Buhle, Jr. and A.P. Somlyo, Ann. N.Y. Acad. Sci. 483 (1986) 295;
 C. Colliex, Ann. N.Y. Acad. Sci. 483 (1986) 311;
 R.D. Leapman, Ann. N.Y. Acad. Sci. 483 (1986) 326.
- [8] C.U. Ro, I.H. Musselman and R.W. Linton, Ann. Chim. Acta 243 (1991) 139;
 L. Vanvaeck, J. Bennett, W. Lauwers, A. Vertes and R. Gijbels, Mikrochim. Acta 3 (1990) 283;
 P.F. Schmidt and R.H. Barkhaus, Prog. Histochem. Cytochem. 23 (1991) 342.
- [9] B.P. Tonner, Nucl. Instrum. Methods 291 (1990) 60;
 B.P. Tonner and G.R. Harp, Rev. Sci. Instrum. 59 (1988) 853; in: Synchrotron Radiation in Materials Research, Mater. Res. Soc. Proc. 143 (1989) 279; J. Vac. Sci. Technol. A 7 (1989) 1;
 G.R. Harp, Z.L. Han and B.P. Tonner, J. Vac. Sci.
- Technol. A 8 (1990) 2566; Phys. Scr. T 31 (1990) 25.
 [10] G. De Stasio, W. Ng, A.K. Ray-Chaudhuri, R.K. Cole, Z.Y. Guo, J. Wallace, G. Margaritondo, F. Cerrina, J. Underwood, R. Perera, J. Kortright, D. Mercanti and M.T. Ciotti, unpublished.