MEPHISTO spectromicroscope reaches 20 nm lateral resolution

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The recently described tests of the synchrotron imaging photoelectron spectromicroscope MEPHISTO (Microscope à Emission de PHotoélectrons par Illumination Synchrotronique de Type Onduleur) were complemented by further resolution improvements and tests, which brought the lateral resolution down to 20 nm. Images and line plot profiles demonstrate such performance. © *1999 American Institute of Physics.* [S0034-6748(99)04902-3]

I. INTRODUCTION

We recently reported the first tests of a novel instrument for synchrotron photoelectron spectromicroscopy.[1] This instrument is one of the electron-imaging systems[2] and is called MEPHISTO from the French acronym ''Microscope à Emission de PHotoélectrons par Illumination Synchrotronique de Type Onduleur'' (Photoelectron Emission Microscope by Synchrotron Undulator Illumination). Several design improvements[1] concerning the electrostatic lens configuration, the large voltages in the electron optics, and the insertion of interchangeable apertures in the back focal plane of the objective lens had previously yielded a lateral resolution of 50 nm. As to the spectroscopy performances, x-ray absorption spectra were taken with an energy resolving power up to 2000 (estimated limit depending on the beamline: 10^4). The MEPHISTO spectromicroscope has been successfully tested both in the photoemission and in the transmission modes.[1,3] The present results were obtained in the photoemission mode.

We now present the latest resolution tests, which demonstrate the unprecedented lateral resolution of 20 nm for spectromicroscopy.

A complete description of MEPHISTO in the photoemission mode can be found in Ref. 1. In short, it is composed of: the photon source (monochromatized soft x rays from a synchrotron bending magnet source), the electron optics, the microchannel plate imaging system, the TV camera, and the computer system.

The first electron optics component is the sample itself, kept at high negative voltage, down to -20 kV, followed by the objective lens, three interchangeable apertures of different diameters, the intermediate lens, and the projective lens. Each of the three electrostatic lenses is composed of three elements; the first and third are at ground, whereas the central element is kept at high negative voltage of slightly smaller magnitude than the sample bias. The photoelectrons emitted by the sample surface under illumination are accelerated by the voltage difference between the sample and the first element of the objective lens.

The objective lens is the critical factor for the spatial resolution of this type of instrument. With a perfect objective lens, the resolution would reach the electron diffraction limit (a few Å). However, the resolution is determined both by the objective lens chromatic and spherical aberrations, and is dominated by the chromatic aberration (electrons of different energies are focused at different positions along the optical axis).[4]

Another element in our electron optics system plays an important role in reducing the chromatic aberration: the aperture in the back focal plane of the objective lens. This aperture stops the off-axis electrons, thereby eliminating the electrons that do not reach the focal plane near the focal point; this obviously reduces the chromatic aberration. The result is a marked improvement of the spatial resolution, which strongly depends on the aperture diameter.

In the present configuration, three quickly interchangeable (under ultrahigh vacuum) aperture diameters are available: 150, 50, and 20 μm. In the tests reported here we used

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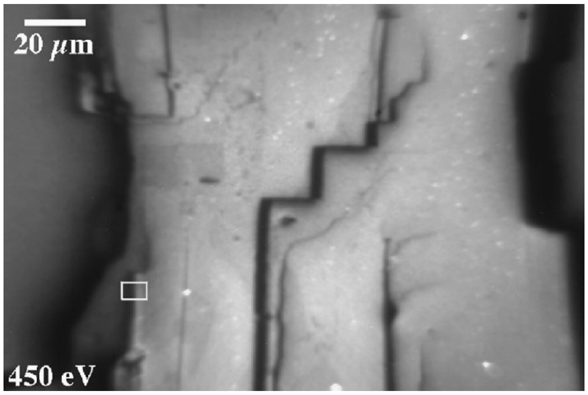

FIG. 1. Low magnification MEPHISTO image of a galena (lead sulfide) crystal sample acquired with monochromatic 450 eV photon energy. Image size 768×512 pixels. The imaged area has a horizontal dimension of 190 μm. The rectangular box on the lower left side indicates approximately the acquisition region of Fig. 2(a).

the 20 μm aperture and we carefully aligned it so that it was perfectly at the focal point. This can be done with following a simple strategy: if the aperture is not perfectly centered, varying the voltage on the objective lens produces a lateral shift of the image. Minimizing this effect made a perfect aperture alignment possible, permitting the improvement in lateral resolution reported below.

Another major improvement was the screening of undesired stray electrons from reaching the microchannel plate detector. These electrons were produced by field emission from the copper wires delivering high voltage to the electrostatic lenses, and were attracted to the microchannel plates (MCP) kept at up to +2 kV. Increasing the image magnification requires an increased projective lens voltage, but the MCP was saturated by stray electrons before reaching the highest magnification. We now introduced a tantalum screen between all high voltage wires and the microchannel plates, eliminating the problem to allow imaging at the highest magnification.

II. RESULTS AND DISCUSSION

Figure 1 shows a low magnification MEPHISTO image of a galena (PbS) crystal sample, acquired with monochromatic photons of 450 eV. The x-ray beam was produced by the Aladdin storage ring at the Wisconsin Synchrotron Radiation Center (SRC), and filtered by the Mark II ''Grasshopper'' monochromator. The rectangular box in this figure indicates the approximate position where the high magnification image of Fig. 2 was acquired.

Note that in Fig. 2(a) appear small dark features that were not detectable at lower magnification. The line in Fig. 2(a) indicates the accurate position where the intensity profile of Fig. 2(b) was measured. Note in Figs. 2(a) and 2(b) that the vertical edge separating the bright from the dark region is not very sharp, as expected;[5] it therefore does not constitute a good resolution test feature. Nevertheless, the small dark feature intersected by the line of Fig. 2(a) has the sharp intensity profile of Fig. 2(b), on which the resolution can be estimated, according to the point-broadening criterion: the full width at half maximum (FWHM) of the Gaussian profile of a point object is the resolution. Obviously, if the object is larger than the resolution, as in the case of the dark feature of Fig. 2(a) this criterion leads to an underestimate of the resolving power. In the present case, we chose the smallest features we could find in the high magnification images, and measured the FWHM of their intensity profiles. One of the results is shown in Fig. 3. This figure reports the same intensity profile of Fig. 2(b), in the area around the sharp feature. As it can be seen in Fig. 3, with monochromatic photons of 450 eV the FWHM of this object is 80 nm. The resolution, therefore, is better than 80 nm. This is not the

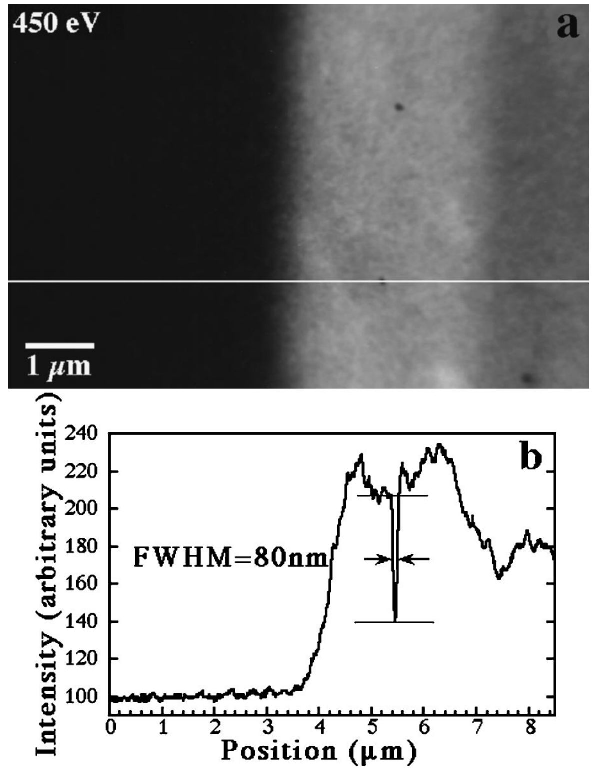

FIG. 2. (a) High magnification MEPHISTO micrograph of the 8.5 μm wide area indicated in the box of Fig. 1. The image size is again 768×512 pixels, and was acquired with 450 eV photon illumination. The magnification was increased by increasing the photoelectron accelerating voltages of the sample and the electrostatic lenses. (b) Intensity profile measured on the image in (a), along the horizontal line indicated there. Note that the small dark feature intersected by the line (a) generates a sharp peak in the intensity profile (b).

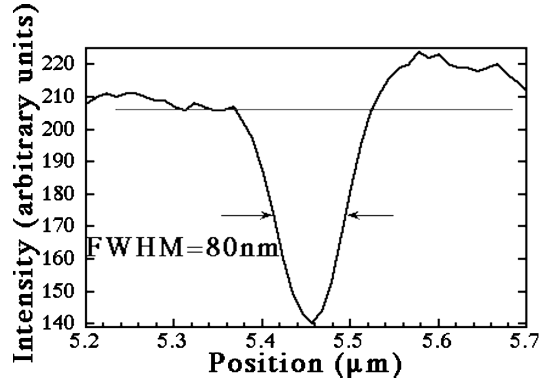

FIG. 3. Same intensity profile of Fig. 2(b), in the region across the sharp peak. The peak profile has a full width at half maximum of 80 nm.

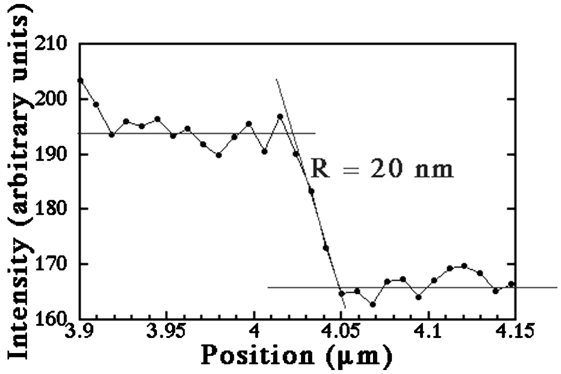

FIG. 4. Sharpest intensity profile, extracted from a batch of over 500 profiles, acquired with MEPHISTO on a grooved stainless steel surface, with unmonochromatized illumination. The resolution was measured according to the most conservative Rayleigh criterion (distance of the 12% and 88% intensity points in the interpolation lines) and gave results ranging between 22 and 35 nm. The profile in this plot is the best one, and gives 20 nm lateral resolution.

best but the average resolution obtained on a data base of several hundred tests performed on the same galena sample, which gave results ranging between 48 and 100 nm resolution, with a mode around 80 nm. This can be considered the real ''working resolution'' of our instrument.

This ''working resolution'' is limited by the physical dimensions of the objects imaged, and by the flux we obtain from bending magnet sources.

To more directly measure the resolution limit of the electron optics, we performed resolution tests on edges instead of point-like objects, and with unmonochromatized photons, gaining several orders of magnitude more photons per unit area on the sample surface.

We measured the resolution using the Rayleigh criterion: the FWHM of the Gaussian fit derivative of a knife edge curve. This is equivalent to the distance of the 12% and 88% intensity points in the interpolation lines shown in Fig. 4.

Figure 4 shows a test performed with unmonochromatized (zero order, from the SRC 6 m toroidal grating monochromator beamline) synchrotron light on a grooved stainless steel surface. This surface was grooved during tribology tests which produced wear scars between two stainless steel surfaces rhythmically rubbed against each other in the presence of the engine oil additive zinc dialkyl-dithiophosphate (ZDDP), used in tribology as an antiwear agent.

The sample had parallel grooves of many different sizes, which were vertical in the sample mounting, and illuminated from the left. They therefore produced high contrast vertical stripes in the MEPHISTO image, and over 500 edges on which we measured the resolution. These edges yielded values of lateral resolution between 20 and 35 nm. Figure 4 shows the best of these tests, reaching the 20 nm level.

The plot of Fig. 4 was extracted from an image obtained with the electron optical components set to the following voltages: $-11\,153$ V (sample), $-10\,370$ V (objective lens), $-9\,748$ V (intermediate lens), $-10\,763$ V (projective lens). The aperture diameter was 20 μm and the width of the image was 7 ± 1 μm, measured displacing the sample laterally on a high precision micrometer. The only significant source of error in our resolution measurement is the sample positioning. Since this has an uncertainty of ±1 μm, at this magnification $\pm15\%$, the uncertainty on the resulting resolution is also 15%, i.e., 20 ± 3 nm.

Note that ZDDP is a very hard material, and the groove edges found on this sample surface are therefore extremely sharp, certainly sharper than any lithographically patterned edges. The availability of such sample was also a key factor in achieving the resolution of Fig. 4.

Also note that our choice of unmonochromatized light for the 20 nm test was dictated by the low photon flux, not by the photon energy. It has therefore no implications on the physics of the experiment (e.g., chromatic or spherical aberrations). On a brighter x-ray source, such as an undulator beamline and/or a third generation synchrotron source the same results can be obtained with monochromatic light.

III. DISCUSSION

We reported the extensive resolution tests that we performed after improving both the instrumentation and the sample choice for such tests. The best test gave 20 nm resolution, and it was performed according to the most conservative Rayleigh criterion: we measured the distance between the 12% and 88% intensity points in the interpolation line of an edge profile.

Although with near-UV microscopy other authors obtained a similar resolution,[6] to the best of our knowledge, 20 nm is the highest resolution achieved by x-ray spectromicroscopy, either in the imaging, scanning, photoelectron, or transmission modes.

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