FINAL TECHNICAL REPORT: DE-FG02-98ER45688

AGENCY: Department of Energy
AGENCY No.: DE-FG02-98ER45688
PERIOD OF PERFORMANCE: 3/15/98 - 9/30/98

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TITLE OF PROJECT: "SpectroMicroscopy Research at the Advanced Light Source"

1. Introduction: Purpose of SpectroMicroscopy

Three closely related techniques, x-ray photoelectron spectroscopy, x-ray absorption spectroscopy, and x-ray fluorescence spectroscopy, have become widely accepted as important tools for the study of the chemical composition and electronic properties of surfaces, overlayers, and interfaces. There is now a major effort to push these spectroscopic techniques into a new realm of applications with very high spatial resolution, at and below 1 micron. This results in a new set of probes which can create images of chemical composition with great subtlety. The field is growing rapidly as high brightness sources of x-rays become available. This 6 month project was used to initiate research applications of soft x-ray spectro-microscopes at the Advanced Light Source. Due to its short duration, only preliminary results were obtained.

The term "spectromicroscopy" is an ugly and unwieldy word to impose on an experimental endeavor, but it has been adopted by a number of disciplines and is likely to remain in use for some time. The word is obviously a contraction of the phrase "spectroscopic microscopy," but there is also a distinction sometimes made between this, and the reverse combination, "microscopic spectroscopy", or "microspectroscopy". Microspectroscopy is a spectroscopic measurement with a small probe beam, which is usually fixed in position on the sample. Spectromicroscopy refers to the case where an image can be formed from the spatial dependence of the features in some spectrum.

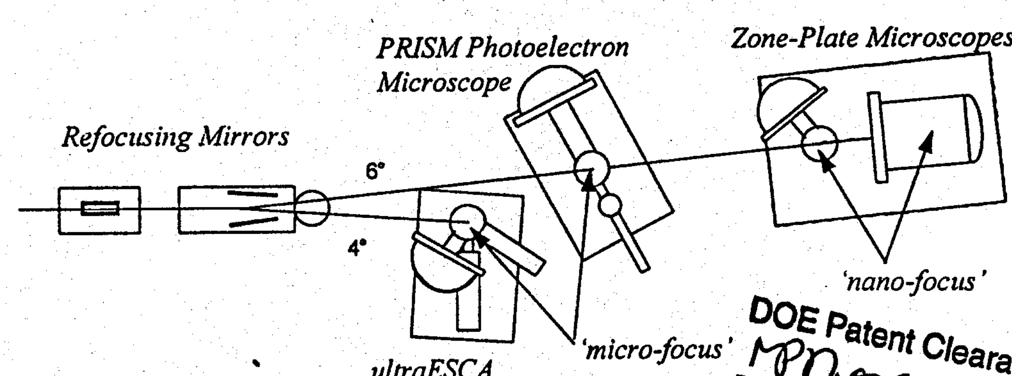

Fig. 1 Schematic layout of the Spectro Microscopy Facility instru[illegible]

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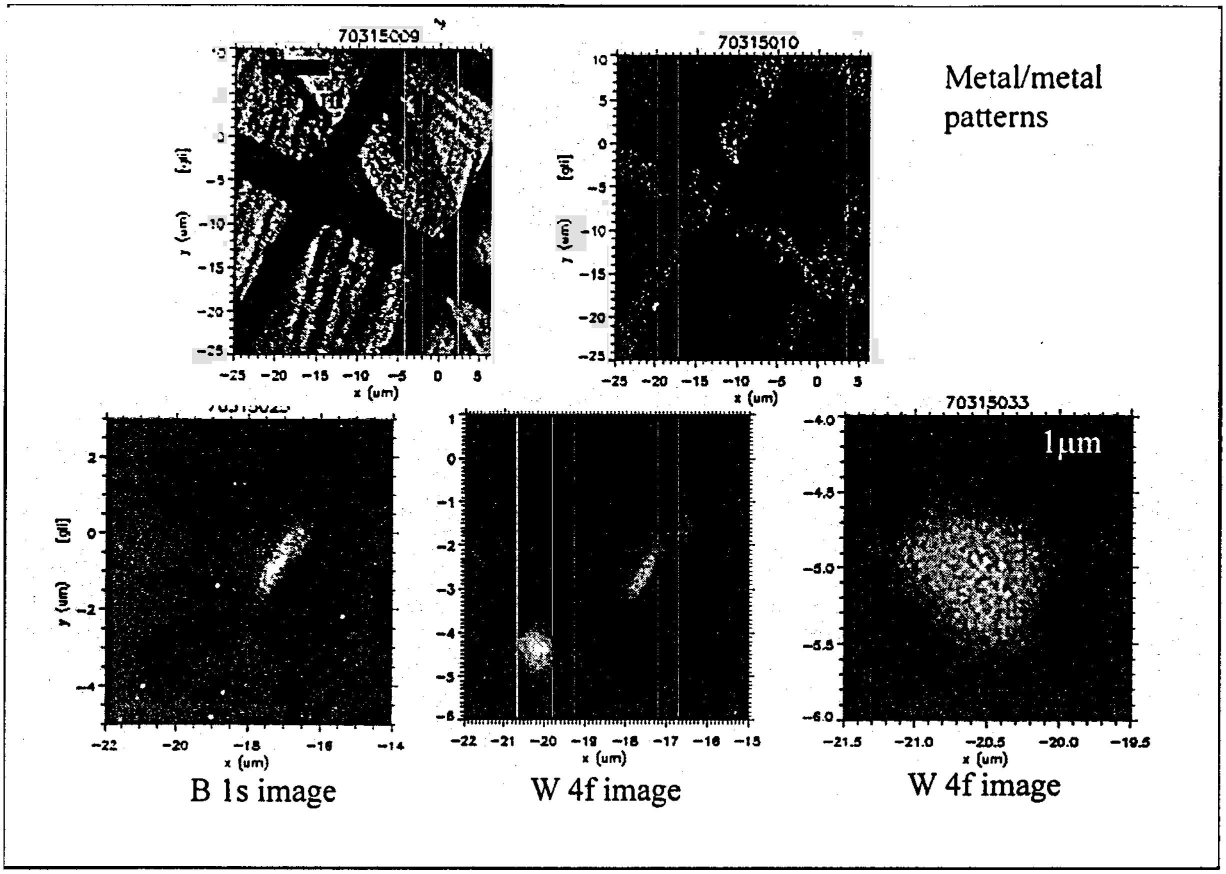

Fig. 2 :Example scanning photoemission images from ALS Beamline 7 SPEM.

Almost every analytical technique can benefit from spatial resolution, so there is a spectromicroscopy in infrared absorption and reflection, mass spectrometry, and visible fluorescence spectroscopy, to name just a few. Many of these techniques have already begun to develop stable technologies and standardization. In the case of electron spectroscopy, the challenges of surface sensitivity, ultra-high vacuum, low signal yield, and x-ray focussing, have presented significant hurdles to the rapid development of widely accessible instruments. It is only recently that the emphasis in the field of photoemission microscopy is shifting from instrument development to applications..

In photoemission microscopy, the goal is to achieve a spatial map of chemical state, as distinguished from atomic or elemental mapping. The chemical combination of elements in the sample can be understood from high resolution core-level spectroscopy, valence band spectroscopy, or fluorescence spectroscopy, sometimes in combination, depending on the complexity or constituents of the sample. The enormous popularity of photoemission spectroscopy in analytical work is related to its ability to reliably determine the chemical state of materials, with high surface sensitivity. There are few techniques that can offer this combination of quantitative compositional information and surface sensitivity, so the interest in high spatial resolution photoemission probes has remained strong throughout the prolonged incubation period of the technology.

There is one important sense in which the term "spectromicroscopy" should be understood to be different from simply taking a spectrum of some kind from a small area. In a mixed-phase sample, the spectrum that results from the combination of materials can be very complex. It may be that no single spectral "line" or feature can be used to identify the chemical components of the sample, but rather the entire spectrum (core-level, valence band, or other) is needed so that data reduction techniques like curve-fitting can be used. In cases like these, which may be the ones for which photoemission microscopes are most sought after, a spectrum is required at each point on the sample surface. This is a much larger data set than is typically encountered in most other microscopies, including surface techniques like Auger and SIMS imaging.

2. Implementation of Spectromicroscopy on BL7.0 of the ALS

The SpectroMicroscopy project is a collection of experiments designed to maximize the benefits of the undulator and monochromator on Beamline 7 at the Advanced Light Source. The experiments have a common theme of combining high spatial resolution and high energy resolution, for either mapping or structure determination with chemical-state selectivity[1,2]. There are a number of ongoing projects. These include the ultraESCA program, X-ray photoelectron diffraction (XPD), the PRISM photoelectron microscope, and two zone-plate microscopes, one for high pressure studies (STXM) and the other for ultra-high vacuum surface studies (SPEM). First results have been obtained from ultraESCA, XPD, PRISM, and STXM, portions of which are summarized here. The UHV zone-plate microscope is under construction, with the first images expected to be taken in the Spring of '96.

The photon energy range of the beamline is from about 60 eV to over 1000 eV. In this range the resolving power of the monochromator can be as high as 8000. The performance of the instruments we use is extraordinary, because the combination of the undulator, monochromator, and detectors are all at the state-of-the-art.

A schematic of the beamline layout is shown in Fig. 1.The beamline has two 'micro-focus' stations, that use adaptive grazing-incidence mirrors to put the monochromatic beam into areas as small as 25 micron diameter (this was measured). We further use one of the micro-focus positions as the object for the zone-plate microscopes, which focus down the x-ray spot to 0.15 micron and lower, depending on the zone-plate used.

What makes spectromicroscopy both interesting and useful, is that it is the process of taking some physical property of a sample and turning it into a spatial image. Examples of the properties used to make images include the x-ray absorption coefficient, photoemission cross-section, fluorescent yield, work-function, and the polarization dependence of both photoemission and absorption. These signals can in turn be interpreted to create microscopic maps of variations in composition, chemical state, crystallographic orientation, molecular adsorption site, and magnetization.

3. Results from this 6 month period:

An example of the results from the scanning photoemission microscope (SPEM) are shown in Fig. 2. At the end of this project, we achieved a spatial resolution of about 0.2 micron in SPEM, and a spectral resolution of 50 meV. This is excellent performance for this early stage of the project. Improvements to the zone-plates that we are using will reduce the spot size. The quality of the images is already very good, with some of them approaching the signal-to-noise ratio seen in conventional SEM images.

4. Publications from this period

"A scanning transmission x-ray microscope for materials science spectromicroscopy at the Advanced Light Source," T. Warwick, K. Franck, J. B. Kortright, G. Meigs, M. Moronne, S. Myneni, E. Rotenberg, S. Seal, W. F. Steele, H. Ade, A. Garcia, S. Cerasari, J. Denlinger, S. Hayakawa, A. P. Hitchcock, T. Tyliszczak, J. Kikuma, E. G. Rightor, H.-J. Shin, and B. P. Tonner, Rev. Sci. Instrum. 69, 2964 (1998).

5. REFERENCES

1 "Photoemission Spectromicroscopy of Surfaces in Materials Science," B. P. Tonner, Synchrotron Radiation News 4, 27 (1991).

2 "Development of Electron Spectromicroscopy," B. P. Tonner, D. Dunham, T. Droubay, J. Kikuma, J. Denlinger, E. Rotenberg and A. Warwick, J. Elec. Spectrosc. and Rel. Phenom. 75, 309-332 (1995).