# **Development of a Fast Scanning X-ray Microprobe**

A.C. Thompson, S.A. McHugo, R.A. Tackaberry, P.E. Denham and R.F. Gunion<sup>1</sup> <sup>1</sup>Center for X-ray Optics, Ernest Orlando Lawrence Berkeley National Laboratory, University of California, Berkeley, California 94720, USA

### INTRODUCTION

The x-ray microprobe at beamline 10.3 of the ALS has recently been upgraded with a new scanning stage and data acquisition system that substantially improves the capability of the beamline. The previous system acquired data in a "step and shoot" mode with no data analysis while images were being acquired. The new system has two modes of data acquisition – "step and shoot" and "slew scanning". Both modes allow a real time display of two-dimensional maps of elements selected for analysis. In the "step and shoot" mode a spectrum from the fluorescence detector is collected at one point and then the stage is moved to the next point in the scan and the next spectrum is taken. On the other hand, in the "slew scanning" mode the sample is scanned repeatedly through the beam in a raster mode and the fluorescent x-rays are detected and stored with a position tag. Elemental maps are produced and displayed as the system is scanning. Since the whole scan area is scanned in several minutes, this mode provides on-line analysis of the whole image field so that experimenters can optimize their beam time and quickly produce publishable results. In both modes the complete energy spectrum from the fluorescence detector is stored for each pixel so the data can be analyzed later for elements that were not looked for while collecting the data. The spectral data are processed with a peak fitting routine to provide elemental images that are calibrated using elemental standard samples.

### SYSTEM DESCRIPTION

The system is based on a two-dimensional scanning stage that has high resolution (100 steps/micron) position encoders on both stages. The horizontal scanning stage uses a solenoidal magnet to move the stage and a DC motor moves the vertical stage. In the 'step and shoot" mode of data acquisition the system acquires data at one point and then moves to the next point. The compete spectra from the Si(Li) detector and the current in two ion chambers (one upstream of the sample and the other downstream of the sample) are recorded at each point. Elemental maps for many different elements can be selected and then displayed as the scan progresses. This mode is used for scanning small areas of the sample around areas of special interest where a detail spatial profile is desired.

In the "slew scanning" mode, the scanning parameters for the stages are downloaded into a programmed controller by the data acquisition computer and then the raster scan data is collected by the system hardware with minimal software overhead. Each x-ray event is tagged with the position of the stages when the event occurred. As the scan progresses the computer stores each event in the correct spectra for each pixel. It also updates the elemental image maps for the elements of interest. This scanning mode has two major advantages. It allows the data in the whole image field to be monitored in real time so that the scan can be stopped when sufficient statistics have been accumulated or modified if the scan parameters are incorrect. In the "slew scanning" mode the data are averaged over the length of each pixel. This produces a more accurate map of the sample area than in the "step and shoot" where it is possible to miss areas of rapidly changing concentration if the pixel size is not about the same as the beam spot size. Typical

scanning parameters in this mode are a sample area of 100 micron x 60 micron with a pixel size of 2 microns and a scanning speed of 5 msec/pixel. With these parameters the system scans the full area in less than two minutes. The system scans the area repeatedly or until the desired number of scans is complete. The fluorescent x-rays are detected with a Si(Li) detector and the data is collected on a SUN computer with a modified data acquisition system from Princeton Gamma Tech. Since the intensity of the x-ray beam from the ALS varies during the experiment, the beam current is also recorded for each pixel as the stage scans. This allows the data to be normalized. The absolute elemental concentrations are determined by running elemental standards from NIST that are run before or after a sample run. The spectral data are processed after the scan with a peak fitting program based on a program from Mark Rivers of the University of Chicago. Quantitative elemental maps are produced using the calibration spectra from the elemental standards.

### RESULTS

The system uses a SUN computer system and software and hardware from Princeton Gamma Tech. Samples are usually mounted in between two layers of thin polypropylene on a 35-mm slide frame. The horizontal-vertical scanning stage is mounted on another linear stage to allow the sample to be positioned at the focal plane. In addition, the stage system is mounted on a rotary stage to allow for stereo and tomographic imaging.

Figure 1 is from a scan of a pair of crossed 25-micron diameter gold-covered tungsten wires. The sample is at 45 degrees to the incident beam and the detector is at 90 degrees to the beam. The Fig. 1a is the spatial profile of the total fluorescence image. The shadowing of the fluorescence signal on the horizontal wire by the vertical wire is visible on the right side of vertical wire. Fig 1b shows the zinc elemental image of the same area. This shows that the two spots on the lower right of the horizontal wire are from zinc particles on the wire.



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Figure 1a. Image of two  $25\mu$ m diameter Au-coated tungsten wires showing the shadowing by the vertical wire.

Figure 1b. Image of zinc over same area.

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Principal investigator: A.C. Thompson, Center for X-Ray Optics, Ernest Orlando Lawrence Berkeley National Laboratory. Email: thompson@lbl.gov. Telephone: 510-486-5590.