Quarterly Summary of Experimental Progress on the VESPERS Beamline

August 2009

Introduction

Since April 2009 some major advances were made in the beamline construction that made it possible to demonstrate XRF mapping and the acquisition of Laue XRD patterns that could be indexed. The pink beam optical train was aligned to produce a focussed spot of under 10 microns; this was used in the calibration of the Laue camera as well as in the first successful mapping of orientation and microscopic strain patterns on a nickel alloy surface. Further refinement of the pink beam optics was prevented by the discovery of a flawed mirror that will be replaced toward the end of the year. After that, attention has concentrated on the refinement of the monochromator optical train. Using the 9% bandpass filter in this train, a beam with a mean energy of 19 kV was able to be focussed to a spot size of 5 x 6 microns. Moreover, the beam flux provided by this train proved to be more than adequate for some micro XRF mapping studies that are described below. It is expected that this train can also be used for many types of Laue XRD studies.

The hardware and software for control of data and motors continues to be improved. User- oriented software is being developed on two levels: (1) software that will operate a wide range of devices using lower level machine languages, (2) software that will combine selected functions within a user friendly environment called “Science Studio”; this will be available eventually to remote users.

For the remainder of this year and up to the end of February 2010, VESPERS will be available to friendly users who propose feasible experiments by “Letter of Intent”. The first peer- reviewed general user proposals for Cycle x (March-June, 2010) on VESPERS has been announced and will close on September x, 2009. Allocation of beamteam member sessions will occur at the first annual VESPERS beamteam meeting that will be held on November 15th (available by phone).

Some details of the XRF and XRD measurements made on VESPERS during this quarter are described below. These measurements effectively completed the “Marquee Experiments” that were originally proposed as a measure of a certain level of readiness of the beamline for user studies.

(a) XRF Studies

An analysis program called Peakaboo, still under development, has been used to fit known sets of K, L and M series spectra for each element into the XRF spectra collected for each sample. The energies, peak separations and intensity ratios are a unique characteristic of each element and thus provide a more certain method for identification than the use of a single “region of interest”. The total peak intensity for each element, once identified, could then be plotted as a function of position of the microbeam to produce a “map” of the elemental spatial distribution.

As a continuation of studies begun in the last Report, spectra of a number of metals and glasses were obtained. The excitation energy window used to collect these was increased by using platinum coatings on all mirrors in the optical train, instead of the silicon substrate. The maximum excitation energy transmitted was thus increased to around 20 kV. An aluminium filter of several hundred microns thickness was used to attenuate the pink beam particularly in the low energy region below 4-5 kV. In Figure 1 the XRF spectrum for Alloy 6061 now covers an energy range up to 20 kV and the Compton-induced background does not be begin to become evident until ~1kV (instead of ~8kV with the Si substrate). The fitted K series of spectral peaks are shown in dark; because of the logarithm intensity scale used, the fitted spectra for less abundant elements do not appear to completely fill the spectral peaks to which they are being fitted. Some medium Z elements are detected in addition to those found previously using the Si substrate.

A NIST SRM glass 6100 was also studied under identical pink radiation conditions; this is a calcium silicate glass to which a large number of elements (61) had been added, with at least some of them near the 500 ppm concentration level target. In Figure 2 the spectrum is shown for an 8 second exposure. In this case, the data has been smoothed by a Savitsky-Golay mathematical filter. Twenty seven elements were detected, all of them known or believed to be present by NIST: These (and their known concentrations) are: Si (32%), Ca(8%), Ti(440 ppm), V(? ppm), Cr(? ppm), Mn (485 ppm), Fe(460 ppm), Co (390 ppm), Ni(460 ppm), Cu(440 ppm), Zn ? ppm), Ga (? ppm), Ge(? ppm), As(? ppm), Se(? ppm) , Rb (430 ppm), Sr (520 ppm), Y (? ppm), Zr(? ppm), Nb (? ppm), Mo (? ppm), Nd (? ppm), Tb (? ppm), Lu(? ppm), Bi (? ppm), Th (455 ppm), and U(460 ppm) . Elements *not* detected were: Ba (? ppm), Dy(? ppm), Er (? ppm),In (? ppm), Hf(? ppm), Ho (? ppm), Ag(254 ppm), Sn (?ppm) W (? ppm), Tl(60 ppm), Te(?ppm) Ta (? ppm), Re (? ppm), Pb (425 ppm), Cs (? ppm), Cd (?

ppm)and Au(25ppm). Elements could either have been undetected because their concentration is unexpectedly low in the glass or because they are located in spectral regions where the K series is not attainable for present VESPERS conditions and the L levels are too low in energy for easy detection. The elements Ru-Ba fall in this class. In summary, using a relatively brief exposure with VESPERS, it was possible to identify the overlapping spectra of a reasonably large number of trace element additives to the glass using the Peakaboo software.

At this point in the development of Peakaboo, the program cannot independently consider *all* possible solutions involving large sets of multiple K and L spectral peaks to arrive at a “best overall fit”; thus, for the present, the order with which peaks are fitted can result in some ambiguities where relatively small peaks are involved. For example, some of the fine structure in the energy range of 4-9 kV is probably due to additional rare earth elements that cannot presently be all treated by the program, while it is still fitting the first row elements.

A third sample to be studied by XRF has been a uranium mine tailings specimen from the Athabasca basin, and this is work that has been carried out by Tom Kotzer and his co-workers. In this study, the 9% bandpass setting was used in the place of pink light and its mean energy was set at 19kV. The XRF spectrum shown in Figure 3 has, as expected, a much lower background for a comparable exposure time, compared to the pink light spectra. We were encouraged to find the peak intensities obtained with the bandpass filter were not substantially reduced from those that we had obtained with pink radiation (about a factor of 3). This results in part from the counting limitations of our Vortex detector in the bath of pink light, but it also illustrates the value of having a high intensity “window” of energy with a somewhat restricted energy range. A large number of elements were able to be detected using Peakaboo, some of which are indicated in Figure 3(b) following smoothing.

A map of a 200 micron square area was made using a 3 second dwell time and with no prior mathematical filtering. These are shown in Figures 5 and 6 and they indicate that the conditions are suitable for obtaining very useful and interesting distributions for relatively short exposures.

(b) XRD Studies

Following the calibration of the CCD detector using pink radiation, a number of test Laue diffraction studies were carried out by Jing Chao and Marina Fuller with nickel alloy specimens that had been exposed to stress corrosion cracking conditions. A typical diffraction pattern is shown in Figure 7(a) for a 10 micron area on the sample. The exposure time needed for this was 0.25 seconds: this shows that VESPERS has a Laue pattern detection sensitivity comparable to or better than that found at beamline 34ID at APS. The patterns were able to be indexed and from this, maps of grain orientation and strains were able to be produced (see Figure 7(b)).