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The stand-alone microprobe at Livermore

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Abstract

Lawrence Livermore National Laboratory (LLNL) and Sandia National Laboratories/California have jointly constructed a new stand-alone microprobe facility. Although the facility was built to develop a method to rapidly locate and determine elemental concentrations of micron scale particulates on various media using PIXE, the facility has found numerous applications in biology and materials science. The facility is located at LLNL and uses a General Ionex Corporation Model 358 duoplasmatron negative ion source, a National Electrostatics Corporation 5SDH-2 tandem accelerator, and an Oxford triplet lens. Features of the system include complete computer control of the beam transport using LabVIEW™ for Macintosh, computer controlled beam collimating and divergence limiting slits, automated sample positioning to micron resolution, and video optics for beam positioning and sample observation. Data collection is accomplished with the simultaneous use of as many as four EG&G Ortec IGLET-X™ X-Ray detectors, digital amplifiers made by X-Ray Instruments and Associates (XIA), and LabVIEW™ for Macintosh acquisition software.

Keywords: Ion microprobe; PIXE; STIM

1. Introduction

Lawrence Livermore National Laboratory (LLNL) and Sandia National Laboratories/California (SNL) have jointly constructed a new stand-alone microprobe facility. The facility is located at LLNL but is jointly run by SNL and LLNL personnel. The new facility is primarily used for Particle Induced X-Ray Emission (PIXE), Scanning Transmission Ion Microscopy (STIM), and Ion Micro-Tomography (IMT) analyses. Applications range from particulate analysis to materials science to biology. Details of the new stand-alone facility and a summary of recent applications will be discussed.

2. Details of the system

A picture of the new microprobe facility is shown in Fig. 1. In the system, H^- ions are

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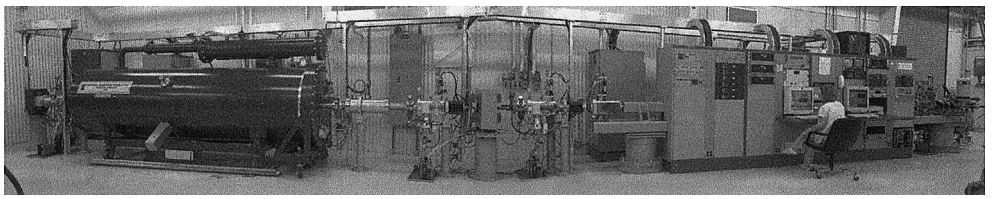

Fig. 1. The new stand alone microprobe at LLNL. The low energy injection magnet can be seen on the extreme far left, the accelerator is center left, the analysis magnet is in the center, while the microprobe beamline is on the right.

produced using a General Ionex Model 358 Duoplasmatron source. The H^- ions are then mass/energy analyzed by the low energy injection magnet and injected into a National Electrostatics Corporation 5SDH-2 tandem accelerator. The maximum operating voltage of the accelerator is 1.7 MV but normal operating voltage is 1.5 MV to produce approximately 3.0 MeV protons. Beam stripping is accomplished using either 1 μg/cm^2 carbon foils or a pumped gas stripper. After exiting the accelerator, the 3.0 MeV protons are mass/energy analyzed by the high-energy analysis magnet and injected into the microprobe beamline. The beamline for this stand-alone system is similar to the beamline used on LLNL's 10 MV tandem accelerator and uses an Oxford lens for final beam demagnification [1]. One unique feature of these beamlines is the use of three cast iron box beams sitting on sand filled piers to provide a rigid beamline structure.

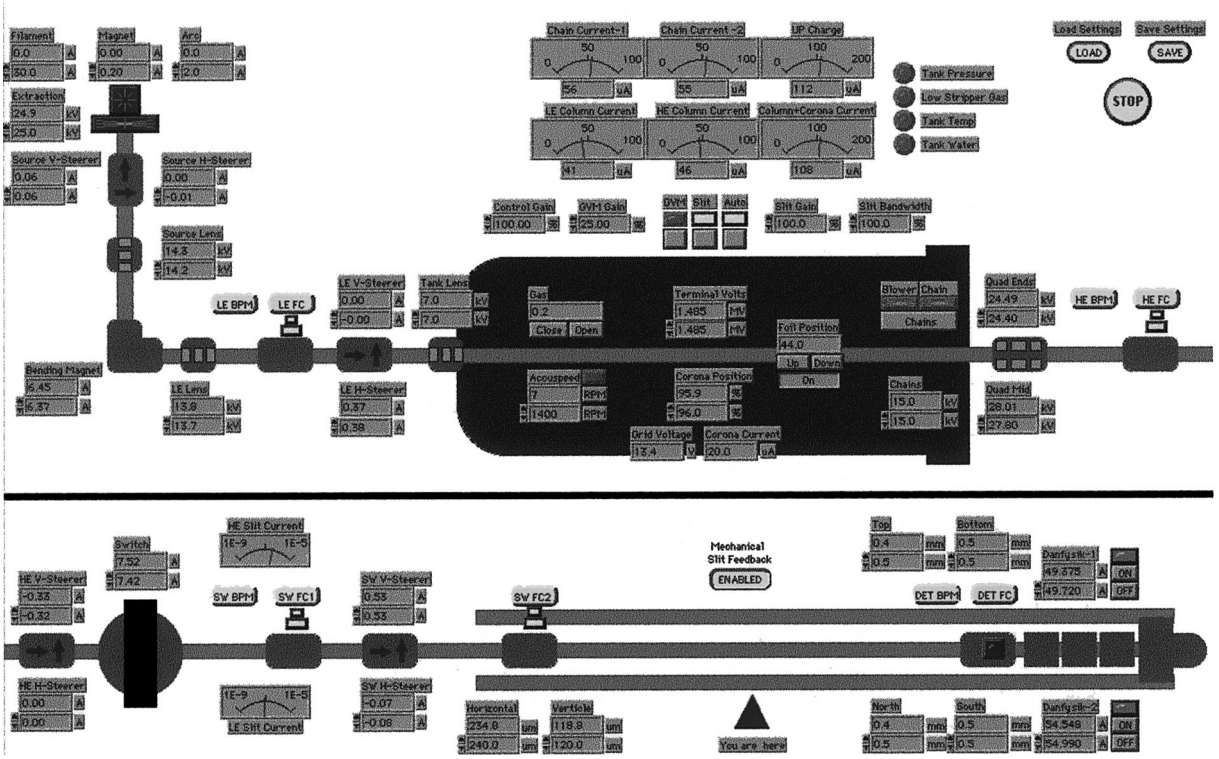

Fig. 2. Display screen for the LabVIEW™ based computer control system.

The box beams are approximately 10 cm wide by 15 cm tall with a wall thickness of 1.3 cm. The upper of the three box beams serves as the beam pipe or vacuum vessel for the ion beam. Because it is made of iron, the upper box beam also provides integral shielding from the effects of stray magnetic fields.

The new microprobe has an object to lens distance of 6.91 m and a lens to sample distance of 0.15 m. With the Oxford lens configured in the Converging-Diverging-Converging mode, demagnification is 88.3 in the horizontal plane and 25.4 vertical plane. Brightness of the beam through the microprobe system is up to 15 $pA/\mu m^2/mrad^2$. Maximum scanning range for 3 MeV protons is 1 mm in the horizontal plane and 1.5 mm in the vertical plane.

Operation of the stand-alone microprobe facility is accomplished using a computer control system. A display screen for the computer control system is shown in Fig. 2. The computer control stem is based on LabVIEW™ for Macintosh and uses CAMAC based Digital-to-Analog Converters, Analog-to-Digital Converters, Input/Output Registers, and Group 3 ControlNet fiber optic communications. Communication between the computer and the CAMAC crate is accomplished using GPIB. All aspects of the beam transport are computer controlled including the ion source, accelerator, beam bending magnets, steerers, lenses, Faraday cups, beam profile monitors, the collimating slits, the divergence limiting slits, and the Oxford quadrupole triplet. Because the computer control system can save and recall operating parameters, system startup is rapid and precise. From initial startup, it is not unusual to begin data taking activities in less than 5 min.

A picture of the target chamber used on the new stand-alone microprobe facility is shown in Fig. 3. The body of the target chamber is rectangular with a depth of 190 mm, a width of 270 mm, and a height of 760 mm. The chamber is pumped

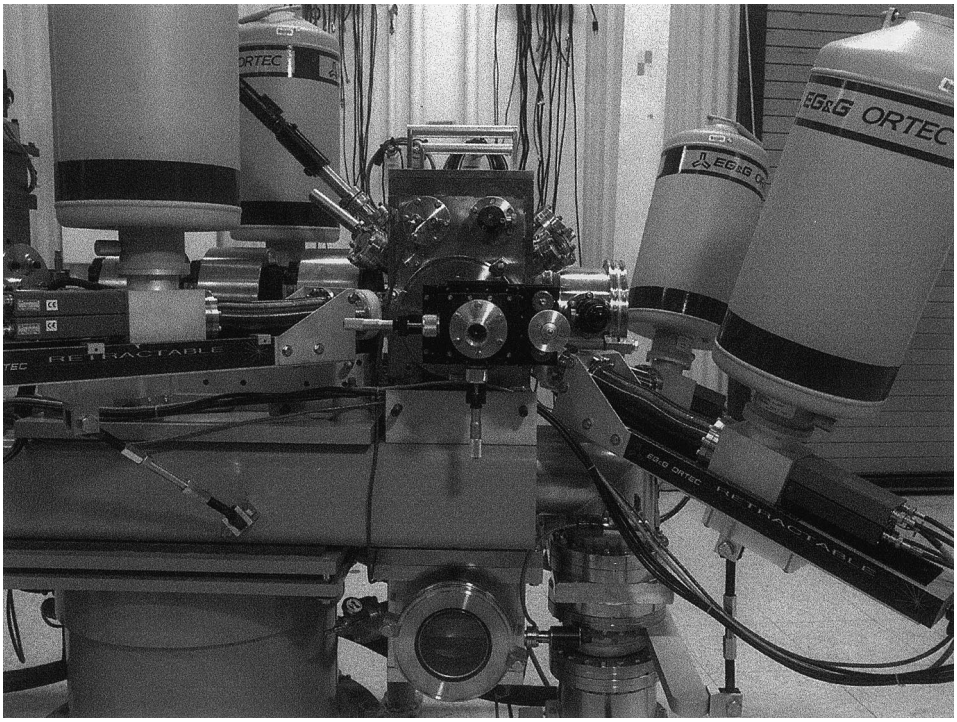

Fig. 3. The target chamber on the new stand-alone microprobe facility. One can see the four EG&G Ortec IGLET-X™ X-Ray detectors surrounding the central target chamber. The Oxford quadrupole triplet can be seen on the left between the two back angle X-Ray detectors.

by a 500 l/s Pfeiffer turbomolecular drag pump. After samples have been inserted, it normally takes less than 30 min to achieve an operating vacuum of approximately 1×10^{-6} Torr. For analyses that cannot be performed in vacuum, an external beam capability is achieved by placing an ultra thin Si_3N_4 window over the beam entry port to the target chamber and then back filling the target chamber to slightly below atmospheric pressure with helium.

Attached to the target chamber are various video cameras. These cameras provide multiple views and magnifications of the sample ladder, beam position, and sample morphology. Depending on the camera and multiplier lens used, magnifications of 1, 26, or 1430 can be achieved as viewed on a 19 in. monitor. At the 1430 magnification, the field of view is 100 by 100 μm. Two microscope lens assemblies are used to image sample morphology and microscopic placement of the beam. The first of these microscope lenses is mounted in front of the sample and images the sample and ion beam by way of a mirror mounted at 45°. To allow passage of the ion beam at normal incidence, the mirror has a 3 mm hole in its center. The second microscopic lens is mounted behind the sample and can only be used with transparent samples. Because the rear viewing microscope can approach closer to the surface of the sample than the front viewing microscope, objectives with a higher magnification and a shorter working distance can be used. The disadvantage of the rear viewing microscope is that it must be removed from the beam centerline to collect beam charge when performing PIXE measurements. Sample illumination is accomplished using a fiber optic illuminator and various viewports. Video signals from any of the cameras can be directed by two video switches to a large monitor, a video micrometer with contrast enhancement, or a video capture card.

Sample movement is accomplished using a Newport motion controller and manipulator. Samples can be moved up to 100 mm along the x-axis and up to 150 mm along the y-axis with 1 μm resolution and 2 μm repeatability. The samples can also be rotated for IMT analyses.

For PIXE analyses, we can simultaneously collect data from as many as four EG&G Ortec IGLET-X™ X-Ray detectors. Each of these detectors has an active area of 200 mm^2 and an energy resolution of better than 160 eV at 5.9 keV. These detectors are capable of measuring X-Ray energies as low as the carbon Kα line and have efficiencies close to 100% for energies up to 45 keV. Two of the detectors are located at back angles while the other two detectors are located at a forward angle. The forward angle detectors have filters to stop beam ion scattering and are limited to the detection of X-rays whose energies are greater than 6 keV. Using all four detectors simultaneously, it is possible to achieve an active detection area of greater than 1 sr. For STIM and IMT analyses, residual ion energies are measured using a retractable, charge-particle silicon surface barrier detector located approximately 5 cm behind the sample.

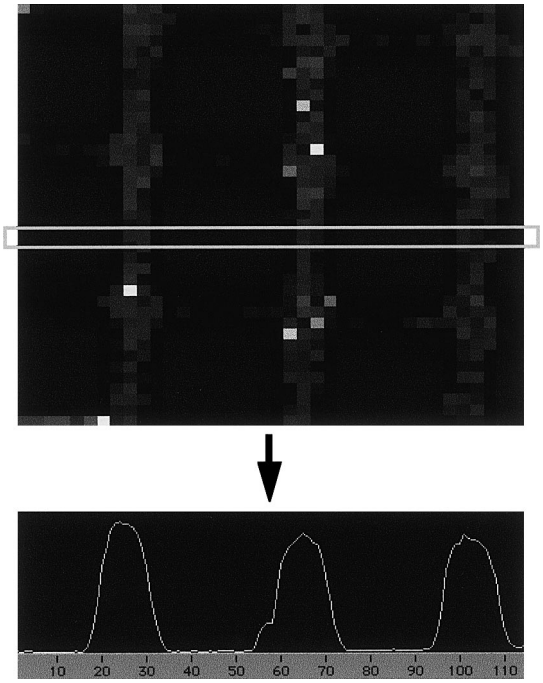

Fig. 4. A two-dimensional and one-dimensional line scan of a focused 250 pA 3 MeV proton beam across an electroformed Cu mesh grid. Grid spacing is 12.75 μm. Cu Kα X-Rays were detected. With the assumption of a gaussian shaped beam and perfectly sharp grid edges, a spatial resolution of 1.2 μm is indicated.

Data acquisition and data analysis on the new stand-alone microprobe facility are accomplished using a LabVIEW™ for Macintosh based system that is discussed elsewhere [2]. On-line histogram and elemental map displays provide a user friendly data acquisition interface while off-line data analysis allows the generation of elemental maps, PIXE spectral analysis, and tomographic reconstruction of IMT data.

As a test of beam spot size on the new stand-alone microprobe facility, we performed a two-dimensional and one-dimensional scan of a focused 250 pA 3 MeV proton beam across an electroformed Cu mesh grid. The result of this scan is shown in Fig. 4. With the assumption of a Gaussian shaped beam and perfectly sharp grid edges, a spatial resolution of less than 1.2 μm is indicated.

One problem that was observed on the new microprobe facility was a vertical drift of the beam spot during long run time analyses. This vertical beam drift was particularly noticeable on hot days. The source of this drift was eventually traced to our Oxford lenses. It was found that as our Oxford lenses warmed up, that they would expand, and that since the lenses sit on a manipulator base, the center of the lenses would shift upwards relative to the center of the beam. This upward shift in the lenses would cause our beam spot to drift down. This shift could be as large as 165 μm over a period of 8 h. To prevent this shift, we now fan cool our Oxford lenses and try to avoid turning them off.

3. Recent measurements

The new stand-alone microprobe facility was originally built to develop a method to rapidly locate and determine elemental concentrations of micron scale particulates on various media using

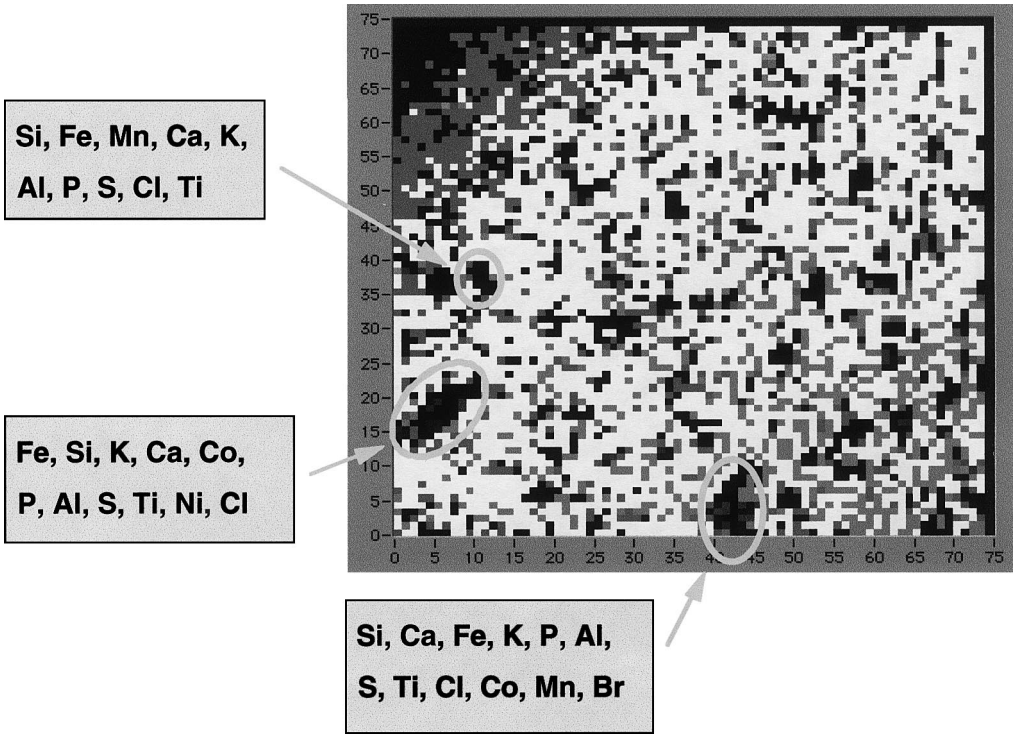

Fig. 5. Large area (1 mm × 1 mm) PIXE scan of a Lake Tahoe water filter. The beam spot size was approximately 13.5 μm. As opposed to bulk PIXE analyses, one can use micro-PIXE to obtain elemental analyses of individual particulates. Elements are listed in order of decreasing concentration.

PIXE [3]. Quantitative analysis of micron-scale particulates on filters is important in several application areas such as determining the composition of air- or water-borne environmental pollutants or for arms control and test ban treaty verification applications. One application area has been analyses of Lake Tahoe water filters.

Lake Tahoe is a large high elevation lake located in the Sierra Nevada mountains on the border between the states of California and Nevada. While the lake is noted for its extremely clear water, the past 40 yrs have seen a dramatic decrease in Lake Tahoe water clarity. One possible explanation for this decrease in water clarity is run-off from the increased urbanization of the Lake Tahoe basin. To study the particulates being discharged into Lake Tahoe, polycarbonate water filters were obtained from the mouths of three streams that empty into Lake Tahoe. One of these streams drained from an area that was largely urban in nature, the second steam discharged from an area that had source material that was largely volcanic in nature, while the final stream discharged from an area that had source material that was largely of granitic origin. Bulk PIXE analyses of the filter papers from these three stream types were obtained and bulk differences in elemental content were seen. To better understand the differences in bulk PIXE analyses, micro-PIXE analyses were performed on the filter papers to look for differences in elemental makeup of individual particulates on the filters. Fig. 5 shows a large area PIXE scan from the 'granite' type stream. While much work remains, it is hoped that analyses of particulates that flow into Lake Tahoe can lead to a better understanding of the decrease in Lake Tahoe water clarity.

Although the new facility was built to develop a method to rapidly locate particulates on media, the new facility has found numerous applications in biology and materials science. Many of these applications have been reported elsewhere and will not be described here [4–7]. One interesting application, however, has been the use of the mi-

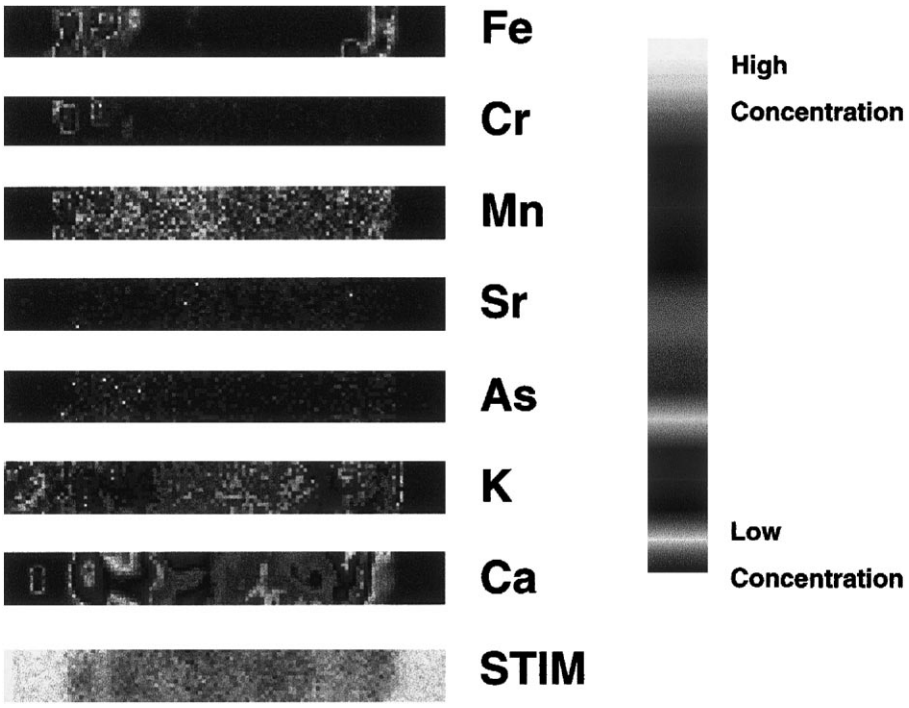

Fig. 6. PIXE and STIM scan of lichen cross section.

croprobe to better understand the distribution pattern of mineral elements in lichen tissues. Fig. 6 shows data from a PIXE and STIM scan of a lichen cross section. While the specific mechanism is not clearly understood, it is generally accepted that lichens receive most of their mineral nutrients from atmospheric out wash, with minimal input from the substrate [8]. The data shown in Fig. 6 suggest that the nuclear microprobe may be useful in elucidating element absorption and transport mechanisms in lichens. This research may also enhance efforts to use lichens in bio-monitoring of air quality.

4. Summary

Lawrence Livermore National Laboratory (LLNL) and Sandia National Laboratories/California have jointly constructed a new stand-alone microprobe facility. Key features of the new system include complete computer control of the beam transport, automated target positioning to micron resolution, the use of video optics for beam positioning/sample observation, and the ability to simultaneously collect data from as many as four energy dispersive X-Ray detectors. With this new facility, we have applied the techniques of micro-PIXE and STIM for use in particulate analysis, materials science, and biology.

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