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Infrastructure Investments in LAMDA: SEM Mill and Vault NSUF 2019 Milestone Report

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June 2019

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OAK RIDGE NATIONAL LABORATORY
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Reactor and Nuclear Systems Division

INFRASTRUCTURE INVESTMENTS IN LAMDA: SEM MILL AND VAULT NSUF 2019 MILESTONE REPORT

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ACRONYMS

ORNL	Oak Ridge National Laboratory
NSUF	Nuclear Science User Facility
LAMDA	Low Activation Materials Development and Analysis
PI	principal investigator
DOE	US Department of Energy
NE	Office of Nuclear Energy
UM	University of Michigan
MIBL	Michigan Ion Beam Laboratory
EBSD	electron backscatter diffraction
FIB	focused ion beam
LWRS	Light Water Reactor Sustainability
HAADF	high-angle annular dark field
RIS	radiation-induced segregation
RTE	rapid turnaround experiment
SEM	scanning electron microscope
S/TEM	scanning/transmission electron microscope (microscopy)
TEM	transmission electron microscope (microscopy)
APT	atom probe tomography

EXECUTIVE SUMMARY

This document summarizes the procurement, installation, and commissioning of a scanning electron microscope (SEM) mill produced by Fischione Instruments and a lead-shielded vault for specimen storage in support of Nuclear Science User Facility (NSUF) awards for use in Oak Ridge National Laboratory's (ORNL) Low Activation Materials Development and Analysis (LAMDA) laboratory. The Model 1061 SEM mill is a tabletop ion milling system for advanced sample preparation of high-quality SEM samples. This device was chosen for its broad range of ion energies allowing for both rapid milling and final polishing of the sample surface. The purchase order with Fischione Instruments was released and awarded by ORNL procurement on September 11, 2018. The instrument was shipped in December of 2018 and was installed in the LAMDA laboratory in March of 2019. During the commissioning of this equipment, electron backscatter diffraction (EBSD) was performed on specimens before and after milling to show the enhanced surface quality. With the purchase of a lead vault dedicated to NSUF library materials co-located in the LAMDA laboratory, the NSUF program will be able to maintain a significant sample library in LAMDA. These specimens will be easily accessible for rapid turnaround experiment (RTE) awards and will reduce the overall cost of year-over-year storage and shipments to and from ORNL hot cell facilities. The contract to fabricate a fourth lead vault dedicated to NSUF specimen storage was awarded by ORNL procurement on August 10, 2018. Installation of the new vault was completed in January of 2019.

1. SEM MILL

1.1 INTRODUCTION

The Fischione Instruments Model 1061 SEM Mill (Figure 1) is a tabletop ion milling and polishing system for advanced sample preparation of high-quality SEM samples. This instrument has two independently adjustable argon ion sources with programmable accelerating voltages capable of being continuously varied from 10 keV for rapid milling down to 100 eV for final specimen polishing. Each ion source is also capable of independent milling angle adjustment from 0 to 10 degrees for easy specimen planarizing. This instrument utilizes Fischione's "TrueFocus" ion sources, which boast of being highly efficient ion sources with a consistent beam current, maintaining small ion beam diameters even at low accelerating voltages. This is significant because the ions are directed only to the specimen, and it prohibits sputtered material from being redeposited from the specimen holder or chamber onto the specimen. With the addition of the high magnification microscope, in-situ images can be collected at any time during the milling process. This ensures that the user is completely aware of the surface condition of the sample. The inert environment and the capability for a liquid nitrogen–cooled sample stage allows for sample finishing in the most ideal environment suited to each individual material. This sample preparation method improves the EBSD quality of samples, as shown in the next section.

The user interface for this instrument allows for simple setup of milling parameters, which are controlled by a 10-inch touch screen, where the user can set the ion beam energy, milling angle, sample motion, sample position, and process termination. Automated operation of the instrument can be setup by creating a recipe in which you have a series of milling sequences. Each sequence in the recipe is completely customizable and can be setup to terminate by either time or temperature. Pauses can also be set into the recipe if user input is required (such as refilling the liquid nitrogen dewar) and the milling process can be paused at any time during operation. These recipes can be saved under the user's profile for future use.

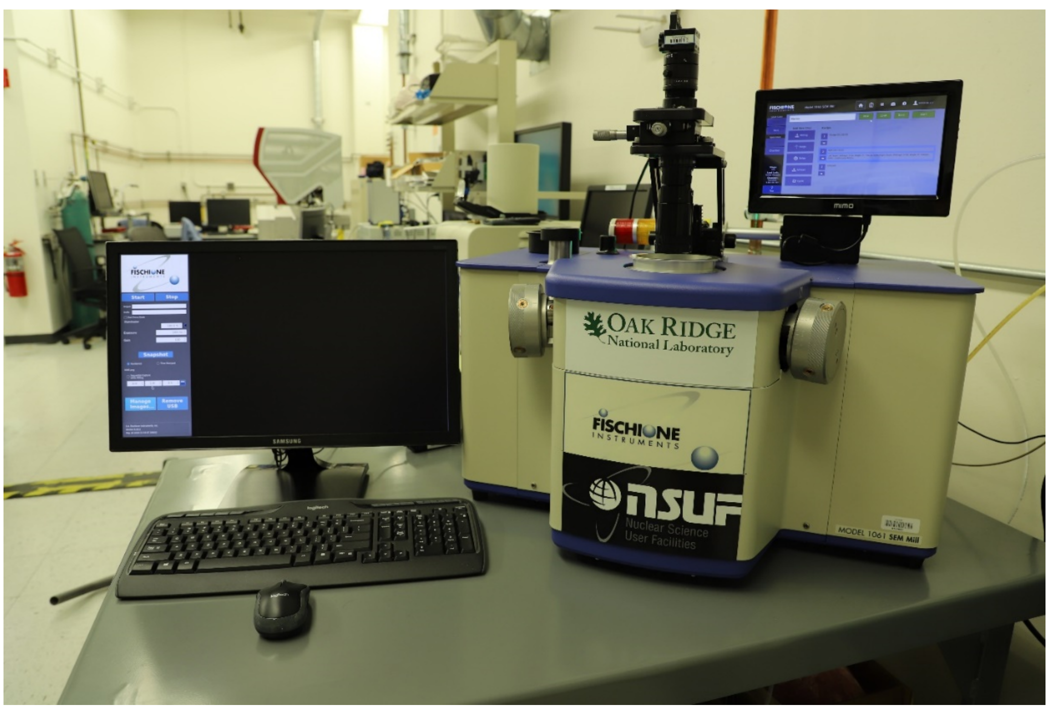


Figure 1. Fischione Instruments Model 1061 SEM Mill in LAMDA Laboratory.

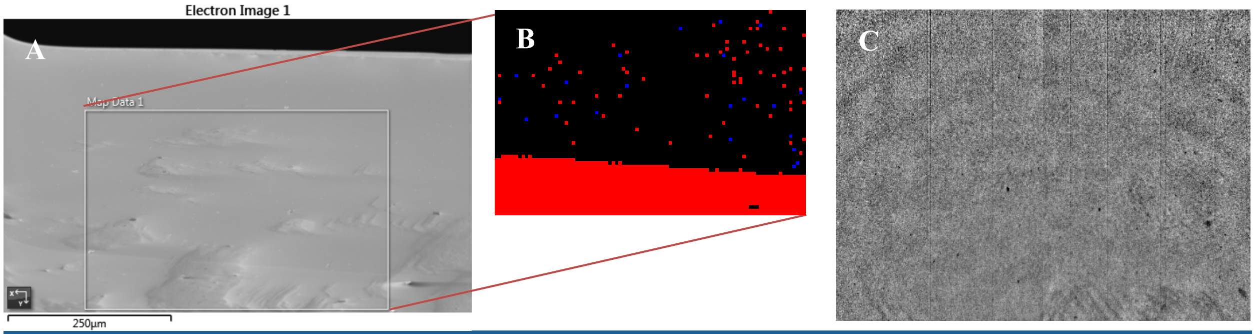
1.2 INSTALLATION AND COMMISSIONING

The purchase order with Fischione Instruments was released and awarded by ORNL procurement on September 11, 2018. The instrument was shipped in December of 2018 and was installed in the LAMDA laboratory in March of 2019. After the installation, steps were taken to ensure that the chamber vacuum was stable, and then two specimens were selected to test the instrument. The two specimens were pulsed laser deposited silicon carbide (SiC) on a commercially available Si (111) single side polished substrate. These specimens were fabricated by removing their native oxide layer via chemical etching, then the substrate was heated to 750°C at which point approximately 70 nm of SiC was deposited through laser ablation of a SiC (111) target, with the growth being monitored using reflection high energy electron diffraction. These specimens were used for past experiments and nearly two years old, resulting in a thick oxide layer. To test the capability of this instrument, EBSD maps were created from the same location of both samples before and after milling in the Model 1061 SEM Mill.

For both specimens, the EBSD parameters remained identical. Both used an accelerating voltage of 30 keV, a 16 nA beam current, and a 70° tilt. For specimen 1, 15-minute EBSD maps were created using a 5 µm step size for a large mapping. For specimen 2, 5-minute EBSD maps were created, also using a 5 µm step size for a smaller mapping. The ion milling parameters were similar for both specimens. Each specimen used both ion beams operating at 2 keV accelerating voltage with the stage rotating at 1 rpm for 30 minutes. The only difference was that specimen 1 had an ion beam tilt of 5° compared to the ion beam tilt of specimen 2, which was at 10°.

Comparison of Figures 2 and 3 shows the effects that ion milling has on the surface of specimen 1. Figures 2(a) and 3(a) show the effect of ion milling on the surface topology, with the post-milled surface being much more uniform. Figures 2(c) and 3(c) show representative Kikuchi patterns for the pre-milled and post-milled conditions, respectively. Since the EBSD Oxford software uses these patterns to identify the crystal phase and orientation, it is useful to ensure that the patterns are as definite as possible. Comparison of these two Kikuchi patterns taken from the center of the map portray the enhanced surface characteristics after this milling operation. This can also be seen clearly by comparing Figures 2b and 2d, as well as Figures 3b and 3d. Figure 2(b) shows an EBSD map of the area that includes many locations with zero solutions, indicating that the Kikuchi bands were not distinct. Figure 3(b) shows that after ion milling, there were far fewer locations with zero solutions. Specimen 2 (Figures 4 and 5) show similar conclusions.

In summary, the Model 1061 SEM Mill has been successfully procured and installed within ORNL’s LAMDA laboratory. Preliminary testing has concluded that the SEM mill is operating according to designated specifications. The SEM mill is now available for sample preparation of irradiated materials.

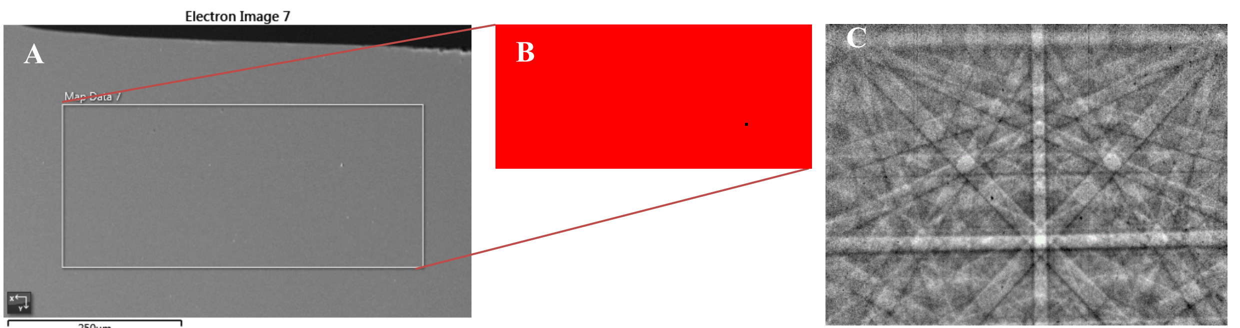


Full Map Statistics

D

				Band Contrast				MAD			
Phase	Color	Fraction (%)	Count	Mean	Standard Dev.	Min	Max	Mean	Standard Dev.	Min	Max
SiC [3C]	red	25.40	1441	162.76	31.30	27.00	195.00	0.37	0.15	0.18	1.47
SiC [4H]	blue	0.37	21	42.43	7.29	29.00	58.00	1.33	0.23	0.92	1.98
Zero Solutions	black	74.23	4211	40.77	6.59	0.00	71.00				

Figure 2. Pre-milled specimen 1 EBSD data showing (a) the pre-milled surface topography, (b) the EBSD map of the area, (c) the Kikuchi pattern from a point near the center of the map, and (d) the statistics for this mapping.

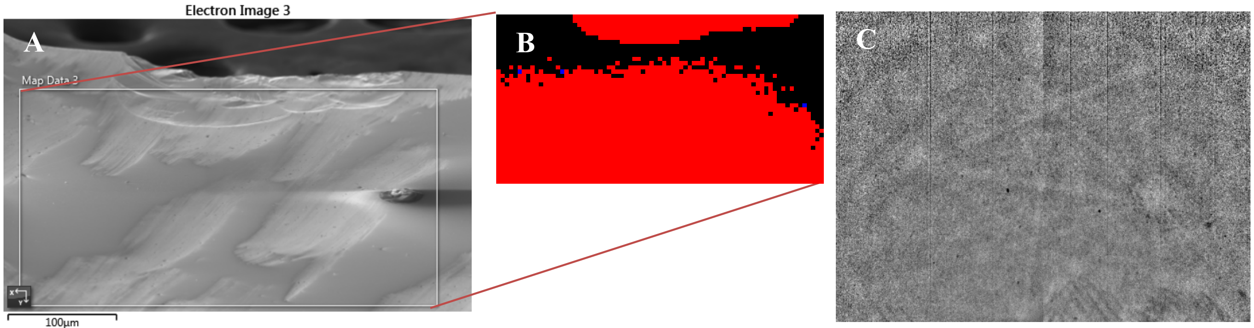


Full Map Statistics

D

				Band Contrast				MAD			
Phase	Color	Fraction (%)	Count	Mean	Standard Dev.	Min	Max	Mean	Standard Dev.	Min	Max
SiC [3C]	red	99.98	4887	191.51	5.57	123.00	209.00	0.17	0.02	0.08	0.26
SiC [4H]	blue	0.00	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zero Solutions	black	0.02	1	79.00	0.00	0.00	79.00				

Figure 3. Post-milled specimen 1 EBSD data showing (a) the post-milled surface topography, (b) the EBSD map of the area, (c) the Kikuchi pattern from a point near the center of the map, and (d) the statistics for this mapping.

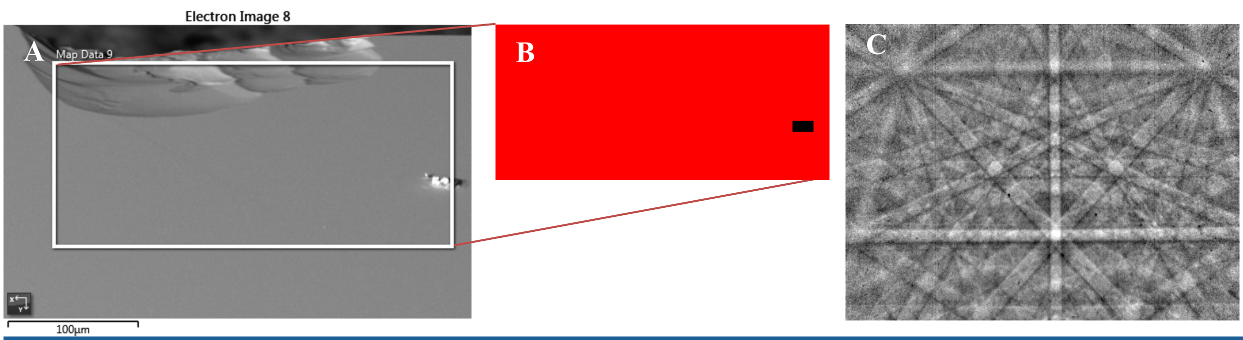


Full Map Statistics

D

Phase	Color	Fraction (%)	Count	Band Contrast Mean	Band Contrast Standard Dev.	Band Contrast Min	Band Contrast Max	MAD Mean	MAD Standard Dev.	MAD Min	MAD Max
SiC [3C]	red	72.01	2218	150.40	33.06	51.00	235.00	0.46	0.11	0.15	1.08
SiC [4H]	blue	0.10	3	80.33	6.65	71.00	86.00	1.30	0.08	1.19	1.38
Zero Solutions	black	27.89	859	67.31	9.96	0.00	133.00				

Figure 4. Pre-milled specimen 2 EBSD data showing (a) the pre-milled surface topography, (b) the EBSD map of the area, (c) the Kikuchi pattern from a point near the center of the map, and (d) the statistics for this mapping.



Full Map Statistics

D

Phase	Color	Fraction (%)	Count	Band Contrast Mean	Band Contrast Standard Dev.	Band Contrast Min	Band Contrast Max	MAD Mean	MAD Standard Dev.	MAD Min	MAD Max
SiC [3C]	red	99.56	1819	185.13	5.16	122.00	209.00	0.21	0.04	0.11	0.40
SiC [4H]	blue	0.00	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Zero Solutions	black	0.44	8	88.75	41.28	0.00	170.00				

Figure 5. Post-milled specimen 2 EBSD data showing (a) the post-milled surface topography, (b) the EBSD map of the area, (c) Kikuchi pattern from a point near the center of the map, and (d) the statistics for this mapping.

2. LEAD SHIELDED VAULT

The LAMDA laboratory at ORNL is a high demand, world-class irradiated materials research facility made available to university, industry, and other national laboratory researchers as an NSUF partner facility. It specializes in the study of materials with a low radiological threat and houses a broad range of instrumentation for materials testing and characterization. Operationally, LAMDA laboratory accepts samples with low or no radioactivity (<100 mR/hr at 30 cm beta/gamma) and low or no removable contamination (<100,000 dpm/100cm^2 beta/gamma, <2000 dpm/100cm^2 alpha). The advantage of bringing irradiated materials into this laboratory is to perform testing and characterization using advanced tools that would otherwise be too costly or extremely difficult to use or maintain in a hot cell facility with remote handling.

With a constant demand on the facility to support post-irradiation examinations, inventory controls are in place to ensure that background radiation levels remain as low as reasonably achievable. As new materials arrive in the lab, specimen inventories are managed through disposal or shielded storage, or they are returned to hot cell facilities for long-term storage. Each hot cell shipment is costly, and the use of hot cell storage can dramatically increase the cost and timeline of post-irradiation examinations primarily funded by NSUF RTEs. By purchasing a lead vault dedicated to NSUF library materials that is co-located in the LAMDA laboratory, the NSUF program can maintain a significant sample library in LAMDA. These specimens will be easily accessible for use on RTE awards, and they will reduce the overall cost of year-over-year storage and shipments to and from ORNL hot cell facilities.

Room 136D in Building 4508 was identified as a suitable room to support six lead cabinets for storage of the LAMDA specimen inventory. The design for lead cabinets and room layout (Figure 6) was completed in 2016, and the first three cabinets were fabricated and installed in the same year.

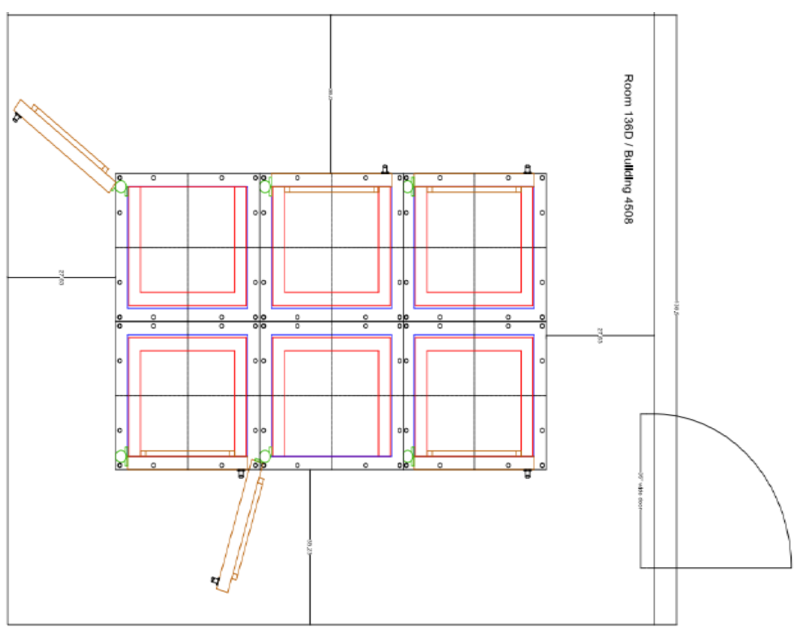


Figure 6. Building 4508, Room 136D layout of 6 lead storage cabinets.

The lead cabinet stands 60.5 inches tall, 36.75 inches wide and 33.6 inches deep, with 2 inches of lead shielding surrounding the entire cabinet (Figure 7). The cabinet contains 5 drawers with hinged doors, and each drawer can hold up to 25 lead pigs, for storage of an estimated 1,000 specimens.

Figure 7. First of three lead cabinets fabricated in 2016.

The contract to fabricate a fourth lead vault (Figure 8) dedicated to NSUF specimen storage was awarded by ORNL procurement on August 10, 2018. Installation of the new vault was completed in January 2019.

Figure 8. Fourth lead vault located in room 136D.