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RPPR Final Report
as of 17-Sep-2018

Agency Code:

Proposal Number: 68972MSREP **Agreement Number: W911NF-16-1-0531**
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Final Report for Period Beginning 01-Sep-2016 and Ending 30-Apr-2018
Title: Acquisition of Multi-functional Nanoprobe Station-based Measurement System for Comprehensive In Situ Materials Characterization and Measurement in SEM
Begin Performance Period: 01-Sep-2016 **End Performance Period:** 30-Apr-2018
Report Term: 0-Other

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RPPR Final Report
as of 17-Sep-2018

STEM Degrees: 1 **STEM Participants:** 1

Major Goals: 1. Acquire and set up a multi-functional nanoprobe station-based measurement (NPSM) system in a scanning electron microscope (SEM). This includes designing of the system, purchasing of multiple instruments and components from different manufacturers and vendors, and machining of required parts.
2. Assemble, install, and test the NPSM system to realize comprehensive functions of in situ materials characterization (structural and compositional) and property measurements (electrical, optical, and mechanical) in the SEM.
3. Provide user training for basic operations and advanced structure characterization and property measurements.
4. Include the NPSM system into a shared user facility, supporting research programs in areas of interest to DoD and education programs in STEM fields.
5. Enhance the STEM curricula at UNC Charlotte in materials characterization and measurements.
6. Enhance research involvement of undergraduate, women, minority students, and other underrepresented groups into research.
7. Provide proactive programs to ensure broader impacts to K-12 students, including high school teachers.

Accomplishments: 1. Goal #1 was accomplished. The system was designed based on the requirements of proposed NPSM system. The design was adjusted during the installation to fit the actual dimension of the SEM chamber. Major instruments and components have been purchased and machined. Additional parts might need to purchase during the testing stage of the system.
2. The assembling and installation in Goal #2 was accomplished. Most system testing was also completed. So far, we realized the operation of micromanipulator and nanoprobe shuttle to control the different probes, the electron beam induced deposition (EBID) using micromanipulator controlled gas injection system, the cathodoluminescence (CL) measurement using micromanipulator controlled optical fiber and external optical spectrometer, the sample transfer using microgrippers, and electrical measurement using probe shuttle and Keithley parameter analyzer.
3. For Goal #3, so far 7 graduate students including 5 female students from different research groups received basic operation training on manipulation of the nanoprobe station. Recently, 1 undergraduate student and 1 high school student has been trained intensively on this system and they worked on a summer project testing different functions of the NPSM system. User manuals and instructions have been developed for future training of more users on multiple functions of the system.
4. Goal #4 was completed. The system was included into our current SEM laboratory, a shared user facility in Mechanical Engineering Department. Currently, two research groups are using the system in their research projects. The system will continue to serve more research groups, enroll new users, and provide education demonstration.
5. The curricula development in Goal #5 was achieved and will be further developed in future courses. System was introduced in PI's undergraduate course of "Introduction of Engineering Materials" and graduate course of "Materials Characterization and Analysis". Labs on NPSM system will become a part in undergraduate course of "Microscopy for Engineering" from Fall 2018 and will be a major content for graduate course of "Materials Characterization and Analysis" from Spring 2019. We will further integrate the NPSM system into materials and nanostructure related courses.
6. Goal #6 was achieved and we will keep improving the involvement of underrepresented groups into research. The PI supported one undergraduate student, Kylen Blanchette from UNC Charlotte, with additional URAP fund in summer 2017. He had research experiences on materials synthesis and characterization and had a demonstration of the NPSM system. In summer 2018, the system hosted a summer URAP project supporting one undergraduate student, Douglas Lawrence. Douglas explored and tested different functions of the NPSM system.
7. Gola #7 was achieved and we will further enhance related programs for broader impacts. The PI supported one female high school student, Divya Sureshkannan from Providence, using additional HSAP fund in summer 2017. The PI also hosted a high school student volunteer, Andrew Peterson from Charlotte Country Day School, in summer 2017. Both students had research experiences on materials synthesis and characterization and received a demonstration of the NPSM system. In summer 2018, the system hosted a summer HSAP project supporting one high school student, Jackson Harwood from Lake Norman Charter High School. Jackson worked with Douglas tested a variety of functions of the NPSM system. Demonstration has been provided other graduate, undergraduate, high school students. Additionally, the PI applied and received an internal fund of #3,800 to organize workshops themed "Plenty of Room under the Microscope" for high school students and teachers. The workshops were prepared with classroom kits, presentation, and videos. The workshops will be arranged in August 2018.

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Training Opportunities: So far, 7 graduate students including 5 female students from four research groups have received basic operation training on manipulation of the NPSM system. In addition, 1 undergraduate and 1 high school students received intensive training for their summer project using different functions of the NPSM system. 2 other undergraduate and 3 high school students had demonstration on the NPSM system. Currently, two research groups are getting training to use the NPSM system in their research projects. Graduate students from other research groups will soon get training depending their needs in research projects.

Results Dissemination: The system was briefly introduced in PI's undergraduate course of "Introduction of Engineering Materials" and graduate course of "Materials Characterization and Analysis" in Spring 2017 to both undergraduate and graduate students at UNC Charlotte. A demonstration was performed to summer students from URAP and HSAP programs and high school volunteer. Total 3 undergraduate student and 4 high school students participated the testing and demonstration of the NPSM system with different functions in sample manipulation, electrical measurement, optical measurement, and electron beam induced deposition. The fund supported URAP and HSAP summer project in Summer 2017. It was presented in Charlotte Research Scholars (CRS) Summer Symposium: Kylen Blanchette, Divya Sureshkannan, Andrew Peterson, and HaiTao Zhang, "Synthesis, Characterization, and Measurement of One-dimensional Molybdenum Oxide Nanostructures", CRS Summer Symposium, Aug. 2017, Charlotte NC. The system hosted one URAP and HSAP summer project in 2018. This project was presented in CRS Summer Symposium: Douglas Lawrence, Jackson Harwood, Haitao Zhang, Terry Xu, "In Situ Testing of Nanostructures within Scanning Electron Microscope (SEM)", CRS Summer Symposium, Jul. 2018, Charlotte NC. The NPSM system and its capabilities are integrated into PI's undergraduate materials course and microscopy course, and the graduate materials characterization course. It will be delivered to a large group of students starting from Fall 2018.

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

PARTICIPANTS:

Participant Type: Undergraduate Student
Participant: Kylen Blanchette
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: High School Student
Participant: Divya Sureshkannan
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: High School Student
Participant: Andrew Peterson
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N

Other Collaborators:

Additional Information for Final Report

1. Status of the Proposed System

The proposed multi-functional nanoprobe station-based measurement system (NPSM) has been designed, purchased, and fully installed. Figure 1 shows the schematic drawing of the proposed system, and Fig. 2 shows the photos of the installed system.

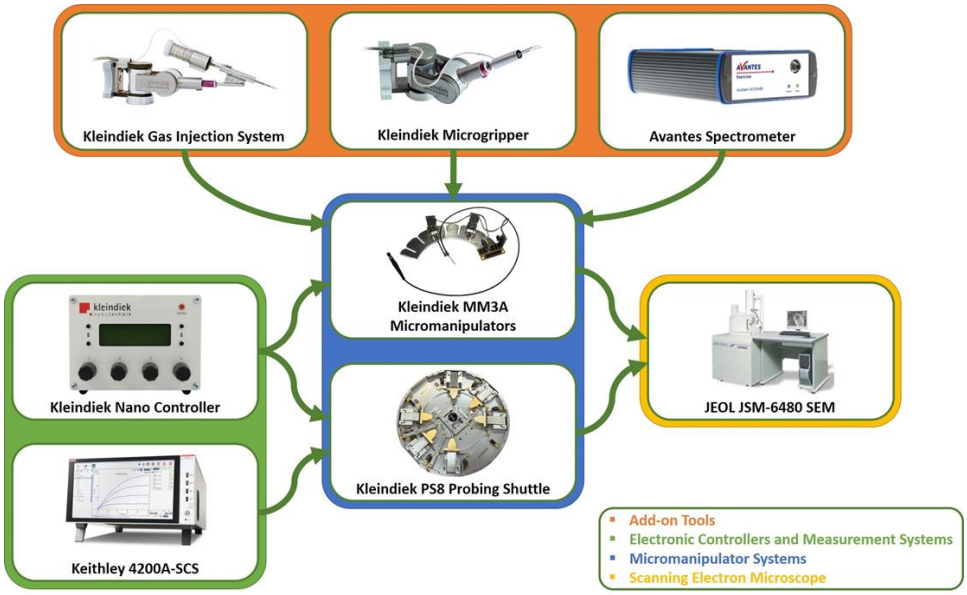


Fig. 1 Schematic drawing of NPSM system in a SEM for *in situ* characterization and measurement (not all manipulators and parts are displayed). (Some images are from vendors' websites: https://www.kleindiek.com/; https://www.tek.com/keithley-4200a-scs-parameter-analyzer; https://www.jeol.co.jp/en/; https://www.avantes.com/products/spectrometers/starline/item/304-avaspec-uls2048l-starline-versatile-fiber-optic-spectrometer.)

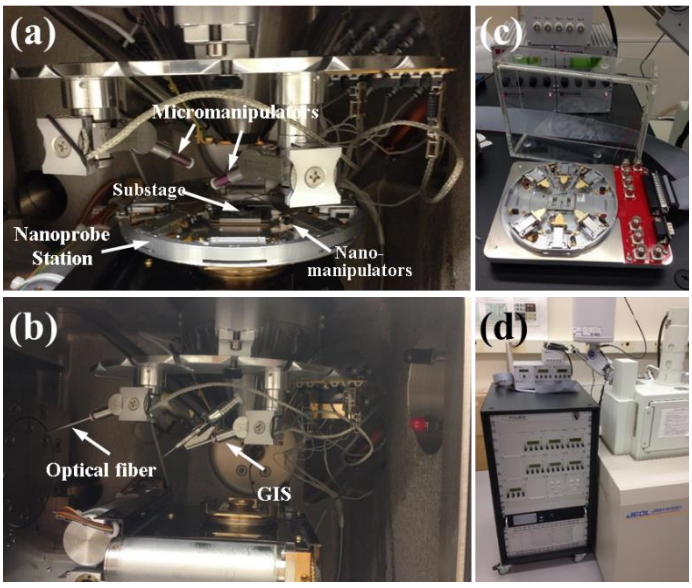


Fig. 2 Photos of (a) installed nanoprobe shuttle and micromanipulators inside the SEM chamber, (b) micromanipulators with add-on tools, (c) nanoprobe shuttle, and (d) electronic rack beside the SEM system.

Most designed functions have been realized and tested including sample transfer, electrical measurement, optical measurement, and electron beam induced deposition. Detail information is provided in following subsections.

1.1 Sample transfer using microgrippers

With the resolution of SEM, nanostructures can be identified. The nanostructures can then be transferred using microgrippers attached to a micromanipulator. Figure 3a shows a SEM image of the microgrippers inside the SEM chamber. With three-dimensional movement of the micromanipulator, we can control the microgrippers to get close to the desired nanostructure, pick it up, and transferred to another location (Fig. 3b). The sample transfer using microgrippers could facilitate device fabrications using nanostructures as building blocks. It is good for transferring vertical oriented nanostructures and nanostructures dispersed on soft substrates.

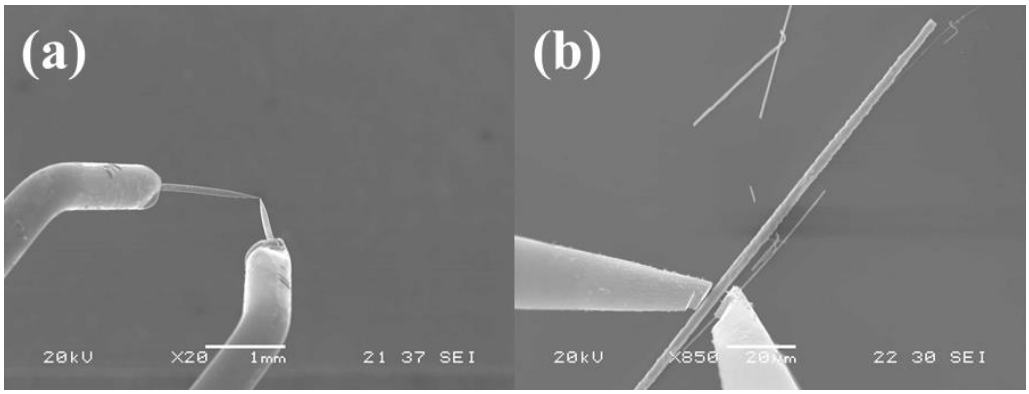


Fig. 3 SEM images of (a) microgrippers at low magnification and (b) microgrippers pick up a nanostructure.

1.2 Electrical measurement using nanoprobe shuttle and parameter analyzer

The electrical measurement is achieved by employing a nanoprobe shuttle with 6 nanoprobes and Keithley 4200A-SCS parameter analyzer. Four of the nanoprobes are able to perform four-/two-probe and transistor measurements on nanostructures. Figure 4a shows a SEM image of four nanoprobes with tungsten tips. With the nanoscale resolution of the nanoprobe movements, the probes can be accurately located onto individual nanostructures (Fig. 4b-c). Currently, the major obstacle for the electrical measurement is the oxide layer on the surface of the tungsten tip. Our tests show the electrochemical etched tungsten tips are prone to be oxidized even with a short time exposed to air. The oxide layer is insulating cause problems of large contact resistance. In the future study, we will focus on solving this problem using prior surface cleaning and *in situ* tip cleaning to remove the oxide layer and to realize accurate electrical measurement.

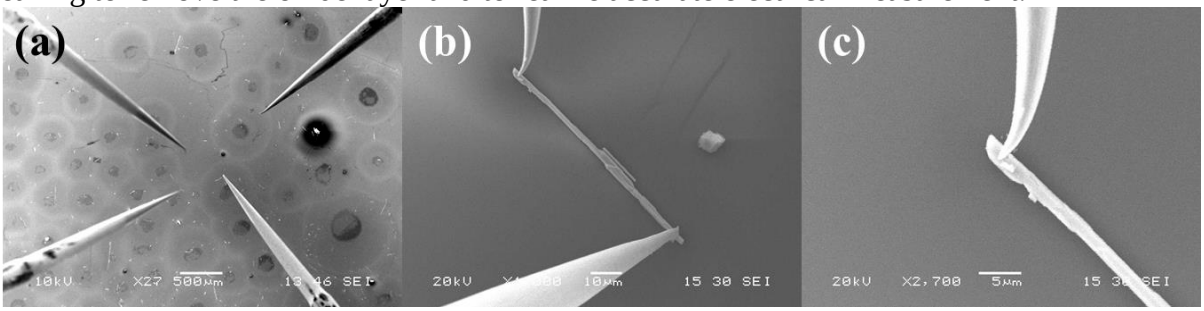


Fig. 4 SEM images of (a) four nanoprobes, (b) two nanoprobes placed onto two ends of a nanostructure, and (c) one probe making contact with the nanostructure**.**

1.3 Cathodoluminescence measurement using optical fiber and spectrometer

Cathodoluminescence (CL) measurement uses electron beam in SEM to excite optical emissions from the sample. To perform the CL measurement, an optical fiber (1mm in diameter) is attached to a micromanipulator to collect the emitted lights. The collected lights are transported to a spectrometer for spectroscopic analysis. The micromanipulator allows for fine maneuverability of the optical fiber to get close to the detected area to enhance the collection of light signals (Fig. 5).

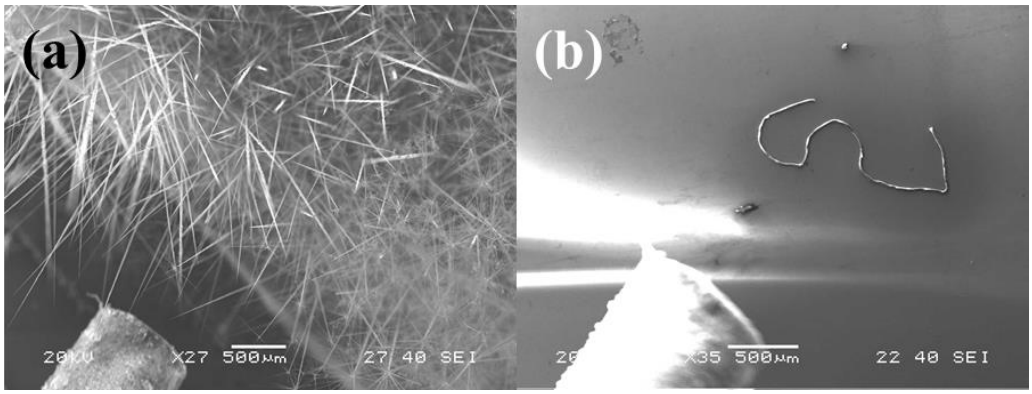


Fig. 5 SEM images of CL measurements using optical fiber getting close to (a) ZnTe nanowire arrays and (b) GaN thin film (features on the surface are dusts).

CL measurement was performed on different samples from ZnO powder, GaN film on a sapphire substrate, to ZnTe nanowires on a SiO_2 substrate. Figure 6 shows typical CL spectra with UV and visible range emissions. Due to the weakness of the light emission from nanoscale materials, the CL measurement requires large spot size of the electrons. The measurement is sensitive to repeating times, integration time, and smooth pixel numbers. Conditions should be optimized for different research projects.

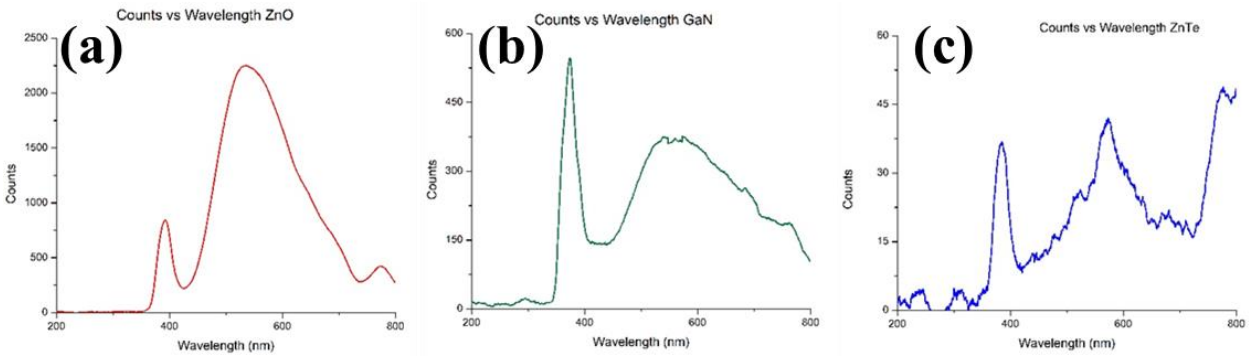


Fig. 6 CL spectra for (a) ZnO powders, (b) GaN thin film, and (c) ZnTe nanowires.

1.4 Electron beam induced deposition using gas injection system

Electron beam induced deposition (EBID) was realized using a gas injection system (GIS) with $W(CO)_6$ as the precursor for W deposition. The GIS is attached onto a micromanipulator, capable of 3D movement to move its nozzle to the vicinity of targeted area. The accurate temperature control and valve control of the precursor allow fine tuning of the flow of the precursor. Electron beam then is able to induce localized deposition specific locations. Figure 7a shows different

patterns of deposition, such as line and rectangle at selected areas. Figure 8b demonstrated deposition across a nanostructure. The deposition can be controlled by growth parameters, such as beam acceleration voltage, beam size, beam dwell time, and magnification, etc. Figure 8c shows one example of parameter study of the effect of beam voltage at a constant spot size of 50. As reveal in Fig. 8c, the deposition at 5KV show a uniform wide line, while with the increase of the acceleration voltage the center line becomes narrower but the overall deposition area increases. This phenomenon matches well with the fundamental theories of electron-matter interaction and secondary electron generation. The deposition can be finely tuned for different research requirements based these principles.

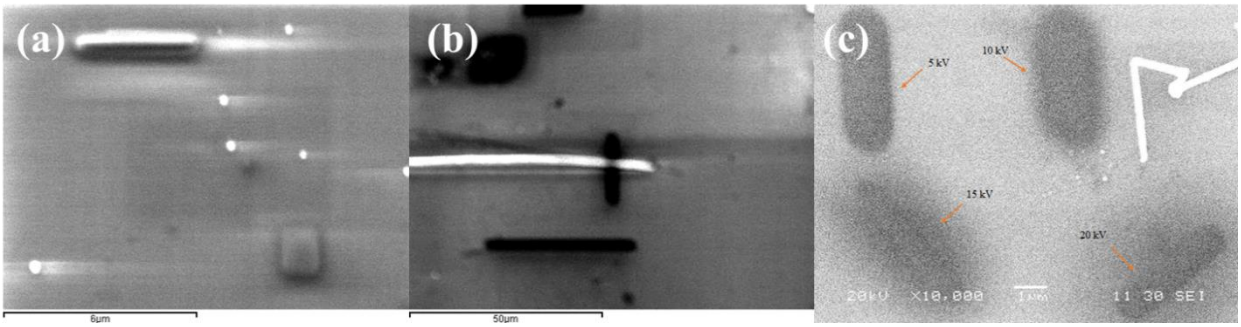


Fig. 7 SEM images of EBID of (a) a line and a rectangle, (b) a line across a nanostructure and (c) lines at different acceleration voltages, from 5KV to 20KV with a constant spot size of 50.

2. Education Enhancement, Student Support, and Dissemination

During the reporting period, undergraduate research apprentice program (URAP) and high school apprentice program (HSAP) fund was requested to support the summer research program at UNC Charlotte. Three students include undergraduate, female student, and high school students participated the summer project in summer 2017. They received research training in safety, sample preparation, materials growth, characterization, measurement and experiment design. They presented their project results in the Charlotte Research Scholars Summer Symposium at UNC Charlotte. Figure 8 shows some of their activities during the summer program, including materials growth using chemical vapor deposition (CVD) method (Fig. 8a), sample preparation (Fig. 8b), demonstration of NPSM (Fig. 8c), and poster presentation in the symposium (Fig. 8d-e).

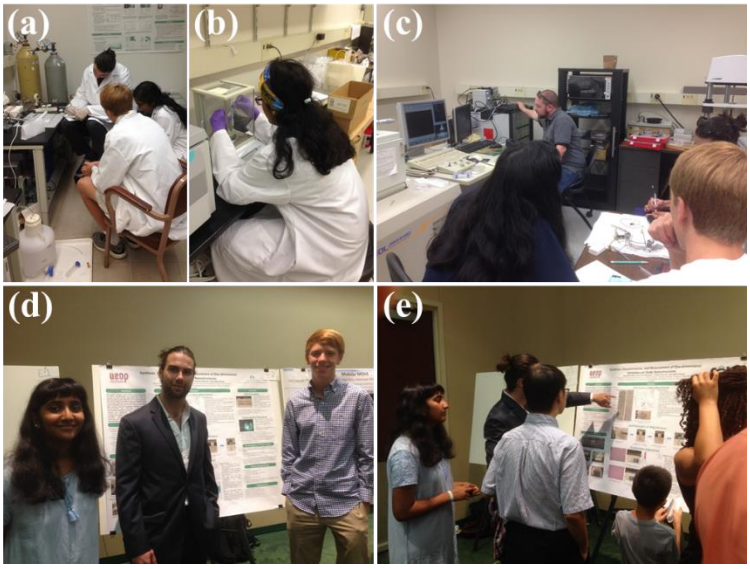


Fig. 9 Photos of activities of URAP/HRSP students and a middle school research volunteer working in the supported summer project. (a) Performing materials growth using a CVD system, (b) preparing sample for materials growth, (c) attending a demonstration of NPSM system, (d) participating the Charlotte Research Scholars Summer Symposium, and (e) presenting the poster to a judge and other audience.

In summer 2018, the NPSM system hosted the 2018 URAP and HSAP projects. One undergraduate and one high school students have worked on the NPSM system extensively to test different functions from sample transfer, electrical measurement, CL measurement, to EBID. Two research groups are currently applying the NPSM system in their research. Two graduate students get trained and start to explore the capabilities of the NPSM system. Demonstrations of NPSM system have been provided to other summer students. The NPSM system will be integrated into PI's undergraduate materials course and microscopy course, as well as the graduate materials characterization course from Fall 2018. Workshops demonstrate the NPSM system for high school students will be completed in August 2018.

3. Development of Training Manuals and Instructions

To serve the internal and external facility users and provide education courses to students (including graduate, undergraduate, and high school students), training manuals and instruction notes have been developed during the funding period. They will provide the users and audience a good procedure guide for operation, brief introduction of working principles, and technical supports. Some of current drafts of manuals and instructions are shown in the "Appendices" part. They will be further modified to fit the users' needs with the continuous testing and optimization of operation procedures and parameter settings. More manuals and instructions are under preparation with further utilization of the NPSM system in different research projects.

Appendices

Appendix 1. Basic Operation Procedures

1.1 MicroManipulators (MM3a) Loading and Operation:

1. E-board stays on the bracket with 5 ports and extra splitters for add-on tools.
2. Connect the E-board using the cable attached to the SEM backside flange.
3. Connections:
    a. E-board
        i. Port 1: vacuum splitter (VS) to left-side MM3a (top) and GIS V control (side). The MM3a is used to attach the GIS tool.
        ii. Port 2: GIS T control
        iii. Port 4: VS to right-side MM3a (top) and MGS (side).
        iv. Port 5: FMS
    b. AirSplitter (AS 5) backside
        i. #1: NanoController (NC) 1
        ii. #2: TCS
        iii. #4: NC 2
        iv. #5: FMS
4. Loading add-on tools: add-on tools need to be mounted on an MM3a or attached to it. When loading the tools onto the bracket, the tip of the MGS should be facing up on the bracket. In the SEM chamber, the bracket is loaded onto the flange support upside-down. **Note:** For the GIS, its nozzle should always face downward to avoid the precursor from getting into the central over-flow tube. So mount the GIS onto the MM3a after the bracket is loaded onto the flange support. (This step needs to practice).
5. Operation using NC 1 and NC 2.

	↻ (clockwise)	↺ (counterclockwise)
Knob #1	Right	Left
Knob #2	Up	Down
Knob #3	Extend	Retract
NC 1 Knob #4 (Valve control for GIS)	Open (partially, slow, speed 4, c001)	Close (all the way, fast, speed 6, C064)
NC 2 Knob #4 (Gripper control for MGS)	Close (Max: speed 4, c001. Same for open)	Open (Opening limit: ~ 6 μm)

6. Change between preset speeds: press “Up” and “Down” to select different Speed Pages for Knobs #1-4. Note the Page Name (the upper right corner) is not the real speed number. On one page, different knobs can have different speeds.
   Coarse mode: Speed 4: c001, Speed 5: c008, Speed 6: c064
   Fine mode: f001-f128

ATTENTION: For vertical motion (B axis), take caution that downward motion will take larger steps due to the additional weight of the add-on tool.

7. Change the Speed Setting: Use “Up”/”Down” button to select the Speed Page to change. Press “Menu” button on NC then press “UP” or “Down” to get to the “15. Speed” menu. Turning the corresponding knob to change its speed setting. Press “Menu” to confirm and return.
8. TSC: Push button to turn heat on. Adjust heating temperature via knob.
9. Approach to the surface:
    a. Locate the tools above the substrate surface (a few mm away).
    b. Focus on the tip of the tool using short WD (turn ↻ “Focus” knob) to locate the front of the tool.
    c. Next, focus onto the sample surface using long WD (turn ↺ “Focus” knob). Bring the focus point up slightly over the surface (turn ↻ “Focus” knob 1-2 turns) with sample surface still clear in the image.
    d. Lower down the tool slowly to the surface until its tip becomes clear in the image. Use coarse speed when it is far away, and slow speed, then fine speed. Increase magnification gradually to see the details and adjust the tip position to the center of the image. **Note:** Both “Down” and “Extend” movements will move the tool closer to the sample surface. Use them carefully.

1.2 Procedures for Preparing GIS:

1. Opening the reservoir: Open the reservoir by turning the cap ↺ slightly to stop. Watch the metal dots on the side of the cap while turning. Pull the cap out slowly.
2. Loading the precursor: Make sure to not let the precursor material get into the 'overflow' tube inside the reservoir.
3. Closing the reservoir: Put the cap onto the reservoir with the metal dots aligned with the slots on the reservoir. Push the cap all the way in and turn the cap ↻ slightly to stop.
4. Be careful while handling the GIS, after filling and closing the reservoir, as to not let the precursor material fall into the overflow tube. (The GIS nozzle should always face downward)
5. The valve should be open during pumping down (when using solid/liquid precursors) in order to remove most of the air from the reservoir. Heating should be off. Opening the valve (turning Knob 4 ↻) should be done with speed 4 c001 steps. When the valve is closed tight, there are a number of steps to travel to open the valve in order to change the chamber pressure. The number of steps depends on the valve temperature.
6. After the SEM system is pumped down to normal operation condition, tightly close (turning Knob 4 ↺) the valve using speed 6 (c064).
   ATTENTION: Opening the valve in speed 6 (c064) can kill the electron source. Therefore, you must only use c064 to close with the correct direction. Best seal is achieved at temperatures of 40°C and above.

7. Turn the knob to change the setting temperature (clockwise to increase and counterclockwise to decrease). Pressing the knob toggles the controller's heat on or off.
8. Before the deposition, open the valve using speed 4 c001.
9. After the deposition, tightly close the valve using speed 6 c064. Turn off the heat.

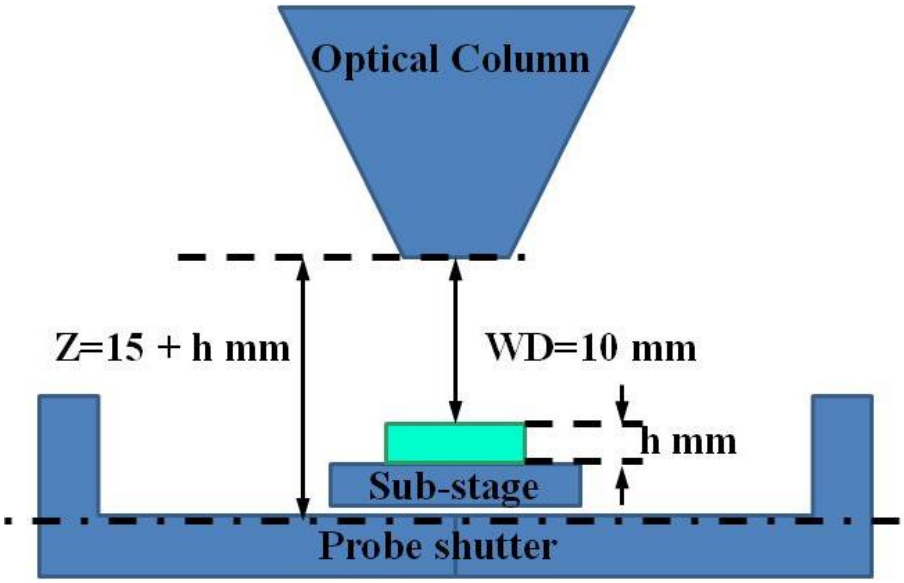
Note: There is no indication if the valve is closed or not. Use speed 6 c064 for several turns to make sure the valve is tightly closed. There is no worry for overtightening.

Adjusting Step Size with Amplitude Wizard

1. Move the stage down to z= 40 mm, to ensure ample testing room.
2. Opening the Amplitude Wizard: Press the menu button once to open. Use "Up"/"Down" to scroll to menu setting "07 Amplitude Wizard".
3. Setting up Wizard: Use knob #2 to select which axis to adjust. Typically this will be Axis B (Up/Down). Turn knob #3 to the right until the arrow on the screen moves all the way to the right, the Wizard is now activated.
4. Using the Wizard: Once the Wizard is activated, the machine will beep indicating the tool has started to move. Watch both the IR Camera and SEM carefully. Once movement is observed, press either the "Up" or "Down" button on the Nano device. Immediately after pressing, the tool will start to move downward. Again, watch both the IR Camera and SEM for the movement. Press either the "Up" or "Down" button when motion is observed.
5. Once the Amplitude Wizard is completed, two numbers will be displayed for the minimum amplitude in the positive and negative direction. Typically for the B axis, the minimum amplitude in the positive direction will be greater than the minimum amplitude for the negative direction. This difference in value compensates for the force of gravity in the down direction.
6. Testing new minimum amplitudes: Press "Menu" to return to the control screen. Test the Up/Down direction (recommended speed setting 5). The differences between steps for positive and negative movements should be smaller. However, on higher coarse speeds (speed 6) there will still be a difference in the Up/Down direction.
7. Repeat the process if there is still a large difference between the Up and Down directions.

Note: Every time you change the attachment to the MM3a, you need to use the Amplitude Wizard to compensate the weight change in UP/Down direction.

1.3 NanoProbe Shuttle Loading and Operation:



How to approach the probe tip onto the surface?

1. First, locate the probe tip < 1mm above the sample surface.
2. Under the SEM, use the focus to find the front of the probe tip. Turn "Focus" knob ↻ (clockwise) reducing working distance (WD) to focus on the probe tip. Identify and remember the tip location above the sample.
3. Turn "Focus" knob ↺ (counterclockwise) increasing WD to focus onto the sample surface.
4. Move the probe down slowly toward the surface of the specimen (speed 2?) until the tip is clearly shown.

1.4 Spectrometer with AvaSoft 8 program:

1. Setting up:
    a. Choose an optical fiber that is thin enough to be clamped onto an MM3a and flexible enough as to not disrupt the motions of the MM3a. Make sure the optical fiber can move within SEM chamber without disrupting any components.
    b. Connect the fiber to the port on the right internal wall of the SEM. Connect another optical fiber from the external port of the SEM to the spectrometer. Use adapters where needed.
    c. Connect the spectrometer usb cord to an appropriate PC and launch the 'AvaSoft 8' program.

d. Changing Optical Fiber Slit: On the Avantes Spectrometer, remove two screws holding current slit with screwdriver in slit kit. Use the Torx T8 to pick up the slit from the Avantes machine, place into the empty space in the slit kit, and unscrew the Torx T8. Then pick up desired slit with Torx T8, place in Avantes, place two screws back, and remove Torx T8.
e. **Note:** Increasing slit size will increase signal, however it increases chance of saturation.

2. Using 'AvaSoft 8' to measure a spectrum:
    a. Adjust the micromanipulator until the optical fiber is within range of the testing specimen. Make sure to not block the electron beam with the optical fiber, this will disrupt the signal.
    b. On the AvaSoft 8 program, click the start button at the top left to begin measuring.
    c. Click the auto-adjust button in the graph window to automatically adjust the x and y axes to meet the measured spectrum. Make sure the graph is set to measure scope.
    d. In order to find a signal, spotsize will likely need to be at 70 or above. If no signal is detected either the optic fiber is not pointed at the sample or aperture should be increased. Recommended electron beam voltage is 20 kV.
    e. In the settings of the AvaSoft 8 program adjust the integration time, the number of counts, and the number of smoothing pixels in the settings until a clear graph is obtained. Increasing integration time will increase signal strength, increasing average count will increase smoothness of graph, and increasing smoothing pixels will also increase smoothness. However, increasing smoothing pixels past a certain number will decrease resolution (the number of smoothing pixels is dependent on optic fiber diameter).

    Note: If the saturated label appears in the status bar next to the graph, decrease the integration time. If at the lowest integration time the data is still saturated, a smaller optic cable must be used.

    f. Adjust the SEM magnification and increase the spotsize to receive a stronger signal. In order to optimize magnification, zoom in and out until the strongest signal is obtained.
    g. When a desired signal is acquired, turn off the electron beam on the SEM. The signal should flatten but 'noise' will still be present. Click the 'Save Dark Data' button to save the noise as a reference.
    h. Turn back on the electron beam. Click the 'Save Reference Spectrum' button to save the unfiltered spectrum.
    i. Change the graph to measure scope minus dark. This will filter out the 'dark' noise and provide a smoother measurement.
    j. Repeat steps g. through i. whenever the SEM image is changed or any parameters in the AvaSoft settings are changed.
    k. The data and graph can be exported via the file tab in the graphical window.

1.5 Operating Keithley System and Clarius Program:

1. Connecting testing unit to the Keithley system:
    a. Connect the appropriate testing unit pin outs to the relative SMU force inputs of the Keithley system via the provided triaxial cables. Note that SMU 1 contains a preamplifier for use with small signal devices. For ground unit inputs, use the GNDU Force input of the Keithley system.
2. Running Clarius on the Keithley system:
    a. Open the Clarius program.
    b. Select the appropriate test from the testing library.
    c. Click ‘Configure’ to open the configuration window. Insert appropriate testing parameter values. Make sure the selected SMU inputs selected in the Clarius system match that of the triaxial cables connected to the testing unit.
    d. Click ‘Run’ to begin the test.
    e. Click ‘Analyze’ to open the analyzation window. Data from the test will be tabulated within a table in the analyzation window. Relative data points will also be graphed within the analyzation window. All data and graphs can be saved or exported for later analysis.

1.6 Keithley-to-NanoProbe Station Connection and Control

Cable connections from NanoProbe Station to Keithley. The leads from the flange connect to the Signal Switching Units (SSUs). From there the Keithley can be connected to the triax OUT connectors. Using the software, the tip signals can then be routed to the BNC ports on the rack's front panel, to the triax OUT ports (i.e. to the Keithley) or to the LCT. That covers the actual measurement signals.

Since we have 6 tips: Tip 2, 3, 4, and Tip 6, 7, 8. Tip 2 will be saved for connection of possible MicroGripper. So we will use Tip 3, 4 and Tip 6, 7 for electrical measurement. We can use the two-wire resistor measurement to test if each tip is connected to the corresponding port on the front panel.

Use Remote Module in APT to control Keithley. The Keithley Remote Module allows driving the Keithley from within the APT software interface. The required configuration steps are described in the APT manual. In order to remote-operate a Keithley 4200 parameter analyzer, it is necessary to launch the KXCI terminal on the Keithley’s PC. Please note that the KXCI interface cannot be run simultaneously with the KITE software, and it has to be configured for GPIB interfacing. Once the Keithley is configured and the KXCI interface is running, it is automatically recognized by the APT software. Clicking the ‘activate’ button initiates communication.

About the Tip-SMU mapping, my understand is it will not affect the hardware connection. It is used for display in the Remote Module, i.e., when you choose a tip in the program the

corresponding SMU will also be displayed if the mapping is on. This only needs to be done once the hardware connections are completed.

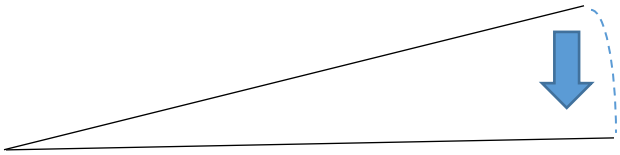
Appendix II. Nanoprobe Shuttle Installation

Nanoprobe Shuttle Installation

1. Carefully remove the container from the shuttle by pulling out the side pins.

ATTENTION: The container has no bottom. Handle with care and do not pick up the container from the bottom to prevent damage to the shuttle.

2. For APT testing outside of the SEM:
    a. Plug in the Ribbon Cable located on side of Kleindiek computer into the shuttle.
3. For APT testing inside the SEM:
    a. Remove the APT from the shuttle by sliding the APT, unplugging the controller connection, and lifting the APT from the shuttle.
    b. Mount the APT via the attached SEM mount to the SEM stage, and plug in the controller connection found inside the SEM chamber.
4. Turn on the Kleindiek computer system by turning on the power strip and turning on the power knob on the top right of the Kleindiek controller tower.
5. Open the Advanced Probing Tools software on the desktop. Select which probe to move in the top right by clicking on the diagram. The sample stage can be selected as well.
6. Connect the Shutter Express usb cord to the usb port for the Kleindiek.
7. The Shutter Express is used to control motion on the B Axis (Up/Down). Turn the knob clockwise to move the probe up, counter clockwise to move the probe down. Note that the B axis motion is not linear.



B axis is rotational on the Z axis.

8. A and C axis rotations are controlled on screen. Hold down the right mouse button and drag the mouse in the desired direction. A axis motion moves the probe left/right, C axis motion extends/retracts the probe.
9. Speed Settings: Speed can also be changed via the software on a preset scale 1-6. 1 and 2 are fine motions, 3 is coarse with fine, and 4, 5, and 6 are coarse motions. Individual axis speeds range from f001-f128 to C001-C064.

Connecting With Keithley Computer

1. The APT can be connected to the Keithley. Use triaxial cables to connect to the desired SMU force ports on the back of Keithley. Note that connections are dependent on the test to be performed and which probes will be used.
2. In order for the SMU cables to connect to the Shuttle/Kleindiek control tower, attach adapters to the triaxle SMU cable.
3. For APT testing outside of the SEM:

    a. Connect the triaxial cables from the desired ports on the Shuttle to the appropriate SMU ports on the back of the Keithley computer.
4. For APT testing inside the SEM:
    a. Connect the triaxial cables from the desired ports on the Kleindiek control tower to the appropriate SMU ports on the back of the Keithley computer.
5. Use the Keithley configuration window to map the SMU inputs for the desired test.

Appendix III Cathodoluminescence Procedures

Equipment:

- Avantes AvaSpec-2048L Spectrometer
- JEOL JSM-6480 SEM
- Kleindiek MM3A micromanipulator
- Optical Fiber
- Computer with AvaSoft 8 program

3. Setting up:
    a. Choose an optical fiber that is small enough to be clamped onto a micromanipulator (MM3A) and flexible enough as to not disrupt the motions of the MM3A. Make sure the optical fiber can move within the SEM chamber without disrupting any components.
    b. Connect the fiber to the port on the right internal wall of the SEM. Connect another optical fiber from the external port of the SEM to the Avantes AvaSpec-2048L spectrometer. Use adapters when needed.
    c. Connect the spectrometer usb cord to an appropriate PC and launch the 'AvaSoft 8' program.

    Note: When using the Kleindiek PC, connect the usb cord to the port near the power button.

    d. Changing the Optical Fiber Slit: Choose a slit size that will produce a strong signal without saturation. To remove a slit, first remove the two screws holding the current slit on the spectrometer with the provided Torx key. Use the Torx T8 to maneuver the slits between the spectrometer and the slit kit. Remove the undesired slit and replace it with an appropriate slit size. Tighten the two screws to hold the new slit in place.

    Note: Increasing the slit size will increase the signal strength, but also increases the chance of saturation.

4. Positioning:
    a. Using the SEM, maneuver the sample to a suitable testing area (Flat areas are easier for capturing wavelengths). Adjust the sample height to a working distance (WD) of approximately 20 mm (Space is needed to maneuver the optical fiber above the sample).
    b. Using the Kleindiek controller, move the optical fiber until it is in view of the SEM above the sample. Maneuver the fiber downwards so that the tip of the fiber points towards the sample and is fixated on the center column of the electron beam. Make sure to not block the electron beam with the optical fiber, this will disrupt the signal.
5. Using 'AvaSoft 8' to measure a spectrum:

a. On the AvaSoft 8 program, click the start button at the top left to begin measuring, as shown in Figure 1.
b. Click the auto-adjust button in the graph window to automatically adjust the y axis to meet the measured spectrum as shown in Figure 1. Make sure the graph is set to measure scope. Manually adjust the x axis to show the wavelength range desired by selecting the drop-down button in the scale options shown in Figure 1. Select edit, then adjust the axes accordingly.

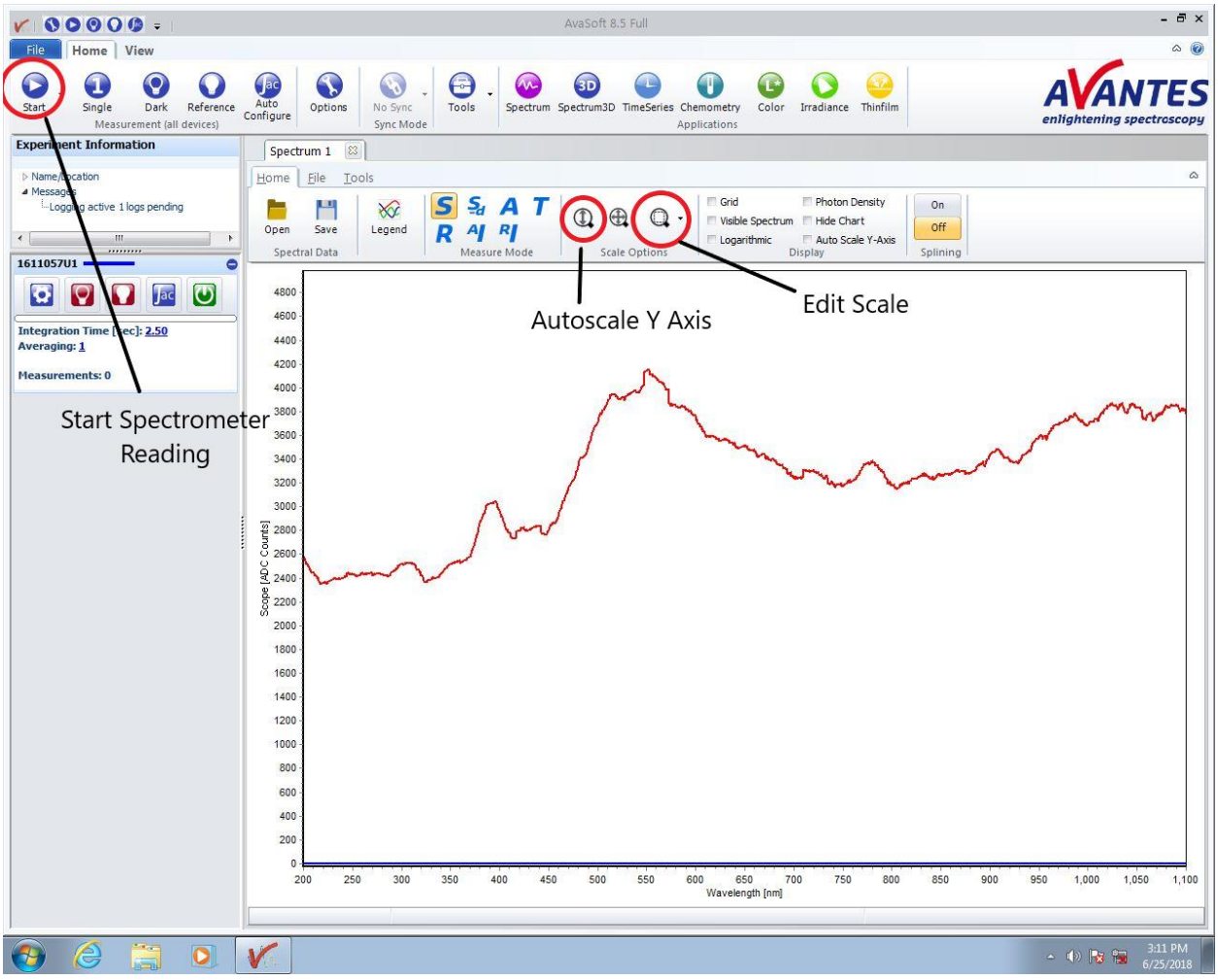


Figure 1 depicts the start features and scaling options for the Avasoft Program.

c. To produce a readable signal, spotsize (SS) will likely need to be at 70 or above. If no signal is detected, the optic fiber may not be pointed at the sample fixated below the electron beam or the aperture may need to be increased to 3. Recommended electron beam voltage is 20 kV.
d. In the settings of the AvaSoft 8 program adjust the integration time, the number of counts, and the number of smoothing pixels in the settings until a clear graph is obtained (Figure 2). Increasing integration time will increase signal strength,

increasing average count will increase the smoothness of graph, and increasing smoothing pixels will increase smoothness but decrease resolution. Note that the number of smoothing pixels is dependent on optic fiber diameter, these values can be found in the spectrometer user's manual.

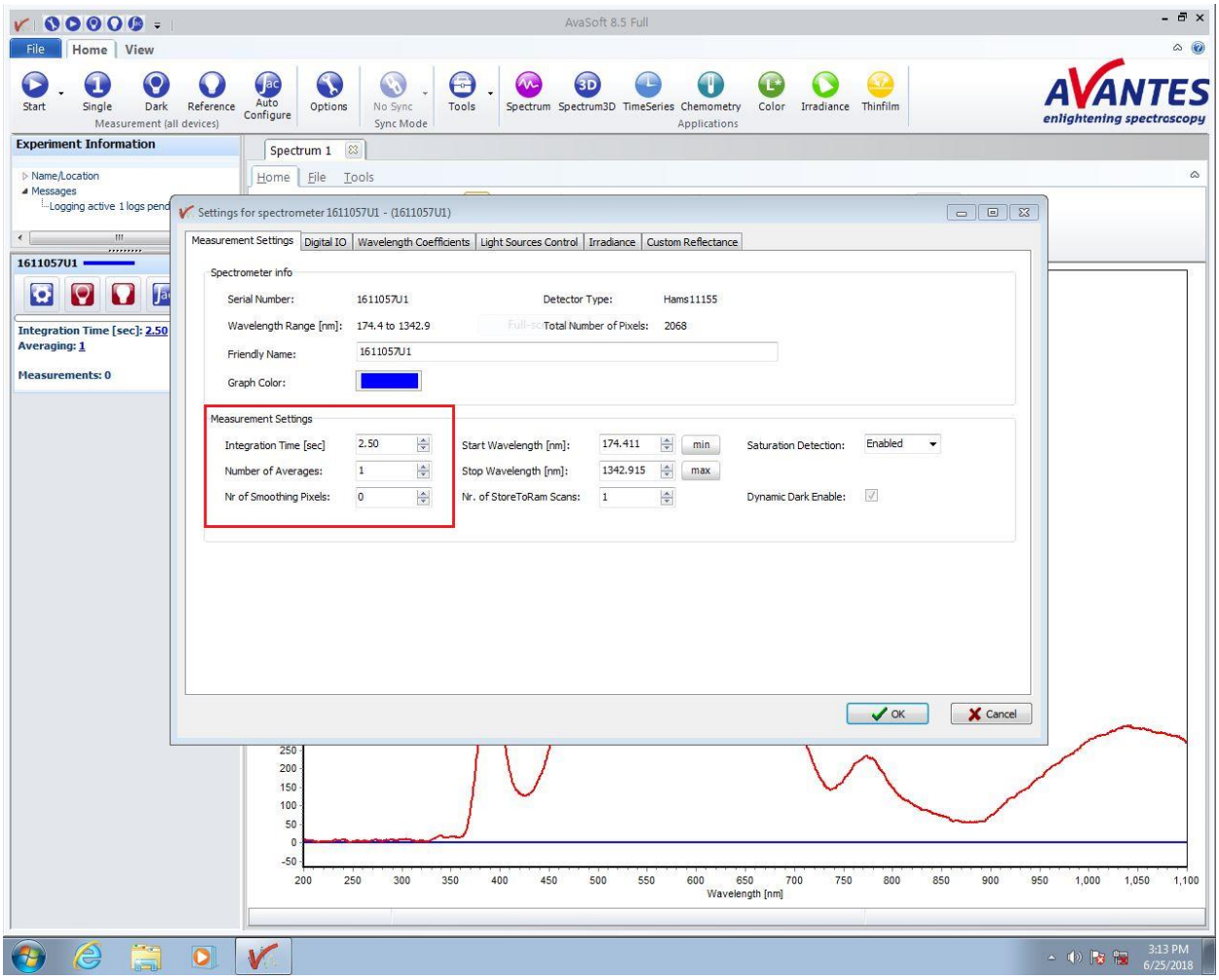


Figure 2 demonstrates the settings menu for the spectrometer.

Note: If the saturated label appears in the status bar next to the graph, decrease the integration time. If at the lowest integration time the data is still saturated, a smaller slit size must be used.

e. Adjust the SEM magnification and increase the spotsize to receive a stronger signal. In order to optimize magnification, zoom in and out until the strongest signal is obtained.
f. When a desired signal is acquired, turn off the electron beam on the SEM. The signal should flatten but 'noise' will still be present. Click the 'Save Dark Data' button to save the noise as a reference per Figure 3.

g. Turn back on the electron beam. Click the ‘Save Reference Spectrum’ button to save the unfiltered spectrum per Figure 3.

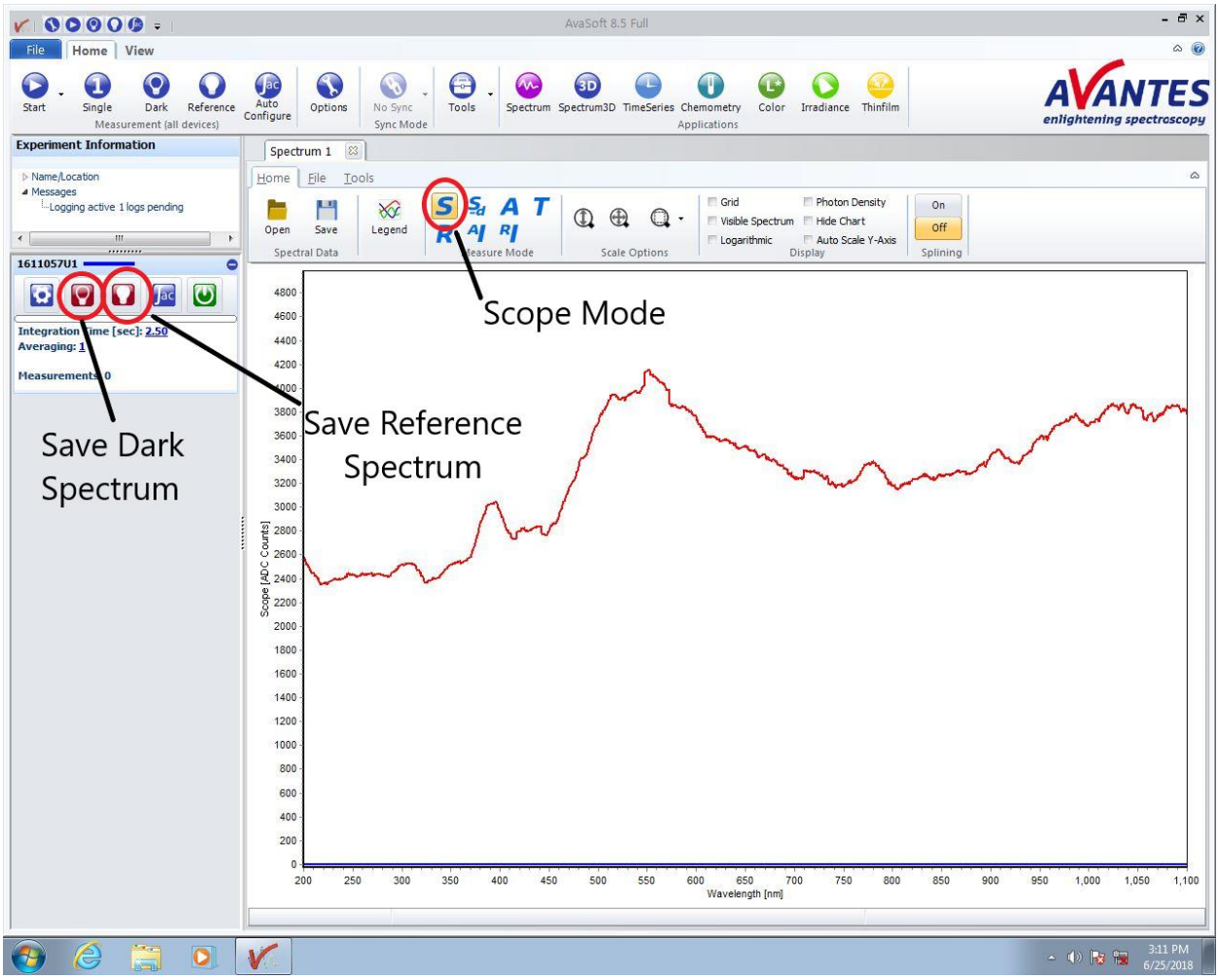


Figure 3 shows how to get a proper spectrometer measurement with dark and reference spectrum features.

h. Change the graph to measure scope minus dark, as shown in Figure 4. This will filter out the ‘dark’ noise and provide a smoother measurement.

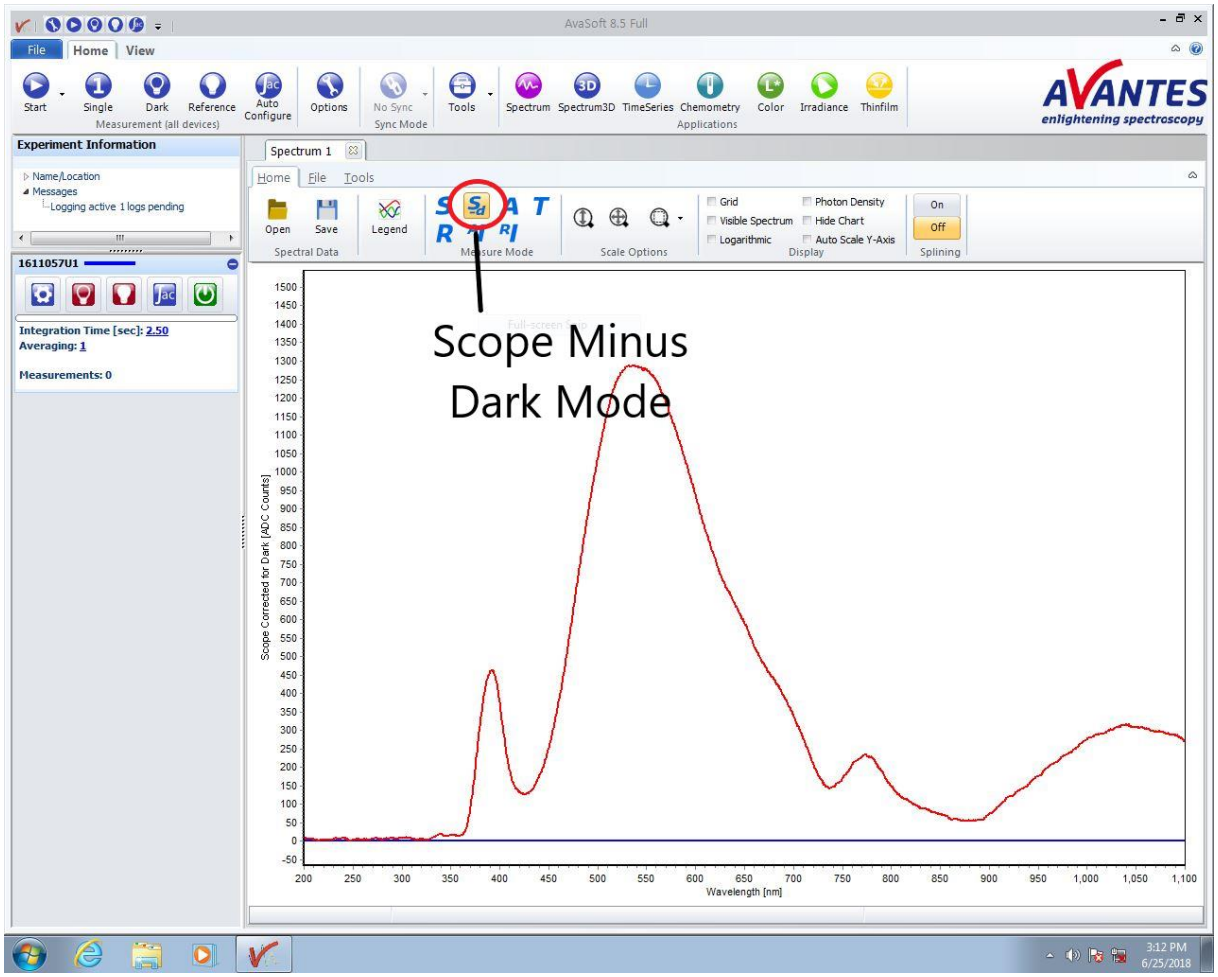


Figure 4 illustrates the scope minus dark feature.

i. Repeat steps g. through i. whenever the SEM positioning or settings are changed or any parameters in the AvaSoft settings are changed.
j. Select single in the top left to get a single measurement if desired.
k. The data and graph can be exported via the file tab in the graphical window.

Appendix IV Electron Beam Induced Deposition (EBID) Procedures

Equipment:

- JEOL 6480 SEM
- Kleindiek MM3A Micromanipulators
- Gas Injection System (GIS)
- Energy Dispersive Spectroscopy (EDS)/INCA

Parameters:

- Acceleration Voltage: 5 – 20 kV
- Spot Size: 20 – 70 (larger size induce faster deposition rate but lower feature resolution)
- Source Heating Temperature: 50℃ (adjustable for flow control)

Procedure:

1. Open GIS valve slowly (speed = C001): ~ 400 units
2. Evacuate SEM chamber
3. Close GIS valve quickly (speed = C004) and heat GIS to 50℃
4. Position GIS into frame of view using a large working distance (WD)
5. Focus on sample surface
6. Capture pre-deposition image of sample surface
7. Center the electron beam through hole of GIS nozzle on desired position above the sample surface
8. Open GIS valve slowly
9. Use INCA program to control electron beam for deposition for line or area depositions.
10. Close GIS valve quickly