REPORT DOCUMENTATION PAGE

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Approved for public release; distribution is unlimited.

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14. ABSTRACT

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RPPR Final Report
as of 07-Dec-2021

Agency Code: 21XD

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Agreement Number: W911NF-21-1-0024

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Final Report for Period Beginning 28-Dec-2020 and Ending 27-Aug-2021

Title: Snapshot Hyperspectral Imaging and Scanning Electrochemical Microscope for Nanoelectrode Reshaping Studies

Begin Performance Period: 28-Dec-2020

End Performance Period: 27-Aug-2021

Report Term: 0-Other

Submitted By: PhD Christy Landes

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Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees:

STEM Participants:

Major Goals: This proposal requested funds to build an integrated scanning electrochemical and high-speed snapshot darkfield spectroscopic microscope capable of simultaneous electrochemical imaging, manipulation, and high sensitivity spectroscopy of single plasmonic nanoelectrode structures.

Accomplishments: All components required to build the proposed SECM instrument have been acquired. Details are provided in the uploaded document.

Training Opportunities: Although not directly supported through this DURIP equipment grant, a graduate student was trained in advanced optical and electrochemical methods as well as introduced to more advanced instrument planning including purchasing and budgeting.

Results Dissemination: Nothing to Report

Honors and Awards: Nothing to Report

Protocol Activity Status:

Technology Transfer: Nothing to Report

Partners

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I certify that the information in the report is complete and accurate:
Signature: Christy Landes
Signature Date: 12/6/21 9:46PM

Final Performance Report

Snapshot Hyperspectral Imaging and Scanning Electrochemical Microscope for Nanoelectrode Reshaping Studies

Christy Landes, Rice University

The funds provided by this DURIP grant were used to acquire the components for a hybrid snapshot hyperspectral imaging/scanning electrochemical microscopy setup and a nanoelectrode manufacturing station. As shown in Figure 1A, the microscope body (1) is set up with a manual stage (2) for coarse positioning of the sample and a piezo-stage with a piezo-controller (3) for scanning the sample. A second piezo-stage controls the approach of a nanoelectrode tip to the sample, while the tip is connected to a bipotentiostat (4) to measure electrochemical currents from individual nanoparticles. A widefield total internal reflection excitation path (5) is used to illuminate the nanoparticles, using the white light laser purchased from NKT Photonics (Figure 1B). This laser was chosen because it has a wide spectrum in the visible region, ranging from 450 nm into the infrared, allowing hyperspectral imaging using a complementary metal oxide semiconductor (CMOS) detector (Figure 1A, 6) to characterize nanoparticle shapes and sizes.

The nanoparticles are illuminated using a widefield total internal reflection excitation geometry, enabled by a high numerical aperture (NA) oil immersion objective (Figure 1C). A major challenge in simultaneous tip-scanning and hyperspectral measurements is scattering of light on the nanoelectrode tip, which would cause a high background in the hyperspectral measurement. To avoid this background, the nanoparticles are illuminated using an evanescent field with a penetration depth less than the working distance of the scanning electrochemical microscope, thus only illuminating the nanoparticles, and not the tip. This evanescent field is achieved using a 1.54 NA objective, which allows the laser to

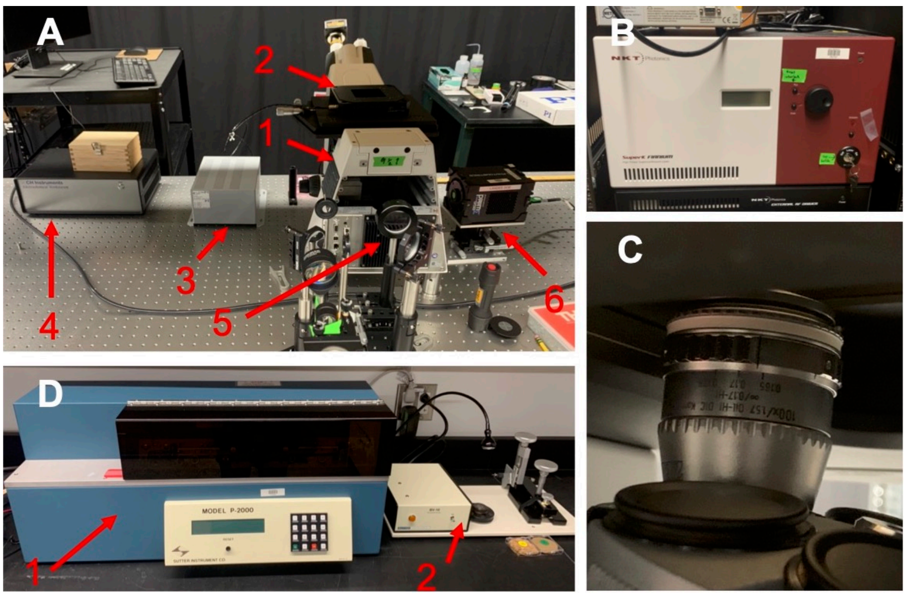

Fig. 1: Components purchased using DURIP fund. (**A**) Components for microscope setup. 1: Microscope body, 2: Manual and piezo stages, 3: Piezo controller, 4: Bipotentiostat, 5: Optics for widefield illumination, 6: CMOS camera (not purchased using DURIP funds). (**B**) White light laser. (**C**) High-NA objective. (**D**) Nanoelectrode manufacturing station. 1: Pipette puller, 2: Polisher

be introduced at a shallow enough angle to cause total internal reflection at the substrate-electrolyte interface.

We set up a nanoelectrode manufacturing station for the scanning electrochemical microscopy component of the hybrid setup (Figure 1D). A platinum wire is inserted into a glass capillary and attached to the pipette puller. By heating the capillary, the glass begins to melt, and the pipette is pulled to generate a nanosized glass tip, with the platinum wire embedded. Once cooled, the tip of the molten glass is then polished to expose the platinum wire, generating a nanosized, perfectly smooth electrode tip. These small electrode tips are necessary to generate a sufficiently low spatial resolution to measure currents from individual nanoparticles.

We have not yet completed the scanning electrochemical part of this setup due to long lead times for components and restrictions on laboratory capacity imposed by Rice University due to the COVID-19 pandemic. All necessary components have now been received and a dedicated graduate student is making excellent progress on completing the system.