Development of a nano-scale scanning impedance micrsocopy system

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There is growing recognition that the properties of many materials depend strongly on nano-scale structure and processes. Purposeful manipulation of materials on the nano-scale can lead to unique properties, spawning novel candidates for an increasing range of applications. Continued progress hinges critically on the ability to perform nanometer scale characterization. Specifically in the domain of electrochemical systems, an ability to probe fundamental electrochemical processes at the nanometer scale is desired. In this presentation, we will disclose the development of an impedance microscopy system that allows highly localized measurements of electrochemical impedance to be acquired at the sub-micron length scale. Nanometer scale visualization and measurement of impedance is valuable for a wide variety of electrochemical investigations, ranging from fundamental electrode/electrolyte studies to Li-ion battery and fuel cell systems to coatings and corrosion research.

Electrochemical impedance spectroscopy (EIS) is a valuable characterization tool in the research and development of diverse electrochemical systems. In fuel cell and battery research, it has been used to distinguish between various sources of cell loss; for example ohmic losses in the electrodes and electrolyte, activation overpotentials due to reaction kinetics, and mass transport effects. While these measurements resolve the electrochemical phenomena *mechanistically*, they cannot resolve the phenomena *spatially*. In other words, standard EIS measurements produce bulk, or system averaged results.

In contrast, our scanning impedance microscope system uses a local probe to obtain spatially resolved EIS measurements at the sub-micron scale. As diagrammed in figure 1, the system is constructed from a commercially available atomic force microscopy (AFM) system coupled to an electrochemical impedance measurement system. A conductive tip is used for the AFM tip and serves as the probe electrode for the EIS measurements. Successive measurements across a sample surface are obtained by moving the AFM tip. Measurements are currently obtained in a point-by-point fashion rather than in a continuous scanning manner. Single frequency or complete EIS spectra can be obtained during the scans. Custom developed software interfaces automate the communication and synchronization between the hardware systems.

Current resolution of the impedance microscope system is demonstrated at below 200nm. Figure 2 shows an example measurement output from the system of a gold structure on a silicon substrate. An insulating silicon nitride film coats the silicon substrate. The gold feature is approximately 2-3 μm in width and connects to a bulk electrode.

Details on the resolution capabilities of the system will be presented. Application of the measurement system for spatially resolved electrochemical investigations of fuel cells on the sub-micron scale will be presented.

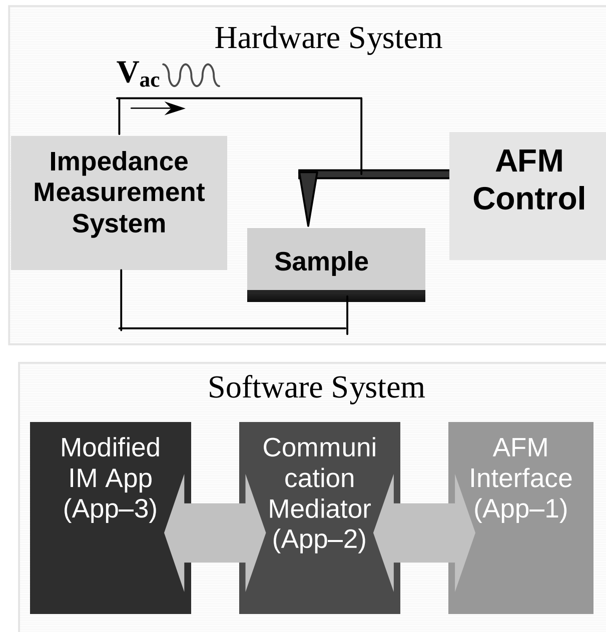

Figure 1: Schematic diagram of the hardware and software configurations for the scanning impedance microscope system. A conductive AFM tip provides the local probe electrode for the impedance measurements. The counter electrode is a bulk electrode attached to the sample of interest.

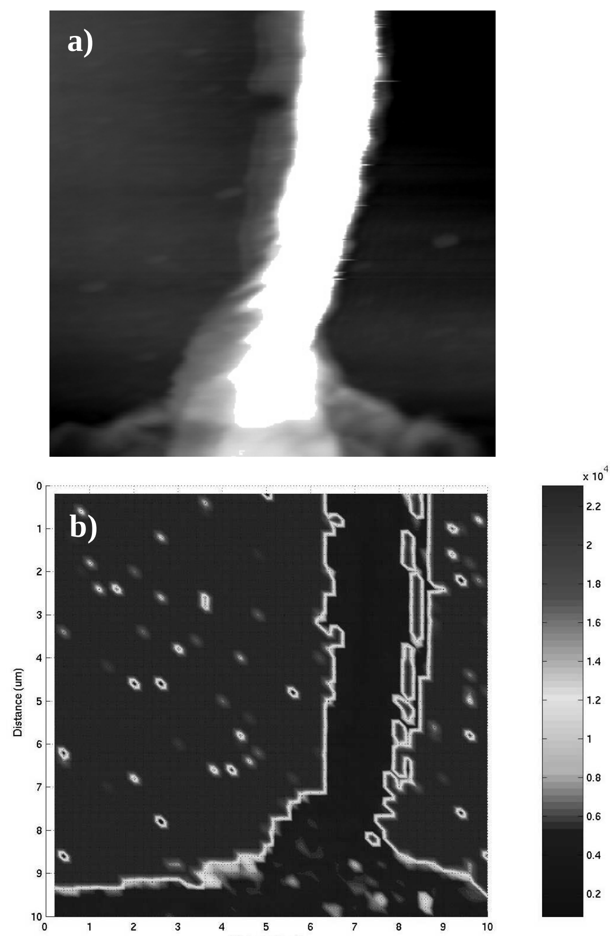

Figure 2: a) AFM topography image of a gold feature defined on a silicon substrate. The silicon substrate is coated with an insulating nitride film. The gold finger connects to a large area bulk electrode at the bottom of the image. Gold finger is 200 nm high. b) Impedance microscopy image of the same area. The real component of the impedance is plotted at a frequency of 1000 Hz. A pixel step of 200nm in x and y has been used for this scan, resulting in 2500 individual impedance measurement points across the image. Both images are 10 μm x 10 μm.