Simultaneous Topographical and Electrochemical Mapping using Scanning Ion Conductance Microscopy–Scanning Electrochemical Microscopy (SICM-SECM)

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Lately, scanning ion conductance microscopy (SICM), has emerged as a versatile non-contact imaging tool. To obtain spatially-resolved electrochemical information, scanning electrochemical microscopy (SECM), also known as the chemical microscope, has been developed [1]. Hybrid SICM-SECM techniques have been developed, in which the SICM compartment provides the accurate probe-sample distance control, while the SECM compartment measures the faradaic current for electrochemical information collection [2].
In this work, we demonstrate the use of an Atomic Force Microscopy (Park NX10) in combination with an ammeter for concurrent topography imaging and electrochemical mapping. The SICM-SECM probe consisted of a Au crescent electrode (AuE) on the peripheral of a nanopipette. High resolution probe-substrate distance control was obtained by the ion current feedback from SICM, while simultaneous electrochemical signal collection was achieved via the AuE from SECM. As a proof-of-concept experiment, a Au/Pyrex pattern standard sample was imaged with the SICM-SECM technique. The Au bar and the Pyrex substrate were clearly resolved from the SICM topography image, with the bar height and pitch width closely matching the actual values. In terms of the electrochemical property mapping, higher Faradaic current was seen when the probe was scanned over Au bar as a result of redox cycling, while lower Faradaic current was observed when the probe was over Pyrex substrate due to hindered diffusion. The capability of the SICM-SECM technique described here holds promise of many exciting applications in the field of electrochemistry, material science and battery research.

[1] P. K. Hansma, et al., *Science*, 1989, **243**, 641.
[2] Shi, W. et al., *Faraday discussions*, 2016, **193**, 81-97.

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Supplementary Pages (Optional)

In **Figure 1**, representative SICM-SECM images are shown. In **Figure 1a**, topography image of the Au/Pyrex pattern is shown. **Figure 1c** (top) shows the line profile of the topography image. The measured pitch width is 20.06 µm, which matches the actual pitch width (20 µm). The measured height of the Au bar is 302.03 nm, which is, again, agrees with the actual feature height of 300 nm. In **Figure 1b**, electrochemical activity map of the same region seen in **Figure 1a** is shown. The absolute value of the Faradaic current over Au is ~4.5 nA, while over Pyrex, the absolute value of the Faradaic current is ~3.6 nA. An overall ~981 pA Faradaic current difference is observed. Correlation of the topography and Faradaic current images reveals the expected contrast, with enhanced Faradaic current over the conductive Au regions, consistent with positive feedback due to redox cycling, and reduced Faradaic current over insulative Pyrex trenches, consistent with negative feedback due to hindered diffusion.

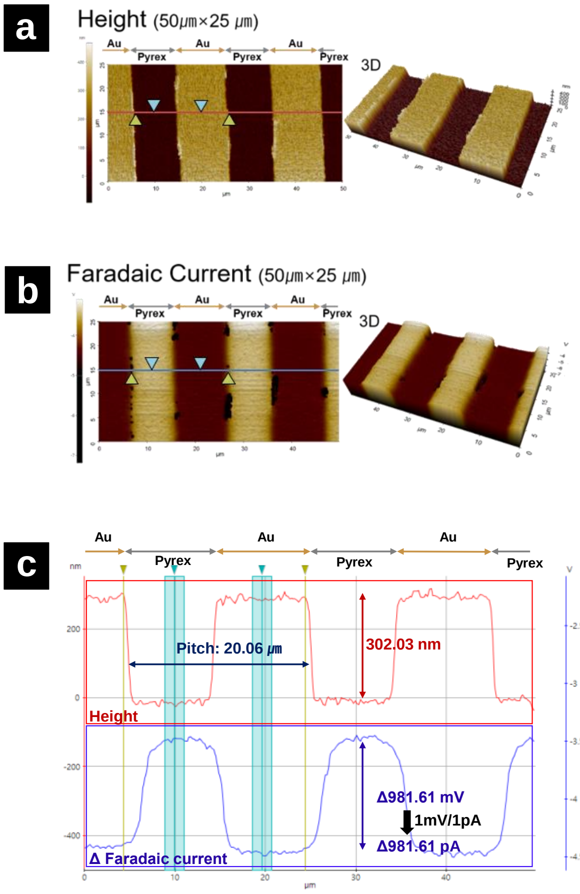

Fig.1 Representative SICM-SECM images. a) SICM topography image; b) SECM Faradaic current image. c) Line profile along the line seen in a) and b). Image size: 50 µm × 25 µm.