doi:10.1017/S1431927619004215
Microsc. Microanal. 25 (Suppl 2), 2019

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

Gabriela Mendoza[1*], Byong Kim[2], Keibock Lee[2]

[1.] Park Systems, Reforma Latino, Ciudad de Mexico, Mexico.
[2.] Park Systems, Inc., Olcott St., Santa Clara, CA, USA
* Corresponding author: gabriela@parksystems.com

Since the inception of scanning tunneling microscopy [1], electrochemists have sought to take advantage of scanned probe microscopy techniques to manipulate the spatial position of a probe with high resolution to facilitate simultaneous high resolution topographical, conductometric, and amperometric/voltammetric imaging of surface and interfaces. Lately, scanning ion conductance microscopy (SICM) [2], has emerged as a versatile non-contact imaging tool and been employed for a variety of applications. SICM has been used to investigate the surface topography of both synthetic and biological membranes, ion transport through porous materials, dynamic properties of living cells, and suspended artificial black lipid membranes. In addition, integration of complementary techniques with SICM has led to many exciting new applications, including scanning near-field optical microscopy and patch-clamping [3]. Powerful as it is, SICM remains insensitive to electrochemical properties, or, in other word, SICM is inherently chemically-blind and has no chemical specificity.

To obtain spatially-resolved electrochemical information, scanning electrochemical microscopy (SECM), also known as the chemical microscope, has been developed. SECM has been widely employed to examine localized electrochemical properties and reactivity of various materials/interfaces, such as electrode surfaces and interfaces, membranes [4], and biological systems. Despite its many applications, SECM, however, lacks reliable probe-sample distance control, and the probe is usually kept at a constant height during conventional SECM scanning. As a result, any variation in surface topography will result in changes in probe-sample distance, and thus leading to convolution to the measured faradaic current, which will complicate the subsequent data interpretation [4]. To address the above-mentioned issues for SICM and SECM, 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.

In this work, we demonstrate the use of an AFM (Park NX10) in combination with an ammeter for concurrent topography imaging and electrochemical mapping. The SICM-SECM probe utilized here 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 applications in the field of electrochemistry, material science and battery research.

References:

[1] G Binnig et al., Appl. Phys. Lett. **40** (1982), p. 178.
[2] PK Hansma et al., Science **243** (1989), p. 641.
[3] W Shi et al., Faraday Discussions **193** (2016), p. 81.
[4] W Shi and LA Baker, RSC Adv. **5** (2015), p. 99284.

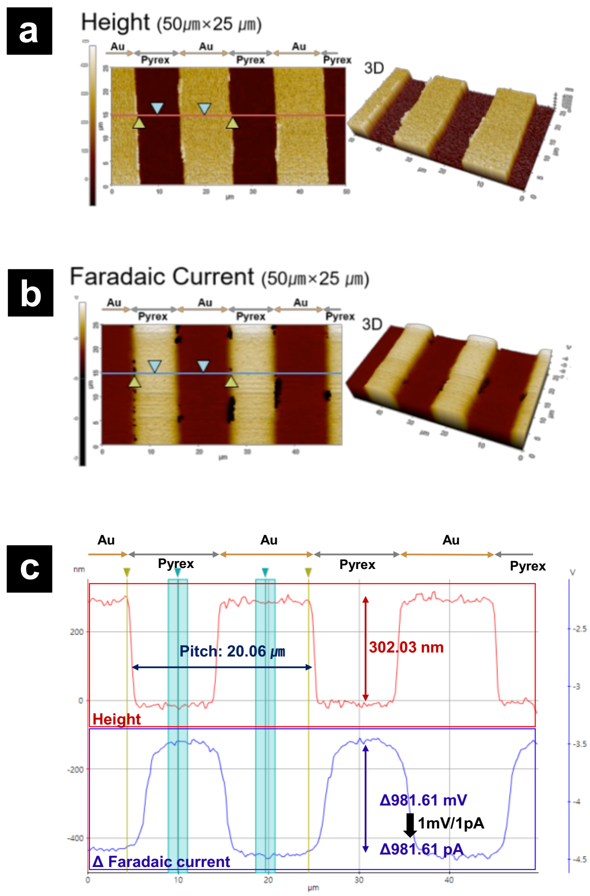

Figure 1. Representative SICM-SECM images. a) SICM topography image; b) SECM Faradaic current image. c) Line profile along the line seen in a) and b). Image size: 50 µm × 25 µm.