A Scanning Electrochemical Microscopy Module for Commercial AFM Systems: AFM-SECM for Simultaneous Topography and Electrochemical Imaging

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Scanning probe microscopic (SPM) techniques provide a powerful toolbox for obtaining topographical surface information with high lateral resolution. Among those techniques, atomic force microscopy (AFM) is the most versatile as there is no limitation in respect to the nature of the sample and imaging can be obtained in buffered solution. However, AFM provides limited information about the chemical or biological nature of the surface. This information is essential for in-situ investigations of chemical or biochemical processes in AFM based studies of complex dynamic samples. Examples herefore are corrosion studies and biomedical research. In contrast, laterally resolved (electro)chemical information on a sample can be obtained with scanning electrochemical microscopy (SECM), where a biased ultramicroelectrode is scanned at a defined distance across the sample surface.. However, conventional SECM suffers the lack of sufficient spatial resolution and the convolution of topography and electrochemical response due to the current-dependent positioning of the microelectrode.

Within the last decade several approaches have been reported, for directly integrating a micro- or nanoelectrode into an AFM probe [1,2]. In order to maintain the functionality of both techniques, the integrated electrode is recessed from the end of the AFM tip [1]. Consequently, the electrode is located at a defined distance to the sample surface, which is now defined by the length of the actual AFM tip. Thus, by applying a potential to this AFM-SECM probe and recording the Faradaic current related to electroactive surface processes laterally (electro)chemical information can be directly correlated to the topographical information obtained by the AFM measurement. So far, combining AFM with SECM required customized solutions, as no commercial SECM module for AFM systems was available and therefore, the technology could only be used by a limited number of researchers.

Recently we have succeeded in bringing a SECM module on to the Agilent 5500 AFM platform, providing a dedicated mount with integrated preamplifier for AFM-SECM probes and a bipotentiostat, which allows to control the potential of the sample and the AFM tip-integrated electrode. This mechanism not only greatly minimized the effort required for experimental setup, but also enabled the capability of multifunctional imaging and surface modification with combined AFM-SECM modes. The advantage of the combined technique is that measurements are not limited to amperometry but can be extended to a multitude of electroanalytical techniques during AFM imaging. For example, localized impedance measurements using AFM-alternating current measurements [3]. Moreover recent activities in AFM-SECM are targeted towards high-resolution electrochemical imaging, novel electrode materials such as integrated boron-doped diamond electrodes [4], conductive polymer electrodes and integrated sensing schemes.

Here we will present selected examples of AFM-SECM demonstrating the versatile capability of this techniques ranging from surface modification to the investigation of electrocatalytic films and corrosion studies.

References

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