Simultaneous *in situ* Scanning Probe Microscopy and Transient Measurements at the Solid / Liquid Interface

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Scanning Probe Microscopy (SPM) has been proven in the last decade to be a very useful technique to investigate the solid / liquid interface in real space [1-4]. Since a simple imaging of surfaces at the solid / liquid interface is usually done near thermodynamical equilibrium, most of the commercial EC-SPM instruments are designed to operate in the dc limit, i.e. they have a very limited bandwidth [5]. This limitation of commercial EC-SPM's has become a serious problem in the recent years in further development of research at the solid / liquid interface. SPM systems are more and more used as preparative tools to nanostructure surfaces [6-12]. A major problem with most nanostructuring techniques is the tip substrate interaction, which requires a much better control and stability of the tip and working electrode potentials, as well as a high bandwidth of both electrodes. Recent results show, that these requirements can be achieved [10-12]. Transient techniques, like impedance spectroscopy or differential capacity measurements, can hardly be performed on surfaces which are well characterized by SPM. Distance Tunneling Spectroscopy (DTS) or Voltage Tunneling Spectroscopy (VTS) at the solid / liquid interface require very fast measurements of current transients in order to satisfy the stability conditions of the real tip position above a particular surface position, i.e. require a high bandwidth [13]. Such measurements cannot be performed at present with the required accuracy to determine detailed information from the measurements using commercial instruments.

Thus, we have developed a novel bipotentiostat [14], which provides all required features to combine SPM with transient techniques *in situ* at the solid / liquid interface. The bipotentiostat has been designed to operate with any commercial or homebuilt SPM electronic. Also SPM electronics which are usually used for application in ultrahigh vacuum, like the Omicron SPM electronic, can be used. This flexibility is unique at present, and allows to combine the best SPM electronic with advanced electrochemical features.

We present the novel features of the instrument, and demonstrate its capabilities by various measurements on Au (111) and n – Si (111) : H surfaces. The bipotentiostat provides the possibility to operate the SPM tip as a real electrochemical nanoelectrode, which allows a variety of novel experiments, for example localized electrodeposition of nanostructures with lateral widths of only a few nanometers.

We have used the instrument to nucleate and grow metal clusters with diameters of about 10 nm on single crystal surfaces and image them with SPM, correlate impedance spectroscopy up to 100 kHz on single crystal silicon surfaces with complementary SPM images, and performed high resolution and fast *in situ* spectroscopy at various single crystal surfaces.

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