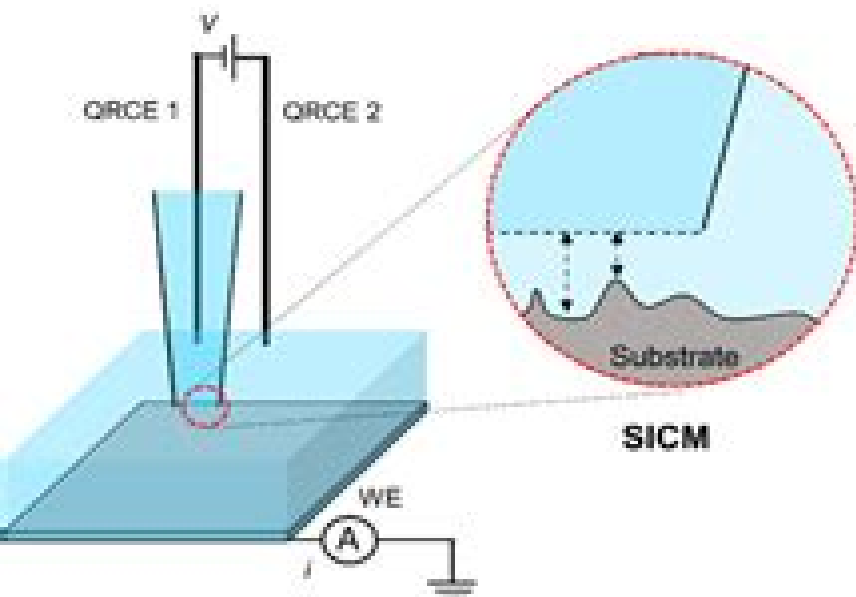
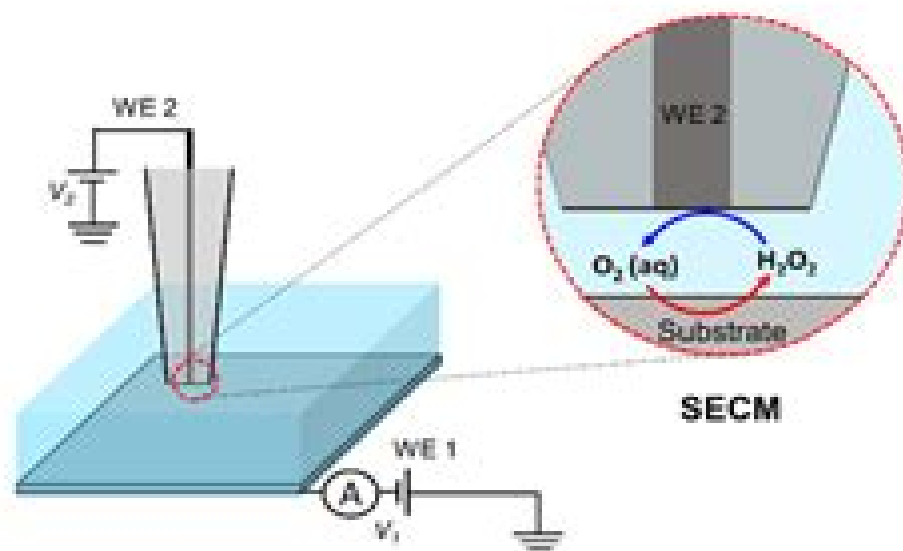


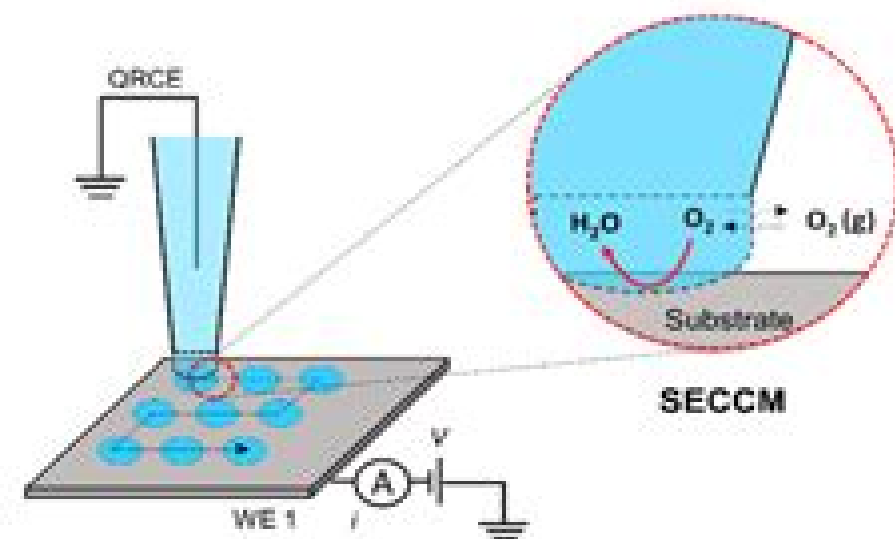
(a) Scanning Ion Conductance Microscopy



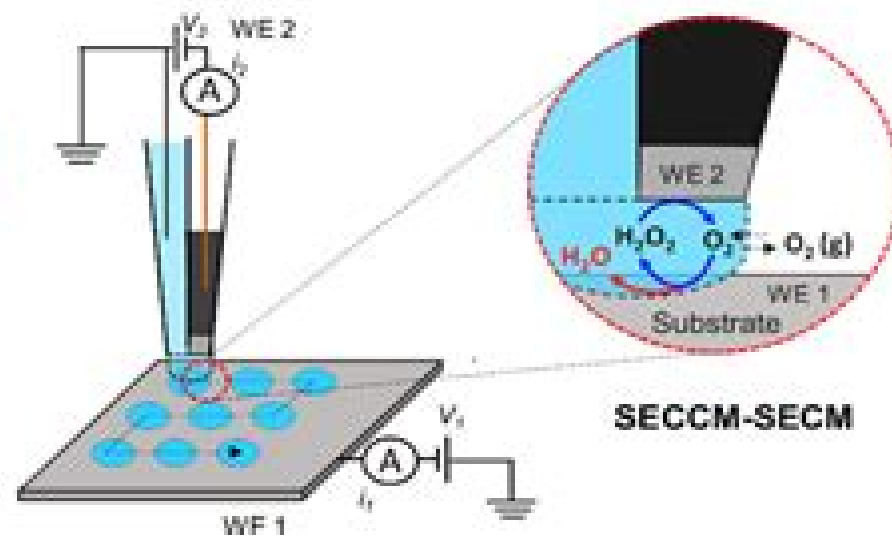
(b) Scanning Electrochemical Microscopy



(c) Scanning Electrochemical Cell Microscopy



(d) Combined Scanning Electrochemical Cell Microscopy and Scanning Electrochemical Microscopy



Scanning Electrochemical Microscopy

Patrick Vollmar



Scanning Electrochemical Microscopy:

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This thesis focuses on in situ studies at the solid liquid interface by combining scanning electrochemical microscopy SECM with gold and platinum mercury amalgam microelectrodes It is shown that stripping voltammetry experiments at imaging amalgam microelectrodes provide laterally resolved insight on the electrochemistry of biogeochemically relevant processes SECM provides information on electroactive surface processes with high spatial resolution and offers the opportunity to study heterogeneous electron transfer reactions Thereby chemical species of interest such as metal ions can be electrochemically detected at mercury amalgam electrodes Platinum and gold mercury amalgam microelectrodes were developed for the detection of biogeochemically relevant analytes such as manganese and iron during SECM imaging experiments at the mineral water interface establishing the fundamental basis of SECM imaging with amalgam microelectrodes SECM experiments were performed for the quantitative determination of Mn²⁺ during the dissolution of microstructured manganese carbonate rhodochrosite precipitates at mildly acidic conditions SECM images along with spatially resolved quantitative data on the Mn²⁺ concentration were obtained This measurement concept was then extended to the investigation of the corrosion behavior of diamond like carbon DLC protected zinc selenide ZnSe waveguides applied in mid infrared attenuated total reflectance spectroscopy at strongly oxidizing conditions The corrosion behavior of DLC coated and uncoated ZnSe crystals was studied obtaining laterally resolved information on the oxidative degradation of ZnSe at defects of the DLC layer utilizing SECM in combination with square wave anodic stripping voltammetry SWASV at

gold amalgam microelectrodes. Thereby insight on the corrosion behavior of ZnSe and concentration profiles of Zn^{2+} at oxidizing conditions was obtained. These results corroborate the utility of SECM imaging with amalgam microelectrodes for addressing relevant analytical questions. Finally the developed amalgam microelectrodes were applied for SECM studies of iron reducing proteins separated from *Shewanella* microbes in native polyacrylamide gels. After calibration of Pt/Hg microelectrodes in bulk solution for the targeted analytes iron and sulfur species SECM approach curves recorded above the native gel enabled positioning of the amalgam electrode in close proximity above protein bands with suspected iron reducing activity. This technique enabled the semi quantitative determination of the anaerobic respiratory activity associated with microbial proteins/protein complexes responsible for the reductive dissolution of manganese and iron oxides above microbial protein bands separated in a native gel matrix.

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