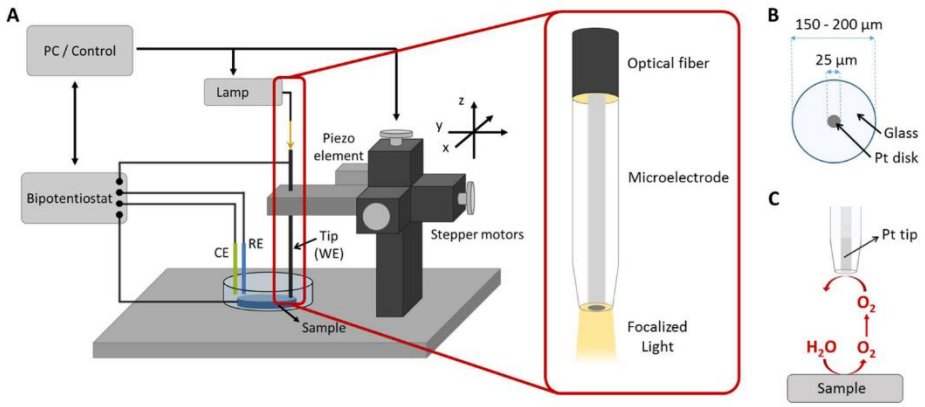
Analysis of Materials for Energy-Conversion Devices by Means of Scanning Photoelectrochemical Microscopy

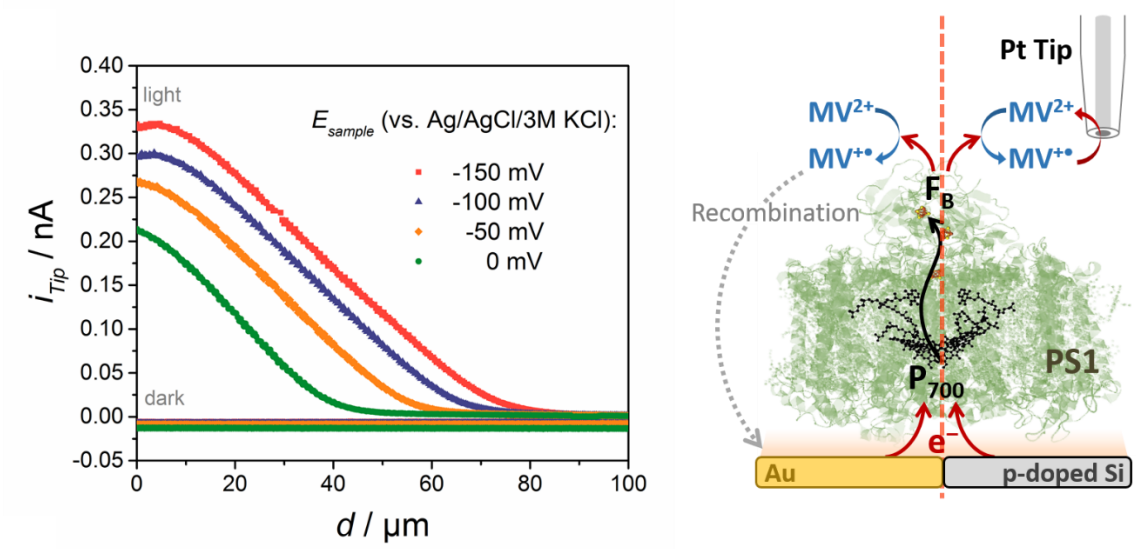
Scanning electrochemical microscopy (SECM) is a powerful tool for the analysis of a wide variety of surfaces and has been applied to the study and screening of different photocatalysts. SECM is a scanning probe technique used for the study of local electrochemical properties of a surface. In addition, the technique can be applied as a rapid method for the evaluation of libraries of photocatalysts and in-depth characterization of photoactive systems. In SECM three-dimensional images of surfaces are obtained by scanning a small microelectrode tip across a surface and recording an appropriate response.

The evaluation of photoactive materials under controlled irradiation conditions is of great importance. Under global irradiation of the sample the recorded photocurrent is generated at the entire sample surface. Hence, local changes of photoelectrocatalytic activity cannot be derived therefore impeding the characterization of complex samples. Moreover, in the case of performing SECM area scans under global sample irradiation, processes like degradation or photo-corrosion of the analyzed sample can influence the response obtained at the SECM tip. To overcome these issues, localized irradiation of the interrogated sample was introduced. With the aim of combining the advantages of localized illumination of the sample with SECM analysis of the surface, scanning photoelectrochemical microscopy (SPECM) was implemented. In SPECM, a light source is coupled to the microelectrode capillary, using the glass sheath of the microelectrode as a light guide, for the focalized irradiation of the sample, thus enabling high-sensitive multi-purpose photoelectrochemical measurements. The versatility of the technique allows the use of microelectrodes of diverse sizes and materials for the interrogation of different samples.



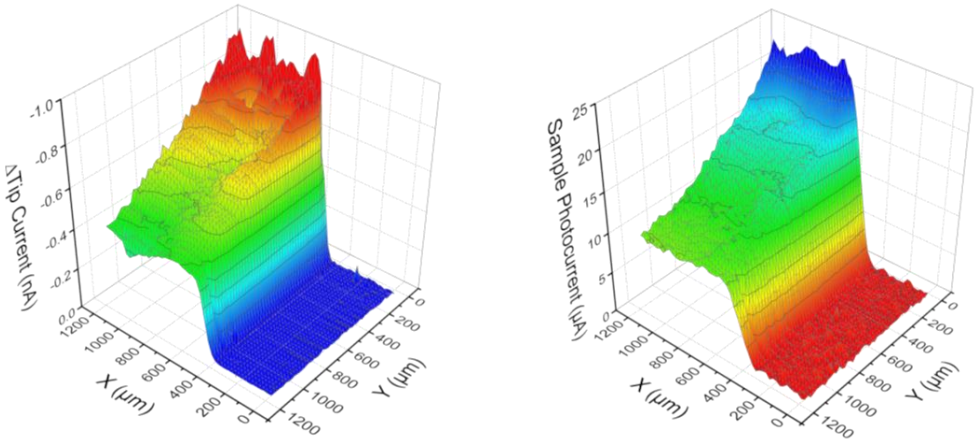
Scanning photoelectrochemical microscopy setup. © ACS, 2017 *Anal. Chem.* 2017, 89, 1222−1228

The technique has been recently used for the analysis of inorganic and photosystem-based energy conversion devices. For example, SPECM was used to directly detect the formed charge carriers and monitor their concentration profile near a photosystem 1-based photocathode. The analysis of the modified surfaces by SPECM enabled the evaluation of electron-transfer processes by simultaneously monitoring photocurrent generation at the biophotoelectrode and the associated generation of reduced charge carriers.



Approach curves recorded while moving the SPECM tip towards a photosystem 1-based photocathode. © Wiley-VCH, 2017 *Small* 2017, 1604093

In addition, SPECM has been used for the characterization of semiconductor materials for solar water splitting. A better understanding of the properties and electrochemical processes involved in photoelectrochemical water splitting is necessary for the design and development of energy conversion devices with improved efficiency.



Tip current and sample photocurrent recorded for an area scan of the edge of a TiO_2 layer deposited on a silicon wafer.

Module time line

Introduction to the technique:

Theoretical introduction to the principles of scanning electrochemical microscopy, microelectrode probes, and the characteristics of the scanning photoelectrochemical microscopy system.

Experimental setup:

Characterization of the microelectrode used as electrochemical probe and set up of the system for performing the characterization of the samples.

Analysis of samples:

Selected samples consisting of diverse materials for energy-conversion purposes will be analyzed by means of SPECM. The theoretical background about the evaluated samples will be briefly discussed.

Approach curves under dark and light conditions will be performed. In addition, a 2D scan of the sample will be done, revealing local hot-spots with increased catalytic activity on the sample.

Data collection and evaluation:

The collected data will be adequately plotted, evaluated and discussed.