Scanning electrochemical microscopy (SECM) offers a unique means with which to probe the chemical and electrochemical activity of solid-liquid interfaces with spatial resolution. In conventional SECM a microelectrode probe is scanned laterally above the surface of the substrate, and is used to monitor processes locally in solution and map corresponding surface reactivity. A variety of applications have been identified, and in particular, a great deal of attention has been given to the monitoring and screening of electrocatalyst performance.

We begin by presenting our research highlighting the use of amperometric SECM in the micron-scale characterization of model fuel cell catalyst films and commercial membrane electrode assemblies. We then explore the possible application of potentiometric approaches to measuring catalyst activity in-situ.

Moving forward, the challenge is to use chemical mapping as a means to study structure-activity relationships in nanostructured catalysts using nanoscale SECM. One promising approach is the combination of SECM with atomic force microscopy (SECM-AFM), which allows sensitive topographical and electrochemical measurements to be made simultaneously. The key to achieving this is the fabrication of dual function SECM-AFM probes in which a nanoelectrode is integrated into the tip. A number of approaches have been demonstrated, each with different merits. We present our recent development of novel probes that are relatively simple to fabricate by modification of commercially available metallic needle probes. We demonstrate that imaging using the ‘lift-mode’ approach successfully enables surface electrochemical mapping with a resolution in the 100 nm range.