

# Applications of scanning probe electrochemistry – Materials

## SCAN-Lab

November 2020



# Background

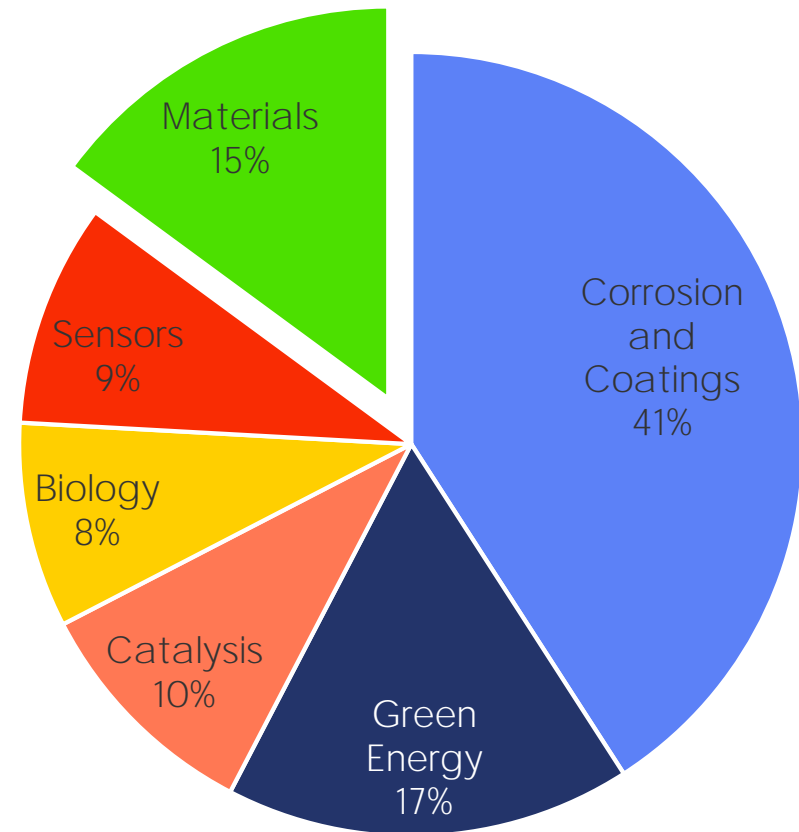


# Background

15% of commercial scanning probe electrochemistry instruments are applied in **materials research**. This includes research on semiconductors, ceramics, alloys, and low dimensionality materials.

This document will further investigate the role of scanning probe electrochemistry in materials research.

Publication Fields - All Techniques





# Why is scanning probe electrochemistry applied in materials?

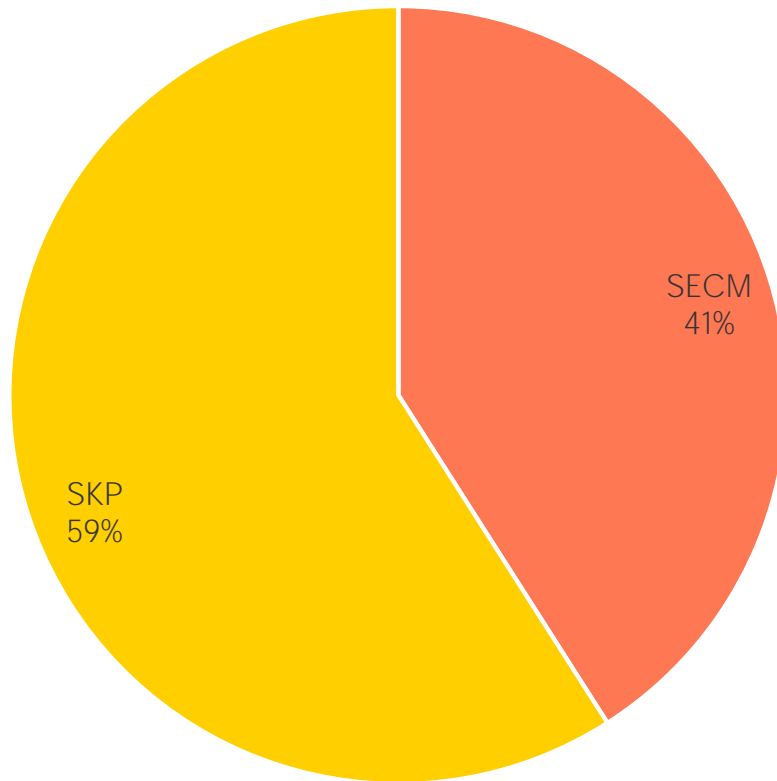
Scanning probe electrochemistry is used in fundamental R&D of **novel materials** to:

- Understand local electronic transport through 2D materials
- Evaluate the electrochemical characteristics of 2D materials
- Perform local functionalization and modification of a material to understand the fundamental effect
- Study the effect of the grain structure of material on its electronic characteristics
- Perform high throughput screening of the electrochemical characteristics of a library of novel materials
- Investigate the effect of alloy composition on electronic properties, e.g. work function
- Measure the effect of crystallographic orientation on electronic properties
- Screen the electronic properties of a sample after processing to determine the presence of defects and contaminants



# What techniques are used?

Materials – Commercial Instruments

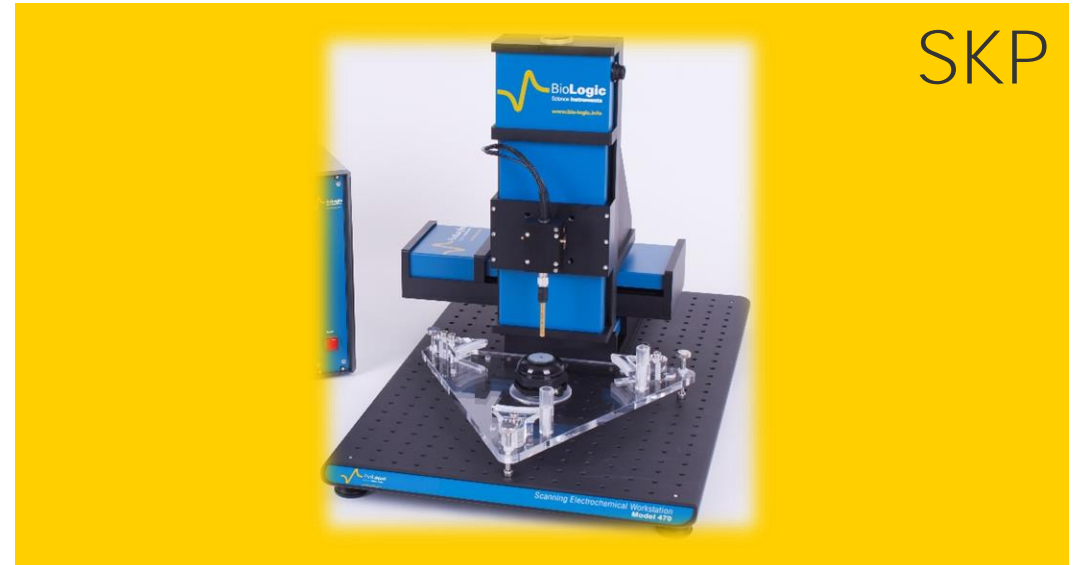
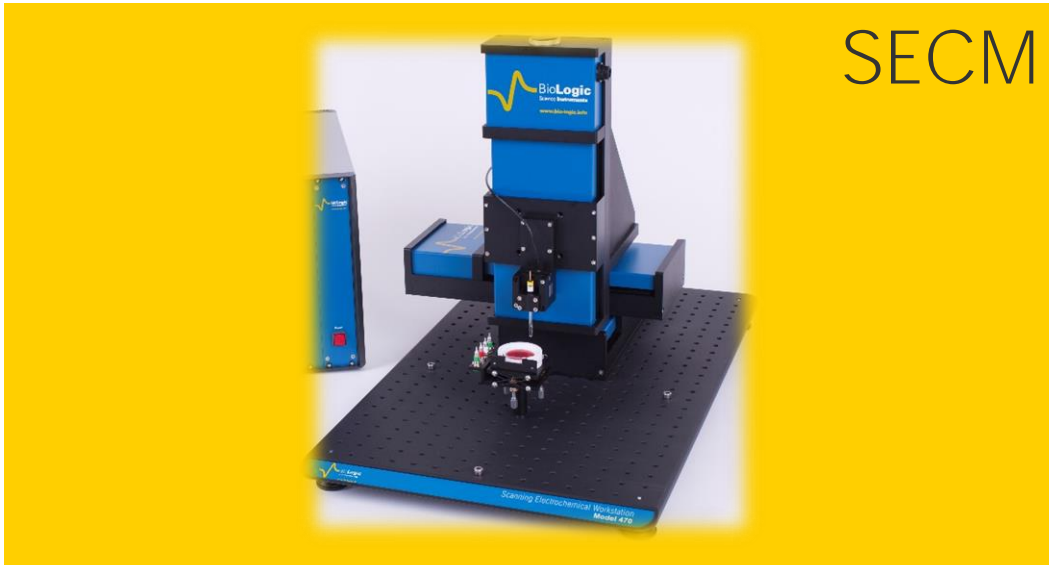


In the study of materials the two scanning probe electrochemistry techniques which are of interest are **SKP** and **SECM**. SKP is slightly more popular. SECM is of particular interest for low dimensionality materials.

Source: Analysis of all scientific publications retrieved through Google Scholar citing commercial instruments (all known manufacturers). Each research group was only counted once per technique.



# What techniques to consider for materials?





# Low Dimensionality Materials



# What are the research problems?

Low dimensionality materials, like graphene, graphene oxide, and MoS<sub>2</sub> are **difficult to make electrical contact** to in order to measure their electrochemical properties. Even when contact can be made the contact resistance affects the accuracy of the measurements.

**Solution: Measurement without electrical contact**

The electrochemistry of low dimensionality materials is **heterogeneous** in nature. It is dependent on whether an edge or bulk site is measured, and even on the number of layers beneath the flake. Bulk electrochemical measurements can be difficult to interpret to obtain local data.

**Solution: Direct local electrochemical information**

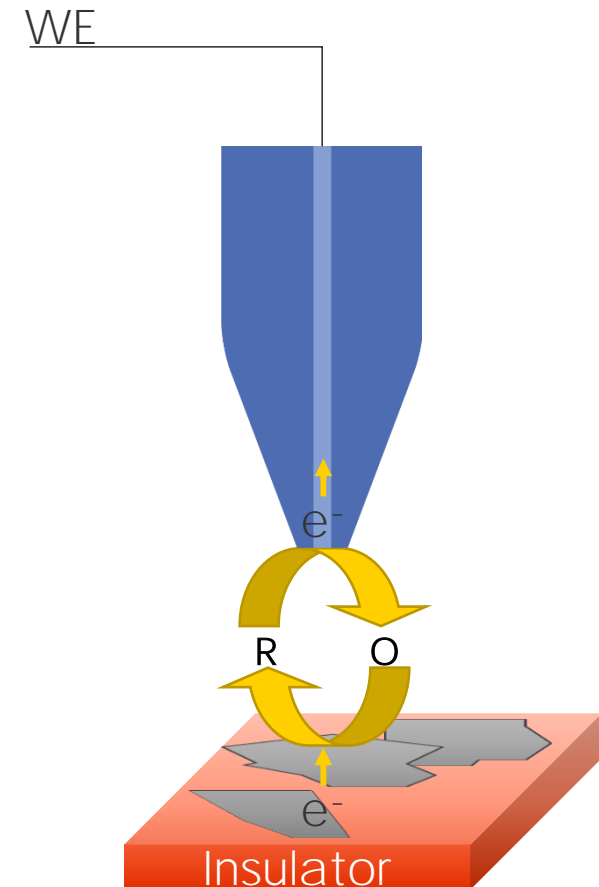




# Solution: Measurement without electrical contact

## How this is met by scanning probe electrochemistry:

Scanning Electrochemical Microscopy (SECM) **does not require electrical contact** to the material of interest. It is very useful for conductive materials in/on an insulating matrix, like graphene on an insulating surface. With the SECM probe over the flake its electrochemical activity can be measured with **standard dc electrochemistry experiments**. SECM can be used in ac mode to perform **Electrochemical Impedance Spectroscopy** studies of the flake. This is all done without any special sample preparation as would be required for bulk electrochemical techniques.



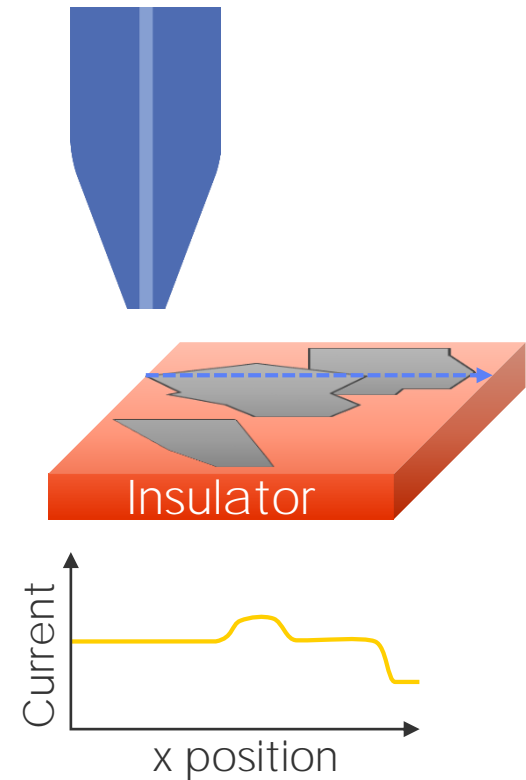


# Solution: Direct local electrochemical information

## How this is met by scanning probe electrochemistry:

Bulk electrochemical measurements result in an average for the entire sample. For heterogeneous materials this is difficult to interpret, and is exacerbated for low dimensionality materials.

Using SECM **individual sites of a sample are measured**. Standard electrochemical and EIS techniques can be performed at known locations to compare the effects of heterogeneities. It is more common to apply a **single dc bias or ac frequency** to the probe and raster scan the sample to visualise varying activity or impedance. This is correlated to known sample features.





# Materials



# What are the research problems?

For novel materials understanding how **sample heterogeneities** such as grain boundaries, and inclusions, affect the electrochemical, and electronic nature of a sample is important. For these samples the average activity measured by global techniques is inappropriate.

**Solution: Measurement of the local electronic and/or electrochemical activity**

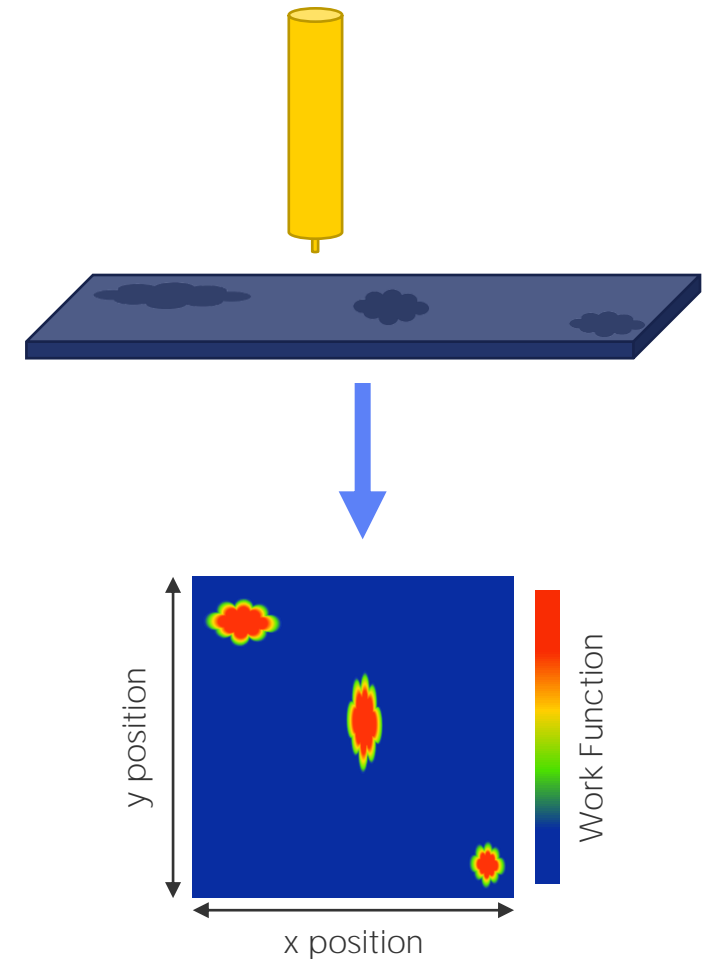
Whenever the composition is changed the electrical and electrochemical properties can change. With a seemingly endless number of possible elemental combinations available to new materials, materials discovery is a **game of chance**. The **time** required to measure each individual composition effectively limits the novel materials discovered.

**Solution: A method to quickly screen electrochemical characteristics**

# Solution: Measurement of the local electronic and/or electrochemical activity

## How this is met by scanning probe electrochemistry:

In all scanning probe electrochemistry techniques the probe is held close to a sample surface to detect the local electrochemistry under the probe. Scanning the probe produces a map of local electrochemical characteristics, allowing the **local electrochemical properties** of a novel material to be visualized, and correlated with the material features and **heterogeneities**. SECM and SKP are typically used to perform materials research in this manner. In SECM the local electrochemical activity or impedance is measured, while in SKP the local work function is measured.



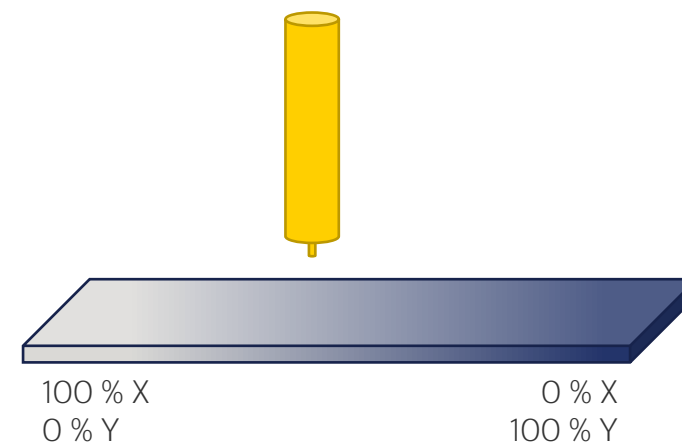


# Solution: A method to quickly screen electrochemical characteristics

## How this is met by scanning probe electrochemistry:

To quickly screen the effect of material composition on a property **high throughput screening** of combinatorial libraries is used. Scanning techniques can measure an entire library in a single experiment, **without changing samples**.

Scanning probe electrochemistry can be used to map combinatorial libraries *in situ*, individually probing the electrochemistry of a wide range of compositions in a **single experiment**. Performing screening in a single measurement allows hugely reduced screening times compared to traditional electrochemical screening methods.





# Conclusions



# Summary

- Samples of interest in materials research can be **difficult to prepare** for measurement by traditional techniques, this can be avoided using scanning probe electrochemistry
- For samples with **local heterogeneities** their effect cannot be determined through bulk measurements, however it is seen in scanning probe electrochemistry
- In materials discovery the **vast number of sample compositions** to measure make traditional measurements too time consuming, this can be overcome with scanning probe electrochemistry





# Why SCAN-Lab?

The scanning probe electrochemistry instruments available through SCAN-Lab allow the effective screening of the local electronic characteristics for materials design and discovery. SCAN-Lab products offer unparalleled user experience, with exceptional support from our team of experts.



# Learning Center Article

A series of Learning Center articles has been created to help determine the most appropriate technique for a given research problem. This includes an article dedicated to materials research:

## [Scanning Probes & Materials Research](#)



# Acronyms

- SECM: Scanning ElectroChemical Microscopy
- SKP: Scanning Kelvin Probe