Visualizing the Electrocatalytic Sites of Electrodes using *In-situ* Scanning Electrochemical Microscopy

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Investigating electroactive surfaces in-situ and establishing correlations with their electrocatalytic features continue to be focal points in the field of electrochemistry. Scanning electrochemical microscopy (SECM) is an advanced electroanalytical scanning probe technique capable of imaging/visualizing electroactive substrate topography and local active spots with high resolution. Unlike to the scanning tunnelling microscope (STM), this SECM provide imaging of in-situ electrochemical reaction that occurs via redox-reaction in association with an ultramicroelectrode tip over the test system surface. Present lecture covers the basic of the SECM instrumentation and operation procedures. In further, applications of SECM toward surface imaging of fictionalized carbon nanomaterials will be demonstrated [1-6]. Figure 1 is a typical SECM imaging of in-situ formation of nickel hexacyanoferrate (NiHCF) crystalline particle on the Ni-metal enriched multiwalled carbon nanotube modified electrode (electrocatalyst for thiol oxidation) in aqueous solution explored [4]. In this technique, a redox-competitive SECM (RC-SECM) technique has been adopted for the visualization. A variety of functionalized nanomaterial-modified electrodes will be covered in this presentation.

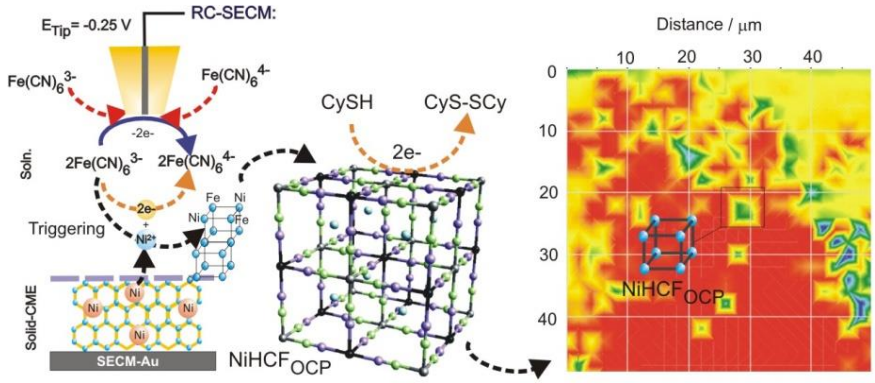

Figure 1. In-situ scanning electrochemical microscopy (SECM) was employed to investigate the surface imaging of nickel hexacyanoferrate (NiHCF) formation on intrinsic nickel-containing multi-walled carbon nanotubes (Ni-MWCNTs) in an aqueous environment under open-circuit conditions (OCP) [4].

RELATED REFERECES: [1] S.Nisha, V. Lakshminarayanan, A.S. Kumar*, *Langmuir* 36 (2020) 9; [2] M.Gandhi, D.Rajagopal, A S.Kumar*, *Appl. Surf. Sci.* 562 (2021) 150158; [3] S.Monisha, A. Mary Saral, A.S.Kumar*, *J. Electroanal. Chem.* 901 (2021) 115757; [4] S.Saikrithika, A. Shaju, B. Dinesh, A.S.Kumar*, *Electrochim. Acta* 405 (2022) 139806; S.Srinivas, [5] S.Srinivas, A.S. Kumar*, *J. Phys. Chem. C* 126 (2022) 8296; [6] S.Srinivas, S.M.Senthil Kumar, A.S.Kumar*, *Langmuir 39 (*2023)12563.

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