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3D nano-rheology microscopy: *operando* nanomapping of 3D mechanical nanostructure of SEI in Na-ion batteries

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The Solid Electrolyte Interphase (SEI) is a nanoscale thickness passivation layer that is formed as the product of electrolyte decomposition through a combination of chemical and electrochemical reactions in the cell and defines the fundamental battery properties - its capacity, cycle stability and safety. While local mechanical properties of SEI hold a clue to its performance, their *operando* characterisation is difficult as one has to probe nanoscale surface features in electrochemical environment that are also dynamically changing. Here, we report novel 3D nano-rheology microscopy (3D-NRM) that uses a tiny (sub-nm to few nm) lateral dithering of the sharp SPM tip at kHz frequencies to probe the minute sample reaction forces. By mapping the increments of the real and imaginary components of these forces, while the tip penetrates the soft interfacial layers, we obtain the true 3D nanoscale structure of sub–µm thick layers [1]. 3D-NRM allows to elucidate the key role of solvents in SEI formation and predict the conditions for building SEI for robust, safe and efficient Li-ion and Na-ion batteries.

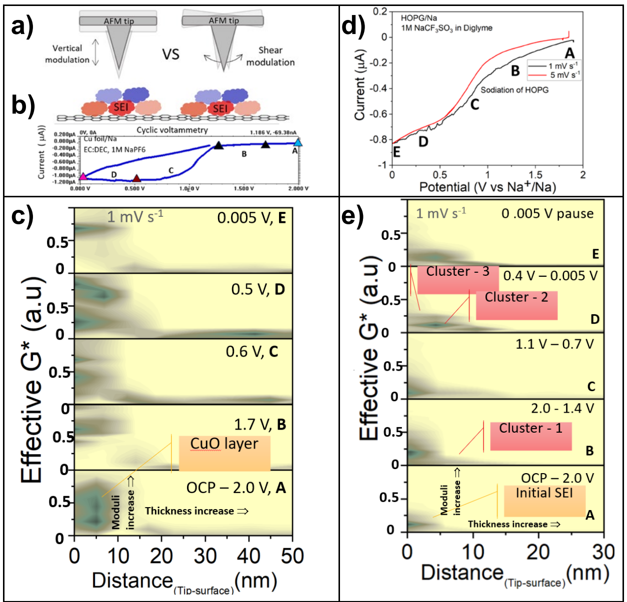

Fig.1 a) Principle of 3D-nanorheology, b) cyclic voltammetry (CV) of Na/Cu anode system, c) average SEI layer mechanical moduli G^* vs thickness, *d*, at different stages of charge-discharge cycle, d) CV of Na/HOPG anode cycle, d) *G**vs*d* for Na/HOPG at different CV points.

Here, we discuss the extension of these studies on smooth HOPG and inhomogeneous and rough copper anodes as sodium ion battery electrodes. Essentially, the new approach allows nanoscale characterisation of SEI with a few nm precision on the electrodes with 1000 nm roughness, and quantitatively evaluate the real and imaginary parts of the elastic moduli over the whole thickness of SEI layer. The observation of the change in moduli and the tip-surface distance helps to evaluate the growth of SEI as a function of the electrolyte, additives, electrode material and charge-discharge rate. We believe that such evaluation of key interfacial nanomechanical properties of SEI will allow us to develop the electrochemically and mechanically robust SEI surface passivation layer and the development of efficient and safe rechargeable batteries.

[1] Y Chen, W Wu, S Gonzalez-Munoz, L Forcieri, C Wells, SP Jarvis, F Wu, R Young, A Dey, M Isaacs, M Nagarathinam, RG Palgrave, N Tapia-Ruiz, OV Kolosov, Nature Comm 2023, 14, 1321.
[2] SJ O'Shea, ME Welland, JB Pethica, Chem. Phys. Lett. 1994, 223 (4), 336;