

Type of presentation: Invited

IT-7-IN-2863 The opportunities and challenges of liquid cell electron microscopy

Ross F. M.¹

¹IBM T. J. Watson Research Center, Yorktown Heights, NY 10598, USA

Email of the presenting author: fmross@us.ibm.com

Liquid samples, particularly samples containing water, have traditionally been difficult to examine using transmission electron microscopy because of the incompatibility between the microscope vacuum and the high vapour pressure liquid. But in recent years, advances in sample design have allowed us to enclose liquids in a form that permits examination by TEM. Microfabricated devices are constructed in which two electron transparent membranes are spaced 100nm-1μm apart. A liquid is introduced between the membranes, allowing imaging of structures and processes *in situ*. The technique of liquid cell electron microscopy has been adopted by many laboratories worldwide, and is of interest to the microscopy, materials and biology communities because it enables data to be obtained at a spatial and temporal resolution not accessible with other techniques.

In this presentation we focus on the use of liquid cell electron microscopy to examine the mechanisms of electrochemical processes in aqueous electrolytes. Liquid cell microscopy is well suited for electrochemistry because electron-transparent electrodes can be included during device fabrication. Images and movies of the transient structures that form during nucleation or dissolution can then be correlated with electrochemical parameters (voltage, current) controlled or measured by a potentiostat. We show measurements made during deposition and stripping of metals (Cu, Zn) on Au or Pt electrodes. After nucleation and coalescence, we measure the propagation of the growth front outwards from the electrode and into the liquid layer. We will show that an initially planar growth front roughens and becomes unstable, forming dendrites or ramified patterns. Such growth instabilities can affect the charging of batteries and the electrodeposition of thin films and multilayers. We will show how the development of diffusion fields works together with kinetic roughening to cause the onset of growth instabilities. Techniques have been developed to control the onset of instability, including pulse plating, electrolyte flow and the use of additives to alter interface parameters. We will examine these approaches using liquid cell microscopy.

In any liquid cell experiment, obtaining quantitative data that is suitable for matching with models involves understanding the pitfalls and artifacts that can occur during liquid cell EM. We therefore discuss electron beam effects, in particular the strong changes in solution chemistry that can be induced by the beam. Beam-induced radiolysis of water can lead to phenomena such as particle and bubble formation. These can be minimised with low-dose techniques, but may also be useful in forming patterned structures and in measuring the properties of nanoscale bubbles.

Acknowledgement: The results presented here have been funded, in part, by the US National Science Foundation under grants 1129722, 1225104 and 1066573.