In-situ Scanning Electron Microscopy Observation of Lead Dendrites Grown in an Electrochemical Cell

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Electron microscopy observation of the structures and processes of materials in liquid phase is booming in recent years, including a wide range of topics such as biological materials, synthesis of nanoparticles and electrochemical reactions[1]. In particular, in-situ electron microscopy observation has deepened nanoscale understanding of the chemical reactions or physical mechanisms. de jonge et al. successfully imaged the labeled single molecules in a liquid cell with a resolution of several nanometers using scanning transmission electron microscopy (STEM)[2]. White et al. has visualized and analyzed lead (Pb) dendrites in an aqueous solution using in-situ TEM[3]. Sacci et al. utilized an electrochemical cell for in-situ TEM to analyze lithium dendrite nucleation and solid-electrolyte interface (SEI) formation at the edge of a gold working electrode during electro-deposition[4]. In electrochemistry, understanding of the mechanism of reactions at the interface of electrode-electrolyte is very important, especially on a nanometer scale. During electroplating, small islands form and afterward turn into a thin layer. As the energy storage devices like lithium-ion batteries or fuel cells, dendrite structures or SEI formation would dominate the performance of these devices.

We developed an electrochemical cell for in-situ scanning electron microscopy (SEM) using an in-lens FE-SEM setup (S-5200, Hitachi Hightechnologies), having two electrode terminals to observe the processes of electro-plating and stripping of lead dendrites simultaneously with measuring the cyclic voltammetry. Pb dendrites on an Au electrode in an electrolyte of 1.5 M $Pb(NO_3)_2$ solution were grown and decomposed during a cyclic of voltammogram. The in-situ SEM observation revealed the initial formation of Pb islands and the subsequent Pb dendritic growth, as shown in Fig. 1. The electrolyte concentration and the scan rate of applied voltage changed the dendrites formation and decomposition. The correlation of concentration site and local structural differences will be discussed.

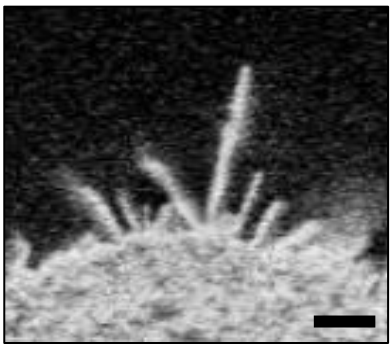

Fig. 1 SEM image of lead dendrites growing on gold electrode in a $Pb(NO_3)_2$ solution, at -1.92 V (relative potential to the counter Au electrode). Scale bar: 2 μm.

References:

[1] de jonge, N., et al., Nat. Nanotechnol. 6 (2011) 695.

[2] de jonge, N., et al., Proc. Natl Acad. Sci. USA 106 (2009) 2159.

[3] White, E., et al., ACS Nano 6 (2012) 6308.

[4] Sacci, R.L., et al., Chem. Commun. 50 (2014) 2104.