X-ray Tomography Study on Li-Ion Battery Electrodes

Sung-Chieh Chao (趙崧傑), Yu-Chan Yen (嚴佑展), and Nae-Lih Wu (吳乃立)

Department of Chemical Engineering, National Taiwan University, Taipei, Taiwan

As anode material for Lithium-ion battery (LIB), Si is an interesting material for replacing graphite anode material because of its high theoretical capacity exceeding 3000 mAh/g and low alloying potential versus Li. However, this material exhibits dramatic volume expansion and shrinkage during lithiation and de-lithiation process. As a result, most recent studies have been focusing on microstructural issues aiming to cope with the volume expansion effect. Ironically, evidence on microstructural change on Si anode has been scarce. The object of this work is to characterize the bulk material change of Si-based material by *in situ* TXM.

A tomographic transmission X-ray microscopy (TXM) was constructed at beam line 01B1 of the National Synchrotron Radiation Research Center (NSRRC) in Taiwan, R.O.C. The microscope, operating in the photon energy range between 8~11 KeV utilizes zone plate optical system to achieve tomographic images at 60 nm resolution with a 15x15 μm field of view and a depth of field of 50 μm. The advantages of hard X-ray imaging are high penetration for nondestructive and in-situ imaging, short wavelength for high resolution, rich image contrast mechanism, element specific and no charging effect on image resolution, which is a powerful tool to *in situ* monitor the bulk material change during charging and discharging process.

Our preliminary study has shown that nanoporous NiSi-Si composite material shows much improved cyclability as compared with Si. The improvement is contributed by preset void. Fig. 1(a) shows that the Ni signal indeed can be detected inside the particle of the material before etching. Also, the area shown light color may indicate that there are some voids formed intrinsically during ball milling process even without etched off the Ni.

Our final goal is to characterize the volume change during lithiation and de-lithiation process of Si material, however, because of its low atomic number, the image is barely distinguishable from Si to the other materials. In order to see the image more clearly, the metal oxide SnO, having the same microstructural issue as Si but has higher atomic number, was used for this fundamental study. Form fig. 1(b), it shows the image of SnO electrode before any electrochemical treatment. All particles tend to aggregate with each other at fresh state, however, those tiny SnO particles gradually separated with each other after charge-discharge (Fig. 1(C)). The whole process of apparently microstructural change was successfully detected by *in situ* TXM, provides strong evidences by means of clear images and movies.

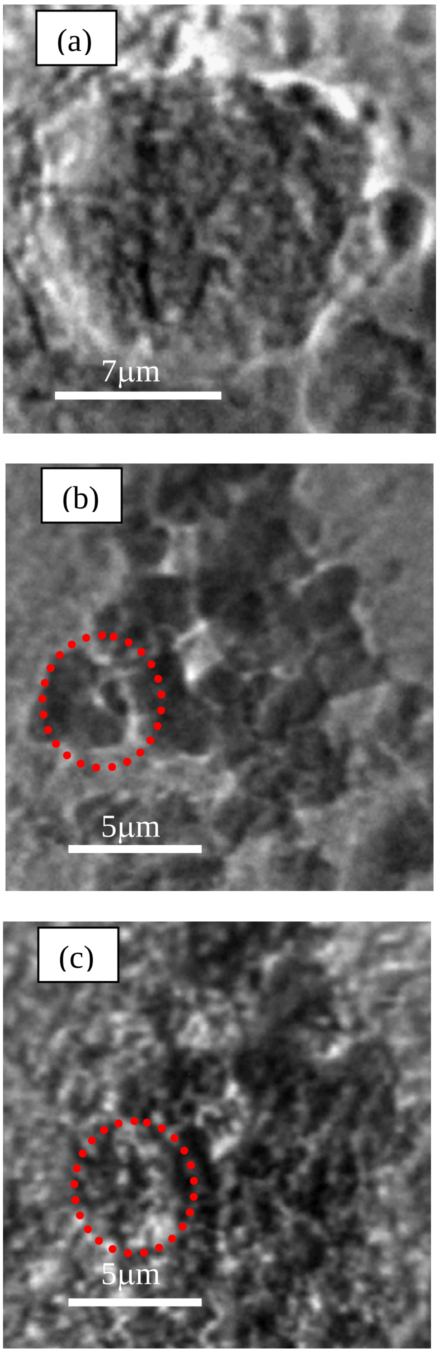

Figure 1. TXM images. (a) NiSi/Si/Ni composite; (b) SnO clusters at fresh state; and (c) SnO clusters after two cycles. The red dotted circle denotes the emphatic region.