Transmission Electron Microscopy (TEM) Study of the Degradation Mechanism of Amorphous Vanadium Oxide Based Thin Film Battery

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Solid-state thin film batteries (TFBs) consisting of amorphous V_2O_5/Pt/Ti/Si have been fabricated by specially designed dual target magnetron radio frequency (rf) and direct current (dc) sputtering systems at room temperature using *in-situ* growing process.[1] It is shown that the discharge capacity of the TFBs is degraded with increasing cycle number; the efficiency is dropped by ~54 % of the initial value after 450 cycles.

To investigate the degradation mechanism, the structural properties of as-deposited and cycled LiPON and a-V_2O_5 films are characterized by transmission electron microscopy (TEM) and x-ray diffraction (XRD) measurements. TEM and XRD results (not shown here) show that the as-deposited LiPON and V_2O_5 are amorphous. Fig.1 shows a cross-section high-resolution electron microscopy (HREM) image and a TED pattern (inset) obtained from the as-deposited V_2O_5 film. The HREM image reveals an amorphous feature.

It is, however, shown that cycling causes the occurrence of crystallites (2 – 6 nm across) not only in the V_2O_5 film, but also in the LiPON film. Fig.2 (a) shows a bright field TEM image obtained from a region including the LiPON/V_2O_5/Pt layers after 450 cycles. The image reveals a number of dark blobs, which are distributed across the whole LiPON and V_2O_5 layers. These blobs are associated with the V_2O_5 crystallites formed during cycling. This was confirmed by dark field TEM images taken using their associated spots shown in inset TED patterns of Fig .2 (b) and a HREM image in Fig.2 (b). For the LiPON layer, the pattern exhibits a ring consisted of weak spots (indicated by the arrow), in addition to a diffuse ring which is a characteristic feature of amorphous structure. The weak spots are evidence for the presence of the nano-crystallites, as confirmed by a HREM image, showing crystallites 2 – 4 nm across (indicated by the arrows in Fig. 3).

Based on the structural and electrochemical results, the cycle-induced degradation of capacity is correlated to the structural change of the LiPON and V_2O_5.

1. E. J. Jeon, Y. W. Shin, S. C. Nam, W. I. Cho and Y.S.Yoon, *J. Electrochem. Soc.* **148**(4), (2001)

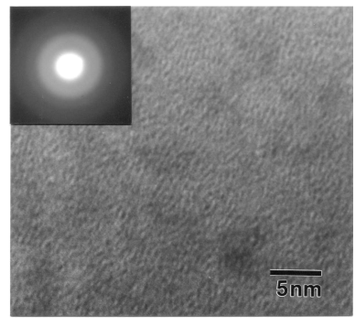
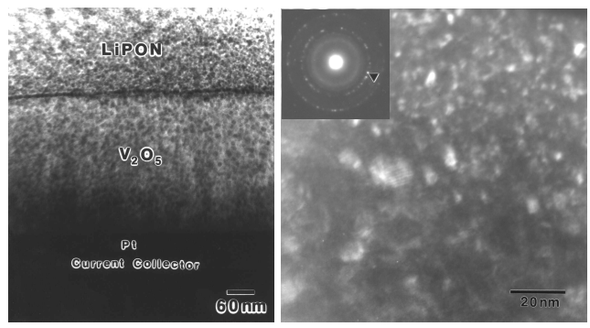


Figure 1. A cross-section high-resolution electron microscopy (HREM) image and a TED pattern (inset) obtained from the as-deposited V_2O_5 film.



(a) (b)

Figure 2. (a) Bright field TEM image obtained from a region including the LiPON/V_2O_5/Pt layers and (b) dark field TEM image of V_2O_5 after 450 cycles.

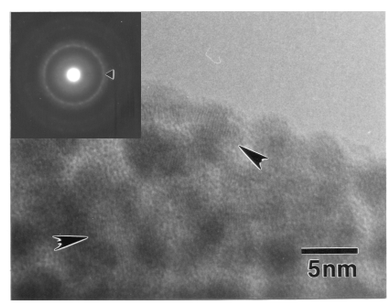


Figure 3. A cross-section high-resolution electron microscopy (HREM) image and a TED pattern (inset) obtained from the LiPON electrolyte after 450 cycling.