Application Note

Testing report of Vacuum Sample Holder

Part One: sample protection

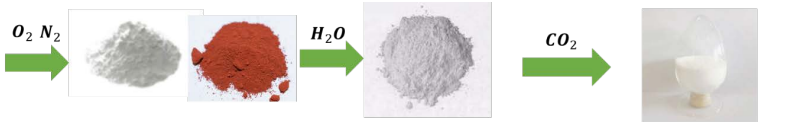
Background

Scanning electron microscopy (SEM) is the best tool for inspection of material surfaces at nanoscale level and has been widely used in the development and quality control of various materials. However, because some materials are sensitive to oxygen or water in the air, they need to be prepared in a glove box and then transferred to SEM with special protective devices.

The Vacuum Sample Holder (VSH) is such a protective device, that can transfer the samples from the glove box to SEM without being exposed to oxygen or water in the air. It opens automatically when moved into SEM and closes automatically when moved out of the electron microscope, which support sample protection. It can realize the sample transfer between the glove box and the electron microscope or different vacuum machines.

In order to illustrate the working mode and protective effect of VSH, a set of control experiments were designed. Lithium metal, a commonly used cathode material for lithium battery, was selected for testing, and the effects of lithium metal sheet and lithium foil materials were compared.

Normally, lithium reacts very quickly in the air, as:



Li

$4Li + O_2 = 2Li_2O$
$6Li + 3N_2 = 2Li_3N$

$Li_2O + H_2O = 2LiOH$
$2Li + 2H_2O = 2LiOH + H_2$
$Li_3N + 3H_2O = 3LiOH + NH_3$

$Li_2O + CO_2 = Li_2CO_3$

Experiment 1 Li surface changes without VSH protection

After the lithium is placed in air for 30 minutes, the above reactions seem to have no obvious changes on the surface, as shown in Figure 1,2. However, the microscopic changes are great. The SEM photo is shown in Figure 3 below. There are obviously distributed white particles on the surface, which are oxidation products. Figure 4 shows the Oxygen counts increasing to 250 cts on the surface when exposed for 30 minutes.

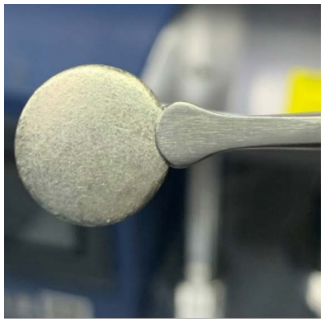


Figure 1. Li in Ar

Figure 2. Li in Air for 30min

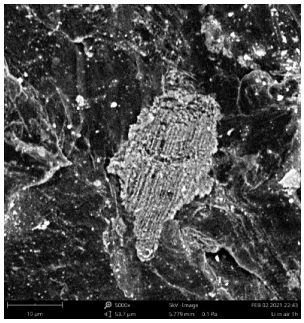


Figure 3. SEM image of Li

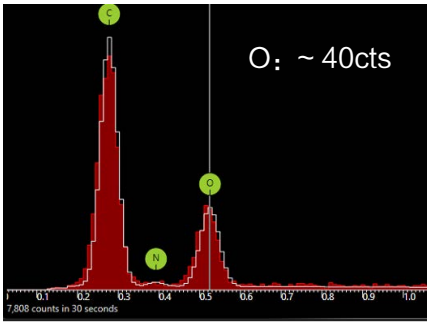
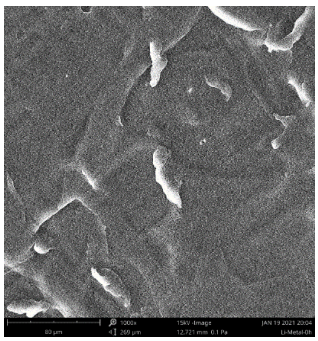


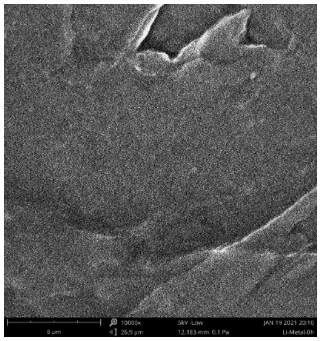
Figure 4. Oxygen count number

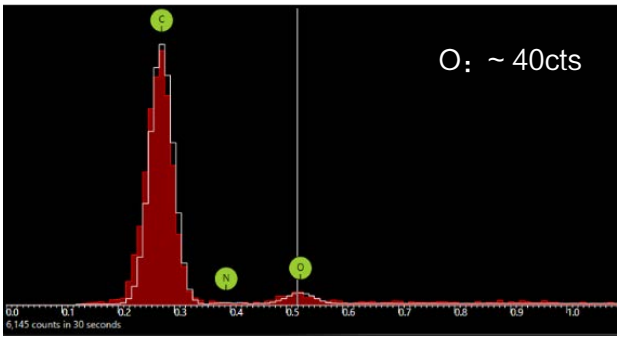
Experiment 2 Lithium surface observation within VSH

The lithium metal sheets (400 micron) were placed in VSH in the glove box and immediately transferred into the electron microscope for observation (i.e., 0 min as shown in the figure below). In addition, the lithium metal sheets were placed into the VSH in the glove box, then the VSH with sample were stored outside of the glove box for 24 hour and 48 hours respectively, and then moved into the electron microscope for inspection.

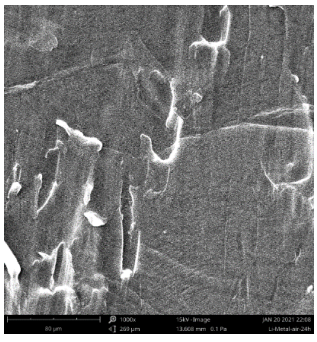
0h

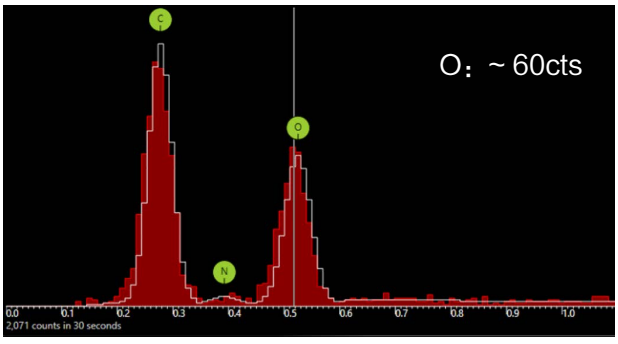




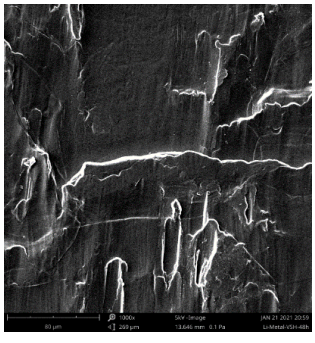


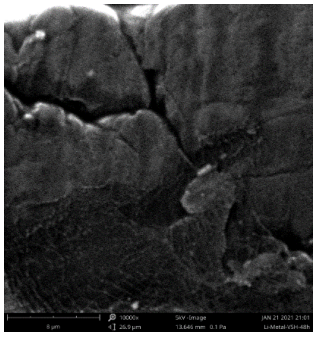
24h

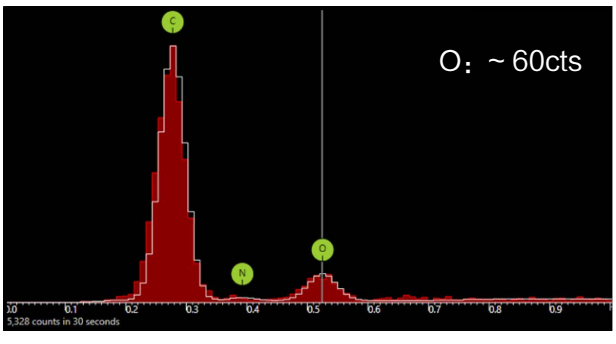




48h





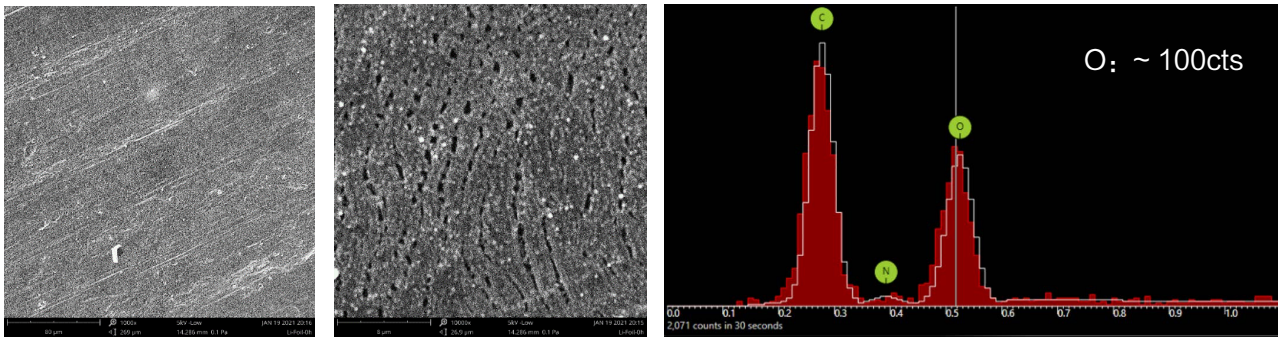


It can be observed that the surface morphology of lithium metal does change, but from the results of oxygen count, the increase of Oxygen is not obvious. So it can be seen that oxidation is not obvious. So even after 48 hours the sample is still very well protected and can be used for research purpose.

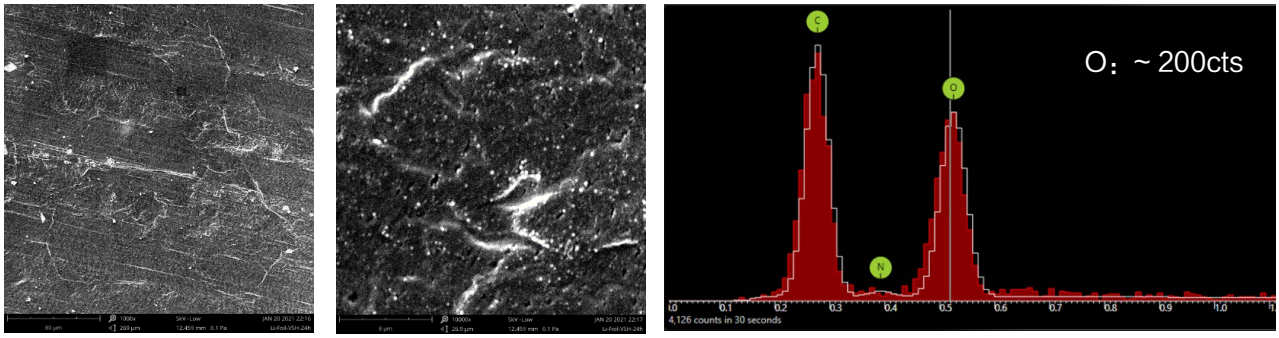
Experiment 3 Lithium foil surface observation within VSH

Then, lithium foil of 20 micron thickness is tested, which is more sensitive to the air. Similarly, the lithium foil were transferred to VSH in the glove box, then it is moved to SEM to observe immediately (that is, the image below at 0 minutes), another two samples were prepared at the same way and closed in VSH and then placed in air for 24 hours and 48 hours respectively, and then moved into SEM. The results were below.

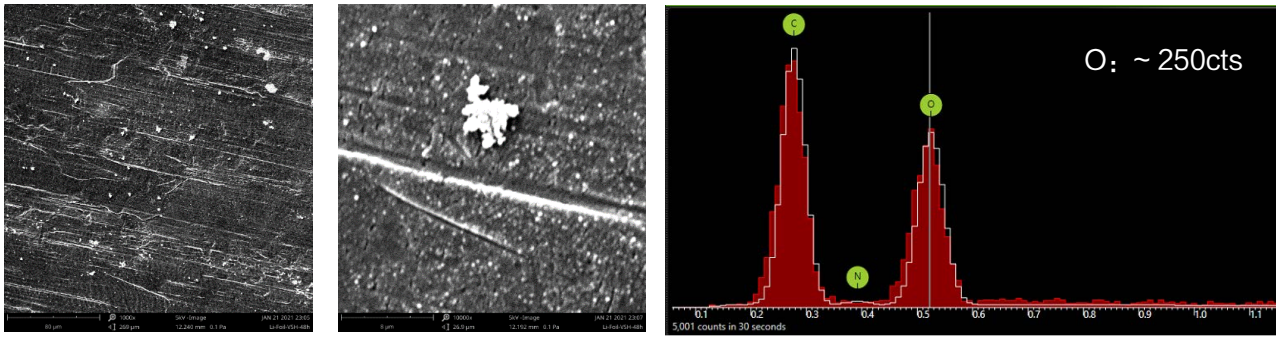
0h



24h



48h



The change of the surface and oxygen count can be observed obviously from above images. Oxygen count increased 100 cts in 24 hours.

Conclusion

1. with the results of SEM and EDX, for the lithium sheet sample, even after 48h of VSH , the microstructure and composition of the metal surface almost did not change.
2. For the same element Li, lithium foil (20 micron) is more sensitive to air than lithium sheet (400 micron). Even though, The VSH protects this sensitive material for at least 24 hours.

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