Plastic Bag Method for Active Sample Loading into Transmission Electron Microscope

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Abstract

A plastic bag method was developed to observe air sensitive samples on microstructure and phases distribution without air exposure during holder transfer process into the Transmission Electron Microscope (TEM). As an example, a type of lithium aluminum hydride (Li_3AlH_6) was observed in TEM to demonstrate the effectiveness of this method. Results show that plastic bag method is a simple and practical TEM transfer method utilized to reduce air contact for a series of air sensitive materials.

Keywords: plastic bag method, transmission electron microscope, air exposure, diffraction patterns

Recently many active samples are requested for the observations of microstructures by using transmission electron microscope. Commonly they are safely deposited under an inert gas atmosphere in glove box. However, the essential technical issue is to protect the oxidation during specimen loading.

Li_3AlH_6 is an active material for promising research of hydrogen storage materials, which has a hydrogen capacity of 2.6wt% [1]. It has a dehydrogenation reaction: $Li_3AlH_6 \rightarrow LiH + Al + H_2$. Due to the influence of the oxygen and water vapor in air, sample can be oxidized and then experienced less release and recharging of hydrogen. Therefore, generally, Li_3AlH_6 is deposited in the argon-filled glove box (oxygen below 5 ppm). Nonetheless, when the TEM observation is required, prevention of the oxidization during the transfer from glove box to TEM is one of important problems to be solved.

Powder Li_3AlH_6 was prepared from decomposition of $LiAlH_4$ (Aldrich, 95% purity) at 179℃ and confirmed by X-ray diffraction. Mo grids were used during observations in a 200kV TEM (JEOL 2010).

Figure 1 shows the illustration diagram of plastic bag method. The plastic bag is made of polyethylene and has a thickness of 0.040 mm. It consists of main body and joints fabricated by heated wire welding. It should be guarantied that the edge of bag is well-sealed with no leakage. In the glove box, the TEM holder can be sealed in the main body of plastic bag by a clip at the position between main body and joint section as shown in Figure 1. This can ensure that the TEM holder can be transferred to the TEM aside without any air exposure. The joint section of the plastic bag should be connected to the entrance of TEM by tape. With flushing argon gas (purity 99.999%) to the joint section at a pressure of 0.2

MPa, the transfer from plastic bag main body to TEM can be done appropriately. Following 3-5 minutes of flushing gas, the holder can be loaded into the TEM with releasing the clip. It should be noted that the dimension of plastic bag can be modified due to any type of TEM holders, such as a high voltage microscope holder.

A sample of Li_3AlH_6, which has not been protected by plastic bag method during transfer of TEM holder, was observed by TEM as shown in the Figure 2 (a). The transfer of holder would introduce a short air exposure to sample for around 5 min. In Figure 2 (a), no clear diffraction spots or rings from crystals were observed exclude a typical halo rings from amorphous phases. A very high peak of O was detected from the exposed Li_3AlH_6 as shown in the Figure 2 (b), indicating that only in 5 min, oxidization of small amount of powder Li_3AlH_6 could be over.

Therefore, some special vacuum transfer TEM holders were designed to protect sample during transfer of holders. Those holders, such as environmental cell and vacuum transfer holders are capable to transfer a specimen into TEM under vacuum or controlled atmosphere [2, 3]. However, an acceptable alternative is the plastic bag method: simple and practical way to reduce the effect of sample oxidization in the transfer process [2, 4-8].

Figure 3 (a) shows a bright-field image of Li_3AlH_6 with the corresponding diffraction patterns when plastic bag method was used. The corresponding diffraction patterns obviously showed a typical index of diffraction spots from Li_3AlH_6 which definitely demonstrate the existence of Li_3AlH_6 crystalline(s). Figure 3 (b) showed the EDS spectrum detected from Li_3AlH_6 prepared using a plastic bag. A high peak of Al and only a little peak of O were detected. Although O could be detected from sample transferred with plastic bag

method but compare with sample without using plastic bag method, the small quantity of O shows that plastic bag could reduce air contact. From the totally different diffraction patterns shown in Figure 2 (a) and Figure 3 (a) indicate that plastic bag method was good enough and also necessary to be used for sample observation without air exposure.

Easy fabrication and low cost for the excellent features of this plastic bag method were considered. From the results shown above, this plastic bag method can be used to be an effective means of reducing air contact to sample in TEM experiments.

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References

[1] X.Liu, G.S. Mcgrady, H.W. Langmi and C.M. Jensen: J. Am. Chem. Soc. 131 (2009) 5032-5033.

[2] P. Vullum, M. Pitt, J. Walmsley, B. Hauback and R. Holmestad: J. Phys.: Conf. Ser. **126** (2008) 012015

[3] http://www.gatan.com/index.php

[4] C. M. Andrei, J. C. Walmsley, H. W. Brinks, R. Holmestad, S. S. Srinivasan, C. M. Jensen and B. C. Hauback: Appl. Phys. A **80** (2005) 709-715.

[5] H. Yao, H. Kawasaki, S. Isobe, Y. Wang, N. Hashimoto and S. Ohnuki: Mater. Tran. **51**, No. 5 (2010) 1016-1019

[6] S. Isobe, H. Yao, Y. Wang, H. Kawasaki, N. Hashimoto and S. Ohnuki: Int. J. Hydrogen Energy **35** (2010) 7563-7567

[7] S. Isobe, A. Ono, H. Yao, Y. Wang, N. Hashimoto and S. Ohnuki: Appl. Phys. Lett. **96** (2010) 223109

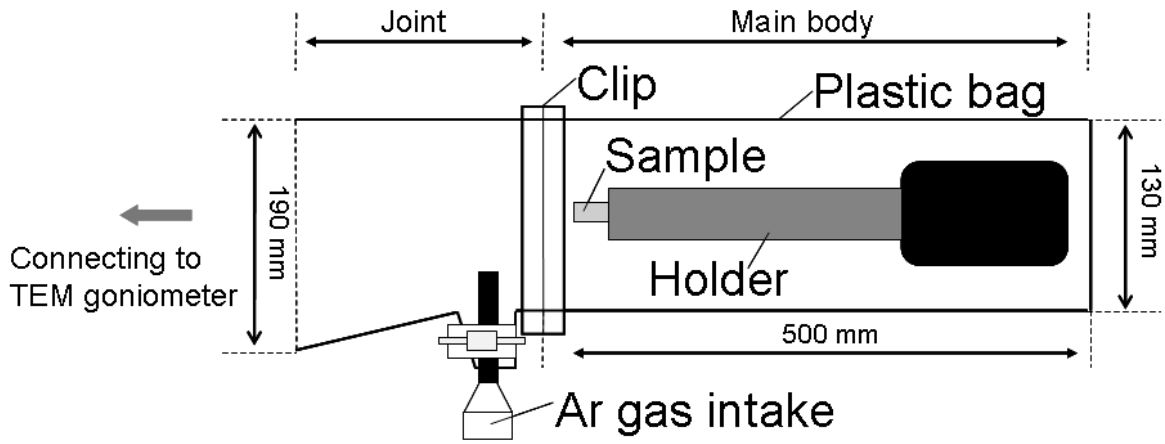
[8] P.E. Vullum, M.P. Pitt, J.C. Walmsley, B.C. Hauback and R. Holmestad: J. Alloys Compd. **509** (2011) 281-289

Figure captions

Figure 1 Schematic illustration of the plastic bag method.

Figure 2 (a) A bright-field image and corresponding diffraction patterns from a Li_3AlH_6 particle after exposure to air for 5 mins, (b) EDS spectrum from the particle.

Figure 3 (a) A bright-field image and corresponding diffraction patterns from a Li_3AlH_6 particle under the plastic bag method, (b) EDS spectrum from the particle.



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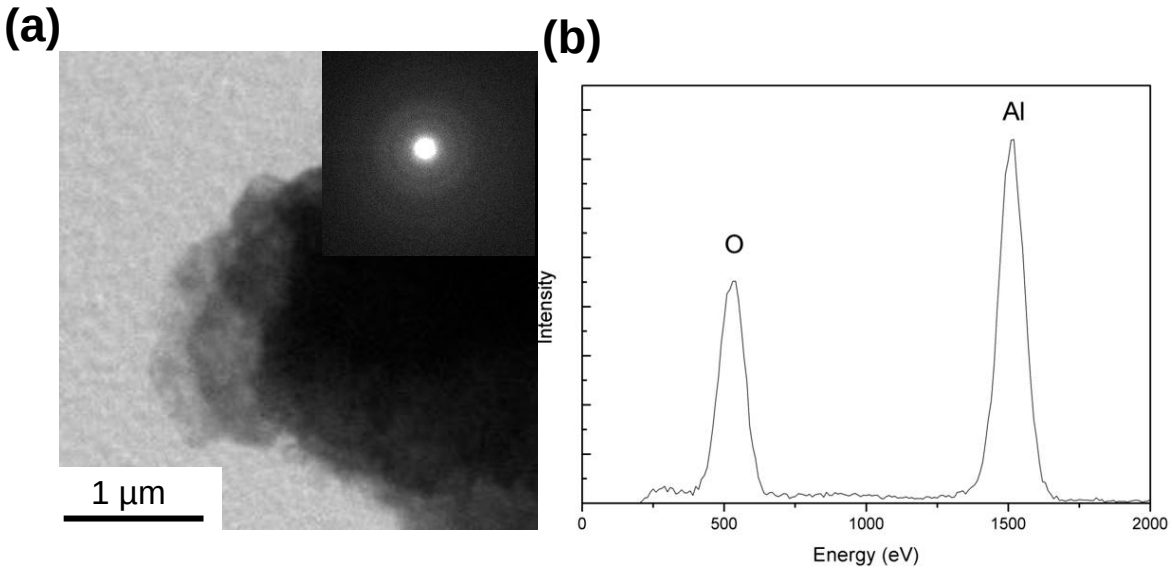

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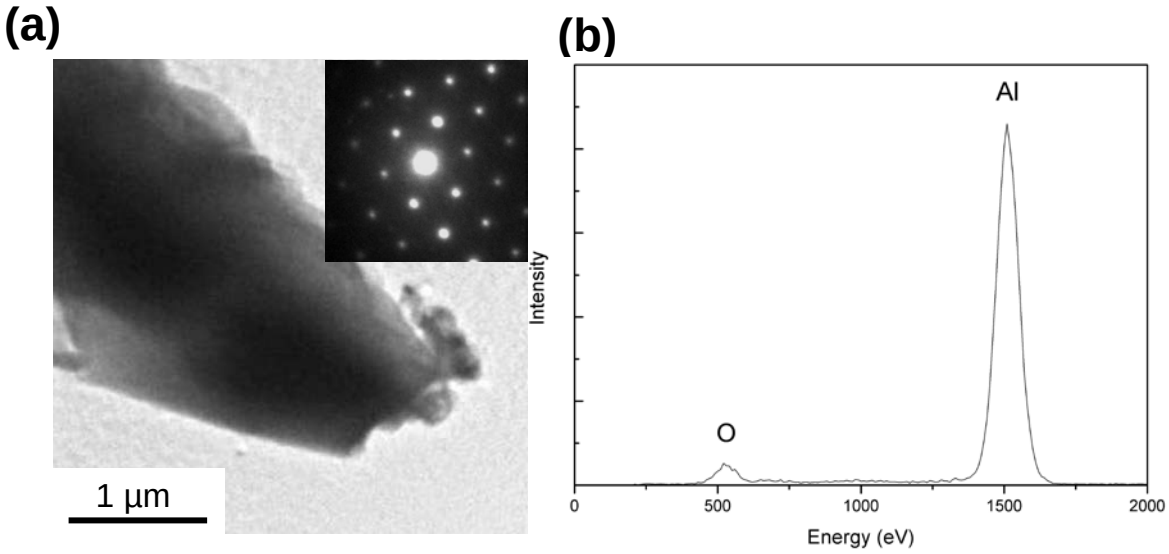

Figure 3 (a) A bright-field image and corresponding diffraction patterns from a Li_3AlH_6 particle under the plastic bag method, (b) EDS spectrum from the particle.