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Low Dose Electron Microscopy of Interlayer Expanded Molybdenum Disulfide Nanocomposites.

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Mg rechargeable batteries represent a safe and high-energy battery technology. However there is a lack of suitable cathode materials due to the slow solid-state diffusion of the highly polarizing divalent Mg ion. Recently, a different method has been proposed [1] i.e. interlayer expansion as a general and effective atomic-level lattice engineering approach to transform inactive intercalation hosts into efficient Mg storage materials without introducing adverse side effects. As for this work, the corresponding characterization using electron microscopy is reported. Transmission electron microscopy has been performed in the TEAM 05 microscope (NCEM-LBNL) in conditions of low dose rate in TEM mode together with a routine in MacTempas ® in order to apply the exit wave reconstruction procedure with 40 experimental images [2]. The samples are prepared by inserting a controlled amount of PEO (polyethylene oxide) into the lattice of MoS_2 in order to increase the interlayer distance. PEO is a rather beam sensitive substance that evaporates as the sample is observed in high dose rate. Four samples are prepared as described elsewhere [1]. Briefly, samples *peo1*- and *peo2*- have different MoS_2: peo molar ratios i.e., $Li_{0.16}MoS_2(PEO)_{0.49}$ and $Li_{0.13}MoS_2(PEO)_{0.98}$, respectively. Sample *com*-MoS2 represents the commercially available compound and *res*-MoS2 is a control sample that was determined to be $Li_{0.21}MoS_2(H_2O)_{1.0}$.

Figure 1 shows a typical experimental image of the *peo1*- sample taken with a dose rate of approximately 20 $e^-/Å^2s$ for a 1 s exposure. Some features are still visible and in order to recover all of the sample characteristics, an exit wave reconstruction procedure (EWR, MacTempas ®) is performed with 40 experimental images that differ in the focussing condition (-20 nm to + 20 nm). The result for this series of images is given in Fig. 2 that shows the corresponding phase image. As can be seen, all important features are now visible i.e., layers of both the inorganic MoS_2 compound and the polymer. Attempting to image this composite structure fails when the dose rate increases beyond 100 e^-/A^2s, the polymer becomes unstable and the sample changes in a rather short time. Figure 2 shows a summary of observations for the involved samples with the layer spacings that are indicated. The *com*-MoS_2 sample shows the expected value for the layer spacing which is similar to the control sample (*res*-MoS_2) i.e., 0.61 and 0.62 nm respectively. Interestingly, the interlayer distance in the *peo1*- and *peo2*- samples gives values rather close to the expected interlayer expansion, it varies from 1.22 nm (*peo1*-) to 1.40 nm (*peo2*-). This work is completed with image simulation and modelling of the observed samples.

References:

[1] Y. Liang, H. D. Yoo, Y. Li, S. Jing, H. A. Calderon, F. C. Robles, L. C. Grabow, Y. Yao. Nano Letters, under revision, February 2015.

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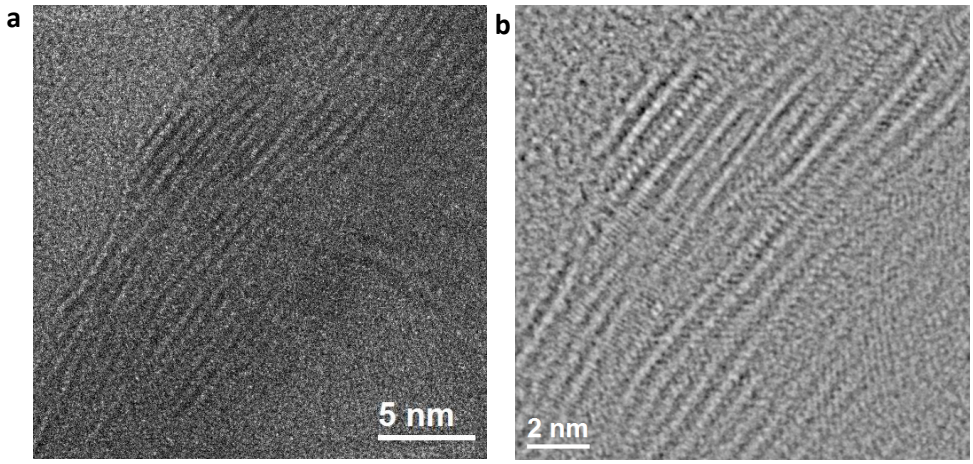

Figure 1. *peo1*-MoS_2 sample. (a) Low dose experimental image. (b) Phase image after EWR procedure.

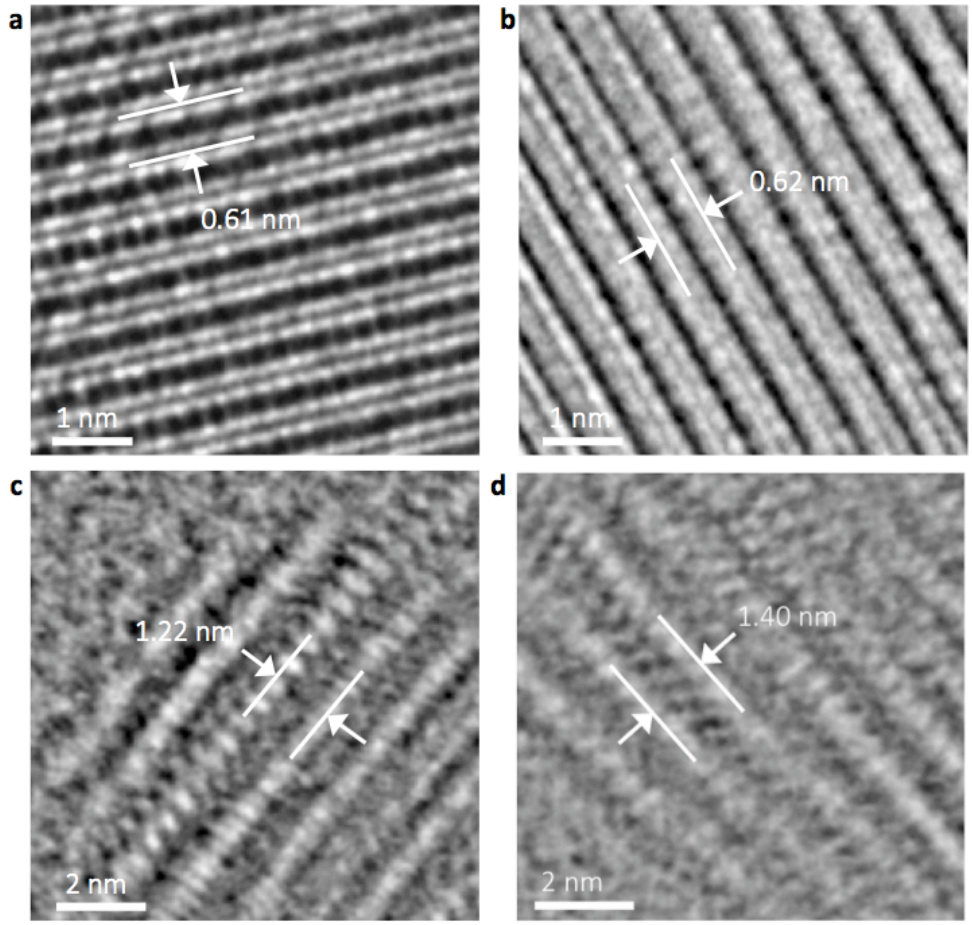

Figure 2. Representative phase images of the investigated samples. (a) Commercial MoS_2, (b) *res*-MoS_2, (c) *peo1*- and (d) *peo2*-MoS_2, respectively.