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Structures of Layered Materials After Reaction with Li/Na

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Understanding structural changes that occur at the atomic scale of the layered materials during reaction of Li /Na may help improve the performance of solid-state batteries using these materials [1-2]. New strategies and novel experimental designs are catalyzed by the availability of TEM holders for in-situ investigations. There are a growing number of reports pertaining to reactions of Li and Na with wide variety of materials ranging from transition metal dichalcogenides (TMDCs) to TM oxides (TMOs) both from bulk exfoliated flakes as well as chemically synthesized nanostructures. Qualitative features have been invariably shown in terms of change in contrast during reaction with Li, which may be a signature of insertion of Li into TMDCs/TMOs [3]. However, The viewing direction can complicate the issue in particular for reactions with nanostructures with a range of possible orientations in addition to the challenges of performing the in-situ reactions inside TEM itself [4]. There are limited reports from structural viewpoint demonstrating the changes in the layered materials with the insertion of Li/Na.

In this paper, we report a study of reactions between Li/Na with mechanically exfoliated flakes and powders of WS_2inside a FEI Tecnai F-30 TEM in-situ employing Nanofactory TEM- STM holder. The structural characterization of pre- and post-reacted specimens have been performed using SAD and HRTEM. The chemistry of the post-reacted WS_2 after Li/Na reaction has been studied using STEM-EELS and STEM-XEDS. The HRTEM images of the post-lithiated materials have been acquired using a Cs-corrected Titan ETEM. These results are compared with experimental images obtained using MoS_2, and initial computer modeling using DFT calculations for both Li and Na reactions.

Figure 1 shows the BF TEM images and electron diffraction patterns of reacted WS_2 powders. The pre-reacted WS_2 was indexed to be 2H-WS_2 along zone axis [0001]. Several extra reflections are observed apart from the 2H-WS_2 after Li/Na reactions clearly suggests the formation of new phases. This interpretation is supported by high-resolution phase-contrast images from the same specimen. The same region of the reacted specimen has been examined for chemical and structural changes. The STEM-EELS line profiles of Li/Na, W, and S along the reaction zone show undulations in the stoichiometry of W and S and in the content of Li/Na. This finding is also corroborated with HRTEM images coupled with image simulations using JEMS [5]. These results will also be compared with the structural changes in other layered materials, in particular MoS_2 and graphite.

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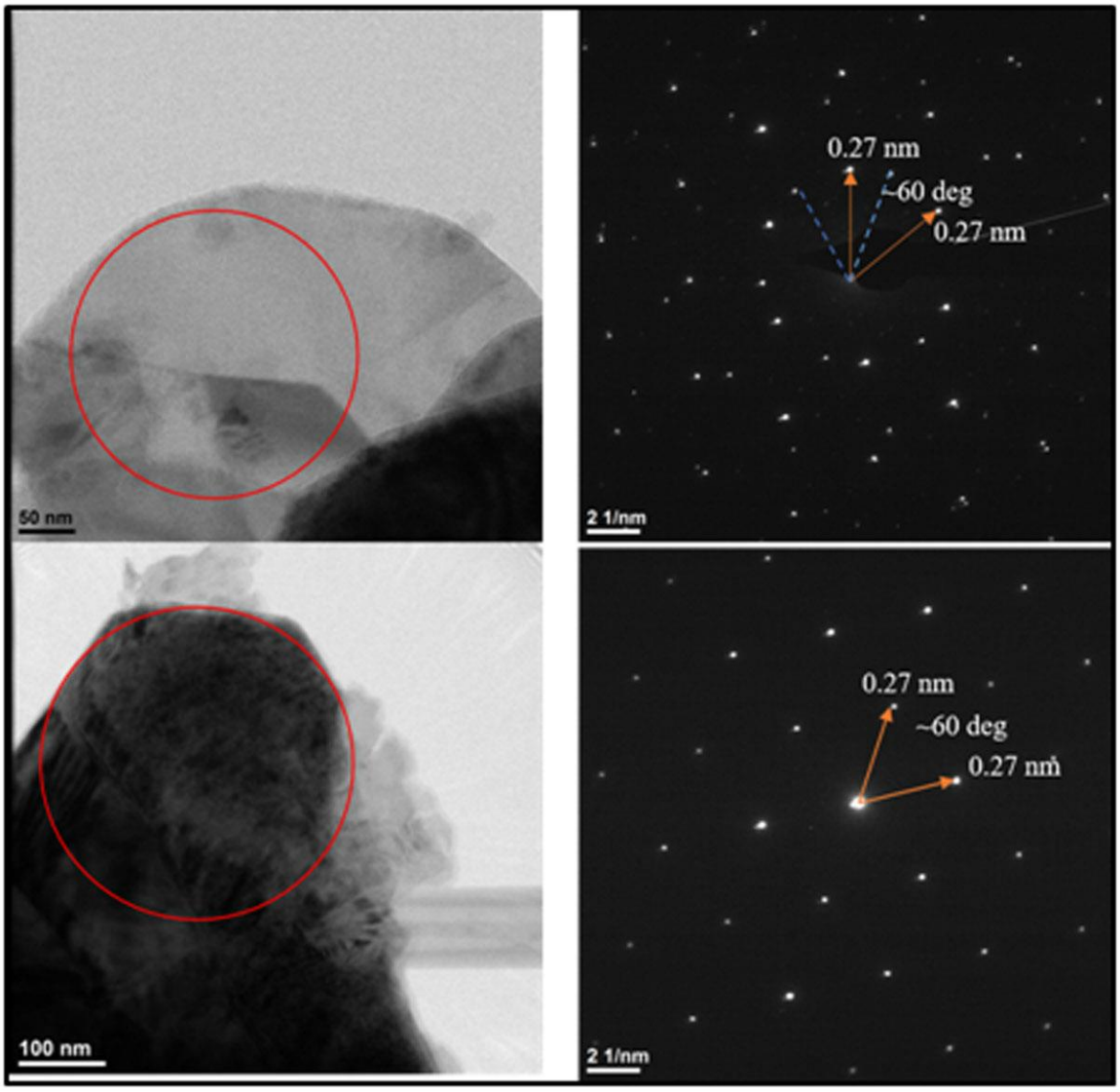

Figure 1. BF TEM images and corresponding electron diffraction patterns of reacted WS2 powder sample.

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