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Electron Irradiation Tolerance of Molybdenum Disulfide Two-dimensional Nanolayers Investigated from Electron Diffraction

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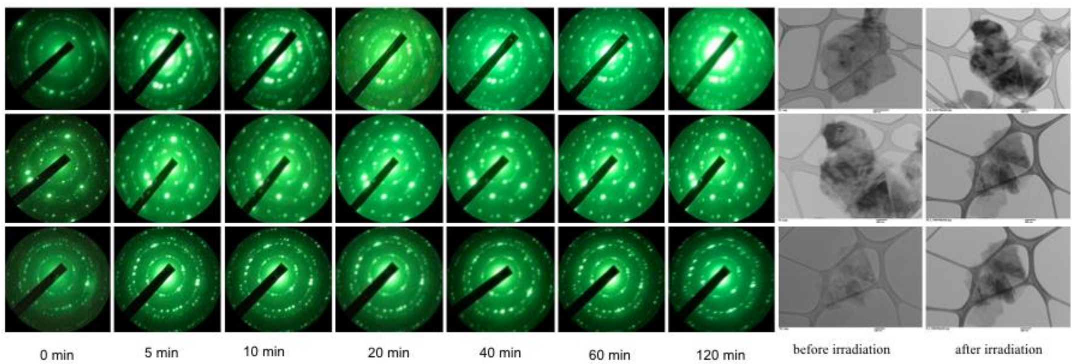
Transition-metal dichalcogenide (TMDC) materials, including MoS_2, WS_2, $MoSe_2$, and WSe_2, are layered semiconductors whose two-dimensional (2D) nanolayers have novel potential applications in novel nano-optoelectronics, optical sensors, catalysts, energy storages, and environments. MoS_2 2D nanolayers show thickness-dependent semiconducting behaviors, being the most interesting and important among the TMDC 2D nanolayers, having potential applications in the next generation flexible electronics, elastic energy storage, field-effect transistors, electronic switches, electronic devices and optoelectronic devices. Their unique properties are tuned by various defects. Therefore, it is necessary to investigate their tolerance under irradiations which create defects. Among various irradiations, electron irradiation was investigated recently on MoS_2 nanosheets [1-5]. It is believed that high-energy electrons generate defects in MoS_2 nanosheets. Additionally, TEM is a power tool to study microstructures and properties of MoS_2 nanosheets during which MoS_2 are exposed to electron irradiation. Therefore, it's also necessary to know the electron irradiation tolerance of the materials under electron beams of TEMs to avoid potential damages during observation. Here, selected-area electron diffraction was employed to in-situ study the electron irradiation on crystallographic structures of MoS_2 nanolayers.

MoS_2 2D nanolayers were prepared by the ultrasonication method. MoS_2 nanolayers were then dropped onto TEM grids and dried in air. TEM images and SAED patterns were taken on a JEOL microscope (JEM1400 Flash). During the whole procedures, the MoS_2 nanolayers were searched, focused and observed below a current density of 10 pA/cm^2. The chosen MoS_2 nanosheets were then irradiated under the electron beam with energy of 80 keV and 120 keV. The thickness of the nanolayers were roughly characterized from its scattering absorption constant [6].

Figure 1 shows SEAD patterns of MoS_2 nanosheets under electron irradiation with energy of 120 keV. BF-TEM images indicated that the examined nanosheets are consisted of several large nanosheets and some tiny nanosheets. The thickness of the main pieces should be about 100 nm and Kichichi lines were observed. Obviously, the SAED patterns changed significantly even after 5 min electron irradiation under 120 kV with electron current density of 200 pA/cm^2, indicating structural change. The diffraction spots diffused with the increasing irradiation time under the dose, indicating more defects were produced under the irradiation. However, TEM images indicated that the main frame of the nano-sheets did not change significantly under the irradiation. A possible explanation is that the electron irradiation only caused the S deficiency. Under lower irradiation doses, such as 50 pA/cm^2, the SAED patterns slightly changed with irradiation time. Therefore, electron irradiation with energy of 120 keV degraded the crystallographic structures of MoS_2 nani-sheets and the degradation also depended on the irradiation doses.

Figure 2 shows SAED pattern of irradiated MoS_2 nanolayers at lower energy. Obviously the low-energy irradiation does not degrade the crystallographic structure of MoS_2 nanolayers. The conclusion is in agreement with theoretical models and STEM experiments [2]. MoS_2 monolayers and bilayers should be more sensitive to electron irradiation. More work is being carried out and will be compared with the reported results.

In summary, the structural stability of MoS_2 nanosheets were in-situ investigated from selected area electron diffraction. The nano-sheets were stable at low-energy electron irradiation (80 keV) while degraded under high-energy electron irradiation. The conclusion is in agreement with reported and would



help us to avoid structural damages to MoS2 nano-sheets that are characterized on TEM.

Figure 1. SEAD patterns of MoS_2 nanolayers irradiated at 120 kV under (top) 200 pA/cm^2 (mid) 100 pA/cm^2 and (bottom) 50 pA/cm^2 current density.

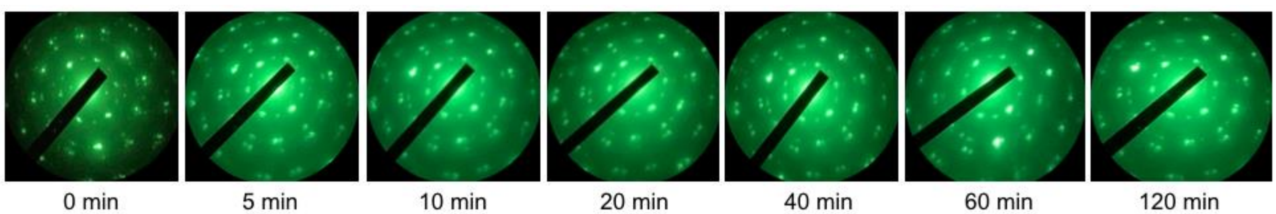


Figure 2 SAED patterns of MoS_2 nanolayers irradiated at 80 kV under 200 pA/cm^2.

References:

[1] H. P. Komsa, et al., Phys. Rev. Lett. 2012, 109, 035503.
[2] A. Garcia et al., Ultramicroscopy, 2014, 146, 33.
[3] E. Rotunno, et al., 2D Mater. 2016, 3, 025024.
[4] W. M. Parkin, et al., ACS Nano 2016, 10, 4134
[5] G. Y. Zhao, et al. Appl. Sci. 2019, 9, 678.
[6] S. Rubino, et al. Microsc. Microanal, 2016, 22, 250.
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