Mitigation of beam damage on MoS2 using electrostatic beam blanking in TEM

Mr Joakim Kryger-baggesen[1], Mrs. Noopur Jain[2], Mr. Mark van Rijt[2], Mr. Ruud Krijnen[2], Mr. Marteen Wirix[2], Mr. Pritam Banerjee[3], Mr. Joachim Dahl Thomsen[5], Mr. Jakob Kibsgaard[1,4], Mr. Christian Damsgaard[1,3], Mr. Joerg Jinschek[1,3], Mr. Stig Helveg[1]
[1]Center for Visualizing Catalytic Processes (VISION), Department of Physics, Technical University of Denmark, Kgs. Lyngby, Denmark, [2]Thermo Fisher Scientific, Achtseweg Noord 5, 5651 GG Eindhoven, Eindhoven, The Netherlands, [3]National Center for Nano Fabrication and Characterization, Technical University of Denmark, Kgs. Lyngby, Denmark, [4]Surface Physics and Catalysis, Department of Physics, Technical University of Denmark, Kgs. Lyngby, Denmark, [5]Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Forshungszentrum Jülich, 52428 Jülich, Jülich, Germany

Background

Transmission electron microscopy (TEM) has allowed for impressive opportunities to visualize matter at the atomic scale. In such experiments, the constituent atoms typically respond dynamically to the electron beam irradiation, alter the sample and, hence, modulate image contrast and intensities. While mitigation strategies have focused much on electron energy and dose variation, there is a growing emphasis on the role of the electron dose rate.

That is, for a broad class of materials, the electrons incident on the specimen is considered to cause phonon or electron excitations because the impingement rate is higher than the subsequent relaxation rate. Consequently, accumulation of energy and or charge leads to bond rupture and atom displacements. In this picture, pulsing the electron illumination seems beneficial as it enables a sharply defined maximum electron impingement rate to match the onset causing sample alterations.

Here we show how a structured electron beam can suppress beam-induced alterations of a two-dimensional MoS2 by 50% compared to the continuous mode.

Method

The present experiments are conducted on a Thermo Fisher Scientific SPECTRA ULTRA microscope operated at 300 kV. An electrostatic beam blanker (ESBB) system developed by Thermo Fisher Scientific, is used to structure the electron beam with a 10 ns temporal electron delivery window with repetition rate of 1MHz. The microscope was operated in pulsed and continuous mode. In each mode, an electron diffractogram of the MoS2 was recorded at similar electron dose-rate and total dose. In pulsed mode the beam blanker was tuned to generate 1 electron per pulse. The area exposed was kept the same during acquisition to compare the behavior of the specimen in both modes.

Results
The electron diffractograms of MoS2 show for increasing electron dose and dose-rate had different evolution of the central diffraction peak intensities in pulsed and continuous modes. In continuous mode the diffraction peaks faded to ~60% of the original intensity due to loss of crystallinity caused by beam damage. In pulsed mode the diffraction peak intensities faded only by ~1%. A similar effect was observed by (1,2) delivering dose electron-by-electron and allowing relaxation time reduces beam damage. Further we found that the effect of pulsing decreased when increasing the number of electrons per packet. This is the first time that this effect is shown with only a beam blanker and on a nanosecond time scale.

Conclusion
Thus, the comparison of pulsed and continuous electron beams suggests that the electron-induced degradation of MoS2 is a multi-electron excitation rather than a single-electron scattering event, consistent with ref. (3,4) and that excitations on the microsecond timescale must be circumvented to maintain structural integrity of the MoS2 sample. We anticipate that beam blanker pulsed electron delivery may benefit the stability of other materials and enable novel experiments with beam sensitive materials in general.(5)

Keywords:

Beam damage, structured electron illumination

Reference:

1. C. Kisielowski, P. Specht, B. Freitag, E. R. Kieft, W. Verhoeven, J. F. M, van Rens, P. Mutsaers, L. Jom, S. Rozeveld, J. Kang, A. J. McKenna, P. Nickias, D. F. Yancey, Advanced Functional Materials, 29, 11 (2019)
2. E. Vandenbussche, D. Flannigan, Nano Letters 19(9) 6687-6694 (2019)
3 . T. Susi, J. C. Meyer, J. Kotakoski. Nat. Rev. Phys. 1, 405 (2019)
4 . F.-R. Chen, D. van Dyck, C. Kisielowski, L.P. Hansen, B. Barton, S. Helveg, Nat. Commun 12, 5007 (2021)
5. We thank Felix van Uden for preparing the microscope and Maarten Waaijer, Erik Kieft, Boy Markus, Bert Freitag and the rest of the Thermo Fisher Scientific team for participating in the initial discussions. Further we thank Tim Booth from the 2DTU group at DTU for assistance in providing MoS2 samples. The Center for Visualizing Catalytic Processes is sponsored by the Danish National Research Foundation (DNRF1462).