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Variation in Band Gap Contrast in Natural Molybdenum Disulphide (MoS_2) with BSE Collection Angle and Stage Bias using a Segmented Annular BSED.

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Molybdenum disulphide (MoS_2 or the naturally occurring 'molybdenite') is a wide and variable band gap material. In the crystalline form it exhibits a near-perfect basal cleavage. Backscattered electron imaging of freshly cleaved surfaces exhibit a contrast that details lattice strain effects that give rise to variation in the band gap. This effect is particularly well illustrated in Rutherford backscattered ion images (figure 1) relative to conventional BSEI (Figure 2) but the image contrast is consistent. MoS_2 has been widely studied as a potential material in modern photonics and well understood imaging is important.

Imaging of a freshly cleaved MoS2 sample using a segmented annular BSED (DBS) with and without stage biasing has provided images with markedly different contrasts as a function of working distance, signal collection angle and stage bias. One set of images, from the different detector rings, are provided in figure 3, collected with a high stage bias (4kV). The first image from the innermost annulus shows strong but diffuse regions in the sample. Imaging with the second annulus, this 'diffuse' contrast is reversed. With the third annulus, the image "contrast' has less dynamic range but sharply defines complex band gap variation. Finally, in the image from the outermost annulus a rupture in the sample has contrast that is reversed relative to the images but otherwise the image retains the well-defined band gap contrast. Monte Carlo modelling constrains the depth of image data to ~ 0.2 micron (CASINO v4.2) at a beam landing energy of 5 keV. Modelling of the DBS collection angles using proprietry FEI software (1) indicates collection angles of A: 7 – 12, B: 12 – 19, C: 19 – 27, and D: 27 – 34 degrees for the BSED rings imaged in figure 3, respectively.

The sample has been also imaged with a landing energy of 1keV and 10keV, a range of WD and stage bias of 0-4 kV in 1kV increments, and using the ET-SED, upper column 'mirror' detector and the pole-piece TLD. A simple polished rock sample has been imaged under the same conditions and Z contrast is maximized in the image from the outermost BSE ring with the stage biased at 4kV (figure 3). With a 4kV stage bias the emitted electrons clearly have significantly modified energy characteristics, changing the nature of the collected signals, as is illustrated. It seems that the lower energy SE are directed up the column towards the 'mirror' detector above the TLD. The optimum mass and band gap contrast is found to lie in the outer rings under these conditions, suggesting that the band gap contrast relates to the higher energy SE that are usually coincident onto the ET-SED. Under biased stage conditions, the annular BSED also collects components of the emitted SE signal. The inversion of contrasts in the inner BSED rings will be discussed. A further uncertainty is the reversal of contrast of the hole in the MoS_2.

References

(1) FEI is acknowledged for access to their DBS modelling software.
(2) Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy (DOE) under contract DE-AC0494AL85000.

Figure 1: Rutherford backscattered ion image of band gap variation in a cleaved MoS_2 sample using a 37 kV He ion.

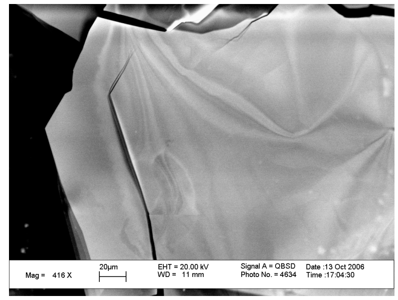

Figure 2: BSEI of band gap variation in the same cleaved MoS_2 sample using a conventional FESEM at 20 kV.

(a) 0V stage bias – MoS_2 (b) 4kV stage bias – MoS_2 (c) signal profile from (b) (d) 4kV stage bias - rock

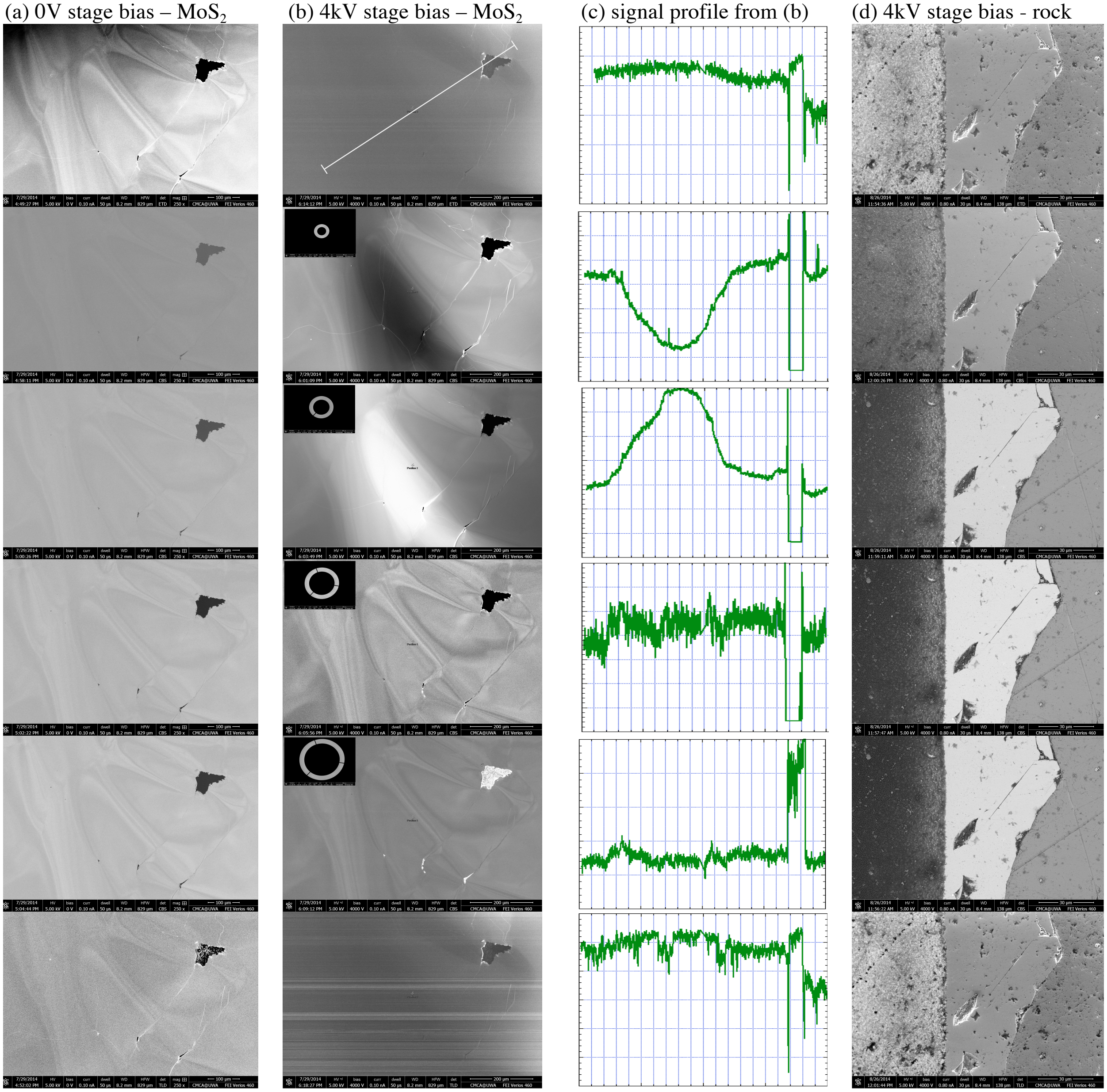

Figure 3: From top: ET-SE, DBS-A, DBS-B, DBS-C, DBS-D and TLD images of biased (4kV) and unbiased cleaved MoS_2 and a polished rock sample (with vertical carbon ink line down the left-hand side) together with the signal profile along the line shown in the top ET-SEI from the biased condition.