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Controlling Beam-Sample Interaction in Low Dimensional Materials by Low Dose Rate Electron Microscopy

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In recent years it became possible to image the atomic structure of low dimensional materials with single atom sensitivity. Clearly, the development of atomic resolution aberration-corrected electron microscopy using low voltages below 100 kV plays a key role in this achievement [1] since it relaxes limitations that are set by beam-sample interactions. It is commonly argued for graphene and similar materials that voltage-dependent threshold values for the displacement of single atoms dominantly contribute to sample degradation. However, our understanding of beam-sample interactions will remain partial if it is ignored that electron beam-induced sample excitations reach far beyond the considered atom displacements and that they are commonly reversible [2]. In fact, the energy deposited by the electron beam onto a sample is greatly current dependent, too, and can be varied by more than six orders of magnitude [2]. In two-dimensional materials, however, beam currents larger than 10000 e/Å^{2}s must be used to detect single light atoms in single images with exposure times around one second. The Figure 1 gives an example for the detection of a boron vacancy (V_B) in a BN double layer [1]. In this reference it is shown that displacement damage does not limit the imaging process of this point defect at all. Instead, at least 2 vacancy configurations coexist in the images with formation energies that differ by ~ 400 meV. If one adopts the view that there is a temperature equivalent for each energy, as schematized in Figure 1, the local temperature during observation would exceed several thousand Kelvin. It can be greatly reduced if the beam current is lowered, which necessarily degrades resolution and sensitivity.

Low dose rate in-line holography [3] at variable voltages allows us to overcome the dilemma how to maintain atomic resolution and single atom sensitivity while addressing beam sample interactions. The method exploits best practices developed for the imaging of radiation sensitive, biological objects and reversible object excitations. In Figure 2 it is compared with the established way of acquiring single high resolution images. Note that the low dose rate approach greatly reduces object motion (compare Fig. 2 a-c with Fig. 2g where 50 images were merged), allows for testing if beam-induced object alterations occur (Fig. 2e,f), preserves the structure of small particles (Fig. 2e,f) and creates a similar contrasts with a lower total dose (compare Fig. 2a - c with Fig. 2g).

This contribution highlights investigations of graphene, BN, MoS_2 and other materials with low dose rate in-line holography using acceleration voltages between 80 kV and 300 kV. They show that the approach is generally applicable and maintains the genuine structure of low dimensional material, small nanoparticles and even molecules to an end that is currently explored. In particular, it is of great benefit to experiments performed at elevated pressure and temperature [4].[5]

[1] N. Alem, O.V. Yazyev, C. Kisielowski, P. Denes et al. PRL 106, 126102 (2011)
[2] C. Kisielowski, L.-W. Wang, Petra Specht et al. Phys Rev. B 88, 024305 (2013)
[3] C. Kisielowski, P. Specht, S.M. Gygax, B. Barton, H.A. Calderon, Micron 68, 186 (2015)

[4] S. Helveg, C.F. Kisielowski, J.R. Jinschek, P. Specht et al., Micron 68, 176 (2015)
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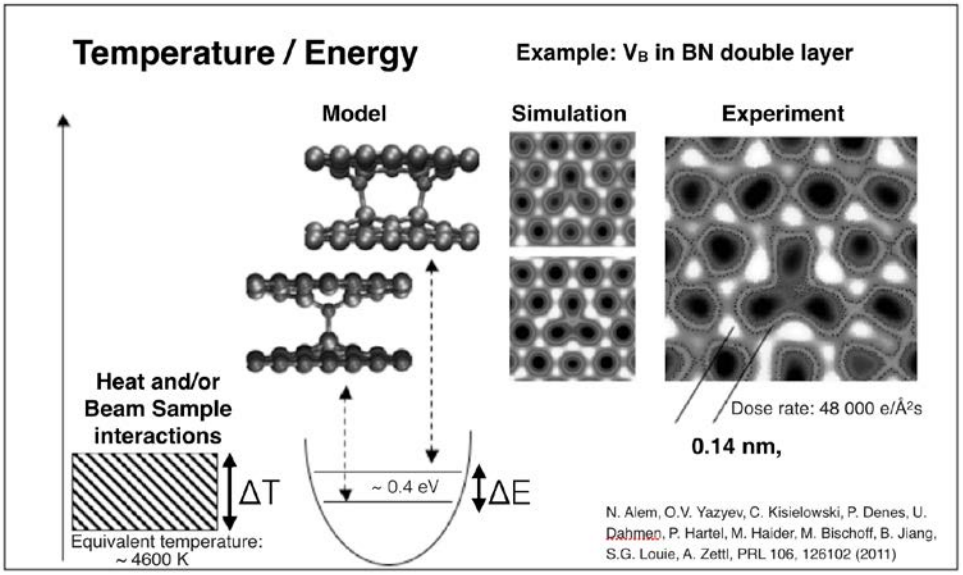

Figure 1:

A schematic representation of two V_B configurations on equivalent temperature and energy scales. If large beam currents are applied, the two structurally different configurations coexist in the images. Their formation energies differ by 0.4 eV, which corresponds to an equivalent temperature of ~ 4600K.

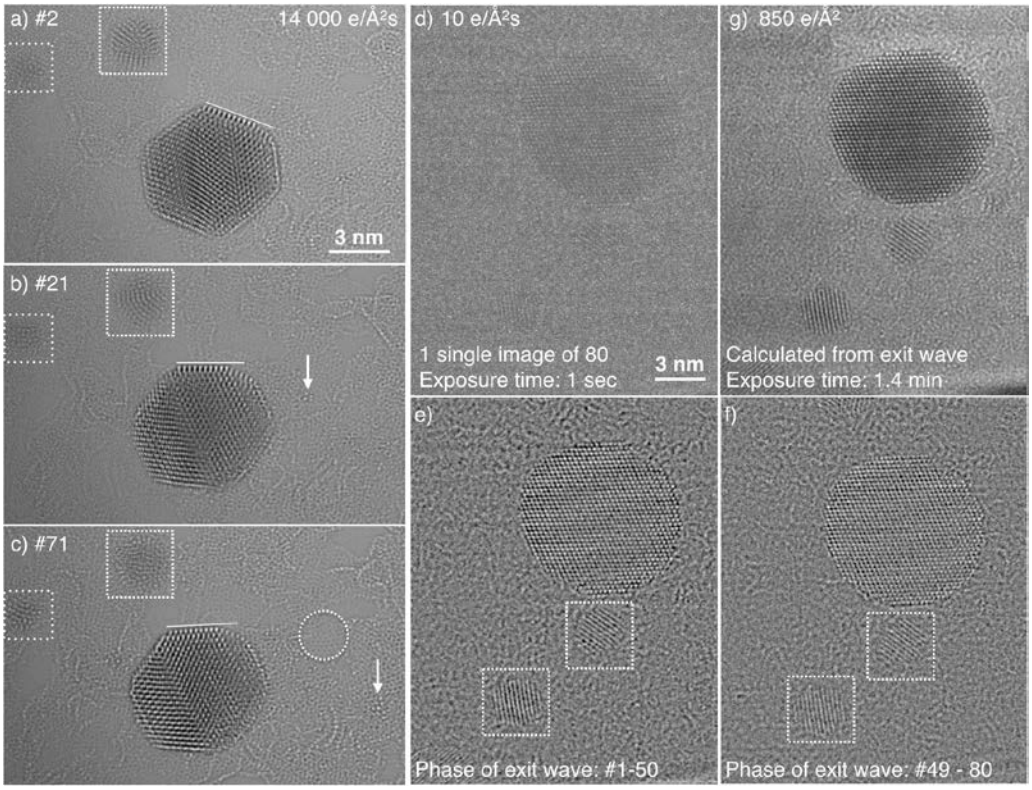

Figure 2:

The traditional approach to acquire single high resolution images (a - c, 1 sec exposure time) is compared with low dose rate in-line holography (d-g) [3]. Gold particles on an amorphous carbon support are shown. For details see text.