Low Dimensional Materials and Catalysts

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Exploring optimum parameters for off-axis holograms at 80 kV with C_s-corrected Titan 80-300

D. Geiger[1], J. Biskupek[1], U. Kaiser[1]

[1]Uni Ulm, Ulm, Germany

dorin.geiger@uni-ulm.de
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Studying 2D structures like graphene [1] has become in the last years of major scientific interest because of their specific mechanical and electrical properties. A large number of applications e.g. as sensor, in nanoelectronics, hydrogen storage or lithium-ion batteries etc. require deep knowledge of their properties at atomic level. One of the most important investigation technique applied in this field represents the high resolution transmission electron microscopy (HRTEM) at 80 kV (or lower) (figure 1).

In order to reconstruct the object exit wave, off-axis electron holography [2] is one of the most appropriate methods. Using C_s-corrected transmission electron microscopes [3], significant progress was achieved in the last decade, especially in decreasing the phase detection limit and enhancing the spatial resolution [4]. Higher hologram contrast is required to reduce the quantum noise [2]. This can be realized by adjusting the TEM illumination to obtain increased beam coherence. For the Titan microscopes, the illumination system consists mainly of the Schottky field emission gun and three condenser lenses, C1, C2 and C3 followed by the minicondenser. To achieve the necessary interference fringe contrast, the electron beam has to be adjusted to a high elliptical spot. This is only possible by switching off the second condenser lens C2, among others because of the performance of the condenser stigmator.

By taking the C1 – C3 condenser lens setting, the coherent elliptical illumination could be optimized but the coherent dose rate remains under conventional extraction voltages too low. Because our instrument is not equipped with a high brightness gun, we analysed other possible electron gun emission settings to achieve better performances. A large range of gun extraction voltages were investigated to find an optimum. Special gun settings, allowing better energy dispersion for improved EELS measurements at 80kV, were already applied at the Titan instrument in Ulm [5]. At lower extraction voltages (down to 1.7 kV), the energy spread of the emitted electron beam should be significantly lower [6]. Consequently, an additional gain in contrast could be achieved at higher spatial frequencies of interest.

In parallel, different gun lens excitations were tested as well, trying to reduce further the quantum noise. This makes it possible to use non prohibitive exposure times per single hologram. Certain minimal exposure times are necessary, even if we process hologram series to increase the total exposure time, and on this way to improve the signal to noise ratio. This happens among others because of the object drift, which is, at least for the current stages, unavoidable at atomic resolution, and should be numerically corrected a posteriori by using single reconstructed holograms. If these are too noisy, the drift compensation programs cannot work reliably.

The effectively achieved performance could be analysed in figure 2 and 3, where the dependence of the hologram contrast on the fringe spacing and on the biprism voltage is shown. Although the hologram contrast is really competitive, the necessary high minimum exposure time per hologram of about 5 to 8 seconds requires a very high stability of the TEM stage, which is difficult to achieve. The resultant stability of all other subsystems of the Titan microscope (excepting the stage) is remarkable, allowing good fringe contrast (~15%) of empty holograms with fringe spacing of ~0.036nm at ~1.3Mx magnification even for exposure times of 20 seconds or more.

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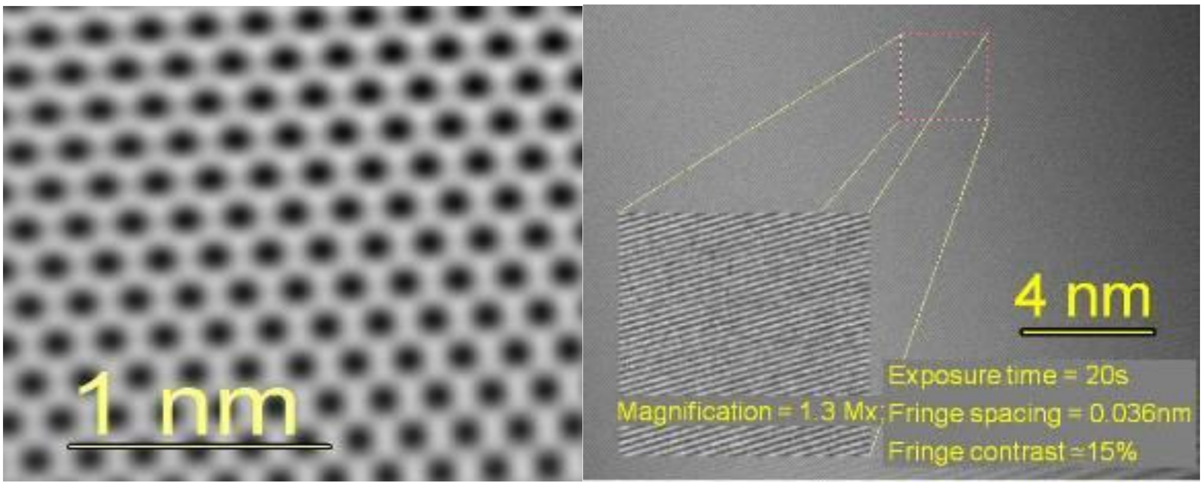

Figure 1. Conventional HRTEM image of graphene (left) and a detail of an empty hologram showing interference fringes of 15% contrast (right) taken at 80kV with the Cs-corrected Titan 80-300 in Ulm.

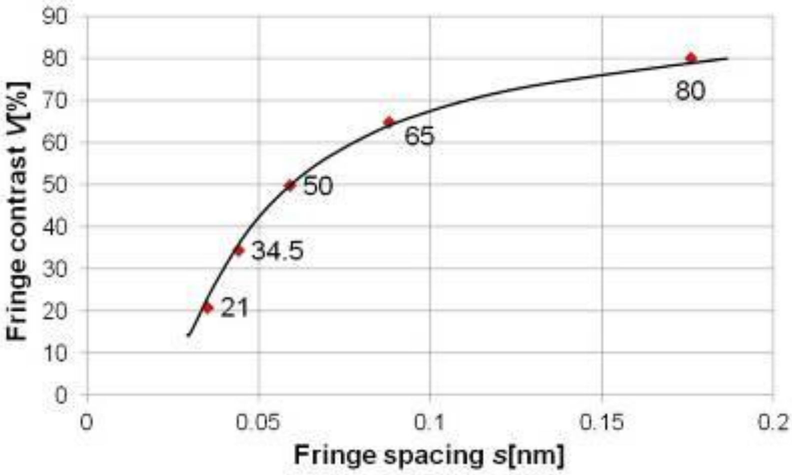

Figure 2. Fringe contrast as function of fringe spacing for Cs-corrected Titan 80-300 in Ulm (accelerating voltage 80kV, exposure time 4s, magnification 1.3 Mx).

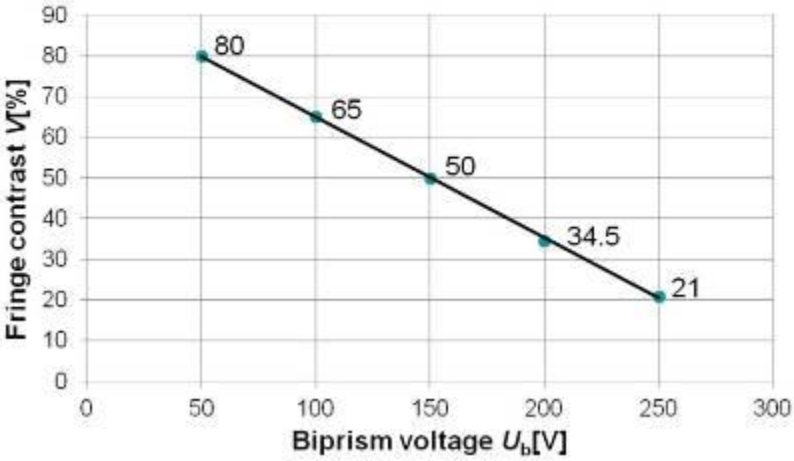

Figure 3. Linear fringe contrast dependence on the biprism voltage for Cs-corrected Titan 80-300 in Ulm (accelerating voltage 80kV, exposure time 4s, magnification 1.3 Mx).