Low-voltage TEM - current status and future prospects

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Recent technical advances in transmission electron microscopy (TEM) [1,2] created trends towards lower accelerating voltages such as 80kV or 60kV. This enables for the first time atomically-resolved structure analysis of materials of low-dimensions and/or low atomic number Z. At higher voltages, the low intrinsic contrast and the high susceptibility of these materials to electron-beam induced knock-on damage prevented defect analysis of materials such as graphene, carbon nanotubes, and fullerenes. Basic questions in physics and chemistry such as — What is the exact atomic structure of defects ? — or — How is the dynamic behaviour — or — What are the structures of amorphous materials? — and the like constantly addressed over the past by our respected colleague and mutual friend, David Cockayne [3-4], microscopist now begin to answer in direct space on the atom-by-atom level [5-11]. Also the theoretical result that it is the tolerable electron dose that limits the achievable specimen resolution in high-resolution TEM images [12] is now exemplified: Microscopists now can use an electron beam with different energies and doses to probe in graphene structural rearrangements of defects and grain boundaries and determine knock-on thresholds from atomically resolved image sequences on the quantitative level [13]. This means we now begin to understand the difficult process of the collision process between the beam electrons and the target atoms, first for selected materials such as C^{12} and C^{13} graphene layers [13]. We visualize bonding effects in high-resolution TEM images of covalently bonded light elements (B, C, N) [14]. Moreover, we use graphene and carbon nanotubes as substrates for radiation-sensitive compounds and take advantage of the dynamics of atom knock-on processes under the electron beam to understand fundamental new transformation routes between carbon nanostructures atom-by-atom [15,16,17]. Further, graphene can now serve as an extreme thermal platform for physisorbed carbon species whose transformations can be imaged under the influence of Joule heat and electron irradiation atom-by atom [18] or simply serves as an substrate for biological structures [19].

Recently microscope develoments are addressing electron energies down to 30keV [20] or 20keV [21] because many low-Z materials require imaging at energies appreciably lower than 60keV. As an example Fig. 1 shows Os-cluster-filled single-wall carbon nanotubes imaged with 20keV, 40keV and 80keV electrons. In Fig.1 (right, 20kV) a stable and (left, 80kV) a destroyed carbon nanotube after exposure to comparable electron doses is shown together with an intermedate situation at 40kV acceleration voltage (middle). At 40kV and 20kV, the chromatic aberration limits predominately the resolution because this aberration is not corrected so far in our prototype microscope. We demonstrate by means of image calculations [22,23] that at 20kV the contrast, even for graphene, a one-carbon-atom-thin material, cannot be described by means of the weak phase-object approximation, and that correction of chromatic aberration is a prerequisite for obtaining high-resolution, high-contrast zero-loss and energy-filtered inelastic images [23]. In EELS mode we take advantage of the exceptionally low background noise at low voltages enabling the investigation of plasmons in single [24] and multi-layer graphene using angle-resolved EELS.

We outline the fully-corrected transmission electron microscope for spatial imaging, diffraction and spectroscopy using low-energy electrons optimised for the range between 20 and 80keV.

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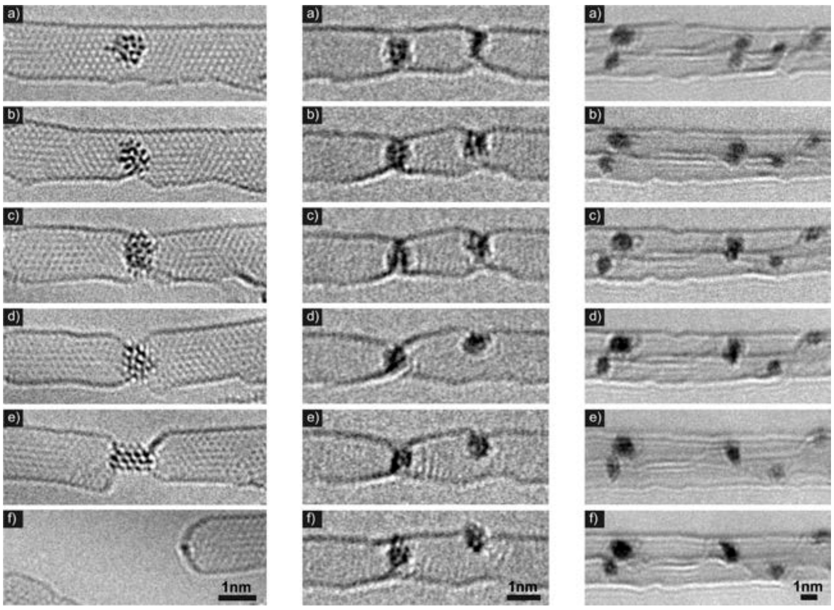

Figure 1. *Spherical aberration-corrected HRTEM images of Os-cluster filled SWNTs imaged at electron doses of*
a) $1x10^8 e/nm^2$*,*
b) $4x10^9 e/nm^2$*, c)* $6x10^9 e/nm^2$*,*
d) $7x10^9 e/nm^2$*, e)* $8x10^9 e/nm^2$*,*
f) $9x10^9 e/nm^2$
at 80kV (left), 40kV (middle) and 20kV (right) with the prototype Zeiss-SALVE microscope (Libra-based) equipped with an electrostatic monochromator and an imaging energy filter.