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Sample Dependent Resolution of Transmission Electron Microscopy in Water or Ice

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Transmission electron microscopy (TEM) and scanning TEM (STEM) of specimens in liquid water or ice are essential for nanoscale characterization of structures and processes in materials science, chemistry, biology, and other fields. But despite their frequent usage, it is often unclear what spatial resolution can be achieved for a given sample. The resolution for experiments in water is typically not limited by the optics of the electron microscope but by two factors determined by the sample. The first factor is spatial broadening and broadening of the energy spread of the electron beam due to electron scattering in the water layer [1, 2]. Secondly, these specimens typically withstand a limited accumulated electron density only, so that the resolution is determined by the achievable signal-to-noise-ratio (SNR) [3]. Both factors depend on the materials the sample is composed of, the used contrast mechanism, and the detection settings. In order to achieve the best possible resolution for a particular experiment and minimize beam-sample interactions, it is thus advantageous to calculate the resolution as function of sample parameters and optimize the detection settings.

A theoretical model was developed for calculating the optimal spatial resolution for TEM and STEM in liquid water or amorphous ice as function of the different microscope settings and sample parameters [4] (Fig. 1A). The model combines the usually well-known electron-optical resolution with calculations of the SNR-limited resolution that depends on the materials properties, and of both spatial- and temporal beam broadening. The resolution was calculated as function of the sample thickness and the vertical position of the object under observation in the water layer. Calculations were performed for samples containing either gold nanoparticles or carbon nanoparticles in a layer of water or amorphous ice. These two types of samples represent two important classes of experiments. The first involving nanomaterials of high atomic number (Z), for example, gold nanoparticles or catalytic nanoparticles in water. The latter representing samples of low-Z materials in water such as biological cells, and protein complexes.

It was found that the opening angle of the used detector plays a crucial role and requires optimization because it determines the amount of contrast, and moreover its settings differ between the detection of carbon or gold. Optimizing the angle can also be used to reduce the effect of spatial beam broadening in thick samples for BF STEM (Fig. 1B). The most critical parameter in an experiment is the total number of accumulated electrons per unit irradiated area, which is usually termed the electron dose. Calculations demonstrate that the spatial resolution scales with the electron dose as $D^{-1/4}$ (Fig. 1C). This finding contradicts general belief that the resolution scales with $D^{-1/2}$ but in those earlier calculations, the SNR was considered as constant, while it should in fact be considered as variable in the calculations since it depends on the object size. The scaling law has important implications. On the one hand, it follows that even small resolution improvements require a large increase of the electron dose. This is also experimentally observed, for example, in single particle tomography, where over 10^4 identical images recorded at low SNR are averaged to achieve a spatial resolution better than 0.5 nm. On the other hand, sample damage by electron beam irradiation can be reduced by giving up only a small amount of resolution. For example, a factor of 3 lower resolution would gain two orders of magnitude in dose. The demonstrated calculation method is applicable to a broad range of other materials and sample types.

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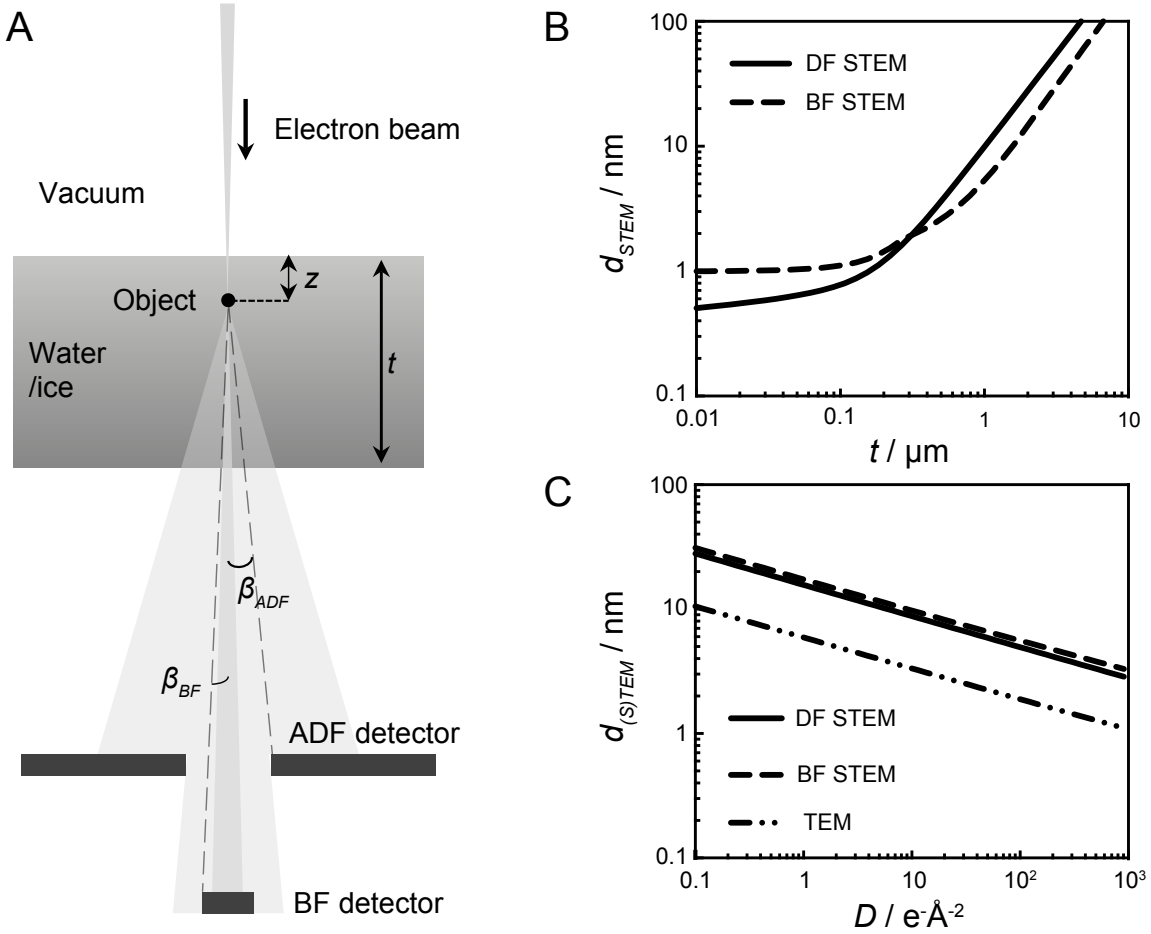

Figure 1. Calculation of the sample-limited spatial resolution of STEM and TEM. (A) Schematic representation of STEM of an object at depth z within a liquid water or ice layer of thickness t. The opening semi-angle of the annular dark field (ADF) detector is indicated as β_{ADF} and that of the BF detector as β_{BF}. For TEM, a lens projects the electron beam on a camera positioned below the sample. (B) Resolution as function of the water thickness for DF- and BF STEM of a gold nanoparticle at the bottom of a water layer. DF STEM provides the best resolution for thin liquid layers but as the liquid thickness increase, beam broadening effects become prominent. For thicker liquids, BF STEM has an advantage over DF STEM because the influence of beam broadening is reduced. (C) Resolution as function of the electron dose for DF- and BF STEM, and phase-contrast TEM of a carbon object in the middle of a water layer of 0.1 µm thickness. TEM provides the best resolution. Improving the resolution by an order of magnitude requires four orders of magnitude more electron dose.

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