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Advancing Nanotomography of Polymer Systems

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Polymer materials, like block copolymers and polymer blends, have a wide range of properties that are dependent on their structure at the nanoscale [1]. Electron tomography (ET) is mostly used for understanding these relationships, since it can provide intuitive real-space representations of three dimensional (3D) morphologies. In ET, the 3D morphology of an object generally is reconstructed from a series of projections which are acquired by rotating a specimen within a (scanning) transmission electron microscope (STEM) [2]. However, there are several limiting factors on the reconstruction quality [3]. For example, the maximum tilt range is limited by the sample holder and the sample stage, which leads to incomplete information, i.e. the missing wedge in the Fourier space. Moreover, the signal-to-noise ratio of the reconstruction is proportional to the applied electron dose. However, polymers systems are sensitive to electron beam irradiation [4] and therefore, should not be irradiated with too high dose levels, otherwise, the systems will be damaged. The reconstruction quality is degraded by the above factors which in turn hamper structure interpretation of an unknown object and subsequent quantitative analysis. In this work, we focus on advancing data acquisition and processing for reliable and accurate quantification at a predetermined electron dose.

The general approach is based on simulating the entire electron tomography workflow from image formation, over tilt series acquisition and alignment to reconstruction, as shown in Figure 1a. For this we use a realistic model of rod-like filler nanoparticles in a polymer matrix. The ideal projections are calculated according to mass-thickness contrast at three different dose levels [5], i.e. $100\ e/Å^2$, $10^4\ e/Å^2$ and $10^6\ e/Å^2$. The maximum tilt range used is changing from ± 50° to ± 80° with 5° steps. Two linear tilt schemes (1° and 2° increments) and one Saxton tilt scheme (3° at 0° tilt increments) are compared. The influence of the microscope optics is approximated using a Gaussian point spread function with the FWHM given by from the first zero of the contrast transfer function. Shot noise is added to the final projections as determined from our CryoTitan electron microscope. Reconstruction was performed by weighted back projection implemented in Matlab. To evaluate the reconstruction quality, we focus on the edge intensity profile which is a key factor to distinguish different phases in the sample [6]. Therefore, we introduced a modified hyperbolic tangent function as edge profile model to determine contrast between two phases, the edge position, the mean density of background, and the steepness of the edge intensity profile (Figure 1b). It should be point out that the steepness is related to the resolution. Using this model, we fit the averaged edge profiles in X, Y (tilt axis), Z (optical axis) direction respectively, which were obtained by averaging all the edge profiles in the same direction.

Our results show that the maximum tilt range has the most significant influence on the quality of reconstruction among series acquired at same total electron dose level; however, the tilt schemes used here have a more limited influence on the reconstruction quality. Global edge intensity profiles show that the original intensities of the object are not reproducible despite normalizing intensities and the edge position is elongated in Z direction. As shown in Figure 2, the steepness is anisotropic at lower tilt range in X, Y (tilt axis) and Z direction, but it reaches roughly the same level using a maximum tilt angle of ±75°.

We have demonstrated how the reconstruction quality can be affects by different acquisition schemes at different electron dose levels. As expected, with high dose and a large tilt range, quantitative analysis is possible and reliable. However, it is still a big challenge to quantify reconstructed information at low dose.

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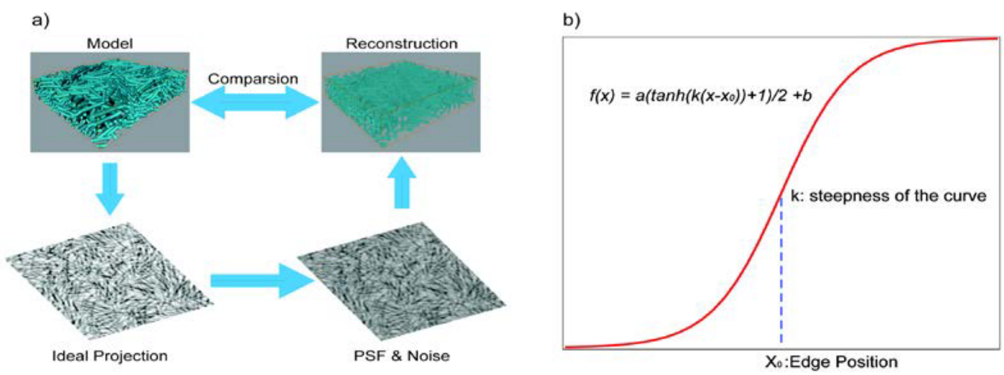

Figure 1. a) Schematic diagram of simulation approach about electron tomography; b) Hyperbolic tangent function is used to fit edge profiles: parameter a represents contrast, b represents background, k represents the steepness of the profile, and x_0 represents the edge position.

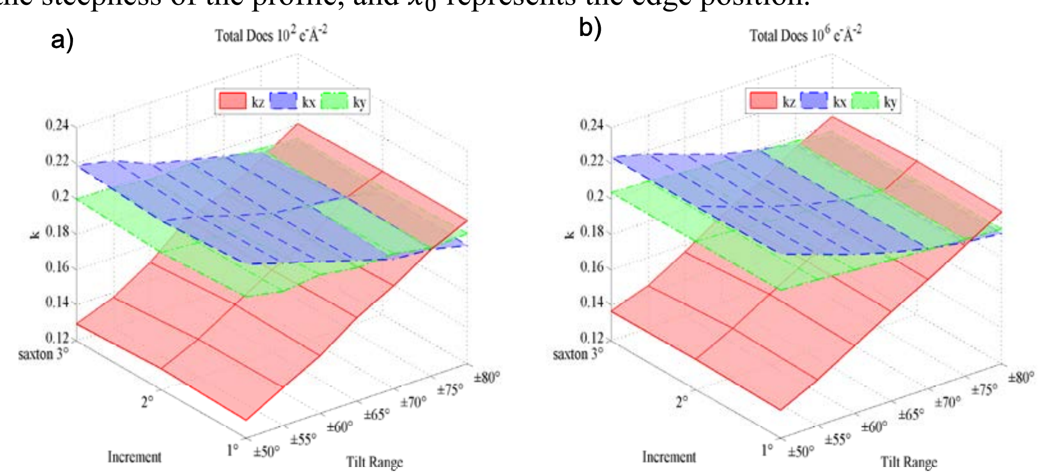

Figure 2. Quantitative comparisons of the steepness of global edge profiles in X (kx), Y (ky) and Z (kz) direction, which are reconstructed with different acquisition schemes, and at 100 $e/Å^2$ total dose (a) at and $10^6 e/Å^2$ total dose (b).

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