Quantitative scanning electron microscopy in the transmission mode: a development for nanoanalytics

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Scanning electron microscopes (SEM) offer except standard signals like secondary or backscattered electrons also signals in the transmission mode in case of thin electron-transparent specimens. Among them a very valuable signal is the annular dark-field (ADF) signal because it is related to the local mass thickness of the specimen in a rather straightforward manner [1]. Therefore, this signal is well suited for quantitative studies. Since important components like highly sensitive ADF detectors and continuous quantitative probe current monitoring are commercially as yet not available for SEM, quantitative studies require hardware extensions of commercial SEM as well as the development of software for data processing.

In this paper we present quantitative investigations made with polystyrene spheres at room temperature for which a high-resolution field-emission "in-lens" SEM (S-5000; Hitachi Ltd., Japan) equipped with a home-made ADF detector capable of single electron counting [2] was used. Two software packages were developed: (i) The program MONCA [3] is a Monte Carlo simulation of electron scattering for estimation of the exact relationship between the local mass thickness of the specimen and the related ADF signal. In case of polystyrene sample, the performance of MONCA was demonstrated up to ~800 nm thickness with a good agreement between theoretical and experimental data. (ii) The program MASDET [4] was developed specifically for mass determination of biological/organic samples in a highly automated manner. Moreover, it allows to calculate the mass and mass thickness profile (mapping) using the data provided from MONCA.

Polystyrene spheres with diameters from 70 nm up to 1 μm were attached laterally to a grid which allows for the advantageous imaging of individual spheres without any background. For the imaging, 30 keV primary electrons were used with a very low probe current of ~3 pA and a short pixel-time of 12 μs to keep the applied electron dose as low as possible. Fig. 1a shows the ADF image of a polystyrene sphere with a diameter of 140 nm where about the upper half of the sphere is free of any spurious background. The image data and the reference signal of the probe current monitored by the objective aperture are used to calculate the fraction of scattered electrons and are compared qualitatively (Fig. 1b) and quantitatively (Fig. 1c, d) with the Monte Carlo simulated data by MONCA [3]. They show a fairly good agreement in the whole thickness range of the sphere.

In a previous quantitative study [5] we investigated a wedge-shaped test specimen made of the embedding resin Epon. With this material we found a close agreement between experimental and simulated data up to a thickness of ~0.5 μm. Presently, studies with other organic and inorganic materials are in progress with the aim to pave the way for quantitative studies of various nanostructures.

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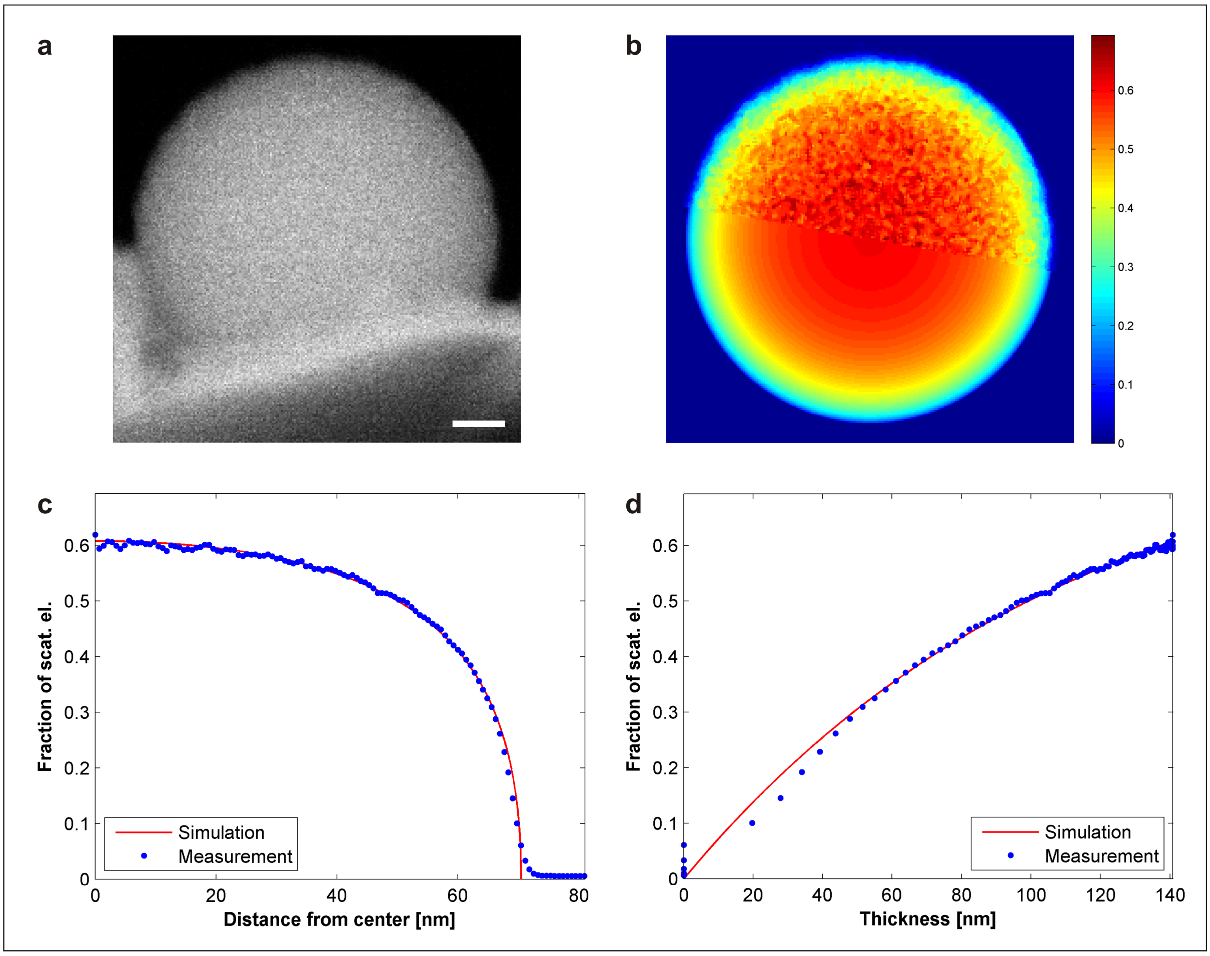

Figure 1. Experimental and Monte Carlo simulated data of a polystyrene sphere with a diameter of 140 nm. (a) ADF image of an individual sphere attached laterally to a TEM mesh grid. (b) The pseudo-colored image of the fraction of scattered electrons hitting the ADF detector; upper half: experimental data low-pass filtered by 3×3 pixels, lower half: Simulated data for the polystyrene sphere calculated by the program MONCA [3]. (c) Numerical data of the polystyrene sphere shown in (b) after rotational averaging. The diagram illustrates the dependence of the fraction of scattered electrons hitting the ADF detector vs. distance from the center of the sphere. (d) Diagram illustrating the dependence of the fraction of scattered electrons hitting the ADF detector vs. the projected local thickness of the sphere. The scattered electrons at distances higher than ~70 nm (in plot c) and at the thickness ~0 nm (in plot d) are due to minor deviations of the polystyrene sphere from a perfect spherical shape. The bar represents 20 nm.

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