# Size measurement of nanoparticles by means of transmission scanning electron microscopy (TSEM)

T. Klein, C. G. Frase and E. Buhr

Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, 38116 Braunschweig, Germany

Tobias.Klein@ptb.de
Keywords: nanoparticle, size measurement, TSEM, SEM, Monte Carlo simulation

Technical properties of nanoparticles as well as their potential risk for health and environment are related to their size. Accurate size measurement is thus a prerequisite for further development in this field and for evaluation of potential dangers.

Scanning electron microscopy (SEM) is a standard means to characterise nano-structures and nanoparticles. An SEM equipped with a transmission detector can be used for dimensional metrology [1] offering the possibility of complementary bright field and dark field imaging as well as an improved signal to noise ratio compared to the detection of backscattered electrons. In order to accomplish accurate size measurements the image formation process has to be taken into account. This can be done using Monte Carlo simulation of electron specimen interaction. Electrons passing through a specimen can be modeled by first-principles scattering theory in contrast to simulation of secondary electron generation which usually includes semi-empirical factors [2]. Scanning electron microscopy in transmission mode (TSEM) is thus a serious option for size measurement of nano-particles [3].

The feasibility of the TSEM approach is shown for three important material classes (ceramics, metals and polymers) by investigating silica spheres, gold nanoparticles and latex spheres. As an example, a bright field image of silica spheres (Merck Monospher) on holey carbon film is shown in figure 1. It was recorded by the transmission detector of a standard SEM (Zeiss Leo Supra 35VP) using an accelerating voltage of 30 kV. The detector is available from Zeiss as a supplementary add-on. It consists of a bright field and a dark field solid state detector which are usually used for detection of backscattered electrons.

In order to evaluate the measured signal profile shown in figure 2, Monte Carlo simulations of signal generation are done using the software package MCSEM (Monte Carlo simulation of scanning electron microscopy) [4,5]. One advantage of this program is its modular design simulating image formation in separate steps: Beam shape and energy of incident electrons are taken into account and geometry as well as elemental composition of specimen and an optional substrate can freely be chosen. Elastic scattering is simulated using classical Rutherford cross sections and tabulated Mott cross sections [6] for high energy and low energy electrons, respectively. Inelastic scattering is modeled by Bethe approximation [7]. Knowing the energy sensitivity of the transmission detector as well as the acceptance angles of bright field and dark field detection the contribution of every scattered electron to the signal is finally determined.

For a given experiment all experimental parameters apart from specimen properties are known within certain limits. In case of silica particles perfect spheres are presumed whose diameter is the only free parameter in simulation. It is chosen to obtain the best match between measured and simulated signal profile. As can be seen in figure 2 both profiles agree quite well leading to the conclusion of a particle diameter of 161 nm. Simulation of a silica

W. Grogger, F. Hofer, P. Pölt (Eds.): MC2009, Vol. 3: Materials Science,
DOI: 10.3217/978-3-85125-062-6-455 , © Verlag der TU Graz 2009

sphere inhabiting a slightly larger diameter of 163 nm leads to a signal profile that visibly deviates from the measured one (figure 3) demonstrating sensitivity in the nm region.

Of course, good sensitivity does not necessarily mean good accuracy but it is a promising starting point on the route to traceable measurements. To achieve this goal, a comprehensive calibration of the SEM and its electron scanner unit are necessary taking into account possible image distortions, differences of magnification in x- and y-direction and dependence of magnification on working distance, electron energy, etc. Furthermore, automatic image analysis is desirable to evaluate a relatively large number of particles in acceptable time. Finally, the measurement uncertainty has to be determined considering all relevant uncertainty contributions.

1. R. C. Farrow et al., J. Vac. Sci. Technol. B **15** (1997) p2167-72
2. J. R. Lowney, Scanning **17** (1995) p281-86
3. E. Buhr et al., Meas. Sci. Technol. (2009), accepted
4. D. Gnieser et al., Proc. 14th Europ. Microsc. Cong. **1** (2008) p549-50
5. D. Gnieser et al., Proc. 11th Int. Seminar (Skalský Dvur, CZ, 2008) p31-32
6. F. Salvat et al., Comp. Phys. Comm. **74** (1993) p358-74
7. D. C. Joy et al., Scanning **11** (1989) p176-80
8. N. Senftleben (TU Dresden) and D. Gnieser (TU Braunschweig) are greatfully acknowledged for their support in measurements and data modeling. This research was supported by the European Commission through the grant EMRP T3.J1.1.

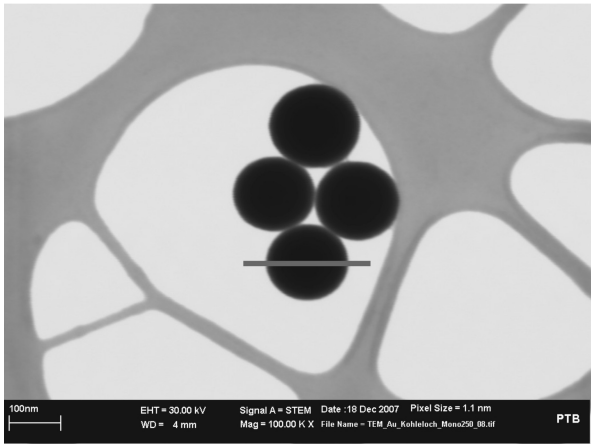


**Figure 1.** Bright field image of silica spheres on holey carbon film. The horizontal line denotes the position where the signal profile shown in figure 2 has been taken.

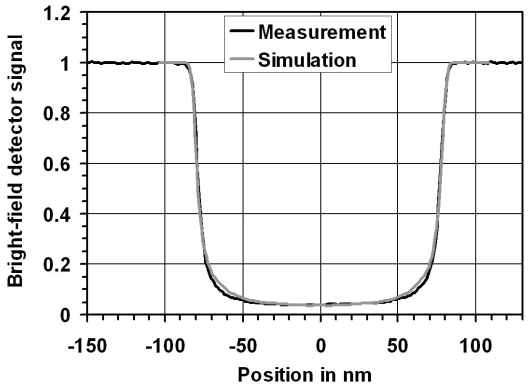


**Figure 2.** The measured signal profile of the silica sphere shown in figure 1 agrees quite well with the simulated profile assuming a diameter of 161 nm.

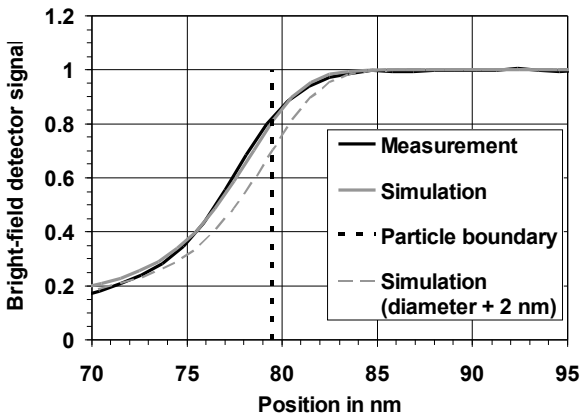


**Figure 3.** Sensitivity to changes of particle size in the nm region can be demonstrated by comparison of best match signal profile to simulated profile of a slightly larger sphere.