

# Transmission Scanning Electron Microscopy (TSEM): A New Means for Characterization of Nanoparticles



T. Klein, C. G. Frase, K. P. Johnsen and E. Buhr

## 1. Abstract

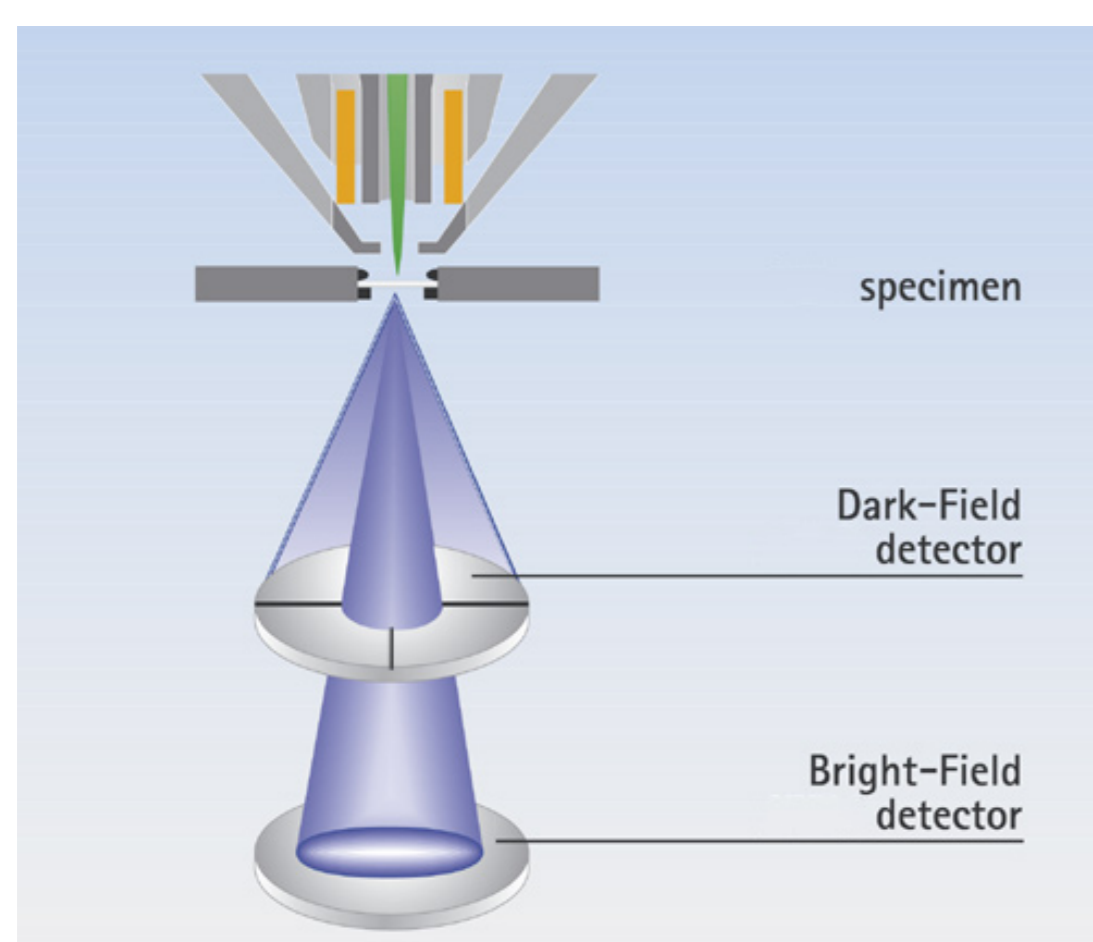
- Accurate and fast size measurement of nanoparticles using a scanning electron microscope equipped with a transmission detector
- Threshold criteria from simulations adapted to particle material and size
- Automated image acquisition and analysis
- Size results traceable to national standards including uncertainty analysis

## 2. Transmission scanning electron microscopy

- Commercially available transmission detector in a Zeiss Supra 35 VP
- Nanoparticles prepared on carbon film (TEM grid)
- Good image contrast at an acceleration voltage of 30 kV
- Automated image acquisition by batch processing

Calibration of SEM pixel size

- 2D-grating with 144 nm pitch (150-2D from ASM Inc.)
- Grating pitch traced back to the SI unit “meter” by laser diffraction

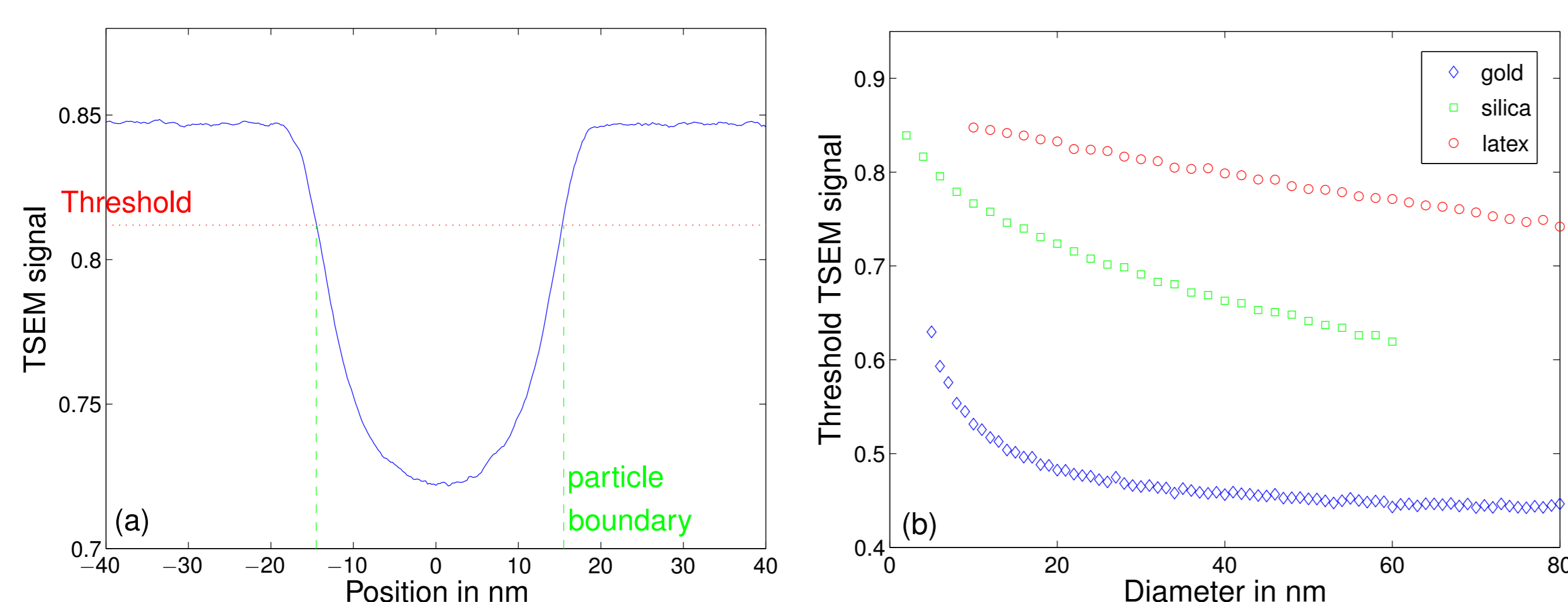


**Figure 1:** Schematic drawing of transmission detector consisting of separate dark-field and bright-field semiconductor detectors, by courtesy of Zeiss.

## 3. Determination of particle boundary

TSEM signal at the particle boundary depends on material and size of the particle due to different electron transmission and scattering properties [1].

- Different threshold grey values at particle boundary for image analysis
- Global thresholding algorithms lead to systematic deviations of measured size
- Individually adapted threshold for every particle necessary for accurate size measurement



**Figure 2:** Simulated TSEM signal of a 30 nm latex sphere (a). Threshold levels as a function of particle size for three different materials (b).

## 4. Simulation

Monte Carlo simulation MCSEM [2] taking into account

- Electron beam: electron energy and beam profile
- Particles: 3D-shape and elemental composition
- Scattering process: Rutherford and Mott cross sections (elastic) and Bethe approximation (inelastic)
- Detector: geometry and energy sensitivity

## 5. Experimental results

Studied particles:

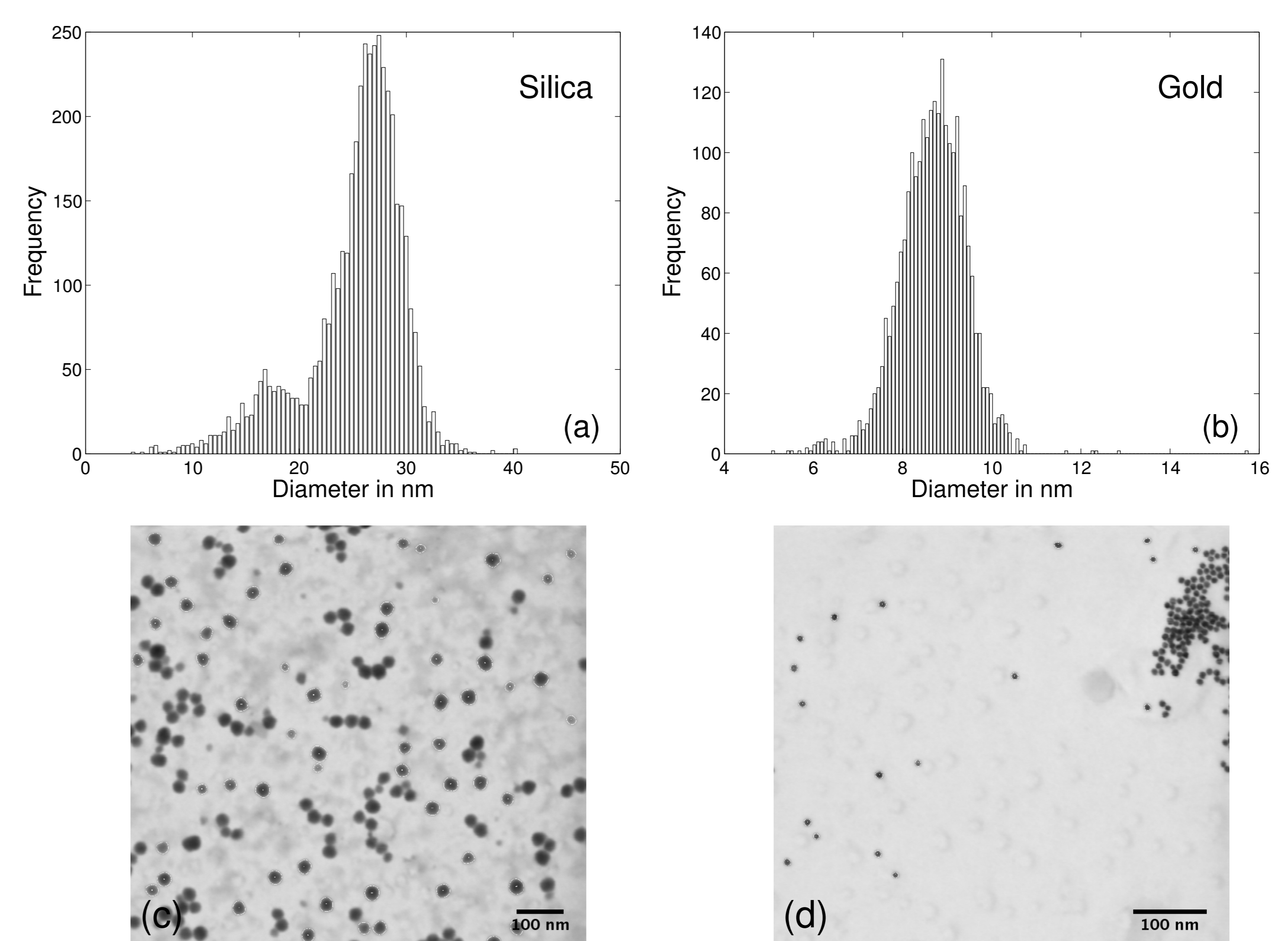
- Silica (IRMM 304) and gold particles (NIST RM 8011)

Parameters:

- 30 kV acceleration voltage, 3 mm working distance, bright-field detection

Results (mean diameter  $\pm$  expanded uncertainty with  $k=2$ ):

- Silica particles: 25.2 nm  $\pm$  1.1 nm
- Gold particles: 8.7 nm  $\pm$  0.9 nm



**Figure 3:** Diagrams (a) and (b) display the distribution of 4400 silica and 2500 gold particles, respectively. Analyzed particles are marked by overlaid outlines in the TSEM images of silica (c) and gold particles (d).

## 6. Conclusion

- TSEM approach feasible for particles down to about 10 nm
- Accurate analysis relies on the simulation of image formation process
- Fully automated measurement and analysis of a particle sample in the course of one day with good statistics
- Traceability to SI unit “meter” achievable

## Literature

- [1] E. Buhr et al., Meas. Sci. Technol. **20**, 2009
- [2] K. P. Johnsen et al., Proc. SPIE **7683**, 2010