A local threshold method for measurement of nanoparticle sizes in the SEM

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Progress in nanotechnology has raised interest in nanoparticle (NP) dimensional reference materials and measurements.[1-3] In the scanning electron microscope (SEM), measurement of NP size requires the assignment of boundaries based on measured intensity associated with secondary electron yield, as in Figs. 1-3. The intensity transition at the boundary is not abrupt. In Fig. 3, for example, the 10 % to 90 % intensity thresholds are separated by about 16% of the spherical NP's 30 nm radius. For smaller NPs, the uncertainty is a larger fraction of particle size.

For complex lithographic features like transistor gates, similar uncertainty can be reduced by applying electron-sample interaction models,[4,5] e.g., by finding a topography for which the modeled and measured intensity profiles match. A transistor gate is ordinarily widest (and may exhibit a rounded "footing") at the bottom. Its top width is characterized by a continuous change of slope from sidewall to top. A simpler approach may suffice for NPs. For them, there is a discontinuous change in slope, with the substrate perpendicular to the beam and the NP tangent to it (a configuration associated with high signal) at the point of first encounter. With a sharp beam, this discontinuity makes approximately a step function, one intensity on the substrate and another on the NP (Fig. 2). This picture is modified by lesser sources of contrast, such as the gradually changing slope and loss of edge bloom as the beam moves deeper inside the edge. A wider beam broadens the step into an s-shaped transition with the intensity at the edge midway between the substrate and NP values (Fig. 3). This motivates a hypothesis: a 50 % threshold edge criterion will have small errors relative to the more complete model.

In this paper we apply the hypothesized threshold criterion to intensity profiles simulated for spherical NPs by a Monte Carlo dielectric function theory model implemented in JMONSEL.[4] Simulations varied NP material and size, substrate material and thickness, beam energy and width. The local 50 % assignment's mean error and its standard deviation for combinations of these parameters were both in the low tenths of nanometers provided the beam energy was in the range 500 eV to 30 keV and beam standard deviation less than or equal to 1 nm. Linear regression indicates that errors are more sensitive to beam energy and secondary electron escape depth (a proxy for NP material) than to the other variables.

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2. A. Nicolet et al., Meas. Sci. Technol. **27** (2016) 035701.
3. V. Kestens et al., J. Nanopart. Res **18** (2016) 171.
4. J. Villarrubia et al., Ultramicrosc. **154** (2015) 15.
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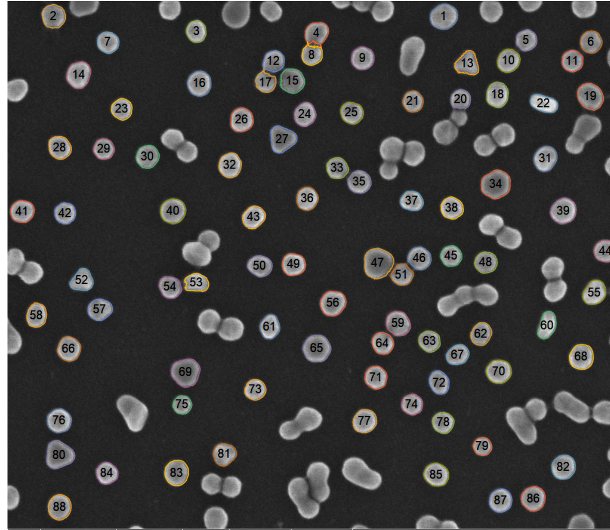

Fig. 1: SEM image (15 keV, 43 pA, horizontal field of view 847 nm, 4 mm working distance) of Au NPs on a Si surface showing local 50 % threshold edge assignments to particles found by a pattern recognition algorithm. For the same NPs, diameters assigned by the maximum entropy method averaged approximately 3 nm larger.

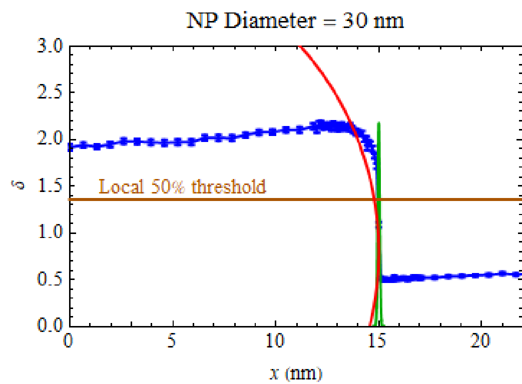

Fig. 2: Simulated intensity profile (blue) of a 30 nm diameter Au NP (red) on a Si substrate at 1 keV with negligible beam size (indicated by the green Gaussian curve). Error bars indicate the 95 % confidence interval of the Monte Carlo simulation repeatability. Departure of the intensity from an ideal step results in 0.04 nm edge assignment error.

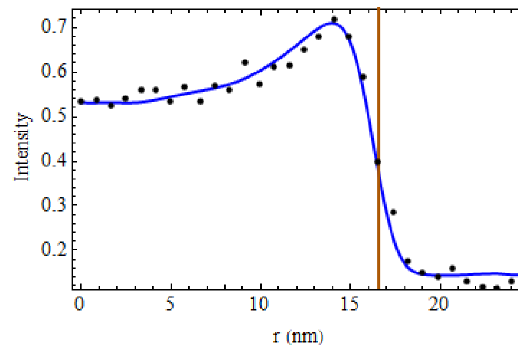

Fig. 3: Comparison of measured (points) and modeled (blue curve) intensity profiles from particle center to beyond the edge of Fig. 1's NP #77. The modeled beam standard deviation was 1 nm. At this beam energy and size, the error of the 50 % threshold relative to the dielectric function theory edge is –0.2 nm, small enough that the total error is likely dominated by unrelated larger components.