Measurement of Silica Particles by Transmission Electron Microscope

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Introduction

As advanced inspection systems apply UV and DUV illumination to resolve smaller structures, there is an industry need for new particle standards resistant to damage at these shorter wavelengths. We intend to provide such a traceable size standard based on silica nanoparticles.

We present the traceable measurement of 14 silica nanoparticle solutions, ranging from nominal diameters of 32 nm to 1500 nm, using established methods in transmission electron microscopy (TEM), NIST standard reference materials (SRMs), and semi-automated digital image processing.

Measurement Method

The "internal standard method" overcomes the problem of variable magnification between TEM images [1,2,3]. The method calibrates each image's magnification using standard reference materials (SRMs) [4,5] imaged within the same field of view as the particles under measurement.

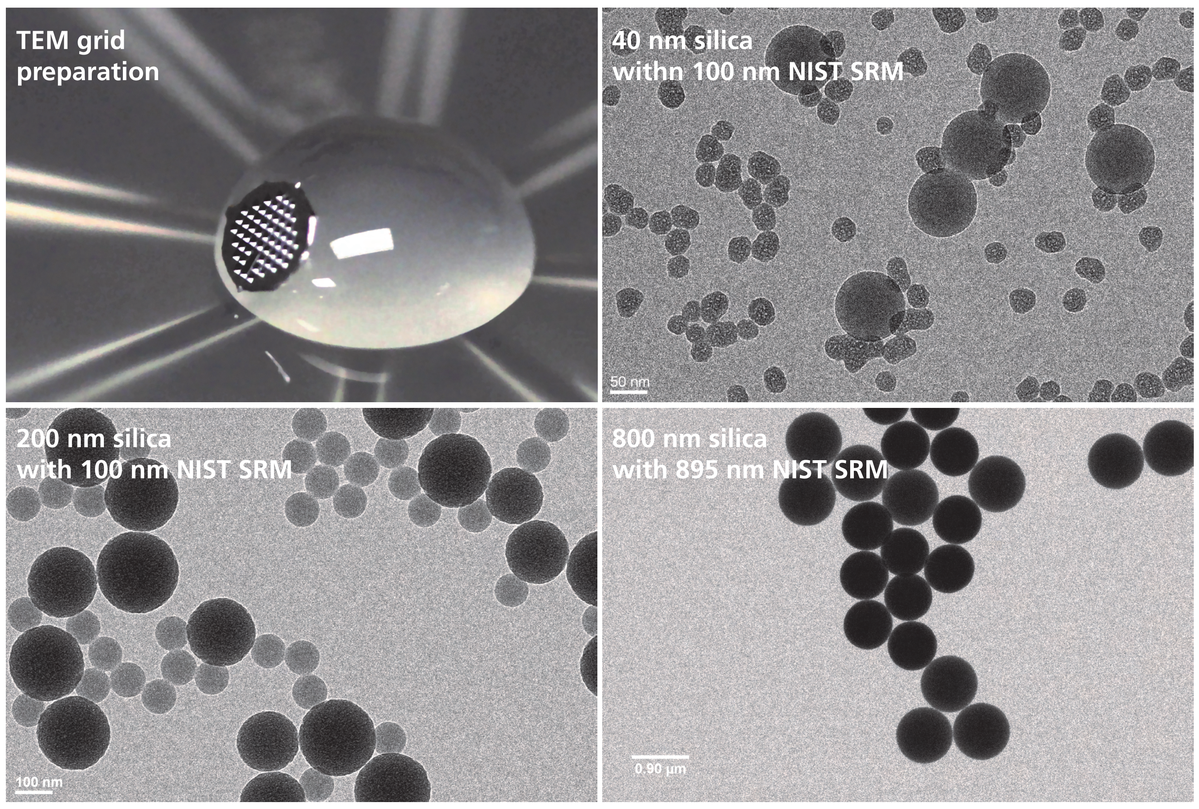
The Fiji [6] image analysis platform isolates and measures particles in images made on an FEI Technai TF-20 FEG/TEM. From these measurements, the method calculates a magnification correction for each image by dividing the mean reference particle diameter in each image by the SRM's published mean diameter [4,5].

$$M_i = \frac{1}{D_r}\sum_k \frac{d_{r,k}}{N_r} = \frac{\bar{d}_{r,i}}{D_r}$$

The average value for each silica size's calibrated diameter can then be expressed as:

$$\bar{D}_m = \frac{1}{N_m}\sum_i \sum_j \frac{d_m(i,j)}{M_i} = \sum_i f_{m,i}\frac{\bar{d}_{m,i}}{\bar{d}_{r,i}} D_r$$

where N_m is the total number of silica particles measured, $f_{m,i}$ is the fraction of measured particles in the ith image, and $\bar{d}_{m,i}$ is the average measured diameter for silica particles in the ith image.



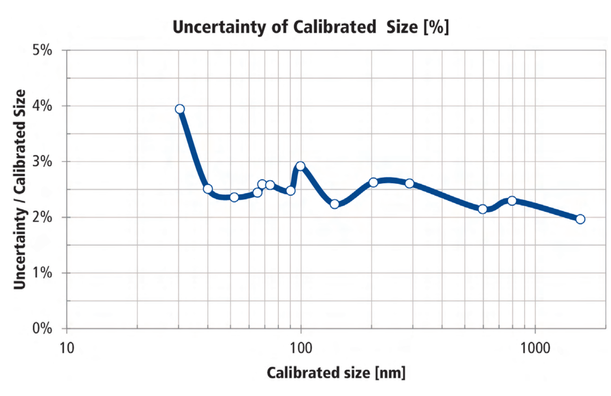
Results

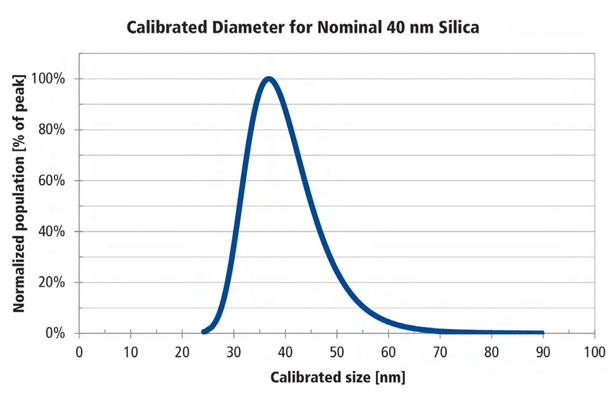
TABLE 1. Uncertainty contributions and sizing results by the nominal silica sizes 32 nm to 90 nm.

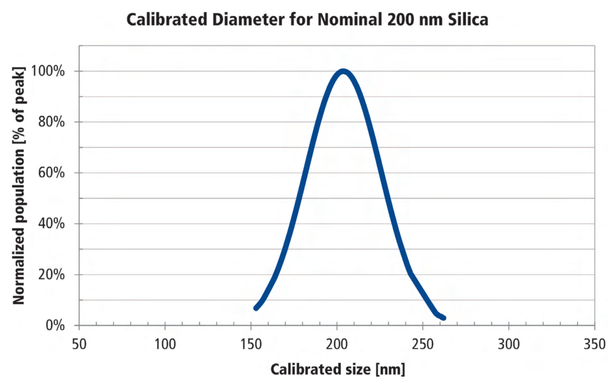
Parameter	Type	Term	32	40	50	60	70	80	90
Pixel size [nm]			0.39	0.39	0.39	0.58	0.58	0.58	0.58
Particle count, silica		N_m	2699	2676	1702	1042	798	962	512
Particle count, SRM		N_r	46	91	92	95	95	84	79
Finite sample, silica [nm]	A	$u_A(\bar{d}_m)$	0.11	0.14	0.19	0.31	0.42	0.48	0.53
Pixilation, silica [nm]	B	$u_{pixel}(\bar{d}_m)$	0.11	0.11	0.11	0.17	0.17	0.17	0.17
Edge finding, silica [nm]	B	$u_{edge}(\bar{d}_m)$	0.01	0.01	0.02	0.03	0.03	0.03	0.04
Shrink rate, silica [nm]	B	$u_{shrink}(\bar{d}_m)$	0.04	0.05	0.06	0.08	0.08	0.09	0.11
Finite sample, SRM [nm]	A	$u_A(\bar{d}_r)$	0.44	0.13	0.12	0.15	0.22	0.19	0.25
Pixilation, SRM [nm]	B	$u_{pixel}(\bar{d}_r)$	0.11	0.11	0.11	0.17	0.17	0.17	0.17
Edge finding, SRM [nm]	B	$u_{edge}(\bar{d}_r)$	0.13	0.12	0.12	0.14	0.14	0.14	0.14
Shrink rate, SRM [nm]	B	$u_{shrink}(\bar{d}_r)$	0.13	0.12	0.12	0.12	0.12	0.13	0.12
NIST SRM [nm]	B	$u(D_r)$	0.30	0.40	0.52	0.65	0.68	0.73	0.90
Mean maximum diameter (nm)			30.4	40.0	51.9	65.1	68.3	73.8	90.2
Expanded uncertainty, k=2 (nm)			1.2	1.0	1.2	1.6	1.8	1.9	2.2

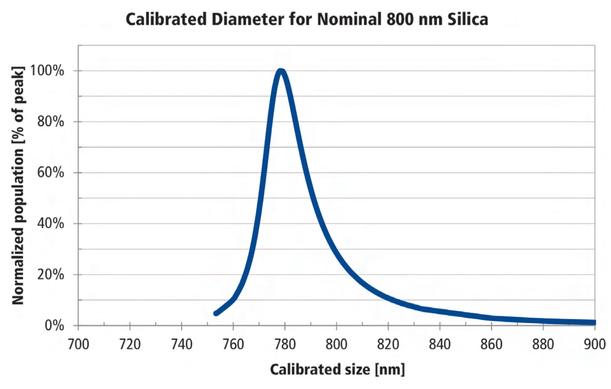
TABLE 2. Uncertainty contributions and sizing results by the nominal silica sizes 100 nm to 1500 nm.

Parameter	Type	Term	100	150	200	300	600	800	1500
Pixel size [nm]			0.58	1.14	0.77	1.14	4.50	4.50	4.50
Particle count, silica		N_m	450	1374	170	126	487	195	141
Particle count, SRM [nm]		N_r	103	248	420	733	101	119	331
Finite sample, silica [nm]	A	$u_A(\bar{d}_m)$	0.98	0.44	1.68	2.36	1.93	3.16	4.29
Pixilation, silica [nm]	B	$u_{pixel}(\bar{d}_m)$	0.17	0.33	0.22	0.33	1.30	1.30	1.30
Edge finding, silica [nm]	B	$u_{edge}(\bar{d}_m)$	0.05	0.03	0.05	0.06	0.72	0.96	1.86
Shrink rate, silica [nm]	B	$u_{shrink}(\bar{d}_m)$	0.12	0.17	0.25	0.36	0.79	1.05	2.04
Finite sample, SRM [nm]	A	$u_A(\bar{d}_r)$	0.22	0.20	0.23	0.24	0.75	3.71	2.74
Pixilation, SRM [nm]	B	$u_{pixel}(\bar{d}_r)$	0.17	0.33	0.22	0.33	1.30	1.30	1.30
Edge finding, SRM [nm]	B	$u_{edge}(\bar{d}_r)$	0.14	0.10	0.08	0.10	1.51	1.51	1.49
Shrink rate, SRM [nm]	B	$u_{shrink}(\bar{d}_r)$	0.12	0.12	0.12	0.12	1.18	1.18	1.17
NIST SRM [nm]	B	$u(D_r)$	0.99	1.38	2.02	2.88	5.33	7.11	13.9
Mean maximum diameter (nm)			99.4	139	204	290	596	796	1557
Expanded uncertainty, k=2 (nm)			2.9	3.1	2.7	7.6	13	18	31









Uncertainties

Both particle types share similar type A uncertainties.

$$u_A(\bar{D}_m)^2 = \sum_i \left[\left(f_{m,i}\frac{\bar{d}_{m,i}}{\bar{d}_{r,i}}D_r\right)^2\left(\frac{u_A(\bar{d}_{m,i})^2}{\bar{d}_{m,i}^{\,2}} + \frac{u_A(\bar{d}_{r,i})^2}{\bar{d}_{r,i}^{\,2}}\right)\right]$$

Each particle type also shares the same three type B uncertainties.

$u_{pixel}(\bar{d})$	Pixelation effects: We quantify this at half of the image pixel size with a rectangular distribution [7].
$u_{edge}(\bar{d})$	Edge finding: Studies at each TEM pixel size estimate the effect of various grayscale thresholds to isolate particles for measurement.
$u_{shrink}(\bar{d})$	Shrink rate differences: A timed study of relative size changes in particle types due to electron beam exposure finds no significant difference. The standard error on the difference, 0.133%, serves as the uncertainty.

The final B term is the SRM's contribution to the uncertainty, $u(D_r)$.

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