

National Bureau of Standards Certificate Standard Reference Material 484 Scanning Electron Microscope Magnification Standard (A Stage Micrometer Scale)

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This Standard Reference Material is intended for use in calibrating the scanning electron microscope (SEM) magnification scale to an accuracy of 5% or better within the range of 1,000 to 20,000X. Each SRM bears an identification number and has been individually measured.

The certified distance between the centers of specific lines opposite the Knoop indentation (see the sketch) and the identification number are given in the table below. The enclosed SEM photograph shows the area used in the measurement of this specific SRM. This certification is valid within an area 24 μm wide centered about a line extending from the Knoop indentation.

Identification Number:

| Line Pair | Nominal Distance μm | Certified Distance μm | Uncertainty μm |
|-----------|-----------------------------------|-------------------------------------|------------------------------|
| 0 → 1 | 1 | | .039 |
| 0 → 2 | 2 | | .039 |
| 2 → 3 | 3 | | .039 |
| 3 → 4 | 5 | | .039 |
| 0 → 6 | 50 | | .71 |

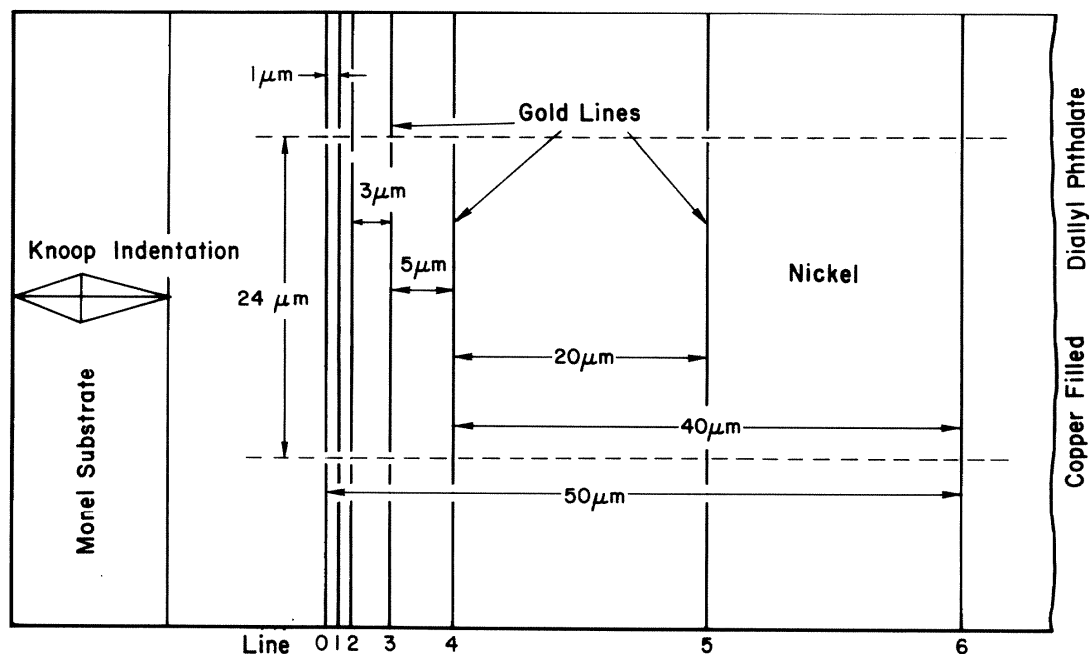
The distances between the lines were determined from measurements made on photographs taken with an SEM in which each SRM was compared by substitution with a Master Standard. The Master Standard had previously been calibrated by the NBS Dimensional Technology Section using a polarizing helium-neon laser interferometer. The uncertainty of this calibration, based on 81 independent measurements, is 0.004 μm for distances from 1 to 5 μm and 0.009 μm for distances from 10 to 50 μm .

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The operating conditions of the SEM were monitored and a resolution of $0.050\ \mu\text{m}$ was maintained using an SEM Resolution Test Specimen. (This test specimen, RM 100, is also available from the Office of Standard Reference Materials.) Each reported interval has been corrected for photographic recording system distortion and SEM magnification drift using the Master Standard. The uncertainty values are based on a random selection of samples which were measured 2 to 3 times at intervals that were as long as 3 months.

The polished surface of each SRM has been carefully ground and polished using metallographic techniques. The carbonaceous contamination (a product of SEM electron beam bombardment) can be removed by hand polishing on micro cloth using metallographic grade MgO powder as an abrasive. An alternate cleaning method is to D.C. sputter etch for less than 30 seconds in an argon atmosphere. This cleaning process does not alter the certified spacing of the lines by more than $0.010\ \mu\text{m}$.

A recommended procedure for calibrating the magnification of the SEM using SRM 484 is given on the following page. It is suggested that the user extend the calibration to adjacent areas outside of the certified area on the standard for routine use as a "Working Standard."

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Recommended Procedure for Calibration of SEM
Magnification Using SRM 484^a

1. Rigidly mount SRM484 on SEM stub with electrically conductive cement or clamp onto stage of SEM. (The SRM, in particular the polished surface, must be kept clean. Prior to use the surface should be inspected and cleaned if necessary.)
2. Surface of SRM 484 should be normal to electron beam.
3. To minimize the distortions produced by the recording system the specific lines on SRM 484 to be used in the calibration should be chosen so that the distance between them corresponds as closely as possible to the length of the object to be measured with both images positioned in the same area on the cathode ray tube (CRT). A millimeter scale taped onto the edges of the CRT on the x and y directions will assist in the re-location on the respective images.
4. Allow a 30 minute or more warm-up of electronic circuits to achieve operational stability.
5. A vacuum of 10^{-4} mm of Hg or better is necessary to keep the contamination rate on the surface of the SRM as low as possible.
6. SEM resolution should be a minimum of 0.05 μm .
(Good edge resolution is not a necessary requirement.)
7. Adjust final lens current at a resettable value. Cycle lens circuit OFF-ON 3 times to minimize hysteresis effects.
8. Adjust electron gun voltage (between 5 to 30 kV), saturate filament, and check filament alignment.
9. Adjust lens apertures and stigmator for optimum operation (minimum astigmatism).
10. The working distance can be reproducibly obtained by focusing on the gold lines with Z axis control at highest possible magnification to minimize depth of focus. An alternate method is to use "Y" mode and adjust for maximum signal.
11. Add "Black Level" for contrast if necessary, S/N ratio should be 2:1 minimum.
12. Take photograph under best conditions. Allow Polaroid prints to dry 15 to 20 minutes or more to minimize distortion from paper, emulsion and coating shrinkage.
13. Measure the perpendicular distance between each pair of lines (using center of each line image) on the photograph with a TEM Diffraction Plate Reader or use an equivalent instrument the precision of which is suitable for this purpose (about 0.1 mm).
14. Repeat measurements 3 times on each photograph to determine the average spacing.
15. Magnification =
$$\frac{\text{Distance between lines as measured on photograph}}{\text{Certified distance between same lines}}$$
16. To determine SEM stability and reproducibility, repeat all steps at hourly or daily intervals or after adjustments and repairs.

*A Procedure for Calibrating the Magnification Scale of An SEM Using SRM 484. (To be published as an NBS Technical Note.)

Some Parameters that May Effect the Magnification of An SEM

1. Photographic material - shrinkage/expansion, photographic enlarging.
2. Recording camera lens- distortions, change of CRT image size.
3. Recording CRT-distortion of faceplate, beam deflection.
4. Scan generator - nonlinear, aging components.
5. Scan coils - nonorthogonal.
6. Magnification switch resistor chain - different for each magnification.
7. Zoom control on magnification - resetability.
8. Working distance - long (small angle beam deflection) vs. short (large angle beam deflection) for same magnification.
9. Specimen tilt - not perpendicular to beam.
10. Specimen tilt correction - relative to tilt axis.
11. Specimen image drift - charging or stage drift.
12. Depth of focus.
13. Reproducibility of specimen height control.
14. Lens strength combinations - particularly for 3 lens instruments.
15. Lens astigmatism and hysteresis.
16. Electron gun stability.
17. Extraneous magnetic and electrostatic fields.