National Institute of Standards & Technology

Certificate

Standard Reference Material 484e

Scanning Electron Microscope Magnification Standard

(A Stage Micrometer Scale)

This Standard Reference Material (SRM) is intended for use in calibrating the magnification scale of a scanning electron microscope (SEM) within the range of 1000× to 20,000×. The SRM is individually certified and bears an identifying serial number. The SRM consists of thin gold layers separated by layers of nickel of nominal thicknesses of 1, 2, 5, 10, 30, and 50 µm mounted such that the layers are viewed in cross-section so the gold layers appear as thin gold lines in a nickel substrate. The SRM is mounted in copper-filled epoxy within a cylinder of 304 stainless steel 11 mm × 0.65 mm high.

The certified region of each SRM is located relative to a Knoop indentation. Distances between the centers of the gold lines are certified, and an SEM photomicrograph showing the certified region (see the figure on page 2) accompanies each SRM. The certification is valid within 15 µm to either side of an imaginary line extending from the Knoop indentation mark normal to the gold lines.

The distances between gold lines were measured by an SEM that uses a scanning specimen stage whose displacement is determined using a helium-neon interferometer measurement system. The SRM is scanned through the fixed electron beam. A minicomputer system simultaneously records the stage position and peak height of the back-scattered signal. The certified value for each distance is an average of nine scans; three scans at each of three locations within the certified region.

Six distances are certified on each SRM, and their values are attached. The total uncertainty, U, for each certified distance includes allowances for two random components: long-term measurement variability and lack of parallelism between the gold lines defining the certified distances. Comparison with line-scale interferometry shows that systematic error is negligible.

The total uncertainties are equal to statistical tolerance limits. The interpretation of the tolerance limit is that at least 95% of the distances (within the certified region) will be within the interval defined by the certified value ± U at the 95 percent confidence level.

Table of Uncertainties of Spacing on SRM 484e

Nominal Spacing	Total Uncertainty, U
1 µm	± 0.058 µm
2	0.056
5	0.061
10	0.079
30	0.102
50	0.251

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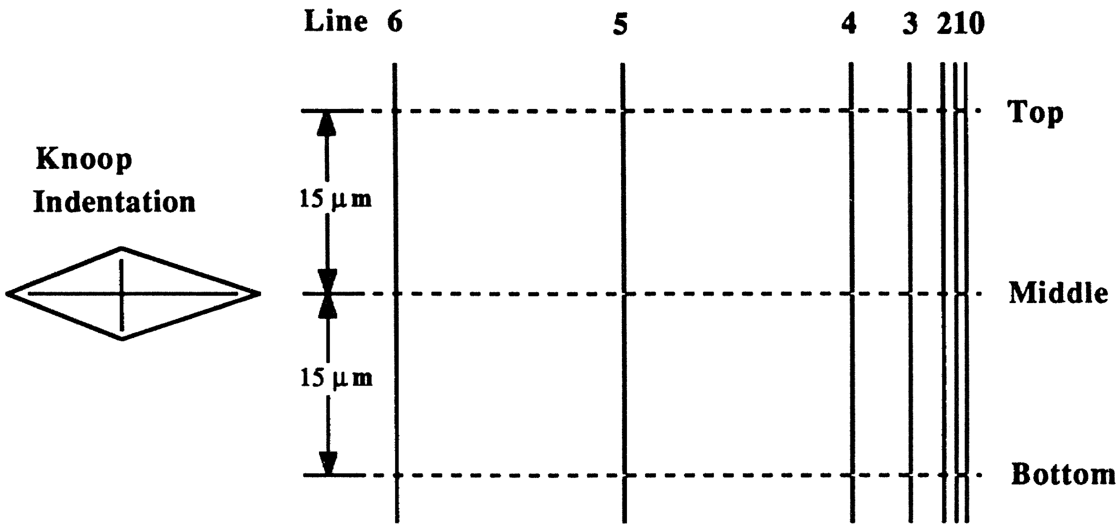

Figure 1. Diagram of Certified Region of SRM

In addition to the certified distance between line pairs, a table of values of the distance between line pairs at the top, middle and bottom of the certified region is also supplied. These values are not certified, but are supplied as information to the user of this SRM. Each value is the average of the three scans made at that position for certification of the SRM. The standard deviations of the averages of three scans (column four in table of information values) reflect the long-term variability of the certification measurement process.

The SRM user should refer to NIST Special Publication 260 (in preparation) for details regarding the fabrication, certification, and statistical analyses related to SRM 484e.

The technical direction and physical measurements leading to certification were provided by Joseph Fu of the Precision Engineering Division, with guidance on statistical analysis provided by M.C. Croarkin of the Statistical Engineering Division.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie.

The surface of each SRM has been carefully ground and polished using metallographic techniques. Cleaning should not be attempted unless signal strength is inadequate. Cleaning techniques that remove surface material sufficient to obliterate the Knoop indentation will void the certified distance values.

A recommended procedure for calibrating the magnification of the SEM using SRM 484e is given on the following page and in ASTM E766, "Practice for Calibrating the Magnification of SEM Using SRM 484". It is suggested that the user extend the calibration to adjacent areas outside of the certified area on the SRM for routine use as a "Working Standard." A list of parameters that may affect the resultant magnification of an SEM is given on page 4.

The operational steps indicated by the manufacturers of scanning electron microscopes to calibrate the magnification scale are different and often do not consider all the instrument parameters that may change the resultant magnification (see next page). The following procedure details the use of NIST SRM 484e to calibrate one particular SEM, but may be used as a guide for calibration of other SEM's.

Outline of Procedure for Calibrating SEM Magnification Scale

1. After the surface of the SRM 484e has been inspected for cleanliness, rigidly mount it on an SEM stub with electrically conductive cement or clamp it onto the SEM stage.

2. The surface of the SRM 484e should be normal to the electron beam.

3 A clean vacuum of 10^{-2} Pa (10^{-4} Torr), or better, is necessary to keep the contamination rate as low as possible.

4. Allow a 30-minute, or more, warm-up of electronic circuits to achieve operational stability.

5. Adjust electron gun voltage (between 5 to 50 kV), saturate filament, and check filament alignment.

6. Adjust all lens currents at a resettable value. Cycle lens circuit OFF-ON 3 times to minimize hysteresis effects.

7. Adjust lens apertures and stigmator for optimum resolution (minimum astigmatism).

8. SEM resolution should be minimum of 0.05 µm (500 Å), or better.

9. Position the SRM, at a nominal magnification of 1000x, so that the image of the Knoop indentation is centered at one edge of the viewing cathode ray tube (CRT). The width of the gold line calibrated region extends 15 µm above and below this indentation.

10. The same working distance or magnification scale of the SEM can be reproducibly obtained by focusing on the image of the gold lines with Z axis control, at the highest possible magnification, to minimize depth of focus. An alternate focus method is to use single line wave form ("y" mode) and adjust Z axis of maximum signal height.

11. To minimize the effect of linear distortions produced by the recording system, the procedure is as follows: The SRM is substituted for the unknown sample and photographed. The lines on the SRM to be used in the calibration should be chosen so that the distance between them matches the length of the object to be measured with both images positioned in the same area on the CRT. A millimeter scale taped onto the edges of the CRT in the "x" and "y" directions will assist in the relocation of the respective images.

12. Add contrast, if necessary, S/N ratio should be 2:1 minimum.

13. Measure the perpendicular distance between the lines using the CENTER of each line image. The measurement may be made by automated image analyses of the CRT image or measurement of the spacing recorded on a photographic recording of the CRT image. If photographic recording is used, the prints (if using Polaroid), should be dried 15 to 20 minutes or more to minimize effects due to emulsion and coating shrinkage. The photographs may be measured with a TEM Diffraction Plate Reader, or an equivalent instrument, the precision of which (about 0.2 mm) is suitable for this purpose.

14. Repeat measurements 3 times on each photograph to determine the average spacing.

15. $$\text{Magnification} = \frac{\text{Distance measured between image lines on photograph}}{\text{Certified distance between same lines}}$$

16. To determine the SEM stability and reproducibility, repeat all steps at hourly or daily intervals, or after adjustments and repair.

PARAMETERS THAT INFLUENCE THE RESULTANT MAGNIFICATION OF AN SEM

The parameters listed below may interact with each other. They are considered, in order of their location in the instrument, from electron source to the recorded photograph or analysis of the image.

1. Electron gun high-voltage instability can change the wavelength of the electrons and thus the final focus.
2. Different condenser-lens strength combinations change the focal point of the final lens.
3. Uncorrected final lens astigmatism can give a false indication of exact focus.
4. Residual magnetic hysteresis, particularly in the final lens, can change the focal conditions for a given indicated lens excitation.
5. Long depth of focus, particularly at low magnification and small beam divergence controlled by lens and aperture selection, can lead to incorrect focus.
6. Nonorthogonal deflection (x-y axis) can be produced by scan coils.
7. Scan generator circuits may be nonlinear and/or change with aging of circuit components.
8. Zoom control of magnification can be nonlinear.
9. Nonlinearity of scan rotation accessory can distort magnification at different degrees of rotation.
10. Distortion of the electron beam sweep may occur from extraneous magnetic and electrostatic fields.
11. The percent error in magnification may be different for each magnification range.
12. A tilted sample surface (not perpendicular to the beam axis) will introduce foreshortening.
13. The tilt correction applied may not be relative to the tilt axis of the sample or of a particular area on the sample surface.
14. Signal processing, particularly differentiation or homomorphic processing, can give a false impression of focus. DC suppression (sometimes called differential amplification, black level/gain, dark level or contrast expansion) may be used because of the isotropic effect on the image.
15. The objective lens on some instruments may be electrically coupled to the magnification meter; thus, focus and magnification are operator dependent.
16. For the same apparent magnification, two different combinations of working distance and beam scan-raster will produce different linear magnification.
17. Thermal and electronic drift of circuit components related to the above parameters can affect magnification with time in a random manner.
18. Distortion of faceplate and nonorthogonal beam deflection of the CRT can produce nonlinear magnification.
19. Camera lens distortion and change of photo image-to-CRT ratio can lead to magnification errors.
20. Expansion or contraction of photographic material, photographic enlarging, and control of contrast, can all have a significant effect on final apparent image magnification.