National Institute of Standards & Technology

Certificate

Standard Reference Material 484f

Scanning Electron Microscope Magnification Standard
(A Stage Micrometer Scale)

Serial No.

This Standard Reference Material (SRM) is intended primarily for use in calibrating the magnification scale of a scanning electron microscope (SEM) within the range of 1000x to 20,000x. 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 0.5, 0.5, 1, 3, and 5 µm such that when viewed in cross-section, 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 x 0.65 mm high.

The certified region of each SRM is located relative to a Knoop indentation. Spacings between the centers of the gold lines are certified, and SEM photomicrographs diagramed in Figure 1, showing the certified region, are provided with 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 certified spacing values and uncertainties for this serialized unit are given in Table 1 below. The uncertainty for each certified spacing includes allowances for two random components; long-term measurement variability as estimated from forty-five repeated determinations on a check standard, and instrument imprecision. Comparison with line-scale interferometry shows that systematic error is negligible. The uncertainties shown in the table, are calculated from the three standard deviation limits to random error.

Table 1.
Certified Spacing Values and Uncertainties

Line Pair	Nominal Spacing, µm	Spacing Values, µm	Uncertainty, µm
0→1	0.5		± 0.021
1→2	0.5		± 0.020
2→3	1		± 0.026
3→4	3		± 0.035
4→5	5		± 0.052
(0→5)	(10)		(± 0.100)

NOTE: Values in parentheses are not certified but are given for information only.

The technical direction and physical measurements leading to certification were provided by J. Fu of the Precision Engineering Division, with guidance on statistical analysis provided by M.C. Croarkin of the Statistical Engineering Division. Specimens were produced by D.B. Ballard, M.E. Taylor Engineering, Brookeville, Maryland.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by N.M. Trahey.

Gaithersburg, MD 20899
March 24, 1992

William P. Reed, Chief
Standard Reference Materials Program

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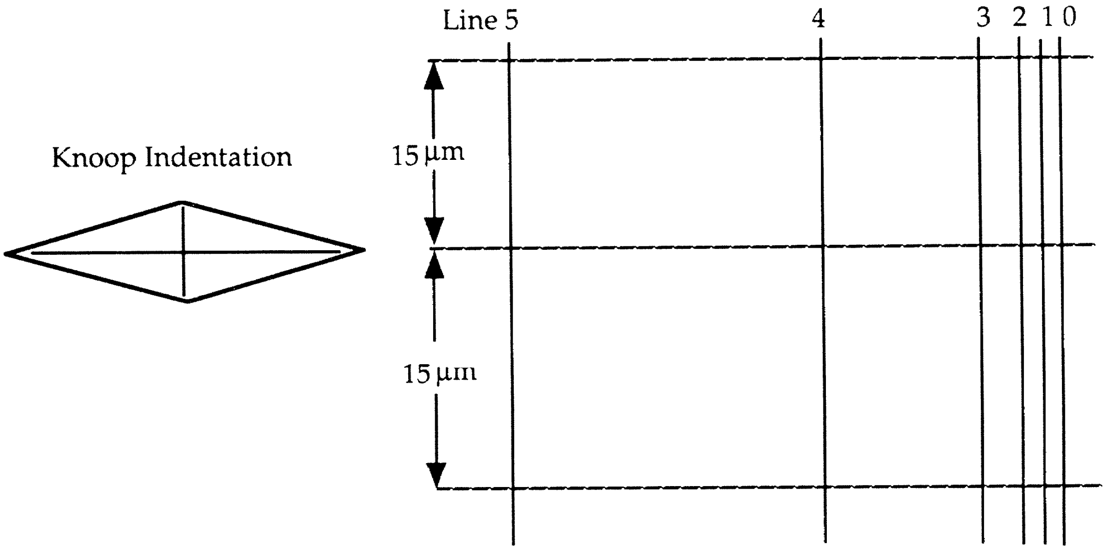

Figure 1. Diagram of Certified Region of SRM

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 across the fixed electron beam to calculate the distances between peaks from stage positions and a peak-finding algorithm. The certified value for each distance is an average of nine measurements at seven locations.

The surface of each SRM has been carefully ground and polished using metallographic techniques. Cleaning should not be attempted as it could void the certified spacing values.

A recommended procedure for calibrating the magnification of the SEM using SRM 484f is given on the following page and in ASTM E766, Practice for Calibrating the Magnification of SEM Using SRM 484 (current version). 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 pages 4 and 5 of this certificate.

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. The procedure on page 3 details the use of NIST SRM 484f to calibrate one particular SEM, but may be used as a guide for calibration of other SEMs.

13. Make the measurement 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. Measure the spacing between the lines at three locations within the calibrated region and average the values to determine the spacing.

15. Magnification = $\dfrac{\text{Spacing measured between image lines on photograph}}{\text{Certified spacing between same lines}}$

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

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.

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 photograph 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.

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