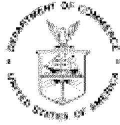

National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 484g

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 1000 x to 20 000 x. 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 µm, 1 µm, 3 µm, 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 in diameter by 6.5 mm high.

The certified region of the SRM is located relative to a Knoop indentation (see Figure 1). Spacings between the centers of the gold lines are certified. SEM photomicrographs, showing the certified region, are provided with the SRM. The certification is valid within 15 µm on either side of an imaginary line extending from the Knoop indentation mark normal to the gold lines.

Table 1. Certified Spacing Values and Uncertainties

Line Pair	Nominal Spacing (µm)	Certified Spacing Values (µm)	Uncertainty (µm)
0→1	0.5		± 0.034
1→2	0.5		± 0.039
2→3	1		± 0.038
3→4	3		± 0.044
4→5	5		± 0.051
(0→5)	(10)		(± 0.059)

Note: Values in parentheses are not certified and are given for reference only.

Expiration of Certification: The certification of this SRM is deemed to be indefinite, provided the SRM is handled and stored in accordance with the Care and Handling and Cautions to User sections of this certificate. Any physical damage or other alteration of the specimen surface, including any processes that remove surface material sufficient to obliterate the Knoop indentation, will void the certification.

Specimens were produced by D.B. Ballard, M.E. Taylor Engineering, Brookeville, MD.

Guidance on statistical analysis was provided by M.C. Croarkin of the NIST Statistical Engineering Division.

The technical direction and physical measurements leading to certification were provided by J. Fu of the NIST Precision Engineering Division.

The support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.J. Gettings.

Gaithersburg, MD 20899
Certificate Issue Date: September 10, 1996

Thomas E. Gills, Chief
Standard Reference Materials Program

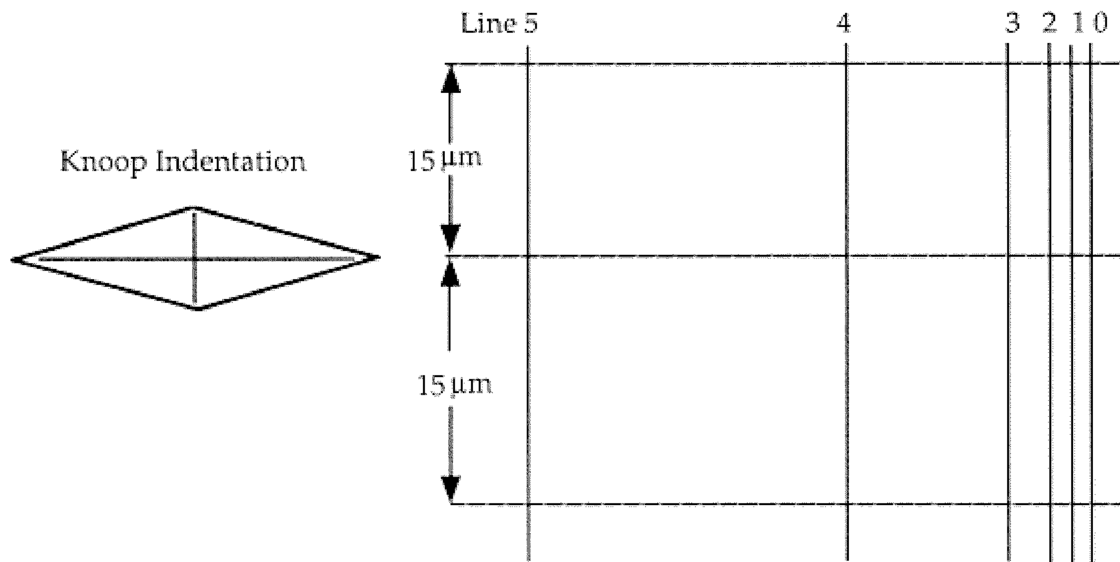

Figure 1. Diagram of Certified Region of SRM 484g

Certification Technique: 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 is used to calculate the exact peak location. The certified value for each distance is an average of nine measurements at seven locations.

Certified Value Uncertainties: The uncertainty for each certified value is calculated as $U = (2\,u_c)$ and includes allowances for long-term measurement variability, disparities within the certified region, instrument imprecision, and differences between realizations by scanning electron microscopy and line-scale interferometry. All uncertainty components are Type A and are calculated according to the ISO and NIST Guides [1]. The uncertainty interval, $\pm\,U$, defined by the certified value, is a two standard deviation prediction interval for a measurement at any point within the certified region of the specimen.

Reference Value: A reference spacing value is given for the 10 µm line spacing, line pair (0-5). This value is not certified as it was calculated by summation of the certified spacing values.

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

Care and Handling of SRM 484g: When unwrapping and rewrapping the SRM unit, the following procedure must be followed.

1. Place the polished surface down onto a clean piece of lens tissue. Do not use silicone-treated tissue.

2. Fold the tissue edges upward around the SRM unit while holding the tissue against the side of the unit. The polished surface of the unit should not slide over the tissue surface.

3. Twist the tissue pigtail down against the back side of the SRM unit.

4. Store the wrapped SRM unit in its plastic capsule when not in use.

Recommended Calibration Procedure: The following is the recommended procedure for calibrating the magnification of an SEM using SRM 484g. This procedure can also be found in the current version of Reference [2]. 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 a SEM can be found on pages 4 and 5 of this certificate.

Note: 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. This calibration procedure details the use of SRM 484g to calibrate one particular SEM, but may be used as a guide for calibration of other SEMs.

1. Rigidly mount the SRM unit on an SEM stub with electrically conductive cement or clamp it onto the SEM stage after its surface has been inspected for cleanliness.

2. Make sure the surface of the SRM unit is normal to the electron beam.

3. Evacuate to a vacuum of 10^{-3} Pa (10^{-5} Torr), or better, to keep the contamination rate as low as possible.

4. Allow a 30 min or more warm-up of electronic circuits to achieve operational stability, unless it has been established that a shorter time will suffice.

5. Adjust electron gun voltage (between 5 kV and 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. Assure SEM resolution is a minimum of 50 nm (500 Å), or better.

9. The calibrated region extends 15 µm above and below the Knoop indentation as shown in Figure 1. To view the calibrated region, position the SRM unit at a nominal magnification of 1000 x, so that the image of the Knoop indentation is visible at one side of the viewing cathode ray tube (CRT). All practice sessions should be conducted on these gold lines away from this calibrated region to reduce contamination to the region and extend the lifetime of the SRM.

10. Reproduce the same working distance or magnification scale of the SEM by focusing on the image of the SRM unit's 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. Perform the following steps to minimize the effect of linear distortions produced by the recording system: a) substitute the SRM for the unknown sample then photograph, b) choose lines on the SRM to be used in the calibration so that the spacing between them matches the length of the object to be measured with both images positioned in the same area on the CRT, c) tape a millimeter scale onto the edges of the CRT in the "x" and "y" directions to assist in the relocation of the respective images.

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

13 Make the measurement by automated image analysis of the CRT image or measurement of the spacing by a photographic recording of the CRT image. If photographic recording is used, the prints (if using Polaroid), should be dried 15 min to 20 min 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 different locations within the calibrated region and average the values to determine the spacing.

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

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 SRM: The parameters described below may interact with each other. They are listed 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. Non-orthogonal defection (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 isotopic 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 non-orthogonal 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.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, D.C. (1994).

[2] ASTM E 766-93, Practice for Calibrating the Magnification of SEM Using SRM 484, **Vol. 03.01** ASTM Book of Standards, West Conshohocken, PA 19428.