Department of Commerce
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National Bureau of Standards
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National Bureau of Standards

Certificate

Standard Reference Material 484d

Scanning Electron Microscope Magnification Standard

(A Stage Micrometer Scale)

This Standard Reference Material (SRM) is intended for use in calibrating the scanning electron microscope (SEM) magnification scale to an accuracy of 5% or better within the range of 200X to 20,000X. Each SRM bears an identification number and has been individually measured.

The certified distances between the centers of specific lines opposite the Knoop indentation (see the Figure on page 2) are provided with each SRM, together with a photomicrograph that shows the area used in the measurement. The certification is valid within an area 30 μm wide centered about a line extending from the Knoop indentation.

The distances between the lines were measured by an SEM that uses a scanning specimen stage, the displacement of which is determined by a helium-neon laser interferometer measurement system. A measurement consisted of scanning the SRM beneath the fixed electron beam while logging the back-scattered electron output signal versus the stage displacement on a minicomputer-based data acquisition system. Line positions and distances between lines were subsequently calculated. The reported value for each distance is an average of multiple scans within the calibration region.

The total uncertainty for the distances between line pairs is the linear sum of the limit of random uncertainty and a systematic uncertainty of 0.5% of the distance measured. The systematic uncertainty is the result of perturbation to the electron beam position caused by the motion of the magnetically permeable SRM material through the objective lens stray field. Any measurement technique which relies on movement of the SRM will be limited in accuracy by this systematic uncertainty. The uncertainties for each nominal distance are given in the following table.

Table of Uncertainties of Distances on SRM 484d

Nominal Distance	Total Uncertainty
1 μm	± 0.027 μm
2	.033
3	.038
5	.048
10	.085
20	.14
30	.20
50	.33

The technical direction and physical measurements leading to certification were provided by G. G. Hembree and J. Unguris of the Mechanical Production Metrology Division, with guidance on statistical analysis provided by M.C. Croarkin of the Statistical Engineering Division.

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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 L.J. Kieffer.

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Stanley D. Rasberry, Chief
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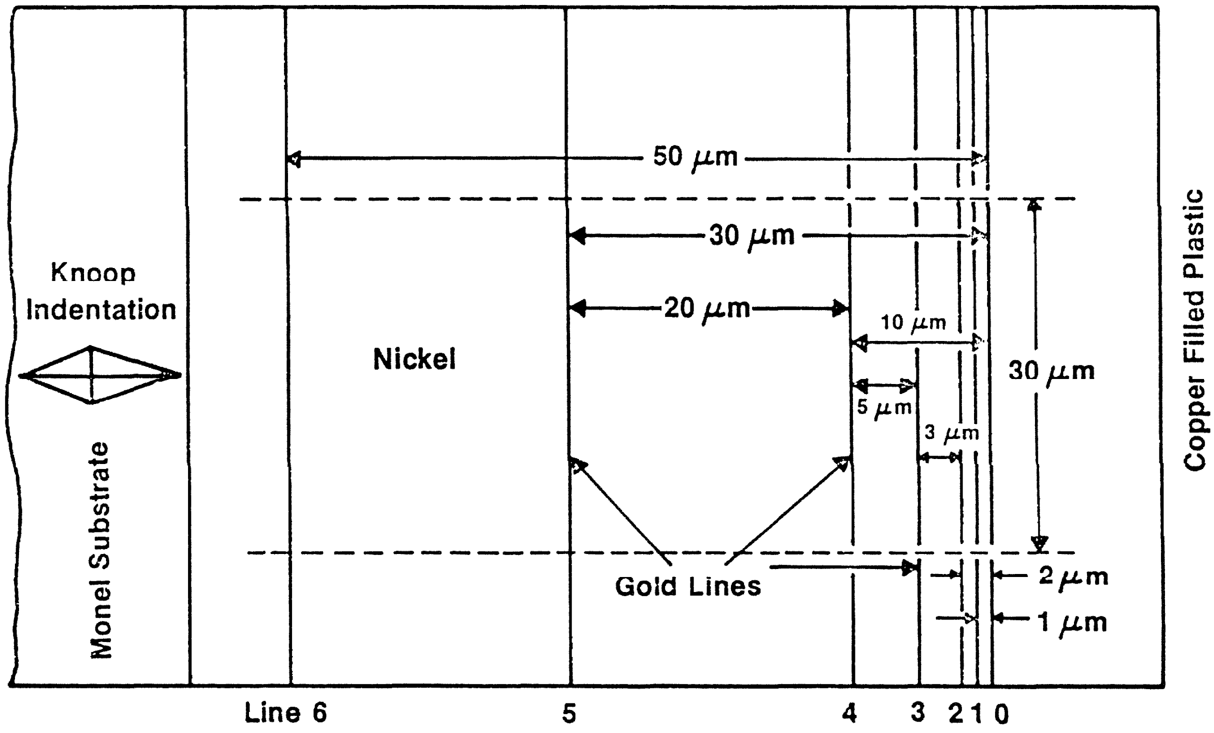

The surface of each SRM has been carefully ground and polished using metallographic techniques. The carbonaceous contamination (a product of SEM electron beam bombardment) can be removed by light hand-polishing on a stationary surface covered with micro cloth, using metallographic 0.05-µm γ-alumina powder. While this cleaning process does not alter the certified spacing of the lines by more than 0.010 µm, such cleaning should not be attempted unless signal strength is inadequate. Other cleaning techniques that remove surface material sufficient to obliterate the Knoop indentation will void the calibrated distance values.

A recommended procedure for calibrating the magnification of the SEM using SRM 484d is given on the following page and in ASTM E766, Practice for Calibrating the Magnification of SEM using SRM 484d. 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 NBS SRM 484d 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 484d 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 SRM 484d should be normal to the electron beam or the tilt axis of the stage should be perpendicular to the gold lines of the SRM.
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 30 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 a 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 area 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 for 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. After photo recording, if using Polaroid, allow prints to dry 15 to 20 minutes, or more, to minimize effects due to emulsion and coating shrinkage.
14. Measure the perpendicular distance between the lines using the *CENTER* of each line image on the photograph with a TEM Diffraction Plate Reader, or use an equivalent instrument, the precision of which (about 0.2 mm) is suitable for this purpose.
15. Repeat measurements 3 times on each photograph to determine the average spacing.
16. $\text{Magnification} = \dfrac{\text{Distance measured between image lines on photograph}}{\text{Certified distance between same lines}}$
17. To determine the SEM stability and reproducibility, repeat all steps at hourly or daily intervals, or after adjustments and repairs.

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.

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 and magnification variation.
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.