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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Practice for Scanning Electron Microscope Beam Size Characterization[1]

This standard is issued under the fixed designation E 986; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice provides a reproducible means by which one aspect of the performance of a scanning electron microscope (SEM) may be characterized. The resolution of an SEM depends on many factors, some of which are electron beam voltage and current, lens aberrations, contrast in the specimen, and operator-instrument-material interaction. However, the resolution for any set of conditions is limited by the size of the electron beam. This size can be quantified through the measurement of an effective apparent edge sharpness for a number of materials, two of which are suggested. This practice requires an SEM with the capability to perform line-scan traces, for example, *Y*-deflection waveform generation, for the suggested materials. The range of SEM magnification at which this practice is of utility is from 1000 to 50 000 × . Higher magnifications may be attempted, but difficulty in making precise measurements can be expected.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 7 Terminology Relating to Metallography[2]

E 766 Practice for Calibrating the Magnification of a Scanning Electron Microscope[2]

3. Terminology

3.1 *Definitions:* For definitions of terms used in this practice, see Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Y-deflection waveform*—the trace on a CRT resulting from modulating the CRT with the output of the electron detector. Contrast in the electron signal is displayed as a change in *Y* (vertical) rather than brightness on the screen. This operating method is often called *Y*-modulation.

4. Significance and Use

4.1 The traditional resolution test of the SEM requires, as a first step, a photomicrograph of a fine particulate sample taken at a high magnification. The operator is required to measure a distance on the photomicrograph between two adjacent, but separate edges. These edges are usually less than one millimetre apart. Their image quality is often less than optimum. Operator judgment is dependent on the individual acuity of the person making the measurement and can vary significantly.

4.2 Use of this practice results in SEM electron beam size characterization which is significantly more reproducible than the traditional resolution test using a fine particulate sample.

5. Suggested Materials

5.1 SEM resolution performance as measured using the procedure specified in this practice will depend on the material used; hence, only comparisons using the same material have meaning. There are a number of criteria for a suitable material to be used in this practice. Through an evaluation of these criteria, two samples have been suggested. These samples are nonmagnetic; no surface preparation or coating is required; thus, the samples have long-term structural stability. The sample-electron beam interaction should produce a sharply rising signal without inflections as the beam scans across the edge. Two such samples are:

5.1.1 *Carbon fibers,* NIST—SRM 2069B.[3]

5.1.2 *Fracture edge of a thin silicon wafer*, cleaved on a (111) plane.

6. Procedure

6.1 Inspect the specimen for cleanliness. If the specimen appears contaminated, a new sample is recommended as any cleaning may adversely affect the quality of the specimen edge.

6.2 Ensure good electrical contact with the specimen by using a conductive cement to hold the specimen on a SEM stub, or by clamping the specimen on the stage of the SEM. Mount the specimen rigidly in the SEM to minimize any image degradation caused by vibration.

6.3 Verify magnification calibration for both *X* and *Y* directions. This can be accomplished by using Practice E 766.

6.4 Use a clean vacuum of 1.33 by 10^{-2} Pa (10^{-4} mm Hg) or better to minimize specimen contamination resulting from

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[1] This practice is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.11 on X-Ray and Electron Metallography.

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[2] *Annual Book of ASTM Standards,* Vol 03.01.

[3] Available from National National Institute of Standards and Technology, Gaithersburg, MD 20899.

electron beam and residual hydrocarbons interacting during examination. The presence of a contamination layer has a deleterious effect on image-edge quality.

6.5 Allow a minimum of 30 min for stabilization of electronic components. The selection of optimum SEM parameters is at the discretion of the operator.[4]

6.6 Select electron gun voltage within the desired range of operating conditions.

NOTE 1—The performance measurement must be repeated for each kV setting used.

6.7 Saturate the filament and check both filament and gun alignment for any necessary adjustment.

6.8 Set all lens currents at a resettable value with the aid of a suitable digital voltmeter, if available.

6.9 Cycle lens circuits OFF-ON two to three times to minimize hysteresis effects. An alternate procedure may be used to drive the lens through a hysteresis loop—increase current above operating current, decrease below operating current, then back up to operating current.

6.10 Adjust lens apertures and stigmator for optimum resolution (minimum astigmatism). Because of its higher resolution, the secondary electron imaging mode is most commonly used. This procedure may also be used to characterize SEM performance in the backscattered electron imaging mode.

6.11 Locate a field on the chosen specimen that shows the desired edge detail. (See Fig. 1.)

6.12 Select the highest magnification that is sufficient to allow critical focusing of the image and shows image-edge transition from white to black contrast (for example, *fuzziness*) of at least 5-mm horizontal width in the photographed image.

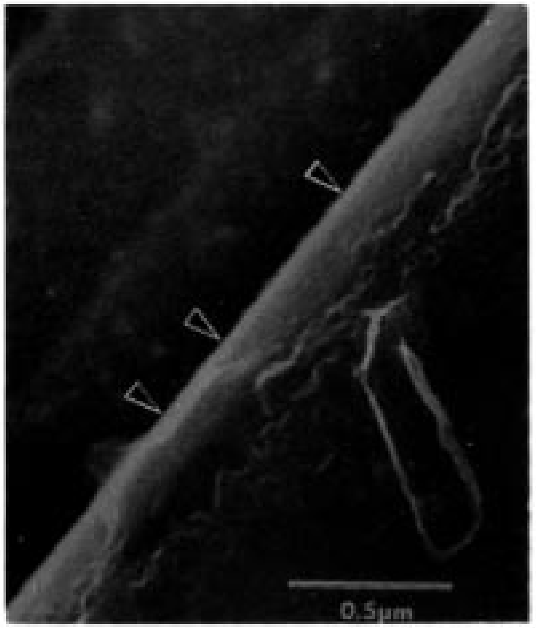

FIG. 1 Edge of Graphitized Natural Cellulose Fiber Used to Produce Line Traces (Fig. 3)

6.13 Rotate the specimen, not the scan, and shift the field of view on the specimen so that the desired edge is oriented perpendicular to the horizontal scan direction near the center of the CRT.

6.14 Make sure that no gamma or derivative processing is employed.

6.15 Obtain a line-trace photograph across the desired edge using a recording time of at least 2 s and a bandwidth of 4 kHz (if SEM is equipped with bandwidth control). (See Fig. 2.)

6.15.1 **Caution**—Slow scan rates in the line-trace mode may cause burning of the CRT-screen phosphor for improperly adjusted SEM-CRT screens.

6.16 Locate the maximum and minimum *Y*-axis deflections across the edge of the specimen in the line-trace photograph. (See Fig. 2.)

6.17 The difference between these values is the full-edge contrast produced in the line trace. From this contrast value, compute the *Y*-axis positions that correspond to contrast levels of 20 and 80 % of the full-contrast value.

$$20\ \%\ \text{level} = 0.2 \times (\gamma_{max} - \gamma_{min}) + \gamma_{min} \quad (1)$$

$$80\ \%\ \text{level} = 0.8 \times (\gamma_{max} - \gamma_{min}) + \gamma_{min} \quad (2)$$

6.17.1 These levels are illustrated schematically on Fig. 2. Locate these positions in the line-trace photograph and measure the horizontal distance (*D*) in mm on the photograph between these points. The slope of the line trace should have a ratio (*Y/D*) of 2 to 4. The distance (*D*) should range between 2 to 4 mm. The performance parameter (*P*), expressed in nanometres, is then defined as follows:

$$P = (D \times 10^6)/M \quad (3)$$

where *M* is the SEM calculated and corrected magnification using an acceptable standard.

6.18 Photograph the field selected for later reference to aid in the location of the image edge used for the performance measurement.

6.19 Repeat the line-trace photograph and measurement process outlined in 6.15 through 6.17 at two additional edges in the material studied. Three waveform traces using a graphite-fiber edge are shown in Fig. 3.

6.20 Average the three results to produce the performance parameter (*P*).

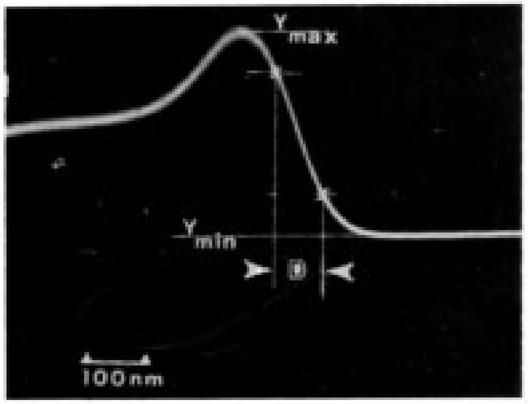

FIG. 2 Typical Waveform With 20 and 80 % Contrast Levels Illustrated

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[4] Newbury, D. E., "Imaging Strategy for the SEM–A Tutorial," *SEM*, Vol. 1, 1981, pp. 71–78.

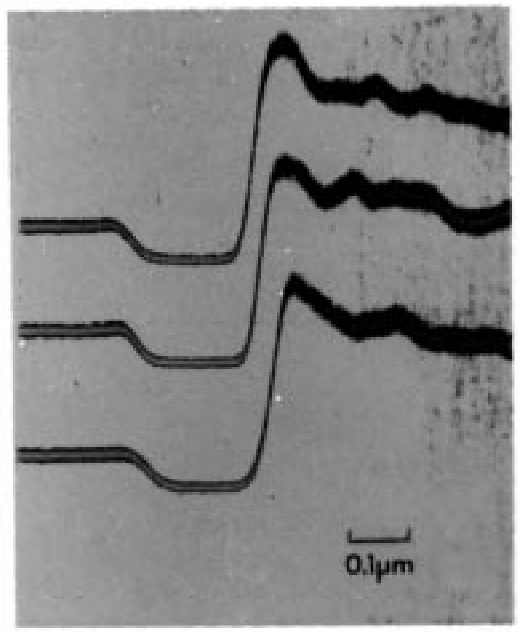

FIG. 3 Set of Waveforms Measured to Determine Performance Parameter (P) (Eq 1)

$$[P = (P_1 + P_2 + P_3)]/3 \quad (4)$$

7. Precision and Bias

7.1 At the present time, it is not possible to give a specific value for the precision and bias of the performance test based on extensive experience. However, the sources of error and their best estimates of uncertainties at a SEM magnification of 80 to 100 000 × under controlled operating conditions and with experienced operators, are as follows:

Source	Uncertainty, %
SEM magnification (M)	±10
Measurement variation between operators	±2
Measurement of waveform (D)	±2
Approximate overall uncertainty	11

8. Reproducibility

8.1 Reproducibility of the performance parameter may be determined by repeating the steps in Section 6 at intervals determined by the user's requirements. Measurement of performance is recommended after repair or realignment of the electron optical functions or after major changes in instrument-operating parameters, for example, beam voltage or lens settings, or both. A listing of instrument parameters that influence the performance is included in the Annex of Practice E 766.

9. Keywords

9.1 edge sharpness; electron beam size; E766; graphite fiber; magnification; NIST–SRM 2069B; performance; SEM; specimen interaction; waveform