Designation: ~~E986 – 04 (Reapproved 2010)~~ E986 – 04 (Reapproved 2017)

Standard Practice for Scanning Electron Microscope Beam Size Characterization[1]

This standard is issued under the fixed designation E986; 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.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E7 Terminology Relating to Metallography

E766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

3. Terminology

3.1 *Definitions:* For definitions of terms used in this practice, see Terminology E7.

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 limited by the S/N ratio of a beam with such a small diameter and low current. 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.

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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 E766.

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 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, vacuum stability, and thermal equilibrium for the electron gun and lenses. The selection of optimum SEM parameters is at the discretion of the operator.[4] For measuring the ultimate resolution, these will typically be: high kV (~30max.), short working distance (5 to 10 mm), smallest spot size, and long scan time.

6.6 Any alternative set of conditions can be used to measure probe size, but they will measure beam diameter under those specific conditions, not ultimate resolution.

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. Allow time for stabilization.

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

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.) Avoid tilting the stage since this will change the magnification due to image foreshortening.

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.

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 60 s. (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 analog 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.

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

[3] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

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