

SEMI F73-1102

TEST METHOD FOR SCANNING ELECTRON MICROSCOPY (SEM)

EVALUATION OF WETTED SURFACE CONDITION OF STAINLESS STEEL COMPONENTS

This test method was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on August 29, 2002. Initially available at www.semi.org September 2002; to be published November 2002.

1 Purpose

1.1 This document defines a uniform procedure for testing the wetted surfaces of stainless steel components intended for installation into high purity gas distribution systems. This procedure characterizes the occurrence, frequency, and in some cases the identity of microscopic surface defects and contaminants that may appear on the wetted surfaces. It should be noted that there has been no direct correlation made between the results of this test method and contamination of process gases or product yields in processes served by high purity gas distribution systems. Application of this test method is intended to yield comparable and reproducible results among various users of this method for the purposes of qualification of components.

1.2 The objective of this method is to describe a general set of instrument parameters and conditions that will achieve precise and reproducible measurements of important parameters regarding the surface condition.

2 Scope

2.1 This procedure applies to the wetted surfaces in stainless steel tubing, fittings, valves, and other components to determine the effectiveness of surface finishing and cleaning processes. The technique describes counting of surface defects including pits, inclusions, inclusion stringers, scratches, residual process marks, grain boundaries and contamination on the wetted surfaces. However, any surface damage produced during sample preparation is to be excluded from such assessment.

2.2 This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety health practices and determine the applicability or regulatory limitations prior to use.

3 Limitations

3.1 This test method is primarily intended to determine the frequency of occurrence (surface density) and identity of microscopic surface defects, down to a size range of $\geq$ approximately 0.1 micrometer, that may be

detrimental to high purity gas distribution systems. Such defects may not be identifiable by visual or magnified optical inspection, but concentrations of microscopic surface defects may cause visually apparent defects such as haze.

3.2 This test method requires sectioning of the specimen(s) used for test purposes and is therefore destructive.

3.3 The method must be applied to representative examples of process lots to determine quality of the processes employed and/or lots processed. As the areas examined and measured by this method are very small, the results may not be representative of all areas or all lots processed.

3.4 This test method may be subject to operator bias in selection of representative areas and definition of countable defects.

3.5 Detection of countable defects is affected by operator selection of SEM operating conditions and image recording conditions. This test method assumes that the operator is sufficiently proficient in operation of the SEM to minimize this limitation, per instructions in the procedures.

3.6 SEM imaging will reveal surface finish flaws, but may not show features that are well rounded by an electropolishing or other surface leveling process.

3.7 Energy dispersive X-ray spectroscopy (EDS) may be used in this test method to analyze surface contaminants at least approximately one micrometer in thickness and inclusions at least approximately one micrometer in size. EDS is not an appropriate technique for analysis of the oxide passive layer on the stainless steel surface, as EDS analyzes down to a depth of the order of 1 micrometer below the surface, and the oxide passive layer is only 0.001 to 0.01 micrometer deep.

4 Referenced Standards

4.1 ASTM Standards¹

ASTM E 7 — Standard Terminology Relating to Metallography

ASTM E 766 — Standard Practice for Calibrating the Magnification of a Scanning Electron Microscope

4.2 ANSI/IEEE Standard³

759 — Standard Test Procedures for Semiconductor X-ray Energy Spectrometers

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *EDS* — Energy Dispersive X-ray spectroscopy, sometimes called EDX.

5.1.2 *FWHM* — abbreviation of Full Width Half Maximum; the width of an EDS peak measured at half its maximum height.

5.1.3 *SEM* — Scanning Electron Microscopy.

5.2 Definitions

5.2.1 *defect* — any surface feature that is either characteristic of the material, or a result of material processing or product fabrication, that is deemed capable of generating and/or trapping and releasing particles, or otherwise be detrimental to the contained fluids. Specific features that may be considered to be defects, with definitions that relate to the purposes of this document and/or obtained from reference sources noted include:

5.2.1.1 *contamination* — three dimensional alien material adhering to a surface. (SEMI Compilation of Terms)

5.2.1.2 *grain boundary* — an interface separating two grains, where the orientation of the lattice changes from that of one grain to that of the other. (ASTM E 7)

5.2.1.3 *inclusion* — indigenous or foreign material within the metal, usually referring to non-metallic

compound particles such as oxides, alumina, sulfides or silicates.

5.2.1.4 *pit* — a surface cavity or crater with a defined edge not caused by impact.

5.2.1.5 *process marks* — a surface texture or pattern that is characteristic of the surface finishing process employed.

5.2.1.6 *scratch* — an elongated mark or groove cut in the surface by mechanical means, not associated with the predominant surface texture pattern. (adapted from SEMI Compilation of Terms)

5.2.1.7 *stringer* — a microstructural configuration of alloy constituents or foreign nonmetallic material, or trace thereof, lined up in the direction of working. (adapted from ASTM E 7)

5.2.2 *wetted surface* — surfaces of a component contacting the contained fluids. (adapted from SEMI Compilation of Terms)

5.2.3 *working distance* — the distance between the surface of the specimen being examined and the front surface of the objective lens. (ASTM E 7)

6 Summary of Method

6.1 Obtain a SEM photomicrograph of a representative area of the wetted surface of the sample at a magnification of 200×. Increase the magnification to 1000× and take another SEM photomicrograph within this area, and another at a pre-selected magnification within the range 3000 to 3600×. Obtain 3000–3600× photomicrographs at two additional representative areas, not necessarily within the 200× magnification region.

6.2 If inclusions or contaminants are noted on any 3000–3600× magnification photos, EDS spectra of representative example(s) may be obtained.

6.3 Perform grid overlay defect counting on the three 3000–3600× photos. Report results in tabular form; provide photomicrographs and EDS spectra with identification of elements detected.

7 Interferences

7.1 Interferences may be encountered as overlaps in EDS spectral peaks. Specific overlaps that may be encountered include but are not limited to:

7.1.1 *Molybdenum and Sulfur* — Overlap in EDS spectra of Mo L peaks and S K peaks prevents unambiguous determination.

7.1.2 *Manganese and Chromium/Iron* — Overlap in EDS spectra of Mn K α peak with Cr K β peak, and Mn

1 American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555 Website: www.astm.org

2 American National Standards Institute, New York Office: 11 West 42nd Street, New York, NY 10036, USA. Telephone: 212.642.4900; Fax: 212.398.0023 Website: www.ansi.org

3 Institute of Electrical and Electronics Engineers, IEEE Operations Center, 445 Hoes Lane, P.O. Box 1331, Piscataway, New Jersey 08855-1331, USA. Telephone: 732.981.0060; Fax: 732.981.1721

K β peak with Fe K α peak interferes with determination of low levels of Manganese in Iron-Chromium matrix.

8 Apparatus

8.1 *Instrumentation* — Any SEM instrument used for this method must be capable of a minimum point-to-point resolution of 30 nm, as measured with a NIST⁴ traceable standard. Any commercially available SEM with image recording capabilities of at least 100 cm² image and an EDS analyzer capable of 170 eV or less FWHM resolution for Mn K α may be employed.

8.2 The SEM shall have a sample stage capable of aligning the sample to provide multiple areas of view with the orientation of the electron beam approximately normal to the surface, and to optimize secondary electron and X-ray signal collection efficiencies.

8.3 *Grid Overlay* — A transparent grid overlay of grid size equal to 1.814 micrometers multiplied by the pre-selected magnification in the range 3000 to 3600 (grid size would be 6.35 mm [= ¼ inch] square for 3500 $\times$ magnification). The grid lines shall be as fine as possible but clearly visible. The grid overlay shall be designed to overlay the image of a photomicrograph with 252 grids (18 by 14 grids for the 3.5 by 4.5 inch image of a standard Polaroid photomicrograph) with minimal interference from alphanumeric information recorded with the photomicrograph, but without reference to any features in the photomicrograph. The rows and columns of the grid overlay may be indexed with alphanumerics to identify and locate specific features in the photomicrographs (see examples in Appendices 1 and 2). It is suggested that a fixed reference point for the edges of the photomicrograph be established to be consistently used in all overlays.

9 Reagents and Materials

9.1 *Sample Preparation Materials* — Equipment required to section the test specimen without damaging or contaminating the surfaces to be analyzed. A clean, dry hacksaw or dry bandsaw is recommended, using a slow cutting speed to avoid excessive sample heating.

9.2 *Sample Mounting Materials* — Sample mounting holders specific to the SEM instrument used. Conductive paste or tape used to adhere the sample to the holder must be vacuum stable and applied so that the area of analytical interest is not contaminated.

10 Safety Precautions

10.1 This test method does not purport to address the safety considerations associated with use of high voltage, vacuum, electron and X-ray producing equipment.

10.2 The method assumes a SEM and EDS analyst with adequate skill level as well as knowledge of instrumentation and associated safety precautions.

11 Test Specimen

11.1 Specimens are to be sectioned to appropriate size for the particular SEM instrument. Any sample preparation technique used shall avoid introducing contamination onto the surface to be measured. In addition, preparation must avoid heating of the sample in excess of approximately 100°C to avoid oxide growth or change in surface composition. Slow speed dry cutting is recommended.

11.2 After preparation, samples shall be protected from contamination by means such as sealing in noncontaminating bags or wrapping in clean aluminum foil. Appropriate cleaning of the analysis surface to remove sample preparation residues is allowed but not required. Any such cleaning procedures should be stated in the report. After sectioning, samples should be analyzed promptly.

11.3 The samples shall be analyzed in the uncoated condition; ie, without sputtering with a conductive coating.

12 Preparation of Apparatus

12.1 The SEM shall be in good condition to assure proper performance in accordance with the manufacturer's specifications.

13 Calibration and Standardization

13.1 Instrument calibration for magnification and EDS performance shall be performed in accordance with ASTM E 766 and ANSI-IEEE 759 and/or the instrument manufacturer's recommendations. The magnification should be calibrated at the working distance selected. Calibration frequency shall be per instrument manufacturer's recommendations.

14 Procedure

14.1 The sample is to be mounted in accordance with manufacturer's recommendations and in a manner consistent with high vacuum analytical procedures. The sample shall be oriented such that the areas of interest for SEM imaging can be viewed at 70 to 90° incidence angle of the electron beam to the surface with optimum secondary electron collection efficiency, and the ability to re-orient the sample for optimization of X-ray

⁴ National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899, (301) 975-NIST (6478) TTY (301) 975-8295, website: www.nist.gov.

collection efficiency. Any adhesives used to mount samples must not contaminate the surface to be analyzed. To remove loosely adhered surface particles, the sample may be blown off immediately before introduction into the SEM with clean, dry, 0.1 micrometer filtered gas.

14.2 Place the sample in the SEM chamber for pumpdown. Activate the electron beam when vacuum conditions meet manufacturer's recommendations. The instrument accelerating voltage should be a consistently selected value within the range 15 to 25 KeV; 20 KeV is suggested. The working distance should be within the range recommended by the instrument manufacturer. Sample position (tilt angle and orientation) may be adjusted to optimize EDS detector collection efficiency for EDS analysis.

14.3 Increase the magnification to $\geq 400\times$ for initial focus; adjust instrument parameters for astigmatism and other anomalies. Decrease magnification to $200\times$ and move the sample while viewing until an area judged as representative of the whole is in view. The area to be analyzed should be as free of particles and defect features produced by sample preparation as possible.

14.4 Refocus and record images of this area at $200\times$, $1000\times$, and a pre-selected, consistently used magnification within the range 3000 to $3600\times$. Move to another representative area and record an image at the same 3000–3600 $\times$ magnification. Move to a third representative area and record another image at the 3000–3600 $\times$ magnification.

14.5 Each area selected for recording should include a defect or metallurgical feature to demonstrate proper focusing, contrast adjustment and resolution capability. Contrast adjustment should be performed per the technique described in Section 4.7.2.1 of *Scanning Electron Microscopy and X-Ray Microanalysis*.⁵ All defects and/or features for which the change in signal ΔS due to the contrast exceeds the noise N by a factor of 5 minimum must be clearly distinguishable in the image recorded (see Section 4.5 of reference 5).

14.6 If any inclusions or contamination are noted in the 3000–3600 $\times$ images, EDS analysis of representative defect(s) may be performed if requested by the company for which the test method is performed. Collect X-ray signals for a sufficient length of time to obtain an integrated count of $\geq 250,000$ within the range of 0 to 10 keV.

⁵ Scanning Electron Microscopy and X-Ray Microanalysis: A Text for Biologists, Materials Scientists, and Geologists; Joseph I. Goldstein, Dale E. Newbury, Patrick Echlin, David C. Joy, A. D. Romig, Jr., Charles E. Lyman, Charles Fiori, Eric Lifshin; Second Edition, Plenum Press, New York (1992).

15 Calculations or Interpretation of Results

15.1 The 3000–3600 $\times$ recorded images shall be placed under the transparent overlay grid. The number of grid squares that contain all or part of a surface defect shall be counted and summed for each photomicrograph. Each such grid square shall only be counted once; it is not the intent of this Test Method to count individual defects. If a defect appears in more than one adjacent grid square, each grid square occupied shall be counted.

NOTE 2: Examples of scanning electron micrographs overlaid with an alphanumeric indexed grid showing specific types of defects with comments on their locations and whether they should be counted may be viewed at <http://www.semi.org/web/wstandards.nsf/url/SurfaceAnalysisExamples>.

15.2 Surface film residues resulting from improper cleaning are generally diffuse and difficult to quantify. These residues, if present, are counted as one per image. Particles that loosely adhere to the surface must be presumed to be artifacts of sample preparation or exposure, and therefore shall be ignored.

15.3 Peaks appearing in the EDS spectra shall be identified and the spectra labeled to indicate whether it is a representative area, or identified as a defect from a photomicrograph.

16 Reporting Results

16.1 A tabular summary of defects counted per 3000–3600 $\times$ photomicrograph shall be presented. Additionally, the table should indicate an average of defect counts and the maximum from the three 3000–3600 $\times$ images. An example of a table reporting defect counting results and the photomicrographs from which the defect counts were taken are shown in Appendix 1.

16.2 All photomicrographs are to be permanently labeled with sample identification, magnification, a magnification scale bar, date, and analyst identification. The photomicrographs should be available for inspection upon request.

16.3 EDS spectra are to be labeled with the same information, plus peak identifications. If any instrument parameters (e.g., tilt angle) were changed for EDS analysis, these must be noted. If peak height differences are such that adequate representation of all peaks cannot be made from the same plot, two plots having different scaling shall be included. Identification of each defect analyzed, either by arrow on photomicrograph or by notation of grid position, shall be made.

17 Related Documents

17.1 ASTM Standards

ASTM F1372-93 — Standard Test Method for Scanning Electron Microscope (SEM) Analysis of Metallic Surface Condition for Gas Distribution System Components

ASTM F1375-92 — Standard Test Method for Energy Dispersive X-Ray Spectrometer (EDX) Analysis of Metallic Surface Condition for Gas Distribution System Components

SUPERSEDED