Designation: F 1372 – 93 (Reapproved 1999)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Method for Scanning Electron Microscope (SEM) Analysis of Metallic Surface Condition for Gas Distribution System Components[1]

This standard is issued under the fixed designation F 1372; 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.

INTRODUCTION

Semiconductor clean rooms are serviced by high-purity gas distribution systems. This test method presents a procedure that may be applied for the evaluation of one or more components considered for use in such systems.

1. Scope

1.1 This test method covers the testing of interior surfaces of components such as tubing, fittings, and valves for surface morphology.

1.2 This test method applies to all surfaces of tubing, connectors, regulators, valves, and any metal component, regardless of size.

1.3 *Limitations*:

1.3.1 This methodology assumes a SEM operator skill level typically achieved over a 12-month period.

1.3.2 This test method shall be limited to the assessment of pits, stringer, tears, grooves, scratches, inclusions, stepped grain boundaries, and other surface anomalies. However, stains and particles that may be produced during specimen preparation should be excluded in the assessment of anomalies.

1.4 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

1.5 *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.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 *NIST Standards:*

SRM 484 F SEM Magnification Standard[2]

SRM 20690 SEM Performance Standard[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *defect*—a pit, scratch, groove, inclusion, stringer, stepped grain boundary, crack, or other surface feature that is either characteristic of the material or a result of its processing that is not a result of the sample preparation.

3.1.2 *grid size*—the grid size (length of the x- and y-axis grid dimension) will be 1.814 µm multiplied by the magnification of the photomicrograph. For example, for a standard 4 by 5-in. photographic image at 3500 × magnification, the grid would be 0.635 by 0.635 cm (0.25 by 0.25 in.).

3.1.3 *groove*—a two-dimensional defect on the surface that has depth and width.

3.1.3.1 *Discussion*—For this kind of defect, the depth is greater than the width, or, conversely, the width is greater than the depth.

3.1.4 *inclusion*— particles of a foreign material in a metallic matrix (see Fig. 1).

3.1.4.1 *Discussion*—These particles are usually compounds (such as oxides, nitrides, carbo-nitrides, sulfides, or silicates), but may be of any substance (and is essentially insoluble in the metal matrix).

3.1.5 *number of anomalies*—the total number of defects per photomicrograph (see 10.1.1).

3.1.6 *particles that loosely adhere*—particles in which over ¾ of the bulk of the particle is above the plane of the surface.

3.1.6.1 *Discussion*—These particles generally appear very bright, and little detail of the surface of the particle is seen when the contrast and brightness are adjusted to image the sample surface.

3.1.7 *pit*—a small, sharp, roughly circular cavity in the metal surface (see Fig. 2).

3.1.8 *sample angle*—that angle measured normal to the incoming electron beam.

3.1.9 *scratch*— a one-dimensional defect on the surface such as a line on the surface.

---

[1] This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.10 on Processing Environments.

Current edition published Apr. 15, 1993. Approved June 1993. Originally published as F 1372 – 92. Last previous edition F 1372 – 92.

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

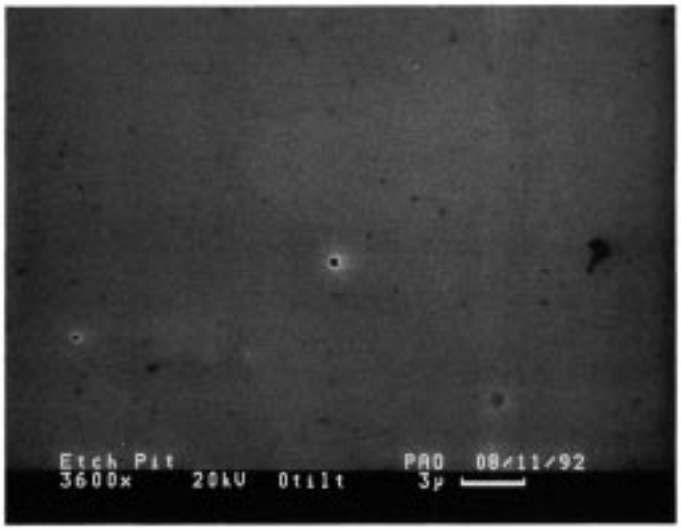

FIG. 1 Example of Inclusion (3600 × magnification)

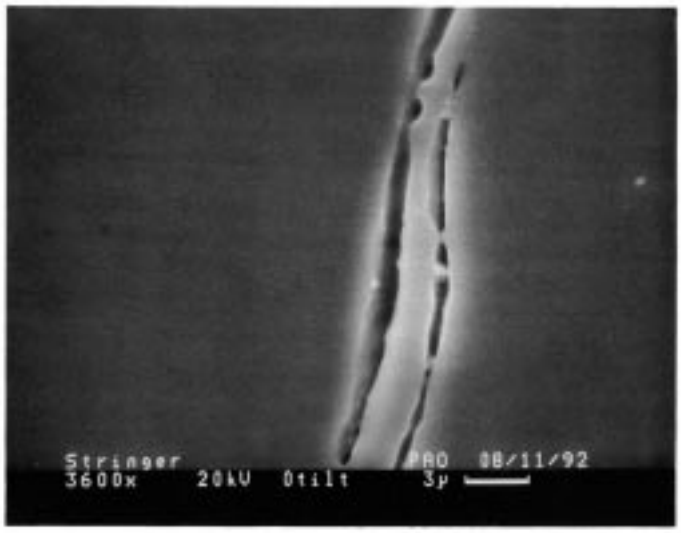

FIG. 3 Example of Stringer (3600 × magnification)

FIG. 2 Example of Pit Defect (3600 × magnification)

3.1.9.1 *Discussion*—For this type of defect, the depth of the defect is no deeper than the width of the defect.

3.1.10 *standard conditions*—101.3 kPa, 0.0°C (14.73 psia, 32.0°F).

3.1.11 *stepped grain boundary*—a grain boundary that has been etched to form a sudden change in height between adjacent grains.

3.1.12 *stringer*—in wrought materials, an elongated configuration of microconstituents or inclusions aligned in the direction of working (see Fig. 3).

3.1.12.1 *Discussion*—In electropolished stainless steel (SST), the stringer defect may have inclusion material on it, or the material may have been removed during electropolishing or cleaning, leaving an elongated void.

3.1.13 *working distance*—the distance between the bottom of the objective lens and the sample.

4. Significance and Use

4.1 The purpose of this test method is to define a procedure for testing components being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for purposes of qualification for this installation.

5. Apparatus

5.1 *Materials*:

5.1.1 *Mounting Stubs*, specific to the instrument used are required.

5.1.2 *Adhesives*, must be vacuum stable, to attach samples to sample stubs. Any adhesive that provides a conductive path is acceptable.

5.1.3 *Photomicrosamples*, must include the following information through the use of electronic notation on the SEM screen or ink on the back of the photomicrograph: sample identification, magnification, and date.

5.1.4 *Scale Marker*, (calibration bar) must be present and clearly visible on all photographs.

5.2 *Instrumentation*:

5.2.1 *Scanning Electron Microscope (SEM)*— The SEM used for this study should have a minimum point-to-point resolution of 30 nm as measured with NIST Standard SRM 20696 or equivalent. A high resolution commercially available SEM with photographic capabilities is recommended. The hard copy photomicrographic medium from which the defect count is taken must have an area of 100 cm^2.

5.2.2 *Instrument Operating Parameters*, shall be as follows: accelerating voltage, 20 KeV; working distance, 10 to 30 mm; sample tilt, 0°; and, final aperture size, 150 μm or less.

5.2.3 Magnification for quantitative pass/fail analysis shall be five randomly chosen areas photographed at 3500 ± 100×.

5.2.4 Instruments will be calibrated every 6 months and calibration verified prior to starting a series of test method measurements using standard laboratory practices and manufacturers' recommendations. Archive or supply magnification calibration check with results.

5.2.5 *Setup and Schematic*, to be furnished by instrument manufacturer.

6. Hazards

6.1 Observe all normal and acceptable precautions regarding use of high voltage, X-ray producing equipment.

7. Sampling, Test Specimens, and Test Units

7.1 Prepare the samples according to 9.1 of this test method to expose the surface.

7.2 Sample preparation shall not cause the temperature of the sample to exceed 90°C (194°F).

7.3 Mount the samples onto SEM compatible mounts in a manner that avoids contamination of the surface to be analyzed.

7.4 Use adhesives, when necessary, in a manner that does not contaminate the area of interest.

7.5 Do not coat samples with a conductive thin layer (for example, gold or carbon).

8. Calibration

8.1 Calibrate instruments regularly using standard laboratory practices and manufacturers' recommendations.

9. Procedure

9.1 *Sample Cutting and Mounting*:

9.1.1 Use any mechanical cutting method that minimizes alteration of the surface. A clean, dry hacksaw is preferred.

9.1.2 After cutting, clean samples in a reagent grade solvent and rinse with a reagent grade isopropyl alcohol (IPA). Place samples in a nitrogen-filled, resealable, non-outgassing container.

9.1.3 Mount samples on the instrument stub.

9.2 Introduce the sample stub into the SEM vacuum chamber.

9.3 Activate the electron beam when vacuum conditions meet those recommended by the manufacturer.

9.4 Move the sample until an area of interest on the sample's surface comes into focus. Make sure that the area of interest is representative of the whole, avoiding gross deformities.

9.5 Orient the sample to the degree that the longitudinal axis of the sample curvature, if applicable, is aligned with the axis of the secondary detector.

9.6 Increase the magnification to 20 000 to 40 000× for final focus, correcting astigmatism, and other instrument anomalies to yield a clear image.

9.7 Decrease the magnification to 3500 ± 100× and record the image on a photographic medium.

9.8 Move to a second random area and repeat the procedures in 9.5 through 9.7 for four additional sample sites. If additional analyses are required, they may be performed at this time, for example, energy dispersive X-ray spectrometer (EDX).

9.9 Turn off the SEM electron beam and remove the sample from the vacuum chamber.

10. Interpretation of Results

10.1 *Data Presentation*:

10.1.1 Overlay the recorded images with a scale as defined in 3.1.4. The grid line should be as fine as possible and still remain clearly visible. The lower left corner of the grid is to correspond with the lower left corner of the photograph. Sum the number of surface anomalies per square (such as pits, scratches, inclusions, and stringers) as the total per micrograph. Defects that appear in one or more adjacent squares shall count as one defect for each square occupied by the defect. Particles that loosely adhere to the surface must be presumed to be artifacts from atmosphere or sample preparation techniques, etc, and therefore will be ignored.

10.1.2 Present the data as photomicrographs (five from each sample) and in tabular form, showing total number of particles counted (per area analyzed) in the grid overlay. Photomicrographs must include the following information through the use of electronic notation on the SEM screen or ink on the back of the photomicrograph: sample identification, magnification, and date. The data table shall include a summation of the total counts for all five micrographs with the average and the maximum count for any one micrograph.

10.1.3 Use illustrations wherever confusion may exist regarding the area of analysis or whenever multiple sites on one sample must be identified.

10.1.4 The EDX spectra and corresponding photographs should be appropriately labeled so that the elemental composition of any specific defect, particle, or anomaly is readily apparent to any third party.

11. Report

11.1 Report the following information:

11.1.1 EDX spectra and related photomicrographs must include the following information: sample identification, date, peak identification, tilt angle, and voltage,

11.1.2 All data reported must identify the SEM equipment manufacturer and model number, and

11.1.3 Any special modifications in equipment or procedure necessary to acquire data must also be documented and fully described.

12. Precision and Bias

12.1 Precision and bias for this test method are being determined.

13. Keywords

13.1 components; contamination; gas distribution; metallic surface condition; SEM analysis; semiconductor processing; surface condition