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Designation: F 1372 – 93 (Reapproved 1999)

# 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).

[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.

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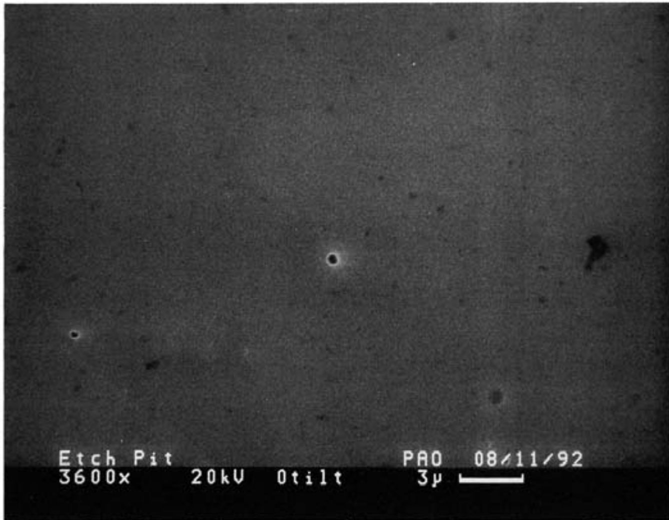


FIG. 1 Example of Inclusion (3600 × magnification)

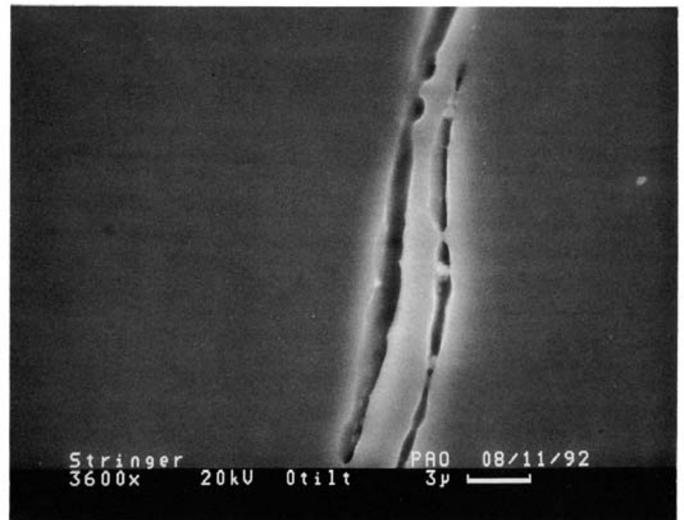


FIG. 3 Example of Stringer (3600 × magnification)

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

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.

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