Designation: F1375 – 92 (Reapproved 2012)

Standard Test Method for Energy Dispersive X-Ray Spectrometer (EDX) Analysis of Metallic Surface Condition for Gas Distribution System Components[1]

This standard is issued under the fixed designation F1375; 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 establishes a procedure for comparing the elemental composition of normal surfaces with any defects found on the surfaces of metal tubing, fittings, valves, or any metal component.

1.2 This test method applies to all steel surfaces of components such as tubings, connectors, regulators, and valves, regardless of size, style, or type.

1.3 *Limitations:*

1.3.1 This test method is intended for use by scanning electron microscope/energy dispersive x-ray spectrometer (SEM/EDX) operators with skill level typically achieved over a twelve-month period.

1.3.2 SEM used for this study should conform to those limitations outlined in Test Method F1372 and should have a minimum point-to-point resolution of 30 nm.

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 *ASTM Standards:*[2]

F1372 Test Method for Scanning Electron Microscope (SEM) Analysis of Metallic Surface Condition for Gas Distribution System Components

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *normal surface*—an area of the sample that does not exhibit any visible defect when viewed under the stipulated SEM magnification. Normal surface is used to provide a baseline for comparison with any area exhibiting a defect.

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

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

3.1.4 *working distance*—the measured distance from the bottom of the objective lens to 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:*

[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.10 on Contamination Control.

Current edition approved July 1, 2012. Published August 2012. Originally approved in 1992. Last previous edition approved in 2005 as F1375–92(2005). DOI: 10.1520/F1375-92R12.

[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.

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

5.1.1.1 Samples shall not be coated with a conductive thin layer (for example, gold or carbon).

5.1.2 *Conductive Paste/Tape,* that will provide a conductive path. Use any means of fixing the sample to a stub. Care should be taken not to contaminate the area of interest.

5.1.3 *Adhesives,* used to attach samples to sample stubs are to be vacuum stable.

5.2 *Instrumentation:*

5.2.1 *Scanning Electron Microscope (SEM)*—Any high resolution commercially available SEM with photographic capabilities of a 100 cm^2 image may be used for these analyses.

5.2.2 *Instrument Operating Parameters ,* shall be as follows: accelerating voltage, 20 KeV; final aperture size nominal 200 µm or less; and working distance and sample tilt, as appropriate to the EDX detector.

5.2.2.1 SEM instrument operating parameters shall be such that collection efficiency for the EDX spectrometer is optimized.

5.2.3 *EDX Spectrometer,* capable of full width half maximum (FWHM) resolution of 170 eV or less (for MnKα), and capable of detecting all elements with an atomic number greater than or equal to that of sodium (Na).

5.2.4 *Printer or Plotter,* capable of accurate spectral reproduction (linear-linear) is required.

6. Hazards

6.1 Observe all normal and acceptable precautions regarding use of high voltage, X-ray producing equipment. Observe standard and routine cryogenic handling procedures.

6.2 Use adhesives in such a manner that they do not contaminate the area of interest.

7. Sampling, Test Specimens, and Test Units

7.1 *Sample Cutting and Mounting :*

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

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

7.1.3 Mount the samples on the instrument stub.

7.2 Conduct sample preparation to ensure that the temperature of the sample does not exceed 90°C (194°F).

7.3 Mount the samples onto SEM compatible mounts in a manner to avoid contamination of the surface to be analyzed. Non-X-ray generating substrates, such as graphite, are preferred as mounting stubs.

8. Calibration

8.1 Calibrate and maintain instruments using standard laboratory practices and manufacturers' recommendations. Calibrate EDX spectrometers according to the manufacturer's specifications so that the energy calibration falls within ± 1 channel.

8.2 Magnification for qualitative and quantitative analysis shall result in incident beam concentration on the surface anomaly, minimizing stray X-ray signal from the background.

9. Procedure

9.1 Follow sample preparation of this test method (7.1) to expose the surface.

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. The area of interest should be representative of a normal surface, avoiding gross deformities.

9.5 Orient the sample (with respect to working distance, sample tilt, etc) to maximize X-ray collection efficiency of the EDX detector.

9.6 Adjust accelerating voltage to provide maximum excitation for the element of interest. Typically, this is 20 KeV for all elements having an atomic number greater than or equal to eleven (the atomic number of sodium) and 10 KeV for those elements with atomic numbers between boron and sodium.

9.7 Collect X-ray signals for a minimum of 100 s from a control area.

9.8 Move sample to that area showing surface anomaly and acquire X-ray signals for a minimum of 100 s.

9.9 Identify peaks and label the spectrum appropriately.

9.10 Print or plot the spectrum (see Fig. 1).

9.11 Photograph the surface anomaly at a magnification best suited to document the anomaly's physical characteristics.

9.12 Repeat 9.5 – 9.11 for all areas of interest.

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

10. Report

10.1 Report the following information:

10.1.1 *Data Analysis*— The first sample data collection must be taken from a selected site that is representative of the best normal surface available for the sample.

10.1.2 *Data Presentation:*

10.1.2.1 All elements not of the base metal will be considered unusual and shall be listed in tabular form with the number of particles demonstrating the presence of those elements being recorded.

10.1.2.2 Data shall be presented in the form of linear-linear printed or plotted spectra (see Fig. 1). The plotted spectral scale shall be such that the smallest peak can easily be discerned. If peak height differences are such that adequate representation of all peaks cannot be made from the same plot, two spectral plots shall be made using different scale factors.