

**SEMASPEC Test Method for
Metallurgical Analysis for Gas
Distribution System Components**

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Abstract: This SEMASPEC defines a method for determining the elemental composition and metallurgical characteristics of metal used to fabricate components for high purity gas distribution systems in the semiconductor industry. The composition and metallurgy of stainless steel may be expected to affect properties of importance to this application including surface roughness, incidence of surface defects, passivation, corrosion resistance, and welding. This procedure is to evaluate components considered for use in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

Keywords: Metallurgy, Defect Sources, Facilities, Gas Distribution Systems, Specifications, Components, Component Testing

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1. Introduction

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

1.1 Purpose

1.1.1 The purpose of this document is to define a method 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.

1.1.2 This document establishes a method for determining the elemental composition and metallurgical characteristics of metal used to fabricate components for high purity gas distribution systems in the semiconductor industry. The composition and metallurgy of stainless steel may be expected to affect properties of importance to this application, including surface roughness, incidence of surface defects, passivation, corrosion resistance, and welding.

1.2 Scope

1.2.1 This method applies to corrosion resistant metallic alloys of the general class stainless steel, containing chromium, nickel, and possibly molybdenum, and manganese and silicon as major alloying additions that are qualified or specified for the materials of components used in high purity gas supply systems for the semiconductor industry. The method is primarily intended for testing to determine conformance to applicable composition and metallurgical specifications as stated in supplier product specifications and/or customer purchase specifications.

1.2.2 Elements to be analyzed and reported are: 1) the alloying additions chromium, nickel and molybdenum (if specified in alloy, as in type 316L), 2) the minor elements and residuals manganese, silicon, copper, cobalt, and stabilizers such as titanium and columbium (niobium), if present, 3) carbon, sulfur and phosphorus, 4) nitrogen and oxygen gases, and 5) any additional minor element additions that may be made as part of the melting and casting practice, such as aluminum and calcium. Available standard analytical and reporting techniques are described for these elements.

1.2.3 Metallurgical characteristics to be analyzed and reported are 1) inclusion contents, 2) grain structure, 3) mechanical properties, and 4) intergranular corrosion susceptibility.

1.3 Limitations

1.3.1 This method is limited to corrosion resistant metal alloys of the general class stated in Section 1.2.

1.3.2 The methods incorporated in this document are not intended to preclude the use of other generally accepted techniques of demonstrated equivalent or superior precision and accuracy.

1.3.3 Inclusion of testing and analysis procedures for any given element or metallurgical characteristic in this document is not to be construed as being a requirement for incorporation of that element or metallurgical characteristic into specifications.

2. Referenced Documents¹

2.1 *General References*

ASTM A479/A479M-88a Standard Specification for Stainless and Heat-Resisting Steel Bars and Shapes for Use in Boilers and Other Pressure Vessels, 1988.

ASTM A484/A484M Rev A-88 Standard Specification for General Requirements for Stainless and Heat-Resisting Steel Bars, Billets, and Forgings, 1988.

ASTM E29-88 Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications, 1988.

2.2 *Chemical Analysis*

ASTM A751-88a Standard Methods, Practices, and Definitions for Chemical Analysis of Steel Products, 1988.

ASTM E353-88a Standard Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys, 1988.

ASTM E572-88 Standard Test Method for X-Ray Emission Spectrometric Analysis of Stainless Steel, 1988.

ASTM E1019-88 Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Steel and in Iron, Nickel, and Cobalt Alloys, 1988.

ASTM E1086-85 Standard Method for Optical Emission Vacuum Spectrometric Analysis of Stainless Steel by the Point-to-Plane Excitation Technique, 1985.

ASTM E1282-89 Standard Guide for Specifying the Chemical Compositions and Selecting Sampling Practices and Quantitative Analysis Methods for Metals and Alloys, 1989.

2.3 *Inclusion Determination*

ASTM E45-87 Standard Practice for Determining the Inclusion Content of Steel, 1987.

ASTM E1122-86 Standard Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis, 1986.

ASTM E1245-89 Standard Practice for Determining Inclusion Content of Steel and Other Metals by Automatic Image Analysis, 1989.

2.4 *Grain Structure Determination*

ASTM E112-88 Standard Test Methods for Determining Average Grain Size, 1988.

ASTM E1382-91 Standard Test Methods for Determining the Average Grain Size Using Semiautomatic and Automatic Image Analysis, 1991.

¹ American Society for Testing and Materials, 1916 Race St. Philadelphia, PA 19103.

2.5 *Mechanical Properties*

ASTM A370–88a Standard Test Methods and Definitions for Mechanical Testing of Steel Products, 1988.

ASTM E8–89 Standard Test Methods of Tension Testing of Metallic Materials, 1989.

2.6 *Susceptibility to Intergranular Corrosion*

ASTM A262–86 Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels, 1986.

3. **Terminology**

3.1 *heat analysis*—chemical analysis of the heat of stainless steel determined by analyzing a sample obtained during the pouring of the heat for the elements designated in a specification.

3.2 *inclusion*—discrete second phases (oxides, sulfides, carbides, inter-metallic compounds) that are distributed in the metal matrix.

3.3 *verification analysis*—chemical analysis of a semifinished or finished product for the purpose of determining conformance to applicable specifications.

4. **Test Protocol**

4.1 *Test Conditions*—Observed or calculated values shall be rounded to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM Recommended Practice E29–88.

4.1.1 Precautions

4.1.1.1 This test method may involve hazardous materials, operations, and equipment. This test method does not purport to address the safety considerations associated with its use.

It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations before using this method.

4.2 *Apparatus*—Materials and instrumentation are specified in the ASTM methods utilized.

4.3 *Test Procedures*—The general requirements of ASTM Standard Specification A484/A484M Rev A–88 for verification analysis, sampling, and test methods shall prevail.

ASTM Standard Specification A479/A479M–88a shall prevail for the determination of conformance of test results to ASTM standard requirements.

4.3.1 Chemical Analysis

4.3.1.1 The definitions, reference methods, practices, and reporting related to the chemical analysis of stainless steel alloys for this application shall be per ASTM Standard A 751.

- 4.3.1.2 The following wet chemical test methods may be used as control test methods and as the basis for standardizing instrumental analysis techniques, as described in ASTM Standard Test Method E 353:

<u>Element</u>	<u>Concentration Range, Percent</u>	<u>Sections</u>
Chromium	0.10 to 35.00	212 to 220
Nickel	10.1 to 48.00	172 to 179
Molybdenum	1.5 to 7.0	242 to 249
Manganese	0.01 to 5.00	8 to 17
Silicon	0.05 to 4.00	46 to 52
Copper	0.01 to 5.00	82 to 89
Copper	0.01 to 5.00	109 to 118
Cobalt	0.01 to 5.00	61 to 70
Titanium	0.01 to 0.35	231 to 241
Sulfur	0.005 to 0.50	37 to 45
Phosphorus	0.002 to 0.35	18 to 29
Phosphorus	0.02 to 0.35	164 to 171
Aluminum	0.003 to 0.20	71 to 81

Apparatus, test procedures, and data analysis are described in the appropriate sections of the ASTM Standard Test Method E 353.

- 4.3.1.3 The method of optical emission vacuum spectrometric analysis per ASTM Standard Method E 1086 may be used for heat analysis or verification analysis of stainless steel samples that can be prepared with a flat surface of 13-mm (0.5-in.) minimum diameter. This method provides analysis of the following elements in the concentration ranges shown:

<u>Element</u>	<u>Concentration Range, Percent</u>
Chromium	17.0 to 23.0
Nickel	7.5 to 13.0
Molybdenum	0.01 to 3.0
Manganese	0.01 to 2.0
Silicon	0.01 to 0.90
Copper	0.01 to 0.30
Carbon	0.005 to 0.25
Phosphorus	0.003 to 0.15
Sulfur	0.003 to 0.065

Apparatus, test procedures, and data analysis are described in the appropriate sections of the ASTM Standard Method E 1086.

- 4.3.1.4 The method of X-ray emission spectrometric analysis per ASTM Standard Test Method E 572 may be used for heat analysis or verification analysis of stainless steel. This method provides for the analysis of the following elements in the concentration ranges shown:

<u>Element</u>	<u>Concentration Range, Percent</u>
Chromium	11.0 to 19.0
Nickel	0.20 to 13.0
Molybdenum	0.05 to 3.00
Manganese	0.40 to 2.00
Copper	0.05 to 3.50
Cobalt	0.05 to 0.50
Columbium (Niobium)	0.30 to 0.70

Apparatus, test procedures, and data analysis are described in the appropriate sections of the ASTM Standard Test Method E 572.

- 4.3.1.5 The following test methods may be used for heat analysis and verification analysis for the determination of carbon, sulfur, nitrogen, and oxygen in stainless steel, as described in ASTM Standard Test Method E 1019:

<u>Element</u>	<u>Concentration Range, Percent</u>	<u>Sections</u>
Carbon	0.005 to 4.5	9 to 19
Sulfur	0.001 to 0.010	20 to 30
Nitrogen	0.001 to 0.200	31 to 41
Oxygen	0.001 to 0.005	42 to 53

Apparatus, test procedures, and data analysis are described in the appropriate sections of the ASTM Standard Test Method E 1019.

- 4.3.1.6 The development of specifications for compositional requirements and the identification of appropriate sampling and quantitative analysis methodologies to be referenced in product specifications shall be guided by ASTM Standard Guide E 1282.

- 4.3.1.7 An example of data for chemical analysis is provided in Table 1.

4.3.2 Inclusion Contents

- 4.3.2.1 The extent and types of inclusions that form as a result of deoxidation practice or limited solubility in solid steel shall be determined by Method A, Plate III, of ASTM Standard Practice E 45, for the four categories of inclusions addressed. Alternatively, inclusion ratings may be obtained by automatic image analysis techniques per ASTM Standard Practice E 1122. Inclusions such as carbides, nitrides, carbonitrides, borides, and intermetallic phases may not be rated using these methods.

- 4.3.2.2 Characterization of the amount, number, size, and spacing of any discrete second phase in stainless steel may be performed by automatic image analysis per ASTM Standard Practice E 1245.

- 4.3.2.3 The definitions, apparatus, methods, practices, and reporting relating to the determination of inclusion contents in stainless steel alloys for this application shall be per the ASTM Standard Practice used.
- 4.3.2.4 An example of data for inclusion contents is provided in Table 2.
- 4.3.3 Grain Structure
 - 4.3.3.1 Estimation of the average grain size in stainless steel shall be performed per ASTM Standard Test Method E 112. The definitions, apparatus, methods, practices, and reporting stated in this ASTM Standard Test Method shall apply.
 - 4.3.3.2 Alternatively, the average grain size may be determined by automatic image analysis per ASTM Standard Test Method E 1382.
 - 4.3.3.3 An example of data for grain structure is provided in Table 3.
- 4.3.4 Mechanical Properties
 - 4.3.4.1 The definitions, practices, and reporting of the mechanical properties of stainless steel alloys for this application shall be per ASTM Standard Test Methods and Definitions A 370.
 - 4.3.4.2 The tension test properties (tensile strength, yield strength, elongation, and reduction of area) of metals for this application shall be determined per ASTM Standard Test Methods E 8. Apparatus, test procedures, and data analysis are described in the appropriate sections of this ASTM Standard Test Method.
 - 4.3.4.3 An example of data for mechanical properties is provided Table 4.
- 4.3.5 Susceptibility to Intergranular Corrosion
 - 4.3.5.1 Susceptibility to intergranular attack due to precipitation of chromium carbides at the grain boundaries shall be detected by the test methods of ASTM Standard Practices A262. Apparatus, test procedures, and data analysis are described in the appropriate sections of this ASTM Standard.
 - 4.3.5.2 Samples shall be screened for susceptibility to intergranular attacks by use of ASTM A262, Practice A, Oxalic Acid Etch Test for Classification of Etch Structures of Austenitic Stainless Steels. Etch structures classified as "Step" or "Dual" shall be acceptable.
 - 4.3.5.3 Etch structures classified as "Ditch Structures" per ASTM A262, Practice A, shall be subjected to further testing, per Practice E, to determine susceptibility to intergranular attack. Acceptance criteria will be per Practice E. Results shall be reported.
- 4.4 *Data Analysis*

Refer to the appropriate ASTM standard referenced above.

5. Illustrations

Table 1 Chemical Analysis

Analyzed composition in weight percent:

Cr	17.34
Ni	11.69
Mo	2.13
Mn	1.62
Si	0.42
Cu	0.10
Co	0.05
Ti	n/a
Cb	n/a
C	0.018
S	0.009
P	0.023
N	0.040
O	0.002
Al	0.005
Ca	n/a

Composition meets the chemical requirements for S31603 (Type 316L) stainless steel per ASTM Standardized Specifications A 479.

Table 2 Inclusion Contents

Rating per ASTM E 45, Method A, Plate III, sampled from billets:

<u>Sample</u>	Inclusion Type							
	Type A		Type B		Type C		Type D	
	<u>Thin</u>	<u>Heavy</u>	<u>Thin</u>	<u>Heavy</u>	<u>Thin</u>	<u>Heavy</u>	<u>Thin</u>	<u>Heavy</u>
1	1	0.5	0.5	0	0	0	0.5	0
2	1.5	0.5	0	0	0.5	0	0	0
3	1	0	0.5	0	0	0	0.5	0
4	1	0.5	0.5	0	0	0	0	0

Table 3 Grain Structure

Average grain size number per ASTM E 112: 5 to 6.

Table 4 Mechanical Properties

Yield Strength (0.2% offset)	117.0 ksi	118.0 ksi
Tensile Strength	131.5 ksi	133.0 ksi
Elongation in 2.00 in.	32%	31%
Reduction of Area	72%	74%

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