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Standard Guide for Metallurgical Analysis for Gas Distribution System Components [1]

This standard is issued under the fixed designation F 1376; 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 guide 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 guide covers corrosion resistant metallic alloys of the general class stainless steel, containing chromium, nickel, manganese, and silicon as major alloying additions and possibly molybdenum, that are qualified or specified for the materials of components used in high-purity gas supply systems for the semiconductor industry. This guide is primarily intended for testing to determine conformance to applicable composition and metallurgical specifications as stated in supplier product specifications or customer purchase specifications, or both.

1.2 Elements analyzed and reported in this guide are as follows:

1.2.1 The alloying additions chromium, nickel, and molybdenum (if specified in alloy, as in type 316L),

1.2.2 The minor elements and residuals manganese, silicon, copper, cobalt, and stabilizers such as titanium and columbium (niobium), if present,

1.2.3 Carbon, sulfur and phosphorus,

1.2.4 Nitrogen and oxygen gases,

1.2.5 Any additional minor element additions that may be made as part of the melting and casting practice, such as aluminum and calcium,

1.2.6 Available standard analytical and reporting techniques are described for these elements.

1.3 Metallurgical characteristics to be analyzed and reported are inclusion contents, grain structure, mechanical properties, and intergranular corrosion susceptibility.

1.4 *Limitations*:

1.4.1 This guide is limited to corrosion resistant metal alloys of the general class stated in the Scope.

1.4.2 The test methods cited in this guide are not intended to preclude the use of other generally accepted techniques of demonstrated equivalent or superior precision and bias.

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

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

1.6 *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.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

A 262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels

A 370 Test Methods and Definitions for Mechanical Testing of Steel Products

A 479/A 479M Specification for Stainless and Heat-Resisting Steel Bars and Shapes for Use in Boilers and Other Pressure Vessels

A 484/A 484M Specification for General Requirements for Stainless and Heat-Resisting Steel Bars, Billets, and Forgings [2]

A 751 Test Methods, Practices, and Terminology for Chemical Analysis of Steel Products

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

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[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.

E 8 Test Methods of Tension Testing of Metallic Materials
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E 45 Test Methods for Determining the Inclusion Content of Steel
E 112 Test Methods for Determining the Average Grain Size
E 353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys
E 572 Test Method for X-Ray Emission Spectrometric Analysis of Stainless Steel
E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Steel and in Iron, Nickel, and Cobalt Alloys
E 1086 Test Method for Optical Emission Vacuum Spectrometric Analysis of Stainless Steel by the Point-to-Plane Excitation Technique
E 1122 Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis
E 1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
E 1282 Guide for Specifying the Chemical Compositions and Selecting Sampling Practices and Quantitative Analysis Methods for Metals, Ores, and Related Materials
E 1382 Test Methods for Determining the Average Grain Size Using Semiautomatic and Automatic Image Analysis

3. Terminology

3.1 *Definitions:*

3.1.1 *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.

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

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

4. Significance and Use

4.1 This guide defines a procedure for testing components being considered for installation into a high-purity gas distribution system. Application of this guide is expected to yield comparable data among components tested for purposes of qualification for this installation.

4.2 This guide establishes a procedure 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.

5. Materials and Manufacture

5.1 Materials and instrumentation are specified in the ASTM test methods utilized in this guide.

6. Procedure

6.1 The general requirements of Specification A 484/A 484M prevail for verification analysis, sampling, and test methods.

6.2 Specification A 479/A 479M prevails for the determination of conformance of test results to ASTM standard requirements.

6.3 *Chemical Analysis*:

6.3.1 The definitions, reference methods, practices, and reporting related to the chemical analysis of stainless steel alloys for this application shall be in accordance with Test Methods A 751.

6.3.2 Use the following wet chemical test methods as control test methods and as the basis for standardizing instrumental analysis techniques, in accordance with Test Method E 353:

Element	Concentration Range, %	Test Method E353, Sections
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

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

6.3.3 Use the method of optical emission vacuum spectrometric analysis in accordance with Method E 1086 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 test method provides analysis of the following elements in the concentration ranges shown:

Element	Concentration Range,%
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

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

6.3.4 Use the method of X-ray emission spectrometric analysis in accordance with Test Method E 572 for heat analysis or verification analysis of stainless steel. This test method provides for the analysis of the following elements in the concentration ranges shown:

Element	Concentration Range,%
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

Element	Concentration Range,%
Columbium (Niobium)	0.30 to 0.70

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

6.3.5 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, in accordance with Test Method E 1019.

Element	Concentration Range, %	Test Method E1019, Sections
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

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

6.3.6 Guide the development of specifications for compositional requirements and the identification of appropriate sampling and quantitative analysis methodologies referenced in product specifications in accordance with Guide E 1282.

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

6.4 *Inclusion Contents*:

6.4.1 Determine the extent and types of inclusions that form as a result of deoxidation practice or limited solubility in solid steel by Method A, Plate III, of Practice E 45, for the four categories of inclusions addressed. Alternatively, obtain inclusion ratings by automatic image analysis techniques in accordance with Practice E 1122. Do not rate inclusions such as carbides, nitrides, carbonitrides, borides, and intermetallic phases using these methods.

6.4.2 Perform characterization of the amount, number, size, and spacing of any discrete second phase in stainless steel by automatic image analysis in accordance with Practice E 1245.

6.4.3 The definitions, apparatus, methods, practices, and reporting relating to the determination of inclusion contents in stainless steel alloys for this application are in accordance with the ASTM practice used (see 6.4.1 and 6.4.2).

6.4.4 An example of data for inclusion contents is provided in Table 2.

6.5 *Grain Size*:

TABLE 1 Chemical Analysis [A]

Analyzed composition in weight, %	
Chromium	17.34
Nickel	11.69
Molybdenum	2.13
Manganese	1.62
Silicon	0.42
Copper	0.10
Cobalt	0.05
Titanium	...
Columbium	...
Carbon	0.018
Sulfur	0.009
Phosphorus	0.023
Nitrogen	0.040
Oxygen	0.002
Aluminum	0.005
Calcium	...

[A] Composition meets the chemical requirements for S31603 (Type 316L) stainless steel in accordance with Specification A 479.

TABLE 2 Inclusion Contents

	Inclusion Type[A]							
	Type A		Type B		Type C		Type D	
Sample	Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
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

[A] Rating in accordance with Practice E 45, Method A, Plate III, sampled from billets.

6.5.1 Perform estimation of the average grain size in stainless steel in accordance with Test Methods E 112. The definitions, apparatus, methods, practices, and reporting stated in this test method apply.

6.5.2 Alternatively, the average grain size may be determined by automatic image analysis in accordance with Test Method E 1382.

6.5.3 An example of data for grain size is provided in Table 3.

6.6 *Mechanical Properties*:

6.6.1 The definitions, practices, and reporting of the mechanical properties of stainless steel alloys for this application are in accordance with Test Methods and Definitions A 370.

6.6.2 The tension test properties (tensile strength, yield strength, elongation and reduction of area) of metals for this application are determined in accordance with Test Methods E 8. Apparatus, test procedures, and data analysis are described in the appropriate sections of this test method.

6.6.3 An example of data for mechanical properties is provided in Table 4.

6.7 *Susceptibility to Intergranular Corrosion*:

6.7.1 Susceptibility to intergranular attack due to precipitation of chromium carbides at the grain boundaries is detected by the test methods of Practices A 262. Apparatus, test procedures and data analysis are described in the appropriate sections of Practices A 262.

6.7.2 Screen samples for susceptibility to intergranular attacks by use of Practices A 262, Practice A. Etch structures classified as“ Step” or “Dual” shall be acceptable.

6.7.3 Subject etch structures classified as “Ditch Structures” as described in Practices A 262, Practice A, must be further tested in accordance with Practices A 262, Practice E, to determine susceptibility to intergranular attack. Acceptance criteria is in accordance with Practice E. Report results.

7. Keywords

7.1 alloy composition; components; composition; contamination; corrosion; gas distribution components; metallurgical analysis; metals; semiconductor processing; stainless steel

TABLE 3 Grain Structure

Average grain size number in accordance with Test Methods 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 in.), %	32	31
Reduction of area, %	72	74