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Standard Guide for Analysis and Reporting the Impurity Content and Grade of High Purity Metallic Sputtering Targets for Electronic Thin Film Applications[1]

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1. Scope

1.1 This guide covers sputtering targets used as thin film source material in fabricating semiconductor electronic devices. It should be used to develop target specifications for specific materials and should be referenced therein.

1.2 This standard sets purity grade levels, analytical methods and impurity content reporting method and format.

1.2.1 The grade designation is a measure of total metallic impurity content. The grade designation does not necessarily indicate suitability for a particular application because factors other than total metallic impurity may influence performance.

2. Referenced Documents

2.1 *ASTM Standards:*

F 1593 Test Method for Trace Metallic Impurities in Electronic Grade Aluminum by High Mass-Resolution Glow-Discharge Mass Spectrometer[2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *finished product, n—for the purpose of this standard*, a "finished product" is a manufactured sputtering target, ready for use.

3.1.2 *material lot, n—for the purpose of this standard*, a "lot" is material consolidated into one ingot, and processed as one continuous batch in subsequent thermal-mechanical treatments.

3.1.3 *target specification, n—for the purpose of this standard*, a specification for a sputtering target source material for electronic thin film applications.

3.2 *Abbreviations:*

3.2.1 *mdl*—minimum detection limit

4. Impurities

4.1 The minimum set of metallic impurity elements to be analyzed shall be developed and listed in the target specification or agreed upon by the purchaser and supplier.

4.2 Acceptable analysis methods and detection limits are to be specified in the target specification. Elements not detected will be counted and reported as present at the minimum detection limit ("mdl") for the method used. Additional elements may be analyzed and reported as agreed upon between the purchaser and the supplier, but these elements shall not be counted in defining the grade designation.

4.3 Certain elements may present particular analysis problems, such as interferences. The limits, analysis method, and mdl may, in such cases, be as agreed upon between the purchaser and the supplier.

4.4 Nonmetallic elements, which shall be analyzed and reported, are carbon, hydrogen, nitrogen, oxygen, and sulfur. Maximum limits for nonmetallic impurities shall be agreed upon between the purchaser and the supplier.

4.5 Acceptable limits and analytical techniques for particular elements in critical applications may be agreed upon between the purchaser and the supplier.

5. Classification

5.1 Grades of metallic sputtering targets are defined in Table 1, based upon total metallic impurity content of the set of elements as specified in 4.1. Impurity contents are reported in parts per million by weight (wt ppm).

5.2 Purity grade and total metallic impurity levels are based upon the set of elements as specified in 4.1.

6. Sampling

6.1 Analysis for impurities and gases shall be performed on samples that represent the finished sputtering target.

6.1.1 Unless otherwise agreed upon between the purchaser and the supplier, impurity analyses for metallic and nonmetallic impurities shall be made by the supplier for one or more sample specimens that are representative of the production lot. If more than one sample specimen is used, the results of the analyses shall be averaged to establish conformance with the grade designation (5.1), other metallic impurity limits (4.2, 4.3, and 4.5), and the agreed upon limits for nonmetallic content (4.4).

7. Analytical Methods

7.1 Analysis for impurities listed as specified in Section 4 shall be performed using methods, techniques and mdls appropriate to the material, grade and special agreements (4.3-4.5):

[1] This guide is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.17 on Sputtered Thin Films.

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[2] *Annual Book of ASTM Standards*, Vol 10.04.

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TABLE 1 Metallic Sputtering Target Grades

Grade	Purity, %	Maximum Total Metallic Impurity Content, wt ppm
4N	99.99	100
4N5	99.995	50
5N	99.999	10
5N5	99.9995	5
6N	99.9999	1

7.1.1 *Trace Metallic Impurities*—The target specification should state the preferred method of analysis. Mdl should be <0.01 wt. ppm for 6N grade, < 0.1 ppm for 5N5 and 5N grade, and < 1 wt. ppm for 4N5 and 4N grade. A discussion of detection limit determination is given in Test Method F 1593 or in Currie.[3]

7.1.2 *Carbon, Oxygen, Sulfur*—By fusion and gas extraction/infrared spectroscopy[4] with mdl typically ≤ 10 weight ppm.

7.1.3 *Nitrogen*—By fusion and gas extraction with mdl typically ≤ 10 weight ppm.

7.1.4 *Hydrogen*—By fusion and gas extraction with mdl typically ≤ 3 weight ppm.

7.1.5 Other analytical techniques may be used provided they can be proved equivalent to the methods specified and have sum of mdl for the list defined in 4.1 less than or equal to one tenth (0.1) of the maximum impurity content listed in Table 1 (for a given grade).

[3] Currie, L. A., "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, Vol 40, 1968, pp 586-593.

[4] Analytical equipment manufactured by Leco Corporation, St. Joseph, MI has been found satisfactory for making fusion and gas extraction analyses for carbon, oxygen, sulfur, nitrogen and hydrogen at the required mdl's.

8. Certification

8.1 When required by the purchaser, the supplier shall provide a certificate of analysis/compliance that documents the finished target.

8.2 The certificate of analysis/compliance shall state the manufacturer's or supplier's name, the supplier's lot number, the grade level (Section 5), impurity levels (Section 4), method of analysis with mdl for each element (Section 7), and any other information as agreed upon between the purchaser and the supplier.

8.2.1 *Impurities Reporting Option 1*—If agreed upon between the purchaser and the supplier, impurity levels may be reported using actual analytical results for the material lot from which the sputtering target is made (11.1.1). All impurity levels, except thorium and uranium, shall be reported in weight ppm. Thorium and uranium are generally controlled at very low levels in material for electronic thin film applications and may be reported in parts per billion by weight (weight ppb). Non-detected trace impurities (from the list as specified in 4.1) shall be reported as present at the mdl concentration (Section 7).

8.2.2 *Impurities Reporting Option 2*—If agreed upon between the purchaser and the supplier, impurity levels may be reported by citing typical results based upon historical data for the same process.

9. Keywords

9.1 electronics; purity analysis; purity grade; sputtering; target; thin film

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