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Standard Specification for High Purity Titanium Sputtering Targets for Electronic Thin Film Applications[1]

This standard is issued under the fixed designation F 1709; 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.

1. Scope

1.1 This specification covers pure titanium sputtering targets used as a raw material in fabricating semiconductor electronic devices.

1.2 This standard sets purity grade levels, physical attributes, analytical methods, and packaging.

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:*

E 112 Test Methods for Determining Average Grain Size [2]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *finished product, n—for the purposes 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 melted into one ingot, and processed as one continuous batch in subsequent thermal-mechanical treatments.

4. Classification

4.1 Grades of titanium sputtering targets are defined in Table 1, based upon total metallic impurity content of the elements listed in Table 2. Impurity contents are reported in parts per million by weight (wt ppm). Higher purity grades, for example "5N5" and" 6N", may be provided, as agreed upon between the purchaser and the supplier.

4.2 Purity grade and total metallic impurity levels are based upon the suite of elements listed in Table 2.

TABLE 1 Titanium Sputtering Target Grades

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

5. Ordering Information

5.1 Orders for pure titanium sputtering targets shall include the following:

5.1.1 Grade (see 4.1),

5.1.2 Special requirements concerning impurities, if required (see 6.1, 6.2, 6.3, 6.4),

5.1.3 Grain size, if required (Section 7),

5.1.4 Configuration (Section 8),

5.1.5 Certification required (Section 12), and

5.1.6 Whether or not a sample representative of the finished product is required to be provided by the supplier to the purchaser.

6. Impurities

6.1 The minimum suite of metallic impurity elements to be analyzed is defined in Table 2. Acceptable analysis methods and detection limits are specified in Section 11. Elements not detected will be counted and reported as present at the minimum detection limit ("mdl"). Additional elements may be analyzed and reported as agreed upon between the purchaser and the supplier, but these shall not be counted in defining the grade designation.

6.2 Cesium, chlorine, phosphorus, and tantalum present particular analysis problems. The limits, analysis method, and mdl may be as agreed upon between the purchaser and the supplier.

TABLE 2 Suite of Metallic Elements to be Analyzed and Reported

aluminum	iron	silicon	vanadium
chromium	lead	silver	zinc
cobalt	lithium	potassium	sodium
zirconium	copper	magnesium	thorium
boron	manganese	tin	molybdenum
tungsten	nickel	uranium	

[1] This specification is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.17 on Sputter Metallization.

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[2] *Annual Book of ASTM Standards,* Vol 03.01.

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

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

7. Grain Size

7.1 The average and the maximum grain size shall be as agreed upon between the purchaser and the supplier.

7.2 Average grain size shall be measured and reported in accordance with Test Methods E 112, or other equivalent method.

7.3 Maximum grain size shall be established by making an optical or scanning electron micrograph of a polished and etched specimen typical of the finished product. The magnification must be calibrated to ± 10 % of nominal using an appropriate gage. At least 50 grains must be resolved in the micrograph. The maximum grain size is the diagonal measure of the largest titanium crystal visible in the field of view, divided by the magnification.

7.4 Average grain size and maximum grain size can alternatively be established using computer-assisted image analysis methods. If image analysis methods are used, then the average grain size is defined as the mean value obtained from the grain diameter distribution data. The maximum grain size is defined as the largest grain diameter recorded in the grain size distribution data set. At least 50 grains must be included in the image analysis data set.

8. Configuration

8.1 Each product shall conform to an appropriate engineering drawing, agreed upon between the purchaser and the supplier.

8.2 Nominal dimensions, tolerances, and other attributes shall be agreed upon between the purchaser and the supplier.

9. Workmanship, Finish, Appearance

9.1 Workmanship, finish, and appearance shall be agreed upon between the purchaser and the supplier.

9.2 Surfaces must be free of any contaminates such as dirt or oils that could adversely effect the performance of the material, as agreed upon between the purchaser and the supplier.

10. Sampling

10.1 Analysis for impurities and gasses shall be performed on samples that are representative of the finished sputtering target.

10.1.1 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. These data shall be averaged to establish conformance with the grade designation (4.1), other metallic impurity limits (6.1, 6.2, 6.4), and the agreed upon limits for nonmetallic content (6.3).

11. Analytical Methods

11.1 Analysis for impurities listed in Section 6 and Table 2 shall be performed as follows:

11.1.1 *Trace Metallic Impurities*—By glow discharge mass spectrometer (GDMS) with a nominal mdl ≤ 0.05 weight ppm.

NOTE 1—The mdl for some metallic species by GDMS is >0.05 weight ppm, as limited by interferences.

11.1.2 *Carbon, Oxygen, Sulfur*—By fusion and gas extraction/infrared spectroscopy [3] with an mdl of ≤10 weight ppm.

11.1.3 *Nitrogen*—By fusion and gas extraction with an mdl of ≤10 weight ppm.

11.1.4 *Hydrogen*—By fusion and gas extraction with an mdl of ≤3 weight ppm.

11.1.5 Other analytical techniques may be used provided they can be proved equivalent to the methods specified and have mdl less than or equal to the specified methods.

12. Certification

12.1 When required by the purchaser a certificate of analysis/compliance that documents the finished target shall be provided by the supplier.

12.2 The certificate of analysis/compliance shall state the manufacturer's or supplier's name, the supplier's lot number, the grade level (Section 4), impurity levels (Section 6), method of analysis (Section 11), and any other information as agreed upon between the purchaser and the supplier.

12.2.1 *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 (10.1.1). All impurity levels, except thorium and uranium, shall be reported in weight ppm. Thorium and uranium are generally controlled at very low levels in this material and may be reported in parts per billion by weight (weight ppb). Nondetected trace impurities (Section 6) shall be reported as present at the mdl concentration (Section 11).

12.2.2 *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.

13. Packaging

13.1 Each piece shall be enclosed in packaging that insures freedom from contamination in handling and shipping. Each piece shall be packed for shipment in a shipping container that insures product integrity during transport.

14. Keywords

14.1 coating; sputtering; target; thin film; titanium

[3] 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.