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Standard Test Method for Trace Metallic Impurities in Electronic Grade Aluminum-Copper, Aluminum-Silicon, and Aluminum-Copper-Silicon Alloys by High-Mass-Resolution Glow Discharge Mass Spectrometer[1]

This standard is issued under the fixed designation F1845; 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 test method determines the concentrations of trace metallic impurities in high purity (99.99 wt. % pure, or purer, with respect to metallic trace impurities) aluminum-copper, aluminum-silicon and aluminum-copper-silicon alloys with major alloy constituents as follows:

aluminum	Greater than 95.0 %
copper	Less or equal than 5.0 %
silicon	Less or equal than 5.0 %

1.2 This test method pertains to analysis by magnetic-sector glow discharge mass spectrometer (GDMS).

1.3 This test method does not include all the information needed to complete GDMS analyses. Sophisticated computer-controlled laboratory equipment, skillfully used by an experienced operator, is required to achieve the required sensitivity. This test method does cover the particular factors (for example, specimen preparation, setting of relative sensitivity factors, determination of detection limits, etc.) known by the responsible technical committee to effect the reliability of high purity aluminum analyses.

1.4 *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]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E1593 Guide for Assessing the Efficacy of Air Care Products in Reducing the Perception of Indoor Malodor

3. Terminology

3.1 Terminology in this test method is consistent with Terminology E135. Required terminology specific to this test method, not covered in Terminology E135, is indicated in 3.2.

3.2 *Definitions:*

3.2.1 *campaign*—a test procedure to determine the accuracy of the instrument, which was normally performed at the beginning of the day or after the instrument modification, or both.

3.2.2 *reference sample*—material accepted as suitable for use as a calibration/sensitivity reference standard by all parties concerned with the analyses.

3.2.3 *specimen*—a suitably sized piece cut from a reference or test sample, prepared for installation in the GDMS ion source, and analyzed.

3.2.4 *test sample*—material (aluminum alloy) to be analyzed for trace metallic impurities by this GDMS method.

3.2.4.1 *Discussion*—Generally the test sample is extracted from a larger batch (lot, casting) of product and is intended to be representative of the batch.

4. Summary of Test Method

4.1 A specimen is mounted in a plasma discharge cell. Atoms subsequently sputtered from the specimen surface are ionized, and then focused as an ion beam through a double-focusing magnetic-sector mass separation apparatus. The mass spectrum (the ion current) is collected as magnetic field or acceleration voltage, (or both) is scanned.

4.2 The ion current of an isotope at mass M_i is the total measured current, less contributions from all other interfering sources. Portions of the measured current may originate from the ion detector alone (detector noise). Portions may be due to incompletely mass resolved ions of an isotope or molecule with mass close to, but not identical with, M_i. In all such instances

[1] This test method 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] 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.

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the interfering contributions must be estimated and subtracted from the measured signal.

4.2.1 If the source of interfering contributions to the measured ion current at M_i cannot be determined unambiguously, the measured current less the interfering contributions from identified sources constitutes an upper bound of the detection limit for the current due to the isotope.

4.3 The composition of the test specimen is calculated from the mass spectrum by applying a relative sensitivity factor (RSF(X/M)) for each contaminant element, *X*, compared to the matrix element, *M*. RSF's are determined in a separate analysis of a reference material performed under the same analytical conditions, source configuration, and operating protocol as for the test specimen.

4.4 The relative concentrations of elements *X* and *Y* are calculated from the relative isotopic ion currents $I(X_i)$ and $I(Y_j)$ in the mass spectrum, adjusted for the appropriate isotopic abundance factors ($A(X_i)$, $A(Y_j)$ and RSF's. $I(X_i)$ and $I(Y_j)$ refer to the measured ion current from isotopes X_i and Y_j, respectively, of atomic species *X* and *Y* as follows:

$$(X)/(Y) = RSF(X/M)/RSF(Y/M) \times A(Y_j)/A(X_i) \times I(X_i)/I(Y_j) \quad (1)$$

where $(X)/(Y)$ is the concentration ratio of atomic species *X* to species *Y*. If species *Y* is taken to be the aluminum matrix ($RSF(M/M) = 1.0$), (X) is (with only very small error for pure metal matrices) the absolute impurity concentration of *X*.

5. Significance and Use

5.1 This test method is intended for application in the semiconductor industry for evaluating the purity of materials (for example, sputtering targets, evaporation sources) used in thin film metallization processes. This test method may be useful in additional applications, not envisioned by the responsible technical committee, as agreed upon between the parties concerned.

5.2 This test method is intended for use by GDMS analysts in various laboratories for unifying the protocol and parameters for determining trace impurities in aluminum-copper, aluminum-silicon, and aluminum-copper-silicon alloys. The objective is to improve laboratory-to-laboratory agreement of analysis data. This test method is also directed to the users of GDMS analyses as an aid to understanding the determination method, and the significance and reliability of reported GDMS data.

5.3 For most metallic species the detection limit for routine analysis is on the order of 0.01 wt. ppm. With special precautions, detection limits to sub-ppb levels are possible.

5.4 This test method may be used as a referee method for producers and users of electronic-grade aluminum-copper, aluminum-silicon and aluminum-copper-silicon materials.

6. Apparatus

6.1 *Glow Discharge Mass Spectrometer* , with mass resolution greater than 3500, and associated equipment and supplies. The GDMS must be fitted with an ion source specimen cell that is cooled by liquid nitrogen, Peltier cooled, or cooled by an equivalent method.

6.2 *Machining Apparatus,* capable of preparing specimens and reference samples in the desired geometry and with smooth surfaces.

6.3 *Electro-Polishing Apparatus* , capable of removing the contaminants from the surfaces of specimens.

7. Reagents and Materials

7.1 *Reagents*—Reagent and high purity grade reagents as required (MeOH, HNO_3 and HCl).

7.2 *Demineralized Water.*

7.3 *Tantalum Reference Sample.*

7.4 *Aluminum Reference Sample.*

7.4.1 To the extent available, aluminum reference materials shall be used to produce the GDMS relative sensitivity factors for the various elements being determined (see Table 1).

7.4.1.1 As necessary, non-aluminum reference materials may be used to produce the GDMS relative sensitivity factors for the various elements being determined.

7.4.2 Reference materials should be homogeneous (see 11.1) and free of cracks or porosity.

7.4.3 At least two reference materials are required to establish the relative sensitivity factors, including a 99.9999 % pure aluminum metal to establish the background contribution in analyses.

7.4.4 The concentration of each analyte for relative sensitivity factor determination should be at a factor of 100 greater than the detection limit determined using a 99.9999 % pure aluminum specimen, but less than 100 ppmw.

7.4.5 To meet expected analysis precision, it is necessary that specimens of reference and test material present the same size and configuration (shape and exposed length) in the glow discharge ion source, with a tolerance of 0.2 mm in diameter and 0.5 mm in the distance of sample to cell ion exit slit.

8. Preparation of Reference Standards and Test Specimens

8.1 The surface of the parent material must not be included in the specimen.

8.2 The machined surface of the specimen must be cleaned by electropolishing or etching immediately prior to mounting the specimen and inserting it into the glow discharge ion source.

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TABLE 1 Suite of Impurity Elements to Be Analyzed[A]

NOTE 1—Establish RSFs for the following suite of elements:

silver	arsenic	gold	boron	beryllium	calcium	cerium	chromium	cesium	copper	iron
potassium	lithium	magnesium	manganese	sodium	nickel	phosphorus	antimony	silicon	tin	thorium
titanium	uranium	vanadium	zinc	zirconium						

[A] Additional species may be determined and reported, as agreed upon between all parties concerned with the analyses.