~~Designation:F1845–97 (Reapproved 2002)~~ Designation: F 1845 – 08

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 F 1845; 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]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

~~E180Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals~~

~~E673Terminology Relating to Surface Analysis[2]~~

~~E876Practice for Use of Statistics in the Evaluation of Spectrometric Data~~

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

~~F1594Specification for Pure Aluminum (Unalloyed) Source Material for Vacuum Coating Applications[5]~~ Guide for Assessing the Efficacy of Air Care Products in Reducing Sensorly Perceived Indoor Air Malodor Intensity

3. Terminology

3.1 Terminology in this test method is consistent with Terminology E 135. Required terminology specific to this test method, not covered in Terminology E 135, 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.

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[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 .

~~Current edition approved Dec. 10, 2002. Published May 2003. Originally approved in 1997. Last previous edition approved in 1997 as F1845–97.~~

Current edition approved June 15, 2008. Published July 2008. Originally approved in 1997. Last previous edition approved in 2002 as F 1845 – 97(02).

[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* ~~, Vol 03.05.~~volume information, refer to the standard's Document Summary page on the ASTM website.

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 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.~~ , 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 ~~(MeHO,~~(MeOH, HNO_3 ~~and HCl....)~~ 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.