



Designation: F 1724 – 96

Standard Test Method for Measuring Surface Metal Contamination of Polycrystalline Silicon by Acid Extraction-Atomic Absorption Spectroscopy¹

This standard is issued under the fixed designation F 1724; 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 covers the quantitative determination of surface trace metal contamination on the surface of polycrystalline silicon using an acid to extract the metals from the surface. The metals content of the acid is then analyzed by graphite furnace atomic-absorption spectroscopy.

1.2 This test method can be used for various rod, chunk, granule and chip sizes, for polycrystalline or single crystal silicon, to determine surface metal contaminants. Since the area of irregularly-shaped chunks, chips, or granules is difficult to measure accurately, values are based on sample weight. Using a sample weight of 300 g allows detection limits at the 0.1 ppbw (parts per billion weight) level.

1.3 The strength, composition, temperature, and exposure time of the acid determine the depth of surface etching and the efficiency of the extraction of the contaminants from the surface. Less than 1 % of the sample weight is removed in this test method.

1.4 This test method is useful for determining the alkali elements, alkali earth, and first series transition elements, such as sodium, potassium, calcium, iron, chromium, nickel, copper, zinc, as well as other elements such as aluminum. The recovery of these elements from the silicon surface is measured as greater than 90 %, using control standards intentionally added to the polysilicon surface.

1.5 This test method suggests a particular sample size, acid composition, etch cycle, testing environment, and instrument protocol. Variations in these parameters may be used, but may effect the recovery efficiency or retention of metals during processing. In practice, this test method is used for sample weights of 25 to 5000 g. For referee purposes, this test method specifies a sample weight of 300 g. This test method includes guidelines to alert the analyst to the interferences and resultant variations in this test method, and includes standard methods for quantifying and reporting these variations.

1.6 This test method specifies the use of graphite furnace atomic-absorption spectroscopy to analyze trace metals content of the acid extract. Other instruments of equivalent sensitivity,

such as inductively-coupled plasma/mass spectrometry, may be used.

1.7 The detection limit and method variation depend on the efficiency of the acid extraction procedure, sample size, the method interferences, the absorption spectrum of each element, and the instrumental sensitivity, background, and blank value.

1.8 This test method uses hot acid to etch away the surface of the silicon. The etchant is potentially harmful and must be handled in an acid exhaust fume hood, with utmost care at all times. Hydrofluoric acid solutions are particularly hazardous and should not be used by anyone who is not familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

1.9 *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.* Specific precautionary statements are given in Section 9.

2. Referenced Documents

2.1 ASTM Standards:

D 5127 Guide for Electronic Grade Water²

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality of a Lot or Process³

2.2 SEMI Standards:

C 7 Specification for Reagents⁴

2.3 Federal Standard:

209E Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones⁵

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *acid blank*—a sample of acid used to establish the background spectrum and trace metal contamination of the extraction acid used in the procedure.

3.1.2 *carousel protocol*—the order and function of

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 14.02.

⁴ Available from Semiconductor Equipment and Materials International, 805 E. Middlefield Road, Mountain View, CA 94043.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

¹ This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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