**Designation: F 1708 – 02**

# Standard Practice for
# Evaluation of Granular Polysilicon by Melter-Zoner Spectroscopies[1]

This standard is issued under the fixed designation F 1708; 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 practice describes a procedure to consolidate granular polysilicon into a solid rod and then to convert the polysilicon rod into a single crystal by a float-zone technique. The resultant single crystal ingot is used for the determination of trace impurities in the polysilicon. These impurities are acceptor and donor components (usually boron, aluminum, phosphorus, arsenic, and antimony) as well as substitutional carbon.

1.2 The useful range of impurity concentration covered by this practice is 0.002 to 100 parts per billion atomic (ppba) for acceptor and donor impurities, and 0.03 to 5 parts per million atomic (ppma) for carbon. The acceptor and donor impurities are analyzed in a slice taken from the single crystal ingot by photoluminescence or infrared spectroscopies. The carbon impurity is determined by analysis of a slice by infrared spectroscopy.

1.3 This practice is applicable only to evaluation of polysilicon granules as produced by thermal deposition of silane, or one of the chlorosilanes, onto high purity seeds of polysilicon in a continuous fluid bed reactor. The granules are near spherical in shape and range in size from 200 to 2500 µm with a mean size of about 900 µm.

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.* Specific hazard statements are given in Section 9 and in 12.1.1.

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 5127 Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry[2]
F 1241 Terminology of Silicon Technology[3]
F 1389 Test Methods for Photoluminescence Analysis of Single Crystal Silicon for III-V Impurities[3]
F 1391 Test Method for Substitutional Atomic Carbon Content of Silicon by Infrared Absorption[3]
F 1630 Test Method for Low Temperature FT-IR Analysis of Single Crystal Silicon for III-V Impurities[3]

2.2 *SEMI Standards:*

C3.14 Specification for Argon[4]
C28 Specifications and Guidelines for Hydrofluoric Acid[4]
C31 Specification for Methanol[4]
C34 Specification for Mixed Acid Etchants[4]
C35 Specifications and Guideline for Nitric Acid[4]

2.3 *Federal Standard:*

209E, Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones[5]

2.4 *ISO Standard:*

ISO 14644-1, Cleanrooms and Associated Controlled Environments C Part 1: Classification of Airborne Particulates[6]

## 3. Terminology

3.1 Most terms used in this practice are defined in Terminology F 1241.

3.2 *Definitions of Terms Specific to This Practice:*

3.2.1 *granular polysilicon, n*—nearly spherical, granules (200 to 2500 µm) of polysilicon as produced in a fluidized bed reactor.

3.2.2 *melter/zoner, n*—an apparatus designed to melt granular polysilicon to a solid rod and then convert the polycrystalline rod to a single crystal ingot by an rf coupled coil.

3.2.3 *PTFE*—an acronym for polytetrafluoroethylene, a chemically resistant polymer.

3.2.4 *silicon pedestal, n*—a piece of single crystal silicon cut from a high purity silicon ingot.

## 4. Summary of Practice

4.1 Granular polysilicon is converted into a single crystal silicon rod in a two-step procedure.

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[1] This practice is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved Jan. 10, 2002. Published March 2002. Originally published as F 1708 – 96. Last previous edition F 1708 – 96.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 10.05.

[4] Available from Semiconductor Equipment and Materials International, 3081 Zanker Rd., San Jose, CA 95134 (www.semi.org).

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

[6] ISO Central Secretariat, C.P. 56, CH-1211 Genève 20, Switzerland; available in the U.S. from American National Standards Institute, 25 West 43rd Street, 4th Floor, New York, NY 10036.

4.1.1 First, the silicon granules are consolidated into a polysilicon rod by melting fluidized granules into the molten (bottom) end of a silicon pedestal during a downward pass of the coil of the zone furnace. After about 12 g of polysilicon has been melted and cooled in the zone process, a polysilicon rod about 0.9 by 6 cm is obtained.

4.1.2 In the second step, a single crystal silicon seed is melted into the tail end of the polycrystalline rod and a single zone pass is done in the upward direction to level the impurities and to convert the silicon to a single crystal rod. This produces a single crystal silicon ingot about 0.9 cm in diameter by 5 cm in length from which a section is sliced for measurement of impurities. The entire consolidation and zoning requires about 30 min to accomplish.

4.1.3 A slice of 2 to 4-mm thick is taken from the center one-third of the single crystal silicon ingot for measurement of impurities by infrared or photoluminescence spectroscopies.

## 5. Significance and Use

5.1 Polycrystalline silicon is used as the starting material for growth of large single crystal ingots by the Czochralski methods. This procedure provides a means to determine the impurity levels in granular polysilicon to be used for crystal growth.

5.2 Although the Czochralski grown ingots are intentionally doped during crystal growth to the desired resistivity and type, the dopant levels in the polysilicon must be known to calculate the amount of dopant to be added.

5.3 Carbon levels in polysilicon must be known so that the concentration of carbon in the ingot can be controlled to a low concentration.

5.4 This practice has applicability in production control, quality assurance, materials research, and materials acceptance.

## 6. Interferences

6.1 The quartz tubes used in this procedure must be of high purity, especially in regards to the impurities to be measured. Boron is of particular concern since it is always present in quartz and may frequently appear in uncharacteristically high concentrations in the polysilicon.

6.2 All chemicals and gases used in this procedure must be free of components to be measured or they may give results extraneously high.

6.3 Loss of single crystal during the zone pass will produce an ingot that may give unsatisfactory results. The quality of the infrared or photoluminescence spectra usually reveals the lack of single crystal.

## 7. Apparatus

7.1 *Acid Exhaust Fume Hood*, to provide for exhaust of acid fumes, a clean air environment (ISO Class 6 minimum, as defined in ISO 14544-1), a drain for acids and water, and a deionized water supply. This hood provides for the cleaning of quartz containers, funnels, and tubes used in the melter/zoner as well as a place to etch silicon pieces and samples used in this practice.

NOTE 1—This class is about the same as Class 1000 as defined in Federal Standard 209E).

7.2 *Laminar Flow Hood*, to provide a flow of clean (ISO Class 6 minimum) air for drying of components and the etched and clean silicon pieces.

7.3 *Quartz Sample Containers*, 200 to 300-mL capacity quartz bottles to contain and transport granular polysilicon samples.

7.4 *Quartz Funnel*, a funnel of sufficient size to transfer granular polysilicon from the sample container to the 15-mm inside diameter quartz tube.

7.5 *Quartz Tube*, a section of high purity quartz tube with 18-mm outside diameter, 15-mm inside diameter, and a length of 55.6 cm. The inside diameter and outside diameter variances should be small to avoid problems with clearances both internally and externally. This tube provides the working enclosure for both the consolidation and the zone leveling within the confines of the working coil of the melter/float zone apparatus.

7.6 *Melter/Float Zone Apparatus*, a radio frequency (rf) generator operating between 2.0 and 3.0 MHz with a copper, water-cooled working coil for rf coupling to the silicon (see Fig. 1). The coil shall have an inside diameter of 20 mm to accommodate the 18-mm outside diameter quartz tube and shall have sufficient power to sustain a molten zone of at least 2 cm. Controls to adjust the power output of the rf generator must be readily available to the operator. The apparatus shall have a carriage to vertically support and move the quartz tube through the coil in a smooth and continuous manner. The upper and lower endpieces of the carriage shall be designed with

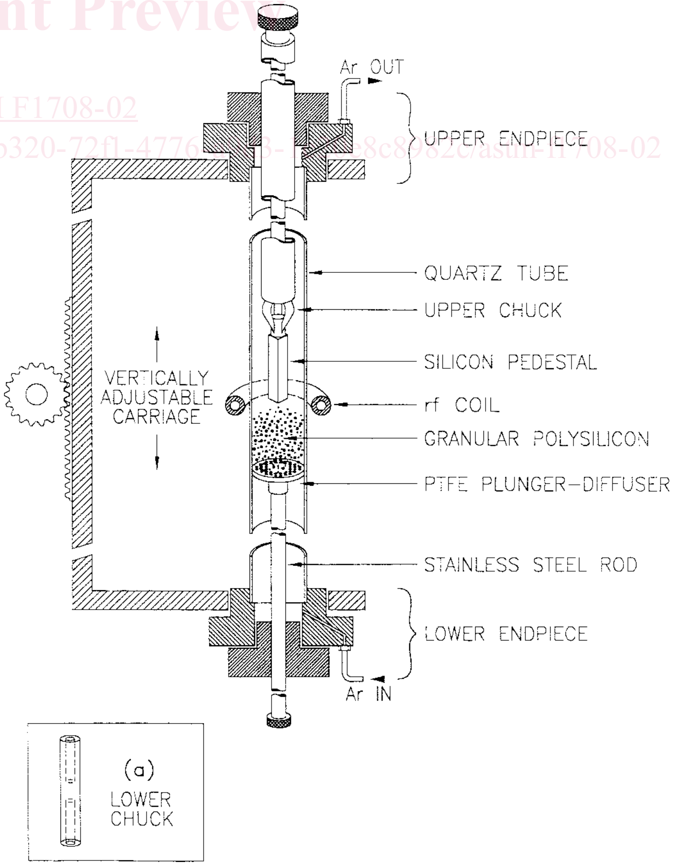

**FIG. 1 Melter/Float Zone Apparatus**