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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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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 Electronic Grade Water[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:

Specification for Gases[4]

Specification for Process Chemicals[4]

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 Practices

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

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.

[1] This practice 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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[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, 805 E. Middlefield Rd., Mountain View, CA 94043.

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*, a hood which can provide for exhaust of acid fumes, provide a clean air environment (Class 1000 minimum), a drain for acids and water, and supplied with deionized water. 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.

7.2 *Laminar Flow Hood*, a hood which will provide a flow of clean (Class 1000 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 chucks to hold silicon pedestals as well as the quartz tubing. These endpieces shall provide a seal to exclude air from the inside of the tube and have connections for argon entry at the bottom endpiece and exhaust at the top. Manual as well as motorized movement of the carriage in the vertical direction while minimizing horizontal motion is essential. This entire apparatus is setup and utilized inside a Class 1000 clean room.

7.7 *PTFE Plunger-Diffuser*, a cylindrical piece of PTFE machined to close tolerance that fits snugly but can be moved freely inside the quartz tube. This PTFE plunger is drilled with an array of small holes for diffusion of argon gas from the bottom endpiece of the zoner carriage, through a bed of polysilicon granules, and finally exhausted through the top endpiece. The bottom of this plunger is machined to fit on the end of a stainless steel rod that is extended through and supported by the bottom endpiece. This rod provides a way to vertically move the PTFE plunger/diffuser plate within the quartz tube.

7.8 *Chucks*, designed to hold silicon pedestals and seed rods.

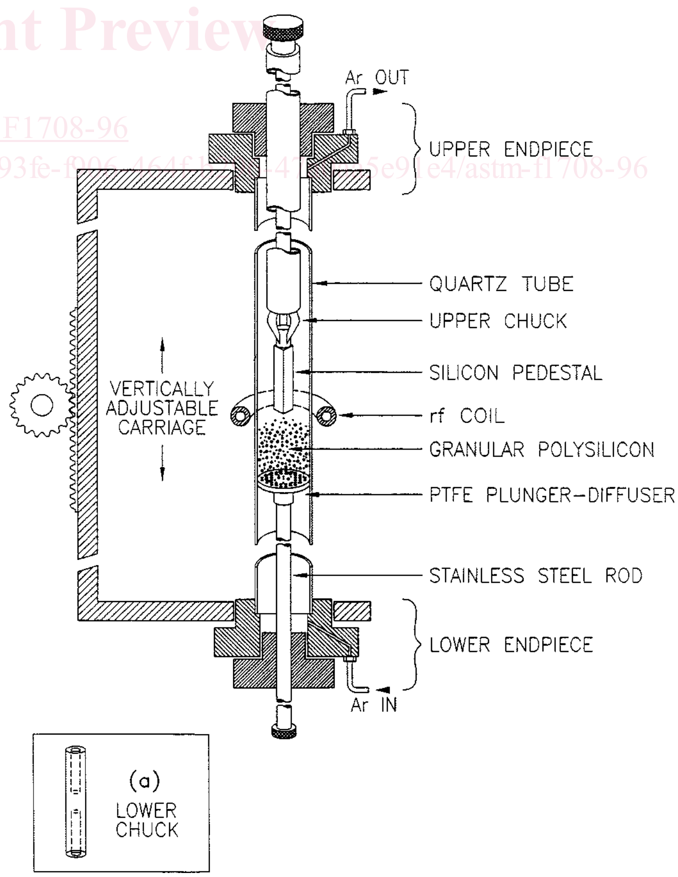

FIG. 1 Melter/Float Zone Apparatus