

SEMI MF1708-1104

PRACTICE FOR EVALUATION OF GRANULAR POLYSILICON BY MELTER-ZONER SPECTROSCOPIES

This guide was technically approved by the Global Silicon Wafer Committee and is the direct responsibility of the North American Silicon Wafer Committee. Current edition approved for publication by the North American Regional Standards Committee on August 16, 2004. Initially available at www.semi.org September 2004; to be published November 2004. Original edition published by ASTM International as ASTM F 1708-96. Last previous edition SEMI MF1708-02

1 Purpose

1.1 Polycrystalline silicon is used as the starting material for growth of large single crystal ingots by Czochralski methods.

1.2 Although 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. This practice provides a means to determine the impurity levels in granular polysilicon to be used for crystal growth.

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

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

2 Scope

2.1 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.

2.2 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 0.02 to 15 parts per million atomic (ppma) for carbon. The acceptor and donor impurities in a slice taken from the single crystal ingot are analyzed by photoluminescence or infrared spectroscopies. The carbon impurity is determined by analysis of a slice by infrared spectroscopy.

2.3 This practice is applicable only to evaluation of polysilicon granules as produced by thermal decomposition and deposition of silane, or one of the chlorosilanes, onto high purity seeds of polysilicon in a continuous or batch fluid bed reactor. The granules are

near spherical in shape and range in size from 200 to 3000 μm with a mean size of about 900–1200 μm .

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health guides and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 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 polysilicon.

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

3.3 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.

4 Referenced Standards

4.1 SEMI Standards

SEMI C3.42 — Specification for Argon

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C31 — Specification for Methanol

SEMI C34 — Specification and Guideline for Mixed Acid Etchants

SEMI C35 — Specifications and Guideline for Nitric Acid

SEMI MF1241 — Terminology of Silicon Technology

SEMI MF1389 — Test Methods for Photoluminescence Analysis of Single Crystal Silicon for III-V Impurities

SEMI MF1391 — Test Method for Substitutional Carbon Content of Silicon by Infrared Absorption

SEMI MF1630 — Test Method for Low Temperature FT-IR Analysis of Single Crystal Silicon for III-V Impurities

4.2 ASTM Standard

D 5127 — Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry¹

4.3 Federal Standard

209-E — Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones²

4.4 ISO Standard

ISO 14644-1 — Cleanrooms and associated controlled environments—Part 1: Classification of airborne particulates³

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Acronym

5.1.1 *PTFE* — polytetrafluoroethylene, a chemically resistant polymer.

5.2 Definitions

5.2.1 Most terms used in this practice are defined in SEMI MF1241.

5.2.2 Other Definitions

5.2.2.1 *granular polysilicon, n* — nearly spherical, granules (200 to 3000 μm) of polysilicon as produced in a fluidized bed reactor.

5.2.2.2 *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.

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

1 *Annual Book of ASTM Standards*, Vol 11.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

2 Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS. (This standard has been superseded by ISO 14644-1 and may no longer be available.)

3 International Organization for Standardization, ISO Central Secretariat, 1, rue de Varembe, Case postale 56, CH-1211 Geneva 20, Switzerland. Telephone: 41.22.749.01.11; Fax: 41.22.733.34.30 Website: www.iso.ch; also available in the US from American National Standards Institute, New York Office: 11 West 42nd Street, New York, NY 10036, USA. Telephone: 212.642.4900; Fax: 212.398.0023 Website: www.ansi.org, and in other countries from ISO member organizations.

6 Summary of Practice

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

6.1.1 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 15 g of polysilicon has been melted and cooled in the zone process, a polysilicon rod about 8 to 12 mm in diameter and up to 70 mm in length is obtained.

6.1.2 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 8 to 12 mm in diameter and up to 70 mm in length from which a section is sliced for measurement of impurities. The entire consolidation and zoning requires about 30 min to accomplish.

6.1.3 A slice 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.

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: ISO class 6 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 556 mm. 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.