

SEMI PV52-0214 (Reapproved 0320) TEST METHOD FOR IN-LINE CHARACTERIZATION OF PHOTOVOLTAIC SILICON WAFERS REGARDING GRAIN SIZE

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1 Purpose

- 1.1 Multicrystalline silicon (mc-Si) wafers consist of a multitude of crystallographically differently oriented grains. The term multicrystalline includes also so-called ‘mono-like’ Si wafers in the context of this Test Method.
- 1.2 The number and size of these grains vary significantly depending on the crystallization method and the wafer’s position in the ingot.
- 1.3 Grain boundaries may be regions of high recombination activity in a multicrystalline wafer. They also may getter unwanted impurities from the interior of the grains and diminish their detrimental impact on solar cell conversion efficiency.
- 1.4 An appropriate solar cell manufacturing process can reduce the detrimental effects of grain boundaries and internal grain defects.
- 1.5 An optimized grain size distribution for a specific solar cell manufacturing process is preferable.
- 1.6 A standardized test method for measuring the grain sizes and their distribution is required to establish wafer specifications regarding grain sizes.

2 Scope

- 2.1 This Test Method evaluates dimensional characteristics of cross-sections of grains of mc-Si as they appear on a wafer surface.
- 2.2 It employs an in-line, noncontacting and nondestructive method for characterizing clean, dry Si wafers that are supported by a mechanism that move the test specimen through the measurement equipment.
- 2.3 The surface condition of the wafers may be as cut or as etched.
- 2.4 The test method covers square and pseudo-square Si wafers for photovoltaic (PV) applications, with a nominal edge length ≥ 125 mm and a nominal thickness ≥ 100 μm .
- 2.5 The test method is intended for in-line high throughput measurements. Therefore it is mandatory to operate the measurement system under statistical process control (SPC) (e.g., ISO 11462) in order to obtain reliable, repeatable and reproducible measurement data.
- 2.6 The test method is based on recording and evaluating images of the wafer surface obtained by a digital camera under directional transmitted or reflected light illumination.
- 2.7 Two procedures for evaluating the grain size characteristics are defined. Both methods are based on obtaining a digital image of the wafer surface displaying the grain structure that is digitally processed. The first procedure straightforwardly evaluates the grain sizes; the second procedure follows a statistical approach used in metallurgy as described in ASTM E112. The constraints of the second procedure must be checked before applying it.
- 2.8 The test may also be used for off-line characterization of Si wafers provided the requirements of the test method are met.
- 2.9 Other measurement techniques may also provide similar information as compared to this Test Method, but they are not the subject of this Test Method.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

- 3.1 This method is only valid for wafers which are cut by a slurry-based technology.
- 3.2 The contrast between the wafer image and the background must be $\geq 10\%$.
- 3.3 Weak contrast of the different grains of a wafer may result in erroneous grain size values.
- 3.4 Variations in illumination (incident angle, irradiance) and orientation of a specimen with respect to light source and camera may impact the precision of repeated measurements.
- 3.5 Residue on the wafer surface also may impact the measurement results.

NOTE 1: The contrast of the different grains seen on a wafer surface depends on surface texture, illumination (wavelength, incident angle, polarization) and orientation of the wafer with respect to light source and camera. Therefore repeated or reproduced measurements of a specimen have to be performed under identical conditions regarding these parameters.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI E89 — Guide for Measurement System Analysis (MSA)

SEMI M59 — Terminology for Silicon Technology

SEMI MF1569 — Guide for Generation of Consensus Reference Materials for Semiconductor Technology

4.2 ASTM Standards¹

ASTM E112 — Standard Test Method for Determining Average Grain Size

ASTM E1382 — Standard Test method for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis

4.3 ISO Standards²

ISO 11462-1 — Guidelines for Implementation of Statistical Process Control (SPC) – Part 1: Elements of SPC

ISO 11462-2 — Guidelines for Implementation of Statistical Process Control (SPC) – Part 2: Catalogue of Tools and Techniques

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

5 Terminology

5.1 Terms and acronyms relating to silicon and other semiconductor technology are defined in SEMI M59.

5.2 Other Abbreviations and Acronyms

5.2.1 MSA — measurement system analysis

5.3 Definitions

5.3.1 *grain* — a single-crystalline volume in the bulk of a material. Also used for denoting a cross section of the grain seen on the surface of a slice through the bulk material.

5.3.2 *grain boundary* — the perimeter of a two-dimensional cross-section of a grain.

6 Summary of Test Method

6.1 The wafer resting on a transport mechanism is moved in the x -direction, the direction of travel, past a directional white light source and a digital matrix camera (see Figure 1).

6.2 For off-line measurement the wafer is positioned on an appropriate support.

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>

² International Organization for Standardization, ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland; Telephone: +41.22.749.01.11, <http://www.iso.org>

6.3 A gray scale picture of the wafer is recorded by the digital camera and processed according to § 13 in order to identify and to outline the grain boundaries.

6.4 Grain area characteristics (maximum, minimum, mean, size distribution) are evaluated according to two procedures—denoted *A* and *B*—in § 14.

6.4.1 Procedure A directly counts the pixels within the grains and reports the number of grains as well as their size statistics.

6.4.2 Procedure B follows a statistical approach as described in ASTM E112 or ASTM 1382 for data analysis of metallurgical samples resulting in an average grain size.³

6.4.3 Procedure A and B may be used alternatively or jointly.

6.5 The results of the evaluation are reported according to § 14.

7 Apparatus (see Figure 1)

7.1 *Light Source* — The wafer is homogeneously illuminated by light sources L_n ($n = 1, 2, \dots, N$) with directional light. The light sources illuminate the wafer under incident angles (with respect to the wafer surface normal) α_n and wavelength λ_n .

7.2 *Camera* — A digital camera capable for taking an image with at least 2048 pixels resolution above the wafer surface. The line of sight of the camera is perpendicular to the wafer surface and the distance camera-wafer surface is ≥ 3 times wafer diagonal. The exposure time of the camera has to be set so that the image sharpness is better than 3 pixels. The noise level (3σ) of the camera shall be $\leq 1\%$ of the peak signal and the dynamic range shall be ≥ 255 gray levels.

7.3 *Computer* — For controlling the measurement system and equipped with software for recording and processing the camera images according to § 13.

7.4 *Wafer Transport* — Consisting of a mechanism that transports the wafer continuously through the measurement apparatus without obstructing the line of sight of the camera and the illumination. The mechanism shall not leave traces or residue on the wafer surface.

8 Safety Precautions

8.1 The entire equipment must be placed in a closed housing and secured with a safety lock that stops the belts and switches the tool off when the housing is opened if lasers are used for illuminating the wafer.

9 Test Specimens

9.1 Clean, dry Si wafers with as-cut or etched surfaces.

10 Preparation of Apparatus

10.1 The suitability of the equipment is determined by performing a statistically based measurement system analysis (MSA) to ascertain whether the equipment is operating within the manufacturer's stated specification; for example, according to SEMI E89.

10.2 Verify that the camera, illumination and wafer are aligned and adjusted according to the manufacturer's specifications.

10.3 Define the SPC control limits for the measurement equipment with a set of selected wafers.

NOTE 2: As this Test Method is intended for a high throughput, high volume measurement the equipment cannot be calibrated for measuring each individual wafer. Therefore careful SPC has to be performed.

³ Schumann, M., Haas, T., Orellana Pérez, T., Riepe, S., Proc. of the 26th European Photovoltaic Solar energy Conference, Hamburg, 2011, H. Ossenbrink, A. Jäger-Waldau and P. Helm, Eds., DOI: 10.4229/26thEUPVSEC2011-2BV.4.22