

SEMI PV43-0113 (Reapproved 1018)

TEST METHOD FOR THE MEASUREMENT OF OXYGEN CONCENTRATION IN PHOTOVOLTAIC (PV) SILICON MATERIALS FOR SILICON SOLAR CELLS BY INERT GAS FUSION INFRARED DETECTION METHOD

This Standard was technically approved by the Photovoltaic – Materials Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on August 17, 2018. Available at www.semiviews.org and www.semi.org in October 2018; originally published January 2013.

1 Purpose

1.1 The purpose of this Test Method is to standardize analytical protocols for the measurement of oxygen concentration in photovoltaic (PV) silicon materials. This will facilitate comparison of test results between laboratories which support research and development activities or monitor or qualify PV silicon materials for purchase, sale or internal use.

2 Scope

2.1 This Test Method can be used to analyze the oxygen content in PV silicon materials for photovoltaic applications. It may also be used for semiconductor grade silicon materials.

2.2 Applications of this Test Method include monitoring or qualifying PV silicon materials to be used for silicon solar cell production.

2.3 Furthermore, this Test Method may be used in research and development of PV silicon materials manufacturing and crystalline and multicrystalline silicon growth processes. It may also be used for correlation of failure or reduced performance and oxygen concentration of crystalline or multicrystalline silicon solar cells.

2.4 The test method described is for use with commercially available oxygen analyzers which are based on the inert gas fusion principle and use infrared detector as the measuring technique.

2.5 The detection range of the method described is from 5 to 500 ppma.

2.6 Not all the information needed to determine the oxygen concentration in PV silicon materials is included in this Test Method. For instance, determination of the optimum temperatures for measuring the oxygen concentrations in a variety of silicon samples or optimization of the oxygen peak parameters are not described in this Test Method.

2.7 This Test Method does cover the particular factors (e.g., specimen preparation, calibration procedures, determination of detection limits) known to affect the most the reliability of oxygen analyses.

2.8 Silicon samples in a range of physical forms can be used, including polysilicon powders, granules, flakes, chunks, and single and multicrystalline wafers and slugs.

2.9 The oxygen determinations described in this Test Method can be used irrespective of all dopant species and concentrations.

2.10 The limit of detection is determined by the instrumental blank value limitations, and may vary with instrumentation and preparation techniques.

2.11 The typical elements present in PV silicon materials for photovoltaic applications such as dopants or unwanted trace or ultra-trace level impurities do not interfere with the determination of oxygen concentrations conducted by this 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 Some impurities from a lower grade flux can cause some adsorption of released oxygen. It is recommended to use only high purity flux.

3.2 Temperature fluctuations in single graphite crucibles can affect the measurement. This can be significantly reduced by using inner crucibles referred further as double-wall crucible.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI PV17 — Specification for Virgin Silicon Feedstock Materials for Photovoltaic Applications

SEMI PV22 — Specification for Silicon Wafers for Use in Photovoltaic Solar Cells

4.2 ASTM Standards¹

ASTM E50 — Standard Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

ASTM E122 — Standard Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process

ASTM E135 — Standard Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

ASTM E691 — Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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

5 Terminology

5.1 Terminology in this Test Method is consistent with terminology in ASTM E135. Required terminology specific to this Test Method, not covered in ASTM E135, is indicated in ¶ 5.2.

5.2 Definitions

5.2.1 *inert gas fusion with infrared detection* — an analytical method in which the analyzed sample is fused in single-use graphite crucible under inert gas flow at a temperature sufficient to release gases, which are then measured using infrared detection.

5.2.2 *PV silicon materials for photovoltaic applications* — the designation is given to silicon solid materials in accordance to SEMI PV17 and SEMI PV22.

5.2.3 *reference sample* — material accepted as suitable for use as a calibration/sensitivity reference standard by all parties concerned with the analyses.

5.2.4 *specimen* — a suitably sized piece cut from a reference or test sample, prepared for introduction into the loading port of the oxygen analyzer, and analyzed.

5.2.5 *test sample* — material (silicon) to be analyzed for oxygen content by this method. Generally, the test sample is selected from a larger batch (lot, casting) of product and is intended to be representative of the batch.

5.2.6 *blank (sample)* — a high purity silicon sample having oxygen content below the sensitivity of the apparatus, such as float zone electronic grade silicon, used to establish the background signal of the detectors.

5.2.7 *graphite crucible* — crucibles made of high purity graphite grades in order to ensure good heating characteristics with minimal dusting.

5.2.8 *high purity graphite powder addition* — the addition of graphite powder designed to optimize furnace performance and facilitate the release of oxygen from the test sample.

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>