

SEMI PV9-0611 (Reapproved 1215) TEST METHOD FOR EXCESS CHARGE CARRIER DECAY IN PHOTOVOLTAIC (PV) SILICON MATERIALS BY NONCONTACT MEASUREMENTS OF MICROWAVE REFLECTANCE AFTER A SHORT ILLUMINATION PULSE

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1 Purpose

1.1 If the free carrier density of a semiconductor is not too high, the excess charge carrier decay time (in short, ‘decay time’) is controlled by impurity centers that have energies located in the forbidden energy gap. Many metallic impurities form such recombination centers in silicon and affect solar cell efficiency by reducing the decay time. For high efficiency cells the decay characteristics must be carefully controlled to obtain the desired high performance device.

1.2 This Test Method¹ covers a procedure for measuring decay time in a variety of types of single crystal and multi-crystalline silicon wafers, bricks and ingots. The procedure is based on the microwave photoconductance decay (μ -PCD) method in which the decay of the conductance after photoexcitation is determined by the decay time of the photogenerated excess carriers.

2 Scope

2.1 This Test Method covers the measurement of excess carrier decay appropriate to carrier recombination processes in *n*- or *p*-type, single crystal or multicrystalline silicon materials. The room-temperature resistivity of the specimen should be greater than a limit that is determined by the sensitivity of the detection system and is normally in a range from 0.05 to 10 $\Omega \cdot \text{cm}$. This Test Method may be applied to the measurement of excess carrier decay in bricks, ingots, or as-cut, lapped, etched, or polished wafers, provided that the sensitivity of the conductivity detection system is adequate.

2.2 Measurement of the excess carrier decay when surface recombination can be neglected and the density of injected carriers is very small and no carrier trapping or depletion modulation occurs (see ¶ 3.7) results in the determination of the low injection bulk excess carrier lifetime (τ_b). In wafers, however, it is often very difficult to completely suppress surface recombination, and the lifetime measurement may depend on the type of surface conditioning used. In addition, the laser power level and wavelength also affect the measured decay time value (see § 0). To enhance the signal-to-noise (S/N) ratio and to avoid the impact of carrier trapping or depletion modulation, high-level excitation is often adopted. In this case, the extraction of the excess carrier lifetime from the excess carrier decay is still possible, but more sophisticated.² Therefore, this Test Method does not purport to measure excess carrier lifetime, but instead covers measurement of decay time under conditions where the surface recombination is not suppressed and/or the excitation level is not low, in which case the decay time does not equal the carrier recombination time.

2.3 In this Test Method, the decay of the wafer conductivity following generation of excess carriers with a light pulse is determined by monitoring the microwave reflectivity of the wafer. The relationship between excess carrier decay and the microwave conductive decay measurement is non-linear over a large range. However, over small variations of carrier density, the relationship can be taken as linear in the ranges of microwave frequency specified in this Test Method. The measured reflected microwave signal following the generation of carriers by a pulse of light with sufficiently fast fall time has the form shown in Figure 1. This figure also shows analysis algorithms for two excess carrier decay times resulting from such conditions, as follows:

¹ This Test Method extends SEMI MF1535 in which the noncontact measurement of photoconductive decay by microwave reflectance was limited to measurements on single crystal silicon wafers. This Test Method is consistent with the method described in ¶ 13.2.2 of EN 50513.

² Lauer, K., Laades, A., Übensee, H., Metzner, H., and Lawerenz, A., “Detailed Analysis of the Microwave-detected Photoconductance Decay in Crystalline Silicon.” *J. Appl. Phys.*, **104** (2008): pp. 104–503.

2.3.1 The primary mode decay time, τ_1 , is influenced by both bulk and surface properties, as well as measurement conditions.

2.3.2 The $1/e$ decay time, τ_e , depends on both the excitation level and the penetration depth (determined by the laser wavelength). The $1/e$ decay time is also strongly influenced by the surface condition, because just after irradiation, the excess carriers are distributed near the surface. On the other hand, τ_e is measured easily and quickly owing to good S/N ratio in the initial part of the decay curve and simplicity of the data analysis.

NOTE 1: In general, the measured decay time is not uniquely related to the excess carrier lifetime because the excess carrier lifetime is not a unique property of the material in any event but depends on, among other things, the injected excess carrier density (see Related Information 1 of SEMI MF1535 for a brief discussion of this issue, which has been widely discussed in the technical literature, and in SEMI AUX017).

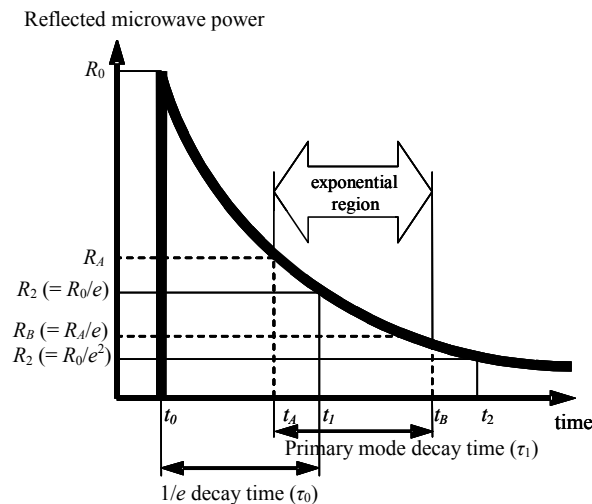


Figure 1
Decay Curve of Reflected Microwave Power and the Definitions of Primary Mode and $1/e$ Decay Times

2.4 This Test Method is appropriate for the measurement of excess carrier decay times in the range from 0.1 μ s to >1 ms. The shortest measurable values are governed by the turn-off characteristics of the light source and by the sampling frequency of the decay signal analyzer while the longest values are determined by the geometry of the test specimen and the surface recombination velocity. With suitable surface conditioning procedures (see Related Information 1), excess carrier decay times as long as tens of milliseconds can be determined even in relatively thin wafers or other specimens.

2.5 In all cases, even though the result of this Test Method is not a real physical property of the test specimen, it is expected to provide information that can be related to the properties of devices for which the material is subsequently used. Thus, this Test Method can be used to determine an effective carrier lifetime as indicated in ¶ 14.2.2 of the March 2009 edition of EN 50513. It is, however, essential to monitor and record all aspects of the measurement so measurements between parties can be properly compared.

2.6 This Test Method is suitable for use in research and development, process control, and materials acceptance applications. However, the results obtained by this Test Method depend on the surface conditioning used (if any), the temperature of the sample to be measured, the laser wavelength, power level, pulse duration, and spot size, used to generate the carriers, and the waveform analysis algorithm used to analyze the decay transient. Therefore, when this Test Method is used for materials specification or acceptance, the supplier and the purchaser need to agree on all of these parameters to produce comparable results.

2.7 Interpretation of measurements to identify the cause or nature of impurity centers is beyond the scope of this Test Method. The identity and density of impurity centers may usually be determined more reliably from deep-level transient spectroscopy (DLTS) measurements made in accordance with SEMI MF978, from other capacitance or

current transient spectroscopy techniques provided that a suitable catalog of impurity characteristics is available,³ or from lifetime spectroscopic methods.⁴

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 If the recombination lifetime of the carriers is such that the excess charge carriers can diffuse to the surface before they recombine, surface recombination influences their decay. Then the effects of recombination at the surfaces of the wafer must be suppressed by surface to measure the decay of excess carriers in the bulk conditioning as described in Related Information 1.

3.1.1 Treatments with surface conditioning solutions must result in a stable surface for the test method to produce reliable results.

3.1.2 A further caution is in order if thermal oxidation is employed. Particularly in high oxygen wafers, oxide precipitates may form in the bulk of the wafer during oxidation. The presence of such precipitates can alter the decay time properties of the wafer (see also ¶ 3.2) thus rendering the test specimen unsuitable for measurement by this Test Method.

3.2 Variations in carrier recombination properties in the direction perpendicular to the wafer surface may result in inaccurate determinations. These variations may arise because of the presence (1) of p - n or high-low (p - p^+ or n - n^+) junctions parallel with the surface or (2) of regions of dissimilar recombination characteristics (such as a wafer with oxide precipitates and a surface denuded region free of such precipitates).

3.3 The recombination characteristics of impurities in silicon are strongly temperature dependent. If comparisons between measurements are to be made (i.e., before and after a process step or at a supplier and a customer), both measurements should be made at the same temperature.

3.4 Different impurity centers have different recombination characteristics. Therefore, if more than one type of recombination center is present in the wafer, the decay of excess carriers may consist of contributions with two or more time constants. Such a decay curve may not be representative of any of the individual centers.

3.5 The recombination characteristics of an impurity center depend on the type and concentration of the dopants in the wafer as well as the position of the energy level of the impurity center in the forbidden energy gap.

3.6 Higher mode decay of photoinjected carriers influences the shape of the decay signal, particularly in its early phases.⁵ These effects are minimized by measuring the decay in an exponential portion of the decay signal. Under certain conditions or on some test specimens, such a portion might not exist. In this case, the decay time is arbitrarily taken as the time for the signal to decay to a specified level (normally $1/e$ where $e = 2.71828$ of the initial signal).

3.7 The measured decay time may be affected by carrier trapping or depletion region modulation.⁶

3.8 The method is not suitable for measurements in bricks or ingots where the decay time is greater than 10 μ s due to the diffusion of carriers into the material beyond the measurement depth of the microwaves used.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI M59 — Terminology for Silicon Technology

³ Schulz, M., ed., "Semiconductors: Impurities and Defects in Group IV Elements and III-V Compounds." Landolt-Börnstein, New Series III/22b, Springer Verlag, Heidelberg (1989): ¶ 4.2.3.1.

⁴ Rein, S., "Lifetime Spectroscopy: a Method of Defect Characterization in Silicon for Photovoltaic Applications." Springer, Berlin, 2005.

⁵ Blakemore, J. S., "Semiconductor Statistics." Dover Publications, New York (1987): § 10.4.

⁶ See, for example; Macdonald, D., Sinton, R. A., and Cuevas, A., "On the Use of a Bias-light Correction for Trapping Effects in Photoconductance-based Lifetime Measurements in Silicon." *J. Appl. Phys.* **89** (2001): p. 2772, and Cousins, P. A., Neuhaus, D. H., and Cotter, P. A., "Experimental Verification of the Effect of Depletion-region Modulation on Photoconductance Lifetime Measurements." *J. Appl. Phys.* **95** (2004): p. 1854.

SEMI MF42 — Test Methods for Conductivity Type of Extrinsic Semiconducting Materials

SEMI MF43 — Test Methods for Resistivity of Semiconductor Materials

SEMI MF84 — Test Method for Measuring Resistivity of Silicon Wafers With an In-Line Four-Point Probe

SEMI MF533 — Test Method for Thickness and Thickness Variation of Silicon Wafers

SEMI MF673 — Test Methods for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage

SEMI MF723 — Practice for Conversion Between Resistivity and Dopant Density for Boron-Doped, Phosphorus-Doped, and Arsenic-Doped Silicon

SEMI MF978 — Test Method for Characterizing Semiconductor Deep Levels by Transient Capacitance Techniques

SEMI MF1530 — Test Method for Flatness, Thickness, and Thickness Variation of Silicon Wafers by Automated Noncontact Scanning

SEMI MF1535 — Test Method for Carrier Recombination Lifetime in Silicon Wafers by Non-Contact Measurement of Photoconductivity Decay by Microwave Reflectance

4.2 *EN Standard⁷*

EN 50513 — Solar Wafers – Data Sheet and Product Information for Crystalline Wafers for Solar Cell Manufacturing

4.3 *Other Documents*

SEMI AUX017 — Contactless Carrier Lifetime Measurements in Silicon Wafers, Ingots, and Blocks

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

5 Terminology

5.1 Definitions of most terms used in PV silicon technology may be found in SEMI M59.

5.2 *Other Acronym Used in This Standard*

5.2.1 *PV* — photovoltaic

5.3 *Other Terms Used in This Standard*

5.3.1 *1/e decay time (τ_e)* — the time duration from the laser pulse injection to the instant that the microwave signal decreases to 1/e of its peak value.

5.3.2 *brick* — one or more squared, cropped, and ground sections from an ingot.

5.3.3 *ingot* — a cylindrical or rectangular solid of silicon resulting from a crystallization process, generally of slightly irregular dimensions.

5.3.4 *primary mode decay time (τ_1)* — the decay time constant obtained from an exponential part of the decay curve (primary mode part) of microwave reflectance.

6 Summary of Test Method

6.1 If wafer surface conditioning is necessary, the test specimen is conditioned using the agreed upon treatment, which may be one of the surface conditioning treatments described in Related Information 1.

6.1.1 Excess hole-electron pairs are locally created in the wafer for a very brief time by a short pulse of light with a fall time ≤ 10 ns with energy slightly greater than the width of the forbidden energy gap at a specified wavelength and specified power density or specified photons/pulse. The decay of the conductivity following cessation of the light pulse is monitored by means of microwave reflectance, and the excess carrier decay time is determined as follows:

6.1.1.1 The primary mode decay time is taken as the decay time constant determined from an exponential portion of the decay curve using agreed upon analysis algorithms.

⁷ Available from CENELEC member bodies, see the CENELEC website at <http://www.CENELEC.eu/> for information.