SEMI PV13-0813
TEST METHOD FOR CONTACTLESS EXCESS-CHARGE-CARRIER RECOMBINATION LIFETIME MEASUREMENT IN SILICON WAFERS, INGOTS, AND BRICKS USING AN EDDY-CURRENT SENSOR

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1 Purpose

1.1 The excess-charge-carrier (hereafter referred to as 'excess-carrier') recombination lifetime is the central parameter to silicon solar cell device design, production, and process control. The measurement of this lifetime as it depends on excess-carrier density yields physically significant results, which allows for design optimization and efficiency prediction in solar cells. The Test Method also describes how this recombination lifetime can be further analyzed in terms of more fundamental parameters of importance to solar cells, such as the bulk lifetime, surface recombination velocity, or the emitter saturation current density of the dopant diffusion. This Test Method includes measurement by quasi-steady-state photoconductance (QSSPC) and transient techniques using an eddy-current sensor.

2 Scope

2.1 This Standard describes methods for measuring the excess-carrier lifetime in silicon wafers, ingots, and bricks with carrier recombination lifetime in the range of 0.1 to 15,000 µs.

2.2 The measurements are applicable in the excess-carrier density range from 1×10^{13} cm^{-3} to 2×10^{16} cm^{-3} for wafer specimens, and 1×10^{13} cm^{-3} to 5×10^{15} cm^{-3} for bulk specimens (thicker than 1 cm).

2.3 The method described here is used for specimens with resistivity in the range from 0.1 Ω·cm to 10000 Ω·cm.

2.4 This Standard describes four measurement methodologies; two each for the assessment of wafered or bulk silicon specimens. All measurement techniques are performed using an inductive-coil (eddy-current) sensor energized at a radio frequency and an illumination source. Two of the methods use a QSSPC technique that requires a light-intensity detector to measure the photogeneration. The scope of this document also addresses the calibration method to be used.

2.4.1 *Transient Mode for Wafers* — This Test Method is appropriate for a silicon specimen in wafer form, of a thickness not to exceed 1 mm. As described in this Standard, the transient mode is used for excess-carrier lifetimes which are long compared to the cut-off time of the illumination.

2.4.2 *QSSPC Mode for Wafers* — This Test Method is appropriate for wafer specimens of a thickness not to exceed 1 mm. As described in this Standard, the QSSPC Mode is used to determine lifetime by analyzing data taken during photo excitation.

2.4.3 *Transient Mode for Bulk Material* — This Test Method shall be used to measure specimens with a thickness greater than 1 cm, and is used to measure carrier lifetimes that are much longer than the cut-off time of the illumination.

2.4.4 *QSSPC Mode for Bulk Material* — This Test Method shall be used for specimens greater than 1 cm, and determines carrier recombination lifetime by analyzing data taken during photoexcitation.

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3 Limitations

3.1 Carrier lifetime is, in general, a strong function of the excess-carrier density.[1] Therefore, in order to compare data, different users need to report the lifetime at the same excess-carrier density.

3.2 Carrier trapping may be present in silicon at room temperature. If trapping of either electrons or holes occurs in the specimen, the excess concentration of the other type of carrier remains high following cessation of the light pulse (in the Transient method), or for low light intensity (for the QSSPC method). Methods avoid reporting lifetime based upon the low excess-carrier density range when a trapping characteristic is present, unless a correction technique is applied[2] (see Appendix 1). The characteristic indicative of trapping is when the measurement lifetime sharply increases with decreasing excess-carrier density, for excess-carrier densities in the range of 1×10^{12} to 2×10^{14} cm^{-3}. This apparently high lifetime at low carrier density is not indicative of high excess-carrier lifetime.[3] Showing the entire lifetime versus excess-carrier density curve is good practice if corrections are required, with both corrected and uncorrected data displayed. Users comparing data need to specify the trapping correction, if any (see Appendix 1).

3.3 If there are junctions in the device, either dopant-diffused junctions or inversion layers created by surface charge, then do not report data at low carrier densities where the depletion region modulation (DRM) characteristic is present, unless a correction is applied as in Appendix 1.[4,5] Users comparing data specify the correction, if any.

3.4 Carrier mobility in silicon has a negative temperature coefficient. Therefore, heating of the specimen appears as a negative photoconductance that integrates the photoexcitation, and then returns to the dark photoconductance baseline with a very long (thermal) time constant. This effect can be seen on thin, heavily-doped specimens especially if high light intensities are used to see a signal from a low-lifetime specimen.

3.5 Specimens very close to intrinsic (undoped) silicon (where sheet resistance is greater than 2000 Ω) can depart from the quadratic calibration curve described in this Standard.[6]

3.6 In the case that the diffusion length and absorption depth of the light in a wafer specimen are much less than the wafer thickness, then the effective thickness to be used when calculating the excess carrier density is twice the sum of diffusion length and absorption depth in place of the wafer thickness.[7]

3.7 For samples where the mobility is unknown, the uncertainty in QSSPC lifetime is proportional to the uncertainty in mobility.

3.8 Bulk measurements by the transient or QSSPC method, with surface excitation and surface sensing, measure only the local near-surface lifetime defined by the excitation depth, diffusion length, and sensing depth. This measured volume extends 1 to 5 mm into the silicon bulk.

3.9 Transient measurements on bulk specimens give a lower bound on the actual bulk lifetime.

3.10 The transfer function from measured to bulk lifetime used for QSSPC bulk measurements needs to be agreed between parties.

3.11 This Test Method describes a methodology for wafers thinner than 0.1 cm, and for bulk material greater than 1 cm. Samples in the range 0.1 to 1 cm can be measured, but the analysis of the data may require computer simulation since simplifying assumptions may not apply. The application of wafer or bulk analysis as described here is approximate only for specimens in the 0.1 to 1 cm range.

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[1] Bothe, K., and Schmidt , J. "Electronically activated boron-oxygen-related recombination centers in crystalline silicon." *J. Appl. Phys.,* 99, 013701, 2006.

[2] Macdonald, Daniel, and Cuevas, Andres "Trapping of minority carriers in multicrystalline silicon." *Applied Physics Letters,* 74 (1999): p. 1710.

[3] SEMI MF28-0707.

[4] Bail, M., Schulz, M., and Brendel, R., "Space-charge region-dominated steady-state photoconductance in low-lifetime Si wafers." *Applied Physics Letters,* 82 (2003): p. 757.

[5] Cousins, P. A., Neuhaus, D. H., and Cotter, J.E. "Experimental verification of the effect of depletion-region modulation on photoconductance lifetime measurements." *Journal of Applied Physics,* 95 (2004): p. 1854.

[6] Macintosh, K., Guo, J. H., Abbot, M. D., and Bardos, R. A. "Calibration of the WCT-100 Photoconductance Instrument at Low Conductance." *Progress in Photovoltaics: Research and Applications* (2008): p. 279.

[7] Bowden and Sinton. "Determining Lifetime in Silicon Blocks and Wafers with Accurate Expressions for Carrier Density." *Journal of Applied Physics,* 102, 124501, 2007.

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3.12 This Test Method is most accurate when the eddy-current sensor is placed sufficiently far (1 to 3 mm in the plane of the sensor) from a specimen's edges.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI M59 — Terminology for Silicon Technology

SEMI MF28 — Test Methods for Minority Carrier Lifetime in Bulk Germanium and Silicon by Measurement of Photoconductivity Decay

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 Methods for Thickness and Thickness Variation of Silicon Wafers

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

4.2 *SEMI Auxiliary Information*

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

SEMI AUX026 — Research Report on Interlaboratory Study to Establish Precision Statements for SEMI PV13, Test Method for Contactless Excess-Charge-Carrier Recombination Lifetime Measurement in Silicon Wafers, Ingots, and Bricks Using an Eddy-Current Sensor

4.3 *ASTM Standards*[8]

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

4.4 *EN Standards*[9]

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

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

5 Terminology

5.1 Acronyms, terms, and symbols related to silicon technology, including those used in this practice, are listed and defined in SEMI M59.

5.2 *Abbreviations and Acronyms*

5.2.1 *B-Cz* — boron-doped Czochralski silicon

5.2.2 *DRM* — depletion-region modulation

5.2.3 *FZ* — float zone (floating zone) silicon

5.2.4 *IQE* — internal quantum efficiency

5.2.5 *QSSPC* — quasi-steady-state photoconductance

5.2.6 *RF* — radio-frequency

5.3 *Symbols*

5.3.1 τ_{eff} — the effective measured carrier recombination lifetime in a specimen; the average time interval spent by the excess charge carrier in the valence or conduction band before an electron-hole recombination event.

[8] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: 610.832.9585, Fax: 610.832.9555, http://www.astm.org

[9] CENELEC, European Committee for Electrotechnical Standardization, Ave. Marnix 17, B-1000 Brussels. EN 50513 specifies a special case of the QSSPC for wafer specimens described here.

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5.3.2 τ_{bulk} — the bulk carrier recombination lifetime in a specimen; the average time interval that would be spent by the excess charge carrier in the valence or conduction band before an electron-hole recombination event if the surface recombination were reduced to negligible levels.

5.3.3 $\Delta n(t)$ — (time-dependent) average excess carrier density.

5.3.4 $G(t)$ — (time-dependent) photogeneration rate for electron-hole pairs.

5.3.5 $S_{front/back}$ — front or back surface recombination velocity.

5.3.6 J_{0e} — emitter saturation current density of a dopant diffused layer.

5.3.7 w — wafer thickness.

5.3.8 D — diffusion coefficient.

5.3.9 $\mu_{n/p}$ — (dopant and excess carrier density dependent) carrier mobility of electrons or holes.

5.3.10 $N_{A/D}$ — concentration of dopant acceptors or donors.

5.3.11 $\Delta\sigma$ — excess conductivity due to photo-excitation.

6 Summary of Test Methods

6.1 *Methods for Wafer Specimens* — A wafer specimen is placed in repeatable proximity to the calibrated eddy-current sensor and illuminated with a time-dependent light profile.

6.1.1 *Transient Mode for Wafers* — After the light is abruptly terminated, the resulting eddy-current sensor output voltage is measured as a function of time. The data from the eddy-current sensor is converted into units of photoconductance, using the instrument calibration (§ 9), and then converted into carrier densities using the mobility functions for silicon as agreed between the parties and recorded in the report. This data is analyzed at each point into the carrier density decay, providing the carrier recombination lifetime as a function of carrier density.

6.1.2 *QSSPC Mode for Wafers* — As the wafer specimen is illuminated, the illumination and eddy-current sensor output voltage are simultaneously measured. The data from the eddy-current sensor is converted into units of photoconductance using the instrument calibration (§ 9). This data is then converted into carrier densities using the mobility functions for silicon as agreed between the parties and recorded in the report. The light intensity incident upon the specimen is converted to photogeneration using a calibration function for the light intensity detector (¶ 9.4.1). This additional input permits the calculation of the excess-carrier lifetime during illumination as well as during the decay after the illumination is terminated.

6.2 *Methods for Bulk Specimens* — A bulk silicon specimen is placed in repeatable proximity to the calibrated eddy-current sensor and illuminated with a time-dependent light profile.

6.2.1 *Transient Mode for Bulk Specimens* — After the light is abruptly terminated, the resulting eddy-current sensor output voltage decay is measured. The data from the eddy-current sensor is converted into units of photoconductance, using the instrument calibration (§ 9), and then converted into carrier densities using the mobility functions for silicon as agreed between the parties and recorded in the report. This data is analyzed at each point during the carrier density decay, providing the carrier recombination lifetime as a function of carrier density. For specimens with high surface recombination, the measured lifetime asymptotically approaches the bulk lifetime as the carriers near the surface are depleted, and the peak carrier density moves away from the surface into the bulk.

6.2.2 *QSSPC Mode for Bulk Specimens* — As the bulk specimen is illuminated, the illumination and eddy-current sensor output voltage are simultaneously measured. The data from the eddy-current sensor, taken during photogeneration, is converted into units of photoconductance using the instrument calibration (§ 9). This data is then converted into carrier densities using the mobility functions for silicon as agreed between the parties and recorded in the report. The light intensity incident upon the specimen is converted to photogeneration using a calibration function for the light intensity detector(¶ 9.4.1). The effects of the surface recombination are reliably calculated in some cases, resulting in a transfer function that permits the reporting of a bulk lifetime value (τ_{bulk}) based on the measured excess-carrier density recombination lifetime.

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7 Apparatus

7.1 *Instrument* — The instrument consists of four components.

7.1.1 *RF-eddy-current Sensor* — A sensor that outputs a voltage that is monotonic in the sheet resistance of a silicon specimen. This sensor has a mechanical configuration that allows it to be placed in known proximity to the silicon specimen.

7.1.2 *A Light Source* — This light source is monochromatic, with wavelength between 350 and 1150 nm, or may be broadband, for example, a broadband light source with filters. In either case, the spectrum of the light source is characterized.

7.1.3 *A Reference Light-intensity Detector (hereafter 'light-intensity detector')* — This light-intensity detector has response in the entire range of the silicon specimen for all wavelengths available in the light. The light-intensity detector time response must be ten times faster than the time response of the light source used in the QSSPC method. For use in the QSSPC method, the reference light-intensity detector must have known spectral response for the range of the light source wavelengths that create electron-hole pairs in the specimen, 300 to 1150 nm.

7.1.4 *A Data Acquisition System* — This system is capable of recording the voltages from the light intensity detector and instrument with sufficient time and voltage resolution. The sampling rate must be ten times higher than the time constant of the lifetime for the transient technique, or the light intensity variation for the QSSPC technique.

7.1.5 A block diagram of the instrument is shown in Figure 1.

7.1.5.1 For wafer specimens, the eddy-current sensor can be placed below the wafer to allow completely unobstructed illumination of the sample from above.

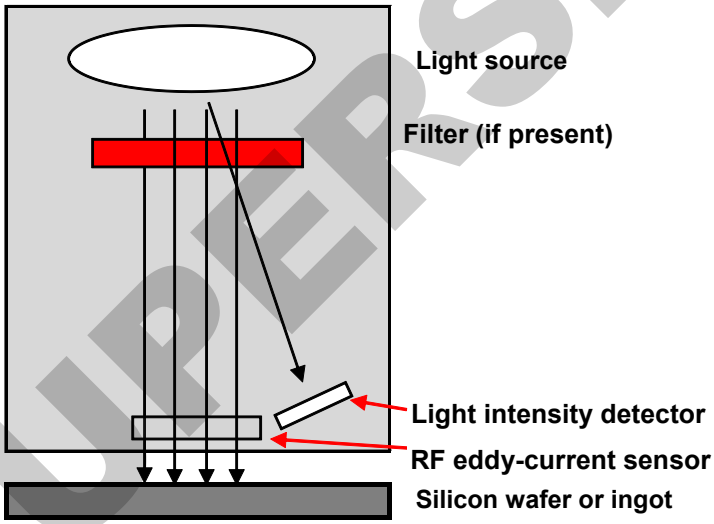

Figure 1
Apparatus for RF-QSSPC and RF-Transient Mode Lifetime Measurements

8 Test Specimen

8.1 The test specimen for the wafer (transient-mode and QSSPC-mode) lifetime measurements may be any silicon wafer of thickness from 10 µm to 1 mm, with area larger than the sense area of the eddy-current sensor. The wafer may be passivated or unpassivated.

8.2 The test specimen for the bulk (transient-mode and QSSPC-mode) lifetime measurements is any silicon specimen of thickness greater than 1 cm, with area larger than the sense area of the eddy-current sensor by at least

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5 mm outside of the sense area. The surface can be as-cut, but best results are obtained for surfaces polished to 1200 grit or finer.[10]

9 Calibration and Standardization

9.1 A wafer set of at least four specimens are prepared from uniformly-doped monocrystalline wafers. Measure the thickness by a method agreed between the parties, for example, SEMI MF533. Then measure the resistivity values by 4-point-probe (SEMI MF84). The sheet resistance is the resistivity divided by specimen thickness. This calibration set spans the intended measurement range of conductance and photoconductance.

9.2 Determine the output of the eddy-current sensor for each calibration wafer under non-illuminated conditions by placing each wafer in repeatable proximity to the sensor. Record the distance between the wafers and the sensor. Using a quadratic fit with least-squares optimization, determine a relationship for conductance as a function of output signal for the calibrated wafer set. The resulting curve is used to convert measured data to absolute units of sheet conductance.

9.3 *QSSPC and Transient Mode for Bulk Specimens* — In addition to obtaining the curve for wafer specimens, repeat the process described in ¶¶ 9.1 and 9.2 using a silicon specimen set of at least four 15 mm thick (or thicker), uniformly doped specimens, to determine a quadratic relationship for bulk conductivity (inverse bulk resistivity) as a function of output signal. In the case of bulk conductivity, the method of SEMI MF43 is used, with temperature controls and corrections as given in SEMI MF84.

9.3.1 *Determine the Sense Depth for the Sensor* — The sense depth is the resistivity (Ω·cm) reported by 4-point-probe divided by the sheet resistance (Ω) reported by the instrument for a bulk sample at least 15 mm thick.

9.4 *Illumination Source Calibration*

9.4.1 Determine a constant of proportionality for photogeneration in the specimen under test as a function of the output signal of the reference light-intensity detector, using one of the following four methods. This calibration is not required for the Transient measurement mode.

9.4.1.1 Use a wafer that will have the identical photogeneration as the device under test; for example, the identical thickness, surface preparation and surface roughness, dielectric (type and thickness), and with carrier recombination lifetime in the range of 100 to 1000 µs. Measure the carrier recombination lifetime of this wafer using the transient method (which requires no reference light-intensity detector) and by the QSSPC method. Determine the calibration for the reference light-intensity detector that duplicates the lifetime measured by the transient method.[11,12]

9.4.1.2 Use a set of wafers with known lifetime based upon measurements with a calibrated instrument. Determine the calibration for the new instrument that duplicates the known values.

9.4.1.3 Using the illumination spectrum of the light source, simulate both the photogeneration in the test specimen and the output signal from the light-intensity detector.[13] This is done by constructing a model solar cell (using PC1D[14] or another program agreed between parties) in which the known spectral response of the light-intensity detector is used to determine its output signal. Then the photogeneration in a model of the specimen (with the correct silicon thickness and dielectric layers) is compared to the light-intensity detector signal output for the same spectral input file from the light source. The ratio of the results is the calibrated photogeneration in the specimen per unit signal from the light-intensity detector for the same light source.

9.4.1.4 If the spectral transmission and reflection of the specimen under test is known, then the photogeneration for the spectrum of the light source can be calculated as the sum of absorbed photons multiplied by the charge on an

[10] Olsen, Espen and Ovrelid, Eivind "Optimizing the Properties of Multicrystalline Silicon Cast in a Pilot Scale Furnace." Proceedings of the 21st European Photovoltaic Solar Energy Conference and Exhibition, Dresden, Germany, WIP-Renewable Energies (2006): pp. 1049–51.

[11] Lago Aurrekoetxea, Rosa, *et al.* "Lifetime Measurements by Photoconductance Techniques in Wafers Immersed in a Passivating Liquid." Journal of the Electrochemical Society, 148 (4) (2001): pp. G200–06.

[12] Trupke, T., and Bardos, R. "Self-consistent determination of the generation rate from photoconductance measurements." Applied Physics Letters, 85 (2004): p. 3611.

[13] Roth, T., Rosenitis, P., Warta, W., and Glunz, S. W. "Photoconductance-based excess carrier lifetime measurements on unpassivated silicon samples." Proceedings of the 24th European Photovoltaic Solar Energy Conference, Hamburg, Germany, WIP-Renewable Energies (2009): pp. 2004–07.

[14] Basore, P.A., and Clugston, D. A. "PC1D/Version 4 for Windows: From Analysis to Design." Proceedings of the 26th IEEE Photovoltaic Specialists Conference, Anaheim, California, Optical Archives, Inc. (1997): p. 377.

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