

SEMI MF391-0310^E

TEST METHOD FOR MINORITY CARRIER DIFFUSION LENGTH IN EXTRINSIC SEMICONDUCTORS BY MEASUREMENT OF STEADY-STATE SURFACE PHOTOVOLTAGE

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^E This Standard was editorially modified in November 2017. A change was made to correct a nonconforming title per the *Procedure Manual*, Appendix 4.

1 Purpose

1.1 Minority carrier lifetime is one of the essential characteristics of semiconductor materials. In epitaxial layers and in thin single crystal wafers, the surface recombination corrections necessary to derive the minority carrier lifetime from the photoconductive decay (PCD) method covered by SEMI MF28 and SEMI MF1535 are excessively large.

1.2 Therefore, other test methods are required to cover the measurement of minority carrier diffusion lengths in specimens of extrinsic single-crystal semiconducting materials or in homoepitaxial layers of known resistivity deposited on more heavily doped substrates of the same type, provided that the thickness of the specimen or layer is greater than four times the diffusion length. Three test methods are described:

1.2.1 *Test Method A* — Constant magnitude surface photovoltage (CMSPV) method. This test method circumvents the influence of surface recombination on the lifetime measurement by maintaining constant front surface conditions.

1.2.2 *Test Method B* — Linear photovoltage, constant photon flux (LPVCPF) method. This test method utilizes only conditions and data points that are not influenced by surface recombination and other non-linear effects.

1.2.3 *Test Method C* — Digital oscilloscope recording (DOR) method. This test method provides the direct control of relaxation processes of surface photovoltage (SPV) appearance and disappearance on minimum radiation intensity and eliminates any non-linear and other distorting effects, which is feasible on these conditions.

1.3 These test methods are suitable for use in research, process control, and materials acceptance.

1.4 These test methods are particularly useful in testing materials to be used in photovoltaic cells and other optical device applications since the diffusion length is derived by methods that are closely related to the functioning of the device.

1.5 Because carrier lifetime is directly influenced by the presence of metallic impurity contamination, these test methods can be interpreted to establish the presence of such contamination. However, such interpretation is beyond the scope of these test methods.

1.6 If a very thin surface region with long lifetime, such as an epitaxial layer or a denuded zone, is on a bulk region with very short lifetime, such as a heavily doped substrate or an internally gettered wafer with oxide precipitates, respectively, the intercept cannot be interpreted as the diffusion length (see ¶ 3.2). Under certain circumstances, the intercept can be related to the layer thickness, providing a nondestructive means for determining the thickness of the layer.

2 Scope

2.1 These test methods are based on the measurement of surface photovoltage (SPV) as a function of energy (wavelength) of the incident illumination.

NOTE 1: The minority carrier lifetime is the square of the diffusion length divided by the minority carrier diffusion constant that is assumed or can be determined from drift mobility measurements. SPV measurements are sensitive primarily to the minority carriers; the contribution from majority carriers is minimized by the use of a surface depletion region. As a result, lifetimes measured by the SPV method are often shorter than the lifetimes measured by the PCD method because the photoconductivity can contain contributions from majority as well as minority carriers. When both majority and minority carrier lifetimes are the same,

both the SPV and PCD methods yield the same values of lifetime¹ provided that the correct values of absorption coefficient are used for the SPV measurements and that the contributions from surface recombination are properly accounted for in the PCD measurement.

2.2 All three test methods covered are nondestructive.

2.3 The limits of applicability with respect to specimen material, resistivity, and carrier lifetime have not been determined; however, measurements have been made on 0.01 to 50 $\Omega \cdot \text{cm}$ *n*- and *p*-type silicon specimens with carrier lifetimes as short as 2 ns.

2.4 These test methods were developed for use on single crystal specimens of silicon. They may also be used to measure an effective diffusion length in specimens of other semiconductors such as gallium arsenide (with suitable adjustment of the wavelength (energy) range of the illumination and specimen preparation procedures) and an average effective diffusion length in specimens of polysilicon in which the grain boundaries are normal to the surface.

2.5 These test methods also have been applied to the determination of the width of the denuded zone in silicon wafers.

2.6 These test methods measure diffusion lengths at room temperature (22°C) only. Lifetime and diffusion length are a function of temperature.

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 The quality of the measurement depends on the accuracy with which the absorption coefficient is known as a function of photon energy (wavelength).

3.1.1 Surface stresses strongly influence the absorption characteristics. These test methods provide absorption coefficient data appropriate to unstressed surfaces typical of those found on epitaxial layers and stress-relieved chemically or chem-mechanically polished wafers.

3.1.2 In heavily doped wafers, the free carrier absorption may affect the SPV measurement at long wavelengths.

3.1.3 The absorption coefficient is temperature dependent; the data given in these test methods are appropriate to room temperature only (22°C).

3.2 For the most accurate measurements, the thickness of the region to be measured must be greater than four times the diffusion length. An estimate of the diffusion length is possible when the diffusion length exceeds twice the thickness. The thickness condition is assessed after the measurement is made.

3.2.1 For measurements on a surface layer (epitaxial layer or denuded region), the intercept may be interpreted as the diffusion length in the substrate if the layer thickness is less than one-half the intercept value.²

3.2.2 If the layer thickness is between one-half and four times the intercept value, estimates of the diffusion length in the surface layer may be made provided that the thickness of the layer is known;² conversely, the layer thickness may be deduced if certain assumptions are made about the ratio of diffusion lengths in the surface layer and substrate regions.

3.3 Unless the total specimen thickness is greater than three times the reciprocal absorption coefficient of the longest wavelength (lowest energy) illumination used, the SPV plot is nonlinear. The upper wavelength limit can be calculated before the measurement is made.

3.4 Variations in long relaxation time surface states may cause a slow drift of the amplitude of the SPV signal with time. This interference can be minimized by (1) allowing sufficient time for the states to approach equilibrium under measurement conditions and (2) making all of the measurements as quickly as possible.

¹ Saritas, M., and McKell, H. D. "Comparison of Minority-Carrier Diffusion Length Measurements in Silicon by the Photoconductive Decay and Surface Photovoltage Methods." *J. Appl. Phys.* 63 (1988): pp. 4562–4567.

² Phillips, W. E. "Interpretation of Steady-State Surface Photovoltage Measurements in Epitaxial Semiconductor Layers." *Solid-State Electron.* 15 (1972): pp. 1097–1102.

3.5 The SPV signal can be masked by a photovoltage produced by the illumination of non-ohmic back contact or of a junction in the specimen. A masking photovoltage of this type can be identified by its large amplitude, a reversal in polarity as the illumination energy changes from large to small, or by the decrease of signal amplitude with increase of illumination intensity at longer wavelengths (smaller energy). In case of method C this masking photovoltage may be detected by SPV signal shape analysis. A junction photovoltage can be eliminated by making the reference potential contact to an unilluminated region of the front surface.

3.6 Lack of spectral purity of the illumination adversely affects the measurements. Although spectral purity requirements have not been definitively established, a spectral bandwidth of 5 nm and (if a grating monochromator is used) an intensity of higher order spectral components of less than 0.1% are expected to provide satisfactory results.

3.7 In some materials the lifetimes and diffusion lengths depend on the intensity of illumination. This occurs even when the density of hole-electron pairs is still much less than the majority carrier density. The principal effect is to give a diffusion length larger than the dark value. This effect can be minimized by working in a linear SPV range in which the SPV signal is directly proportional to the illumination intensity.

3.8 For Test Methods A and C, correction must be made for any differences in losses as a function of energy (wavelength) in the optical path to the specimen and the optical path to the detector. For example, any surface film or coating can introduce an energy dependent absorption or reflection.

3.9 Handling of the test specimens with metal tweezers may introduce metal contamination that can shorten the minority carrier lifetime and result in an erroneous determination of diffusion length. To eliminate the effect of handling on diffusion length measurements, use clean plastic tweezers or a plastic vacuum pick-up.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI C23 — Specifications for Buffered Oxide Etchants

SEMI C30 — Specifications for Hydrogen Peroxide

SEMI C34 — Specification and Guide for Mixed Acid Etchants

SEMI M59 — Terminology for Silicon Technology

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

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

SEMI MF95 — Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer

SEMI MF110 — Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique

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

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

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

4.2 ASTM Standard³

ASTM D5127 — Standard Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

³ 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>

4.3 JEITA Standard⁴

JEITA EM-3509 — Sample Preparation Method for Minority Carrier Diffusion Length Measurement in Silicon Wafers by Surface Photovoltage Method

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

5 Terminology

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

6 Summary of Test Methods

6.1 Test Method A

6.1.1 The specimen surface is illuminated with chopped monochromatic radiation of energy slightly greater than the band gap of the semiconductor sample. Electron-hole pairs are produced and diffuse to the surface of the specimen where they are separated by the electric field of a depletion region to produce the SPV. The depletion region can be created by surface states, surface barrier, *p-n* junction, or liquid junction.

6.1.2 The SPV signal is capacitively or directly coupled into a lock-in amplifier for amplification and measurement.

6.1.3 The photon intensity is adjusted to produce the same value of SPV for all energies of the illuminating radiation.

6.1.4 The photon intensity at each selected energy is plotted against the reciprocal absorption coefficient for the energy.

6.1.5 The resultant linear plot is extrapolated to zero intensity; the (negative) intercept value is the effective diffusion length.

6.1.6 By using feedback from the detector to the light source, and a stepping motor for the monochromator, the procedure may be automated.

6.2 Test Method B

6.2.1 A surface photovoltage produced by chopped white light illumination is first measured for two different photon fluxes to ensure that the SPV is linear in photon flux.

6.2.2 Next, using monochromatic light produced by a set of narrow band filters at constant photon flux within the linear SPV range, the SPV is measured for a series of selected photon energies larger than the band gap of the semiconductor sample.

6.2.3 The reciprocals of the values of SPV that increase monotonically with photon energy are plotted against the reciprocal of the absorption coefficients corresponding to the selected photon energies.

6.2.4 The resultant linear plot is extrapolated to zero intensity; the (negative) intercept value is the effective diffusion length. The values outside the monotonic range are rejected from the analysis to eliminate interference from surface recombination effects.

6.2.5 A small area contact can be used to measure the SPV; by moving the test specimen under the probe, an area map of diffusion length can be made.

6.2.6 The procedure may be automated by using stepping motors for the filter wheel and stage; feedback to the light source is not required.

6.3 Test Method C

6.3.1 The specimen surface is illuminated with IR-radiation rectangular pulses.

6.3.2 The pulse SPV signal is capacitively coupled into a high resistively input amplifier and further into a digital oscilloscope.

⁴ Japan Electronics and Information Technology Industries Association, Ote Center Building, 1-1-3, Otemachi, Chiyoda-ku, Tokyo 100-0004, Japan; <http://www.jeita.or.jp>

6.3.3 The radiation pulse duration should exceed signal rising edge duration by a factor of three, and the time between pulses should exceed falling edge duration by a factor of three. For the correct registration of SPV impulse amplitude and shape the time constant of measuring circuit has to be significantly longer than the complete duration of this pulse by a factor of three.

6.3.4 Rising edge regression is performed by an exponential function to determine steady-state SPV.

6.3.5 The amplitude of this function is the steady-state SPV.

6.3.6 The steady-state SPV is determined for several values of IR-radiation irradiance.

6.3.7 The regression of dependency steady-state SPV on IR-radiation intensity by power function is performed and the derivative value of this function is determined at the point of origin.

6.3.8 The effective diffusion length is calculated from the derivative values at the point of origin for more than one wavelength.

6.3.9 The minority carrier diffusion length determination by method C allows highly automated measurements.

7 Apparatus

7.1 *Light Source and Monochromator or Filter Wheel* — Covering the wavelength range from 0.8 to 1.0 μm (energy range from 1.55 to 1.24 eV) with a means for controlling the intensity (variable AC or DC input, adjustable aperture, or neutral density filters). Both tungsten and quartz halogen lamps have been found to be suitable sources.

7.1.1 If a filter wheel is used (recommended for Test Method B), a minimum of six energies, approximately evenly spaced between 1.24 and 1.55 eV, is recommended. For Test Method B, the output photon flux (at the specimen) at each energy should be equal within $\pm 3\%$. In addition, for Test Method B, provision must be made for two neutral density attenuators to provide white light at two photon flux values with a ratio ϕ_1 to ϕ_2 known to 1%.

7.1.2 If a grating monochromator is used, a sharp cutoff filter that attenuates at least 99% of the light with wavelength shorter than 0.6 μm is required. In this case, calibrated interference filters are required to verify the wavelength calibration of the monochromator.

7.2 *Mechanical Light Chopper* — To operate at a frequency that is low enough to permit a steady-state distribution of carriers to exist in the specimen, low enough to be compatible with the response time of the detector (see Note 2), and high enough to permit effective coupling of the SPV signal into the amplifier.

NOTE 2: A frequency of about 10 Hz is recommended for most applications with Test Method A. Because Test Method B does not require a detector, this condition does not apply for Test Method B and higher frequencies can also be used.

7.3 *Optical Components* — To couple the illumination to the specimen and photon detector. A system of mirrors (or quartz lenses or both) or a system of fiber optic cables can be used. If mirrors or lenses are used, they should be arranged to focus an image of the exit slit on the chopper blade and on the specimen and detector (see Figure 1). In this case a wavelength-independent beam splitter is used to direct some the illumination to the detector; alternatively, the detector signal can be obtained by using the reflection from the back of the chopper blades. Fiber optic cables are preferred for use with Test Method B (see Figure 2).

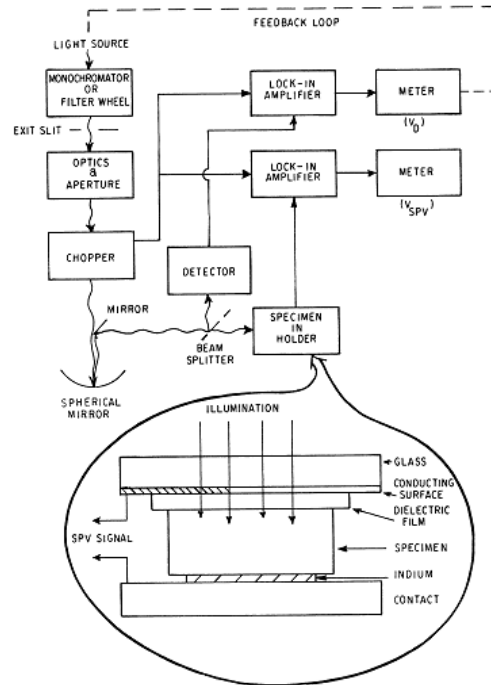


Figure 1
Block Diagram of SPV Equipment and Schematic of Specimen Holder for Capacitively Coupled Contact Set Up for Test Method A

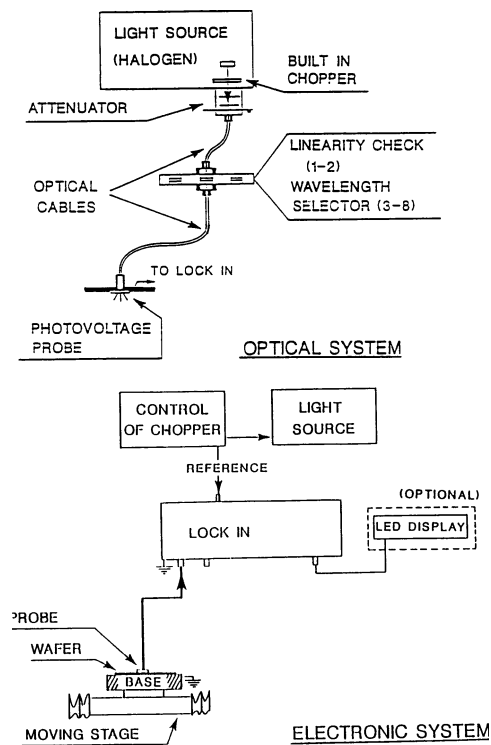


Figure 2
Block Diagram of Optical and Electronic Components of SPV Equipment for Test Method B

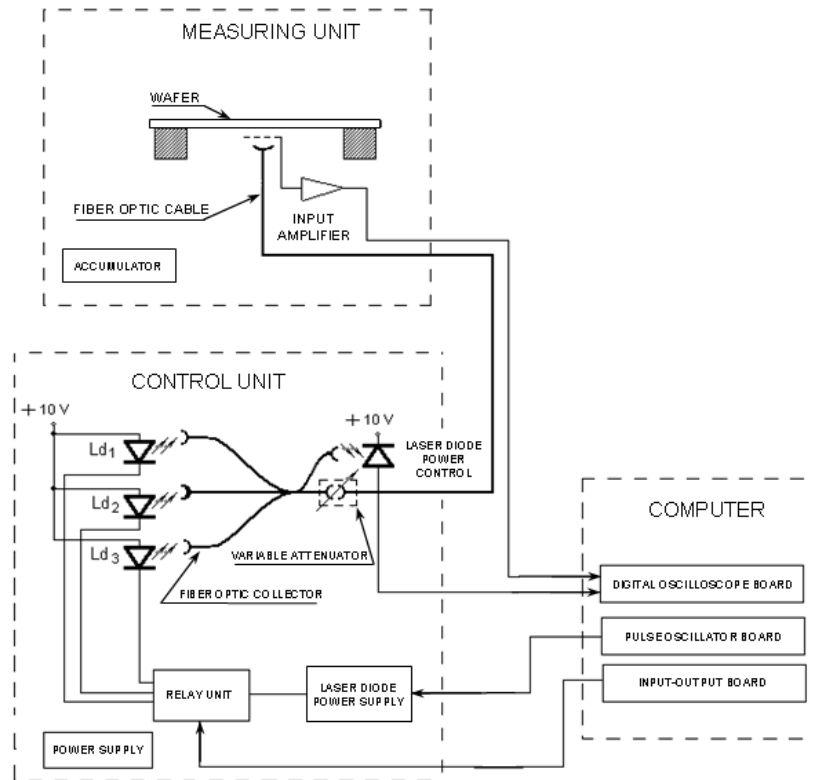


Figure 3
Block Diagram of Optical and Electronic Components of SPV Equipment for Test Method C

7.4 Photon Counter or Detector — With known relative spectral sensitivity (for Test Method A only). Absolute calibration is not required. A thermopile capable of operating at the chopper frequency is satisfactory. A silicon photodiode or pyroelectric detector can also be used.

NOTE 3: The detector calibration is simplified if the optical path to the detector includes a duplicate of the front contact structure of the specimen holder so that the optical paths to the detector and specimen are similar (see ¶ 3.8).

7.5 Specimen Holder — To support the specimen and to provide a transparent capacitively coupled front contact (a glass plate with a tin oxide coating and a 50 μm thick mica dielectric layer have been found to be satisfactory) and a reference potential contact to the back surface or to an unilluminated region of the front surface. For a surface barrier, p - n junction, or liquid junction, direct electrical connection to the illuminated surface of the specimen can be made in place of the capacitively coupled front contact. The holder may provide lateral and rotational motion of the specimen if the front contact covers only a small area of the front surface and if information on the area dependence of the diffusion length is desired.

7.6 Lock-In Amplifiers — Two, to measure the amplitudes of the SPV and detector signals (see Figure 1). A sensitivity of 1 μV full scale and an output noise level of less than 0.1 μV are required. An input impedance of 10 $\text{M}\Omega$ or higher is needed to match the high source impedance of the capacitively coupled specimen. Alternatively, a single dual-input lock-in amplifier can replace the two amplifiers if care is taken to prevent interference between the two signals. This alternative configuration is particularly appropriate for Test Method B (see Figure 2) since the photon flux need not be measured during the SPV measurements.

7.7 Conventional Laboratory Facilities — For cleaning, polishing, and etching specimens, if required.

7.8 Thermometer — Or other temperature measuring instrument, to determine the ambient temperature to $\pm 0.5^\circ\text{C}$.