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Standard Test Methods for Minority Carrier Diffusion Length in Extrinsic Semiconductors by Measurement of Steady-State Surface Photovoltage ¹

This standard is issued under the fixed designation F 391; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

- 1.1 These test methods 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.
- 1.2 These test methods are based on the measurement of surface photovoltage (SPV) as a function of energy (wavelength) of the incident illumination. The following two test methods are described:
- 1.2.1 *Test Method A*—Constant magnitude surface photovoltage (CMSPV) method.
- 1.2.2 *Test Method B*—Linear photovoltage, constant photon flux (LPVCPF) method.
 - 1.3 Both test methods are nondestructive.
- 1.4 The limits of applicability with respect to specimen material, resistivity, and carrier lifetime have not been determined; however, measurements have been made on 0.1 to 50 Ω ·cm n- and p-type silicon specimens with carrier lifetimes as short as 2 ns.
- 1.5 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.
- 1.6 These test methods also have been applied to the determination of the width of the denuded zone in silicon wafers.
- 1.7 These test methods measure diffusion lengths at room temperature (22°C) only. Lifetime and diffusion length are a function of temperature.
- 1.8 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the

responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water ²

- F 28 Test Method for Minority-Carrier Lifetime in Bulk Germanium and Silicon by Measurement of Photoconductivity Decay ³
- F 84 Test Methods for Measuring Resistivity of Silicon Wafer with an In-Line Four-Point Probe ³
- F 95 Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer ³
- F 110 Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique ³
- F 533 Test Method for Thickness and Thickness Variation of Silicon Slices ³
- F 673 Test Methods for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage ³
- 2.2 SEMI Standards:
- C 1 Specification for Reagents ⁴
- C 2 Specifications for Etchants ⁴

3. Summary of Test Method

3.1 Test Method A—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. The SPV signal is capacitively or directly coupled into a lock-in amplifier for amplification and measurement. The photon intensity is adjusted to produce the same

¹ This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved Feb. 10, 1996. Published April 1996. Originally published as F 391 – 73 T. Last previous edition F 391 – 90a.

² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 10.05.

⁴ Available from Semiconductor Equipment and Materials International, 805 East Middlefield Road, Mountain View, CA 94043.

value of SPV for all energies of the illuminating radiation. The photon intensity at each selected energy is plotted against the reciprocal absorption coefficient for the energy. The resultant linear plot is extrapolated to zero intensity; the (negative) intercept value is the effective diffusion length. By using feedback from the detector to the light source, and a stepping motor for the monochromator, the procedure may be automated.

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

4. Significance and Use

4.1 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 to the photoconductive decay (PCD) method covered by Test Method F 28 are excessively large. The CMSPV method (Test Method A) circumvents the influence of surface recombination on the lifetime measurement by maintaining constant front surface conditions while the LPVCPF method (Test Method B) utilizes only conditions and data points that are not influenced by surface recombination and other non-linear effects.

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 lifetime (1) ⁵ 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.

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

- 4.3 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.
- 4.4 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.
- 4.5 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 can not be interpreted as the diffusion length (see 5.2). Under certain circumstances, the intercept can be related to the layer thickness, providing a nondestructive means for determining the thickness of the layer.

5. Interferences

- 5.1 The quality of the measurement depends on the accuracy with which the absorption coefficient is known as a function of photon energy (wavelength).
- 5.1.1 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.
- 5.1.2 In heavily doped wafers, the free carrier absorption may affect the SPV measurement at long wavelengths.
- 5.1.3 The absorption coefficient is temperature dependent; the data given in these test methods are appropriate to room temperature only (22°C).
- 5.2 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.
- 5.2.1 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).
- 5.2.2 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.
- 5.3 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 will be nonlinear. The upper wavelength limit can be calculated before the measurement is made.
- 5.4 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 allowing sufficient time for the states to approach equilibrium under measurement conditions and then making all of the measurements as quickly as possible.

⁵ The **boldface** numbers in parentheses refer to the references at the end of these test methods.

- 5.5 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). A junction photovoltage can be eliminated by making the reference potential contact to an unilluminated region of the front surface.
- 5.6 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.
- 5.7 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.
- 5.8 For Test Method A, 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.
- 5.9 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.

6. Apparatus

- 6.1 Light Source and Monochromator or Filter Wheel, covering the wavelength range from 0.8 to 1.0 μ m (energy range from 1.55 to 1.24 eV) with a means for controlling the intensity (variable a-c or d-c input, adjustable aperture, or neutral density filters). Both tungsten and quartz halogen lamps have been found to be suitable sources.
- 6.1.1 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 ± 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 %.
- 6.1.2 If a grating monochromator is used, a sharp cutoff filter that attenuates at least 99 % of the light with wavelength shorter than $0.6~\mu m$ is required. In this case, calibrated interference filters are required to verify the wavelength calibration of the monochromator.
- 6.2 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.
- 6.3 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 Fig. 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 Fig. 2).
- 6.4 *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 5.8).
- 6.5 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 areal dependence of the diffusion length is desired.
- 6.6 Lock-In Amplifiers, two, to measure the amplitudes of the SPV and detector signals (see Fig. 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 $M\Omega$ 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 Fig. 2) since the photon flux need not be measured during the SPV measurements.
- 6.7 Conventional Laboratory Facilities, for cleaning, polishing, and etching specimens, if required.
- 6.8 *Thermometer*, or other temperature measuring instrument, to determine the ambient temperature to ± 0.5 °C.
- 6.9 Computer Control System, (optional) with appropriate stepping motors to perform the appropriate calculations and control the wavelength selection, stage motion, and (for Test

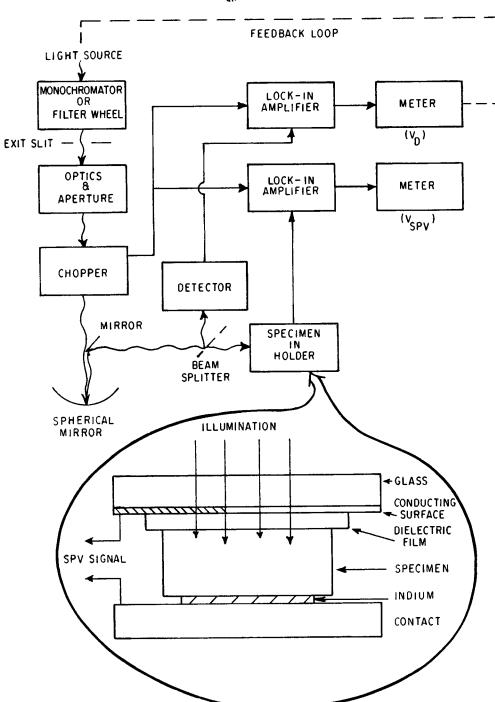


FIG. 1 Block Diagram of SPV Equipment and Schematic of Specimen Holder for Capacitively Coupled Contact Set Up for Test Method A

Method A) feedback from the detector to the light source, as required.

6.10 Low Level Light Source, coupled by fiber optic cable to the SPV system (see Fig. 2), consisting of a variable dc voltage control, incandescent lamp, and 800- μ m thick silicon filter.

7. Reagents

7.1 Purity of Reagents—Chemicals shall conform to the appropriate specifications in SEMI Specifications C 1 and C 2.

Other grades may be used provided it is first ascertained that the chemical is of sufficiently high purity to permit use without degrading the results of the test.

7.2 Purity of Water—Reference to water shall be understood to mean deionized water meeting the resistivity and purity specifications of Type I reagent water in Specification D 1193.

7.3 Etching Solution CP4A—5:3:3 mixed acid etchant in conformance with SEMI Specification C 2.1, for chemical polishing of silicon specimen surfaces (if necessary). To prepare, mix 50 mL of concentrated nitric acid (HNO₃), 30 mL

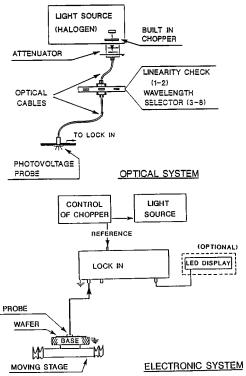


FIG. 2 Block Diagrams of Optical and Electronic Components of SPV Equipment for Test Method B

of concentrated hydrofluoric acid (HF), and 30 mL of glacial acetic acid (CH₃COOH).

7.4 Buffered Oxide Etchant—Mixture of 40 % ammonium fluoride solution (NH $_4$ F) and concentrated hydrofluoric acid (HF) in conformance with SEMI Specification C 2.2, for use in improving the SPV signal in p-type silicon specimen surfaces, if required.

Note 4—A mixture of 6 parts NH₄F and 1 part HF, by volume, has been found to be satisfactory for this purpose.

7.5 Hydrogen Peroxide (H_2O_2) —30 %, unstabilized, in conformance with SEMI Specification C 1.9, for use in improving the SPV signal in n-type silicon specimen surfaces, if required.

8. Hazards

8.1 The acids in the etchants used for chemically preparing or treating the specimen surfaces (when such preparation or treatment is required) are extremely hazardous. All precautions normally used when handling these chemicals should be strictly observed.

NOTE 5—**Precaution:** Hydrofluoric acid solutions are particularly hazardous. Make sure that the user is familiar with the specific preventive measures and first aid treatments given in the appropriate Material Safety Data Sheet.

9. Specimen Preparation

- 9.1 Epitaxial and stress-relieved chem-mechanically polished wafers can usually be measured in the as-received condition.
- 9.2 Surfaces of stress-relieved sawed or lapped single crystal silicon specimens require either chemical polishing with an etch such as Etch Solution CP4A (see 7.3) or chem-mechanical polishing to remove any surface mechanical damage.

- Note 6—If the SPV signal is low, it can often be increased by a treatment that enhances the depletion layer. For n-type silicon, a useful treatment consists of boiling the specimen in $\rm H_2O_2$ for about 15 min. For p-type silicon, a suitable treatment is an etch in buffered HF (see 7.4) for 1 min
- 9.3 Solar cells can be measured in the as-received condition provided that the top layer is thin enough that significant carrier generation is not induced by the illumination.
- 9.4 A liquid junction with a transparent electrolyte can be used (3).
- 9.5 If a thin metal surface (Schottky) barrier is used as the front contact, the optical behavior of the metal must be characterized so that the appropriate correction can be made to obtain the relative internal photon flux.

10. Calibration

- 10.1 The wavelength (energy) of the illumination must be accurately known. Calibrated interference filters provide a convenient means of checking wavelength calibration of a monochromator; if a filter wheel is used, the wavelength of each filter must be known or determined.
- 10.2 For Test Method A, the wavelength (energy) dependence of the photon detector response, if any, must be known or determined. However, absolute calibration of the photon detector is not required.

TEST METHOD A—CONSTANT MAGNITUDE SURFACE PHOTOVOLTAGE (CMSPV) METHOD

11. Procedure

- 11.1 Turn on the light source, chopper, and lock-in amplifiers, and align the optical system using visible light from the monochromator or filter wheel. If a grating monochromator is used, remove the sharp cut-off filter and use a higher order diffraction mode in the visible range.
- 11.2 Set the monochromator or filter wheel to the shortest wavelength (highest energy) to be used, usually 0.8 μ m (1.55 eV).
- 11.3 Adjust the illumination intensity to about half maximum power or 70 % of maximum amplitude.
- 11.4 Mount the specimen in the specimen holder and bring the capacitative or other front contact into the measurement position.
- 11.5 Adjust the frequency and phase of the lock-in amplifier connected to the specimen for maximum signal. If the same amplifier is used for both the specimen and detector, adjust the phase as needed before each reading.
 - 11.6 Note the approximate SPV signal amplitude, V_{SPV} .
- 11.7 Set the monochromator or filter wheel to the longest wavelength (lowest energy) for which data are desired (usually 1.04 μm (1.19 eV) in bulk silicon specimens and 1.0 μm (1.24 eV) in (epitaxial silicon). For specimens with short diffusion length (<20 μm), the longest wavelength may be 1.0 μm (1.24 eV) or less.
- 11.8 Change the illumination intensity to obtain a convenient value of V_{SPV} for the series of measurements. The preferred value is near that noted in 11.6.
- 11.9 Reset the monochromator or filter wheel to the shortest wavelength (highest energy) and reset the intensity to produce the chosen value of V_{SPV} .

- 11.10 Record the value of λ and V_{SPV} .
- 11.11 Read and record the signal level, V_D , from the photon detector.
- 11.12 Increase λ (decrease photon energy) in increments to the maximum λ (minimum energy) desired. For silicon, suitable steps are 0.85, 0.90, 0.95, 0.97, 0.99, 1.00, 1.01, 1.02, 1.03, and 1.04 μm (1.46, 1.38, 1.31, 1.28, 1.25, 1.24, 1.23, 1.22, 1.20, and 1.19 eV). For short diffusion lengths, use only the shorter values of λ (higher values of energy).
- 11.13 Adjust the illumination intensity at each wavelength (energy) to produce the chosen value for V_{SPV} .
- 11.14 Read and record λ (or photon energy), V_{SPV} , and V_D for each value of λ (or photon energy).
- 11.15 Measure and record the specimen thickness in accordance with Test Method F 533. For an epitaxial layer, also measure and record the layer thickness in accordance with Test Method F 95 or F 110.
- 11.16 Optionally, measure the specimen resistivity in accordance with Test Methods F 84 or Test Method F 673.

12. Calculation

12.1 Determine the reciprocal absorption coefficient, α^{-1} , either by direct measurement or from the following relation (4):

$$\alpha^{-1}(\lambda) = (84.732/\lambda - 76.417)^{-2}$$

(1)

where absorption coefficient, α is in cm $^{-1}$, and wavelength, λ , is in μ m.

- 12.2 Determine the relative photon intensity, I_o , in arbitrary units, from V_D for each value of wavelength (energy), correcting for any wavelength dependent losses.
- 12.2.1 If a thermopile of constant energy sensitivity at all wavelengths is used as the photon detector, and if there are no wavelength dependent components in the detector path, determine I_o as follows:

$$I_o = k\lambda \cdot V_D \tag{2}$$

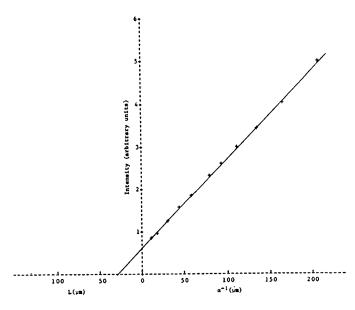
where: k is an arbitrary constant that can be taken as unity. 12.2.2 If the photon detector has a wavelength-dependent sensitivity, apply the appropriate correction factor at each wavelength used.

12.3 Calculate the term (1 - R) from the following relation that is valid for the case of polished silicon surfaces with thin native oxide in the wavelength range from 0.7 to 1.05 μ m (5):

$$(1 - R(\lambda)) = 0.6786 + 0.03565\lambda^{-1} - 0.03149 \lambda^{-2}$$
 (3)

where: λ = the wavelength in μ m.

- 12.3.1 For other materials or if any surface films are present on silicon, (Eq 3) is not valid; determine the term (1 R) by direct measurement.
- 12.4 Calculate the product $I_o \cdot (1 R)$ at each wavelength used and plot this product against α^{-1} . Fit a straight line to the points visually or calculate a least squares fit to the data. Extrapolate the line to the (negative) abscissa and measure and record the magnitude of the intercept on the negative abscissa (see Fig. 3).
- 12.5 The magnitude of this intercept is the effective diffusion length, L_o . If L_o is less than one fourth the specimen



	Reciprocal	
T C Voltage,	Coefficient,	
μV	μm	Intensity
8.40	206.329	5.854
6.85	164.864	4.725
5.90	134.306	4.028
5.20	111.159	3.513
4.55	93.225	3.041
4.10	79.063	2.711
3.35	58.450	2.168
2.90	44.494	1.835
2.40	30.797	1.467
1.95	18.032	1.122
1.85	11.269	0.997
	μ V 8.40 6.85 5.90 5.20 4.55 4.10 3.35 2.90 2.40 1.95	Absorption T C Voltage,

 $L_{\rm o} = 28.9236~\mu m$, Sigma = 0.9716 μm , Specimen: 20P850-32, Date: 12/27/71 SPV Signal = 2.5 mV.

Note 1—This plot is based on a previously used analytic approximation for the absorption coefficient (see Appendix X1).

FIG. 3 Typical Plot and Print Out of SPV Data Obtained Using Test Method A

(or epitaxial layer) thickness, $L_{\rm o}$ can be taken to be equal to the diffusion length, $L_{\rm D}$.

13. Report

- 13.1 Report the following information:
- 13.1.1 Specimen identification,
- 13.1.2 Data (λ , α^{-1} , V_{SPV} , and V_D) for each wavelength used.
 - 13.1.3 Ambient (room) temperature, and
- 13.1.4 Effective diffusion length, $L_{\rm o}$, and a statement as to whether this value is equal to the diffusion length, $L_{\rm D}$, in the layer or specimen.
 - 13.2 If desired, also report the following information:
 - 13.2.1 Specimen thickness.
 - 13.2.2 Epitaxial layer thickness (if appropriate), and
 - 13.2.3 Specimen resistivity (if determined).

14. Precision and Bias

14.1 An interlaboratory test was conducted in which six laboratories performed three determinations of effective diffusion length on each of six silicon specimens. Two of the

specimens were chem-mechanically polished wafers with diffusion length in the 100 to 200-µm range and four were epitaxial layers on more heavily doped substrates of the same conductivity type. In the case of the epitaxial specimens, the layer thickness was less than four times the measured effective diffusion length so that $L_o \neq L_D$. Data from one laboratory was excluded from the analysis because of deviations from the measurement procedure. Results are summarized in Table 1.

- 14.2 Although this test was based on earlier versions of Test Method A (Test Methods F 391, 1978 and 1984 editions) that utilized a different analytic approximation for the absorption coefficient (see Appendix X1) it is not expected that this would affect the estimate of precision.
- 14.3 Because there are no reference standards for diffusion length in silicon or other semiconducting materials, no statement regarding bias can be made.

TEST METHOD B—LINEAR PHOTOVOLTAGE, CONSTANT PHOTON FLUX (LPVCPF) METHOD

15. Procedure

- 15.1 Turn on the light source, chopper, and lock-in amplifier, and adjust the chopping frequency to the preselected value (for example, 13 Hz).
- 15.2 Place the test specimen on the specimen holder with the capacitative contact raised, and center the specimen on the holder.
- 15.3 Set the filter wheel to the high-intensity white light position. Verify that white light is passing through the system.
- 15.4 Lower the capacitative contact to contact the specimen surface.
 - 15.5 Adjust the lock-in amplifier for maximum signal.
- 15.6 Using an attenuator in the optical path, adjust the magnitude of the SPV signal to about 2 mV. Turn on the low

TABLE 1 Summary of Results of Interlaboratory Experiment to Evaluate Test Method A

A. Single-Laboratory Results

Bulk Specimens:

Sample Standard Deviation of Least-Squares Fit:

0.6 to 12 µm

(all 30 values were < 10 % of $L_{o\ avg}$)

Single-Laboratory Sample Standard Deviation:

0.4 to 14.4 um

(all 10 values were < 12.5 % of $L_{o\ avg}$)

Epitaxial Layers:

Sample Standard Deviation of Least-Squares Fit:

0.3 to 2.2µ m

(one value of 60 was > 10 % of $L_{o avg}$)

Single-Laboratory Sample Standard Deviation:

0.2 to 20 um

(three values of 20 were > 50 % of L_o avg; if these three values are excluded from the analysis, the upper limit of the range was 4.3 μ m)

B. Multilaboratory Results					
Sample Number	Diffusion Average Length, µm	Sample Standard Deviation, μ m	Relative Sample Standard Deviation, %		
Bulk 1	132	25	19		
Bulk 2	97	37	38		
Epi 1	10.9	4.8	44		
Epi 2	7.8	6.0	77		
Epi 3	31.8	10.5	33		
Epi 4	22.3	13.8	62		

level light source and adjust its intensity so that the SPV signal is about 1 mV.

- 15.7 Read and record the SPV signal amplitude as V_1 .
- 15.8 Set the filter wheel to the low-intensity white light position and read and record the SPV signal amplitude as V_2 .
- 15.9 Verify that the response is linear by taking the ratio of V_1 to V_2 . If this ratio is not within 5 % of the known ratio of ϕ_1 to ϕ_2 , reset the filter wheel to the high-intensity white light position and reduce the SPV signal amplitude to one half of its previous value by means of the attenuator and repeat 15.7-15.9.
- 15.10 Set the filter wheel to the position that gives the highest energy (shortest wavelength) illumination and reset the intensity to produce an SPV value approximately equal to V₂.
- 15.11 Reset the filter wheel to the position that gives the lowest energy (longest wavelength). Read and record the resulting value of SPV as V_3 and record the corresponding known wavelength as λ_3 .
- 15.12 Set the filter wheel to the next positions in order of increasing photon energy. Read and record the value of SPV at each position as V_4 , V_5 , V_6 , etc., which correspond to the known values of wavelength, λ_4 , λ_5 , λ_6 , etc.
- 15.13 Measure and record the specimen thickness in accordance with Test Method F 533. For an epitaxial layer, also measure and record the layer thickness in accordance with Test Method F 95 or F 110.
- 15.14 Optionally, measure the specimen resistivity in accordance with Method F 84 or Test Method F 673.

16. Calculation

- 16.1 Examine the sequence of SPV values and reject from further analysis any value V_n that is not greater than V_{n-1} .
- 16.2 If the specimen thickness is less than 500 μ m, reject any SPV values obtained for photon energy less than 1.24 eV ($\lambda > 1.00 \mu$ m).
- 16.3 For each wavelength used, determine the reciprocal absorption coefficient, α^{-1} , either by direct measurement or from the following relation (4):

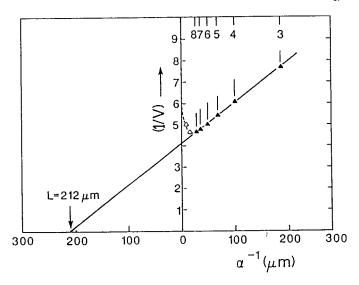
$$\alpha^{-1}(\lambda) = (84.732/\lambda - 76.417)^{-2}$$
(4)

where: the absorption coefficient, α , is in cm⁻¹ and wavelength, λ , is in μ m.

- 16.4 Plot the reciprocal of each SPV value against the value of α^{-1} for the wavelength of the illumination used to obtain the SPV value. Fit a straight line to the points visually or calculate a least squares fit to the data. Extrapolate the line to the (negative) abscissa and measure and record the magnitude of the intercept on the negative abscissa (see Fig. 4).
- 16.5 The magnitude of this intercept is the effective diffusion length, L_o . If L_o is less than one fourth the specimen (or epitaxial layer) thickness, L_o can be taken to be equal to the diffusion length, L_D . If L_o is greater than or equal to the specimen thickness, then find $L_{\rm real}$ according to Table 2, interpolating for non-standard wafer thicknesses if necessary. $L_{\rm real}$ can be taken to be equal to diffusion length, L_D .

17. Report

- 17.1 Report the following information:
- 17.1.1 Specimen identification,



Note 1—The numbers at the top of the graph refer to positions of the filter wheel that provides illumination at the appropriate energies and output photon flux (see 6.1.1).

FIG. 4 Typical Plot of SPV Data Obtained Using Test Method B

- 17.1.2 Data $(\lambda, \alpha^{-1}, \text{ and } V_n)$ for each photon energy used,
- 17.1.3 Ambient (room) temperature, and
- 17.1.4 Effective diffusion length, L_o , and a statement as to whether this value is equal to the diffusion length, L_D , in the layer or specimen.
 - 17.2 If desired, also report the following information:
 - 17.2.1 Specimen thickness,
 - 17.2.2 Epitaxial layer thickness (if appropriate), and
 - 17.2.3 Specimen resistivity (if determined).

18. Precision and Bias

18.1 A single operator has made single center-point measurements on five silicon specimens using four different systems of the same type. The measurements were made in accordance with this test method; in particular, the surface treatments listed in Note 6 were employed. These results (see Table 3 6) provide an estimate of the intralaboratory repeatability that might be expected from this test method when it is used by competent operators.

18.1.1 Two *n*-type and two *p*-type specimens with resistivity at room temperature of about 10 Ω -cm and minority carrier diffusion length (as determined by this test method) of between 130 and 310 μ m were measured. Sample standard deviations ranged from 2.1 to 5.0 μ m (0.7 to 2.2 %). Neither the sample standard deviation nor the relative sample standard deviation showed a correlation with minority carrier diffusion length.

18.1.2 The fifth specimen was n-type with room temperature resistivity of about 0.1 Ω -cm and minority carrier diffusion length of about 16 μ m. The sample standard deviation of the four measurements was 0.54 μ m (3.5 %).

TABLE 2 Diffusion Length Conversion Table

	Measured Values for Various Sample Thicknesses						
$L_{\rm real}$	T = 725 μm	T = 675 μm	T = 625 μm	T = 525 μm			
2000	550.6	505.7	460.8	371.3			
1950	549.9	505.2	460.3	371.1			
1900	549.1	504.5	459.9	370.9			
1850	548.2	503.9	459.4	370.6			
1800	547.3	503.2	458.8	370.3			
1750	546.3	502.4	458.2	370.0			
1700	545.2	501.5	457.6	369.7			
1650	544.0	500.6	456.9	369.3			
1600	542.7	499.6	456.1	368.9			
1550	541.3	498.5	455.3	368.5			
1500	539.8	497.3	454.4	368.0			
1450	538.1	496.0	453.4	367.4			
1400	536.2	494.5	452.2	366.8			
1350	534.2	492.9	451.0	366.2			
1300	531.9	491.1	449.7	365.5			
1250	529.4	489.1	448.1	364.6			
1200	526.5	486.9	446.4	363.7			
1150	523.4	484.5	444.5	362.7			
1100	519.9	481.7	442.4	361.6			
1050	515.9	478.5	440.0	360.2			
1000	511.4	475.0	437.2	358.7			
950	506.3	470.9	434.0	357.0			
900	500.5	466.2	430.4	355.0			
850	493.8	460.9	426.2	352.7			
800	486.2	454.7	421.4	349.9			
750	477.3	447.5	415.7	346.7			
700	467.0	439.1	408.9	342.8			
650	454.9	429.1	401.0	338.2			
600	440.8	417.4	391.5	332.6			
550	424.2	403.4	380.1	325.7			
500	404.6	386.8	366.3	317.2			
450	381.5	366.8	349.5	306.5			
400	354.3	342.9	329.0	293.0			
350	322.5	314.4	304.1	275.7			
300	285.8	280.6	273.9	253.6			
250	244.1	241.5	237.8	225.4			
200	198.3	197.4	195.9	190.1			
150	149.8	149.6	149.2	147.6			
100	100.0	99.98	99.96	99.82			
50	050.0	050.0	050.0	050.0			

TABLE 3 Results of Intralaboratory Experiment to Evaluate Test
Method B

Wafer Number Dopant		Resistivity, Ω·cm	Diffusion Length, µm as Measured by System Number			Mean, µm	Standard Deviation,		
			1	2	3	4		μm	%
A1	Phosphorus	10	140	141	134	139	138.5	3.11	2.2
T2	Phosphorus	0.1	15.9	14.9	16.1	15.4	15.6	0.54	3.5
G2	Phosphorus	10	280	280	282	277	279.8	2.06	0.7
D1	Boron	10	229	231	220	224	226.0	4.97	2.2
T4	Boron	10	306	309	308	302	306.3	3.10	1.0

18.2 This test method has not yet been evaluated by interlaboratory experiment to determine its interlaboratory reproducibility.

18.3 Because there are no reference standards for diffusion length in silicon or other semiconducting materials, no statement regarding bias can be made.

19. Keywords

19.1 diffusion length; minority carriers; polysilicon; silicon; single crystal silicon; surface photovoltag

 $^{^{6}\,\}mathrm{Supporting}$ data are available from ASTM Headquarters. Request RR:F01–1007.

APPENDIX

(Nonmandatory Information)

X1. PREVIOUSLY USED ANALYTIC EXPRESSIONS FOR ABSORPTION COEFFICIENT OFSTRESS-RELIEVED SILICON

X1.1 Earlier editions of these test methods utilized an analytic expression for the reciprocal absorption coefficient developed by Phillips (2) from the data of Runyan (6) as follows:

$$\alpha^{-1} = (0.526367 - 1.14425\lambda^{-1} + 0.585368 \lambda^{-2} + 0.039958 \lambda^{-3})^{-1} \mu m$$
 (X1.1)

where:

 λ = the wavelength of the incident illumination in μm .

X1.2 This expression has been widely employed in constant magnitude SPV measurements because it yielded a much more linear curve than the previous data of Dash and Newman (7) that appears to have been based on measure-ments made on nonstress-relieved specimens.

X1.3 However, since this expression was developed, a number of investigators have measured the absorption coefficient in the wavelength range relevant to SPV measurements. These results have been critically reviewed by Nartowitz and Goodman (4).

X1.4 This work and subsequent work by Saritas and McKell (8) have shown that the expression in Eq X1.1 overestimates α , especially in the region of the wavelength range above 0.9 μ m. Thus when this expression is used to determine α as a function of λ , the test method yields diffusion length values that are too low.

X1.5 Consequently, the use of Eq X1.1 in SPV measurements is no longer recommended.

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