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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Minority Carrier Diffusion Length in Extrinsic Semiconductors by Measurement of Steady-State Surface Photovoltage¹

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 $\Omega \cdot \text{cm}$ *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 Water²

F 28 Test Method for Minority-Carrier Lifetime in Bulk Germanium and Silicon by Measurement of Photoconductivity Decay³

F 84 Test Methods for Measuring Resistivity of Silicon Wafer with an In-Line Four-Point Probe³

F 95 Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer³

F 110 Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique³

F 533 Test Method for Thickness and Thickness Variation of Silicon Slices³

F 673 Test Methods for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage³

2.2 SEMI Standards:

C 1 Specification for Reagents⁴

C 2 Specifications for Etchants⁴

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

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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 μm (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 $\pm 3\%$. 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 %.

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 μm 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 Ω 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 $\pm 0.5^\circ\text{C}$.

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