

SEMI M89-0721

TEST METHOD FOR RECOMBINATION LIFETIME OF THE EPIAYER OF THE SILICON EPITAXIAL WAFER ($p/p+$, $n/n+$) BY THE SHORT WAVELENGTH EXCITATION MICROWAVE PHOTOCONDUCTIVE DECAY METHOD

This Standard was technically approved by the Silicon Wafer Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on March 9, 2021. Available at www.semiviews.org and www.semi.org in July 2021.

1 Purpose

1.1 In recent years, ULSI and other devices have been increasingly miniaturized and integrated. As a result, high-tech devices use more and more epitaxial wafers ($p/p+$, $n/n+$) which have substrates with lower resistivity, because epitaxial wafers are advantageous over other types of silicon wafers in several respects. For example, their features include integrity of active region of device, gettering capacity, resistance for latch up, etc. On the other hand, epitaxial wafers are feared to present serious problems in their manufacturing process, such as heavy metal contamination and defect formation in epilayers. Therefore, it is necessary to standardize the measurement method for recombination lifetime as a means of quality evaluation of epitaxial wafer contamination, and crystal defects etc.

2 Scope

2.1 This Standard specifies the measurement method for recombination lifetime in epilayer of silicon epitaxial wafer (epitaxial wafer; $p/p+[++]$, $n/n+[n++]$) by microwave photoconductive decay method using short wavelength excitation light source (the short wavelength excitation μ -PCD method).

2.2 For evaluation of an epilayer, it is necessary to confine excess carriers in an epilayer. Therefore, samples of the epitaxial silicon wafers should have $p/p+$ (++) or $n/n+$ (++) structure.

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 Although mechanical vibration is not limited for installation environment, the measurement result may be affected depending on the degree of mechanical vibration. Accordingly, caution is required.

3.2 The recombination (effective) lifetime is strongly influenced by the epi-thickness and the injection photon density. For mutual evaluation and data exchange between organizations, it is required to unify the measurement condition in consideration of accuracy of measurement.

3.3 With this technique, the contamination of the sample having an epilayer of 1 μm thick or more can be monitored using lifetime.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI M1 — Specification for Polished Single Crystal Silicon Wafers

SEMI M59 — Terminology for Silicon Technology

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

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

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

SEMI MF391 — Test Method for Minority Carrier Diffusion Length in Extrinsic Semiconductors by Measurement of Steady-State Surface Photovoltage

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

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

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

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

SEMI MF1388 — Test Method for Generation Lifetime and Generation Velocity of Silicon Material by Capacitance-Time Measurements of Metal-Oxide-Silicon (MOS) Capacitors

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

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

4.2 JIS Standards¹

JIS C 6802 — Safety of Laser Product

JIS H 0604 — Test Method for Recombination Lifetime in Silicon Wafers by Measurement of Photoconductivity Decay by Microwave Reflectance

4.3 JEITA Standards²

JEITA EM-3506 (00-KI-19) — The Measurement Method for Recombination Lifetime of the Epilayer of the Silicon Epitaxial Wafer (p/p+, n/n+) by the Short Wavelength Excitation Microwave Photoconductive Decay Method

NOTE 1: This JEITA standard is archived in the Japan Society of Newer Metals³.

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

5 Terminology

5.1 Most definitions of terms and acronyms used in silicon technology can be found in SEMI M59.

5.2 Abbreviations and Acronyms

5.2.1 μ -PCD — microwave photoconductive decay

5.3 Definitions

5.3.1 *1/e lifetime* (τ_e) — the time required for reflective microwave power to decay down to 1/e of the power at the time of optical pulse irradiation. For the short wavelength excitation μ -PCD method, it is the quantity which reflects not only the crystallinity and surface conditions of a sample but also injection photon density.

5.3.2 *1st mode lifetime* (τ_1) — time constant of decay obtained from such decay part (1st mode part) that is deemed to be the exponential function of reflective microwave power. For the short wavelength excitation μ -PCD method, it is the quantity which reflects the crystallinity and surface conditions of a sample.

5.3.3 *differential μ -PCD method* — the method that mount the microwave waveguide for signals equipped with the excitation light source that generates an optical pulse and the microwave waveguide for reference, and detect variation of excess carriers from the difference of the microwave power of both waveguides.

¹ Japanese Standards Association, 4-1-24 Akasaka, Minato-ku, Tokyo 107-8440, Japan; Telephone: +81.3.3583.8005, Fax: +81.3.3586.2014, <http://www.jsa.or.jp>

² 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>

³ Toranomonendenki Bldg., 2-8-1, Toranomon, Minato-ku, Tokyo 105-0001, Japan; Telephone: +81.3.3591.0389, Fax: +81.3.3591.0340, <http://www.jsnm.or.jp/jeita/>

5.3.4 *effective lifetime (τ_{eff})* — the lifetime that is directly obtained from measurement. For the μ -PCD method, it is obtained as a time constant of decay of reflective microwave power. It is the quantity which reflects the crystallinity and surface condition of a sample but also injection photon density. Especially for measurement of epilayer lifetime, it is the quantity which also depends significantly on the epilayer thickness. The following two kinds are mainly used according to the decay part to be adopted.

5.3.5 *excess carrier* — the electrons and holes that are generated by an optical pulse in the sample.

5.3.6 *injection photon density* — the number of photons that are injected by an optical pulse, per unit area ($/\text{cm}^2$). It may be called the carrier injection density.

5.3.7 *p+, p++ and n+, n++* — generally, *p+* and *n+* refer to the substrate with resistivity of $0.05 \Omega \cdot \text{cm}$ or less, while *p++* and *n++* refer to the substrate with resistivity of $0.01 \Omega \cdot \text{cm}$ or less in most cases.

5.3.8 *recombination* — phenomenon in which excess carriers are recombined and disappear.

5.3.9 *surface recombination* — phenomenon in which excess carriers are recombined and disappear on the front or back surface of a sample.

5.3.10 *μ -PCD method* — the method to measure the photoconductive decay by microwave that follow generation of excess carries by an optical pulse.

6 Summary of Test Method

6.1 When an optical pulse with higher photon energy of silicon epilayer is irradiated on the epilayer, it generates surplus free electrons and free holes (excess carriers) in the conduction band and in the valence band, respectively. If the penetration length of the light is shorter than the thickness of the epilayer, and if the resistivity of the substrate is lower than the epilayer as in the cases of *p/p+* (++) or *n/n+* (++) structure, then a potential barrier between the epilayer and the substrate prevents the diffusion of the excess carriers into the substrate. The excess carriers confined in the epilayer will decay mostly by recombination processes via levels formed in the forbidden band of silicon formed by impurities as metallic contamination or crystal defects. This process can be monitored by the changes of reflectivity of irradiated microwaves. Analysis of the shape of microwave reflectivity decay will give a recombination lifetime of the epilayer (μ -PCD method). If the concentration for impurity levels increase, then the recombination lifetime decreases. Therefore, if the recombination lifetime is obtained by performing the test specified in this Standard, the result will give the degree of metallic contamination and crystal defects in an epitaxial wafer.

6.2 In this Test Method, however, if the resistivity of a substrate is low like *p/p+* (++) or *n/n+* (++) wafer, and if an epilayer is thin, then the measured recombination lifetimes become short, and the sensitivity for contamination will become significantly low. The following two mechanisms are possible.

6.3 The volume density of excess carriers in an epilayer increases according as the epilayer becomes thinner, causing non-linear recombination to excess carrier density. Moreover, excess carriers go over potential barrier at the epilayer/substrate interface, flowing into a substrate. These excess carriers recombine rapidly via the recombination centers formed by the high-concentration dopants in highly doped substrate. As a result, the total lifetime observed becomes short.

6.4 The interface levels at the epilayer/substrate interface serves as recombination centers. A certain percentage of the excess carriers which reached the interface by diffusion, recombine, and disappear. For this reason, if an epilayer is thinner, then an observed lifetime is shorter.

6.5 This Test Method cannot generally identify impurity elements. But it can identify some specific impurity elements within a limited range. Moreover, mapping measurement of a sample can sometimes infer the origin of contamination induced in manufacturing process of semiconductors.

6.6 A measuring device is installed in the environment of room temperature ($23 \pm 3^\circ\text{C}$), humidity of 30% to 70% and atmospheric pressure of 860 hPa to 1060 hPa.

7 Apparatus

7.1 A measuring device for recombination lifetime of epitaxial wafer is shown in Figure 1. A measuring device consists of a pulse light irradiator, microwave irradiator/detector, wafer stage, analyzer, and controller/processor. Each component shall have the performance specified below. Conductivity of a sample shall correlate with the reflective

microwave power which is received by the microwave irradiator/detector in a monotonous increase or a monotonous decrease. For that purpose, the phase of microwave is required to be adjusted.

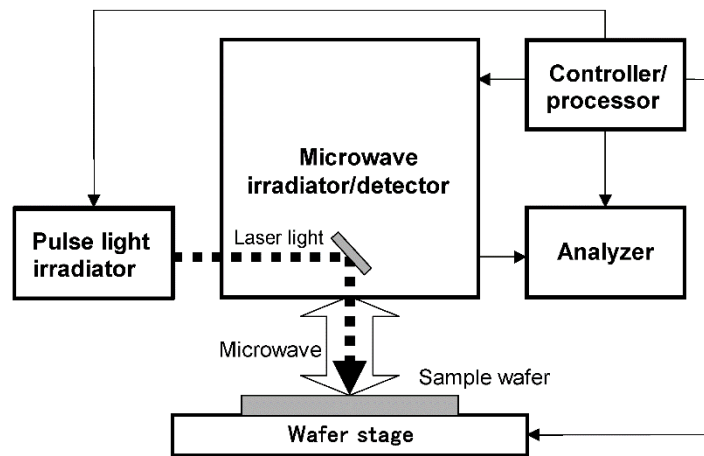


Figure 1
Block Diagram of Measuring Device for Recombination Lifetime of Epitaxial Wafer

7.2 Pulse Light Irradiator — Unit which has the laser whose wavelength is 330 to 550 nm. The pulse width shall be 10 ns or less. The injection photon density shall be $5 \times 10^{15}/\text{cm}^2$ or less.

NOTE 2: Generally, laser with a wavelength of 523 nm is used. Although the laser of other wavelength may be used, recording and reporting are required in that case.

NOTE 3: Generally, injection photon density as $2 \times 10^{13}/\text{cm}^2$ is used. When the injection photon densities other than this density are used, recording and reporting are required.

7.2.1 A pulse light irradiator shall have a cover to prevent direct sight of laser light, and an interlock device for cover opening and closing motion (while an interlock is canceled, a pulse light irradiator shall not be operated).

7.3 Microwave Irradiator/Detector — Unit which has the microwave source whose frequency is 10 to 100 GHz and measures the reflective power at the sample surface.

NOTE 4: Generally, the differential μ -PCD detection system which has a microwave source with a frequency of 26 GHz is used. Although the other microwave frequency and detection systems may be used, recording and reporting are required in that case.

7.3.1 A microwave detector keeps the intensity of reflective microwave leaking from the detector to outside at the level of $1 \text{ mW}/\text{cm}^2$ or less.

7.4 Wafer Stage — Unit which holds a sample at the desired X and Y position relative to the source of a pulse light.

7.4.1 A wafer stage may have the heater which controls the temperature of a sample.

7.4.2 A wafer stage may have a computer-assisted X - Y or the R - θ movement mechanism which enables mapping on the sample surface. It may also have a loader/unloader and a transfer mechanism which measure some samples sequentially.

7.5 Analyzer — It shall have an appropriate signal processor and a display unit. A sampling interval shall be 1/100 or less of the measured lifetime.

NOTE 5: If the sampling intervals are equal to and more than 1/100, then recording and reporting are required.

7.6 Controller/Processor — Although manual measurement is possible, it is desirable to use an appropriate computer-assisted controller/processor in order to control the following: loading/unloading of a sample, stage movement, operation of pulse light source and detector, analysis of decay signal, statistical analysis of data, recording and storage of data, and printing or plotting of results.

7.7 Inspection of Device — When the same position of a standard wafer is measured 10 to 20 times, it shall be confirmed that the repeatability of the peak value of a signal shall be 10% or less and that the repeatability of lifetime is 5% or less.

NOTE 6: Use the $p/p+$ (++) or $n/n+$ (++) wafer which has been controlled in the clean environment. An epilayer thickness should be 10 μm or more.

NOTE 7: (Maximum minus minimum of measured values)/(2 $\times$ averages).

8 Procedure

8.1 Sample

8.1.1 A sample is the epitaxial silicon wafer of $p/p+$ (++) or $n/n+$ (++) structure. For the thickness of the epilayers, refer to ¶ 8.1.2.

8.1.2 The laser described in § 7.2 has the Si penetration length of 1 μm or less. Therefore, it is theoretically possible to measure the lifetime of the sample having an epilayer of 1 μm thick or more. However, measurement of a thin wafer with an epilayer thickness of less than 5 μm is apt to result in the deterioration of the contamination detection sensitivity in epilayer. In this case, the data shall be checked carefully.

NOTE 8: The lifetime of the sample having an epilayer of lower than 1 μm cannot be applied. Thinner epilayer is not enough carrier and the lifetime is influenced by the epilayer interface.

8.1.3 Pretreatment

8.1.3.1 Surface recombination of excess carriers can be suppressed by heat treatments including oxidation in the following gas atmosphere.

- *Oxygen Gas* — Dew point -72°C or less (maybe from liquid oxygen, purity 99.8% or more).
- *Hydrogen Gas* — Purity 99.999% or more; dew point -110°C or less.
- *Nitrogen Gas* — Purity 99.999% or more; dew point -110°C or less.

8.1.3.2 In order to suppress surface recombination and to measure a recombination (effective) lifetime, it is necessary to perform thermal oxidation (950°C to 1050°C) which forms the oxide film with low oxide/epitaxial layer interface level density ($D_{it} < 10^{10}/\text{cm}^2\cdot\text{eV}$). The oxidation conditions shall be as follows. The oxidation treatment shall not produce the oxidation precipitation which might affect a recombination lifetime. Any metallic impurities shall not be included during the process. In order to prevent metallic impurities contamination, the surface of sample shall be cleaned before oxidation, to eliminate metallic impurities thereon. A heat treatment furnace and jigs shall be sufficiently clean.

NOTE 9: If the surface recombination can be suppressed to the extent that it does not affect measurement of the recombination lifetime of the epilayer of the sample, and if a stable surface can be obtained, then other passivation methods may be used. In that case, however, recording and reporting are required.

8.2 Loading of a Sample

8.2.1 A sample shall be loaded on a wafer stage so that the pulse light and microwave can be irradiated to the measuring position of the sample.

8.3 Irradiation of Microwave

8.3.1 Microwave shall be irradiated. Then, the analyzer shall perform sampling of the power of the microwave reflected by the sample. A recorder shall record the result.

8.4 Irradiation of Pulse Light

8.4.1 A pulse light irradiator shall irradiate pulse light to a sample.

9 Calculations

9.1 Analysis of Decay Curve and Calculation of Recombination Lifetime

9.1.1 A recorder shall record variation (decay curve: refer to Figure 2) of the power of the reflective microwave and give a recombination (effective) lifetime. In Figure 2, t_0 represents the moment that excess carriers are injected by

optical pulse into a sample. The time constant of decay obtained from such decay part that is deemed to be the exponential function of the decay curve, is the 1st mode lifetime (τ_1). The reflective microwave power V_A at t_A is supposed to decay by the recombination of excess carriers down to $1/e$ (≈ 0.368), and to reach V_B at t_B . Then, $\tau_1 = t_B - t_A$. Moreover, the reflective microwave power V_0 at t_0 is supposed to decay down to $1/e^2$ (0.135) and to reach V_2 at t_2 . Then, $t_1 - t_0$ is equal to $1/e$ lifetime (τ_e). Furthermore, when the deviation from an exponential function is not large, τ_1 can be replaced by $t_2 - t_1$.

9.1.2 The lifetime may be obtained from a single measurement of decay, but, if the signal-to-noise ratio (S/N) is not sufficiently high, it is recommended to repeat the measurement and average the decay curve. τ_1 may be obtained by fitting an exponential curve to the decay curve.

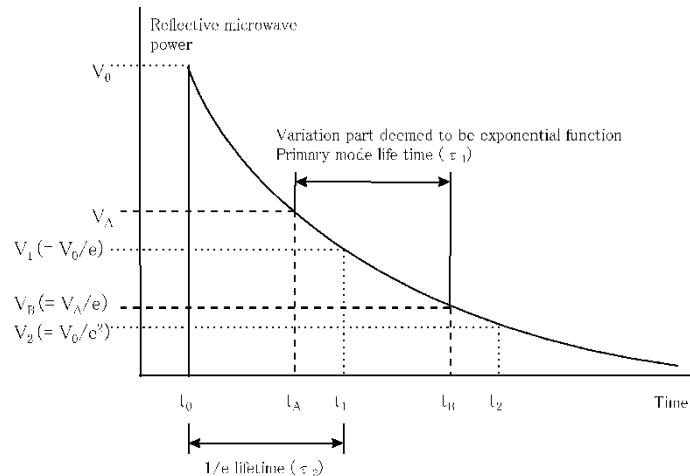


Figure 2
Decay Curve

10 Report

10.1 The following information shall be included in the report:

10.1.1 Test time and date.

10.1.2 Manufacturer and model of device.

10.1.3 Pretreatment (surface recombination suppression) method for sample.

10.1.4 Conditions of sample (epilayer thickness etc.).

10.1.5 Measurement result.

NOTE 10: Specify clearly whether the measured value is 1st mode lifetime (τ_1) or $1/e$ lifetime (τ_e).

11 Precision

11.1 A round robin test was carried out by JEITA using the short wavelength excitation μ -PCD method. The results of this 7-laboratories study are shown in Related Information 1. Table 1 shows the summary of variation between each laboratory for level of contaminated Mo concentration and epilayer thickness using %CV (a coefficient of variation) value. The variation for all conditions was around 10% or less of lifetime.

Table 1 Summary of Variation Between each Laboratory for Level of Contaminated Mo Concentration and Epilayer Thickness

<i>Sample</i>	<i>p/p+</i>
<i>Epi-Thickness</i>	<i>3 μm</i>
<i>Mo Contaminated Level</i>	<i>%CV Value</i>
low	12
medium	9.2
high	9.3
<i>Sample</i>	<i>p/p+</i>
<i>Epi-Thickness</i>	<i>5 μm</i>
<i>Mo Contaminated Level</i>	<i>%CV Value</i>
low	7.1
medium	3.7
high	9.5
<i>Sample</i>	<i>p/p+</i>
<i>Epi-Thickness</i>	<i>10 μm</i>
<i>Mo Contaminated Level</i>	<i>%CV Value</i>
low	4.8
medium	5.3
high	4.6
<i>Sample</i>	<i>n/n+</i>
<i>Epi-Thickness (μm)</i>	<i>%CV Value</i>
1.1	8.8
2.7	4.4
12	4.5

11.2 Also, the long term changing the measurement value was studied by JEITA round robin test which can be referred to in Table R1-2 of Related Information 1. As the result, the ratio of difference of average was around 10% or less for the samples of all levels which were compared with after 7 months values. After all, this result was found that the oxide on the specimen surface used in this experiment suppressed the surface recombination, resulting in the negligible secular change.