SEMI M46-1101[E] (Reapproved 0309)
TEST METHOD FOR MEASURING CARRIER CONCENTRATIONS IN EPITAXIAL LAYER STRUCTURES BY ECV PROFILING

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[E] This document was editorially modified in November 2001 to correct a typographical error. A change was made to ¶ 7.3.3.

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1 Purpose

1.1 The purpose of this document is to specify a method to measure the carrier concentration and carrier concentration vs. depth profile of epitaxial layers by Electrochemical Capacitance Voltage ECV profiling.

2 Scope

2.1 This test method covers a procedure for measuring the carrier concentration of epitaxial layers by ECV profiling. This method focuses on improving the accuracy and repeatability of the measurement by standardizing the test conditions and reporting and by routine calibration of the measurement.

2.2 This test method is intended to cover the majority of routine samples measured. However, because of the number of different materials encountered it cannot cover every contingency.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards and Documents

3.1 *SEMI Standard*

SEMI C1 — Guide for the Analysis of Liquid Chemicals

3.2 *ASTM Standard*[1]

ASTM D1125-95 (1999) — Standard Test Methods for Electrical Conductivity and Resistivity of Water

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

4 Terminology

4.1 *bias* — the potential applied to the sample with respect to a reference electrode.

4.2 *capacitance voltage CV measurements* — electrical measurements where the capacitance of a rectifying barrier is measured as a function of applied bias and is a measure of the net fixed ionized charge per unit volume.

4.3 *carrier concentration* — the net fixed ionized charge per unit volume. Equal to the free carrier concentration if the dopant is fully ionized and the material is free of traps.

4.4 *current voltage IV measurements* — electrical measurements where the current through the rectifying barrier is measured as a function of applied bias.

4.5 *dissipation factor* — the ratio of the real part to the imaginary part of the complex admittance. It is a measure of the non-ideality of the barrier.

4.6 *electrochemical* — chemical reaction in which charge transfer takes place via an external circuit.

4.7 *epitaxial layer* — a layer of single crystal semiconductor material grown on a host substrate which determines its orientation.

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4.8 *excess area* — the difference between the wetted and illuminated areas.

4.9 *flatband potential* — the intercept on the voltage axis of the $1/C^2$ vs. V plot. A measure of the built in field or barrier height.

4.10 *illuminated area* — the area of the sample which can be illuminated during electrochemical etching.

4.11 *rectifying barrier* — a potential gradient formed at the junction between two materials which permits the flow of charge in one direction only.

4.12 *reference electrode* — half cell which has a constant electrode potential, such as a saturated calomel electrode, SCE.

4.13 *rest potential* — the open circuit potential of the sample with respect to the reference electrode.

4.14 *wetted area* — the area of contact between the electrolyte and the sample.

5 Summary of Method

5.1 In this method, the carrier concentration of the epitaxial layer is measured by the CV method.

5.1.1 Ohmic contacts are formed on the sample.

5.1.2 A rectifying barrier, of known contact area, is formed by placing the sample in contact with an electrolyte.

5.1.3 The quality of the barrier is assessed using I-V and C-V measurements.

5.1.4 From the measured C-V data the carrier concentration of the layer is determined.

5.1.5 The sample is anodically etched to produce a new barrier deeper in the material. The etch depth is determined using Faraday's law of electrolysis.

5.1.6 ¶ 5.1.4 and ¶ 5.1.5 are repeated to generate a carrier concentration vs. depth profile.

6 Interferences

6.1 *Thin Layers* — Diffusion of carriers into adjacent regions set a fundamental resolution limit to the measurement. In addition if the layer has a high sheet resistivity this can also affect the value obtained.

6.2 *Double Layer Capacitance* — A limitation imposed by the physical nature of the electrolyte/semiconductor barrier which sets an upper limit to the measured carrier concentration, for accurate measurements, of 1×10^{19} cm^{-3}. The technique can be used beyond this value, but with progressive degradation.

6.3 *Etch Well Uniformity*

6.3.1 *Etch Well Flatness* — Non-uniformity increases the effective area and may result in the measurement not being wholly made in the layer of interest. The main causes of non-uniformity are gas bubbles, inadequate electrolyte circulation and uneven illumination.

6.3.2 *Etch Well Roughness* — Indicates poor etching and has a deleterious effect on the measurements. Generally overcome by selecting a more appropriate electrolyte and/or etching conditions.

6.4 *Non-Ideal Electrical Characteristics*

6.4.1 *Series Resistance* — From the contacts, sample and electrolyte affect the accuracy of the measurements. This effect is minimized by using a highly conductive electrolyte, making large area ohmic contacts and by employing a low measurement frequency.

6.4.2 *Leakage Currents* — Parallel conduction can affect the accuracy of the measurement. Its influence is reduced by employing a high measurement frequency.

6.4.3 *Large Excess Area* — Can give rise to errors on *n*-type material particularly when profiling Hi-Lo structures. The excess area is electrolyte dependent but is mainly affected by the quality of the seal used to define the area. Large excess areas indicate a poor seal and are corrected by replacing the seal.

6.5 *Hi-Lo Structures* — Measurement is subject to inaccuracy due to carrier diffusion and excess area errors. For accurate measurements, chemically etch to the layer of interest before measurement.

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6.6 *Contact Quality* — Non-Ohmic or high resistance contacts have a deleterious effect on the measurement. Contacts should be checked by measuring the resistance between two similar contacts and then reversing the current direction and repeating the measurement.

6.7 *Surface and Deep States* — Cause the measured capacitance to be frequency dependent and/or dependent on the rate at which the bias is changed. Usually resolved by using a high measurement frequency. This should not be confused with the frequency dependence which is more usually attributed to high series resistance.

6.8 *Surface Films* — Have a deleterious effect on the capacitance measurement. Film formation should be avoided by using an appropriate electrolyte, control of the etching bias and time and by electrolyte circulation.

6.9 *Surface Roughness* — The surface of the sample should be smooth and free of features that would affect the action of the seal.

6.10 *Light* — Light can affect the capacitance measurement, so the sample must be placed in a dark environment during the measurement.

6.11 *Sample Loading* — The accuracy of the measurement depends on the repeatability of the area. If a compliant seal is involved, such as a plastic ring, the sample should be held in contact with the seal by applying a controlled and constant force. No relative lateral movement should take place during the loading process.

6.12 *Ring Variability* — The condition of the ring has a large effect on the measurement. Good seals will have a small to zero excess area and exhibit a well defined and reproducible contact area.

7 Apparatus

7.1 *Measurement of Etch Well and Ring Areas* — Measuring microscope with either an XY stage, with a linear resolution of 0.01 mm or better, or a camera and image processing system capable of measuring an area of 0.1 cm^2 to better than 0.5%.

7.2 *Measurement of Etch Well Flatness* — (Optional) A means such as a stylus profiler for checking the flatness and roughness of the etch well. In most cases visual inspection of the etch well, for a mirror like appearance, is sufficient indication of low roughness.

7.3 *Instrumentation*

7.3.1 *Capacitance Meter* — Capable of measuring values up to 300 nF, employing a test signal of amplitude less than 100 mV RMS. The measurement frequency should be typically between 1 and 100 kHz.

7.3.2 *Equivalent Circuit Model* — The meter should assume a parallel circuit model for the electrolyte/semiconductor interface and provision should be made to compensate for the large series resistance of the electrolyte and contacts.

7.3.3 *Potentiostat* — Capable of maintaining the potential of the sample with respect to a reference electrode constant to ±5mV during the measurement and with a reference input impedance of $>10^{13}$ Ohms.

7.3.4 *Light Source* — A source of uniform illumination of above band gap energy for etching *n*-type materials.

7.4 *Sample Holder*

7.4.1 *Electrochemical Cell* — Used to hold the sample, contain the electrolyte and support the sealing ring and other electrodes, see Figure 1.

7.4.1.1 *Sealing Ring* — A corrosion resistant ring which forms a seal between the electrolyte and the sample. This ring defines the measurement area which should be reproducible and well defined. The ring area should not be less than 0.008 cm^2 and the reproducibility between runs should be better than ±3%. Large ring areas (typically ≥0.1 cm^2) are preferred for more accurate measurements.

7.4.1.2 *Reference Electrode* — Used to control the sample bias via a potentiostat.

7.4.1.3 *Counter Electrode* — The bias and test signals are applied between this electrode and the sample.

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