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Surface chemical analysis — Scanning probe microscopy — Standards on the definition and calibration of spatial resolution of electrical scanning probe microscopes (ESPMs) such as SSRM and SCM for 2D-dopant imaging and other purposes

Analyse chimique des surfaces - Microscopie à sonde à balayage - Normes sur la définition et l'étalonnage de la résolution spatiale des microscopes électriques à sonde à balayage (ESPMs) comme SSRM et SCM pour l'imagerie 2D-dopant et d'autres fins

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Foreword

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis,* Subcommittee SC 9, *Scanning probe microscopy*.

Introduction

Electrical scanning probe microscopy (ESPM) is a branch of scanning probe microscopy (SPM) with the capability of electrical imaging at nanometre spatial resolution. ESPM includes electrostatic force microscopy (EFM), scanning capacitance microscopy (SCM), scanning spreading resistance microscopy (SSRM), etc. Because ESPM can observe electrical or electronic properties with molecule-scale resolution, it is applied to many fields such as semiconductors, displays, etc. However, there has been no standard measurement method for the spatial resolution.

In this International Standard, standardized procedures to determine the spatial (lateral) resolution of SSRM and SCM, which are widely used to image the distribution of carrier and other electrical properties in semiconductor devices, are provided with the use of suitable reference materials. This International Standard uses the sharp-edge method to measure the lateral resolution of ESPM in a similar manner to that already used in measuring the resolution in micro-beam spectroscopy and in depth-profiling measurements with Auger electron spectroscopy and X-ray photoelectron spectroscopy (refer to ISO 18516).

Surface chemical analysis — Scanning probe microscopy — Standards on the definition and calibration of spatial resolution of electrical scanning probe microscopes (ESPMs) such as SSRM and SCM for 2D-dopant imaging and other purposes

1 Scope

This International Standard describes a method for measuring the spatial (lateral) resolution of scanning capacitance microscopes (SCMs) or scanning spreading resistance microscopes (SSRMs), which are widely used in imaging the distribution of carriers and other electrical properties in semiconductor devices. The method involves the use of a sharp-edged artefact.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115-2, *Surface chemical analysis – Vocabulary – Part 2: Terms used in scanning-probe microscopy*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-2 and the following apply.

3.1
electrical scanning probe microscopy
ESPM
SPM mode in which a conductive tip is used to measure electrical properties such as capacitance, resistance, electrical field, etc.

3.2
contact mode
mode of scanning the probe tip over the sample surface, adjusting the relative heights of the probe and sample, in which there is always a repulsive force between the probe and the sample

Note 1 to entry: This mode can be, for example, either the constant-height or constant-force mode.

[SOURCE: ISO 18115-2:2013, 6.35]

4 Symbols and abbreviated terms

AC	alternating current
DC	direct current
ESPM	electrical scanning probe microscopy
SPM	scanning probe microscopy
AFM	atomic force microscopy

MIS	metal-insulator-semiconductor
MOS	metal-oxide-semiconductor
SCM	scanning capacitance microscopy
SIMS	secondary ion mass spectroscopy
S/N	signal to noise ratio
SSRM	scanning spreading resistance microscopy
TEM	transmission electron microscope
2D	two dimension
Δx	spatial resolution of ESPM

5 General information

5.1 Background information

ESPM is a branch of scanning probe microscope that can be used to image an electrical or electronic property of a sample surface using an electrically conducting probe. Since this conductive probe is scanned over the sample surface in the contact mode, its lateral resolution is strongly related to the size and shape of the probe apex. Currently, this can be as small as a few nanometres, enabling sub-10 nanometre spatial resolution to be achieved. Such a high resolution, shown in ESPM images, allows the investigation of the two-dimensional distribution of carriers in nanoscale semiconductor devices.

5.2 Target

There are a number of types of ESPM categorized by the methods of electrical characterization. Among these ESPMs, this International Standard is for SCM and SSRM.

5.2.1 Scanning capacitance microscope

Scanning capacitance microscopy (SCM) is a modification of scanning probe microscopy in which a conductive probe is in contact with the surface of a sample, with an applied AC bias, and scanned across it. SCM characterizes the change in electrostatic capacitance between the sample and the probe on the surface of the sample. SCM uses an ultra-sharp conducting probe made from etched silicon (often coated with Pt/Ir or Co/Cr alloy) to form a metal-insulator-semiconductor (MIS/MOS) capacitor with a semiconductor sample if a native oxide exists on the sample. When the conducting probe is in contact to the surface under an AC bias, generated capacitance variations on the surface can be detected using a GHz resonant capacitance sensor. The probe is then scanned across the semiconductor's surface in *x*- and *y*-axes while the probe is operated under the contact mode.

By applying an alternating bias to the metal-coated probe or the sample, carriers are alternately accumulated and depleted within the semiconductor's surface layers under the probe, changing the tip-sample capacitance. The magnitude of this change in capacitance with the applied voltage gives information about the concentration of carriers (SCM amplitude data), whereas the difference in phase between the capacitance change and the applied, alternating bias carries information about the sign of the charge carriers (SCM phase data).[2]

5.2.2 Scanning spreading resistance microscope

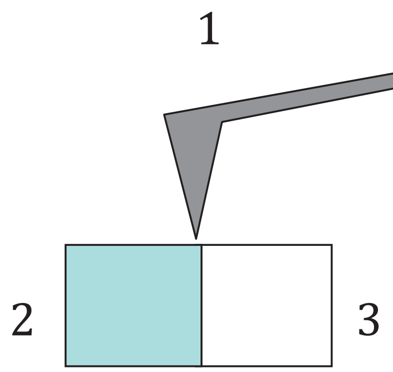
A very challenging task as the size of the semiconductor components shrinks towards sub-100 nm level is the development of new tools allowing two-dimensional (2D) carrier profiling with very high spatial resolution. One of the promising new tools is scanning spreading resistance microscopy (SSRM).

SSRM is based on atomic force microscopy (AFM) and has been developed in recent years to probe the 2D resistivity and carrier distribution in semiconductor devices. In SSRM, a very small conductive tip is contacted on the sample surface to be used to measure the local spreading resistance, which is intimately linked to the local resistivity. Scanning a cross section of the sample provides a 2D map of the local spreading resistance with a spatial resolution set by the tip radius (typically 5 nm ~ 15 nm). The main advantages of SSRM lie in its relative robustness, as it is less sensitive to surface preparation than, for instance, scanning capacitance microscopy (SCM) leading to excellent reproducibility. SSRM also benefits from an excellent dynamic range covering the entire dopant range of interest ($10^{14} \sim 10^{20}$) cm^{-3} with constant sensitivity and from a high spatial resolution (set by the tip radius only) combined with very accurate junction delineation capabilities.[3]

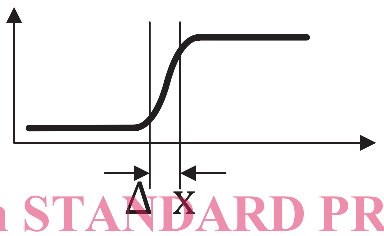
5.3 Measurement method for lateral resolution in SCM and SSRM

The spatial resolution is not only influenced by geometric factors of the conductive probe. Other factors that affect spatial resolution include surface roughness of the sample, contrast of the electrical image from difference in carrier density, pixilation, noise and sensitivity of the detector. The spatial resolution of the ESPM instrument or the image has been determined by a few methods: imaging a regular pattern and measuring the smallest feature and imaging across an electrically abrupt interface, etc.

It is very difficult to fabricate electrically separated layers with two different carrier density. Also, it is crucial to connect, electrically, each plane of the repetitive pattern or the smallest feature. Therefore, the method chosen here is the sharp-edge method based on consideration of ease of use. This method of resolution definition is widely applied for depth-profiling of micro-beam spectroscopy such as secondary ion mass spectroscopy (SIMS). An electrically abrupt interface is line-scanned perpendicularly across the interface by a conductive probe and the detected profile of electrical characteristics is inspected. In the micro-beam spectroscopy, so called 16 % to 84 % width or some other criterion may be applied as the spatial resolution of SCM or SSRM as shown in Figure 1.[1][4] However, the definition of the resolution as 10 % to 90 % width is adopted as a standard method for SCM and SSRM since it has been well agreed academically.[5][6]



a) Side view of the sample and probe



b) Electric output as a function of the scan position

Key

1 conductive probe

2 coating

3 silicon

Δx spatial resolution of ESPM

Figure 1 — Schematic of sharp-edge method applied to SCM and SSRM

In applying this method and to obtain high-resolution data, the following should be noted.

a) It is recognized that in SSRM, the resolution depends on the contrast. Therefore, the resolution should be compared at the same contrast level. A discussion of contrast levels is given in 7.3.

b) The resolution is also a result of mechanical probe-sample contact between the sample and probe. The mechanical probe condition and the mechanical contact may change continuously even for a good contact mode. In general, the probe apex shape, outer metal coating condition and the contact area continuously change, causing the signal to fluctuate. Also, the electrical property of the silicon or its coating will not be perfectly uniform. These inhomogeneities may increase the noise and uncertainty levels. Therefore, the property of calibration sample, the experimental condition, and the S/N ratio should be considered carefully to obtain the optimum result.

5.4 Key parameters in determining the lateral resolution

The measurement of lateral resolution can depend upon a number of experimental factors. They could be physical properties of the SCM or SSRM instrument and the sample surface, such as the conductive probe condition and the surface roughness. The following are the most important:

a) apex size of conductive probe: The apex size of the conductive probe is the most determining factor. Smaller probe sizes result in better spatial resolutions;

b) sample surface roughness: Sample surface roughness has a big effect on the measurement of spatial resolution in SCM or SSRM because the rougher surface changes more the contact between the probe and sample, both mechanically and electrically, generating apparent noise that affects the measured data;

c) contact force: A higher contact force makes the contact area wider and the resolution poorer;

d) signal contrast across the interface: A greater contrast gives a higher resolution value in SSRM; therefore, a signal contrast which exhibits at least 10 times of background noise level should be used in the experiment. If any signal saturates, an erroneously better value of the resolution could result.

6 Measurement of lateral resolution of SCM with the sharp-edge method

6.1 Background information

The sharp-edge method is frequently used in micro-beam techniques such as scanning electron microscopy (SEM) and SIMS to estimate the size of the focused beam on the sample surface.[1][4] An electrically abrupt interface is scanned by a conductive probe and the electrical signal profile across the interface is inspected. As the two regions have different electrical conductivities, the resultant SCM image can exhibit the interface region with spatially varying contrast. If a sectional analysis is applied to the interface region, a line profile with two plateau regions and a slope region between them is obtained. After the signal levels of the upper plateau and the lower plateau are normalized to be 1 and 0 as intensity values, respectively, the horizontal distance between the locations corresponding to intensity level of 0,1 and 0,9 is measured as the lateral resolution. In scientific publications, this criterion was generally used for the definition of spatial resolution of SCM or SSRM.[5][6] The concept of this method is straightforward and easy to implement. However, a difficulty is expected in preparing the sample having such abrupt interface without diffusion of dopants.

6.2 Selection of the sample

In this sharp-edge method, one needs a calibration sample with an electrically abrupt interface where the electrical contrast ideally shall change instantaneously as the interface is scanned by the conductive probe. In practice, there will be an “intermediate region” with a spatial gradient in electrical contrast between two regions of different electrical contrasts. This should be as small as possible and should be a fraction of the probe size.

In this International Standard, the fabrication procedure of the calibration sample is not specified because there may be a number of ways having their pros and cons. Therefore, this International Standard only describes the minimum requirements for the specification of the calibration sample.

Usually, a sample with the carrier density changing across an interface is used as an SCM or SSRM resolution calibration sample. For the best resolution, it is best to make the sample without diffusion. One way to do this is to use a doped silicon wafer on which 200 nm thick amorphous silicon layer is sputter-coated without diffusion, as shown in Figure 2, to make an electrically abrupt interface as fine as 1 nm or thinner. The capacitance or the AC capacitance variation of the coating of the reference sample is different from that of the substrate. The sample is cross-sectioned and polished to a roughness below 1 nm. The roughly polished backside of the polished sample is Pt-coated and later scratched deeper than the native oxide. The scratched surface is glued with GaIn and bonded to a metallic sample mount with silver paste. Then, both sides of the interface are electrically well connected to the sample mount.