Studies of MC2 capacitance standard sample by scanning microwave microscopy (SMM)

Introduction

Demand for nano-electrical characterization has been steadily growing over the last decade, driven by the continuous miniaturization of electronic devices. The knowledge about the local electric material properties, such as conductance, dielectric constant, local capacitance and dopant density, is of paramount importance for the semiconductor and microelectronics industry and research.

Multiple techniques were developed to aid these tasks, such as conducting atomic force microscopy (C-AFM), Kelvin probe force microscopy (KPFM), and other electrical AFM modes measuring local currents and potentials. However, none of the techniques offers measurements of buried structures, and modern integrated circuits are multilayered devices. A technique which allows such subsurface imaging, is essential for fault detection and optimization of manufacturing processes.

Scanning microwave microscopy (SMM) is a scanning probe technique, which combines an AFM with the measurement of local tip-sample microwave impedance, deduced from the S11 parameter, or microwave reflection coefficient[1]. The S11 parameter is a ratio between the microwave power sent to the tip, and the one received back after being reflected at the tip-sample contact. Through the complex impedance such a measurement yields information about the local capacitance and resistance. From the local capacitance one can deduce the dielectric constant and the dopant density in semiconductors. In terms of the dopant density measurement, the SMM is very close to the scanning capacitance microscopy (SCM), however the SMM offers wider range of measurements, since it is not solely relying on the modulation of the depletion capacitance in the sample, and therefore can be used for measurements of not only semiconductors, but also dielectric materials and metals.

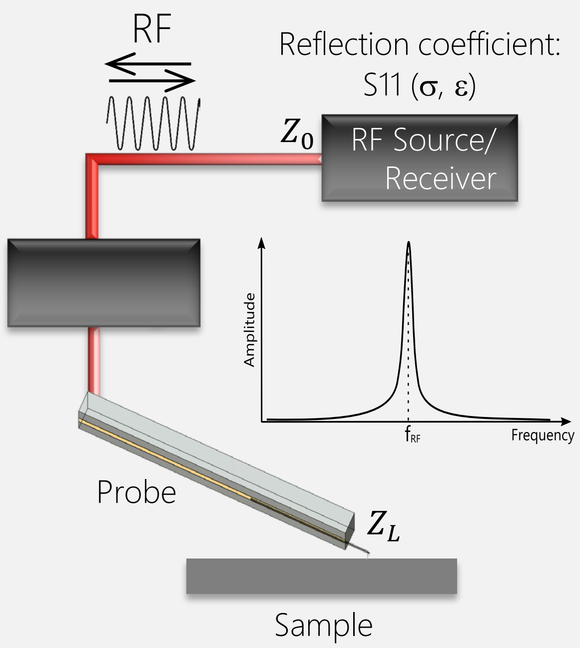

Figure 1. Schematic representation of a scanning microwave microscopy experiment. An RF signal is generated by the RF source and is being guided via a transmission line to a probe, which is in direct contact with a sample. The RF signal, reflected from the tip-sample contact with impedance Z_L, travels back to the electronics via the same path and is being registered by the receiver. The ratio of the incoming and reflected waves is called S11 parameter, which is a function of local electrical properties of the sample, in the place of tip-sample contact. Z_L is usually much larger than Z_0 – the characteristic impedance of the transmission line, therefore a matching circuit is required to prevent full reflection of the wave. Excitation and detection frequency is the same – f_{RF} (see inset).

SMM technique

A typical SMM setup consists of a radio frequency (RF) wave source and a receiver, working in the few GHz range. The source and receiver are usually integrated in the same housing, working on the same port - hence the measurement of the S11 parameter, where the first index and the second index mean the number of the receiving and the sending port[1]. The signal is transferred to the cantilever via a transmission line – a low loss coaxial cable. Because of the characteristic impedance of 50 Ω of the instrument and the much higher tip-sample contact impedance, there is also usually an impedance matching circuit between the tip and the instrument (Fig. 1).

In an S11 measurement, the transmitter and the receiver both work on the same frequency, and there is only one main tone in the frequency spectrum (see inset in Fig. 1).

Microwave basics

The S11 parameter is introduced as a ratio between the difference and the sum of impedances of the load Z_L and the reference Z_{ref} (Eq. 1):

$$S11 = \frac{Z_L - Z_{ref}}{Z_L + Z_{ref}} \qquad (1)$$

The reference impedance is usually 50 Ω, although in most of the calculations it can be arbitrary chosen and be as high as several kΩ. The load impedance is the impedance of the tip-sample contact and is a complex number with a real and imaginary part (Eq. 2) where j is the imaginary unit:

$$Z_L = Re(Z_L) + j \cdot Im(Z_L) \qquad (2)$$

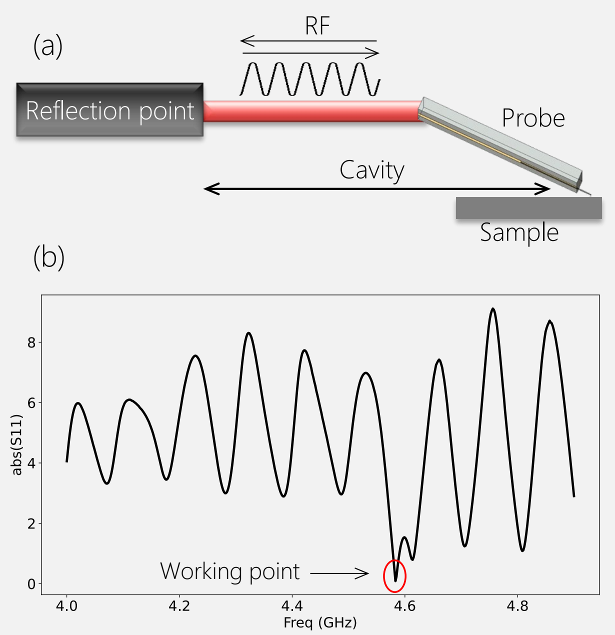

Figure 2. (a) Schematic representation of the reflection of an RF signal from impedance mismatch points and a corresponding frequency spectrum (b). The point in the spectrum with the lowest reflection is the natural working frequency for SMM. At this frequency smallest changes in tip-sample impedance would be the most noticeable.

The real part of the impedance is attributed to the resistance and the imaginary part to the inverse capacitance (Eq. 3):

$$Z_L = R + 1/j\omega_{RF}C \qquad (3)$$

An alternative way to operate is to work with microwave admittance instead of the impedance, then the real part is the conductance, and the imaginary – directly the capacitance. As a result of impedance being a complex number, the S11 parameter is also a complex number (Eq. 4):

$$S11 = Re(S11) + j \cdot Im(S11) \qquad (4)$$

or in the polar coordinates (Eq. 5):

$$S11 = |S11|e^{i\varphi_{S11}} \qquad (5)$$

Meaning that in the S11 measurement there are always two measured quantities, either the real and the imaginary part, or the

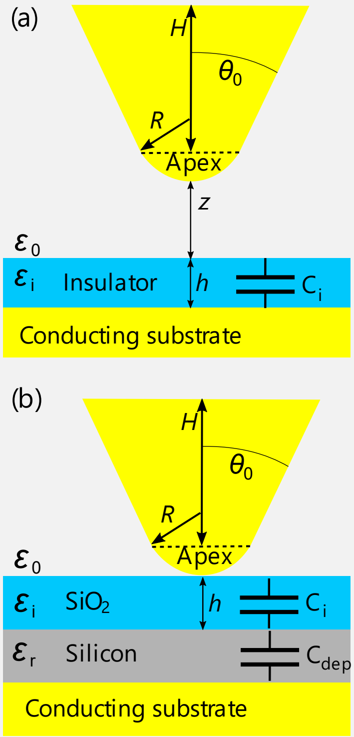

Figure 3. (a) Schematics of the parallel plate capacitor tip-sample geometry with an insulating film and conducting both tip and the substrate. Image adapted from Ref. 2. (b) Schematics of the tip-sample geometry in case of the semiconductor sample with the depletion capacitance C_{dep} and the parallel plate capacitor capacitance C_i connected in series.

amplitude and phase, and the calculations for the extraction of the sample parameters should be done with complex numbers.

From Eq. 1 and Eq. 3 if one plots the $Im(S11)(\omega_{RF})$ function at different capacitances, it becomes evident that the higher the ω_{RF} is, the smaller the smallest detectable capacitance is.

The tip-sample contact due to its high impedance is a natural reflection point for microwaves, and there are other reflection points in the transmission line. These form resonator cavities for microwaves, meaning that on the frequency spectrum of S11 there would be maxima and minima (Fig. 2). For setting up the SMM measurement, we would use the minima on this spectrum as a working point, because this is where the S11 is the most sensitive to changes in the tip-sample impedance.

Capacitance measurement

As we described earlier, the SMM measurement is an impedance measurement, with resistive and capacitive component. In many case the most informative part is the capacitance. In this section we will show, what data can be extracted from the tip-sample capacitance.

The first example is extraction of a dielectric constant of an insulating material. The capacitance of a parallel plate capacitor depends on the dielectric between the plates. As a model we consider a thin film of an insulating material sandwiched between the tip and a conducting substrate (Fig. 3). From the capacitance measurement of such a system, one could extract the dielectric constant of the insulator, using the following equation (Eq. 6)[2]:

$$C_i = 2\pi\varepsilon_0 R\, ln\left[1 + \frac{R(1 - sin\theta)}{z + h/\varepsilon_i}\right] \qquad (6)$$

where R is the tip radius, z – tip height above the surface (in contact equals to zero), h – thickness of the dielectric, and ε_0 and ε_i are dielectric constants of vacuum and dielectric correspondingly.

The analytical extraction of the dielectric constant from such measurement requires precise knowledge of the dielectric thickness, and a conducting substrate below. Often the researchers are facing bulk pieces of the dielectric material, or dielectric films of unknown thickness. In that case the measurement needs to be calibrated with 3 known standards[3]. SMM measurements of the dielectric constant can cover the range between 1 and 1000 and offer better precision than the measurements with electrostatic force microscopy (EFM)[3].

Another popular quantity to extract from the capacitance is the carrier density in semiconductor materials. The capacitance of interest in this case is the depletion capacitance of semiconductor. In a typical sample, this capacitance would be in series with the capacitance of the parallel plate capacitor, which we discussed above (Fig. 3). The depletion capacitance is a function of the carrier density (Eq. 7):

$$C_{dep} = \pi R^2 \sqrt{\frac{\varepsilon_0 \varepsilon_r n e^2}{k_B T}} \quad (7)$$

Where ε_r is the dielectric constant of semiconductor, n – carrier density, e – electron charge, k_B – Botzmann constant, and T is temperature.

In this case the equation for the total capacitance would be (Eq. 8):

$$C_{tip} = \frac{C_{dep} C_i}{C_{dep} + C_i} \quad (8)$$

It should be noted again, that the extraction of the C_{dep} is only possible if the C_i is known, and this is only possible, if the insulator thickness is known. Sometimes it is advisable to wash away the oxide from the surface, and grow a new layer in controlled conditions.

Calibration algorithm

While it is possible to extract the sample parameters from the SMM measurement purely by analytical methods, the calculations require precise knowledge about the sample parameters and are computationally extensive. In semiconductors it is also necessary to know which model for the carrier mobility to use. In practice, a much faster way to obtain quantitative data is calibrating the measurements on known standards[4].

The algorithm of the calibration is shown in Fig. 4. To understand how it works, we should understand that the S11 measured at the port of the receiver S_{11m} is not the same as the S11 at the tip-sample interface. These two measurement planes are connected by the SMM, which can be described with the three error parameters. The schematic on the left of the Fig. 4 shows the two planes, and the error parameters. The arrows on this schematic are the propagation of the microwaves. The e_{01} is responsible for the wave propagation from the source to the tip and back to the receiver, and the e_{00} and the e_{11} describe the internal reflections at the port and at the tip. The first step of the algorithm is to measure 3 known impedances and build a linear system of equations using the Maison's rule (Eq. 9 - 11):

$$S_{11m} = e_{00} + \frac{e_{01} S_{11}}{1 - e_{11} S_{11}} \quad (9)$$

$$\begin{pmatrix} 1 & S_{11}^1 & S_{11}^1 S_{m11}^1 \\ 1 & S_{11}^2 & S_{11}^2 S_{m11}^2 \\ 1 & S_{11}^3 & S_{11}^3 S_{m11}^3 \end{pmatrix} \begin{pmatrix} e_{00} \\ e_{01}^* \\ e_{11} \end{pmatrix} = \begin{pmatrix} S_{11m}^1 \\ S_{11m}^2 \\ S_{11m}^3 \end{pmatrix} \quad (10)$$

$$e_{01} = e_{01}^* + e_{00}\, e_{11} \quad (11)$$

the e_{01} needs to be replaced by the product of e_{00} e_{11} and a new parameter e_{01}^*, otherwise the system is non-linear and difficult to solve. Then after solving the system and finding the error parameters, we can apply the reverse operation and apply the correction to the measured S_{11m} to find the S11 at the tip-sample contact (Eq. 12, 13):

$$S_{11} = \frac{S_{11m} - e_{00}}{e_{01} + e_{11}(S_{11m} - e_{00})} \quad (12)$$

$$Z_{tip} = Z_{ref} \frac{1 + S_{11}}{1 - S_{11}} \quad (13)$$

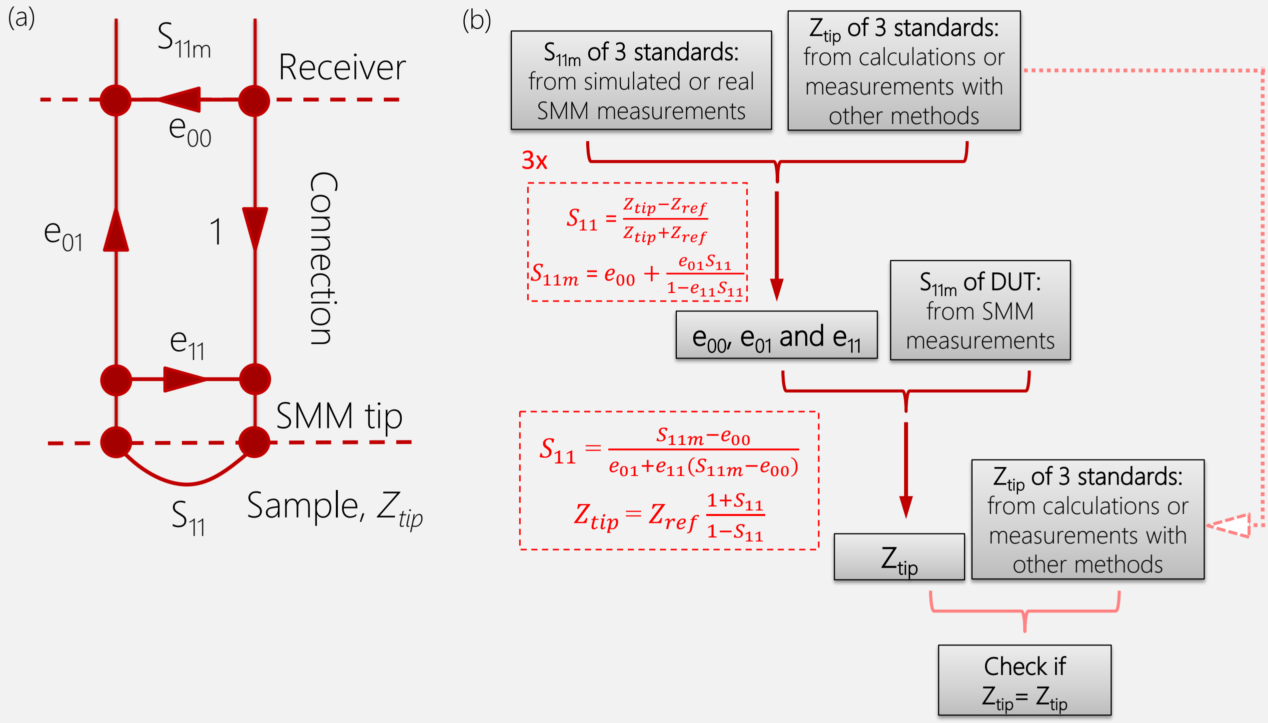

Figure 4. Calibration algorithm for extraction of tip-sample impedance from the S11 measurement. (a) Schematic representation of the with error coefficients and two different planes – the measurements plane of the receiver, and the plane of interest – between the tip and the sample. (b) The calibration algorithm, consisting of determination of error coefficients, and consequent calculation of the tip-sample impedance, using these error coefficients.

Then from the Z_{tip} one could find the resistance and the capacitance according to the Eq. 3.

Calibration example

Let us apply the calibration algorithm to a real sample. As a sample we would consider the capacitance calibration sample developed by NIST and sold by the MC2 Technologies (Fig. 5a). The sample consists of silicon oxide terraces of various heights and golden dots of different diameters deposited on these terraces. As we have learned from the previous sections, the dots of different diameters would have different capacitances, but also the dots of the same diameter, but on the terraces of different heights would also have different capacitances. To apply the algorithm to this sample we first have to analytically calculate the capacitances of three dots marked with red small circles and indicated with numbers 1, 2 and 3 on Fig. 5e. These calculations we would do similarly to Eq. 6, but instead of the tip radius we would use the radii of the dots, and use the calculated values as Z_{tip} from Eq. 13. In the calculation a series capacitance of two capacitors was considered. The first capacitor is formed by a top electrode made from a golden dot, and bottom electrode positioned at the interface between the SiO_2 and Si. The second capacitor is the depletion capacitance of Si substrate. The total capacitance is then calculated using the Eq. 7 and 8.

The capacitances were estimated to be 0.3, 3.6 and 12.4 fF. From here we can calculate the three S11 values, which we need for building the system of Eq. 10. As Z_{ref} we would take 10 kΩ. Once we solved the system and found the error parameters, we apply the algorithm in reverse (Fig. 4), and

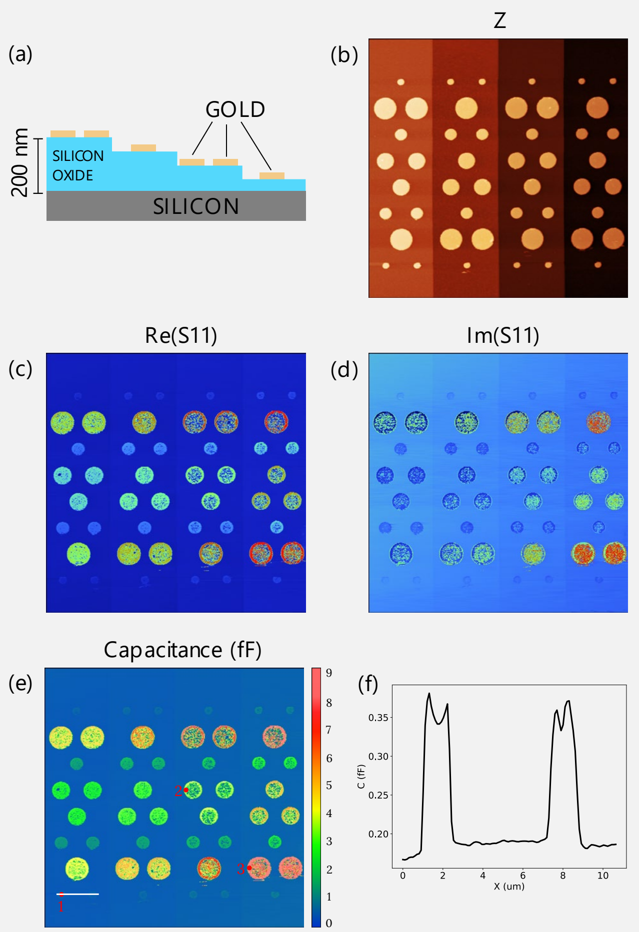

Figure 5. Example of calibration of S11 measurement of the MC2 Technology capacitance calibration sample. (a) Sample schematics showing 4 terraces, each 50 nm high, with patterned golden dots on top. (b) Topography of the sample, measured in contact mode. (c,d) Images of real and imaginary parts of S11 parameter. (e) Capacitance image, recalculated from the S11 parameter. Capacitors indicated with numbers 1, 2 and 3 were selected as calibration standards. Their respective capacitances were theoretically estimated as 0.3, 3.6 and 12.4 fF. These values vere used to calculate the capacitance map. (f) Cross section along the white line in (e), showing the contrast of a scan over two smallest capacitors.
The smallest capacitance change in the image – the step between the terraces is 0.04 fF.
Scan size: 52 x 52 µm².
(b) Range: 0.43 µm.

calculate the values of S11 for the whole image from the image of measured S_{11m}. And from there – the Z_{tip} for the whole image, which, in this case, is mostly determined by the capacitance (Fig. 5e). The values of the calculated capacitances are in good agreement with other measurements of this sample, made by other groups[5].

The data and the Jupyter notebook used in this application note are available for download on our website.

References

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