# Chapter 11 Dopant profiling in semiconductor nanoelectronics

# **11.1 Introduction**

As nanoelectronic device dimensions are scaled down to atomic sizes, device performance becomes more and more sensitive to the exact arrangement of atoms, including individual dopants and defects, within the device. Thus, there is ongoing demand for spatially-resolved measurements of dopant concentration. Due to the predominance of silicon-based micro- and nanoelectronics, the need for dopant profiling in semiconductors is particularly acute. This need has been underscored by the inclusion of dopant profiling in the *International Technology Roadmap for Semiconductors*: "Materials characterization and metrology methods are needed for control of interfacial layers, dopant positions, defects, and atomic concentrations relative to device dimensions. One example is three-dimensional dopant profiling" [1]. The ongoing development of alternative nanoelectronic devices based on emerging low-dimensional materials such as carbon nanotubes and graphene also will benefit from enhanced capabilities to identify and characterize dopants. Ideally, dopant profiling tools are non-destructive, exhibit nanometer-scale or better spatial resolution, and are sensitive to both surface and subsurface features.

A variety of scanning-probe-based, microwave techniques are excellent candidates for dopant profiling. The near-field scanning microwave microscope (NSMM), which we have described in detail throughout this book, is one such technique. An NSMM's combined capabilities to perform non-destructive, contact-free electrical measurements and to measure subsurface defects make it particularly attractive for dopant profiling. Other instruments, such as the scanning capacitance microscope (SCM), the scanning kelvin probe microscope (SKPM) and the scanning spreading-resistance microscope (SSRM) are also useful for characterizing semiconductors. Taken together, this suite of complementary scanning-probe techniques is capable of local measurement of work functions, surface barrier heights, dopant profiles, and resistance. To date, most dopant profiles obtained with scanning probe microscopes have been two-dimensional techniques, measuring only at or near the sample surface. In order to obtain the depth-dependent dopant concentration with such a technique, special sample preparation is required. For example, a cross-sectional sample of a device may be prepared via focused ion beam milling. However, the subsurface sensitivity of near-field scanning microwave microscopy makes the NSMM a strong candidate to provide quantitative, three-dimensional, subsurface resolution, as will be discussed in more detail in Chapter 12.

In Chapter 9, we reviewed a number of different, application-specific models of the tip-sample system, highlighting how such models are required for extraction of characteristic material parameters such as conductivity and complex permittivity. Thus, our discussion of dopant profiling begins with an overview of tip-sample models for semiconductor samples, including a brief review of some basic aspects of semiconductor physics. From there, we will detail approaches for performing quantitative dopant profiling with two radio frequency (RF)

scanning probe systems: scanning capacitance microscopy and near-field scanning microwave microscopy. These approaches require the development of calibration techniques and the selection of suitable reference samples. The chapter will conclude with a review of other scanning probe techniques that are used for spatially-resolved characterization of semiconductors. As these additional techniques are not RF- or microwave-based measurements, our discussion of them will be limited.

#### 11.2 Tip-sample models for semiconductor samples

#### 11.2.1 Capacitive models

Consider a contact-mode, scanning probe measurement of a semiconductor sample. Often, we will assume that the semiconductor is coated by a thin dielectric film. This film may represent a native oxide layer or a thin film that is deliberately deposited or grown on the sample. When the metal probe tip is in contact with the sample, the tip-sample system forms a metal-oxide-semiconductor (MOS) junction. Given the abundant knowledge about the physics of MOS systems, this representation suggests a natural approach to dopant profiling with scanning probe systems. However, the complexity of MOS physics and the deviations of real-world systems from ideal models require careful consideration. Throughout the remainder of this chapter, we develop dopant profiling techniques that assume idealized MOS structures. Though this approach has been successfully and widely applied to silicon, extension to other semiconductors will require a more complex physical description. For example, in the case of compound semiconductors such as GaN and AlGaN, interface states, trapped charges, and Fermi level pinning must be taken into account [2].

In order to perform dopant profiling, we will naturally focus on tip-sample models applicable to the case of semiconducting samples. We begin with a highly simplified picture of the interaction, approximating the system as a parallel plate capacitor. Then, we will present a metal-semiconductor model and a determination of the depletion layer capacitance. From there, we proceed to a more detailed description of the tip-sample junction in terms of a MOS capacitor junction. The simplest representation of a metal scanned probe interacting with an dielectric-covered, semiconducting sample is a parallel plate model, in which the two plates represent the probe tip and the semiconductor substrate, respectively. The parallel plate capacitance is

$$C = \frac{\varepsilon_0 \epsilon_r A}{d} \quad , \tag{11.1}$$

where  $\varepsilon_0$  is the permittivity of free space,  $\varepsilon_r$  is the relative permittivity of the intermediate dielectric, *A* is the area of the capacitor plates, and *d* is the separation between the plates. This simple model assumes uniform permittivity and uniform thickness of the oxide. In addition, this model ignores the tip shape as well as the semiconducting nature of the substrate (unless the semiconductor is degenerately doped). Note that a complete, lumpedelement tip-sample model likely will require inclusion of the stray capacitance, tip resistance, and semiconductor sheet resistance, in addition to the parallel plate capacitance.

A natural extension of the parallel plate model is to include a reasonable approximation of the tip shape. Consider a probe tip in the shape of a truncated cone of aperture angle  $\theta$  that is terminated by a spherical surface of radius *R*, as illustrated in Fig. 11.1. If the tip is positioned a distance z above the oxide surface, then the capacitance is [3]:

$$C = 2\pi\varepsilon_0 R \ln\left[1 + \frac{R(1-\sin\theta)}{z+d/\varepsilon_r}\right]$$
(11.2)

Once again, oxide uniformity is assumed. Given the underlying assumptions in Equations (11.1) and (11.2), we may consider one or more of the geometric parameters to represent effective electromagnetic dimensions as opposed to mechanical dimensions. For example, in Reference [4], the effective electromagnetic probe radius of an NSMM was extracted by use of Equation (11.2).

Figure 11.1. Metal-oxide-semiconductor junction formed by a probe tip and an oxidized semiconductor. The metal probe tip of a scanning probe microscope is characterized by geometric parameters: the effective tip radius R and the effective cone angle  $\theta$ . A native or deposited oxide film of thickness d sits on the semiconductor sample under test. In some systems, such as those based on contact-mode atomic force microscopes, the tip height z is zero and the metal probe is in direct mechanical contact with the oxide layer. Adapted from G. Gomila, J. Toset, and L. Fumagalli, J. Appl. Phys. 104 (2008) art. no. 024315, with permission from AIP Publishing.

#### 11.2.2 Metal-semiconductor models

While elementary capacitive models provide an initial estimate of material parameters, advanced tip-sample models must accurately reflect the semiconducting nature of the sample. Below, we will borrow heavily from established semiconductor physics models and related analyses [5]. In most cases, we will cite established results and provide only cursory discussion. Given the complexity of metal-semiconductor heterostructures, these results necessarily require a number of simplifying assumptions. For instance, many models assume idealized interfaces. Most models have been developed for multi-layer, thin-film geometries and thus do not account for the specialized tip shape found in scanning probe microscopes. As a result, one must proceed with care and attention to detail when selecting an appropriate tip-sample model. Ideally, any model will be complemented and validated by numerical simulations. The advanced models presented here are best thought of as a conceptual starting point that likely will require modification in order to suit specific applications.

Before discussing an ideal MOS system, we consider a metal-semiconductor system, such as a metal tip in a scanning probe microscope that is placed in direct mechanical contact with a semiconductor sample. Such a model would apply, for example, to a NSMM probe in contact with a clean silicon sample that is held in a vacuum environment to prevent the formation of a native oxide layer on the surface. As these experimental conditions are less common than ambient conditions, metal-semiconductor junctions are less common in scanning-probesystems than MOS-like junctions. However, we briefly discuss metal-semiconductor systems here, introducing important concepts such as the depletion layer. At thermal equilibrium, charge in the vicinity of the interface is redistributed such that the Fermi levels of the two materials are coincident. As a result, a depletion layer will develop in the semiconductor at the material interface. The depletion layer is characterized by a depletion width W that is given by [5]

$$W = \sqrt{\frac{2\epsilon_r \epsilon_0}{qN_D} \left( V_{bi} - V - \frac{k_B T}{q} \right)} \quad , \tag{11.3}$$

where  $\varepsilon_r$  is the relative permittivity of the semiconductor, q is the unit charge,  $N_D$  is the dopant density,  $V_{bi}$  is the built-in potential, V is the tip bias voltage,  $k_B$  is the Boltzmann constant, and T is the temperature. The depletion capacitance per unit area associated with the depletion layer in a metal-semiconductor system is given by [5]

$$C_{dep} = \sqrt{\frac{q\epsilon_r \epsilon_0 N_D}{2\left(V_{bi} - V - \frac{k_B T}{q}\right)}} \quad . \tag{11.4}$$

Once again, inclusion of lumped elements in addition to  $C_{dep}$ , such as the stray capacitance, tip resistance, and semiconductor sheet resistance, is necessary to complete the full tipsample model. The form of equation (11.4) suggests that a measurement of the voltagedependence of the depletion capacitance will allow determination of the dopant density, provide that material parameters ( $\varepsilon_r$  and  $V_{bi}$ ) are known or measurable. Indeed, as we will see below, local capacitance versus voltage (*C-V*) measurements (and differential dC/dVversus voltage measurements) are central to dopant profiling with NSMM and related techniques.

Experimentally, *C*-*V* tests require that a small AC voltage  $V_{ac}$  is added to the swept DC bias *V*. In the context of a scanning probe microscope where a DC bias is applied to the probe tip,  $V_{ac}$  can be added to the tip bias as a modulation signal. For dopant profiling applications, the amplitude of  $V_{ac}$  is typically on the order of millivolts while the tip bias is typically on the order of volts. In the presence of  $V_{ac}$ , an AC current will flow between the electrodes of the capacitor, leading to a net change in the charge *Q* at the capacitor's electrodes. The AC current can be integrated over time to find *Q* and in turn, the capacitance can be determined from the definition: C = dQ/dV. As we will see below, in certain cases the sample capacitance may be found by measurement of the relative change in the reflection coefficient in an

NSMM. In such cases, modulation of the tip bias is still useful, as it enables dC/dV (and higher order derivatives) to be determined by use of a lock-in technique.

### 11.2.3 Metal-oxide-semiconductor model

MOS or more generally, metal-insulator-semiconductor (MIS) systems, are more likely to be encountered in scanning probe measurements of semiconductor systems. This is due largely to the utility and prevalence of silicon-based devices and the convenience of characterization under ambient conditions. Well-established MOS diode theory provides a foundation for modeling the probe tip-oxide-semiconductor system. [5]-[7] As in the metal-semiconductor system, the formation of a depletion layer in the semiconductor is characterized by a voltagedependent depletion capacitance. In a MOS system, there is an additional contribution from the oxide capacitance. Coupling between the tip and trapped charges, as well as quantum capacitive effects, also influence the total tip-sample capacitance observed in the junction of a radio frequency scanning probe microscope.

The curve can be conceptually divided into three modes of operation: accumulation, depletion, and inversion. The accumulation regime for an n-type device corresponds to large, positive voltages applied to the metal contact (or probe). In this range of voltages, negatively charged electrons (the majority carriers) in the semiconductor will accumulate at the semiconductoroxide interface. For devices with sufficiently thick oxide layers, the C-V curve will fully flatten at sufficiently large positive voltages. In accumulation, the measured capacitance is dominated by the oxide capacitance. An example of a C-V curve measured by use of a NSMM is shown in Fig. 11.2 for an n-type silicon sample [8]. The accumulation mode corresponds to the far-right side of the figure. As the bias voltage is decreased toward and past zero volts, the electrons are displaced from the semiconductor-oxide interface, creating a depletion region near the surface of the semiconductor that effectively acts as an insulating layer. Thus, when a MOS device is operated in the depletion mode, the total measured capacitance consists of the oxide capacitance in series with the depletion capacitance. Note that in depletion, the depletion width and depletion capacitance are once again voltage-dependent, as was the case in metal semiconductor junctions. Finally, for sufficiently large negative voltages, electron-hole pairs will be generated in the semiconductor and move toward the metal contact, accumulating near the semiconductor-oxide interface. Thus an inversion layer is formed near the surface of the semiconductor within which the holes are the dominant carrier type. At extreme voltages, the C-V curve once again flattens out and the measured capacitance is the series combination of the oxide capacitance and the maximum depletion capacitance. For a p-type device, similar modes of operation exist, albeit with reversed voltage polarity.

Figure 11.2 **Capacitance-voltage curve for n-type silicon.** The relative capacitance of an n-type MOS device is shown as a function of the bias voltage on the metal contact. The measurements were made by use of a near-field scanning microwave microscope and calibrated by use of the techniques described in the text. The tip-sample geometry is as shown in Fig. 11.1 with z = 0 (contact mode). Measurements were made with the probe tip positioned over two areas of known dopant concentration, as indicated in the figure. Multiple measurements are shown for each probe position. Reprinted from H. P. Huber, et.al., *J. Appl. Phys.* **111** (2012), art. no. 014301, with permission from AIP Publishing.

A number of characteristic geometric and material parameters may be extracted from a C-V measurement. For example, if a device is operated in accumulation mode, the oxide thickness can be obtained in a straightforward way. In accumulation, the measured capacitance is the oxide capacitance and may be modeled by a simple expression such as Equation (11.1). Provided that the relative permittivity of the oxide and the metal contact area are known, the oxide thickness is easily determined by use of Equation (11.1). In a scanning probe microscope, topographic images can often provide values for the contact area and thickness. In that case, the relative permittivity of the oxide layer can be determined from the C-Vmeasurement. Equation (11.2) can provide a more precise representation of the tip geometry, but there are additional unknowns (the effective tip radius and cone angle) that can't be determined from topographic images. In this case, C-V measurements performed on a series of oxide steps of different thicknesses allows us to determine of the permittivity in addition to the geometric parameters of the tip [4]. While electromagnetic material parameters determined by use of macroscopic C-V measurements represent average quantities in MOS systems, the corresponding values extracted from C-V measurements made with a scanning probe microscope will be local with a lateral spatial resolution on the order of the probe dimensions. In general, appropriate analysis of the measured C-V curve for a MOS capacitor enables estimation of several material and device parameters, including the flatband voltage, the threshold voltage, and the difference between the metal and semiconductor work functions.

Here, the primary goal is to determine the semiconductor dopant concentration. As in the metal-semiconductor model, this objective requires calculation of the semiconductor depletion layer capacitance. In the standard approach [5]-[7],  $C_{dep}$  is given by the derivative of the surface charge density  $Q_S$  with respect to the surface potential  $\Psi_S$ . Initially, the potential in the semiconductor  $\Psi$  is introduced and defined with respect to the intrinsic Fermi level within the semiconductor. The value of  $\Psi$  at the surface is  $\Psi_S$ . If the space charge density varies only in one direction, as in the center of a parallel-plate-like geometry, then the potential  $\Psi$  may be determined from a one dimensional Poisson equation of the form

$$\frac{d^2\Psi}{dz^2} = -\frac{\rho(z)}{\varepsilon_r \varepsilon_0} \qquad , \tag{11.5}$$

where  $\rho(z)$  is the one-dimensional space charge density distribution and z is normal to the semiconductor-oxide interface. Equation (11.5) can be solved analytically, though the existence of solutions depends on the form of  $\rho(z)$ . For example, in Reference [5], this approach is used to find the depletion capacitance for an ideal MOS structure under flat-band conditions ( $\Psi_S = 0$ ):

$$C_{dep} = \sqrt{\frac{\epsilon_r \epsilon_{0p_0 q^2}}{k_B T}} \qquad , \tag{11.6}$$

where  $p_o$  is the equilibrium density of holes in the bulk of the semiconductor. Note that the total capacitance is the series combination of the depletion capacitance and the oxide capacitance. In the context of a scanning probe microscope, the one dimensional approximation is of limited value, as carriers are likely to be redistributed in the lateral dimensions, as well as z [9]. Given the complexity of the charge distribution in the probe-sample junction of a scanning probe system, numerical solutions may complement or even supersede approximate analytical solutions to the Poisson equation.

Before proceeding to discussion of scanning-probe-based dopant profiling techniques, it is vital to note limitations of the simplified MOS picture that we have introduced. To date, scanning-probe-based dopant profiling has primarily been applied to silicon. However, the silicon-silicon oxide interface is unusually free of trapped charge and interface states. In contrasts, most compound semiconductor surfaces and oxide interfaces contain high densities of mid-gap states whose charge contributes significantly to the magnitude of surface band bending and the formation of the depletion region. Though these states tend to be deep within the gap and thus unresponsive to high-frequency microwave signals, they will alter the dependence of the depletion capacitance upon DC and low-frequency tip bias voltages. As a result, extension of scanning-probe-based dopant profiling techniques to material systems beyond silicon will require refinement of the tip-sample models to account for trapped charge and interface states. Additional details on interface states can be found in References [2], [10], and [11].

#### 11.3 Dopant profiling with scanning capacitance microscopy

Scanning capacitance microscopy is similar in many respects to other scanned probe techniques, but the SCM probe detects changes in local capacitance as opposed to current, force, or other aspect of the probe-sample interaction [12]. SCM is an RF measurement tool in that the capacitive sensor that is integrated into the probe structure typically operates at a frequency near 1 GHz. Often, the capacitance may be detected by integrating the sensor with a resonant LCR circuit and measuring changes in the resonant behavior, reminiscent of a resonant NSMM. The sensor measures the differential capacitance  $\Delta C$  induced by an alternating voltage signal of amplitude  $V_{ac}$  that is applied to either the probe tip or the sample. The alternating voltage signal, oscillating at a frequency  $\omega_{ac}$ , also serves the

reference for a lock-in amplifier, which is used to perform the measurement of  $\Delta C$ . A slowly swept DC bias V is applied, in addition to  $V_{ac}$ , in order to measure the  $\Delta C$ -V relationship. SCM has long been applied to two-dimensional dopant profiling problems. [13]-[23]

As we saw in Chapter 9, the extraction of electromagnetic material parameters by use of high-frequency scanning probe microscopy relies on accurate modeling of the probe-sample interaction. Likewise, interpretation of SCM data requires reliable modeling in order to extract parameters from the measured  $\Delta C \cdot V$  data [15]. Often, these models require complex numerical simulation involving a large number of parameters, including probe tip shape, oxide layer thickness, oxide permittivity, and the SCM operating voltage. In many cases, the numerical simulations are implemented in software packages. One example of a model for two-dimensional dopant profiling with a SCM is a quasi-three-dimensional model in which the probe-sample capacitance is represented by a set of capacitive MOS ring-shaped capacitors arranged in a concentric geometry that corresponds to the probe tip geometry [13], [15]. Each MOS capacitance is calculated by use of Equation (11.5), with the space charge density distribution approximated by either Boltzmann or Fermi-Dirac statistics. The use of an approximation for the space charge density and other simplifying assumptions in the solution of Poisson's equation can speed computation time, but unfortunately this can also compromise the accuracy of the extracted dopant concentration [16]. Provided that one can tolerate the increased calculation time, a three-dimensional, finite-element solver can provide more accurate solutions of the three-dimensional Poisson's equation [17]. Note that in some cases, the effects of air gaps between the tip and sample, and the effects of condensed water on the sample, were needed in the model to improve agreement between SCM and measurements by other macroscopic techniques such as secondary ion-mass spectrometry [13]. In Fig. 11.3, several different models were used to calculate the conversion of measured dC/dV curves into estimates of dopant density. Clearly, the estimates are sensitive to the model used. In order to build confidence in both the interpretation of measurements and parameter extraction by use of a given model, calibration is required.

Figure 11.3. **Comparison of dopant density conversion curves for different models.** Calculated curves for converting dC/dV measurements to estimates of dopant density for five different models of a MOS capacitor. The models are denoted by the labels (A) through (E). Details of the models can be found in Reference [16] and the citations within. Reprinted with permission from J. J. Kopanski, J. F. Marchiando, and B. G. Rennex, *J. Vac. Sci. Technol. B* **20** (2002) pp. 2101-2107. Copyright 2002, American Vacuum Society.

Calibration techniques and reference samples are critical for establishing accurate, absolute dopant profiling. Without calibration, SCM and other RF scanning probe techniques are limited to measurement of relative dopant concentration. The ideal reference sample for dopant profiling will be topographically smooth, minimizing the contribution of geometric effects to the probe-sample capacitance. In addition, the ideal reference sample should provide distinct, contrasting regions of varying dopant concentrations across the dynamic range of interest: roughly speaking, between  $10^{14}$  cm<sup>-3</sup> and  $10^{20}$  cm<sup>-3</sup>. Finally, the ideal reference sample will include both p-doped and n-doped regions, though in practice separately realized p-doped and n-doped reference samples have often been utilized. One approach is to create a multilayer structure in which each layer is uniformly doped at a known concentration. As needed, buffer layers may be introduced between the uniformly doped stripes, e.g.,  $10^{14}$  cm<sup>-3</sup>,  $10^{15}$  cm<sup>-3</sup>,  $10^{16}$  cm<sup>-3</sup>, etc. [18], [19] A conceptual illustration of such a reference sample is shown in Fig. 11.4. This type of reference sample was also described in Chapter 9 in relation to material characterization techniques.

Figure 11.4. **Reference sample for dopant density calibration.** The reference sample is fabricated by depositing a series of silicon layers with known, uniform dopant concentration in a multilayer geometry. When this sample is imaged in cross section with a scanned probe, the sample appears as a series of stripes of varying dopant concentrations (e.g.  $10^{14}$  cm<sup>-3</sup>,  $10^{15}$  cm<sup>-3</sup>...  $10^{19}$  cm<sup>-3</sup>). In practice, separate p-doped and n-doped samples of this type have been utilized for calibration of dopant profiles.

Fig. 11.5 shows an SCM image of a variably doped n-type gallium nitride sample [20]. The stripes corresponding to different doping levels are clearly distinguished in the raw SCM image, shown in Fig. 11.5(a). Comparisons of SIMS measurements of a reference sample are compared to the SCM measurements in Fig. 11.5(b), providing a calibration approach that converts SCM measurements to estimates of the absolute dopant concentration. Systematic, cross-sectional SCM imaging of multilayered reference structures with varying layer thicknesses reveal best-case lateral resolution on the order 10 nm for SCM imaging [20], [21]. Note, however, that both the sensitivity and resolution of SCM images are strongly dependent on tip geometry.

Figure 11.5. Scanning capacitance microscopy of variably-doped, n-type gallium nitride. (a) Cross-sectional SCM amplitude image of the multilayer gallium nitride sample. Dopant concentrations for each layer are labeled. (b) Processed SCM data (dashed line) are compared to SIMS data (solid line). Reprinted with permission from J. Sumner, R. A. Oliver, M. J. Kappers, and C. J. Humphreys, *J. Vac. Sci. Technol. B* **26** (2008), pp 611-617. Copyright 2008, American Vacuum Society.

# 11.4 Dopant profiling with near-field scanning microwave microscopy

As we have described above, measurement of the depletion layer capacitance-voltage relationship provides an avenue for estimating dopant concentration in metal-semiconductor and MOS structures. Since NSMM measurements of such structures include a significant contribution from the local depletion layer capacitance, NSMM provides a useful capability for local capacitance-voltage spectroscopy and in turn, dopant profiling. These capabilities are only realized when other contributions to the tip-sample interaction, including any parasitic reactance, are accounted for by use of calibration and modeling. Below, we introduce approaches to dopant profiling by use of NSMM. In the first approach, the dopant profiling procedure has three main components: (1) measurement of the voltage dependence of the reflection coefficient, (2) calibration of the instrument so that the reflection coefficient can be converted to quantitative values of the capacitance, and (3) extraction of the dopant concentration from the capacitance-voltage relationship. We focus on the determination of the capacitance-voltage relationship in order to connect the procedure to the underlying physics, standard semiconductor characterization approaches, and dopant profiling with other scanned probe techniques such as scanning capacitance microscopy.

The discussion below assumes an experimental setup similar that of References [4] and [8] and generally follows the methods laid out in those references. Naturally, alternative test platforms will require corresponding modifications to the dopant profiling procedure, though the general concepts will remain intact. Measurements are made by use of a resonant NSMM operating in a reflection mode. The operating frequency is near, but not equal to, the resonant frequency. Further, the NSMM is assumed to be based on a contact-mode atomic force microscope in which the probe tip is in direct mechanical contact with the sample. During measurements, a DC bias is applied to the tip. The tip is laterally positioned above a point of interest and the complex reflection coefficient  $S_{11}$  is measured as a function of the DC tip bias by use of a vector network analyzer. The effects of the measurement platform may be partially removed by calibrating the network analyzer at coaxial reference planes located outside of the microscope head. Alternatively, the system may be calibrated such that the complex impedance is obtained in place of  $S_{11}$ , as described in Chapter 7 [24], [25]. Here, our approach is based on measurements of the raw, uncalibrated, complex reflection coefficient  $S_{11}$ .

The conversion of the raw  $S_{11}$  data to quantitative capacitance measurements requires a known reference sample, an accurate model of the tip-sample interaction, and a calibration procedure. The reference sample must incorporate features with a known impedance. Note that if a three-term error model is used for impedance calibration of NSMMs that operate in a reflection mode, then a minimum of three reference features are required for a one-port calibration, though the inclusion of additional reference features will decrease the statistical uncertainty in the calibrated measurements. Ideally, all of the features will be located close together such that the calibration requires only a single NSMM calibration image to be acquired and subsequently analyzed. It is also useful to incorporate the reference features into the same wafer or substrate as the sample under test, if possible. The calibration procedure below requires that the reference features are on either the same device substrate

or a similar substrate that can be assumed to have similar electromagnetic material properties as the device substrate. Specifically, the model of the tip-sample interaction must be valid across both the reference sample and the sample under test. Here, we will use a set of microcapacitors, illustrated in Fig. 11.6(a), as the reference sample [3]. This reference sample was fabricated using standard microfabrication techniques. The sample comprises a series of circular gold pads patterned upon a series of steps in a silicon dioxide staircase on a doped silicon substrate. The NSMM tip is in direct electrical and mechanical contact with one of the gold contacts, forming the top electrode of a microcapacitor while the doped substrate forms the bottom electrode. The capacitance  $C_{diel}$  is calculated by use of equation (11.1) with the geometric parameters (pad area A and oxide thickness d) determined from topographic images of the sample. In addition, the oxide is assumed to be uniform and have a known relative permittivity. Assuming that the leakage current through the oxide is negligible, the impedance of the microcapacitor is given by the standard definition  $Z = 1/j\omega C_{tot}$ , where  $\omega = 2\pi f$  and f is the operating frequency.

Figure 11.6. **Microcapacitor reference sample.** (a) A side-view schematic of the microcapacitor reference sample. The oxide layer is patterned to create a staircase with step thicknesses of 50 nm, 100 nm, 150 nm, and 200 nm, for example. Circular gold pads are patterned on top of the steps. On each step, there is a sequence of pads with diameters equal to 1  $\mu$ m, 2  $\mu$ m, 3  $\mu$ m, and 4  $\mu$ m. (b) A lumped-element model of the tip-sample interaction, including the capacitance  $C_{diel}$ , a parasitic background capacitance  $C_{back}$ , and the cantilever-sample capacitance  $C_{cant}$ . Reprinted from H. P. Huber, et. al., "Calibrated nanoscale capacitance measurements using a scanning microwave microscope," *Rev. Sci. Instrum.* **81**, (2010) art. no. 113701, with permission from AIP Publishing.

A simple, lumped-element circuit model is used to represent the reference sample, as shown in Fig.11.6(b). Following a similar analysis as was presented in Chapter 8, the total microcapacitor capacitance  $C_{tot}$  is defined by the parallel combination of the oxide capacitance  $C_{diel}$  and the substrate or back capacitance  $C_{back}$ :

$$\frac{1}{C_{tot}} = \frac{1}{C_{diel}} + \frac{1}{C_{back}}$$
(11.7)

To find the calibrated capacitance measured by the probe, two measurements are made for each microcapacitor: the reflection coefficient with the probe positioned on the gold pad  $S_{11}^{Au}$ and the reflection coefficient with the probe positioned on the oxide surface  $S_{11}^{Ox}$ . The relative amplitude signal  $\Delta S_{11}$  is defined simply as the difference between the scattering parameters

$$\Delta S_{11} = S_{11}^{Au} - S_{11}^{0x} \quad . \tag{11.8}$$

The use of a relative value is necessitated by the fact that the absolute reflection coefficient amplitude will depend on the test platform, the nature of the NSMM resonance, and the selected operating frequency. Further, by performing this subtraction, we are excluding the effects of the parallel stray capacitance  $C_{cant}$  from the cantilever body, albeit under the assumption that  $C_{cant}$  is unchanged by movement from the gold pad to the oxide. Note that sensitivity of  $C_{cant}$  to changes in probe position can be reduced by increasing the aspect ratio of the asperity on which the probe tip is formed. This increased aspect ratio reduces the capacitive coupling between the cantilever body and other parts of the probe platform with the sample. In Reference [4], it has been shown that there is a linear relationship between the relative amplitude signal and the total microcapacitor capacitance:

$$C_{tot} = \alpha |\Delta S_{11}| , \qquad (11.9)$$

where a is a calibration constant. The two unknown calibration parameters,  $C_{back}$  and a, in Equations (11.7) and (11.9) can now be determined from measurements of reference microcapacitors.

Once  $C_{back}$  and a are known, the absolute capacitance as a function of voltage and tip position can be calculated from voltage- and position-dependent measurements of  $\Delta S_{11}$ . Once this dependence is known, the local dopant concentration can be determined from numerical models or from analytical solutions for the semiconductor potential  $\Psi$  in Poisson's equation. Essentially, once the NSMM measurement has been calibrated and converted to a voltagedependent capacitance (or voltage-dependent dC/dV) measurement, estimation of the dopant concentration from the C-V curve follows the same procedure as SCM, as described in the previous section. As the NSMM capacitance calibration relies on a relative measurement referenced to the reflection coefficient on the oxide surface  $S_{II}^{Ox}$ , it is limited to applications in which there is an oxide or other suitable reference surface. Given the widespread use of silicon substrates and the presence of native silicon oxides, there are many applications that meet this requirement. Additional requirements for successful and sustainable capacitance calibration include a stable test platform. Given the current state of commercial network analyzers, test cabling, and other hardware, it is generally straightforward to assemble a nominally stable test platform. However, microwave measurements in general and resonant NSMMs in particular are highly sensitive to environmental factors, such as temperature and humidity. A stable tip shape is also a necessity for sustainable, calibrated NSMM capacitance measurements, as capacitance is inherently geometry dependent. In the case of a contactmode, AFM-based NSMM, significant mechanical wear can occur in a short period of time, thus invalidating the calibration procedure. Custom, metal-coated nanowire probes have demonstrated reduced mechanical wear over large numbers of scans. [26]

Calibrated, spectroscopic dC/dV measurements are compared to modeled data in Fig. 11.7. The different curves in Fig. 11.7(a) correspond to different regions of known dopant concentration on an n-type reference sample similar to the one shown in Fig. 11.4. The numerical simulation package, FASTC2D [27], initially calculates the spectroscopic curves based on best-known values of measurement parameters and then adjusts a set of variable

parameters, including tip geometry, surface insulator thickness  $t_{ox}$ , and the relative permittivity of the oxide  $\varepsilon_r$ , to fit the experimental data. To achieve reasonable agreement, the voltage amplitude had to be significantly increased from a nominal value of 800 mV to a value of 3000 mV, broadening the modeled response. Additionally, the flatband voltage in the model had to be adjusted by + 0.8 V to align with the measured NSMM data. Overall, approximate agreement between the model and measured data is seen in Fig. 11.7(a). Finally, the FASTC2D package is also used to model the n-type calibration curve shown in Fig. 11.7(b). The solid black line is an initial calculation, based on nominal values of  $t_{ox}$  (1 nm) and  $\varepsilon_r$  (3.9), with the DC bias set in the model to produce the maximum differential capacitance signal at a concentration of  $10^{16}$  cm<sup>-3</sup>. The dashed line represents a fit found by reducing the ratio  $\varepsilon_r/t_{ox}$  to 0.5 and setting the DC bias to produce the maximum differential capacitance signal at a concentration of  $10^{15}$  cm<sup>-3</sup>.

Figure 11.7. **Modeling of capacitance voltage measurements.** (a) Experimentally measured, voltage-dependent dC/dV curves (markers and solid lines) are compared to numerically modeled values (dashed lines). (b) Experimentally determined calibration curves for an n-type calibration sample. Reprinted from H. P. Huber, et.al., *J. Appl. Phys.* **111** (2012), art. no. 014301, with permission from AIP Publishing.

## 11.5 Dopant characterization with other microscopy techniques

In addition to the SCM and NSMM, several additional scanning probe tools are commonly applied to local characterization of defects and dopants in semiconductors [28], [29]. As these additional tools are based on DC or low-frequency techniques, they are not, strictly speaking, applicable to microwave and RF measurements. However, they provide complementary insight into semiconductor micro- and nano-electronics in general and RF nanoelectronics in particular. Here we will briefly review three such techniques: scanning spreading resistance microscopy, scanning kelvin probe microscopy, and scanning tunneling microscopy. In general, these techniques are two-dimensional and surface-sensitive. Thus, sample cross-sectioning is required for subsurface, depth-dependent profiling. As in the case of SCM and NSMM, extraction of material parameters such as dopant concentration requires accurate physical models, reliable calibration techniques, and well-known reference samples.

From an instrumentation point of view, the SSRM is similar to a contact-mode, conductive atomic force microscope [30]. During SSRM measurements, a sharp, conductive cantilever probe is in direct mechanical contact with the sample. With a small ( $\sim 100 \text{ mV}$ ) bias applied to the probe, the SSRM measures the DC resistance between the probe and a backside contact on the sample. If the force applied by the cantilever probe on the sample exceeds a threshold value, then the measured resistance will be approximately equal to the local spreading resistance, which is, in turn, inversely proportional to the local carrier concentration. Because

the SSRM applies high forces, a mechanically hard probe such as a diamond-coated tip is usually used [31].

SKPMs are often based on non-contact, cantilever-based AFMs [32]. A DC voltage is applied to the probe tip, inducing an electrostatic force between the probe and the sample. If the DC bias voltage is equal to the surface potential, then the electrostatic force will be zero. Thus, the surface potential can be measured by adjusting the tip voltage such that the electrostatic force is nulled. In practice, the force can be detected by modulating the bias voltage at a mechanical resonance frequency of the cantilever and using a lock-in technique to detect the resulting oscillations of the cantilever. Note that if a non-contact, dynamic AFM mode is used for distance following, then the fundamental resonance frequency will be required. The work function of the sample may be determined from the surface potential, provided that the work function. This method has been used to perform dopant profiling on Si samples, for example [33].

At the limit of device scaling, as device dimensions become comparable to a few atomic diameters, device performance depends upon the identity and placement of individual atoms. At this scale, the STM's capability to perform local electronic spectroscopy on individual atoms is a powerful tool [34], [35]. In an STM, a conducting tip is placed within a nanometer of a conducting sample. When there is a potential difference between the tip and the sample, a small, but measurable current will flow due to quantum mechanical tunneling. The magnitude of the tunneling current will depend upon the tip-sample separation, the potential difference, and the local electronic density of states of the sample. The latter dependence is the basis in scanning tunneling spectroscopy (STS), a technique in which the tunneling conductivity is measured as a function of the potential difference between the tip and the sample. If the tip-sample height is fixed, then STS provides a method for measuring the density of states with atomic-scale resolution. This enables the identification and electronic characterization of individual atoms, enabling the measurement of the role of individual dopants in nanoelectronic devices [36].

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Figure 11.1



Figure 11.2



Figure 113



Figure 11.4



Figure 11.5



# Figure 11.6



Figure 117