

# Microwave imaging at the nanoscale: quantitative measurements for semiconductor devices, materials science and bio-applications

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This article will examine a novel high frequency electrical characterization microscope for the nano-scale imaging of semiconductor devices, advanced materials and biological samples. The scanning microwave microscope (SMM) can extract quantitative material properties at Giga-Hertz (GHz) frequencies with nanometre accuracy. The SMM interfaces two well-known measurement tools, the atomic force microscope (AFM) for materials characterisation and the vector network analyser (VNA) from telecommunication network testing for reliable microwave signal measurements. The AFM allows for nanometre lateral resolution imaging, and the VNA provides high precision impedance and admittance measurements at broadband frequencies from MHz to GHz. We give a review of this microscope including nanoscale imaging of semiconductor devices and failure analysis, as well as calibrated impedance imaging of advanced materials. Finally, we show the first calibrated complex impedance image of bacteria at 20 GHz, as well as a microwave image of a living endothelial cell acquired in liquid buffer media. This new microscope combines advanced nanotechnology imaging with microwave electronics and 3D sample modeling, for the study of advanced materials and for the first time also to bio-molecules and cells in living condition.



## Introduction

The Scanning Microwave Microscope (SMM) merges the nanoscale imaging capabilities of an atomic force microscope (AFM) with the high-frequency broadband (from MHz to GHz) impedance measurement accuracy of a vector network analyser (VNA) (Figure 1). The typical frequency range of the combined SMM is between 1-20 GHz (Huber et al. 2010). It allows characterising electric and magnetic properties of materials at microwave frequencies with nanometer lateral resolution. Using the microwave signal, impedance nanoscale imaging and doping profiling of the 'Device Under Test' (DUT) can be performed. Typically the SMM is operated in reflection mode, whereby the ratio of the reflected and incident electromagnetic waves, the so called  $S_{11}$  scattering parameter, is measured by the VNA at each pixel of the AFM tip-sample contact point. As such a microwave image is generated pixel by pixel, simultaneously to the topographical image of the DUT. Imaging speeds are fast resulting in a typical acquisition time of one minute per image.

There are two main different imaging modes in SMM. The first is quantitative dopant profiling by

means of differential capacitance (also called dC/dV), which is a widely used technique for semiconductor failure analysis and detecting leakages in solid state devices with nanometer resolution (Huber et al 2012). The dC/dV mode relies on a low frequency (kHz) modulation of the GHz  $S_{11}$  signal using the Dopant Profile Measurement Module (DPMM). It allows tuning the semiconductor depletion zone with low frequency and probing the doping concentration through the native oxide interface at GHz frequency. The second SMM mode is complex impedance imaging and it is based directly on the scattering  $S_{11}$  signal at GHz frequency (Gramse et al 2014). This complex impedance mode has been recently developed and is currently used in materials science and life science investigations. The  $S_{11}$  signal is detected by the VNA and depends on the tip-sample relative electrical impedance. A calibration workflow is required that allows converting the raw  $S_{11}$  into calibrated capacitance and resistance images of the DUT.

SMM has been extensively used to gain new insights in the semiconductor industry, and significant advances in SMM measurement and calibration

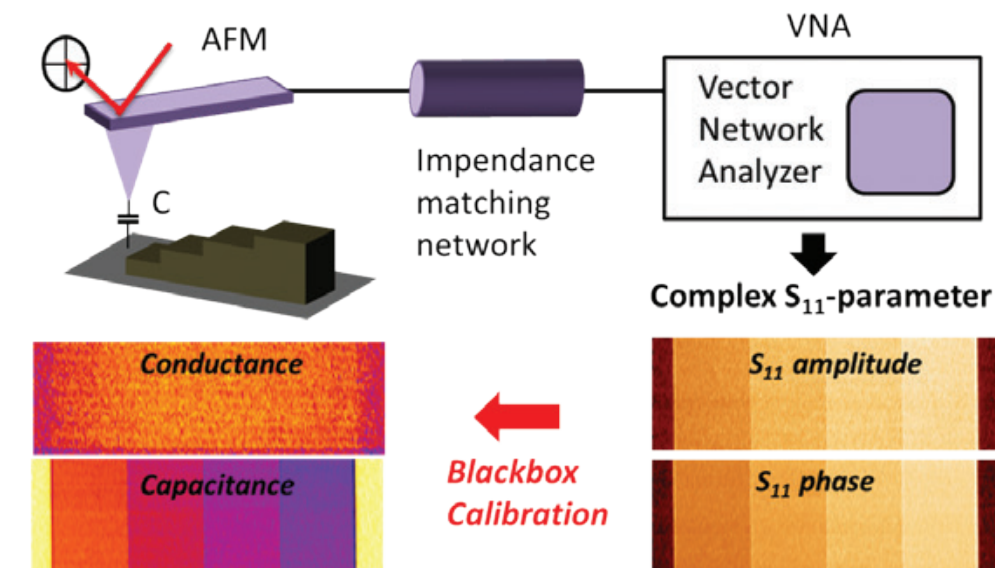


Figure 1. Scanning microwave microscope (SMM) sketch (upper row) and complex impedance calibration (lower row). The SMM sketch shows the main components including AFM cantilever with standard laser deflection readout, the impedance matching network consisting of the 50Ω shunt resistor, half-wavelength resonator and coaxial cables, as well as the VNA (vector network analyser) for GHz signal readout. The VNA measures the amplitude and phase of the complex  $S_{11}$  parameter, which are shown in the  $S_{11}$  amplitude and  $S_{11}$  phase images. Based on a standard VNA three error parameter black-box calibration the conductance and capacitance images are derived.

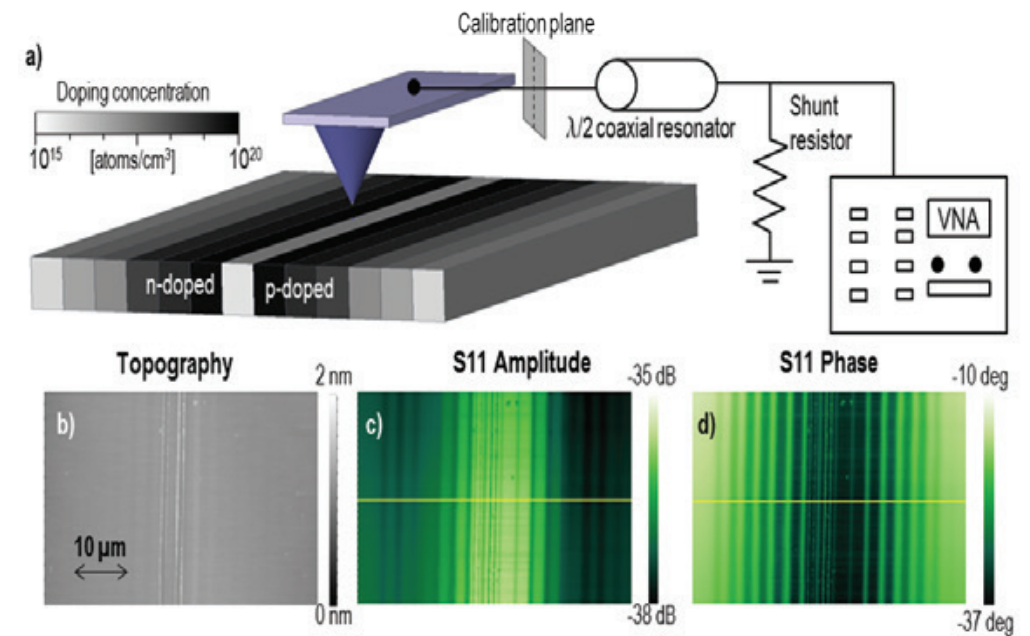


Figure 2. Sketch of the SMM experimental setup and of the doped Si sample with flat topography. The sample consists of 10 different n-implant areas and 10 different p-implant areas with a width of 2 μm each, with doping concentrations ranging from  $4 \times 10^{15}$  to  $1 \times 10^{20}$  atoms/cm<sup>3</sup>. The SMM raw images of this sample are shown including flat topography (b),  $S_{11}$  amplitude (c) and  $S_{11}$  phase (d).

workflows were achieved studying well-defined semiconductor devices (Schweinboeck & Hommel 2014). Originating from the requirement of the semiconductor industry to identify failures and leakages in electronic devices with nanometer precision and low impedance values, the SMM has since proven to be a powerful tool also for materials science applications (Hoffmann et al 2014; Kasper et al 2014a). In particular, the capability of sub-surface imaging of buried nanoscale structures has been demonstrated, as well as conductivity measurements of 2D layers (Gramse et al 2015). Recently, the high frequency characterisation of biological materials has attracted considerable interest, as SMM represents a convenient non-invasive evanescent imaging technique that complements AFM (Tuca et al 2016). While AFM measures the surface topography, the SMM can see inside materials using the electromagnetic wave penetration capabilities at GHz frequency.

In the following we present dopant profiling measurements for semiconductor device applications, complex impedance measurements for materials science investigations, and permittivity

measurements of single bacteria at 20 GHz including live cell imaging in liquid media. On top of standard SMM, we present augmented SMM solutions where the standard mode is extended with other electronic measurement devices from Keysight that opens new and advanced SMM measurement capabilities. Finally, we combine SMM with the Keysight EMPro 3D modeling capabilities at GHz frequencies that can be used to calculate E-fields and complex impedance values in order to assist quantitative SMM data interpretation.

## Dopant profiling and semiconductor failure analysis

For quantitative dopant profiling with dC/dV, the DPMM is attached to the VNA and a well-established calibration workflow is used that allows extracting the doping concentration of a semiconductor sample using a dopant calibration sample (Figure 2) (Huber et al 2012). The calibration workflow converts the dC/dV voltage signal to doping concentration (atoms\*cm<sup>-3</sup>) values. First, a calibration sample is scanned with SMM, acquiring the dC/dV image from which the dopant calibration

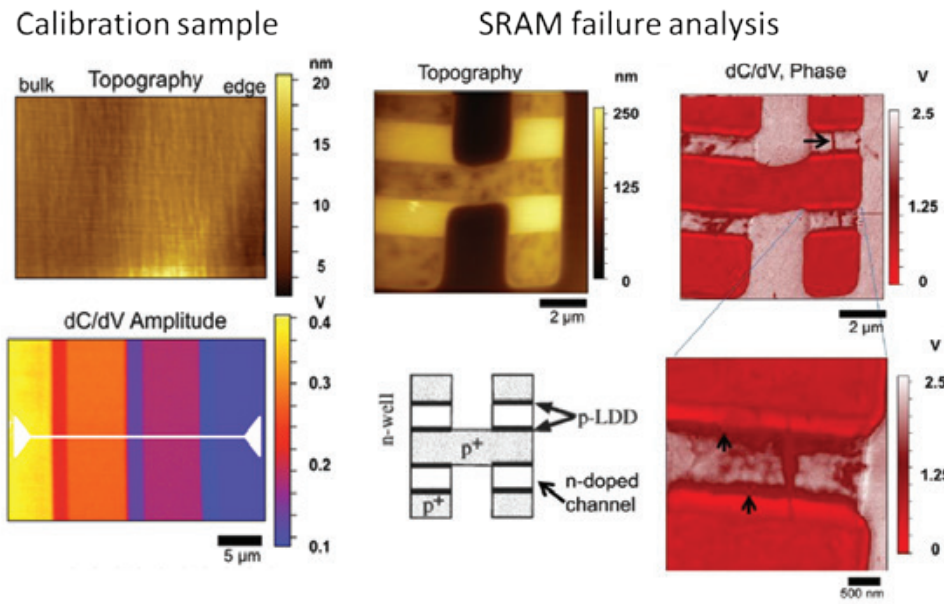


Figure 3. Dopant profiling calibration sample including topography and dC/dV image of dopant stripes (left column). Right column: imaging SRAM defect structures and p-n junctions. Topographical image with sketch of the doping polarities (left). dC/dV phase image (right column) with defect structures in the n-doped channels with a zoom in below showing p-n junctions (cf. arrow).

curve is generated (Figure 3, left column). In a second step, the dC/dV image of the DUT is acquired under the same SMM conditions. Based on the dopant calibration sample the dC/dV image of the DUT can be transferred in quantitative dopant concentrations (Schweinboeck & Hommel 2014). Figure 3 shows the application to semiconductor failure analysis. Defects or leakages on real devices can be detected using the dC/dV image which is

acquired simultaneously to the topographical image, as shown on a conventional bipolar p-n SRAM sample. Several p-n defect structures within the n-doped channels can be identified in the dopant profiling image (cf. arrows in Figure 3). A sketch depicts p- and n-doped areas in Figure 3, showing a clear agreement with the various doping types and concentrations in the dC/dV image. The various regions of interest can be clearly distinguished and

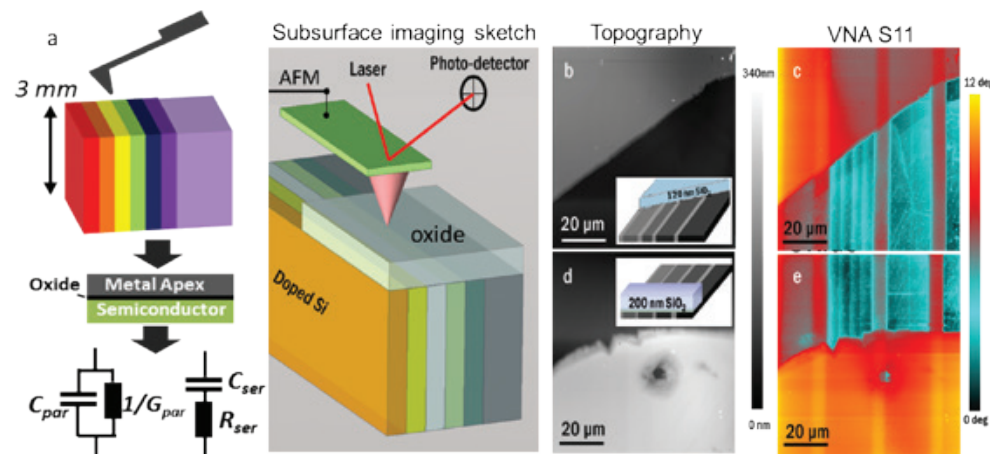


Figure 4. Sub-surface imaging. Dopant sample in cross-section with metal-oxide-semiconductor (MOS) structure and experimental setup for sub-surface imaging of a doped Si substrate partly covered with SiO<sub>2</sub> (a). (b), (d) Topographical images of a doped Si substrate partly covered with 120 nm and 200 nm thick oxide (inset is a sketch of the sample). (c), (e) show the reflection S11 phase images with the sub-surface dopant features observed even below the oxide.

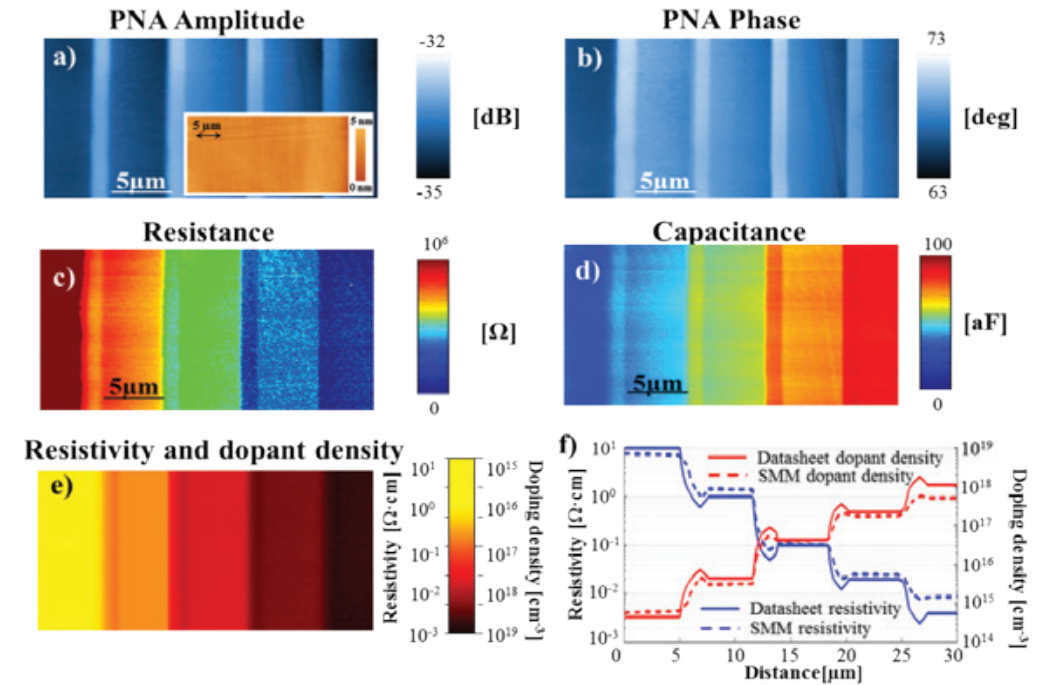


Figure 5. Calibrated capacitance and resistivity. SMM raw data (a and b) and calibrated impedance (c and d) of a n-type doped Si sample. The sample has a flat topography (inset in a) and different n-doped areas with doping concentrations. The 5  $\mu\text{m}$  wide doping areas are separated by 1  $\mu\text{m}$  wide bulk interface layers. The resistivity and doping concentration (e) are calculated from the SMM resistance. (f) Comparison with data sheet.

the doping polarity and density analysed. No cross talk is obtained between topography and dopant profiling image. Bipolar p/n junction interfaces are observed with a width of 100 nm. The exact width of the p-n junction depends on the SMM tip voltage bias that can be further used to investigate the physics of the depletion zone interface.

### Sub-surface imaging: seeing below the surface

Figure 4 shows that SMM can use the penetration capabilities of GHz microwaves to perform non-destructive in-depth imaging of structures located underneath the sample surface (Gramse et al 2015). This capability, named as sub-surface imaging, is an important benefit when the area of interest is buried under the sample surface. Figure 4 (left column), shows a silicon test sample with varying dopant density ( $10^{15}$ – $10^{19}$  atoms $\cdot\text{cm}^{-3}$ ) where the calibrated impedance can be determined from the SMM images. The right column shows the sub-surface imaging proof and concept. The dopant sample is covered with dielectric layers of SiO<sub>2</sub>

(100–400 nm thickness). SMM, with a noise level of 1 aF, allows sensing the electrical properties of doped silicon structures buried under oxides with more than 400 nm thickness. SMM can be therefore used to accurately perform quantitative nanoscale probing of dielectric materials and to perform calibrated capacitance characterisation of buried structures. This finds applications in the field of semiconductor failure analysis studies including process optimizations for integrated chip fabrication.

### Calibrated capacitance and resistivity: quantitative materials properties from SMM

Based on the S<sub>11</sub> complex impedance calibration workflow, the SMM images are converted into capacitance and resistance (Figure 5) (Gramse et al 2014). Using a tip-sample analytical model that includes tip radius, microwave penetration skin depth, and semiconductor depletion layer width, the resistivity of the DUT can be extracted from the



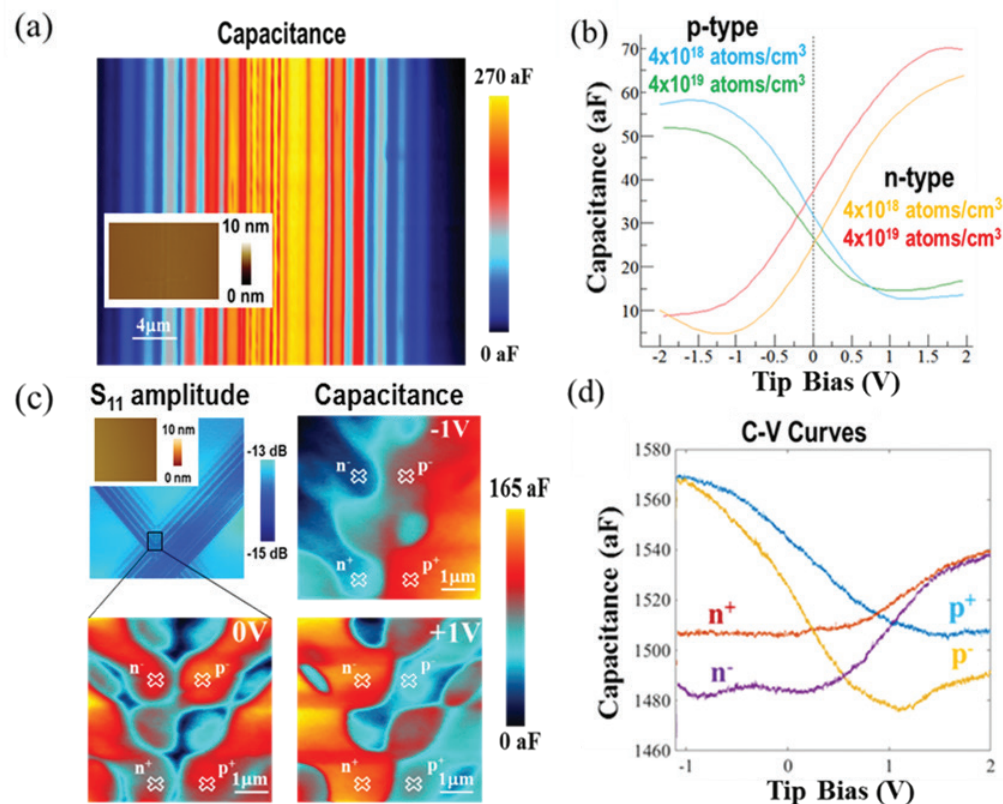


Figure 6. Capacitance-voltage (C-V) spectroscopy experiments. (a) Calibrated capacitance and topography (inset), of a bipolar doped Si staircase sample with n- and p-implanted areas. (b) C-V curves generated from the image acquired at different tip-bias voltages. (c) S<sub>11</sub> amplitude with topography (inset), and zoomed-in calibrated capacitance of a flat p-n junction structure. The three capacitance images have been acquired at -1V, 0V, and +1V tip bias, respectively. (d) Pointwise C-V spectroscopy curves in the four different spots labeled with crosses in (c).

calibrated SMM resistance as recently demonstrated (Brinciotti et al 2015). The method has been tested on two doped silicon samples and in both cases the resistivity and doping concentration are in quantitative agreement with the data-sheet values over a range of  $10^{-3} \Omega \text{ cm}$  to  $10^1 \Omega \text{ cm}$ , and  $10^{14} \text{ atoms cm}^{-3}$  to  $10^{20} \text{ atoms cm}^{-3}$ , respectively (Figure 5). The method does not require sample treatment like cleavage and cross-sectioning, and high contact imaging forces are not necessary, thus it is easily applicable to various semiconductor and materials science investigations. In Figure 5, the calibrated resistance and capacitance images show lower and higher values for the highly doped areas, respectively. Using an analytical method the SMM resistance image can be converted into a resistivity and dopant density image, which are true materials property images. Figure 5 compares the SMM resistivity and

doping concentration with the data-sheet values of the sample; a quantitative agreement is obtained for the entire range of doping concentrations. The lateral resolution of SMM is limited by the AFM tip probe apex radius while the bulk penetration is mainly determined by the skin-depth. The skin depth and therefore the microwave penetration depth can be changed by varying the SMM frequency. In this way, frequency dependent depth profiling can be realized with the SMM.

### Capacitance-voltage spectroscopy for further physics investigations

The nanoscale lateral resolution of SMM allows performing imaging of doped regions, interfaces, and junctions that often play a critical role in several semiconductor devices. Figure 6 shows a bi-polar

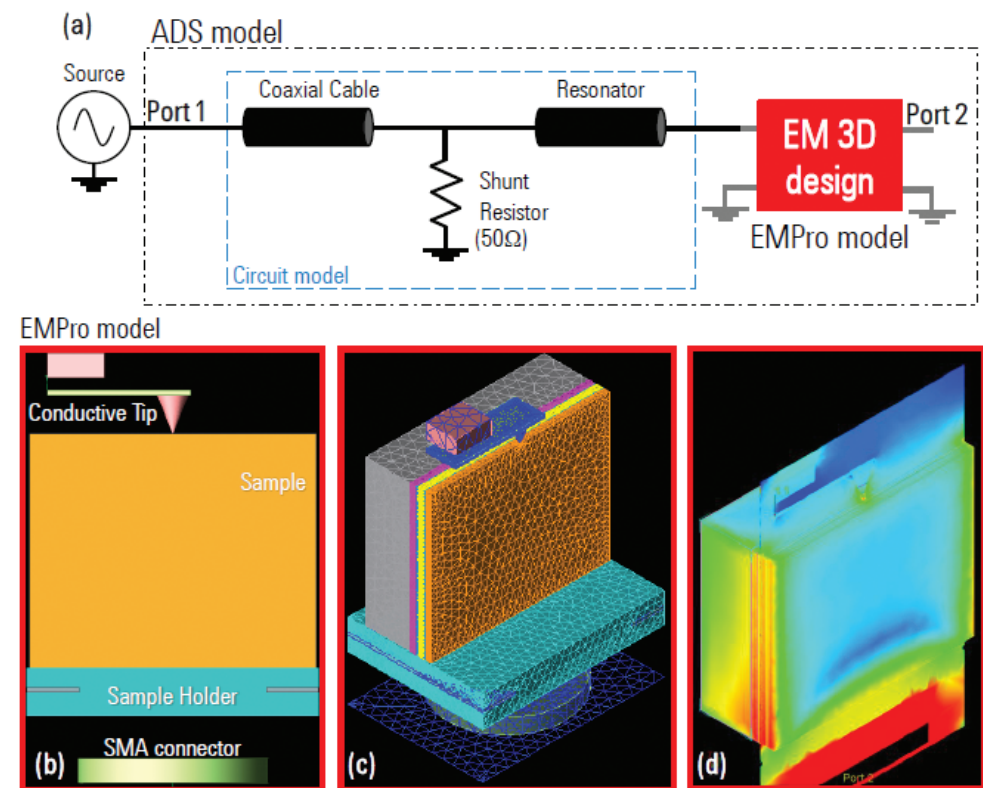


Figure 7. SMM modeling with ADS and EMPro. (a) Integrated 2D circuitry model (ADS) and 3D EM design block (EMPro model). (b) 3D CAD model of the cantilever and sample holder. (c) Meshed view in EMPro advanced mode. (d) E-Field distribution at 10 GHz.

doped silicon sample imaged with SMM to obtain calibrated capacitance images at different tip DC-voltage bias (Brinciotti et al 2016). The capacitance depends on the doping density and reaches up to 300 aF, with high capacitance values observed on highly doped silicon regions. The same area was imaged with different DC tip-bias voltages between -2V and +2V. By plotting the calibrated capacitance versus the tip-bias voltage the C-V curves can be generated for different locations. The conductive AFM-tip and the thin native silicon dioxide layer (~1 nm) with the doped silicon substrate below form a Metal-Oxide-Semiconductor (MOS) structure. In this configuration, for n-type doped silicon regions, an increase in capacitance at high positive voltages is measured, which is in line with the standard depletion zone model. The C-V curve for the p-type stripes shows high capacitance values at negative tip bias and low capacitance at positive tip bias which

follows also the textbook model. A second region of interest on the same silicon sample includes multiple p-n junction structures. The topography of the area is flat, and the structures are only visible in the electrical SMM images. Also in this case, the same area has been imaged multiple times in different DC tip bias conditions. From the capacitance images the different polarity of the n-type and p-type region can be determined. These results show that the C-V curves can be either determined pointwise at particular positions or also from the entire capacitance image acquired at different DC tip-bias voltages (Moertelmaier et al 2014).

### Nanoscale modeling with ADS and EMPro: GHz electric field distribution

Calibrated SMM experiments can be compared to quantitative modelling using two software

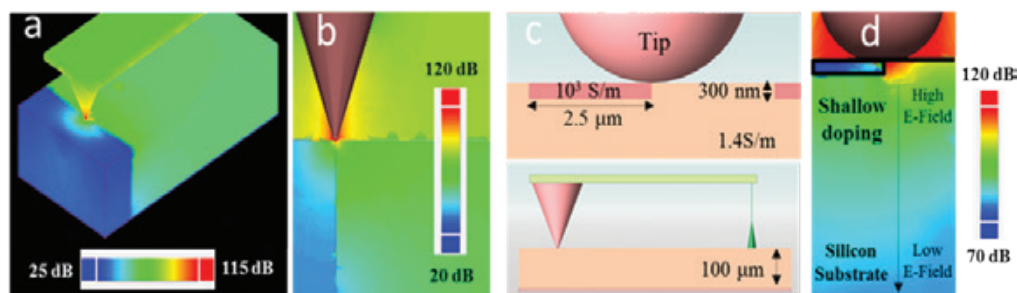


Figure 8. Nanoscale EMPro modeling. (a) 3D E-field distribution of the tip-sample system. The E-field is lower in the doped region with high-conductivity (shown in blue), than in the Si bulk of the sample (shown in green). (b) Cross-section of the 3D E-field distribution. (c) EM-Pro model geometry (bottom), with a zoom on a shallow doped structure (top). (d) Cross-section of the E-field distribution showing the tip-sample region. The E-field is highest close to the tip and decreases inside the sample.

packages, ADS and EMPro (Figure 7) (Kasper et al 2014b; Medina et al 2015). ADS is an electric circuit simulation tool that provides optimisation, tuning, network parameters, and the broadband frequency response for standard electronic components. ADS was used to model the broadband frequency  $S_{11}$  response of the coaxial cables and the impedance matching network, as well as the effect of the AFM tip in contact with different materials samples. The ADS frequency sweep simulations were compared to experimental SMM data and a quantitative agreement has been achieved. With the 3D modeling tool EMPro, calibrated SMM impedance images and electrical E-field distributions can be compared to 3D microwave finite element modelling. As

such, 3D electromagnetic full-wave simulations using EMPro can validate the calibrated complex impedance SMM measurements (Figure 7). EMPro supports two simulation engines, Finite Element Modelling (FEM) and Finite Difference Time Domain (FDTD). In FEM, the structure is included in CAD (Computer Aided Design) and meshing is done until the solutions for the Maxwell's equations in the mesh tetrahedrons converge to a certain threshold. Integrating the obtained electric and magnetic fields, the voltage and current is calculated in each mesh point. The ratio of voltage to current calculated by EMPro at the port gives the impedance, which corresponds to  $S_{11}$ . Both EMPro and ADS modelling are used to gain insights into the sub-surface imaging

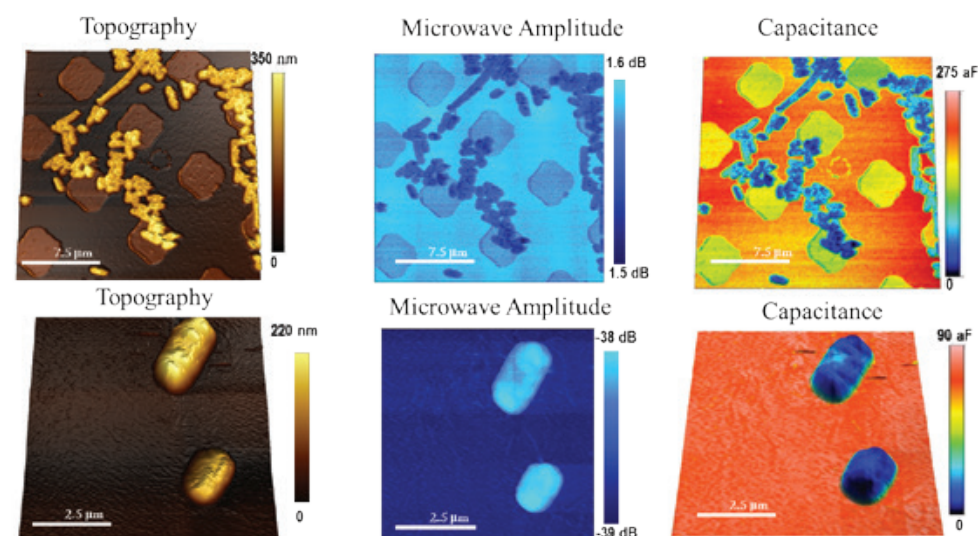


Figure 9. GHz image of single bacteria. Multitude of *E. coli* bacteria imaged with the SMM in air. The first column shows the AFM topography image, the second column shows the raw SMM  $S_{11}$  image. The last column shows the calibrated capacitance map overlaid on the 3D topography image.

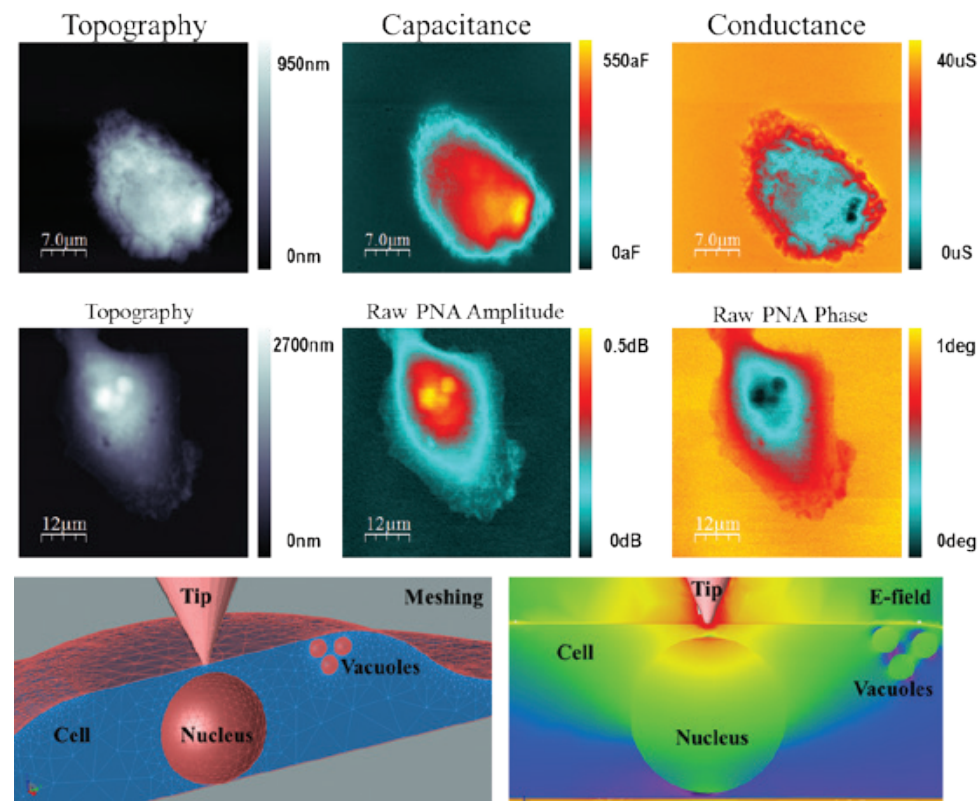


Figure 10. Upper row: Cell complex impedance imaging in air and liquid. Individual CHO (Chinese Hamster Ovary) cell imaged in air in intermittent contact mode showing topography, calibrated capacitance and conductance. Centre row: SMM images of individual ARPE-19 (Arising Retinal Pigmented Epithelia) cell fixed on Si<sup>+</sup>/SiO<sub>2</sub> substrate and imaged in liquid solution of medium, serum, and antibiotics. Lower row: Tip-cell interaction with 3D EMPro model mesh of the cantilever, tip, cell and Si substrate (left). EMPro simulation of E-field over cell surface and penetration into the cell (right).

capabilities of oxides and semiconductor materials at different frequencies and different experimental conditions. As such the modelling packages are useful for proper experimental SMM planning as well as further data interpretation at a quantitative level (Oladipo et al 2013).

Figure 8 shows how the 3D-field solver EMPro was used for nanoscale modeling of the E-field and impedance values of the SMM tip-sample system at 19 GHz (Brinciotti et al 2016). Figures 8a and 8b show a 3D distribution of E-field magnitude at the tip-sample system on the interface between two doped silicon regions having different doping concentrations. This geometry model describes, in a simplified way, the structure of the uni-polar n-type doped calibration sample, where the differently doped regions extend over the entire sample thickness. The E-Field has a maximum, as expected,

near the very end of the conductive tip. The part of the sample where the field is low (shown in blue in the color map), corresponds to the region with higher doping concentration. Accordingly, the field is higher (shown in green), in the region with lower conductivity (i.e. lower doping concentration). The modeling of the bulk sample was compared with a shallow doped sample where the doping extends only within few hundreds of nanometers from the sample surface (Figures 8c and 8d). For this reason a different CAD geometry has been designed to model the shallow stripes which are 300 nm high and 2.5 μm wide, with a conductivity of 1000 S/m. Figure 8d shows a cross-section of the tip-sample E-field distribution. The E-Field has a maximum close to the SMM tip, decreasing proportionally to the depth in the bulk region, whereas it reaches a relative minimum value inside the shallow doped



stripe, which is 300-nm thick. The penetration depth of the E-field is roughly half a micrometer into the sample, which is comparable with the doping depth.

## GHz images of single bacteria: capacitance and complex permittivity

The SMM was used to measure *Escherichia coli* (*E. coli*) bacteria in air and in liquid using intermittent contact mode for imaging soft materials (Figure 9) (Tuca et al 2016). Quantitative SMM calibration is achieved resulting in complex impedance images of bacteria in air including capacitance (aF; attoFarad) and conductance ( $\mu\text{S}$ ; microSiemens) images. *E. coli* bacteria were immobilised onto a silicon substrate and imaged with the SMM either in the dry state or in buffer solution. Figure 9, upper row, shows an ensemble of bacteria spread over the surface of Si and  $\text{SiO}_2$  pillars while the lower row shows two individual bacteria on Si. The topography images show *E. coli* bacteria with lengths of 2-3  $\mu\text{m}$  and a height of 300-350 nm. The SMM raw data includes the VNA amplitude image showing a good contrast between the bacteria,  $\text{SiO}_2$  pillars and the Si substrate.

The calibrated capacitance images show individual bacteria with a capacitance ranging from 20-100 aF depending on the tip diameter and the frequency. The capacitance of the bacteria as well as the  $\text{SiO}_2$  pads is lower than the capacitance of the substrate. This is consistent assuming a simple parallel plate capacitor model where the capacitance decreases with increasing distance of the tip and the substrate. The conductance channel shows no variation between bacteria and substrate (data not shown), which is expected from the non-conductive  $\text{SiO}_2$  pads and indicates that the bacteria have no electrical loss under the measurement conditions. In a subsequent study we quantified the electric permittivity of single bacterial cells at microwave frequencies and nanoscale spatial resolution by means of near-field scanning microwave microscopy (Biagi et al 2016). To this end, calibrated complex impedance and admittance images have been obtained at 19 GHz and analysed with a methodology that removes the non-local topographic cross-talk contributions and thus provides quantifiable intrinsic dielectric images of the bacterial cells. Results for single *Escherichia coli* provide a relative electric permittivity of  $\sim 4$  in

dry conditions and  $\sim 20$  in humid conditions, with no significant loss contributions. Present findings, together with the ability of microwaves to penetrate the cell membrane, open an important avenue in the microwave label-free imaging of single cells with nanoscale spatial resolution.

## Cell conductivity at humidity and living cell imaging at GHz frequencies

Figure 10 shows the topography and calibrated capacitance and conductivity images of single Chinese Hamster Ovary (CHO) cells acquired in air (upper row) and in liquid (middle row; living cell) (Tuca et al 2016). Due to their large dimensions, they can be easily spotted on the silicon substrate and individually scanned in contact mode. The topography images show a diameter of roughly 30  $\mu\text{m}$  and a height of more than 1  $\mu\text{m}$ . The obtained capacitance variation is induced by cell dielectric properties that are different from the Si substrate dielectric properties. Figure 10, lower row, shows the microwave interaction at the tip/sample contact point using EMPro. A 3D cell-like structure has been implemented in CAD including the cell nucleus, three vacuoles, and a 10nm thick membrane. The simulated values of admittance were obtained at different frequencies and positions over the cell, in order to investigate how different cell compartments influence the electric field distribution. Different dielectric values were used for the individual cell compartments including the lipid bilayer of the cell membrane ( $\epsilon_r=3.2$ ), the cell nucleus with DNA ( $\epsilon_r=8$ ) and a protein vacuole ( $\epsilon_r=3$ ). Depending on the cell and tip geometry, the simulated tip-cell complex admittance was determined as roughly  $Y=110 + j250 \mu\text{S}$ , which translates to conductance values of roughly 50  $\mu\text{S}$  and capacitance values of 500 aF. Those modeled EMPro values fit very well to the experimental SMM results. Figure 11 shows a set of CHO cell measurements with controlled variation of the ambient humidity, ranging from low humidity (2% RH) to high humidity (60% RH). The calibrated conductance values obtained at low

humidity are in the order of 10-20  $\mu\text{S}$ , whereas the values obtained at high humidity vary between 30-200  $\mu\text{S}$ , which can be explained with the variation of the conductivity based on the water content in the cells.

Figure 11 shows the variation of the complex permittivity of different aqueous solutions (pure water, PBS, and HBSS) with respect to frequency ranging from 1-20GHz, recorded with the dielectric probe kit (Tuca et al 2016). The choice of the three solutions should match the cell buffer solutions. The real part of the water dielectric constant thereby decreases from  $\sim 78$  at 2GHz to  $\sim 40$  at 20GHz. The imaginary part of the water relative permittivity is proportional to the conductivity, based on  $\epsilon'' = \frac{\sigma}{\omega \epsilon_0}$ , where  $\epsilon_0$  is the permittivity of free space, and  $f$  is the frequency. The dielectric probe measurements reveal a low value of the imaginary part of the relative permittivity ( $<10$ ,  $<1\text{S/m}$ ) at low frequency (2GHz) and a high value ( $\sim 35$ ,  $\sim 40\text{S/m}$ ) at high frequency (20GHz). The SMM cell conductivity can now be compared to the water conductivity at 20GHz. The SMM conductivity  $\sigma$  can be obtained from the conductance  $G$  in a simple first order approximation from the volume of the cell in contact with the SMM tip, and applying the relation  $\sigma = \frac{G}{A}$ , where  $A$  is the contact area and  $l$  is the cell height obtained from the topography image. The effective conductivity obtained from the SMM cell conductance, at  $f=20\text{GHz}$ , is  $\sim 18 \text{ S/m}$  and effective  $\sim 15$ , which is in the range of the bulk water permittivity. Accordingly, the cell conductivity behaviour at different frequencies and ambient humidity is similar to bulk water properties, corroborating the hypothesis that water is adsorbed on the cells' surface and that it is responsible for the microwave energy dissipation over the cells.

## Summary

The Keysight Scanning Microwave Microscope (SMM) consists of an AFM interfaced with a vector network analyser (VNA) allowing measurement of complex materials properties for nano-electronics,

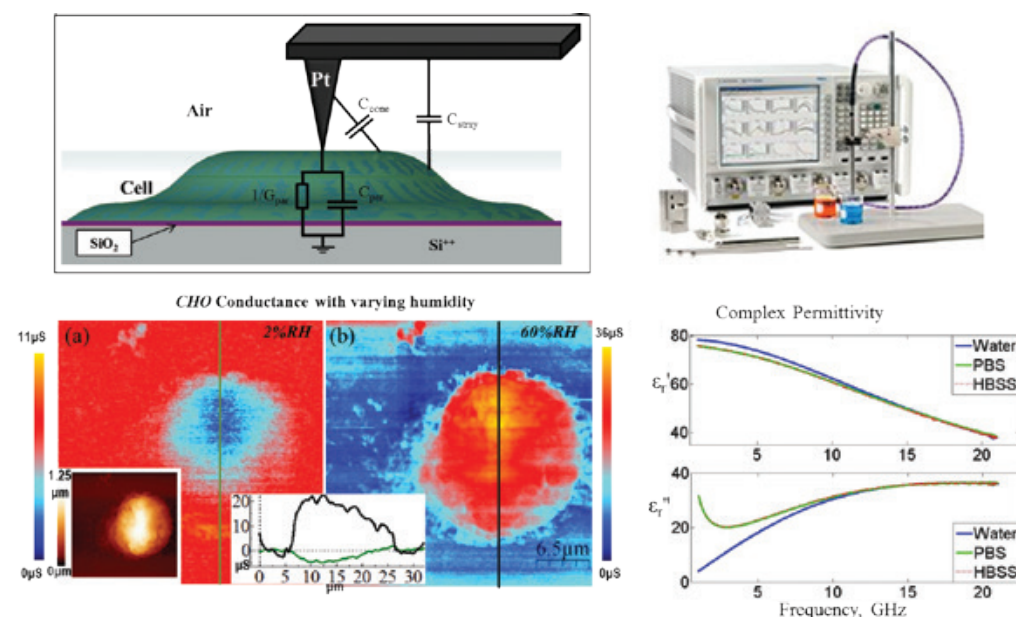


Figure 11. Cell conductivity at different humidity. SMM equivalent electric circuit model of the tip-cell interaction (upper left). Calibrated cell conductance images measured at different ambient humidities (lower left). Inset shows cell topography. Real and imaginary parts of water complex permittivity, 0.1M PBS and HBSS buffers as a function of frequency, over the range 1-20GHz (lower right), experimentally obtained using VNA and dielectric slim probe kit from Keysight (upper right).

materials science, and life science applications. The SMM operates at broadband frequencies between 1-20 GHz. We present novel calibration workflows for complex impedance imaging and dielectric quantification, as well as advanced voltage-spectroscopy measurements. Various nanodevices are studied including dopant profiling layers, semiconductor devices, buried oxide structures, and biomaterials like cells and bacteria. Due to the capability of the electromagnetic wave to penetrate the surface of the sample under study, the technique can be used to selectively image sub-surface features. Calibrated sub-surface and non-contact capacitance imaging of silicon samples is presented and dopant areas can still be detected under a silicon oxide layer. Finally, SMM imaging in buffer solution is presented including life cell imaging, and complemented by 3D EMPPro modeling at GHz frequencies.

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*"I would like to congratulate the graphic designer on the excellent layout and selection of the images. A great job has been done and I look forward to seeing the items in the December issue of Infocus Magazine when it is published"*

**Raymond Coleman**, author of *Virtual Microscopy* (December 2012 issue)