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**Broadband Scanning Microwave Microscopy of Phosphorene**

**Farina, Marco**  
**UNIVERSITA' POLITECNICA DELLE MARCHE**  
**PIAZZA ROMA 22**  
**ANCONA, ANCONA, 60121**  
**ITA**

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PI: Professor Marco Farina  
Department of Information Science  
Marche Polytechnical University, Ancona, Italy 60131  
Tel: +39 (071) 2204837  
Email: [m.farina@univpm.it](mailto:m.farina@univpm.it)

Co-PI: Professor James C. M. Hwang  
Department of Materials Science and Engineering  
Cornell University, Ithaca NY 14853-1501  
Email: [jch263@cornell.edu](mailto:jch263@cornell.edu)

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## Summary

This research project aims to develop a broadband scanning microwave microscope (SMM) for the characterization of the electromagnetic properties of 2D atomic-layered materials. The work was carried out through a close collaboration between the PI at the Marche Polytechnical University in Italy and the co-PI at Lehigh University (later Cornell University after the co-PI moved from Lehigh to Cornell) in the US. Experiments were conducted using both SMMs at Marche and Lehigh (Cornell). In-person visits, virtual meetings, and impromptu email and phone discussions occurred regularly between the two groups.

The main technical accomplishments through the past four years include:

1. Refined the SMM for imaging and quantitative characterization of 2D materials.
2. Invented an inverted SMM (iSMM) to improve its bandwidth, sensitivity, and dynamic range.
3. Developed a protocol to remove topography crosstalk from the SMM signal.

These results are described in the next three main sections. All referred publications related to the project are listed at the end.

## Table of Contents

Summary .....	1
List of Figures .....	2
List of Symbols, Abbreviations and Acronyms .....	3
Introduction .....	4
Refinement of the SMM for Imaging and Quantitative Characterization of 2D Materials and Structures .....	4
Invention of an Inverted SMM .....	8
Real-Time Removal of Topography Crosstalk .....	9
Conclusion .....	11
Refereed Publications .....	12

## List of Figures

Fig. 1. (a) Flat 2DEG, (b) patterned 2DEG, and (c) patterned 2DHG heterostructures. ....	5
Fig. 2. (a) AFM topography image, (b) SMM conductance/capacitance contrasts, (c) SMM conductance image, and (d) SMM capacitance image of a flat 2DEG heterostructure [Fig. 1(a)]. Dashed curves are simulated. ....	<b>Errore. Il segnalibro non è definito.</b>
Fig. 3. (a) AFM topography image, (b) SMM conductance/capacitance contrasts, (c) SMM conductance image, and (d) SMM capacitance image of a patterned 2DEG heterostructure [Fig. 1(b)]. Dashed curves are simulated. ....	6
Fig.4. (a) AFM topography image, (b) SMM conductance/capacitance contrasts, (c) SMM conductance image, and (d) SMM capacitance image of a patterned 2DHG heterostructure [Fig. 1(c)]. Dashed curves are simulated.....	7
Fig. 5. (a) Schematics and (b) 2-port equivalent circuit of an inverted scanning microwave microscope.....	<b>Errore. Il segnalibro non è definito.</b>
Fig. 6. iSMM PtSe <sub>2</sub> images of (a) Re(Y) and (b) Im(Y)/ $\omega$ , with surrounding gold appearing in blue. (c) and (d) are line scans of (a) and (b), respectively. Dotted curves are smoothed trends.....	9
Fig. 7. Measured (green dot) vs. simulated (curves) Re(Y) as a function of PtSe <sub>2</sub> conductivity. Inset shows details around 10 <sup>3</sup> S/m.....	10
Fig. 8. (a) AFM $h^*$ image, SMM $\angle S_{11}^*$ image, and linear scans across the dashed lines on the images. (b) topography-corrected $\angle S_{11}^* - h^*$ image, AFM PFM image, and linear scans across the dashed lines on the images.....	<b>Errore. Il segnalibro non è definito.</b>

## List of Symbols, Abbreviations and Acronyms

$\angle S_{11}^*$ : normalized phase of the reflection coefficient  
 $\Delta \mathbf{S}$ : scattering matrix variation  
 $\Delta \mathbf{S}^*$ : normalized scattering matrix variation  
 $\varphi$ : half cone angle of probe  
 $\sigma$ : electrical conductivity  
2D: two dimensional  
2DEG: two-dimensional electron gas  
2DHG: two-dimensional hole gas  
AFM: atomic force microscopy  
AlN: aluminum Nitride  
Al<sub>2</sub>O<sub>3</sub>: aluminum oxide  
AlGaN: aluminum gallium nitride  
BCl<sub>3</sub>: boron trichloride  
 $C_{BALL}$ : capacitance of probe tip  
 $C_{CANTI}$ : capacitance of probe cantilever  
 $C_{CONE}$ : capacitance of probe cone  
Co-PI: co-principal investigator  
dB: decibel  
G: ground line  
GaN: gallium nitride  
 $h$ : sample topography  
 $h^*$ : normalized sample topography  
 $H$ : probe body length  
HfZrO: hafnium zirconium oxide  
InAlN: indium aluminum nitride  
iSMM: inverted scanning microwave microscope  
PFM: piezoelectric force microscopy  
PI: principal investigator  
 $P_{in}$ : input power  
 $P_{out}$ : output power  
Pt: platinum  
PtSe<sub>2</sub>: platinum diselenide  
 $r$ : probe tip radius  
 $\mathbf{S}$ : scattering matrix  
 $S_{11}$ : reflection coefficient  
 $S_{21}$ : transmission coefficient  
Si: silicon  
SiC: silicon carbide  
SiO<sub>2</sub>: silicon dioxide  
SL: signal line  
SMM: scanning microwave microscopy  
SNOM: scanning near-field optical microscope  
STM: scanning tunneling microscope  
VNA: vector network analyzer  
 $Y$ : probe-sample interaction admittance

## Introduction

The project was planned for phosphorene, but switched to platinum diselenide (PtSe<sub>2</sub>), because monolayer PtSe<sub>2</sub> features a sizable bandgap, high carrier mobility, air stability, and can be synthesized in large areas. With the approval of Drs. Jason Foley and Dr. Kenneth Goretta of AFOSR, the scope of the project was expanded to include other promising 2D materials such as PtSe<sub>2</sub>. Besides PtSe<sub>2</sub>, we have also analyzed and characterized 2D electron and hole gases (2DEG and 2DHG) buried at the interface of AlN/GaN heterostructures. The methodology developed can be extended to many other 2D systems.

A major accomplishment in the project is the invention of an inverted scanning microwave microscope (iSMM). This new technique allows any existing scanning probe microscope such as an atomic force microscope (AFM), a scanning tunneling microscope (STM), or a scanning near-field optical microscope (SNOM), to be converted into an SMM with a simple modification of the sample holder. For example, different iSMMs were developed based on the Keysight SMM at Lehigh (Cornell) and the NT-MDT SMM at Marche. The iSMM exhibited better bandwidth, sensitivity, and dynamic range than traditional SMMs do. A calibration procedure was developed for the iSMM and used for quantitative characterization of PtSe<sub>2</sub>.

The topography crosstalk has been extensively analyzed. This is an artifact caused by the vertical movement of the SMM probe as it traces the sample surface. The movement changes the parasitic impedance of the probe even for a sample with uniform electromagnetic properties. This masks the intrinsic electromagnetic properties of the sample. Therefore, we developed an efficient technique to remove the topography crosstalk and to extract the intrinsic electromagnetic properties in real time. The technique is applicable to a wide range of materials and devices, including 2D materials and semiconductor heterostructures.

## Refinement of the SMM for Imaging and Quantitative Characterization of 2D Materials and Structures

The Lehigh (Cornell) SMM was modified to enable broadband sweep up to 20 GHz instead of a single frequency. The obtained SMM results confirmed the benefit of broadband SMM with improved sensitivity and image quality especially after time-gated postprocessing of data. Using the topography information simultaneously obtained by the AFM and the *in situ* extracted probe geometry, we de-embed from the topography-corrupted SMM data the sheet resistance of 2D electron or hole gas (2DEG or 2DHG) buried at the interface of an AlN/GaN heterostructure, including the lateral depletion of the 2DEG from an etched step. The SMM results are validated by Hall-effect measurements. With an improved setup, the SMM can be used to nondestructively monitor the sheet resistance of 2DEG or 2DHG during device manufacture. These studies help pave the way to 3D microwave tomography on the nanometer scale of multilayer structures.

Fig. 1 illustrates schematically the three types of AlN/GaN heterostructures used: (a) a flat 2DEG heterostructure, (b) a patterned 2DEG heterostructure, and (c) a patterned 2DHG heterostructure. As part of device fabrication, the active regions of 2DEG and 2DHG are isolated by selective-area ion bombardment or BCl<sub>3</sub>-based plasma etching. The former results in a flat surface; the latter results in steps of 39 nm and 200 nm on the 2DEG and 2DHG heterostructures, respectively. Both

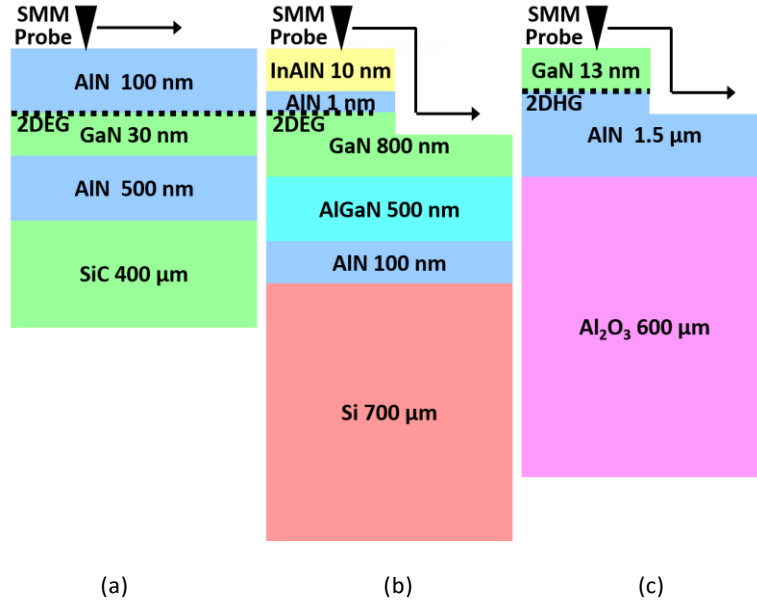


Fig. 1. (a) Flat 2DEG, (b) patterned 2DEG, and (c) patterned 2DHG heterostructures.

flat and patterned heterostructures are scanned at 6.2 GHz, a tradeoff between sensitivity and signal-to-noise ratio. The reflection coefficient  $S_{11}$  is calibrated by modeling the probe geometry with a half ball attached to a truncated cone, which is in turn attached to a cantilever. The slender cone minimizes the cantilever capacitance.

It is commonly assumed that the topography crosstalk on the total interaction capacitance, including that of the ball ( $C_{BALL}$ ), cone ( $C_{CONE}$ ), and cantilever ( $C_{CANTI}$ ), is a function of the probe height  $z$  only. This allows the topography crosstalk to be corrected by the  $z$  values measured by the AFM. However, when the topographic feature size (e.g., step height) approaches the probe size ( $r$ ),  $C_{BALL}$  and  $C_{CONE}$  can also be functions of the lateral distances  $x$  and  $y$  from the feature. Therefore, in this work, we use 3D COMSOL to simulate the topography crosstalk on  $C_{BALL}$  and  $C_{CONE}$  as the probe scans across an active region of 2DEG or 2DHG. The topography crosstalk on  $C_{CANTI}$  is neglected because it is estimated to be on the order of 0.01 fF, much smaller than the total capacitance contrast induced by the 2DEG or 2DHG as shown in Fig. 2, Fig. 3, and Fig. 4. These figures also show agreement between simulation and measurement.

Fig. 2 shows the AFM topography image and SMM interaction conductance and capacitance images for the flat 2DEG heterostructure [Fig. 1(a)]. It can be seen in Fig. 2(a) that the AFM image is featureless except for randomly distributed atomic steps of 0.3 nm. By contrast, the 2DEG region is distinguishable in the SMM images having average conductance contrast of  $0.47 \pm 0.30 \mu\text{S}$  and capacitance contrast of  $0.18 \pm 0.03 \text{ fF}$  with respect to the isolated region and consistent with that simulated as illustrated in Fig. 2(b). However, the conductance contrast can narrow down the range of the 2DEG sheet resistance to  $960 \pm 840 \Omega/\square$ , in general agreement with the Hall-measured value of  $450 \Omega/\square$ .

Fig. 3 shows that the active region of the patterned 2DEG heterostructure [Fig. 1(b)] is distinguishable in not only the SMM conductance/capacitance images, but also the AFM topography image as expected. Comparing the measured and simulated conductance/capacitance

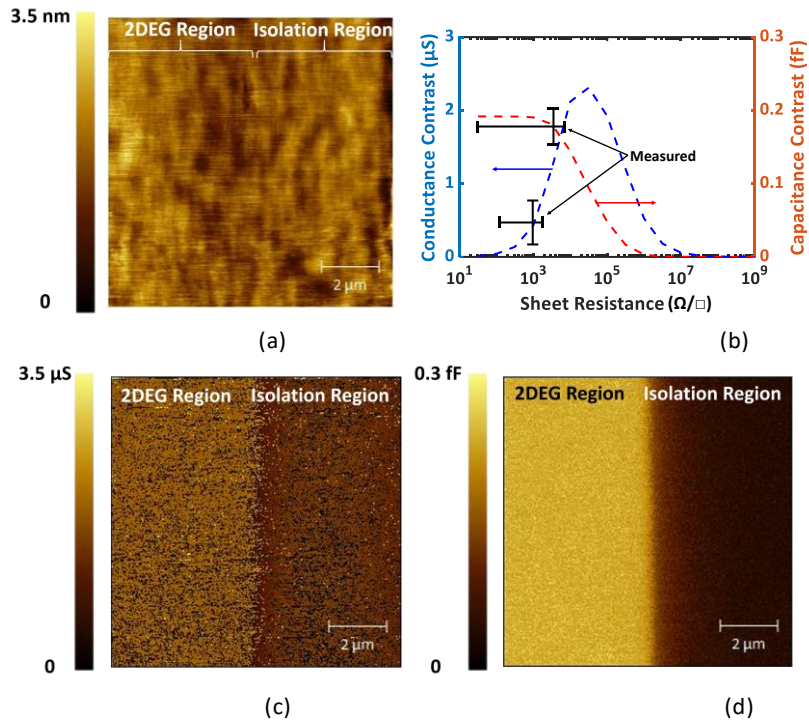


Fig. 2. (a) AFM topography image, (b) SMM conductance/capacitance contrasts, (c) SMM conductance image, and (d) SMM capacitance image of a flat 2DEG heterostructure [Fig. 1(a)]. Dashed curves are simulated.

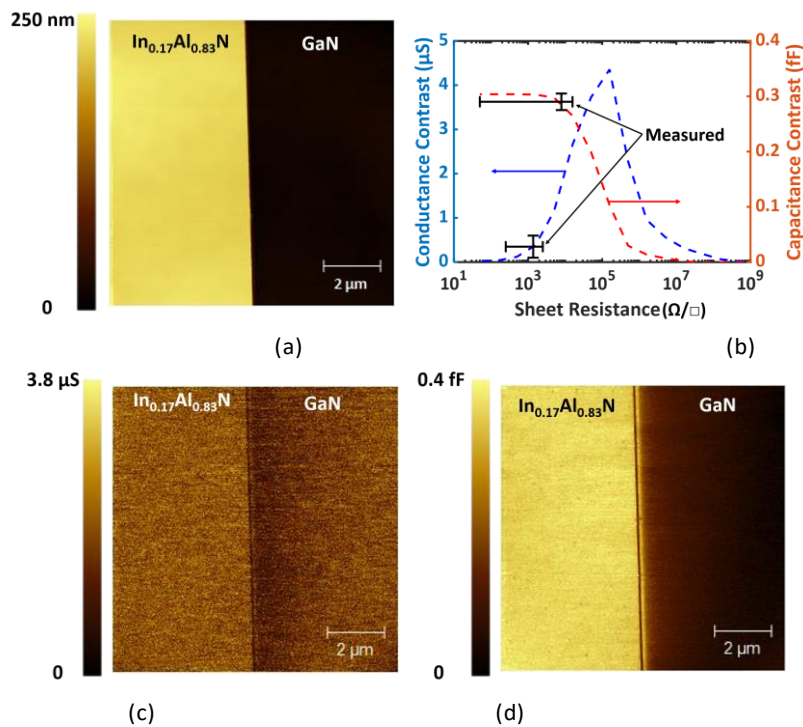


Fig. 3. (a) AFM topography image, (b) SMM conductance/capacitance contrasts, (c) SMM conductance image, and (d) SMM capacitance image of a patterned 2DEG heterostructure [Fig. 1(b)]. Dashed curves are simulated.

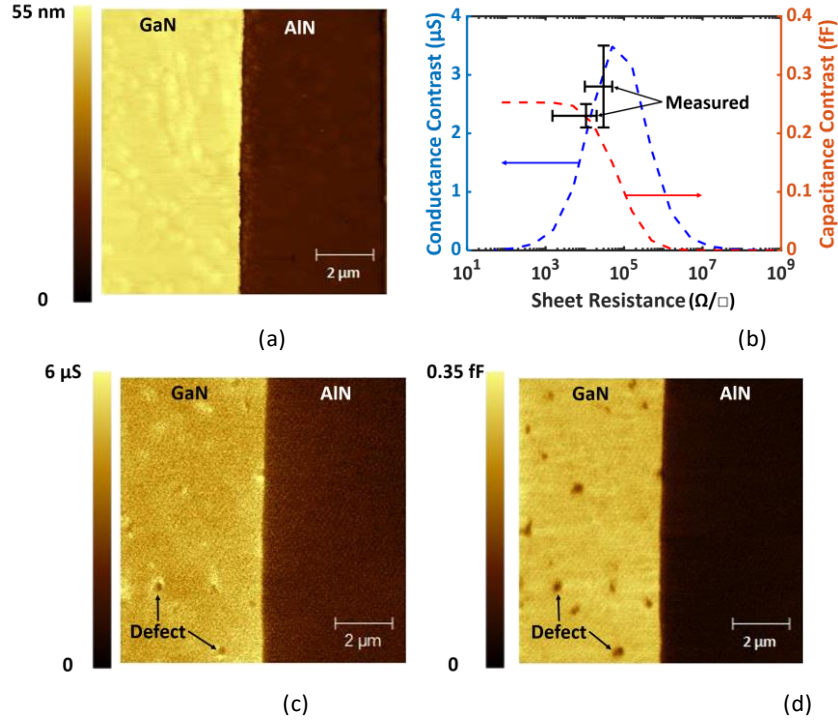


Fig. 4. (a) AFM topography image, (b) SMM conductance/capacitance contrasts, (c) SMM conductance image, and (d) SMM capacitance image of a patterned 2DHG heterostructure [Fig. 1(c)]. Dashed curves are simulated.

contrasts, the conductance contrast can again narrow down the 2DEG sheet resistance range to  $1.4 \pm 1.1 \text{ k}\Omega/\square$ , which is higher than the Hall-measured value of  $200 \Omega/\square$ . The difference may be due to the Schottky barrier between the SMM probe and  $\text{In}_{0.17}\text{Al}_{0.83}\text{N}$ , which partially depletes the 2DEG. Upon close examination, it can be seen that the contrast between  $\text{In}_{0.17}\text{Al}_{0.83}\text{N}$  and GaN is sharp in the AFM image, but an extra fringe beyond the edge is present in the SMM images. The extra SMM fringe can be explained by the finite SMM probe size compared to the step height. When the probe starts to roll off the edge, its overlap with the 2DEG decreases and so does the interaction capacitance. However, when the probe just lands on GaN, its side contacts the 2DEG edge, resulting in a local peak in the capacitance and an extra fringe in the image. The height of this peak and the depth of the valley before the peak may be modulated by the 2DEG depletion width  $d$  from the edge. Comparing the simulated and measured data, it is estimated that  $d \approx 0.2 \mu\text{m}$ . This shows that SMM can be used to quantify not only the sheet resistance of the 2DEG, but also the depletion width of the 2DEG from the etched step. The depletion width may result from both the Schottky barrier and the free surface, so their effects need to be separated by a more detailed analysis.

Similar to Fig. 3, Fig. 4 shows that the active region of the patterned 2DHG heterostructure [Fig. 1(c)] is distinguishable in not only the SMM conductance/capacitance images, but also the AFM topography image. As the probe scans from the GaN mesa down to the AlN region, the average contrast is  $39 \pm 2 \text{ nm}$  in topography,  $2.8 \pm 0.7 \mu\text{S}$  in conductance, and  $0.23 \pm 0.02 \text{ fF}$  in capacitance. It can be seen that the higher sheet resistance of the 2DHG makes it more sensitive to the SMM conductance contrast, so that it is more accurately determined to be  $13 \pm 5 \text{ k}\Omega/\square$ , which agrees with the  $15 \text{ k}\Omega/\square$  value of the Hall measurement. Compared to Fig. 3, the SMM images of the 2DHG do not show any extra fringe near the etched step, probably because the step height of 39

nm is much smaller than the probe radius. On the other hand, the SMM images show defect spots that are absent in the AFM image. These defects appear darker, indicating higher resistance than the surrounding 2DHG. The defect density is approximately  $3 \times 10^7 \text{ cm}^{-2}$ , which is orders-of-magnitude lower than the typical threading dislocation density in AlN and GaN layers grown on foreign substrates such as Si, SiC, and  $\text{Al}_2\text{O}_3$ . This suggests that not all dislocations are electrically active. The nature of these defects needs to be better characterized, because they can be scattering centers for mobile holes.

## Invention of an Inverted SMM

Traditionally, an SMM is modified from an AFM or a scanning tunneling microscope (STM), so that the probe can be scanned at a fixed height (0–1 nm) above the sample through the feedback control of atomic force or tunneling current. In either case, the microwave signal is injected through the probe by a vector network analyzer (VNA), and the signal reflected from the sample is also sensed by the VNA. Such a one-port reflection measurement usually has a dynamic range of 40–60 dB as limited by directional couplers.

To minimize the parasitic interaction and to boost the SMM sensitivity without resorting to a resonance circuit, we invented the iSMM. As shown schematically in Fig. 5(a), in an iSMM, the scanning probe is always grounded and the microwave signal is injected through a transmission line (e.g., a slot line, a coplanar waveguide, or a microstrip line) as part of the sample holder. Unlike the traditional SMM probe, the transmission line can have a broadband impedance match over many decades of frequency. The input and output of the transmission line are connected to the VNA, so that both reflection and transmission coefficients ( $S_{11}$  and  $S_{21}$ ) are measured. Such a two-port measurement usually has a dynamic range of 120–140 dB, making it easier to sense the perturbation when the probe scans across the sample.

With the microwave signal injected through the sample and the probe grounded, the parasitic interaction between the probe body and the surround is greatly reduced, because most of the surround is grounded in any case. Thus, compared to a traditional SMM, an iSMM can have wider dynamic range, higher sensitivity, and broader bandwidth (by making a resonance circuit unnecessary). Additionally, the probe can be a simple, rugged, and bio-compatible metal stylus. Meanwhile, whether the iSMM is modified from an AFM or STM, the original AFM or STM function is intact so that an iSMM image can be obtained simultaneously with an AFM or STM image.

For both SMM and iSMM, the intrinsic interaction between the probe and the sample can be represented by an admittance  $Y$ . The equivalent circuit of the iSMM is well defined as shown in Fig. 5(b) in which ABCD matrices represent the error networks related to cables, connectors, etc.

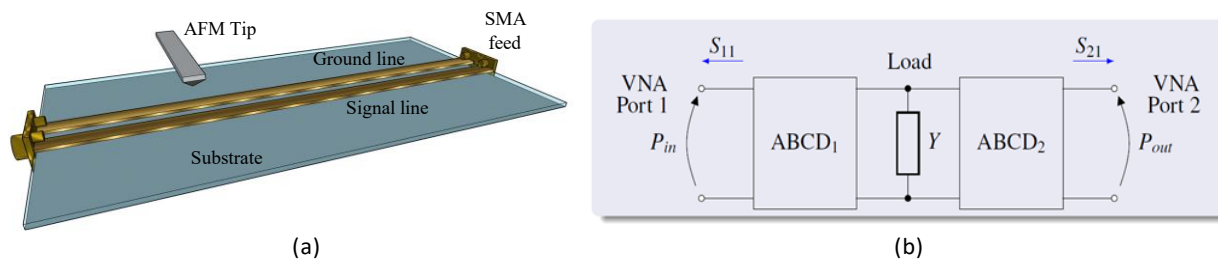


Fig. 5. (a) Schematics and (b) 2-port equivalent circuit of an inverted scanning microwave microscope.

Starting from the ABCD matrices, calibration of reflection and transmission parameters have been developed to de-embed  $Y$  from the measured data and to extract the intrinsic sample parameters. This calibration method has been validated through numerical simulations.

The iSMM was used to characterize exfoliated  $\text{PtSe}_2$  flakes that were transferred onto an iSMM sample holder.  $\text{PtSe}_2$  was selenized at  $400^\circ\text{C}$  from Pt sputtered onto Si. It was then transferred by an adhesive tape to the iSMM holder. The transfer resulted in flakes of multi-layer  $\text{PtSe}_2$  with properties similar to those of bulk  $\text{PtSe}_2$ .

With proper calibration, the intrinsic probe-sample admittance  $Y$  is extracted from  $S_{21}$  measured at 4.7 GHz on  $\text{PtSe}_2$  and split into real and imaginary parts as shown in Fig. 6(a) and Fig. 6(b), respectively. Line scans from these figures are plotted in Fig. 6(c) and Fig. 6(d), respectively, to quantify the  $\text{PtSe}_2$  conductivity. Comparing the measured and simulated real  $Y$  (Fig. 7), it is estimated that the  $\text{PtSe}_2$  conductivity is in the range  $10^3$ – $10^4$  S/m, consistent with the SMM result on 3-nm thick  $\text{PtSe}_2$ . However, it can be seen in Fig. 7 that this range of conductivity is beyond the most sensitive range of the current iSMM.

## Real-time Removal of Topography Crosstalk

For the first time, we developed a technique to remove the topography crosstalk in real time during an SMM scan. The technique was proved on a hafnium-zirconium oxide ( $\text{HfZrO}$ ) ferroelectric sample with a rough surface. By removing the topography crosstalk caused by the rough surface, the ferroelectric domain structure is revealed independent of the topography. The

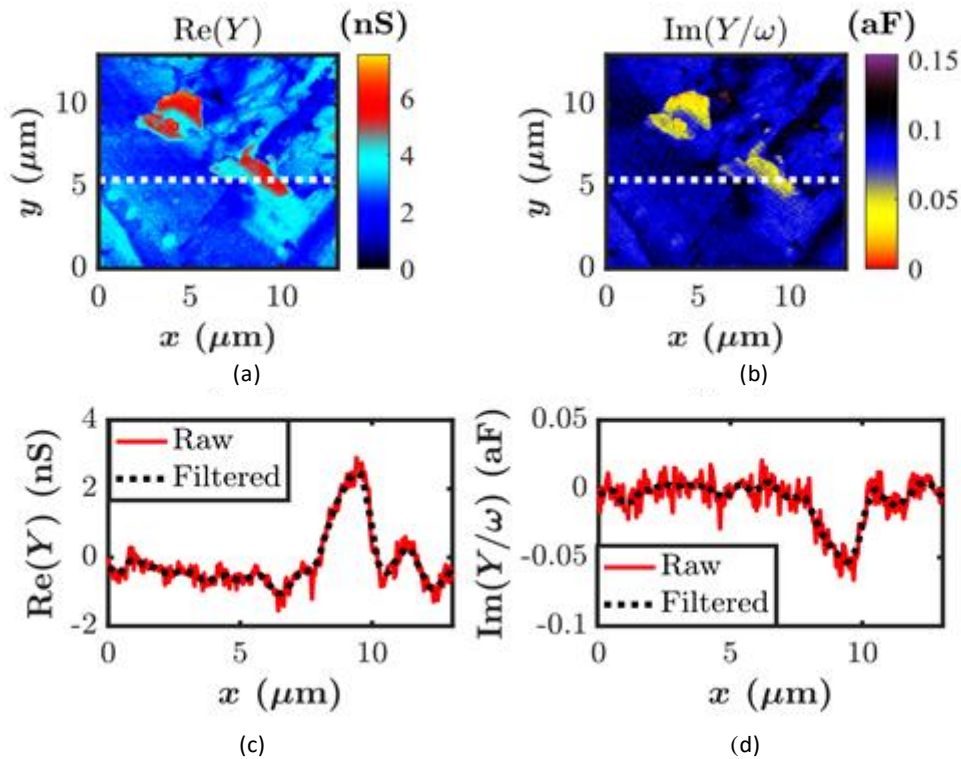


Fig. 6. iSMM  $\text{PtSe}_2$  images of (a)  $\text{Re}(Y)$  and (b)  $\text{Im}(Y/\omega)$ , with surrounding gold appearing in blue. (c) and (d) are line scans of (a) and (b), respectively. Dotted curves are smoothed trends.

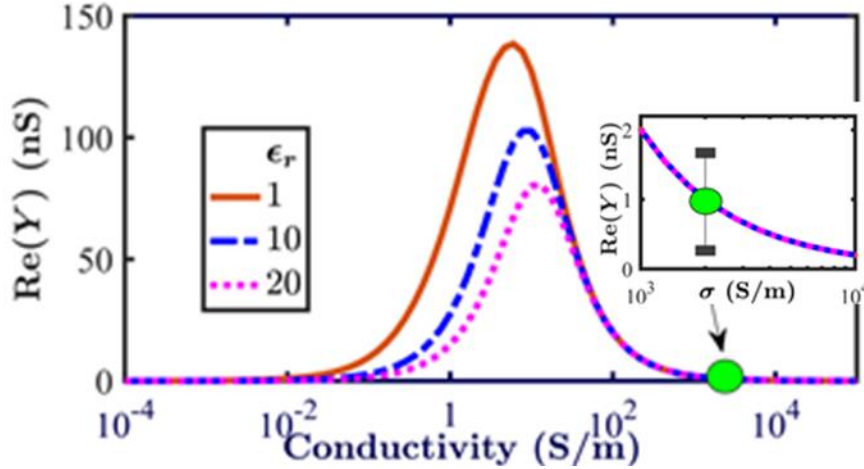


Fig. 7. Measured (green dot) vs. simulated (curves)  $\text{Re}(Y)$  as a function of  $\text{PtSe}_2$  conductivity. Inset shows details around  $10^3$  S/m.

effectiveness of the technique was validated by operating the AFM in the piezoelectric force microscopy (PFM) mode, which is known to be sensitive to ferroelectric domain structure and not the topography.

The SMM signal can be represented by a matrix  $\mathbf{S}$  of scattering parameters as sensed by the VNA through the probe. During an SMM scan, small variations of  $\mathbf{S}$  reflect changes in both the topography and the electromagnetic properties of the sample. Knowing the topography by the simultaneous AFM scan, the topography crosstalk can be approximated by assuming a linear relationship between the perturbation  $\Delta\mathbf{S}$  and the topography without laborious 3D finite-element multiscale simulation. This greatly speeds up the removal of the topography crosstalk, making it possible to be done in real time during the SMM scan. Consequently, this provides instant feedback of the scan area and can be used by the operator to zero in on the most interesting area of the sample. The real-time removal of the topography crosstalk includes the following major steps:

1. Acquire simultaneously SMM and AFM signals in terms of  $\Delta\mathbf{S}$  and  $h$ , respectively.
2. Correct  $\Delta\mathbf{S}$  for the macroscopic tilt of the sample and normalize it between 0 and 1 as  $\Delta\mathbf{S}^*$ .
3. Correct  $h$  for the macroscopic tilt of the sample and normalize it between 0 and 1 as  $h^*$ .
4. Subtract  $h^*$  from  $\Delta\mathbf{S}^*$ .

For proof of concept, the sample is a 6-nm-thick  $\text{HfZrO}$  layer grown by atomic layer deposition on a  $\text{Pt/SiO}_2/\text{Si}$  substrate. The sample surface roughness is on the order of  $1\ \mu\text{m}$ , which reflects mainly the roughness of the substrate. Crosstalk from such a rough topography completely masks the electromagnetic properties of the sample in the SMM image, which mandates the removal of the topography crosstalk. All SMM scans are performed at 4.5 GHz where the reflection coefficient  $S_{11}$  reaches a minimum and the signal-to-noise ratio reaches a maximum.

Fig. 8(a) shows that, because of severe topography crosstalk, the SMM  $S$ -parameter image  $\angle S_{11}^*$  resembles the AFM topography image  $h^*$ , where  $\angle S_{11}^*$  is the normalized reflection coefficient angle and  $h^*$  is the normalized topography. Fig. 8(b) shows that, with real-time removal of the topography crosstalk, the  $\angle S_{11}^* - h^*$  image resembles the AFM PFM image with ferroelectric

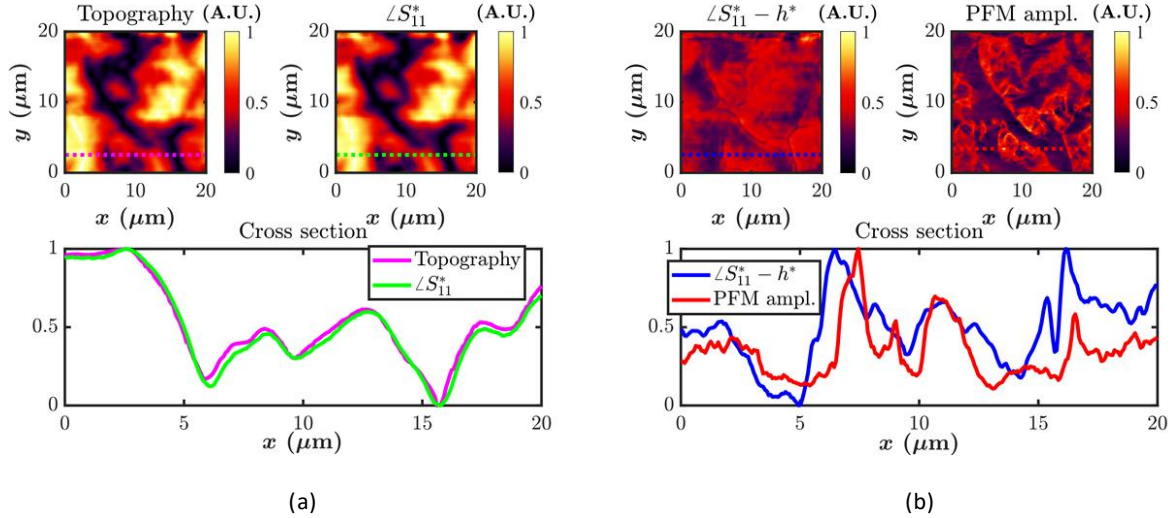


Fig. 8. (a) AFM  $h^*$  image, SMM  $\angle S_{11}^*$  image, and linear scans across the dashed lines on the images. (b) topography-corrected  $\angle S_{11}^* - h^*$  image, AFM PFM image, and linear scans across the dashed lines on the images.

domains delineated. Note that the PFM image is shifted slightly with respect to the SMM image, because, unlike the topography image, it is not simultaneously acquired with the SMM image.

## Conclusion

During the project period, different approaches are developed to refine the SMM for the characterization of 2D materials and structures. The main results obtained are:

1. Refinement of the conventional SMM, including improved bandwidth and sensitivity. The improvements are demonstrated on the 2DEG and 2DHG in AlN/GaN heterostructures for the characterization of sheet resistance, depletion width, and high-resistance point defects. The measure sheet resistances are consistent with Hall-measured values, whether the heterostructure is flat or not. Using this approach, the SMM may be used to nondestructively monitor the sheet resistance of low-dimensional materials in actual device structure during fabrication.
2. iSMM, a new variation of the SMM, has been invented with improved sensitivity, bandwidth, ruggedness, and convenience over the traditional SMM. The associated theory and calibration procedure for iSMM has also been developed. All measured results are compared with that simulated by 3D multi-scale full-wave electromagnetic simulations to quantify the conductivity and dielectric constant of the samples. This enables the characterization of the electrical conductivity of PtSe<sub>2</sub>.
3. A procedure for the fast and effective removal of the topography crosstalk has also been invented, allowing real-time visualization of the electromagnetic properties of the sample in the SMM. The procedure is flexible and easy to use. It does not require any additional measurement besides the sample topography acquired simultaneously by the AFM. It can potentially be applied to a wide variety of samples, including ferroelectric and semiconductor materials.

## Refereed Publications

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