

GaN nanowire coated with atomic layer deposition of tungsten: a probe for near-field scanning microwave microscopy

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Abstract

GaN nanowires were coated with tungsten by means of atomic layer deposition. These structures were then adapted as probe tips for near-field scanning microwave microscopy. These probes displayed a capacitive resolution of ~ 0.03 fF, which surpasses that of a commercial Pt tip. Upon imaging of MoS₂ sheets with both the Pt and GaN nanowire tips, we found that the nanowire tips were comparatively immune to surface contamination and far more durable than their Pt counterparts.

Keywords: atomic layer deposition, gallium nitride, molybdenum disulphide, nanowires, scanning microwave microscopy, tungsten

(Some figures may appear in colour only in the online journal)

Introduction

The need to better characterize advanced materials at the nanoscale has precipitated the development of a multitude of scanning-probe instruments. Since resolution and repeatability generally depend upon both the probe's dimensions and material properties, careful attention to its design is essential for advancing state-of-the-art imaging. The advent of well-controlled nanostructures, including nanoparticles, nanotubes, and nanowires, provides new opportunities for realizing probes capable of exploiting their unique architecture in order to obtain enhanced measurement capabilities. While modified AFM probes were initially dominated by carbon nanotube-based atomic force microscopy tips [1–3], new research has expanded their applications to tip-enhanced Raman spectroscopy [4], scanning tunneling microscopy [5], near-field optical microscopy [6], and scanning electrochemical microscopy (SEM) [7], among many others. In this paper, we present a tungsten (W) atomic layer deposition (ALD) NW-based probe well suited for near-field scanning

microwave microscopy (NSMM). The probe's robust, high-aspect ratio design makes it an ideal platform for use in alternative scanning-probe microscopes.

NSMM provides a non-destructive technique for studying nanoscale-properties for a variety of materials [8–14]. Its broadband illumination extends device characterization into the gigahertz range, complementing scanning capacitance and scanning impedance microscopy. Tracking changes in evanescent fields at the tip-sample interface, typically as a change in the microwave reflection coefficient S_{11} , enables simultaneous mapping of topography, conductivity, and permittivity [15–20]. Recent work emphasizes improved quantitative analysis of NSMM through the use of calibration standards. This approach enables the conversion of the measured S_{11} signal into the complex impedance plane and the separation of conductance and capacitance contributions [21–27]. Reliable calibrations require an understanding of the effective electromagnetic probe radius during scanning in addition to knowledge of the device under study [28, 29]. While this can be assessed through a variety of means

Table 1. ALD surface reactions for buffer, microwave pathway, and capping layers.

Al ₂ O ₃	A	AlOH(see footnote 3) + Al(CH ₃) ₃ → AlOAl(CH ₃) ₂ (see footnote 3) + CH ₄
	B	AlCH ₃ (see footnote 3) + H ₂ O → AlOH(see footnote 3) + CH ₄
W	A	WF ₅ (see footnote 3) + Si ₂ H ₆ → WSiF ₂ H(see footnote 3) + SiF ₃ H + 2H ₂
	B	WSiF ₂ H(see footnote 3) + WF ₆ → WWF ₅ (see footnote 3) + SiF ₃ H

including measuring capacitive approach curves, contact scanning-induced wear may alter probe dimensions thus necessitating an updated calibration between imaging runs.

We reported on the fabrication of a GaN NW probe exhibiting improved mechanical wear-resistance and comparable microwave sensitivity to a widely used commercial Pt tip [30]. Here, by significantly improving the microwave pathway with a W film through ALD, we have increased the capacitive resolution over an order of magnitude from ~ 0.7 fF to ~ 0.03 fF. Owing to the conformal nature of ALD with respect to the evaporative metal coating used previously, the effective tip diameter was reduced by ~ 60 nm, resulting in increased microwave and topographical spatial resolution. These improvements are further detailed through imaging of 2D MoS₂ sheets on a SiO₂ substrate. Here, the NW probe's high-aspect ratio and flexible structure appeared to reduce its sensitivity to surface contamination, yielding improved scanning contrast.

Experimental method

The NSMM used to conduct these scans consists of an Agilent 5400³ atomic force microscope (AFM) that incorporates a vector network analyzer (VNA) to source and receive microwave signals in the 1–20 GHz range. An integrated phase shifter improves broadband sensitivity by matching sample and transmission line (50Ω) impedance with the complete microwave circuit described in detail elsewhere [22, 31]. The GaN NWs used for the fabrication of this probe were grown through catalyst-free molecular-beam epitaxy on a Si <111> substrate with an AlN buffer layer [32–34]. The NWs form a wurtzite crystalline structure and are essentially defect-free owing to their high-temperature, ultra-high-vacuum growth environment. A similar fabrication process, previously established in [27], was used to manufacture the probe. As before, a tipless Si cantilever (12 kHz CLR-10 tip by VISTAprobes) (see footnote 3) was used for the probe's foundation. To create a microwave pathway from the AFM chip body to the tip, the entire structure was coated using ALD with a structure comprised of 30 cycles Al₂O₃ (3.5 nm)/63 cycles W (25.6 nm)/10 cycles Al₂O₃ (1.2 nm) [35–37]. The depositions were conducted in a viscous flow stainless steel ALD reactor with an inner diameter of ~ 23 cm. The reactor body walls were kept constant at 130 °C while the precursor vessels were held at room temperature. N₂ was used as the

³ Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification implies neither recommendation nor endorsement by NIST, nor does it imply that the materials used are necessarily the best for the purpose.

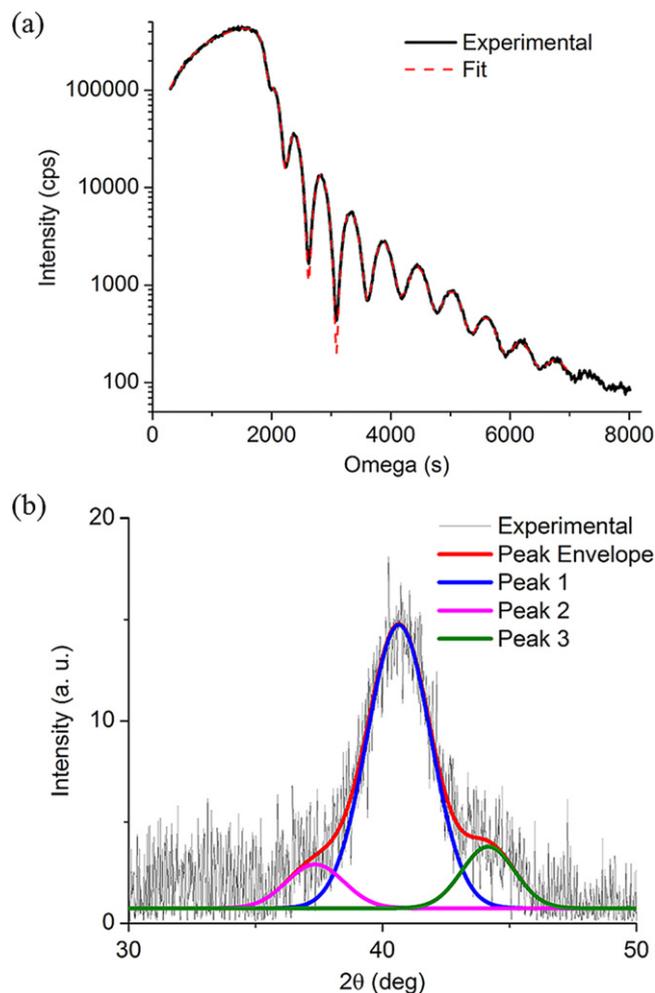


Figure 1. (a) XRR experimental data was used to independently determine the ALD thicknesses. The buffer Al₂O₃ layer is 3.5 nm, the W layer is 25.6 nm, and the capping Al₂O₃ layer is 1.2 nm. (b) XRD experimental results along with fitted peaks. The presence of a sharp center peak as well as a total of three peaks between 30° and 50° indicates the presence of mostly β -phase W. The three peaks are shifted slightly from the calculated peak positions of 34.3°, 39.6°, and 43.6°, indicating the presence of stress in the film [39].

carrier gas, with each precursor line having a flow of 40 sccm. Surface reactions for Al₂O₃ and W are provided in table 1. Deposited thicknesses were independently measured through x-ray reflectivity (XRR) (figure 1(a)), while x-ray diffraction (XRD) data indicated the deposited metal to be comprised primarily of β -phase W [38] (figure 1(b)). Figure 2(a) is an SEM image showing the NW probe, post-ALD. The NW protrudes $\sim 5 \mu\text{m}$ from the Si base and has a radius of ~ 120 nm. The uniform coverage afforded by ALD enables a

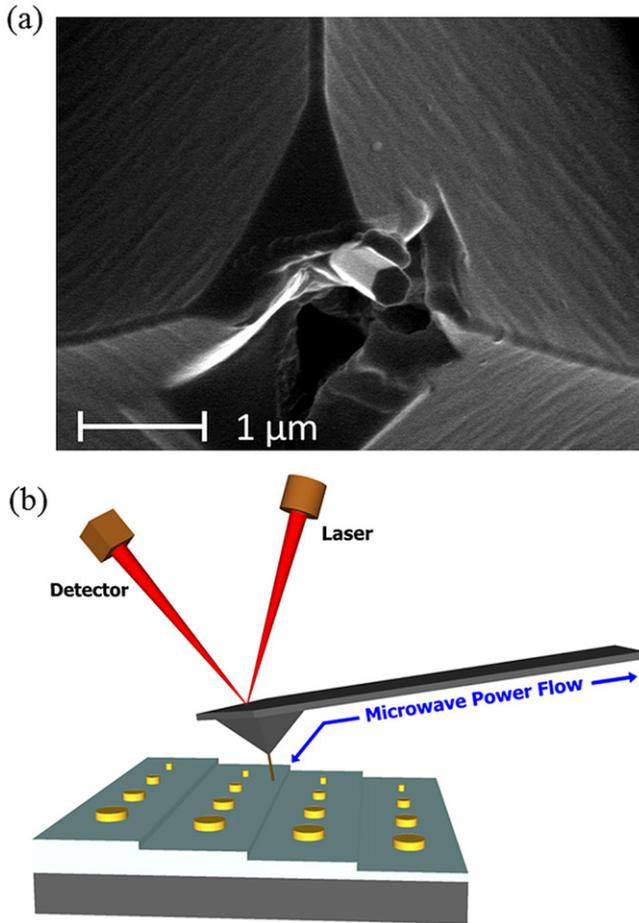


Figure 2. (a) SEM image of fabricated GaN NW probe post W ALD. (b) Illustration of microcapacitor calibration sample with 1–4 μm diameter Au microcapacitors on 10 nm SiO_2 steps with a silicon substrate.

continuous electrical pathway free of pinholes and cracks without significantly increasing the mechanical probe radius during scanning.

Results and discussion

A microcapacitor sample presented in detail previously was used to calibrate the capacitance resolution of the ALD NW probe [22] (figure 2(b)). The sample consists of a silicon substrate with four 10 ± 0.3 nm SiO_2 steps ranging in thickness from 10 nm to 40 nm. Each SiO_2 step is patterned with four circular metal pads comprised of 20 nm Ti/200 nm Au with diameters of 1 μm –4 μm . Prior to scanning, the W-ALD NW probe was brought into contact over the 40 nm thick SiO_2 step, and the resonance peak was tuned to -50 dBm at a frequency of 2.3 GHz. The VNA measuring frequency was offset from the minimum and held fixed during a $50 \mu\text{m} \times 50 \mu\text{m}$ scan. In figure 3(a), the improved microwave contrast of the W-ALD NW probe can be clearly seen relative to the original Ti/Al NW probe (figure 3(b)). The difference in microwave signal, or $|\Delta S_{11}|$, between each Au microcapacitor pad and the surrounding SiO_2 step on which the pad

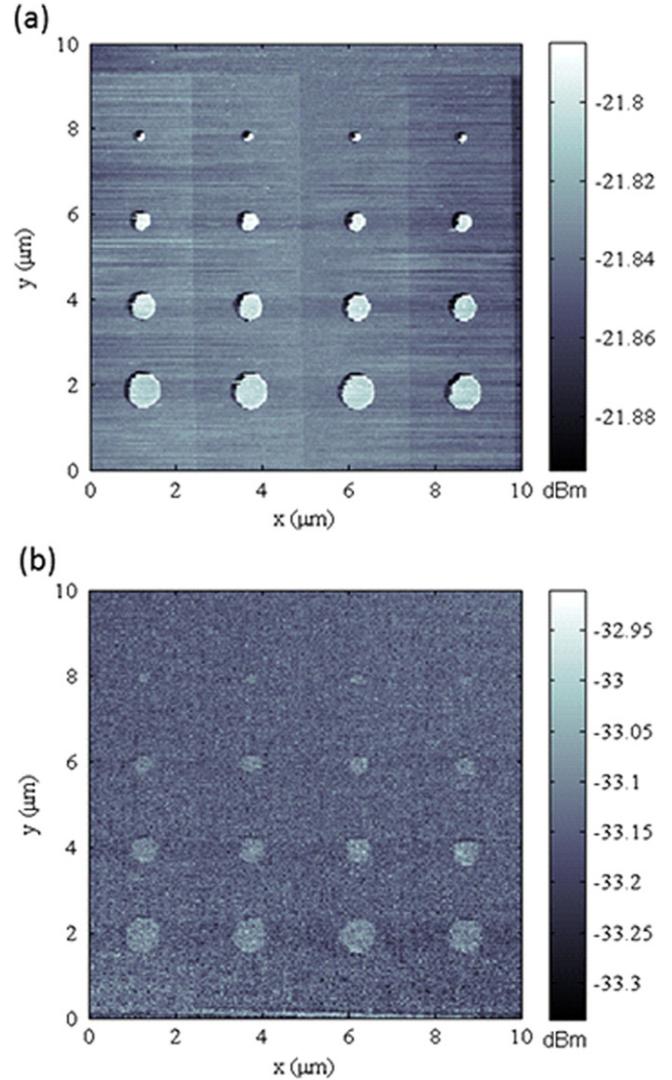


Figure 3. (a) Microwave reflection coefficient S_{11} image taken with the W-ALD NW probe (tip radius of ~ 150 nm reported in [27]) showing contrast for both the microcapacitors and SiO_2 steps. (b) S_{11} image taken with the Ti/Al. SiO_2 steps are not visible while microcapacitor edge resolution is reduced. Both images were recorded at 2.3 GHz with VNA power set to 5 dBm and a scan speed of 0.3 ln s^{-1} .

was recorded. S_{11} is given by

$$S_{11} = \frac{Z_L - Z_0}{Z_L + Z_0}, \quad (1)$$

with Z_L being the load impedance and Z_0 being the source impedance (transmission line impedance with a value of 50Ω). ΔS_{11} thus measures a change in S_{11} and consequently the change in Z_L as the probe passes from a SiO_2 region to a metal pad. Following [12], the $|\Delta S_{11}|$ data were then converted to capacitance using the relation $C_{\text{tot}} = \alpha^* |\Delta S_{11}|$ and fitted with a circuit model. The model consists of stray capacitance caused by the cantilever C_{cant} in parallel with three additional capacitances in series: tip capacitance C_{tip} due to the 12 nm Al_2O_3 ALD passivation layer, dielectric capacitance C_{diel} from the SiO_2 layer under the microcapacitor, and back or parasitic

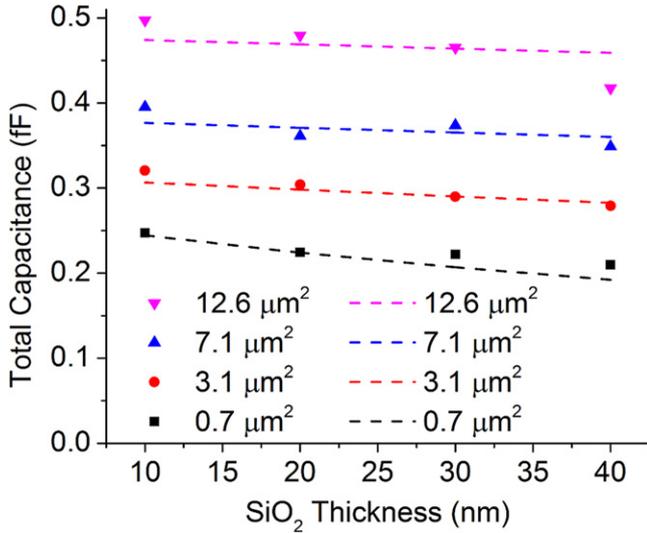


Figure 4. $|\Delta S_{11}|$ between each microcapacitor and background SiO_2 converted to capacitance and plotted as a function of SiO_2 thickness (solid symbols). Experimental data for $3.1 \mu\text{m}^2$ microcapacitors was fitted (dashed lines) using C_{back} and α as fitting parameters. These parameters were then held constant for the other three microcapacitor areas shown.

capacitance C_{back} due to fringing effects and depletion in the silicon substrate. Because $|\Delta S_{11}|$ data are presented as a difference measurement, effects caused by the largely constant C_{cant} may be ignored. C_{back} is treated as a fitting parameter that scales relative to the area of the microcapacitor in question with values on the order of 0.3 fF. C_{tip} and C_{diel} are modeled as simple parallel-plate capacitors governed by

$$C_{\text{tip}} = \frac{\epsilon_{\text{tip}} A_{\text{tip}}}{t_{\text{tip}}};$$

$$C_{\text{diel}} = \frac{\epsilon_{\text{diel}} A_{\text{diel}}}{t_{\text{diel}}};$$

$$\frac{1}{C_{\text{tot}}} = \frac{1}{C_{\text{tip}}} + \frac{1}{C_{\text{diel}}} + \frac{1}{C_{\text{back}}},$$

where A_{tip} is the area of the NW surface, A_{diel} is the area of the microcapacitor, t_{tip} is the thickness of the outer Al_2O_3 ALD layer, t_{diel} is the thickness of the SiO_2 layer, ϵ_{tip} is the permittivity of Al_2O_3 , and ϵ_{diel} is the permittivity of SiO_2 . To obtain the plot shown in figure 4, the model was fitted to the experimental data for the $3.1 \mu\text{m}^2$ microcapacitors with $\alpha = 0.5 \text{ fF}/\Delta\text{dBml}$. The α and C_{back} fitting parameters were held constant and applied to the other three microcapacitor sizes. As expected, increasing microcapacitor area and decreasing dielectric thickness correlate with an increase in measured total capacitance. The capacitance model tracks these trends well with deviations likely attributed to error caused by fringe effects in the parallel-plate capacitors and a variable C_{tip} due to bending of the NW during scanning.

To determine the resolution limit and measurement noise floor attributed to the W-ALD NW probe and microscope, a line scan across the SiO_2 steps without the presence of microcapacitors was taken. Here, the total capacitance model

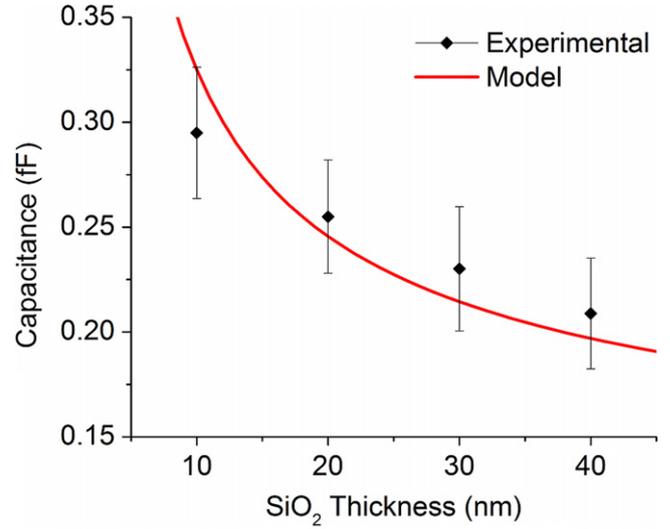


Figure 5. Measured capacitance of each SiO_2 step (black diamonds). Error bars represent measurement noise during scanning across each step. Data are fitted with a capacitance model with fringe capacitance across the SiO_2 layer taken into account. The model assumption that the NW end facet radius is the electromagnetic radius shows good agreement with the measured data to within 10%.

is reduced to C_{cant} in parallel with C_{tip} and C_{step} in series. C_{tip} is once again modeled as a simple parallel-plate capacitor and retains the same value from the model presented above. C_{step} , however, must account for fringing effects, as the ratio of the parallel-plate radius (tip radius) R to half the dielectric SiO_2 thickness d ranges from 6–12. Fringe capacitance for a parallel plate capacitor is dependent upon a dimension characteristic of the width of the electrode, such as the disc diameter. The Kirchhoff–Hutson expression was derived as a first-order approximation for calculating total capacitance, however, its error increases for small R/d values. As such, an improved empirical equation incorporating higher order terms into the Kirchhoff–Hutson expression, with error <1% when compared to numerical simulations, was used [40]:

$$\frac{C_{\text{diel}}}{C_{\text{elem}}} \approx 1 + \frac{2d}{\pi R} \ln \frac{8\pi R}{ed} + \left(\frac{d}{\pi R} \ln \frac{d}{8\pi R} \right)^2;$$

$$C_{\text{tot}} = \left[\frac{1}{C_{\text{tip}}} + \frac{1}{C_{\text{diel}}} \right]^{-1} + C_{\text{cant}},$$

where C_{elem} is the parallel-plate model, d is half the SiO_2 thickness, e is the permittivity of free space, and R is the NW radius. From figure 5, it can be seen that the SiO_2 steps are at the measurement threshold for the W-ALD NW probe, because 10 nm changes in SiO_2 thickness correspond to a ~ 0.03 fF change in capacitance, with measurement noise for each step also corresponding to ~ 0.03 fF. C_{cant} was determined by measuring $|S_{11}|$ as a function of tip-sample separation with a $2 \mu\text{m}$ approach curve. By this method, the stray capacitance contribution of C_{cant} was found from a linear relation to be $0.6 \pm 0.04 \text{ aF nm}^{-1}$, indicating that each 10 nm increase in topography due to the SiO_2 staircase results in a ~ 6 aF decrease in C_{cant} . Because this contribution is

approximately one order of magnitude smaller than the measurement resolution of our system, C_{cant} was once again determined to be negligible and omitted from the model. Using the same value of $\alpha = 0.5 \text{ fF}/\Delta\text{dBm}$ previously calculated, the model provides a reliable first-order approximation for the SiO_2 steps across the thicknesses measured and agrees with the experimental data to within 10%, as shown in figure 5. Improvements can be further made to the fit by decreasing the value of C_{tip} . This is attributed to the likely case where the NW end facet is not flush with the sample during scanning, resulting in a reduced contact area and an increased effective dielectric thickness.

Imaging 2D MoS_2 sheets

To further assess the performance of the W-ALD NW probe, we compared its topographical and microwave resolution against that of a commonly used commercial Pt NSMM probe (4.5 kHz Rocky Mountain Nanotechnology 12Pt400A) (see footnote 3). We chose a MoS_2 sample for imaging measurements due to its potential applications in CMOS-like logic devices and as a transparent semiconductor in photovoltaic and other optoelectronic structures [41, 42]. MoS_2 is a member of the layered transition-metal dichalcogenide materials with crystals composed of vertically stacked layers held together by van der Waals forces. Unlike pure graphene,

single layer MoS_2 is a direct bandgap semiconductor (1.8 eV) and therefore a candidate for replacing Si in transistor designs without the need for increased fabrication complexity. One–four-layer-thick MoS_2 was extracted from a bulk sample by use of the Scotch Tape method and then deposited on 260 nm of SiO_2 grown on a p-type Si wafer [43]. Both probes were scanned over the same MoS_2 patch at $\sim 2.3 \text{ GHz}$ and with a scan area of $10 \mu\text{m} \times 10 \mu\text{m}$. Figure 6(a) shows the topographical results for the W-ALD NW probe. The different layers (each 6.5 \AA thick) are clearly visible with sheet edges well defined. By comparison, in figure 6(d), the topographical results for the Pt probe are shown, and the sheet edges are no longer sharp, while the single-layer region is poorly resolved. In figures 6(b) and (c), the amplitude and phase components of the microwave reflection S_{11} are shown for the W-ALD NW probe. Both single and multilayer MoS_2 sheets are clearly visible in the S_{11} amplitude and phase images. The dashed white oval highlights the transition from one- to two-layer MoS_2 , which can be seen in all three W-ALD NW image modes. The physics underlying the microwave contrast in the MoS_2 sample will be explored in depth in an upcoming publication. Figures 6(e) and (f) show the amplitude and phase results respectively for the Pt tip. These images are set to the same intensity scale as the W-ALD NW results, allowing for a direct comparison between the image contrasts. Although it maintains good multilayer edge resolution, phase contrast is reduced with a loss of sensitivity to

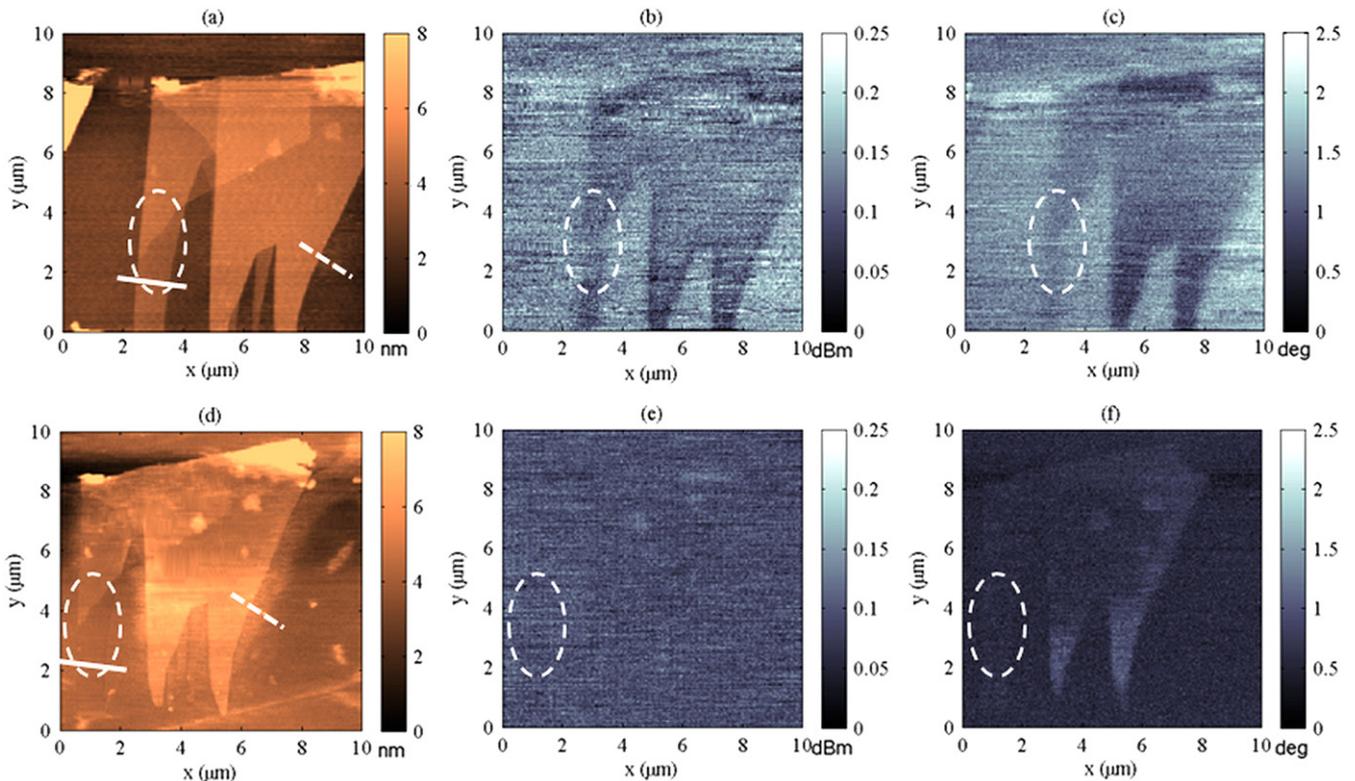


Figure 6. (a) Topography of the MoS_2 sample with the W-ALD NW probe showing high edge definition. Solid and dashed white lines indicate, respectively, location of single-layer and multilayer line cuts for figure 7. (b) and (c) $|S_{11}|$ amplitude and phase, respectively, with the W-ALD NW probe with sensitivity to the one- to two-layer MoS_2 transition (dashed white oval). (d) Topography result from the commercial Pt probe showing reduced edge definition and lack of sensitivity to the single-layer region. (e) and (f) $|S_{11}|$ amplitude and phase, respectively, from the commercial Pt probe with only minimal contrast present in the phase image. All images were taken with the same normal force of a few nanonewtons.

the single-layer MoS₂ sheet. Furthermore, amplitude sensitivity is almost completely eliminated in the case of the Pt tip, with measurement noise dominating the scan.

The MoS₂ sample was further examined in an effort to determine the cause of varying resolution between the two probes. For the above data, the MoS₂ was imaged first by the commercial Pt probe (with the result shown in figure 6(d)) before being scanned by the W-ALD NW probe (figure 6(a)). Figure 7(a) shows a follow-up topography scan with the Pt probe showcasing improved edge definition and single-layer contrast. Line cuts of 2.5 μm across the single-layer region (solid white line) and across the multilayer-SiO₂ region (dashed white line) were taken from the same area for each of the three topography scans. The results are shown in figures 7(b) and (c), respectively. In figure 7(b), the top-line scan (black) represents the first Pt probe result which exhibits no obvious topographical sensitivity to the single layer region. The middle-line scan (red) represents the W-ALD NW probe, and here the single-layer region is clearly shown with a lateral resolution of ~ 100 nm for the 6.5 \AA step. The bottom-line scan (blue) represents the final Pt probe scan. Sensitivity to the single-layer region is now improved with a lateral resolution of ~ 400 nm. In figure 7(c), the line cuts produce a similar trend across the multilayer region. The W-ALD NW probe once again yields the cleanest edge definition followed by the second of the two Pt probe scans.

These line cuts indicate that the MoS₂ sheets may have a glue residue from the Scotch Tape on their surface. During scanning with the Pt probe, such a residue would result in a convolution between the topographical amplitude and phase as the cantilever rotates out of plane, subsequently reducing sensitivity to the MoS₂ edge boundaries. Because these scanning artifacts are reduced after the W-ALD NW probe imaging, we propose that the improvement in sensitivity observed with the commercial probe may be attributed to ‘cleaning’ of the sample surface through the removal of portions of the glue residue. It is important to note that the sample was first scanned with the Pt tip ten times with no apparent change in topography (figure 6(d)), while the first scan with the W-ALD NW probe yielded the resolution shown in figure 6(a). This indicates that the Pt probe performed negligible cleaning, if any, of the sample over the course of repeated scanning. Furthermore, the W-ALD NW probe still exhibits the highest contrast relative to either Pt probe result with a lateral resolution on the order of its tip radius, indicating that its flexible, high-aspect-ratio structure is possibly immune to any remaining glue residue, while providing a stable, uniform tip geometry that enables enhanced microwave contrast.

Conclusion

In conclusion, we report on the fabrication of a GaN NW NSMM probe with a W-ALD microwave pathway. Scanning over a microcapacitor calibration sample has shown the probe to exhibit capacitance resolution on the order of 0.03 fF. Capacitance circuit models were developed to fit trends in

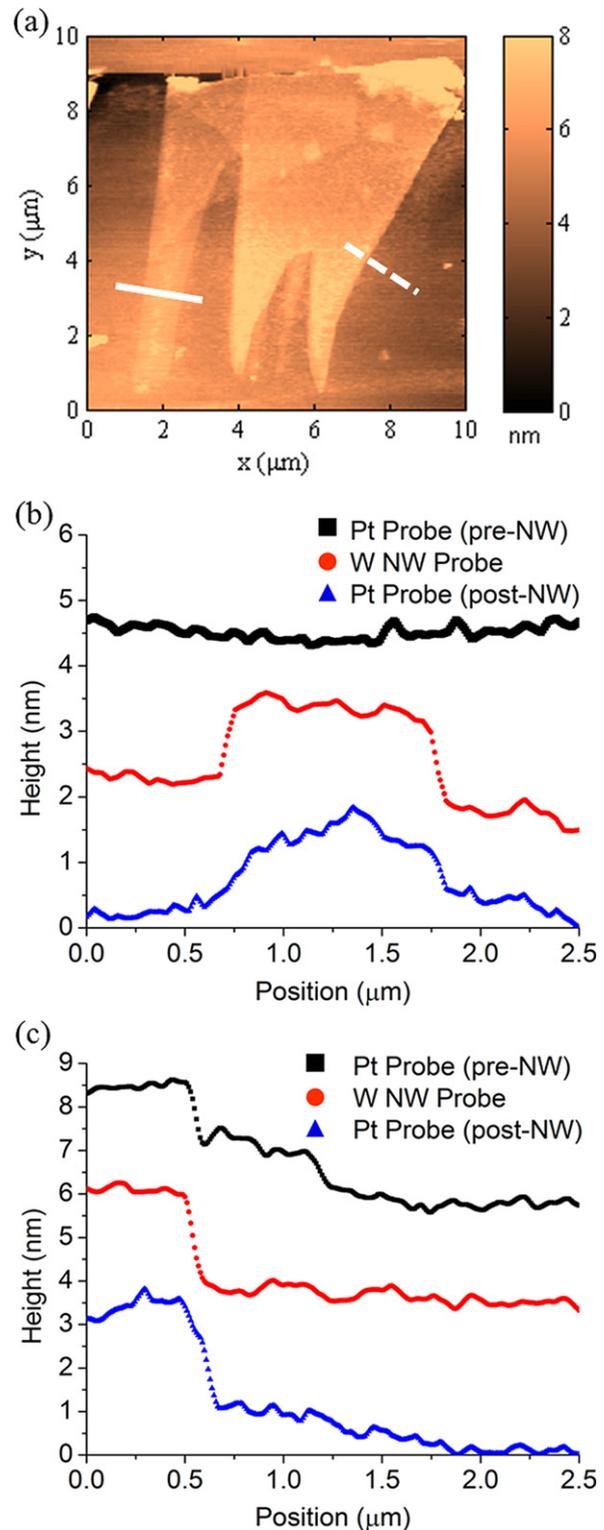


Figure 7. (a) Topography image from the commercial Pt probe after the W-ALD NW scan. Line cuts of 2.5 μm are taken across the single-layer region (solid white line) and the multilayer region (dashed white line). (b) and (c) Results from single-layer and multilayer regions, respectively, with the top black line representing the Pt probe topography data before the W-ALD NW probe scan, the red line representing the W-ALD NW probe, and the blue line representing the Pt probe after the W-ALD NW probe scan. The latter Pt probe line cuts reveal an improved sensitivity to the MoS₂ edge, possibly owing to a reduction in the presence of glue residue. Note: line scans have been offset along the y-axis for viewing clarity.

changing microcapacitor area and dielectric thickness as well as tip interaction with the SiO₂ staircase with agreement to within 10%. Improvements can be made through a better understanding of both fringing fields through the dielectric thin films as well as possible bending of the NW structure and end facet during contact mode scanning. The W-ALD NW probe was shown to yield improved topographical and microwave sensitivity relative to a commercial Pt probe on 2D MoS₂ films, potentially due to its insensitivity to sample contamination in the form of glue residue. Future work will seek to enable the wafer-scale fabrication of NW probes through the incorporation of selective GaN NW growth into a clean-room process flow. The success of ALD as a thin, uniform electrical pathway also opens the possibility of creating a conductor/insulator device structure, thus enabling the formation of a shielded microwave pathway along the entire length of the cantilever.

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