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Mapping electric fields in real nanodevices by *operando* electron holography

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1 Abstract

2 Nano-electronic devices play an essential role in many
3 domains, and their development and improvement attract
4 considerable attention in fundamental and applied research.
5 Access to the local physical processes involved in these
6 nanosystems during their operation is therefore crucial. We
7 show how electric fields in real nanodevices can be studied
8 under working conditions using *operando* electron
9 holography. A specific sample preparation method was first
10 developed to bias electron-transparent nanodevices
11 extracted from production lines whilst ensuring their
12 electrical connectivity and functionality without employing
13 dedicated probe-based holders. Metal-insulator-metal
14 (MIM) nanocapacitors were prepared using this approach
15 based on focused ion beam (FIB) circuit modification.
16 *Operando* electron holography allowed the electric potential
17 to be quantitatively mapped in the active areas, and between
18 devices, whilst biasing the devices *in situ*. Experimental
19 results were compared with finite element method (FEM)
20 modelling simulations to determine local electrical
21 parameters. We demonstrate that electrical properties such
22 as capacitance and surface charge density can be measured
23 at the nanoscale and have been preserved by our sample
24 preparation methodology when compared to macroscopic
25 measurements. This work paves the way for mapping the
26 local electrical properties of more complex biased devices.
27

28 Main text

29 The development of nanometer-scaled electronic
30 devices with reduced dimensions, involving new materials
31 or new architectures such as Magnetic Random Access
32 Memories (MRAM)¹⁻⁴, memristors⁵⁻⁷ or Phase Change
33 Memories (PCM)⁸⁻¹², requires a deeper understanding of
34 their operating properties. While electrical and physical
35 characterizations are widely used to monitor and evaluate
36 both the device performance and the quality of the layer
37 stack, there is a lack of knowledge on how the
38 electromagnetic fields are precisely mediated along devices
39 at the nanoscale level. Correlating local electric fields

40 mapped across a chosen device with its structural properties
41 and chemical composition would greatly help research
42 laboratories and the microelectronics industry in the
43 development of new nano-electronic devices. Furthermore,
44 these local studies could also provide methods to determine
45 the root causes of electrical failure¹³ to improve the devices
46 in terms of reliability, speed and power consumption.

47 Transmission Electron Microscopy (TEM) is a powerful
48 technique for studying the properties of individual
49 nanosystems with very high spatial resolution. Among TEM
50 methods, off-axis Electron Holography (EH) is an
51 interferometric technique that allows quantitative mapping
52 of electrical potentials inside and around the specimen, as
53 well as the measurement of charge distributions¹⁴⁻¹⁸ with a
54 precision down to the elementary charge.¹⁹ EH
55 experiments, when combined with *in situ* biasing, pave the
56 way for nanoscale studies of fundamental electrical
57 phenomena of nanodevices in working conditions.
58 However, despite these exciting prospects, *operando* EH has
59 been used relatively little to investigate the local properties
60 of nanosystems. A few model systems (nano tips²⁰⁻²⁴,
61 quantum wells²⁵, nanowires^{26,27}, p-n junctions²⁸⁻³¹, oxide
62 layers³²) have been studied, but key issues such as specimen
63 preparation, surface damage layers, stray field and electron
64 radiation were identified.^{30,33,34} The investigation of real
65 devices are rarer still^{35,36}, even with dedicated probe-based
66 holders.³⁷ The main problem is that stray fields around the
67 nanoprobe may interfere with the reference wave required
68 in holography²¹ and the potential applied to the active area.³⁸
69 In addition, the contact resistance between nanoprobe and
70 sample is unknown, poorly controllable and causes
71 mechanical instabilities.

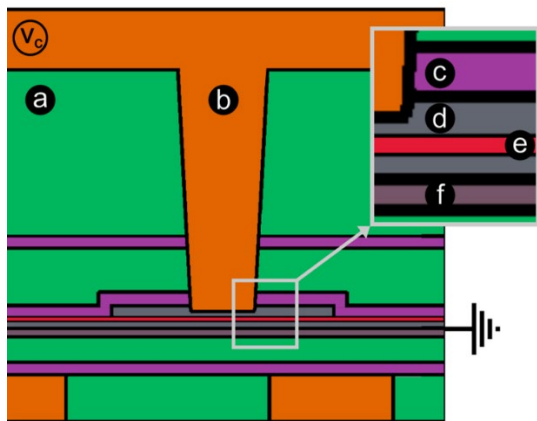
72 *In situ* biasing TEM experiments thus necessitate a
73 specific and complex sample preparation that minimizes
74 preparation artifacts whilst ensuring the electrical
75 functionality of the nanodevice itself. A dedicated setup for
76 applying bias from the macroscopic to nanometre scale is
77 also required to avoid electrostatic discharge that can
78 destroy the fragile specimen. Moreover, the signal from
79 local charge distributions is extremely weak and measuring
80 such a signal on a working device prepared by sample

81 preparation methods such focused-ion beam (FIB) is very
82 challenging. Finally, the analysis of the resulting phase
83 images remains complex due to the integration of the signal
84 along the electron path as well as additional effects (beam-
85 induced charge, stray fields and sample preparation
86 artifacts).

87 Here we present the methodology that we have been
88 developing for mapping the electrical properties of nano-
89 electronic devices extracted directly from production lines
90 without using a probe-based approach. We investigated the
91 potential distribution at the nanoscale of metal-insulator-
92 metal (MIM) nanocapacitors using advanced FIB sample
93 preparation and state-of-the-art EH. Capacitors are, in fact,
94 one of the most important components in all electronic
95 devices. They are widely used in analog and RF circuit
96 applications for energy storage and delivery in memory
97 devices such as flash memories and random access memory
98 (RAM) chips, and processors whose performance is
99 dependent on their miniaturization and operation.
100 Experimental phase measurements will be compared to
101 additional numerical simulations using finite element
102 modelling (FEM) including factors such as specimen
103 geometry and stray fields.

104 Our results demonstrate that electrical properties such
105 as electric field, capacitance and surface charge density can
106 be preserved and investigated at the nanoscale.

107 Experiments were carried out on an array of parallel
108 MIM nanocapacitors extracted from a matrix structure
109 integrated in a STMicroelectronics 28 nm process test chip.
110 The nanocapacitors used in backend upper layers for high
111 frequency circuit applications are made up of a thin 11.5 nm
112 tantalum pentoxide (Ta_2O_5) high- k insulator sandwiched
113 between two TiN electrodes connected to a Cu pillar
114 (above) and a Al layer (below) (Fig. 1). The upper electrode
115 of TiN has a width of 950 nm (in the plane of the image)
116 and 1.7 μm in depth (out-of-plane).



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FIG. 1. Architecture of MIM nanocapacitor: (a) SiO_2 , (b) Cu interconnects; (c) Si_3N_4 ; (d) TiN electrodes; (e) active region of Ta_2O_5 dielectric; (f) Al electrode.

Prior to experiments, individual MIM of the array were measured to have a capacitance of $35 \pm 2 \text{ fF}$ at 1MHz using a nanoprobe (nProber III from ThermoFisher). Assuming the formula for a parallel plate capacitor with a surface area corresponding to the top TiN electrode gives a relative electric permittivity of the thin-layer Ta_2O_5 to be 28 ± 1 , which is in the range of values found in the literature.⁴⁰

The specimen-device for holography experiments was prepared using a dual-beam FIB (Helios NanoLab 1200AT DualBeam from ThermoFisher) with a gallium liquid-metal ion source and gas injection system (GIS). A slab of material containing the MIM array was lifted from a fully processed wafer and prepared for electron transparency and *in situ* biasing (Fig. 2).

To ensure proper charge evacuation during specimen navigation and preparation, a thin conductive layer of gold-palladium alloy was deposited on the sample surface prior to extraction. The specimen surface was then connected to the Si substrate by milling a hole in the pad, allowing the charge from the electron beam to drain to the ground. Top and bottom electrodes were also temporarily connected by Pt deposition to prevent electrostatic discharges harming the nanocapacitors during the deprocessing. The specimen-device was then deprocessed top-down with FIB until the copper lines connecting the nanocapacitors were exposed.

A second layer of Pt was deposited to equalize the potential of the two electrodes reconstructing the top circuit, and to protect the structure during further milling. The lamella was then cut and extracted from the wafer and placed on a dedicated chip (Hummingbird Scientific) over a trench surrounded by the prepatterned Au tracks (Fig. 2(a)) before being welded by W deposition using GIS at both ends to ensure the electrical connection (Fig. 2(b)).

Final thinning of the lamella was performed with a low energy ion beam (8 kV) to minimize the impact of surface damage on the thin device compared to the corresponding embedded device. A uniform thickness of about 100 nm was then obtained for two of the capacitors in the array (Figures 2(c) and 2(d)). The final step in the preparation process was to cut trenches so that the top and the bottom electrodes could be biased independently.

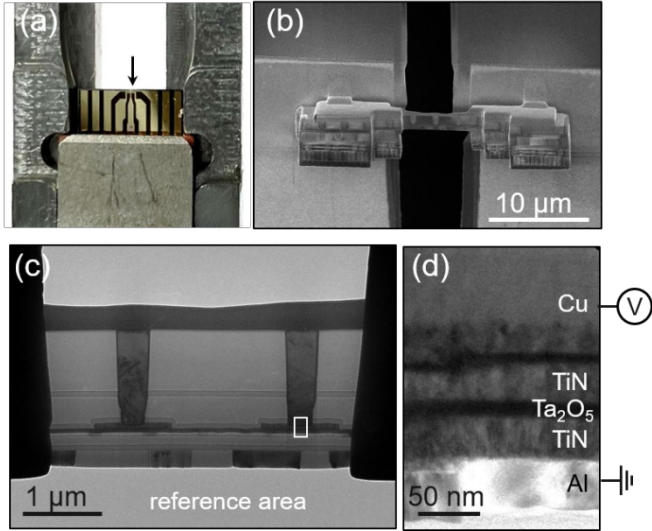


FIG. 2. MIM nanocapacitor specimen-device prepared for operando EH study: (a) chip inserted in sample holder (arrow indicates position of MIM nanocapacitors); (b) SEM image of sample device connected to gold electrodes on chip; (c) TEM image of final specimen-device active area; (d) enlargement of MIM nanocapacitor showing stack.

Such specimen-devices are extremely delicate, with capacitances estimated to be only around 2 fF (the cross-sectional area of the dielectric in the thin lamella is 17 times smaller of the original device). An additional setup consisting of several levels of electrical connections with adapted equipment and software, was therefore developed to protect the thinned nanocapacitors from electrical discharge. Indeed, the electrical resistance of completed specimen-devices was measured prior to TEM experiments to confirm that the nanocapacitors had not been electrically shorted. The chip supporting the connected devices was inserted into a Hummingbird holder designed specifically for biasing experiments (1600 series, Hummingbird scientific) and connected to this additional setup. The bottom electrode was grounded whilst positive, negative or zero biases were applied *in situ* to the top electrode for the electron holography experiments.

The principle of off-axis electron holography is to use an electrostatically charged wire to interfere a beam of highly coherent electrons that has interacted with the specimen, called “object wave”, with a reference wave that has not undergone interaction with any field.^{41–43} The resulting interference pattern (*i.e.* the hologram) contains all of information on the phase shift of the electron wave experienced when interacting with the local electromagnetic fields.^{14,43}

The electrical potential V distribution induces a phase shift ϕ recorded in the x - y image plane:^{44–46}

$$\phi(x, y) = C_E \int V(x, y, z) dz \quad (1)$$

where C_E is an energy-related constant that depends on the accelerating voltage ($6.526 \cdot 10^6 \text{ V}^{-1} \cdot \text{m}^{-1}$ at 300kV) and ξ the direction of the electron path. V is the sum of the mean internal potential V_{MIP} , which is related to the atomic potential and static charges, and V_{Bias} , which is caused by the applied bias. As a result, ϕ can be divided into two sub-contributions, ϕ_{MIP} , and ϕ_{Bias} . Since ϕ_{MIP} does not depend on the bias, it can be measured by grounding the two electrodes and subtracted from the total measured phase shift. It is then possible to isolate ϕ_{Bias} on applying bias.

Operando EH experiments were carried out on a Hitachi HF3300-C (I2TEM) microscope specifically designed for *in situ* electron interferometry studies. It is equipped with a cold field-emission gun (CFEG) for optimal brightness and an imaging aberration corrector (BCOR from CEOS), which allows on- and off-axis aberrations to be corrected over wide fields of view. Observations were performed with the microscope operating at 300 kV in Lorentz mode with a spatial resolution of 0.5 nm⁴⁷, elliptical illumination and 2 post-specimen biprisms to avoid artefacts linked to Fresnel fringes and to allow adjustment of the interference area with respect to the fringe spacing.⁴⁸ The reference wave was placed in the field-free region of the vacuum beneath the Al electrode (cf Fig. 2(c)).

A high-speed 4k by 4k camera (OneView, Gatan Inc.) operating at full-frame rate of 25 images per second was used with a temporal integration window of 250 ms, which corresponds to the sum of the frames displayed in the previous 250 ms. Holograms with an interfringe of 1.5 nm (5 pixels) were acquired using dynamic automation⁴⁹ and π -shift method⁵⁰ over a total exposure time of 120 s each corresponding to the sum of 480 images.

The phase was monitored in real-time during experiments using HoloLive 1.0 (HREM Research Inc.), a plug-in for the image processing package Digital Micrograph (GMS 3.3, Gatan Inc.). In-house scripts and dedicated code implemented within Digital Micrograph were used to analyze the holograms and extract amplitude and phase images during post-processing. A 5th order Butterworth Fourier-space filter centered on the sideband giving a spatial resolution of 3 nm was selected for the amplitude and phase images. To remove the projector and camera distortion-induced phase modulations, a dedicated reference hologram recorded in a vacuum area was used. For each applied bias, a final field of view of 3.5 μm x 1 μm was reconstructed by aligning and combining 3 images.

Figures 3(a) and 3(b) show the experimental amplitude and phase images, respectively, acquired with 0.6 V of bias applied by the power supply. The phase image only contains the electrostatic contribution ϕ_{Bias} from biasing. The mean phase and linear phase ramp measured within the bottom

245 substrate were subtracted from the whole phase image. 275
 246 Isophase contours with a spacing of 0.25 rad, calculated 276
 247 from the phase image, have been superimposed on the
 248 amplitude image.

249 On biasing, the phase presents a sharp variation across
 250 the Ta₂O₅ layer due to the strong local electrical field in the
 251 dielectric, but is relatively uniform within the electrodes, as
 252 expected. There is also an in-plane stray field between the
 253 nanocapacitors due to the finite lateral dimensions of the
 254 upper electrodes (zoom in Fig. 3(b)). This weak stray field
 255 is clearly visible because of the very long exposure times of
 256 our automated holography experiments.⁴⁹

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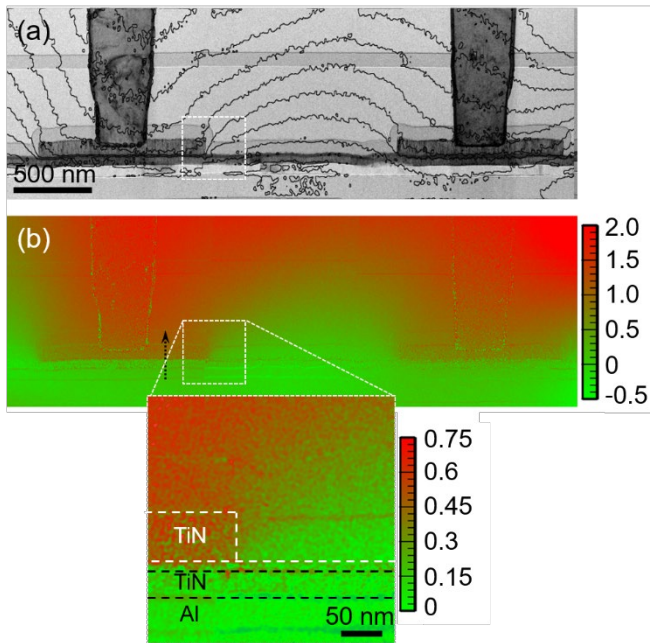


FIG. 3. Operando electron holography: (a) Amplitude image of both MIM nanocapacitors in parallel; (b) Experimental phase image of the induced electrostatic potential for 0.6 V bias applied by the power supply.

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 259 To analyze quantitatively the potential distribution
 260 across the thin oxide layer, profiles were plotted from the
 261 phase images along the black arrow (Fig. 3(b)) for different
 262 biases and averaged over a width of 50 nm parallel to the
 263 interfaces (Fig. 4). The overall phase shift across the
 264 dielectric increases linearly with applied bias, as expected
 265 from Equation (1). On the other hand, the phase is not
 266 completely flat in the electrodes whereas we would expect
 267 them to be at a uniform potential.

268 In fact, the phase curvature is caused by the stray field
 269 above and below the thin lamella since the phase is
 270 integrated along the whole path of the electron beam (cf Eq.
 271 (1)), inside and outside the specimen. Unfortunately, the
 272 stray field cannot be described analytically for complex
 273 geometries so numerical modeling must be performed to
 274 separate the effects of the internal and external fields.

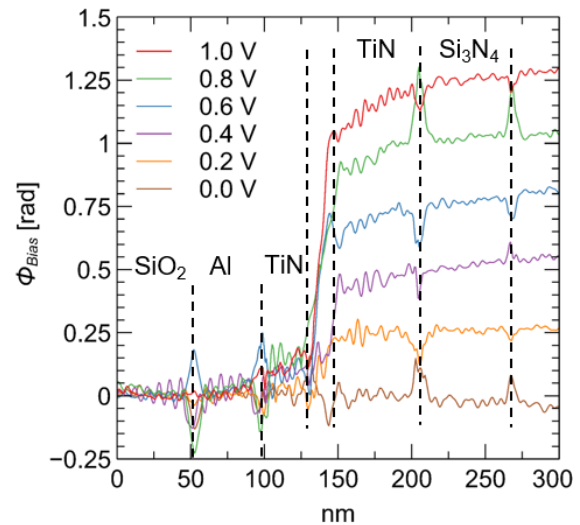


FIG. 4. Phase profiles extracted along the black dotted arrow in Fig. 3(b) for different biases. Ta₂O₅ active layer is between the two TiN electrodes.

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279 Three-dimensional (3D) finite element method (FEM)
 280 modeling was performed with the COMSOL software
 281 package (COMSOL Multiphysics). The model geometry
 282 and dimensions were based on the TEM images of the
 283 observed devices, to approach the reality of the experiments
 284 as closely as possible. Indeed, this is one of the advantages
 285 of TEM experiments that accurate digital twins can be built
 286 relatively easily. The main uncertainty is the exact lamella
 287 thickness, which we measured by electron energy-loss
 288 spectroscopy (EELS) to be 100 ± 10 nm based on theoretical
 289 mean-free paths for elastic scattering.

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291 To limit the size of the model, the array of parallel
 292 devices was nevertheless limited to four and surrounded by
 293 vacuum. From the external boundaries to the active region
 294 of the nanocapacitor, the mesh size dynamically adapts to
 295 the local dimensions, with a minimum mesh size of about 2
 296 nm. The electrical potential is determined in three
 297 dimensions and integrated along the electron path using Eq.
 298 (1) to give the 2D phase map. An internal reference is
 299 applied in exactly the same way as for the experimental
 300 phase images.

301 The simulated phase corresponding to the experimental
 302 case (Fig. 5(a)) is shown in Fig. 5(b) for a lamella
 303 thickness of 100 nm. An excellent fit is obtained for the overall
 304 phase distribution and for the phase profiles across the active
 305 area (Fig. 5(c)). The root mean squared difference between
 306 the simulated and experimental phase profiles is only 54 mrad.
 307 To convert this into a potential, the phase change across the

308 dielectric was determined from the simulated curves. The
 309 result of 0.88 rad.V^{-1} means that the phase error is
 310 equivalent to about 60 mV of applied bias. Simulations also
 311 confirm that the out-of-plane stray field is indeed the cause
 312 of the non-uniform phase in the region corresponding to
 313 the electrodes. Even the in-plane stray field is well
 314 reproduced (see Fig. 5(d)) and the simulations of the out-
 315 of-plane stray field confirm that it is indeed the cause of the
 316 non-uniform phase in the region corresponding to the
 317 electrodes.

318 However, in order to obtain this agreement, the local
 319 applied bias in the model was 0.40V. Indeed, the dominant
 320 source of uncertainty being the lamella thickness, we can
 321 estimate the local experimental bias to be within 40 mV of
 322 this value. The local bias was therefore significantly lower
 323 than the 0.6V indicated by the external power supply. The
 324 discrepancy between the bias applied locally and
 325 macroscopically is most probably caused by leakage currents
 326 and impedances in the connections.

total contribution (red); (d) horizontal phase profiles (black
 arrow 2).

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The agreement between simulations and experiment means that we can be confident in the local electrical parameters extracted from the model, such as the electrostatic potential inside the dielectric layer, the capacitance, and surface charge density. The electric field inside the dielectric layer was found to be $0.35 \pm 0.04 \text{ MV cm}^{-1}$ for the specimen-device with a corresponding capacitance of 2.1 fF, very close to the estimated value from macroscopic measurement, even for the DC bias of the EH experiments. The surface charge density is then 0.12 elementary charge per nm^2 and per Volt. The energy associated with the induced electrical field between both electrode plates is equal to $3.9 \cdot 10^{-16} \text{ J.V}^{-1}$, while the energy of the stray fields between two neighboring devices corresponds to $1.24 \cdot 10^{-18} \text{ J.V}^{-1}$, or 0.3% of the total energy.

These results show that devices extracted from production lines can be prepared for TEM studies whilst preserving their electrical connectivity and functionality. *Operando* electron holography can then be used to quantitatively map the electric potential distribution as a function of bias. That the measured electric field can be modeled with almost no fitting parameters suggests that the device is indeed functioning normally within the microscope.

There is no reason that the same methodology cannot be applied to more complex electronic devices such as MOS transistors or spintronic devices by adapting the sample preparation workflow to achieve a successful circuit modification and enable electrical stimulation of the device. Furthermore, measurements can be correlated to complementary TEM techniques for a full understanding of structural, chemical and electrical properties of individual devices at the nanometer scale. We hope that the ability to study local electric fields quantitatively in newly proposed devices and devices already in production will help efforts to explore fundamental physical processes as well as to develop and improve current devices.

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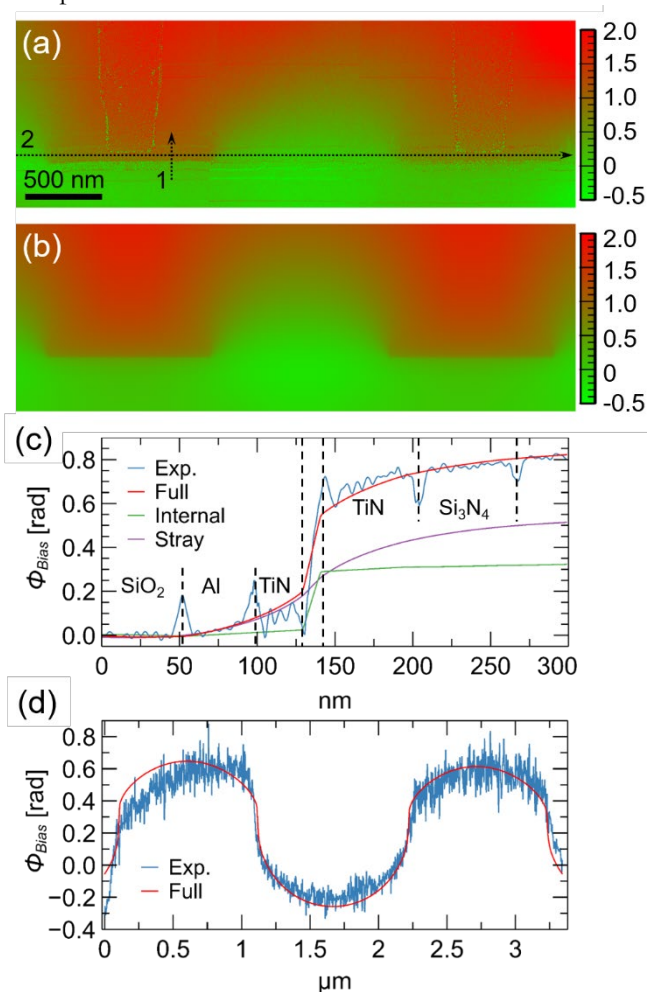


FIG. 5. Quantitative analysis: (a) experimental phase; (b) FEM simulated phase; (c) experimental phase profiles across active layer (black arrow 1), corresponding simulated profiles for the internal field (green), stray field (purple) and

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