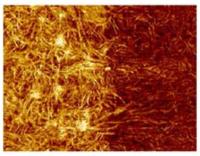
Beating the limitations of surface-bound scanning probe microscopy

Exploring nanoscale 3D physical properties of advanced materials and devices

Oleg Kolosov, Lancaster University, UK

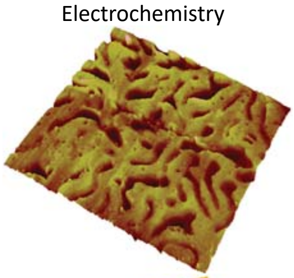
AFM and SPM outstanding images of ... surfaces

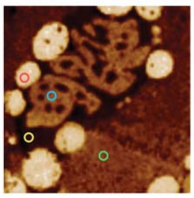
Nanomechanics



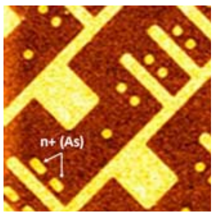
Surface potential (KPFM)

Electrochemistry





Polymers

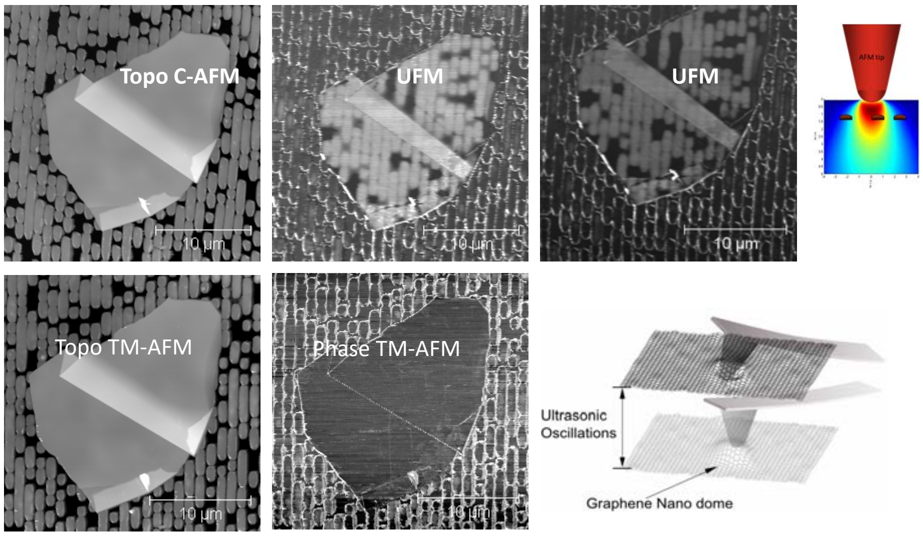


Semiconductors

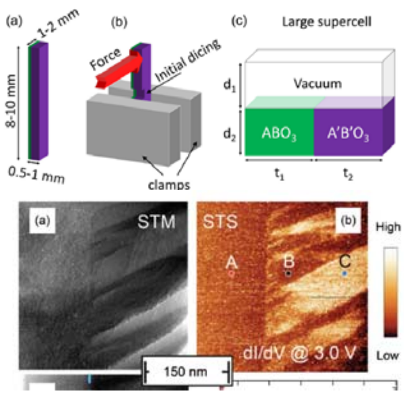
Metals

https://www.bruker.com/en/products-and-solutions/microscopes/materials-afm/afm-modes.html

Subsurface measurements via ultrasonic force microscope (UFM) - graphite nanoflakes

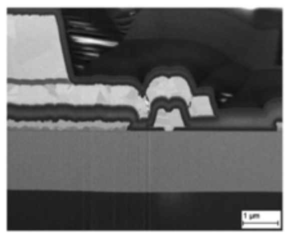


Looking into the 3D structures – mechanical cleavage and FIB



- Wang, A. and T. Chien (2018) Physics Letters A 382(11): 739-748.

The cleavage is a real art and suitable for epitaxies only

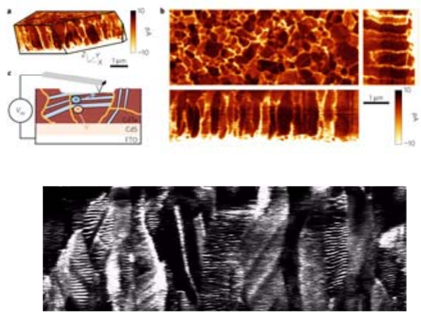


SEM cross-section of a GaN HEMT device.

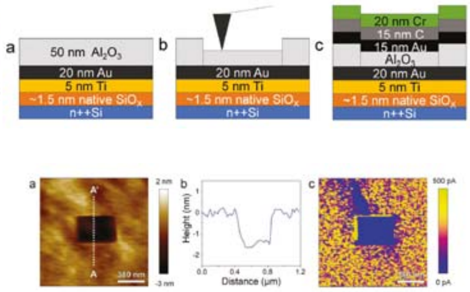
Microelectronics Reliability 54, 1785-1789, (2014)

FIB area is small, not easily used in SPM, and Ga implantation change the properties.

“Slicing” the sample directly in AFM - nanotomography and “Scalpel AFM”



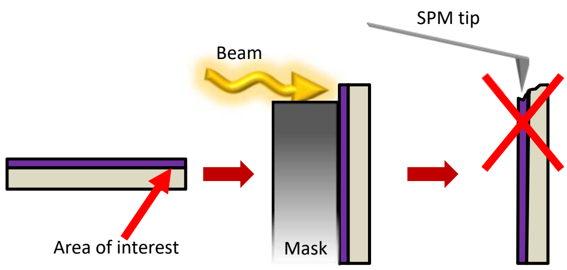
Luria, J. et al, (2016) Nature Energy **1**: 16150.



Chen, S. at al, Advanced Functional Materials 28(52): 1802266.

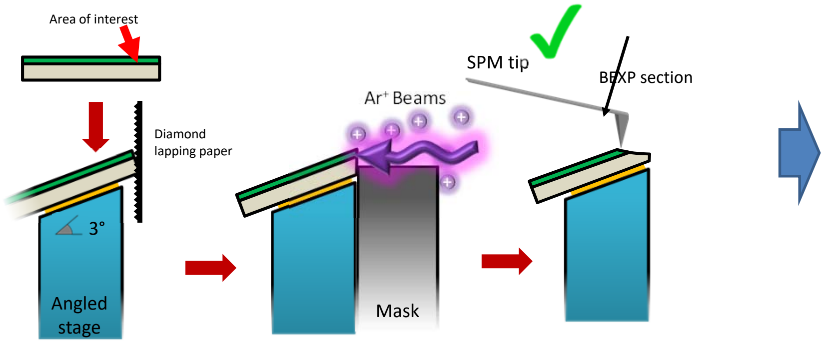
Great tool, but area is rather small, and section of harder materials like Si, iii-v is very difficult.

Can we use Ar Ion Cross-sectioning?



Low damage to the surface, but near surface layers are damaged during the preparation.

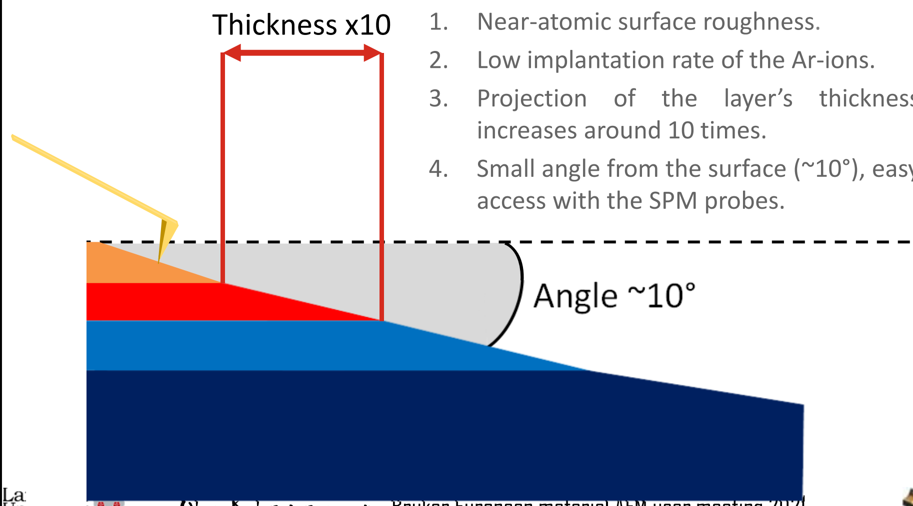
New - SPM friendly nano-cross-sectioning Beam Exit Cross-sectional Polishing (BEXP)



Method and apparatus for ion beam polishing. USA. **9,082,587**.

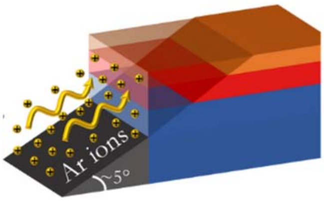
Kolosov, O. V. et al, Nanotechnology **22**(18): 185702.

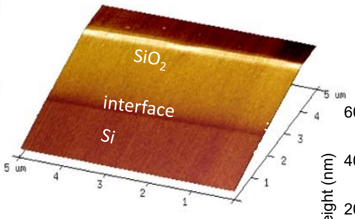
Benefits of using BEXP nano-cross-sectioning

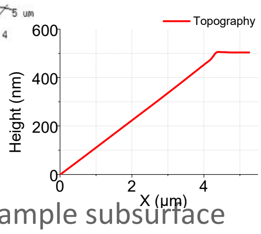


1. Near-atomic surface roughness.
2. Low implantation rate of the Ar-ions.
3. Projection of the layer's thickness increases around 10 times.
4. Small angle from the surface (~10°), easy access with the SPM probes.

Result – BEXP based cross-sectional SPM (xSPM)



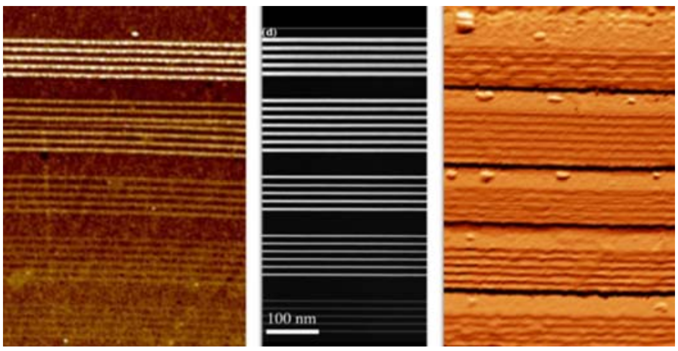




SPM-compatible near-atomically-flat section of sample subsurface layers. Thermal SiO2 on Si with nanomechanical ultrasonic force microscopy map.

US/EU patents- US 9082587, EP 2537017 B1.

3D cross-sectioning for SPM – rivaling TEM resolution

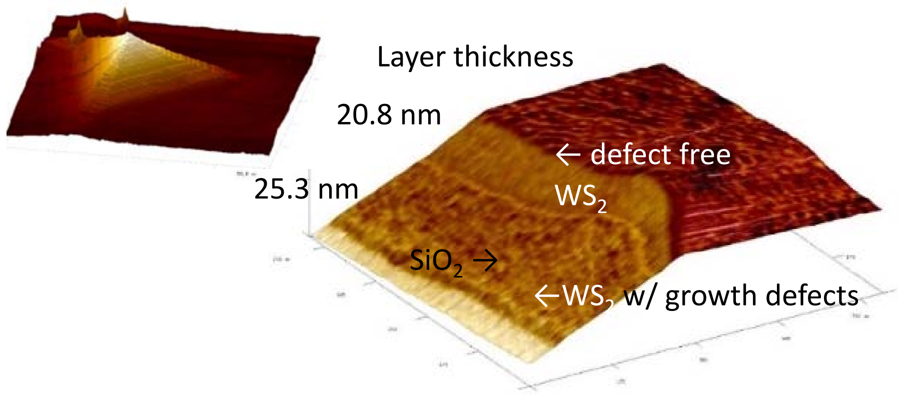


Robson, A. J. et al *Acs Applied Materials & Interfaces* **2013,** *5* (8), 3241-3245.

xSPM vs cross-sectional TEM

Multimode SPM in both Tapping Mode AFM and nanomechanical ultrasonic force microscopy (UFM). Oxidised $Al_xGa_{1-x}As$ layers protrude above GaAs layers by an amount which varies with Al content *x*, allowing identification during imaging.

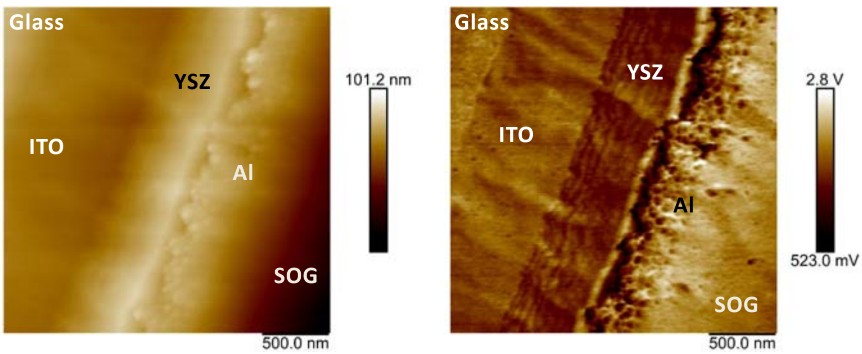
xSPM of CVD layered vdW materials



CVD grown transition metal dichalcogenide (TMD) WS2 on Si substrate.

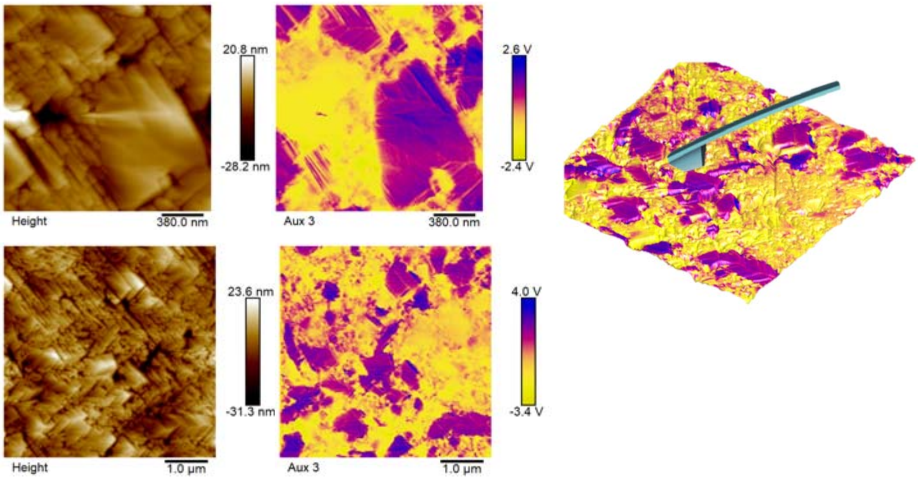
SPM nanomechanical contrast, low defects top layer and the high defect density bottom thickness measured with nm precision.

Gate oxides



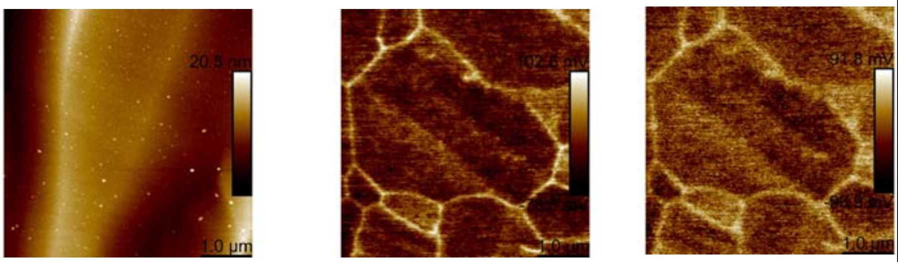
Topography UFM

Energy storage materials - Na ion batteries



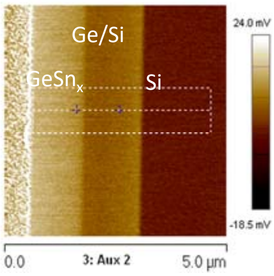
- *NTO*

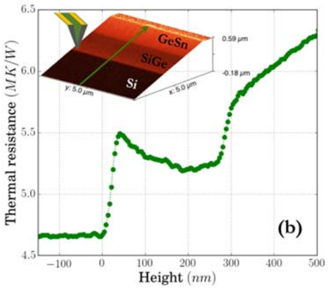
KPFM (surface potential) xSPM mode - thin film solar cells (CdSe/CdS) grain structure

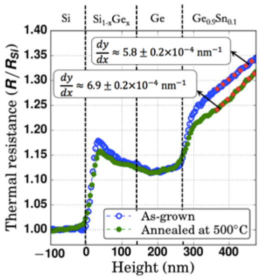


Topography KPFM amplitude KPFM phase

SThM (thermal conductivity) xSPM - $Si/GeSi_x/GeSn_y$ multilayer MBE structure



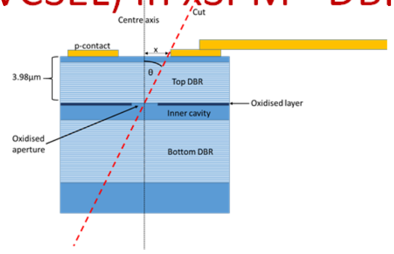


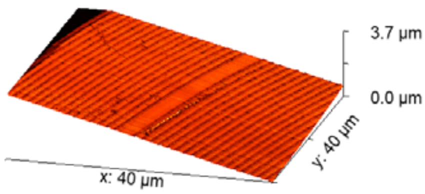


Thermal resistance SThM map and profiles.

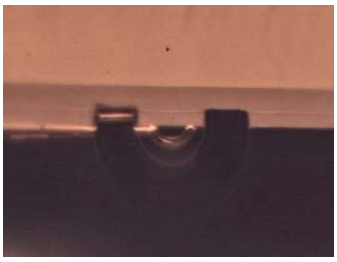
Inset: 3D topography overlaid with SThM response.

Devices – vertical cavity surface emitting laser (VCSEL) in xSPM - DBR and MQW structures.

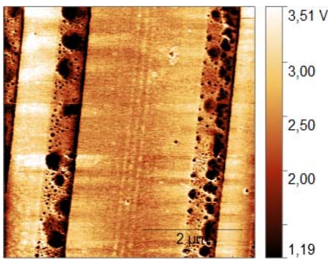




Topography



VCSEL section



UFM - nanomechanics

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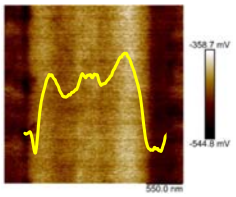
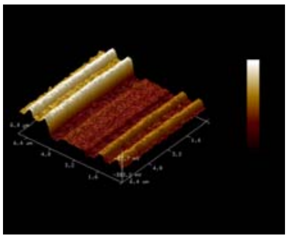
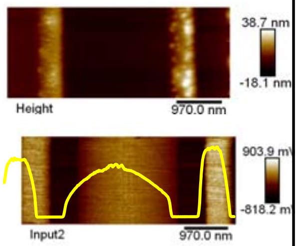
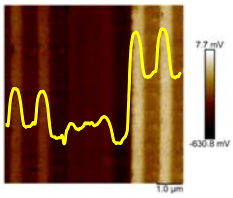
VCSEL active area zoom in – KPFM and SSRM (condutivity) xSPM modes

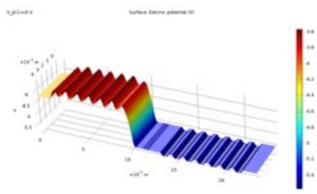








KPFM



KPFM

Note – due to tilted cut the vertical dimension of the structure is expanded from 5-10 times.

The lateral bar corresponds to approximately 100 nm in thickness.

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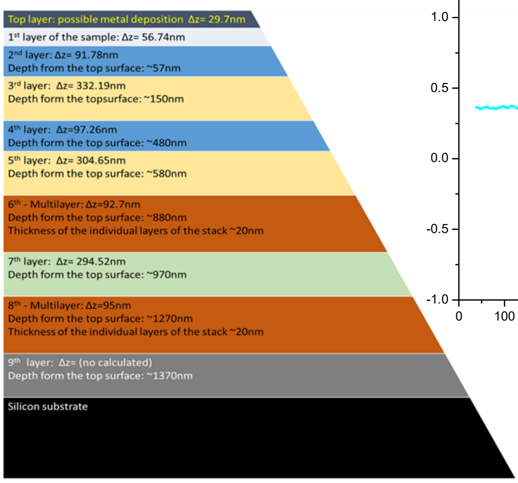
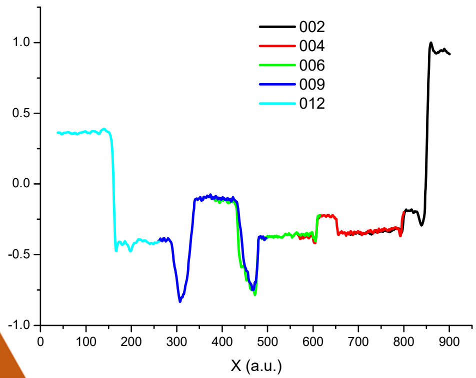
Multiple quantum wells iii-v on Si structures KPFM xSPM cross-section





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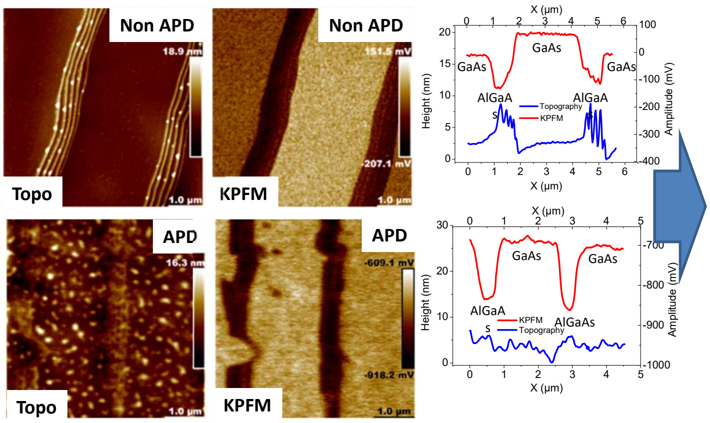
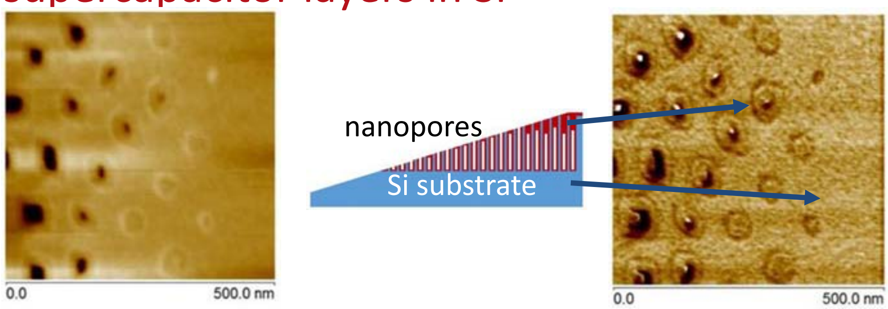
iii-v on Si structures multiple GaAs/AlGaAs/ InGaAs quantum wells - KPFM xSPM



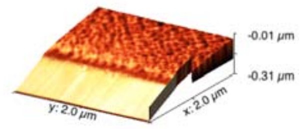
Direct indication - APD disrupts electrical charge distribution in MQW

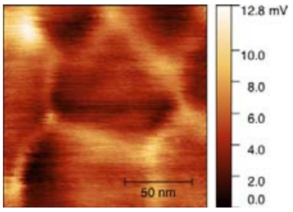
3D mapping of nanowires - nanoporous supercapacitor layers in Si

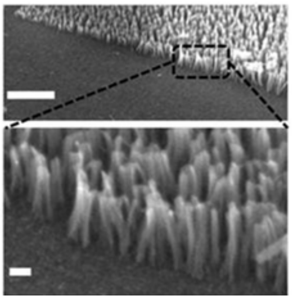


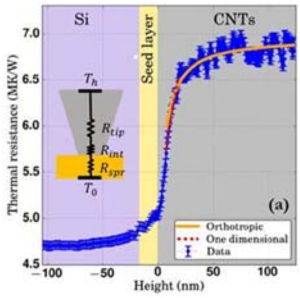
The structure of deep-etch vertical nanopores in the Si is observed from through the thickness of material. The structure of the nanopore-substrate interface and oxide pore clogging are clearly observed. (Sample courtesy M. Prunilla, VTT, Finland.)

Carbon nanotubes CNT thermal interface materials (SThM mode of xSPM)



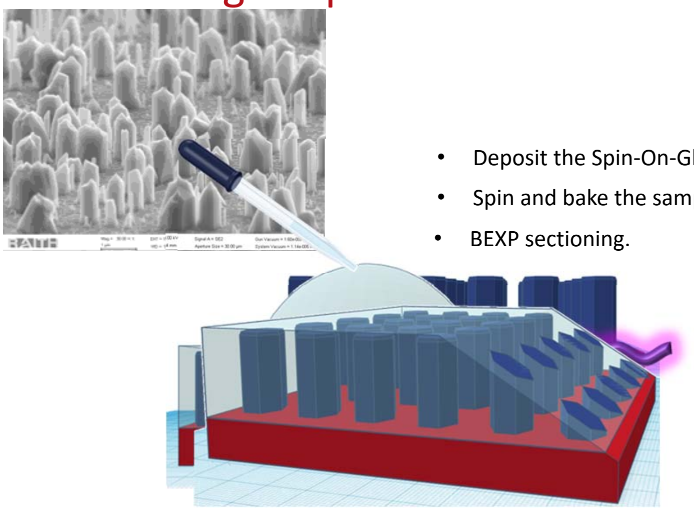






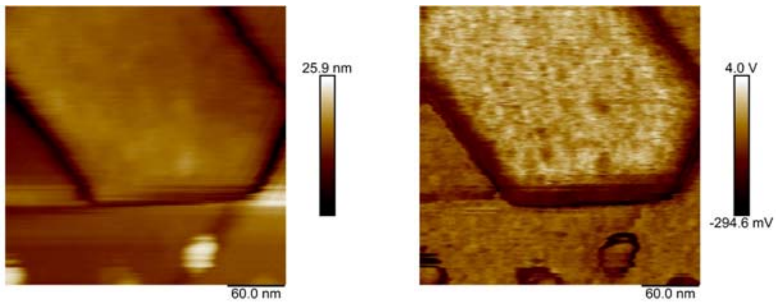
CNT "forest" is "trimmed" creating gradient of CNT height via BEXP™, enabling absolute measurements of intrinsic thermal conductance of the TIM (Sample courtesy O. Bezencenet, Thales, France).

xSPM of non-planar samples - GaN NWs - embedding in Spin-On-Glass



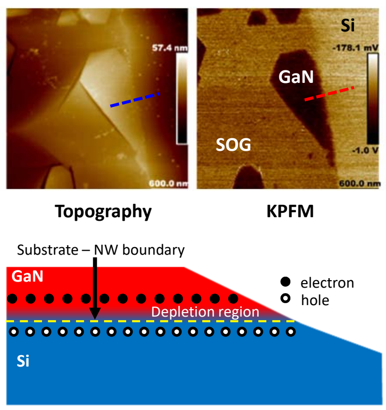
- Deposit the Spin-On-Glass (SOG).
- Spin and bake the sample.
- BEXP sectioning.

UFM nanomechanical mapping of GaN NWs

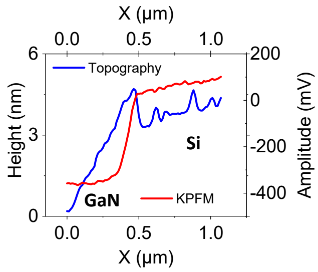


Topography UFM

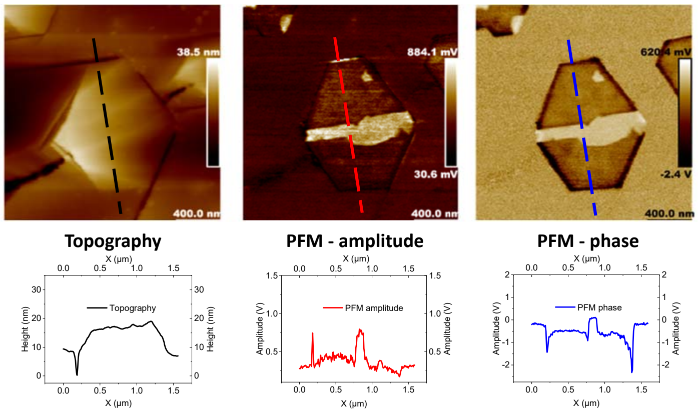
Electron affinity (KPFM) maps of GaN NWs



- CPD dependence with the thickness of the layer.



Piezoelectric response GaN NWs = NW polarity



The domains of opposite polarity exist in GaN NW's. BEXP-SPM can provide insight on what trigger particular polarity growth.

EPSRC Engineering and Physical Sciences Research Council
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