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

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

Looking into the 3D structures – mechanical cleavage and FIB


The cleavage is a real art and suitable for epitaxies only

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”


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.


3D cross-sectioning for SPM – rivaling TEM resolution


xSPM vs cross-sectional TEM

Multimode SPM in both Tapping Mode AFM and nanomechanical ultrasonic force microscopy (UFM). Oxidised AlₙGa₁₋ₙAs 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**

Gate oxides images showing different materials layers.
Energy storage materials - Na ion batteries

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.

VCSEL section

Topography

UFM - nanomechanics
VCSEL active area zoom in – KPFM and SSRM (conductivity) xSPM modes

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.

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

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.

Lancaster SPM group: M. Mucientes, Y. Chen, A. Niblett, L. Forcieri, J. Spiece, C. Evangelì, E. Castanon,
Collaborators (External) P. Pingue, F. Dinelli (CNR, SNS, Pisa), T. Wang (Sheffield), H. Liu (UCL), P. Smowton, S. Shutts (Cardiff).
Thank you!