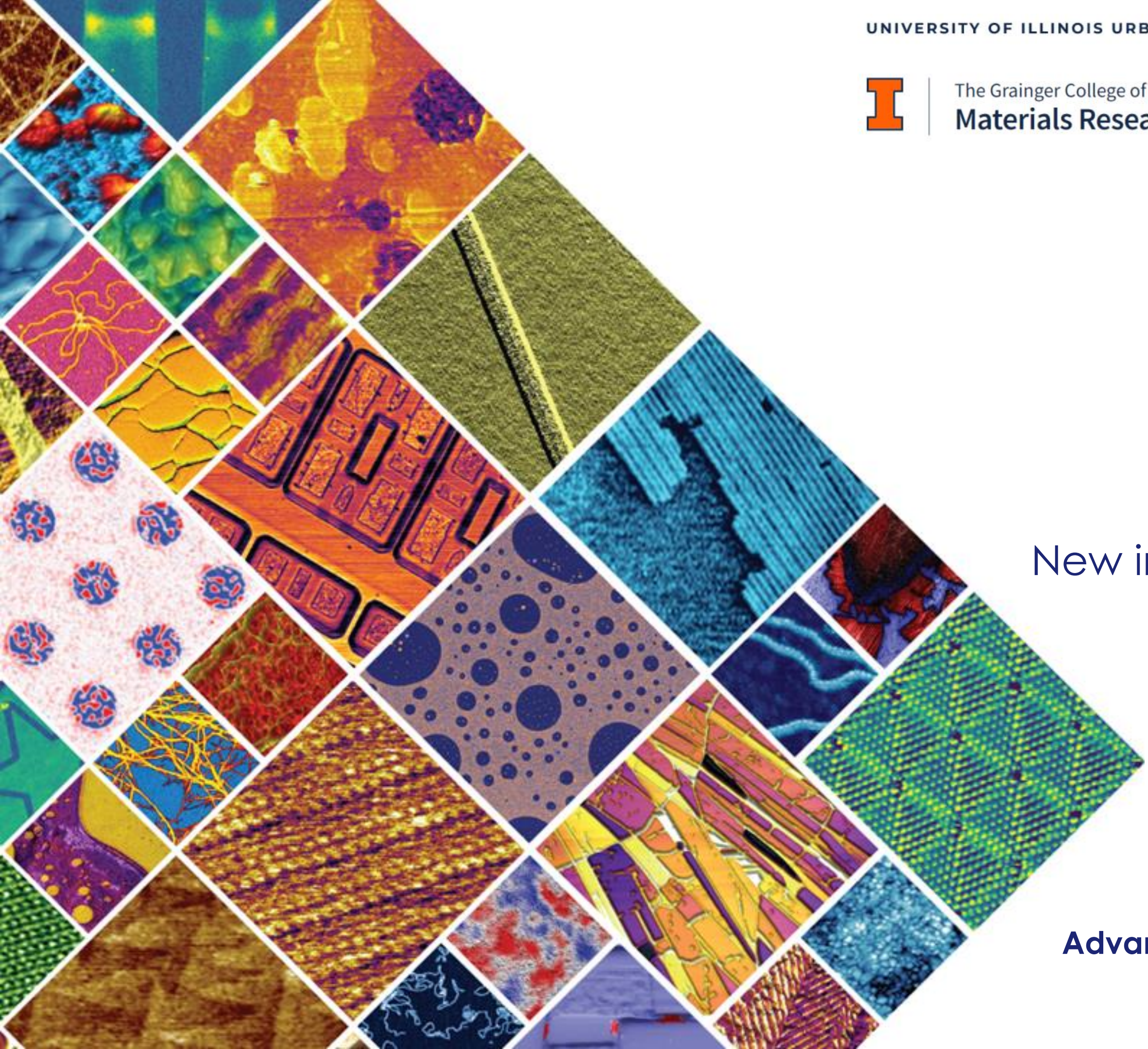


UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

The Grainger College of Engineering
Materials Research Laboratory

OXFORD INSTRUMENTS

# Can AFMs be more accurate and precise?

## New interferometric detection system for tip displacement sensing

**Ted Limpoco, PhD**
B. Ohler, J. Li, R. Proksch, A. Labuda, J. Lefever
Oxford Instruments Asylum Research
Concord, MA ∘ June 2024

**Advanced Materials Characterization Workshop**
**University of Illinois, Urbana-Champaign**

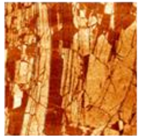
# Innovation through NEW products

**Apr 1, 1999**
**AR Birthday**
**MFP**
Molecular Force Probe


**2000-2001**
Closed-loop Z-scanner
Open-source AFM software


**2002**
**MFP-3D AFM**
First all-digital closed-loop AFM


**2005**
**Variable Field Module (VFM)**
AFM under applied magnetic fields

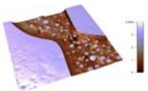

**2006**
**Bimodal Dual AC mode**


**2007**
**High-Voltage Piezoresponse Force Microscopy with DART**

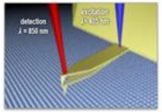

**2008**
**Cypher AFM**
The Highest resolution, Fast Scanning AFM


**2010**
**NanoRack**
AFM under applied mechanical strain

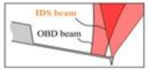

**2011**
**Cypher ES AFM**
Fully integrated environmental control


**2012**
**AR joins Oxford Instruments**
**AM-FM**
Viscoelastic mapping mode


**2013**
**Contact Resonance**
Viscoelastic mapping mode
**Origin AFM**

**blueDrive**
photothermal excitation


**2017**
**Cypher VRS**
First full-featured video-rate AFM


**2018**
**Cypher IDS**
Interferometer-based AFM



**2019**
**Jupiter XR**
Large-sample AFM


**2020**
**Ergo Software**
Workflow based for ease of use


**2021**
**Cypher VRS1250**
Up to 45 frame/s


**2022**
**Cypher L**
Entry-level Cypher
High-voltage and **nanoTDDB** for Jupiter

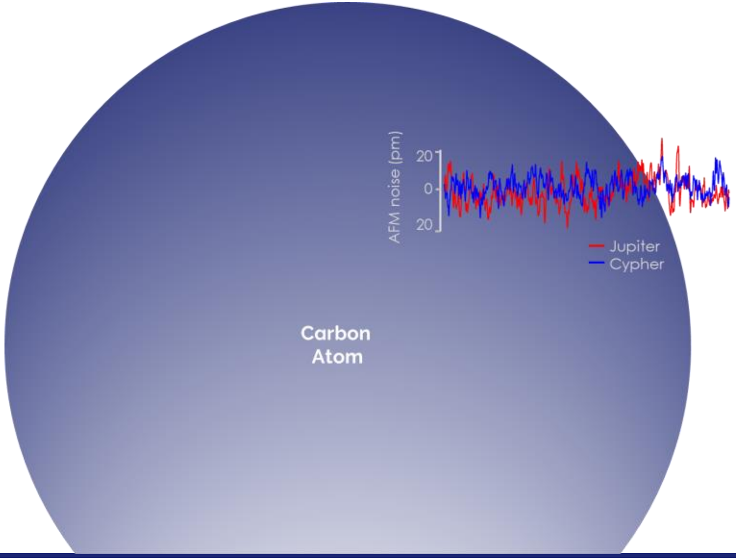
**2023**
**Vero AFM**
with **QPDI** detection

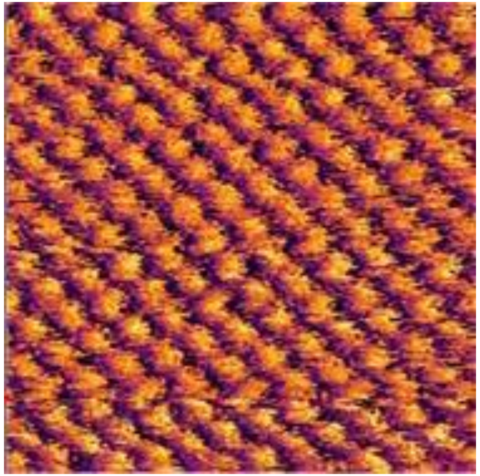

**2024**
CELEBRATING 1999 25 2024 YEARS OF ASYLUM RESEARCH

# AFMs = high resolution visualization

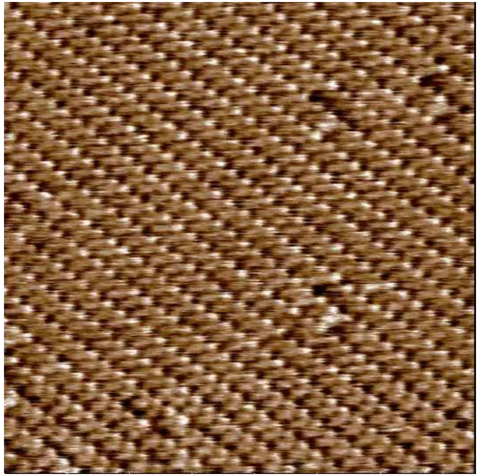
## AFMs visualize surface topography… down to atoms

- Sharp stylus (vs. light, electrons)
- Vertical resolution (z-noise <15 pm)
- Lateral resolution (tip radius <10 nm)

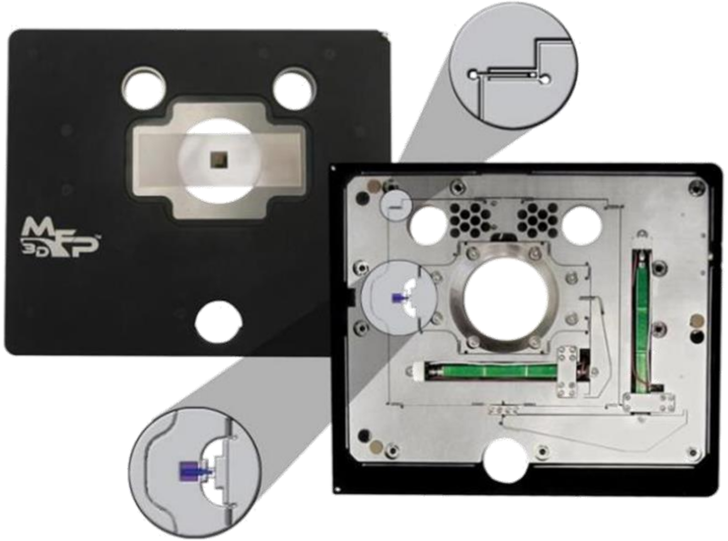



**Graphite lattice**
**1-5 nm scan**
**(contact mode - current image)**

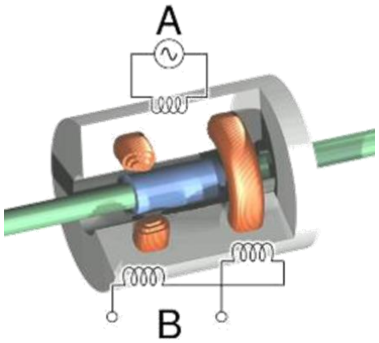


**Calcite point defects**
**10 nm scan**
**(tapping mode in liquid)**

# AFM = high resolution visualization



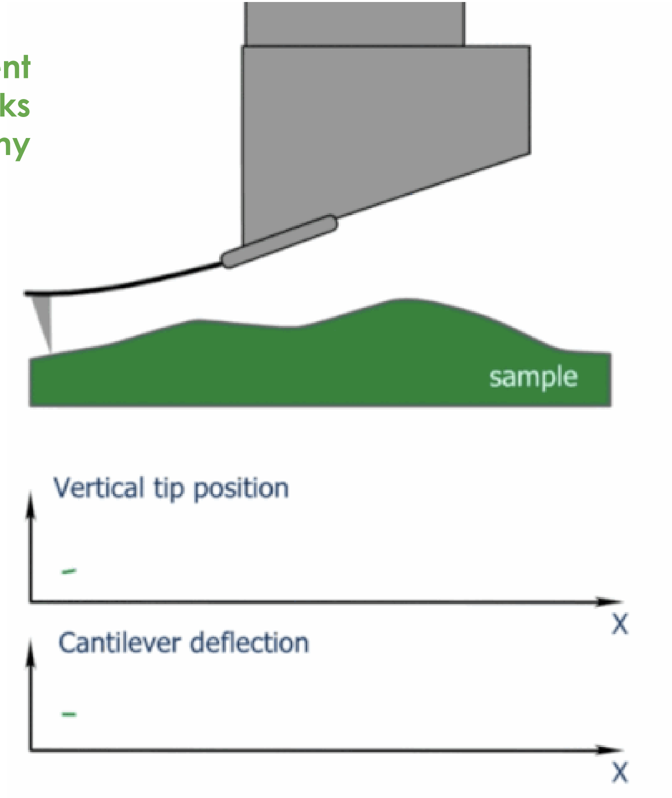

Asylum Research pioneered **closed-loop scanners**, which revolutionized scan accuracy




Asylum Research also developed the **lowest noise position sensors**, improving AFM scanner position

# How is topography obtained?

Z-piezo displacement (voltage) tracks topography




(Z-piezo Voltage)

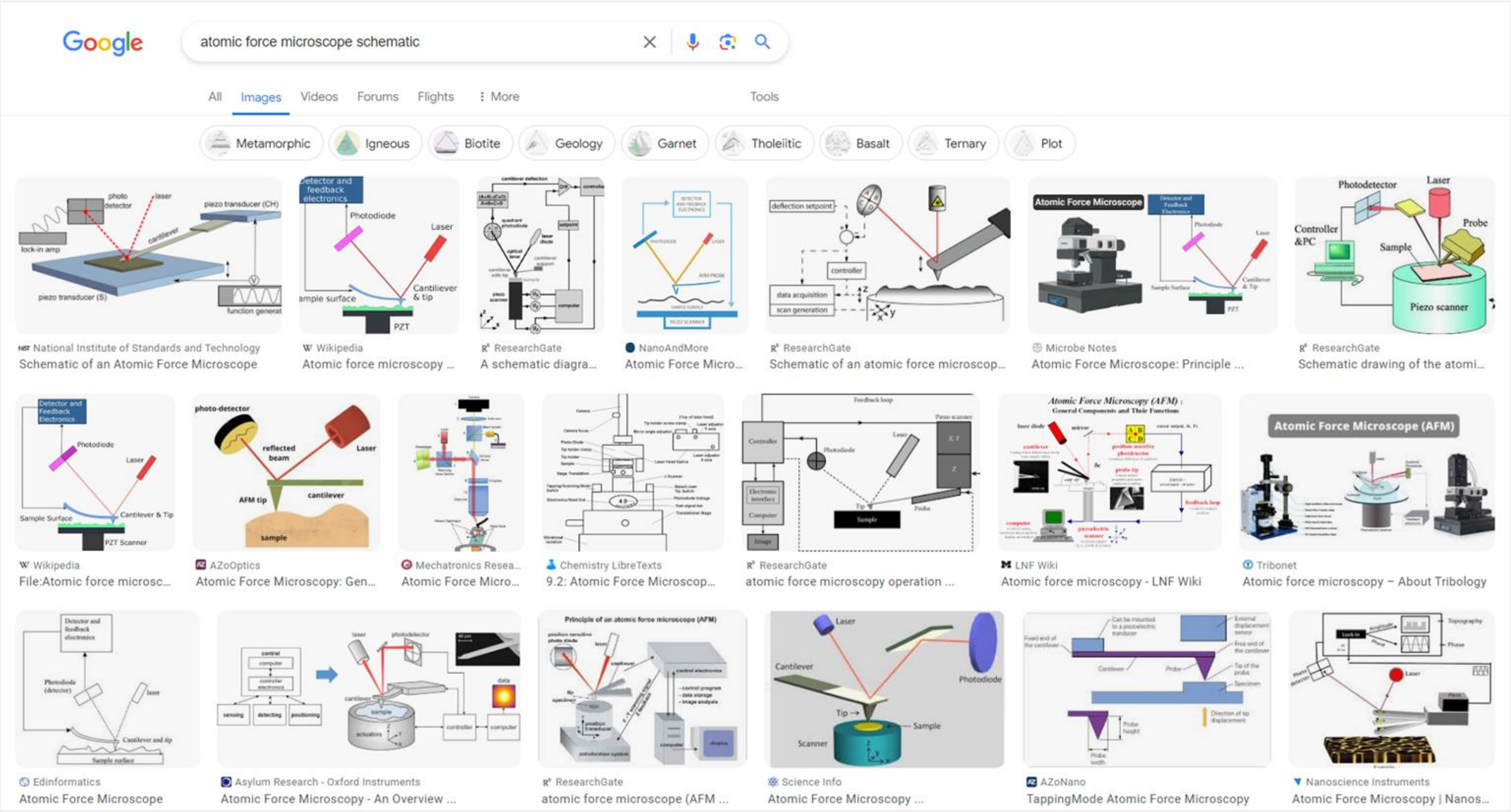
(Set point)

- In contact mode, the **height feedback loop** looks at deflection
- When an AFM tip is rastered across the surface, cantilever **deflection** changes when the tip encounters variations in topographic slope.
- The height feedback loop **adjusts the tip z-position** to keep deflection at setpoint, i.e., to maintain a constant loading **force**
- The actuating **voltage** to adjust (extend/retract) the z-piezo therefore tracks topography.

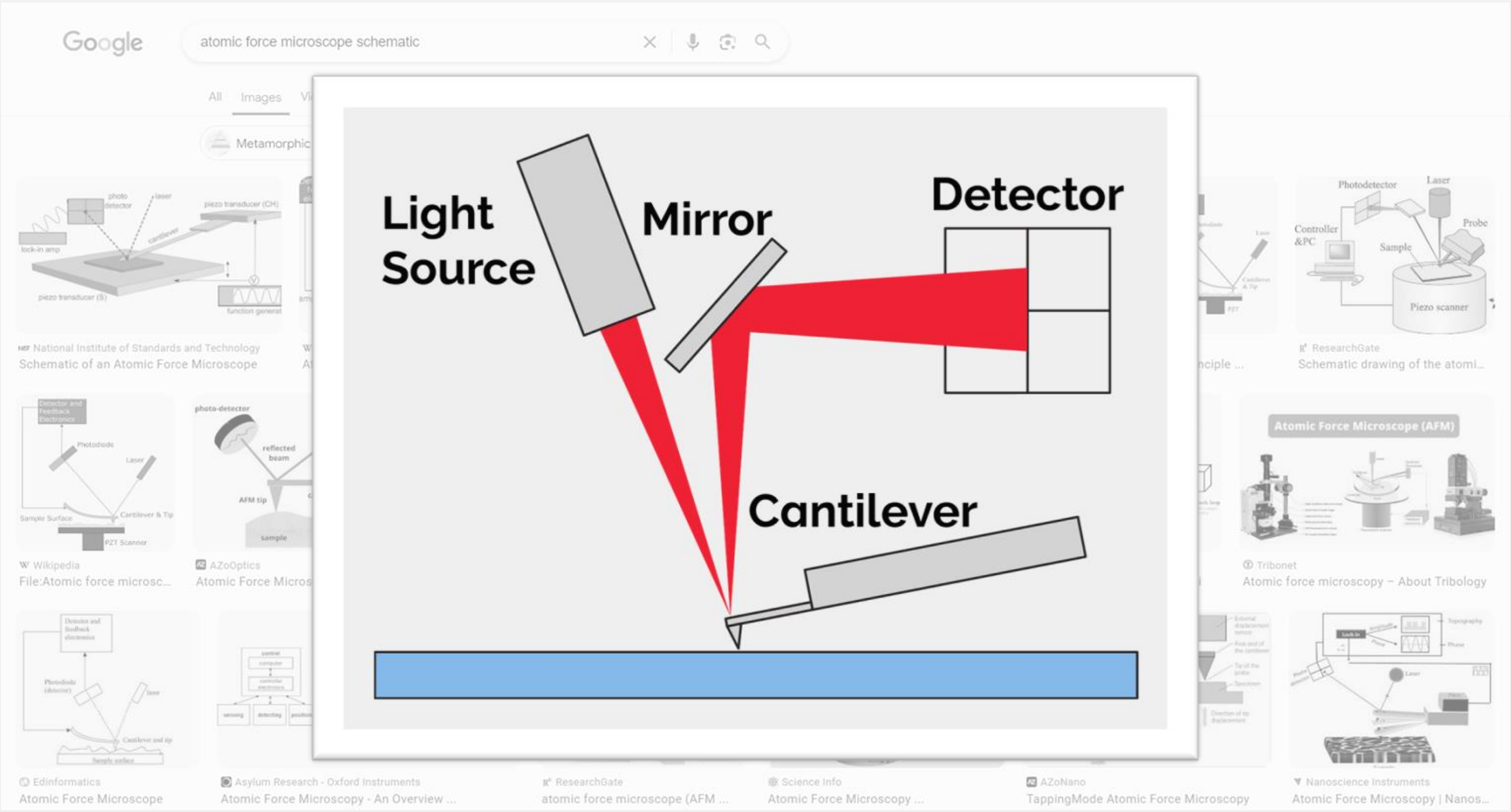


Image source: NTMDT Spectrum Instruments (www.ntmdt-si.com)

# How is deflection detected?



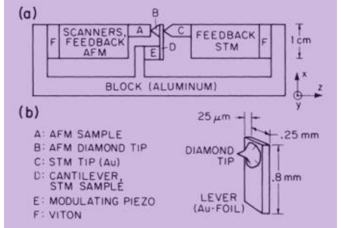
# Optical beam detection (OBD)



# How did we get OBD?

1986

AFM Invented



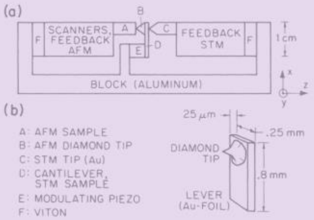

Binnig, G., Quate, C. F. & Gerber, Ch. Phys Rev Lett 56, 930–933 (1986)

Binning, Quate, and Gerber's first AFM used an STM!

# How did we get OBD?

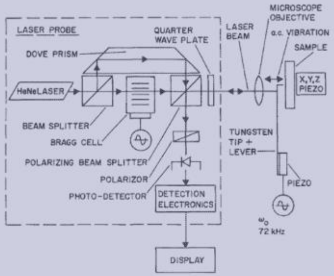
1986

AFM Invented




1987

Interferometric AFM



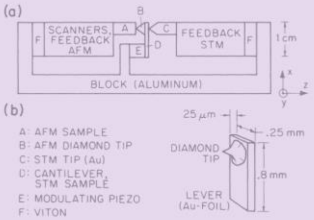

Martin, Y., Williams, C. C. & Wickramasinghe, H. K. J. Appl. Phys. 61 4723 (1987)

Several early AFM designs used interferometric sensing

# How did we get OBD?

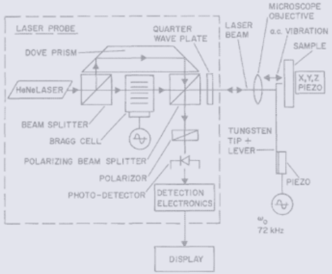
## 1986

### AFM Invented




## 1987

### Interferometric AFM




## 1988

### Optical Beam Deflection "OBD"

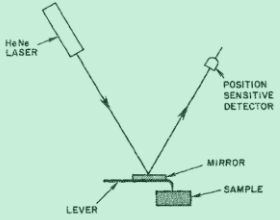
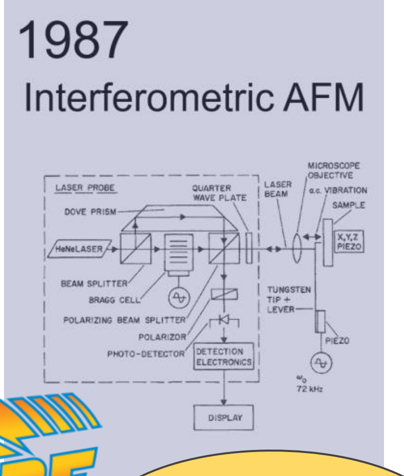



FIG. 1. Cantilever deflection detection scheme.

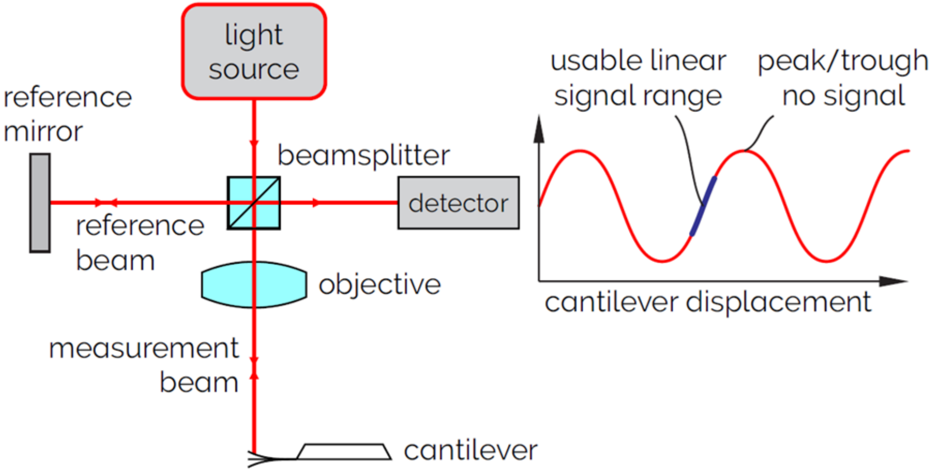
Meyer, G. & Amer, N. M. Appl Phys Lett 53, 1045–1047 (1988)

Why did OBD become the default?

# Early interferometric AFMs had issues…





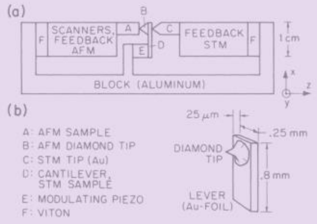



- Limited measurement range
- Poor low-frequency noise / drift
- Complex to build and use

# Revisiting interferometric detection

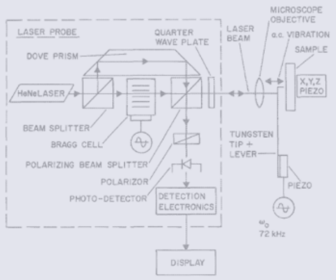
1986

AFM Invented




1987

Interferometric AFM




1988

Optical Beam Deflection "OBD"

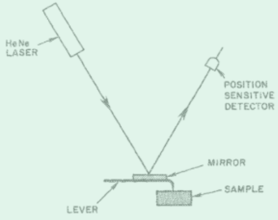
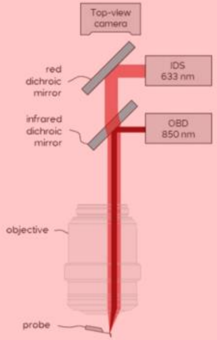



FIG. 1. Cantilever deflection detection scheme.

2018

Cypher IDS



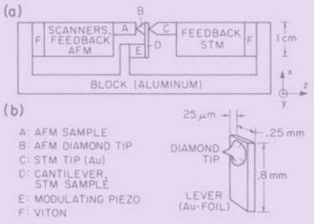

Labuda, A., & Proksch, R. Appl. Phys. Lett. 106 (2015)

30 years later…

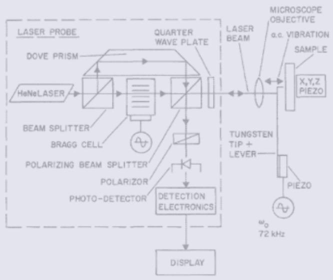
**Cypher IDS**: interferometric detector demonstrating benefits for piezoresponse force microscopy (PFM). But still poor low-frequency noise…

# New in 2023: AFM with QPDI detector

1986
AFM Invented




1987
Interferometric AFM




1988
Optical Beam Deflection "OBD"

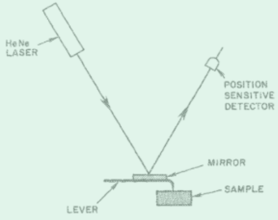
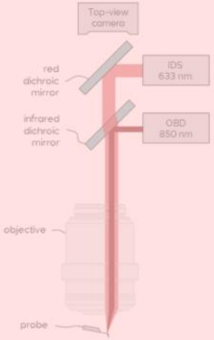
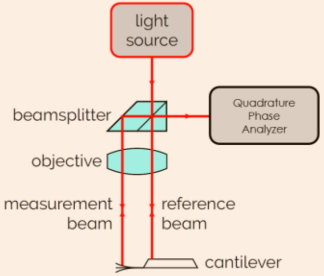



FIG. 1. Cantilever deflection detection scheme.

2018
Cypher IDS




2023
Vero

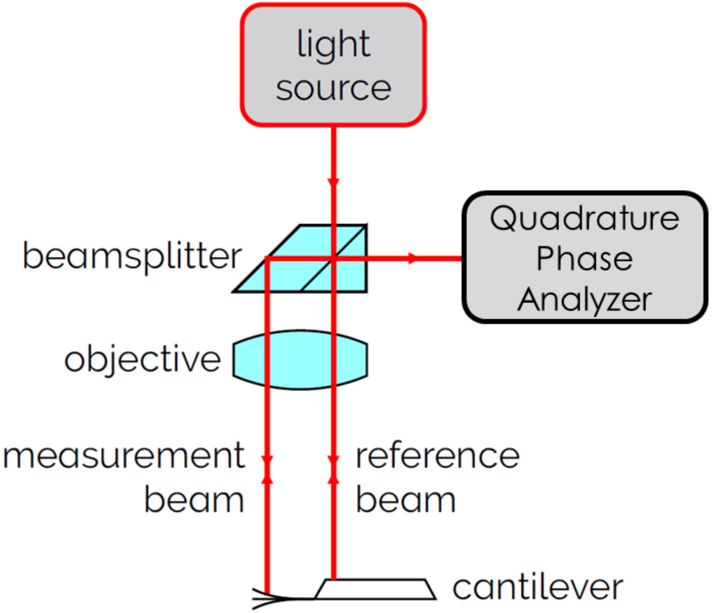



Fall MRS 2023

**Vero**: first AFM to use quadrature phase differential interferometry (QPDI) detector.

👉 **So what is different about QPDI?** 👈

# Quadrature phase differential interferometry (QPDI)




**"Quadrature Phase"** Two interferometric signals are generated, where the second has a 90° phase delay with respect to the first… a bit like running two interferometers in parallel

- Benefit: QPDI can measure very large displacements and do so while maintaining optimal noise performance

**"Differential"** Instead of using a remote reference mirror, the back of the probe substrate acts as the reference, i.e., <1 mm from the AFM tip

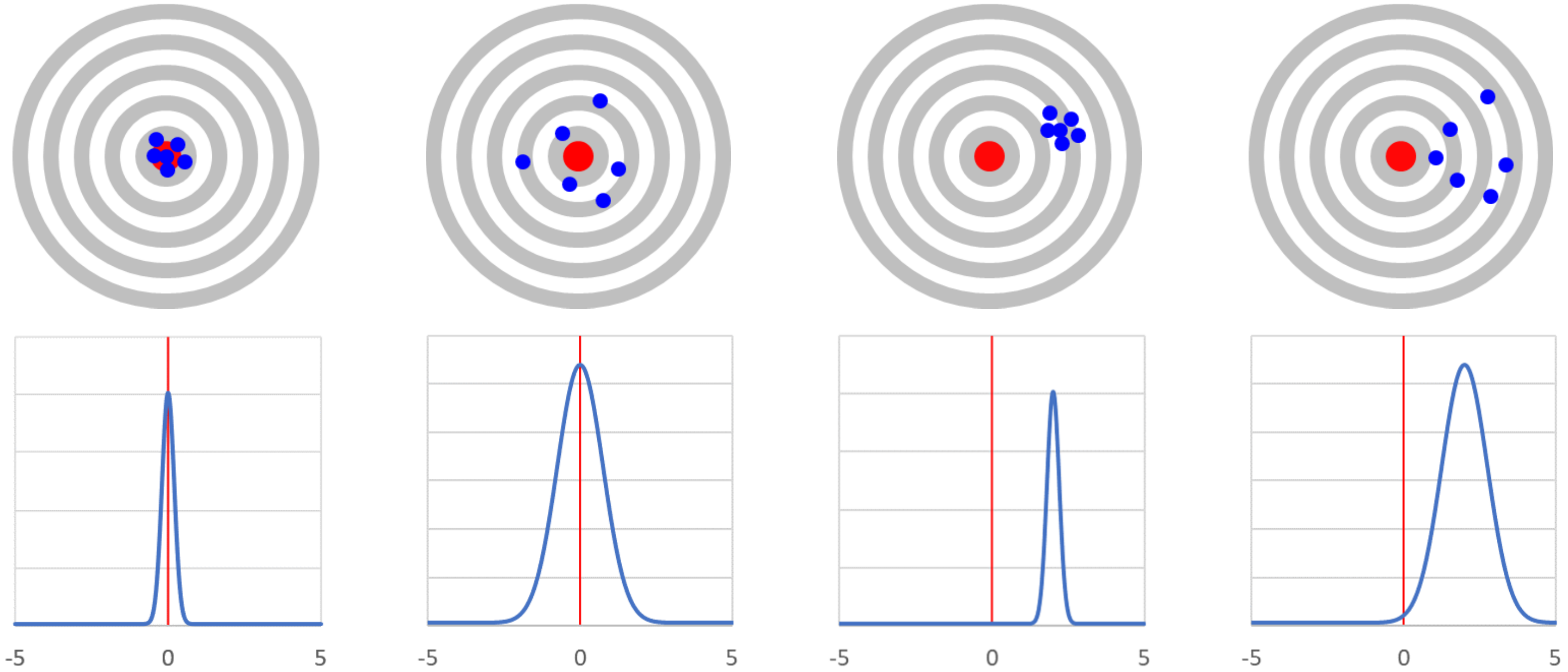
- Benefit: QPDI has dramatically better low-frequency noise and drift

# Vero: very accurate and very precise

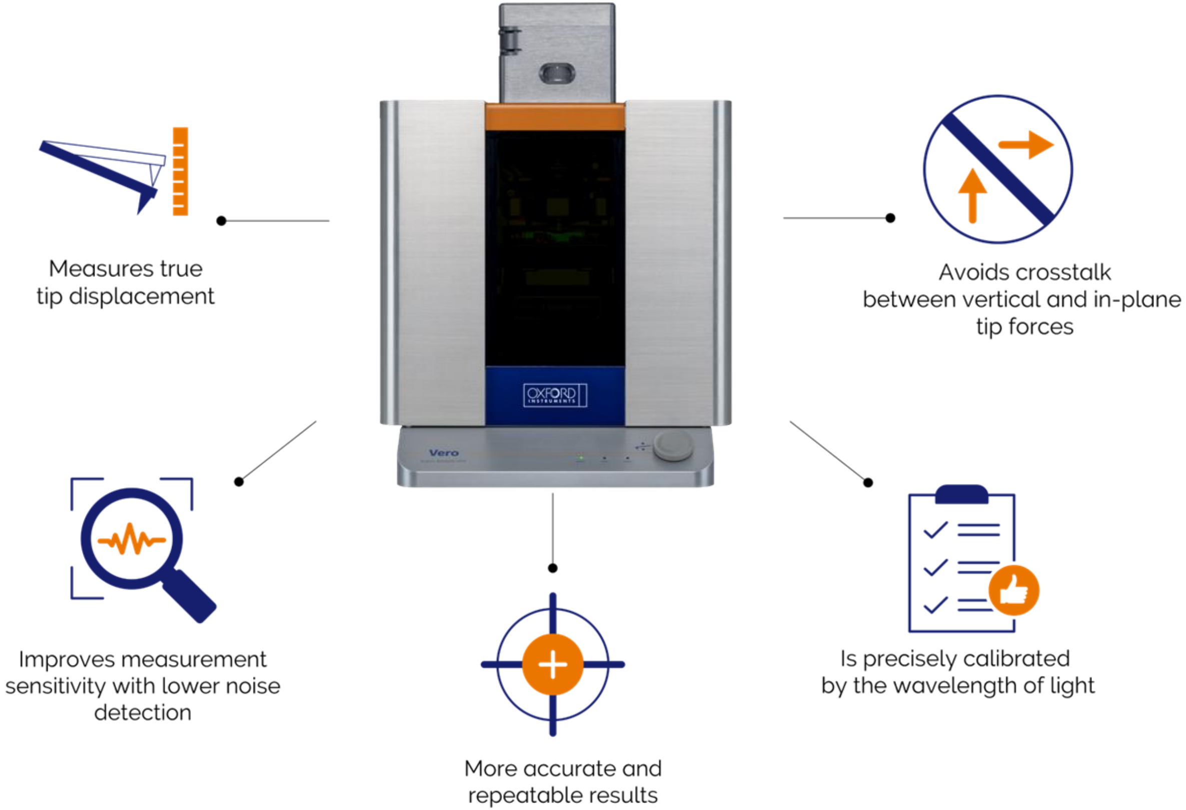

- Vero is a next-generation AFM family from Oxford Instruments Asylum Research
- Vero uses **Quadrature Phase Differential Interferometry** (**QPDI**) to produce more accurate and precise AFM results
- Vero builds on the ultra-high performance, stability, and capabilities of Cypher AFMs

# Accuracy and precision

Accurate and precise | Accurate but less precise | Less accurate but precise | Less accurate and less precise



# Five key benefits



# Five key benefits

# Vero measures true tip displacement

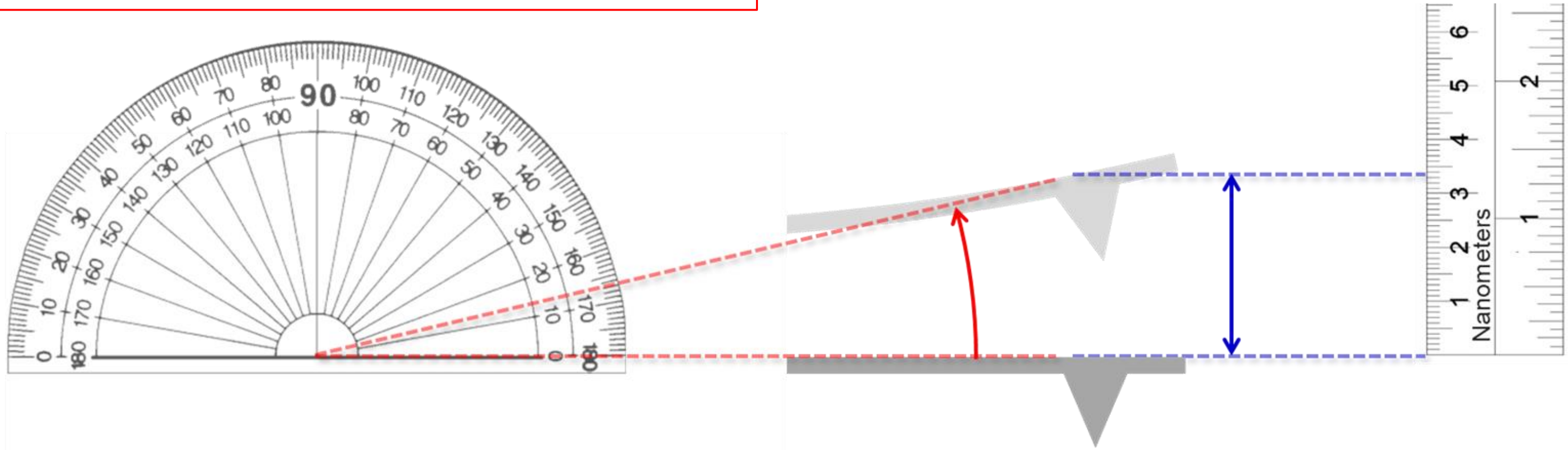
OXFORD INSTRUMENTS

## Optical beam deflection (OBD)

- OBD measures **cantilever angular changes**
- Angular changes must be converted into displacement
- Conversion value (optical lever sensitivity or "InvOLS") is dependent on many factors (e.g., spot position, spot size, cantilever length, etc.)

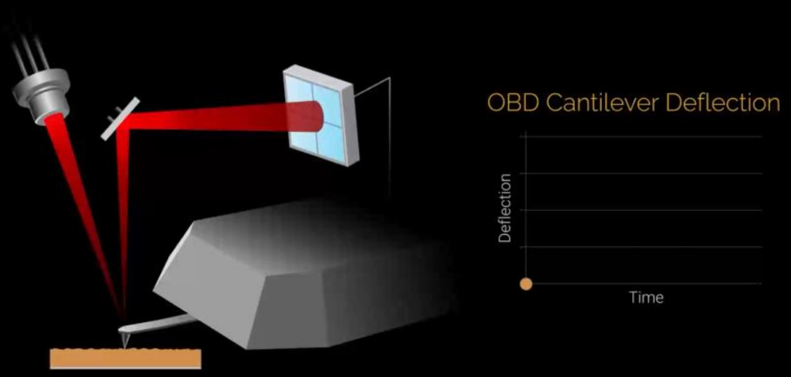
## Quadrature phase differential interferometry (QPDI)

- Vero QPDI measures **tip displacement** directly
- "InvOLS" is a fixed constant (510 nm/V)



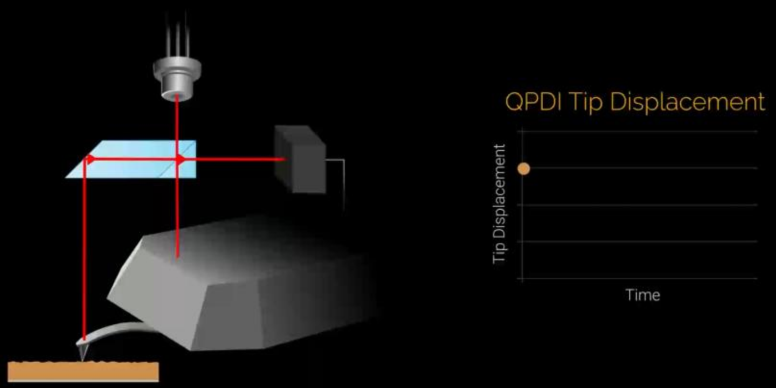
# You can't solve OBD errors with better calibration

## OBD measures angular deflection




- All angular changes are interpreted as tip displacement
- But lots of things can affect cantilever bending besides tip displacement

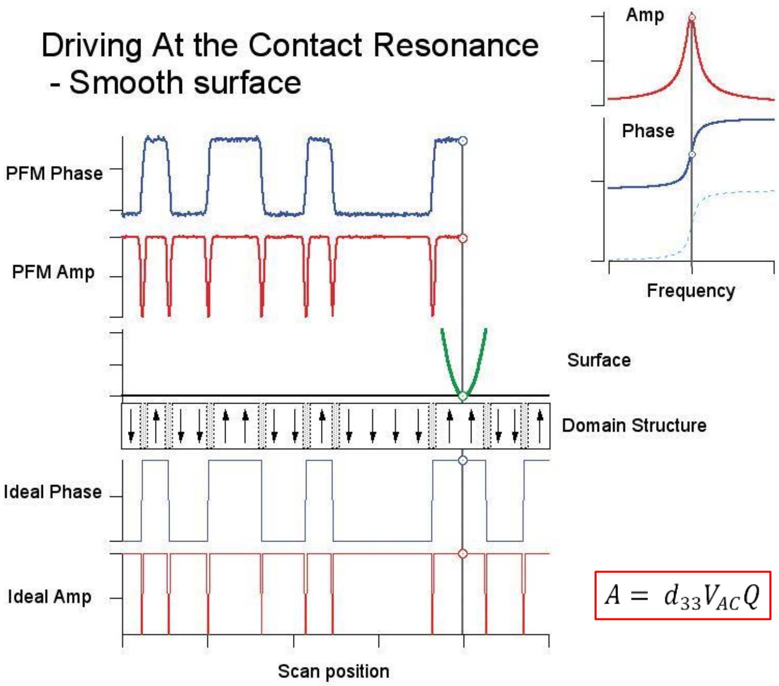
## QPDI measures tip displacement



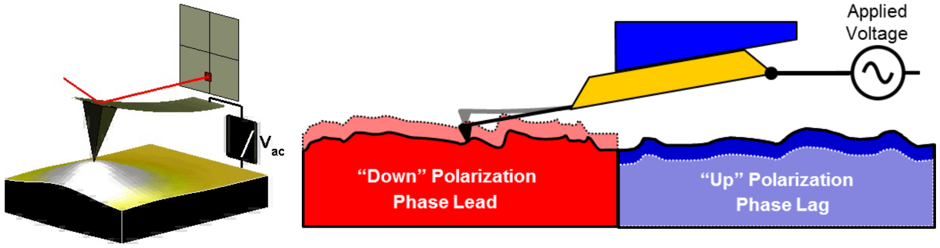

- No tip displacement detected even when there is cantilever bending

**When does this happen?**

# Piezoresponse force microscopy

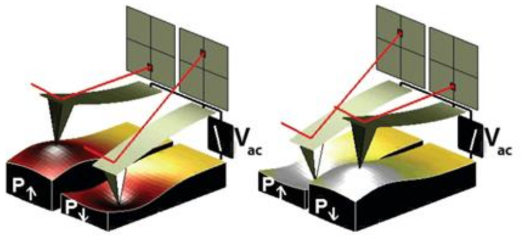



$$A = d_{33} V_{AC} Q$$

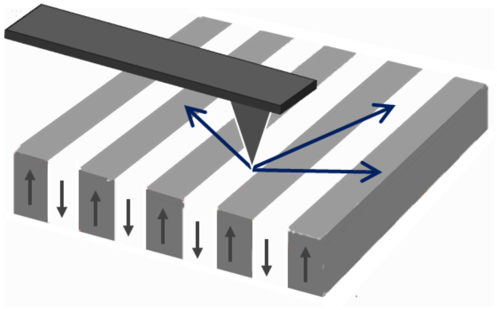



- **Amplitude** is related to the magnitude of the bias and the "effective" vertical piezo coefficient, ($\boldsymbol{d_{eff}}$)

  PFM $\boldsymbol{d_{eff}}$ values range from 0.1 pm/V to 500 pm/V
- **Phase** gives directional information, i.e., domain polarization:



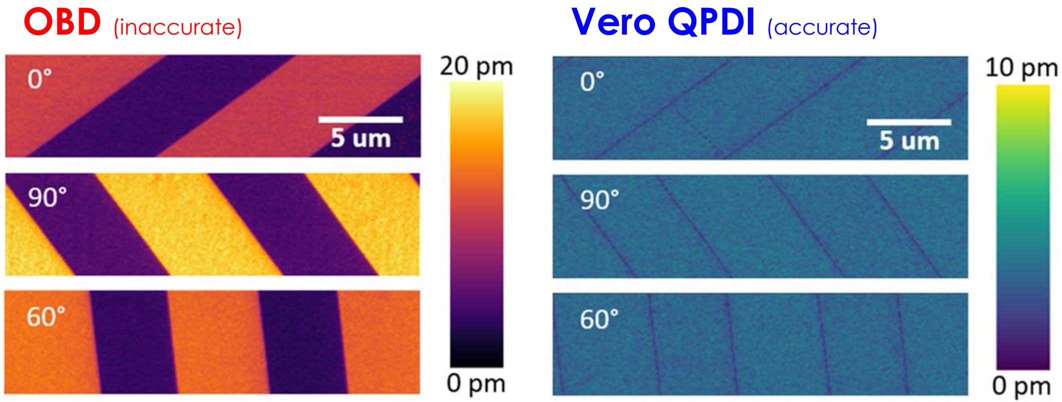
# Accurately removes tip-sample friction



## Periodically poled lithium niobate (PPLN)

- Patterned with up and down domains
- Same material but oppositely polarized
- Magnitude of piezoresponse should be the same

## PFM imaging on PPLN



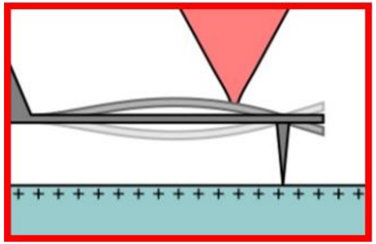

- **Tip-sample friction** contributes to cantilever bending
  - OBD shows inaccurate variation in amplitude of piezoresponse, as well as variation with scan angle
  - Due to variable in-plane forces from friction
- QPDI detection not affected by tip-sample friction

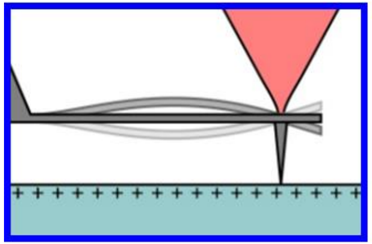


Proksch, R., Wagner, R., & Lefever, J. (2024). Accurate vertical nanoelectromechanical measurements. Journal of Applied Physics, 135(3).

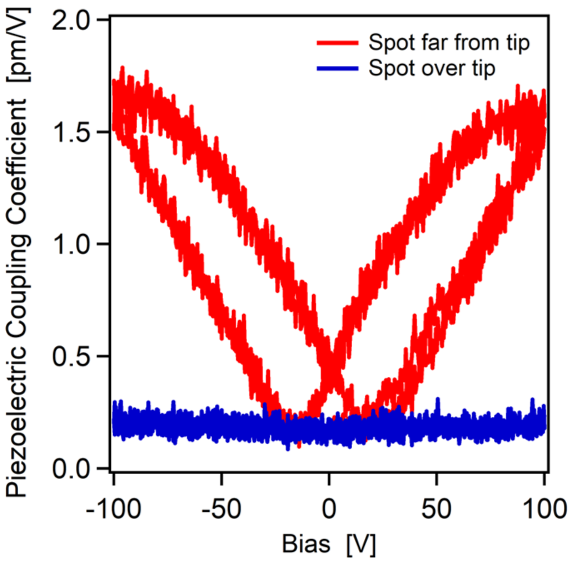
# Accurately removes electrostatic effects

- During PFM, drive bias can drive the cantilever via **electrostatic forces**
- Soda lime glass, though not ferroelectric, can exhibit cantilever oscillation if…
  - OBD is used (measures cantilever angle)
  - QPDI is used if spot isn't over the tip
- PFM with QPDI unaffected if spot is over tip





**QPDI SS-PFM on soda lime glass**



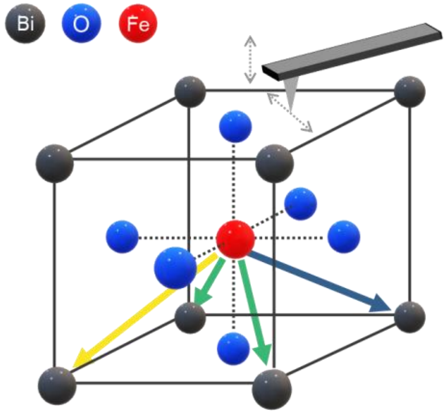

✗ inaccurate
✓ accurate



Collins, L., Liu, Y., Ovchinnikova, O. S., & Proksch, R. (2019). Quantitative electromechanical atomic force microscopy. ACS nano, 13(7), 8055-8066

# Five key benefits

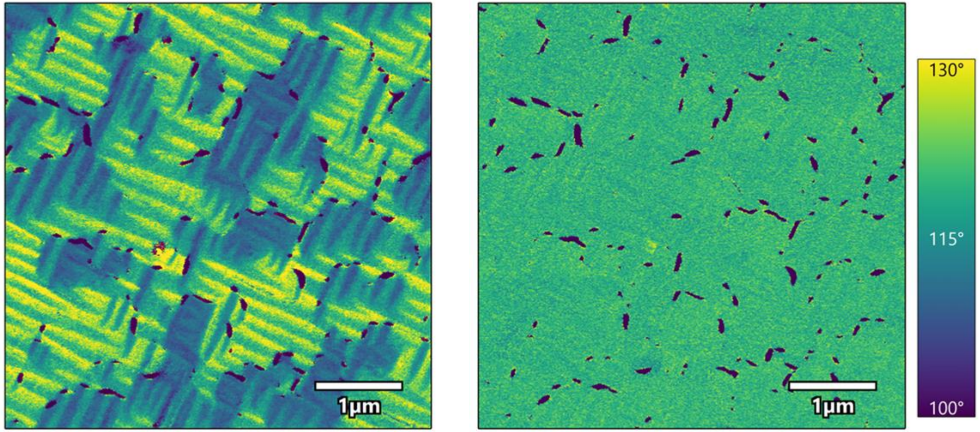
# Accurately removes in-plane piezoresponse




- Many piezoelectric materials exhibit both in-plane and out-of-plane response
- PFM response on BFO (100) has both in-plane and out-of-plane response

## PFM on bismuth ferrite ($BiFeO_3$ or "BFO")

**OBD** (inaccurate) **Vero QPDI** (accurate)




- **Crosstalk** of in-plane response into vertical response
  - OBD-based PFM claims to distinguish between vertical and lateral response, but in-plane tip-sample forces couple into the vertical deflection
- QPDI vertical response unaffected by in-plane forces

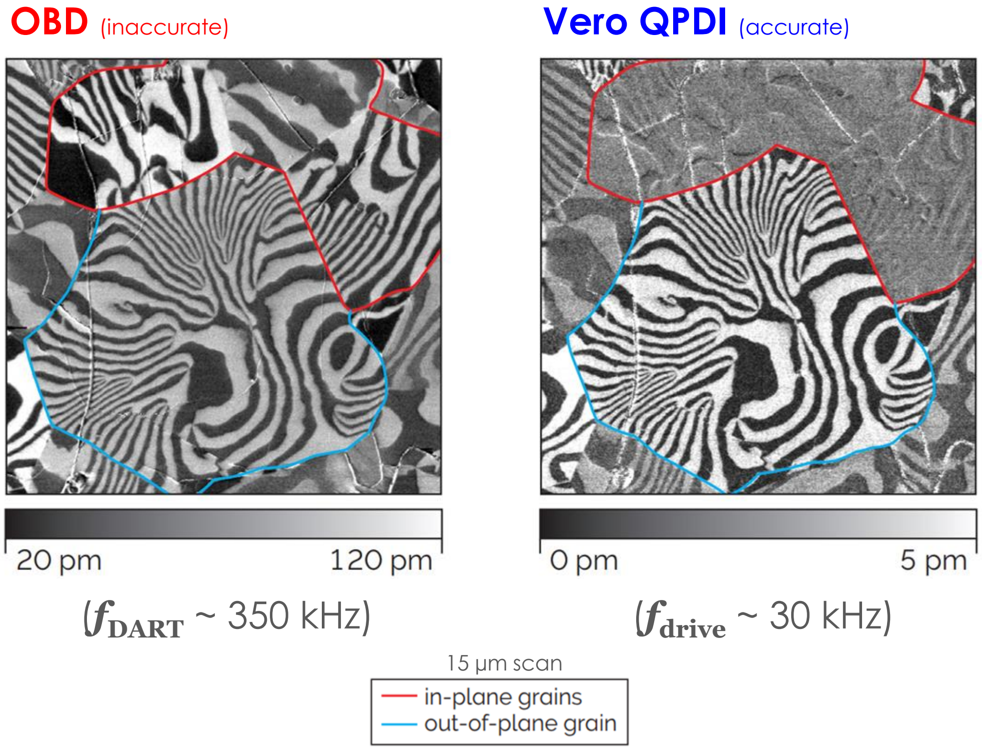


Sample courtesy of Ying-Hao Chu, National Tsing Hua University, Taiwan

# Accurately removes in-plane piezoresponse

- Crosstalk of in-plane response into vertical response
  - Red outline highlights grains that have mostly in-plane response.
  - Blue outline highlights grain with out-of-plane response.
  - In plane response is visible in OBD (artifact) but not in QPDI.
- Note: DART used in OBD to accentuate signal

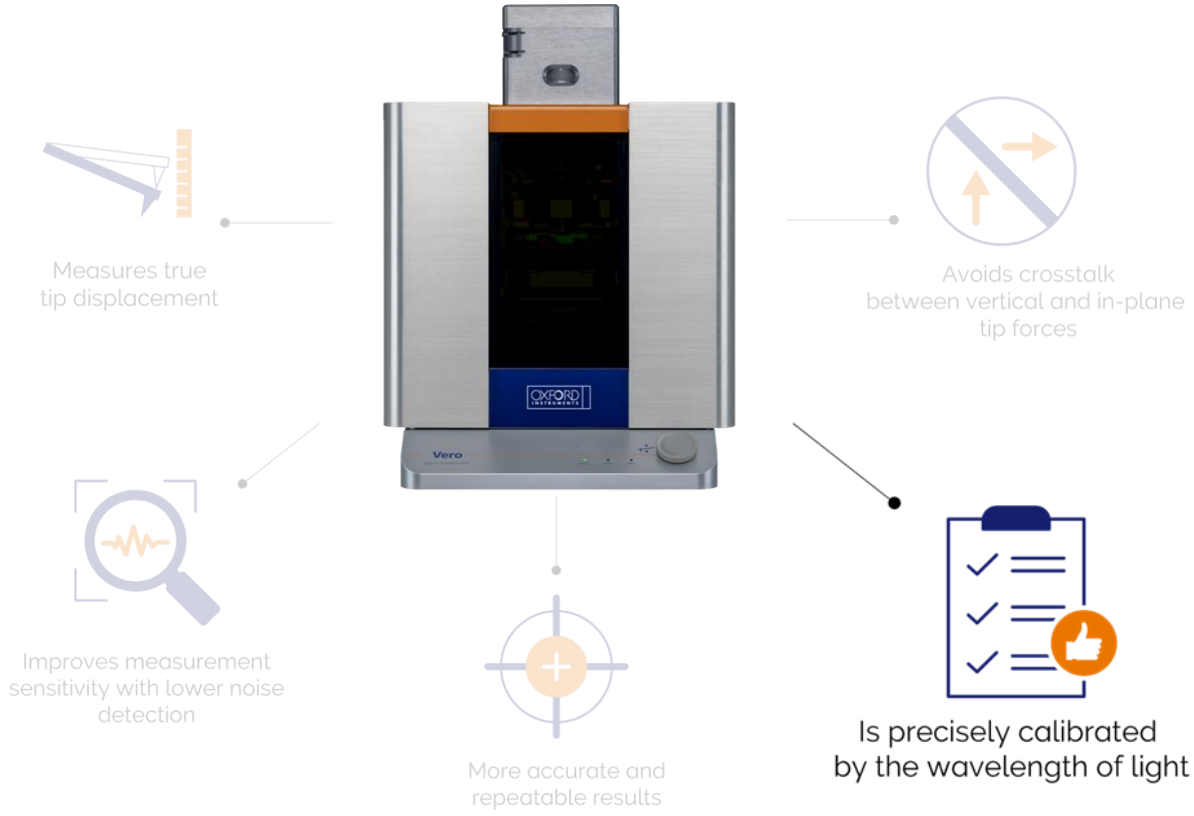
## PFM on erbium manganese trioxide ($ErMnO_3$)






Sample courtesy of Jan Schultheiß and Dennis Meier, Norwegian University of Science and Technology

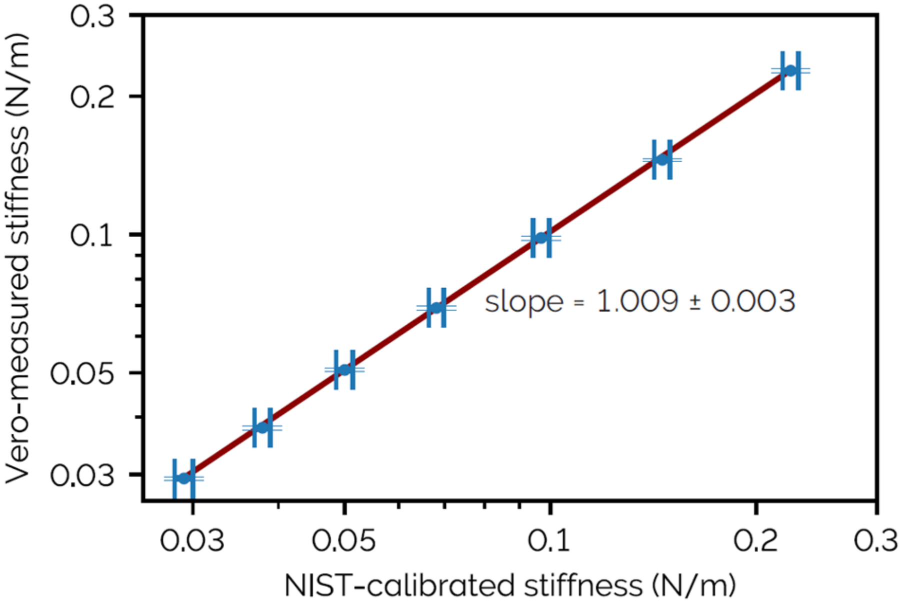
# Five key benefits



# Accurately calibrates spring constants

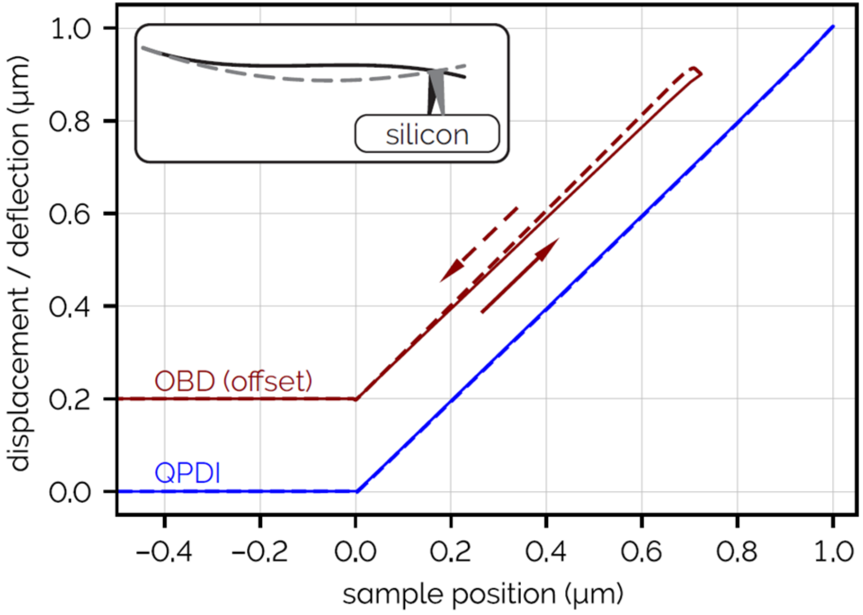
- With OBD, **10-20% error** in cantilever spring constant calibration is typical.
- Vero's built-in interferometer and direct measure of tip displacement reduces this error.
- Sensitivity (InvOLS) is fixed by the wavelength of light used (510 nm/V)

Vero QPDI spring constant calibrations agreed with NIST SI-traceable values **to within 1%**



# Accurately removes buckling in force curves

- Force curve on a hard surface:
  - Extend and retract curves should be linear and overlap if there's no indentation
  - Tip slides and pivots during contact
- Tip-sample friction can cause **bucking** that results in cantilever angular change
  - OBD interprets this as tip displacement, resulting in hysteresis at the turnaround
  - Pivoting of the tip does not change its vertical displacement, so QPDI signal is not affected
  - Tip "plowing" is real and probably affects indentation, but it shouldn't appear in the force curve



# Five key benefits

# Detector with the lowest noise floor

## OBD

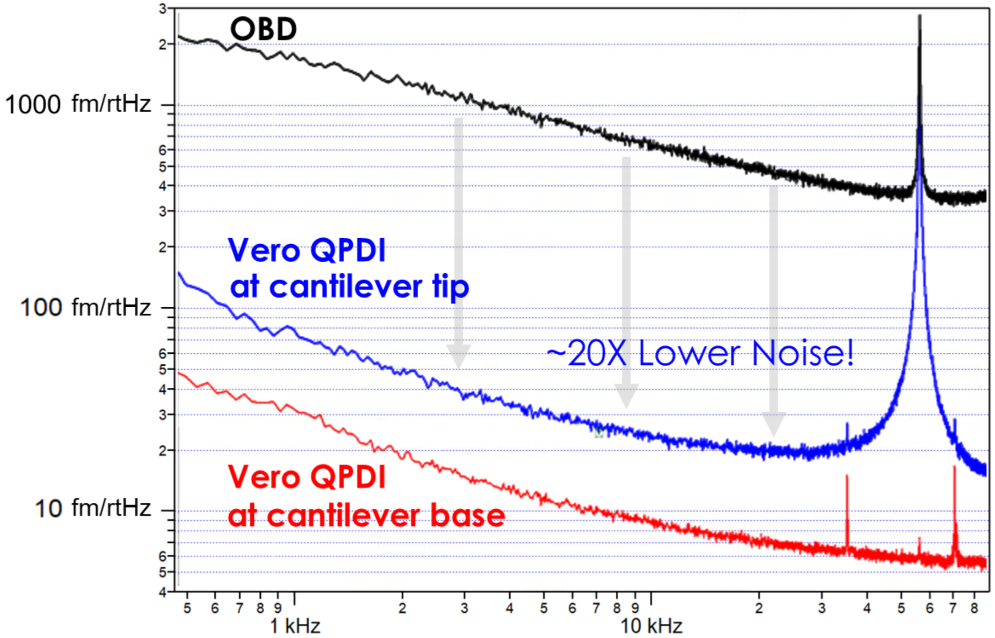
- Noise floor depends on cantilever size vs. spot size
- Best case: ~25 fm/√Hz if carefully optimized, but usually 200-500 fm/√Hz

## QPDI

- Noise floor does not depend on cantilever size vs. spot size
- Consistently <10-20 fm/rtHz above 20 kHz regardless of cantilever or spot size
- Actual **detector noise** can be measured at the base
- So signal at the tip is actual **sub-resonance thermal motion**
- This means what we are seeing is thermally limited motion even for stiffest levers

**Adama AD-2.8-AS probe**

**(w ~ 35 µm, l ~ 225 µm, f ~ 65 kHz, k ~ 2.8 N/m)**

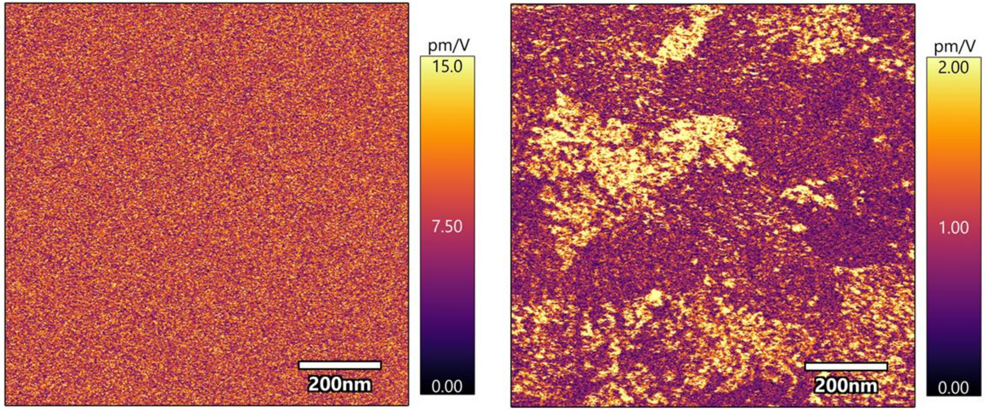


# Precisely measure low-response material

- $HfO_2$ is uniquely compatible with Si and can be naturally **integrated in logic and memory devices**
- Processing into thin films is key to transforming $HfO_2$ into its ferroelectric crystalline state
- Vero clearly **resolves piezoelectric domains** in hafnia even though the response is <2 pm/V
- Identical settings used for these two images. Only difference is detector type (OBD vs QPDI)
- DART or HV - but will not work on samples with low breakdown or coercive voltage

## PFM on hafnia ($HfO_2$)

**OBD** (weak signal) **Vero QPDI** (strong signal)




Technique: single-frequency PFM at 30 kHz, 2 V drive amplitude, and 290 nN setpoint



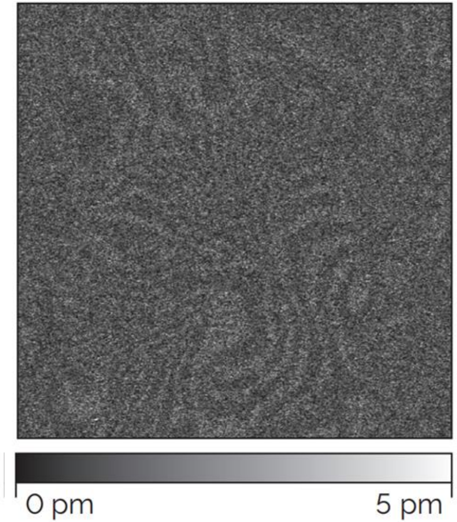
Sample courtesy of NamLab, Germany

# Precisely measure low-response material

## PFM on erbium manganese trioxide ($ErMnO_3$)

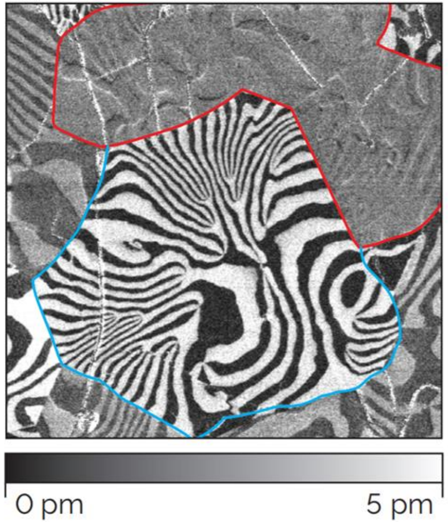
- Identical settings used for these two images. Only difference is detector type (OBD vs QPDI)
- Vero clearly **resolves piezoelectric domains** which highlights much lower noise floor of the QPDI detector
- DART (resonance amplification) an option, but may still contain artifacts

**OBD** (weak signal)




($f_{DART}$ ~ 350 kHz)

**Vero QPDI** (strong signal)

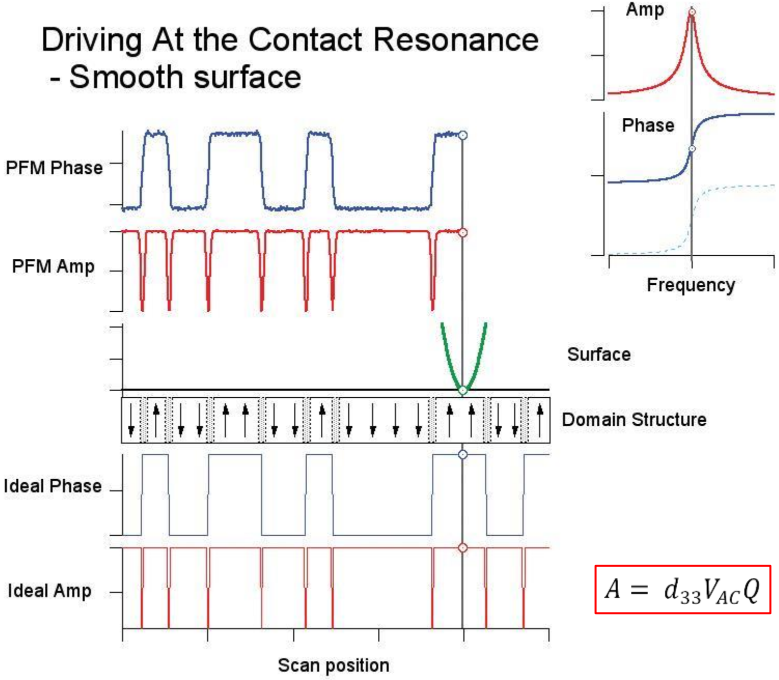



($f_{drive}$ ~ 30 kHz)

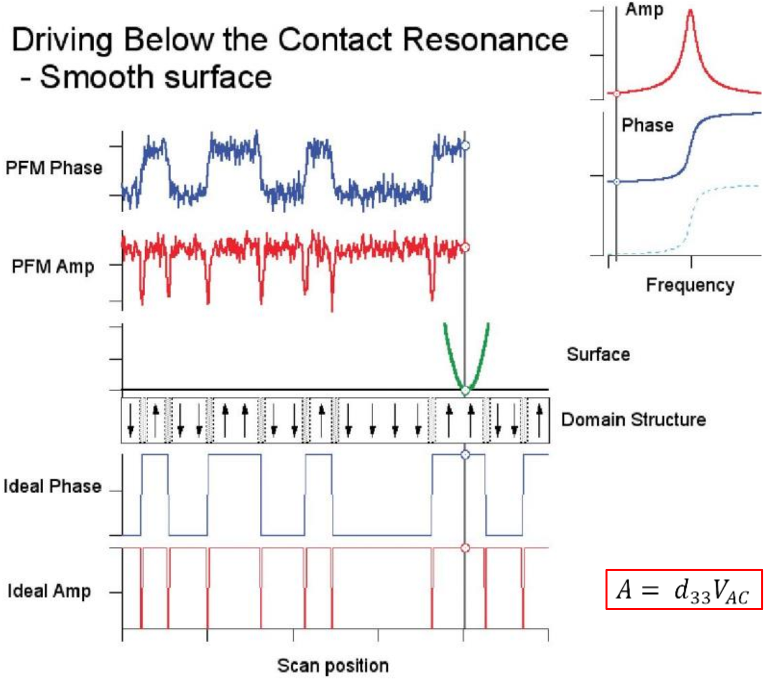


Sample courtesy of Jan Schultheiß and Dennis Meier, Norwegian University of Science and Technology

# On-resonance vs. Off-resonance PFM




$A = d_{33}V_{AC}Q$



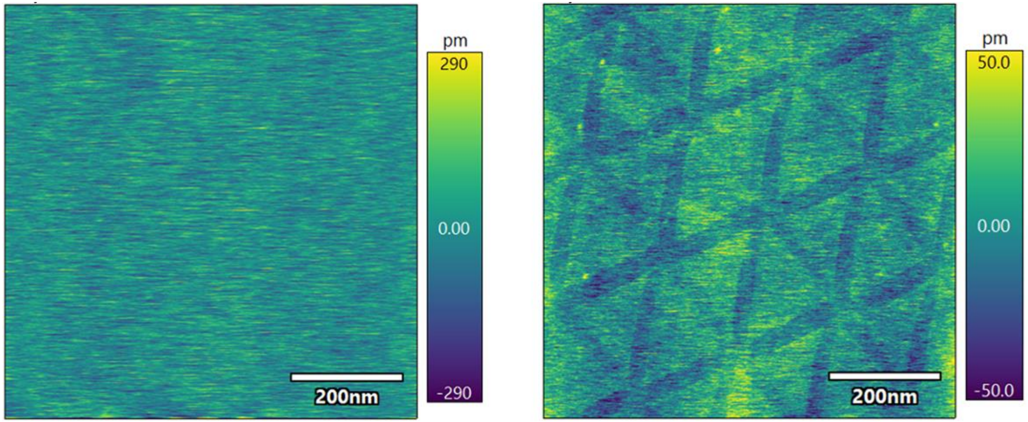

$A = d_{33}V_{AC}$

# Precisely measure tBLG moiré

- **Moiré patterns** form when 2D materials are stacked
- Varying the relative rotation (or **twist angle**) alter their electronic properties
- tBLGs can either be superconductors or correlated insulators within a narrow range of "magic" angles around 1.1 degrees.
- Moire pattern typically not observed in **topography** so other contrast mechanisms used (LFM, PFM)
- Owes to the **low noise floor** of Vero
- What else can Vero potentially see?

## Twisted bilayer graphene (tBLG) moiré

**OBD** (weak signal) **Vero QPDI** (strong signal)

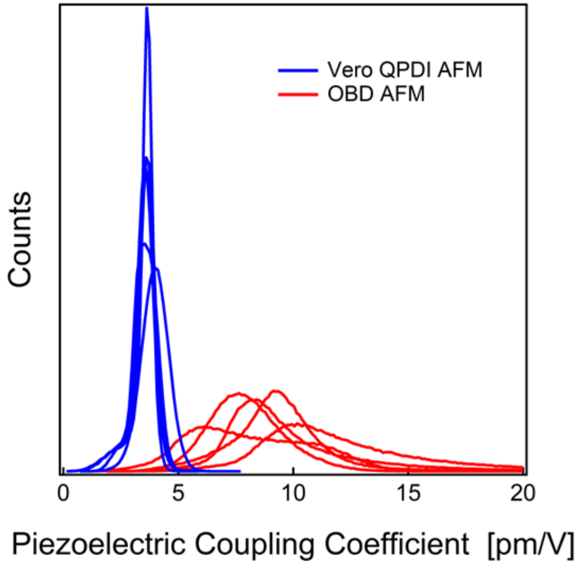


# Five key benefits

# Precisely quantify Sc doping in AlN

- AlN (a piezoelectric material) is used in **acoustic resonators** in wireless devices
- Many advantages, e.g., compatible with high temperature, CMOS processing, etc.
- But has low piezoresponse (6 pm/V)
- Alloying AlN with Sc increases response by up to 4x
- **Processing needs to be optimized**, e.g., balanced with defects at higher Sc concentrations
- Requires **good correlation with Sc levels and piezoresponse** to evaluate processing
- Vero QPDI results show both more precise measurements (narrower distributions) and more repeatable measurements (peaks tightly clustered) compared to OBD

**PFM of scandium aluminum nitride (ScAlN)**




Technique: single-frequency PFM at 30 kHz, 1-5 V drive amplitude, and 290 nN setpoint



Sample courtesy of A. Žukauskaitė and S. Barth, Fraunhofer FEP, Germany

# QPDI improves AFM accuracy and precision

## Accuracy (systematic errors)

- Measurement artifacts
  - Electrostatic artifacts in PFM measurements

  - Pivoting or buckling of tip during force curves

- Calibration errors
  - Error in INVOLS calibration (aka deflection sensitivity)
  - Error in spring constant calibration

## Precision (random errors)

- Measurement noise
  - Noise in OBD deflection signal

  - Dependence of noise on cantilever
- Calibration repeatability
  - User variability in INVOLS calibration
  - User variability in spring constant calibration

# Thank You! Questions???

**Vero**

**AFM Redefined**