Zurich Instruments

Boost Your SPM Applications: From Kelvin Probe to Time-Resolved Measurements

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Zurich Instruments for scanning probe microscopy (SPM)

What will you learn in this webinar?

Part 1: Kelvin Probe Force Microscopy (KPFM)

Live demonstration with Nanosurf Flex AFM and MFLI

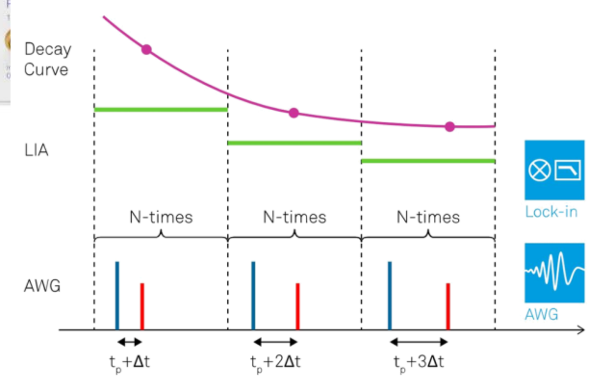
- Amplitude modulated KPFM
- Heterodyne KPFM

→ Multiple modes with single instrument

Part 2: Time-resolved AFM methods

Live Demonstration with UHFLI

→ Electrical pump-probe methods



Part 3: Q&A

Zurich Instruments for scanning probe microscopy (SPM)

Electrostatic Force Microscopy (EFM)

Non-Contact Atomic Force Microscopy (NC-AFM)

Kelvin Probe Force Microscopy (KPFM)

Piezoresponse Force Microscopy (PFM)

Phase-Locked Loop (PLL)

Multifrequency

Resonance Tracking

Sideband Analysis

Electron Spin Resonance-STM

Scanning Near-Field Optical Microscopy (SNOM)

Time-Frequency Domain Analysis

Scanning Probe Microscopy

Band Excitation AWG

Spin Polarised-STM

Tip-Enhanced Raman Spectroscopy

Data Acquisition

Electrical Pump-Probe

Cascaded Feedback Loops

Harmonic Distorsion Analysis

Scanning Tunneling Spectroscopy (STS)

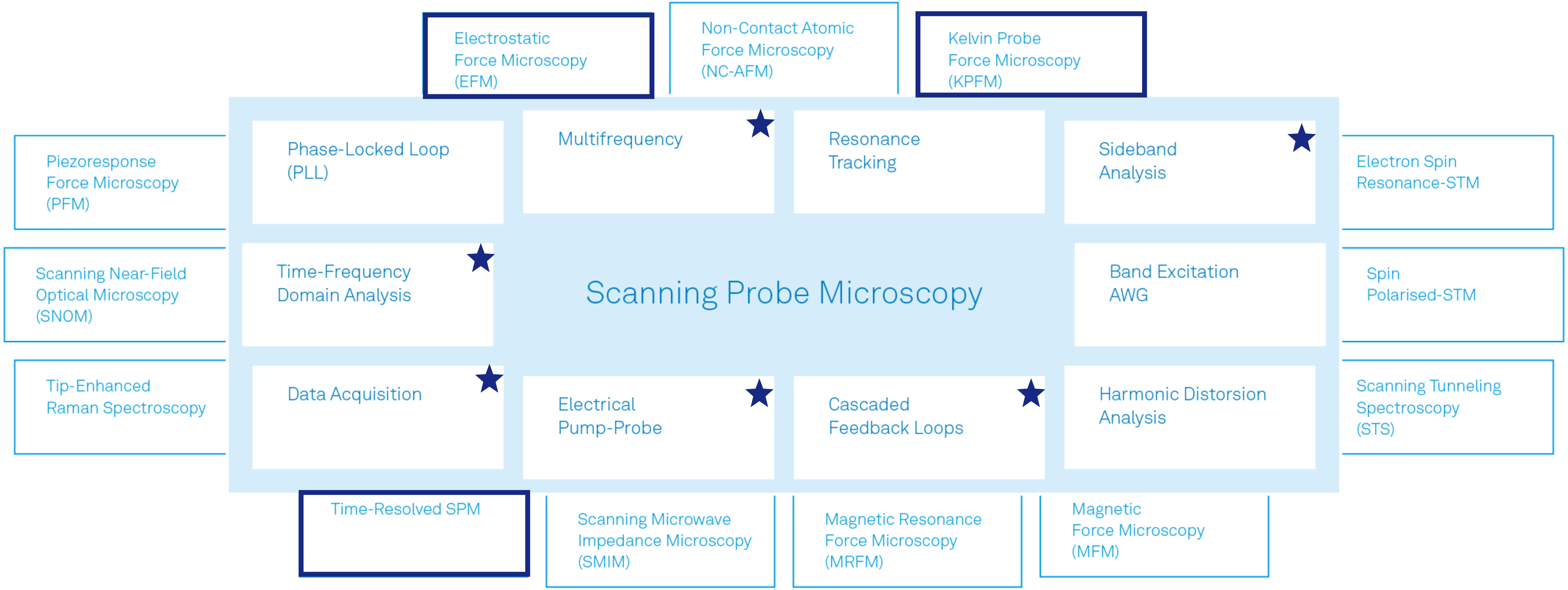
Time-Resolved SPM

Scanning Microwave Impedance Microscopy (SMIM)

Magnetic Resonance Force Microscopy (MRFM)

Magnetic Force Microscopy (MFM)

Zurich Instruments for scanning probe microscopy (SPM)



Zurich Instruments for scanning probe microscopy (SPM)

Time and frequency domain analysis

Probing multiple force contributions

Actuation and demodulation schemes in frequency domain

Actuations

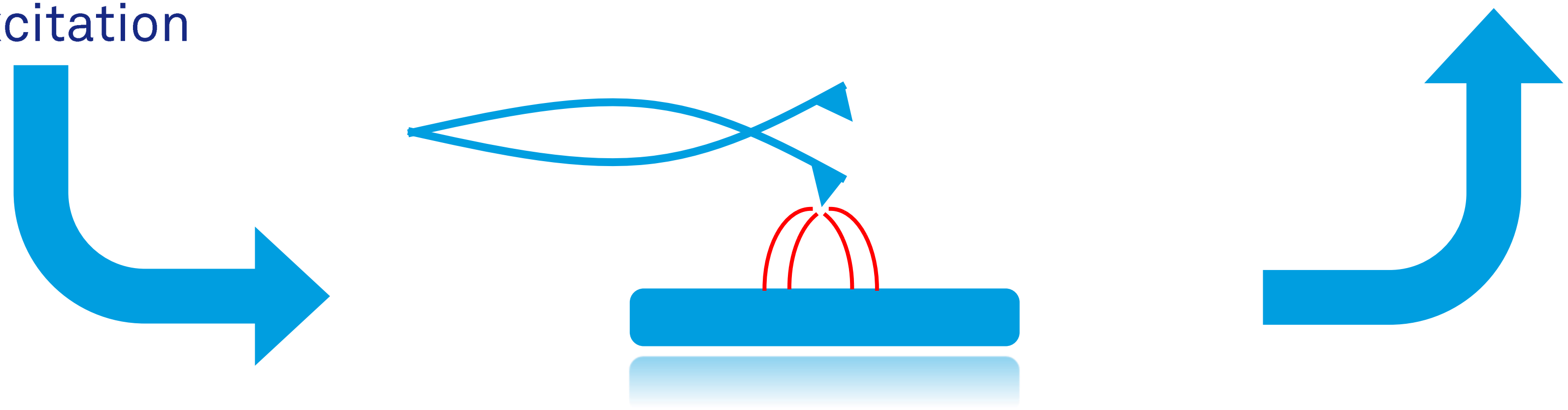
- Mechanical
- Electrical
- Magnetic
- Optical

Observables

- Amplitude
- Phase
- Δf_0
- Dissipation

Multi-frequency Excitation

Total force



Probing multiple force contributions

Actuation and demodulation schemes in frequency domain

Actuations

- Mechanical
- Electrical
- Magnetic
- Optical

Reference

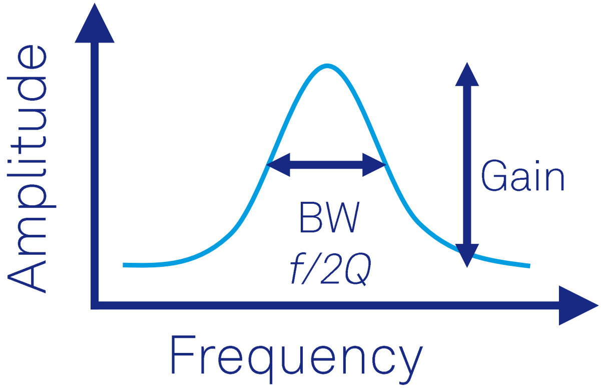
Observables

- Amplitude
- Phase
- Δf_0
- Dissipation

Physical response

- Mechanical response
- Electrical response
- Magnetic response
- Optical response

Multi-frequency Excitation



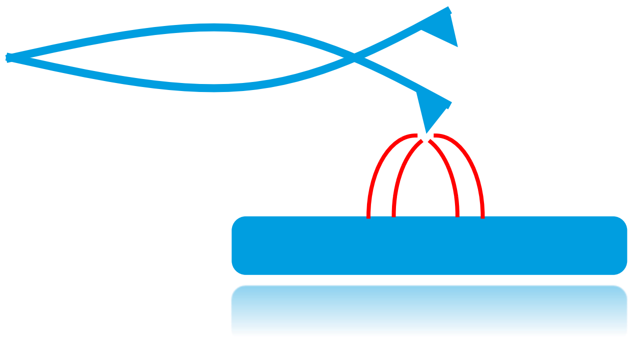
Total force

Single-pass resonance technique

Distinguish between mechanical and electrical contributions

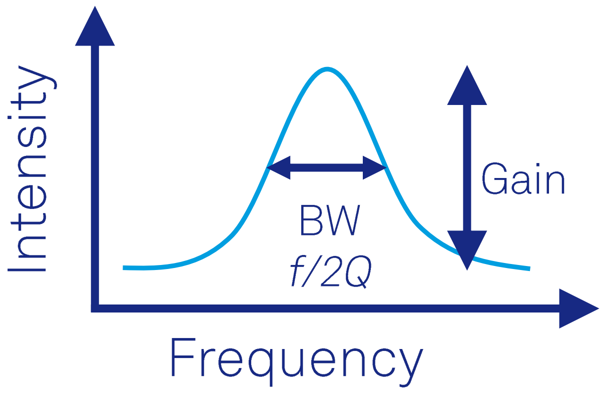
Single-pass technique offers

- Better lateral resolution
  (no tip lift, no long-range blurring effect)
- Faster scan
  (don’t pass twice on the same location)
- Decouple mechanical and electrostatic contribution



Resonance technique offers

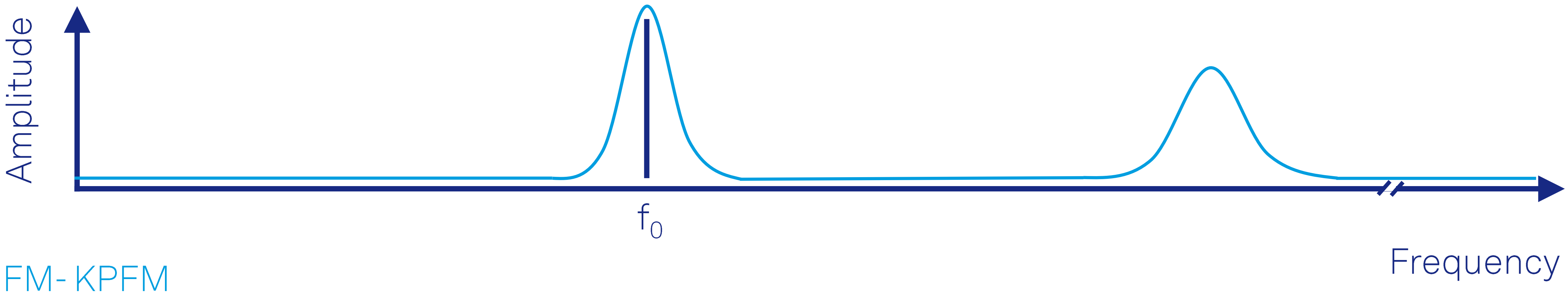
- Amplification factor thanks to resonance enhancement
- Known bandwidth
  (noise rejection)
- Orthogonal information
  (X, Y components in quadrature)



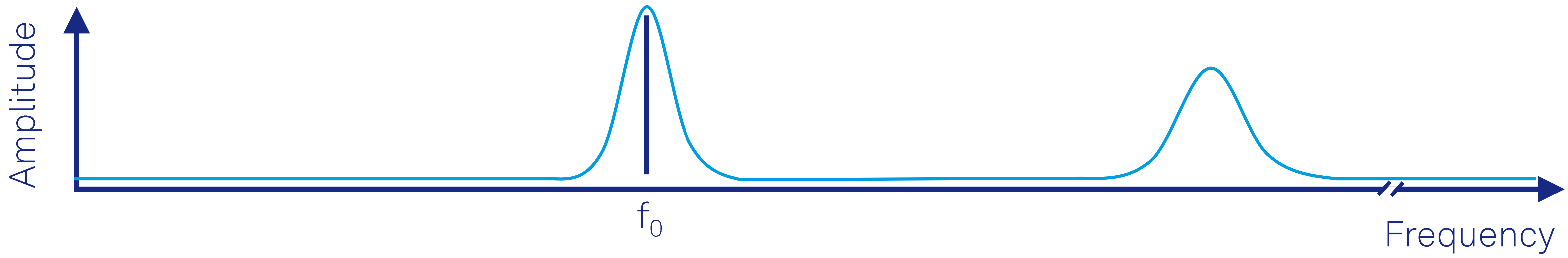
Single-pass resonance technique

Distinguish between mechanical and electrical contributions

AM-KPFM



FM- KPFM

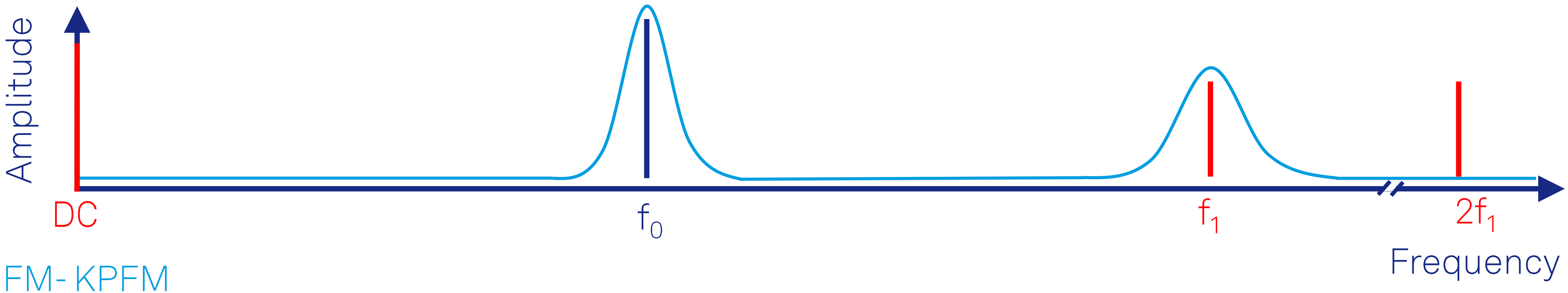


Single-pass resonance technique

Distinguish between mechanical and electrical contributions

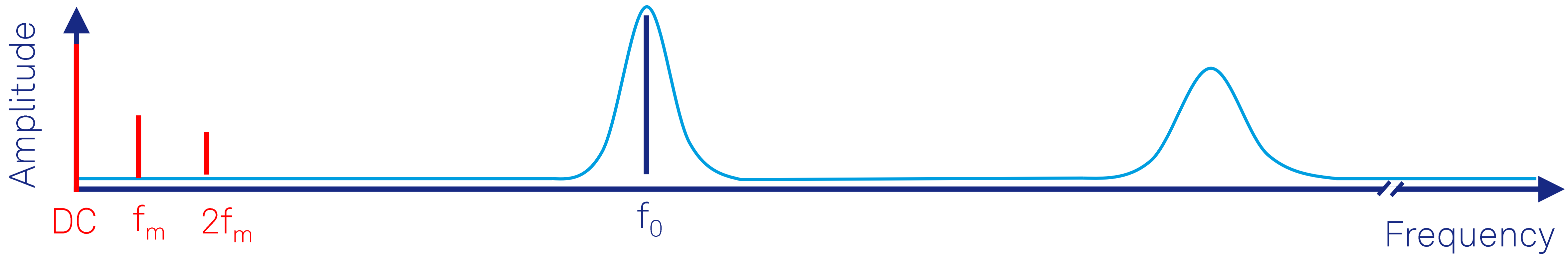
AM-KPFM

→ Amplitude sensitive technique



FM- KPFM

→ Phase sensitive technique

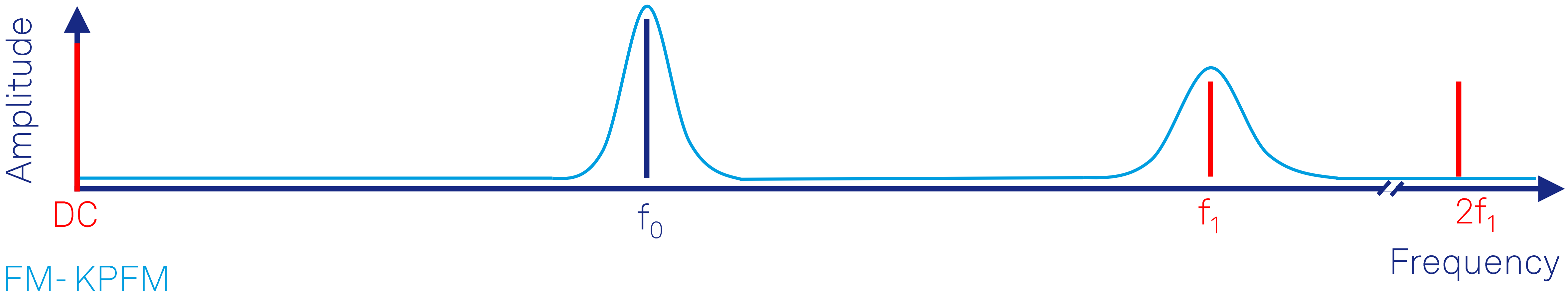


Single-pass resonance technique

Distinguish between mechanical and electrical contributions

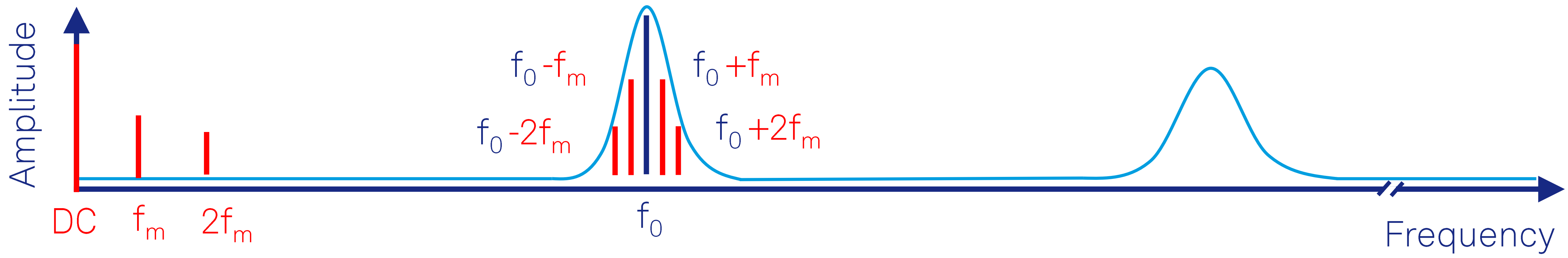
AM-KPFM

→ Amplitude sensitive technique



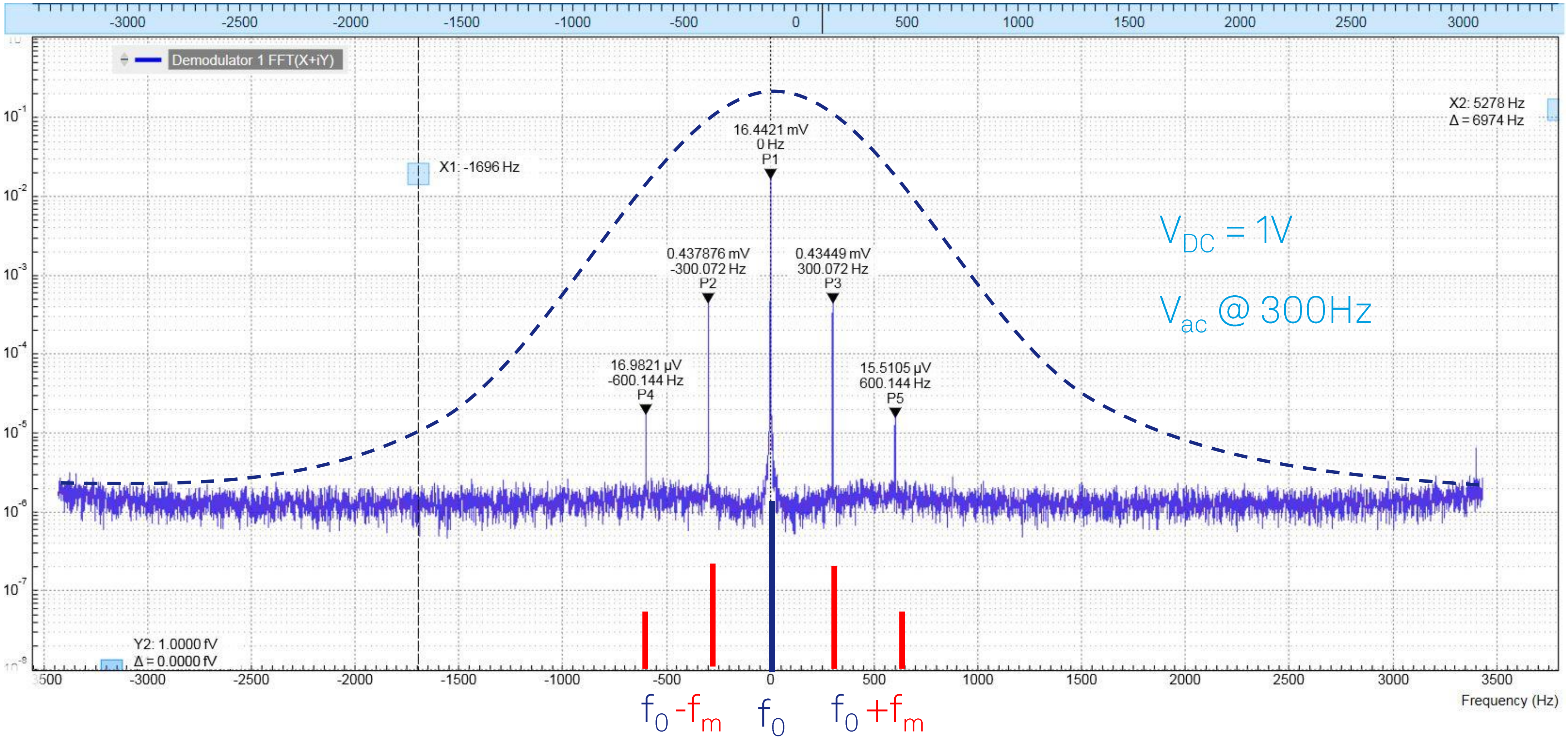
FM- KPFM

→ Phase sensitive technique



Electrical frequency mixing of mechanical resonator

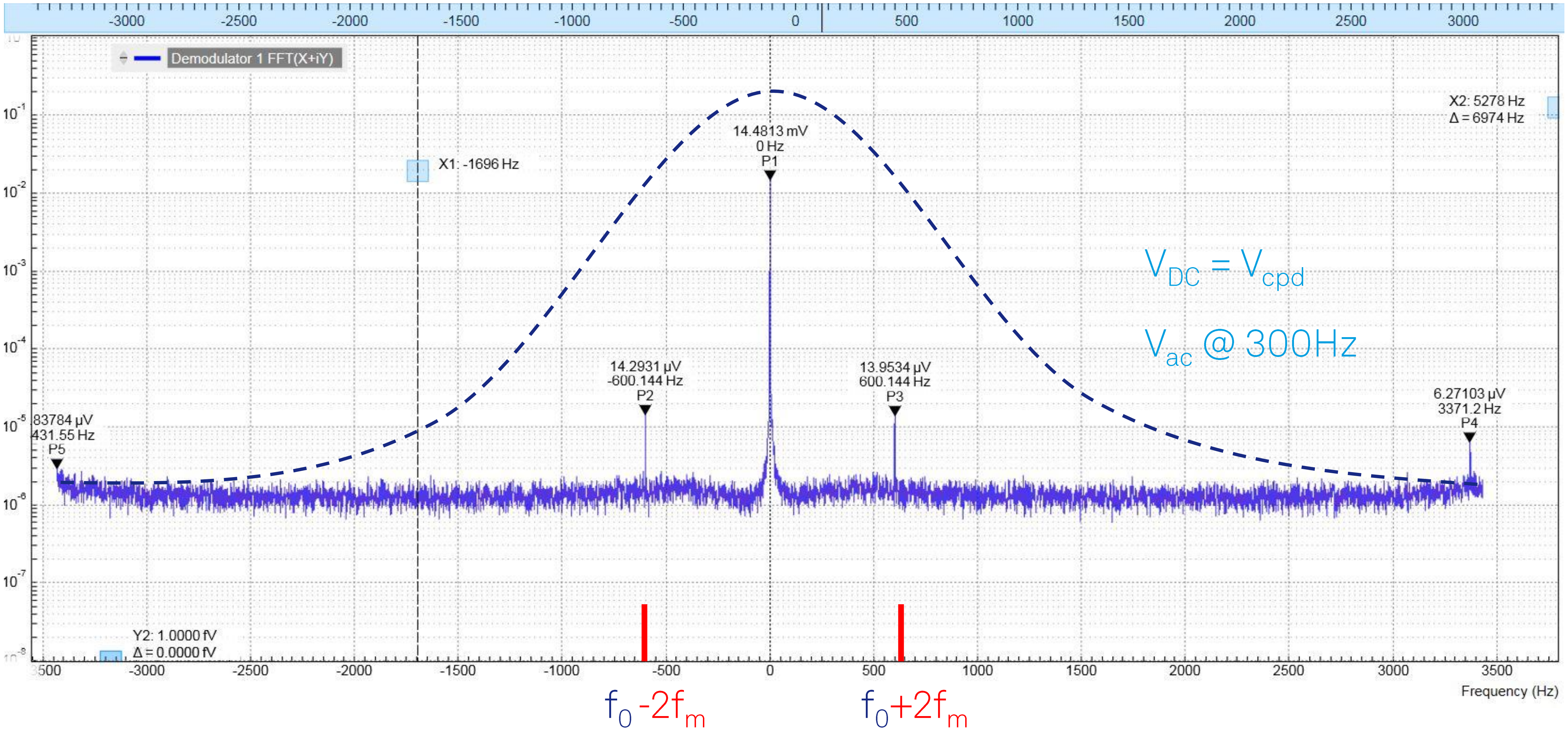
Open-loop spectrum



Electrical frequency mixing of mechanical resonator

Closed-loop spectrum

→ 2ω component not affected by V_{DC}

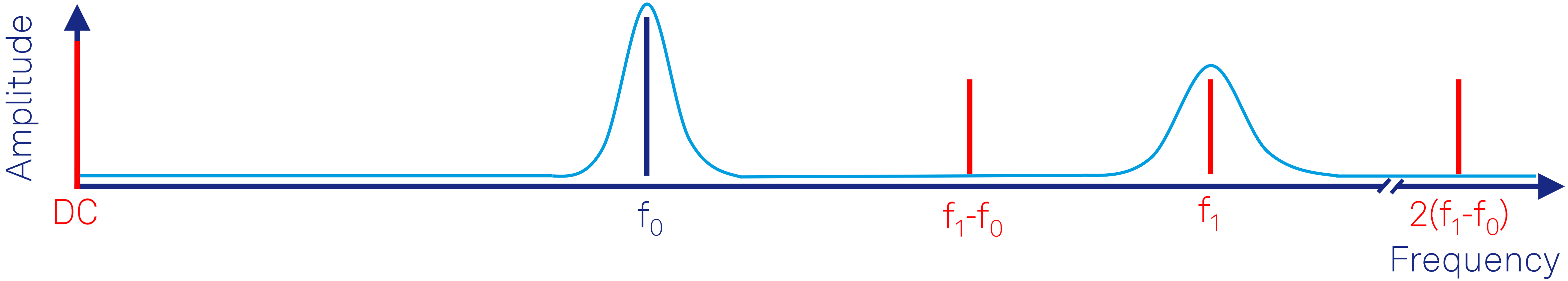


Single-pass resonance technique

Heterodyne FM-KPFM

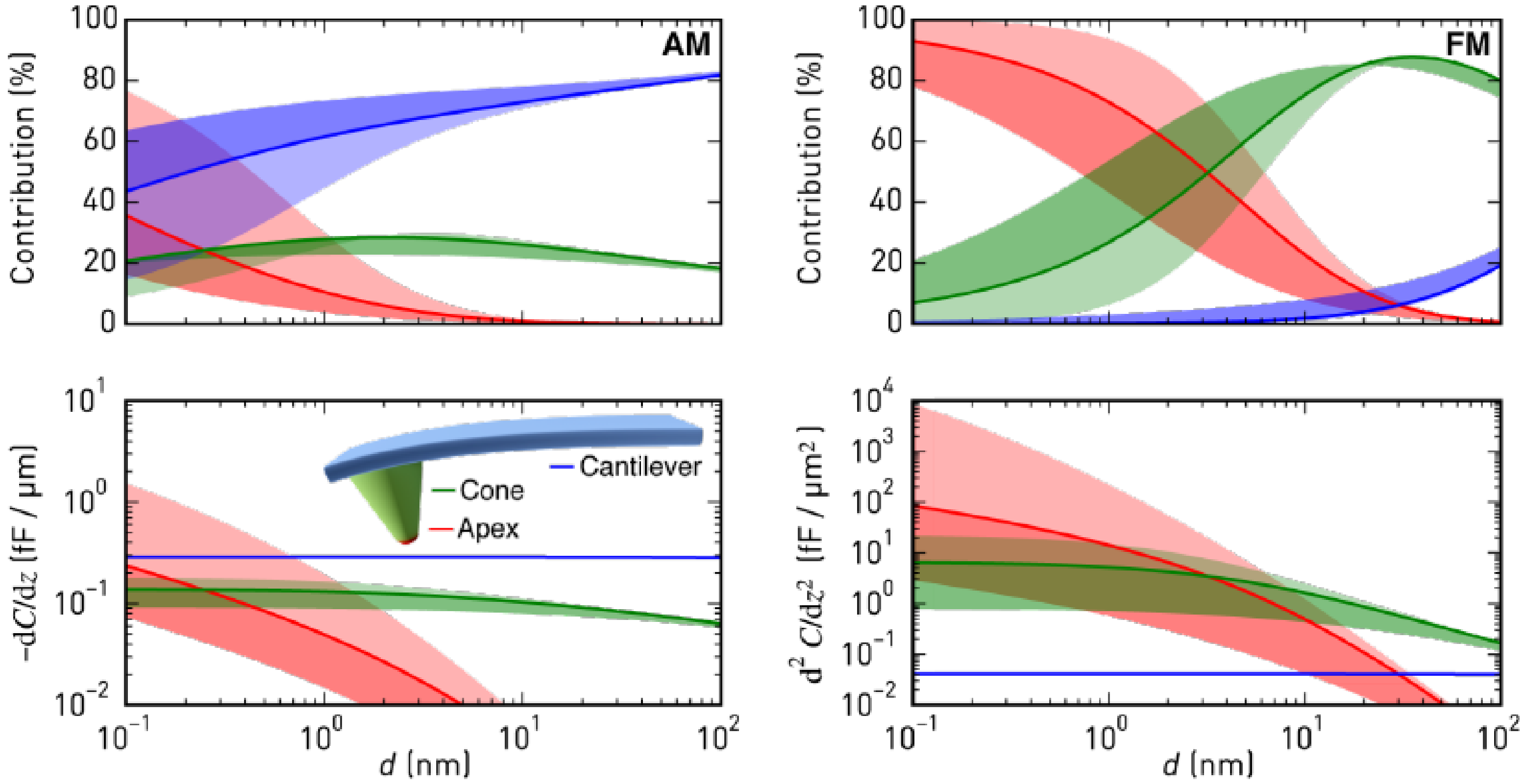
→ The new right sideband (f_0+f_m) is now conveniently located at f_1

→ Good trade-off between high modulation frequency, still measured at resonance, and phase sensitivity of the electrical measurements



Axt, A. et at, Beilstein J. Nanotechnol. 2018, 9, 1809–1819

AM vs FM techniques
Be sensitive to the force or the force gradient



$$F_{el} = -\frac{1}{2}\frac{\partial C}{\partial z}(V_{DC} - V_{CPD})^2$$

$$\frac{\partial F_{el}}{\partial z} = -\frac{1}{2}\frac{\partial^2 C}{\partial z^2}(V_{DC} - V_{CPD})^2$$

T. Wagner et al., Beilstein J. Nanotechnol. **2015**, 6, 2193–2206

How KPFM works

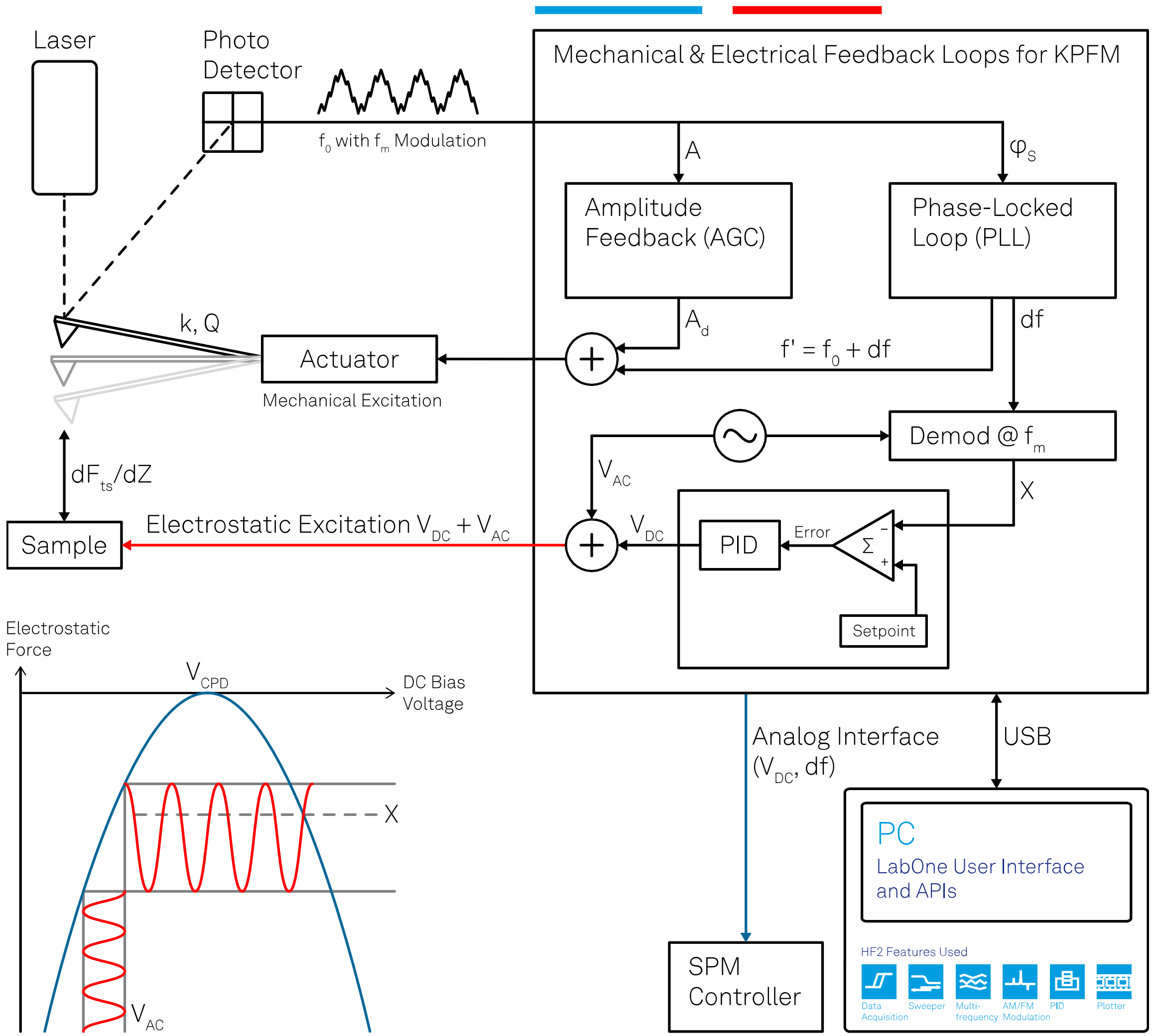
Electro-mechanical mixing

Mechanical loop

- Total force is sensed through the detuning of the mechanical resonator
- Feedback Tapping mode or Non-Contact AFM both) controls mechanical motion
- → In FM mode, phase (or freq) detuning is used as an input to the electric loop

Electrical loop

- Small AC bias voltage added to static Tip-Sample voltage
- Sensed electrostatic force is measured by lock-in technique (X-component)
- → Feedback on DC voltage compensates the measured electrostatic contribution to the total force



Overview of closed-loop KPFM modes

Single-pass (topography & CPD simultaneously)

	Amplitude Modulation (AM)		Frequency Modulation (FM)		
Sensitive to	Force (through amplitude)		Force gradient (through phase)		
KPFM mode	AM-KPFM	1ωD-KPFM (dissipation)	FM-KPFM (sidebands)	2ωD-KPFM (dissipation)	Heterodyne FM-KPFM
Mechanical drive	f_0	f_0 (NC-AFM)	f_0	f_0 (NC–AFM)	f_0
Electrical drive	f_1 or off-resonance	f_0 90° phase shift to mechanical drive	f_m (few kHz)	$2f_0$ 90° phase shift to mechanical drive	$f_1 - f_0$
Electrical detection	X-component at f_1	Dissipation channel	X-component at $f_0 \pm f_m$	Dissipation channel	X-component at f_1
Setpoint	Nullify X_1	Dissipation with no bias	Nullify $X_3 - X_2$	Dissipation with no bias	Nullify X_1
Comments	$V_{AC} < 1$ V	Requires PLL+AGC	V_{AC} drive ~ 2V	Requires PLL+AGC	Can demodulate faster

Y. Miyahara and P. Grutter, Appl. Phys. Lett. 110, 163103 (2017)

Demo: AM-KPFM & Heterodyne-KPFM

Interfacing with the microscope

What MFLI lock-in amplifier does?

Controls the electrical actuation

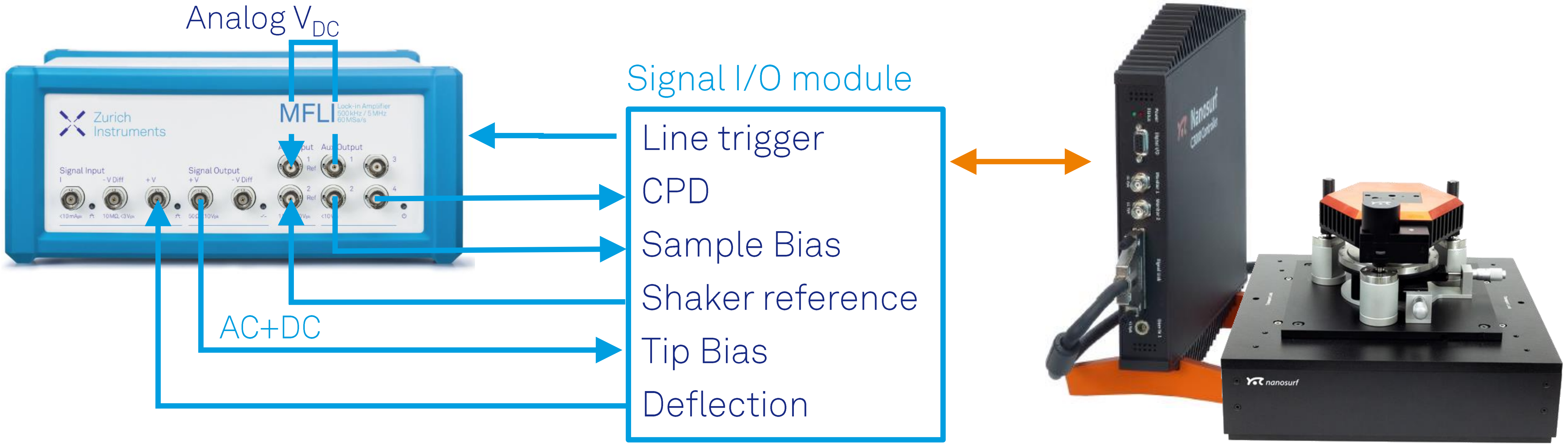
- Electrostatic-feedback
- Tip and sample bias

→ **Enable single pass KPFM**

What Nanosurf Flex AFM does?

Controls the mechanical actuation

- Z-feedback
- Topography



Demo: AM-KPFM & Heterodyne-KPFM

Tip & sample

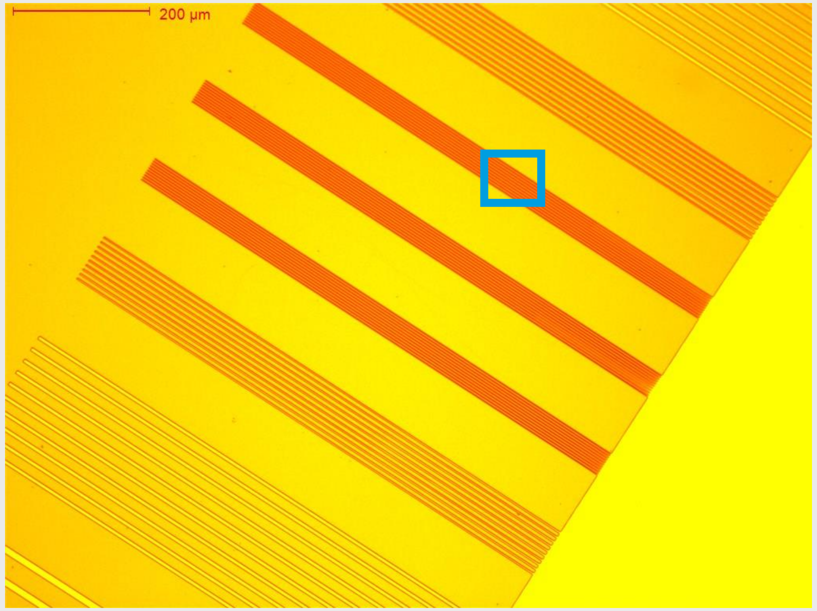
Budget Sensors KPFM grid

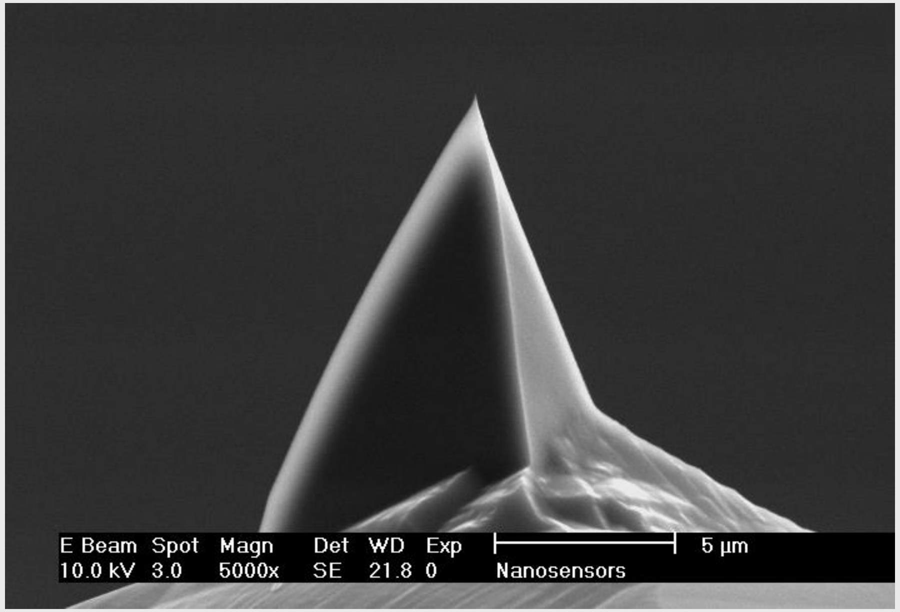
- Al & Au interdigitated line array
- Sample bias through contact pads
- Commercially available calibration standard

→ Better suited for AM-KPFM

Nanosensors PPP-EFM

- Good conductivity (full coated or doped)
- High force sensitivity (<3 N/m)
- Low tip wear with ideally high-aspect ratio

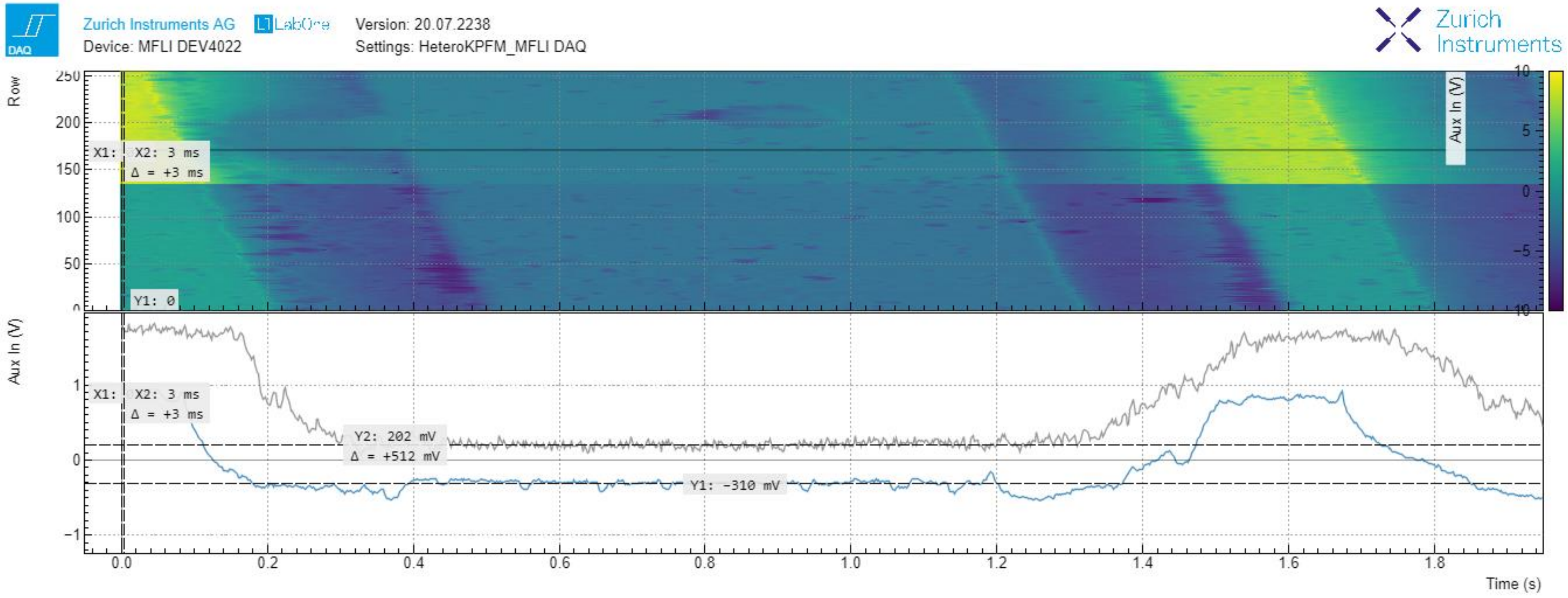




Part 1: Kelvin Probe Force Microscopy (KPFM)

What we leaned so far?

- Optimization process for AM-KPFM and Heterodyne-KPFM in the same image
- Direct mode comparison for 1V DC bias offset on Au electrode
- AM-KPFM works well for large surface inspection, H-KPFM is more quantitative



Part 2: Time-resolved AFM methods

Probing the transients on a surface

Why time-resolved?

- Scanning microscopy: Spatial resolution (~nm)
- Time-resolved: Temporal resolution (~ns)

Pump-probe technique

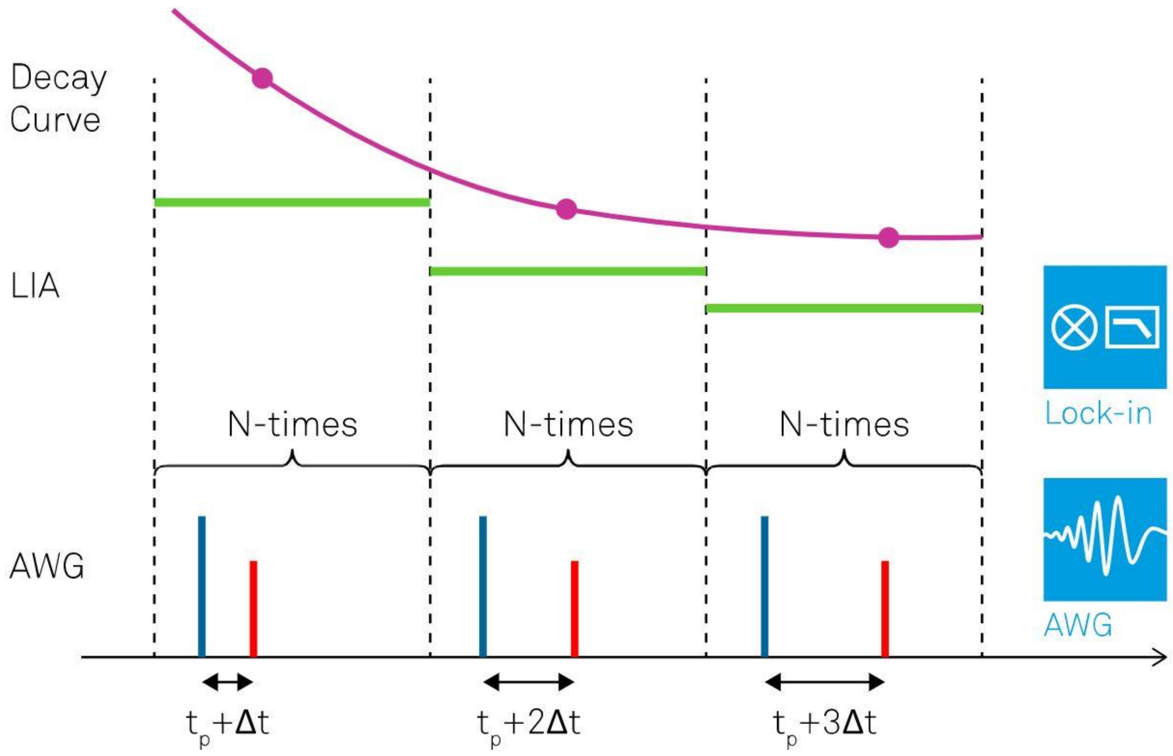
- Pump: Excite the sample at a given time
- Probe: Read out the sample state with a delay

Signal generation by AWG

- 2-channel pulse train with adjustable delay
- Modulation of pump or probe

Measurement by lock-in amplifier

- Lock to frequency and demodulate the signal
- Sweep the delay and sync the signal generation and detection



Electrical pump-probe KPFM

A typical scheme

Sample

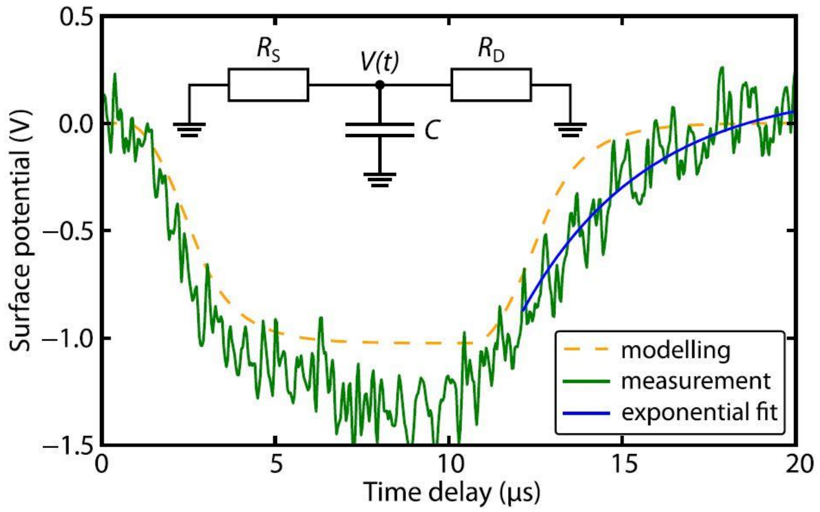
Organic FET → SP during switching

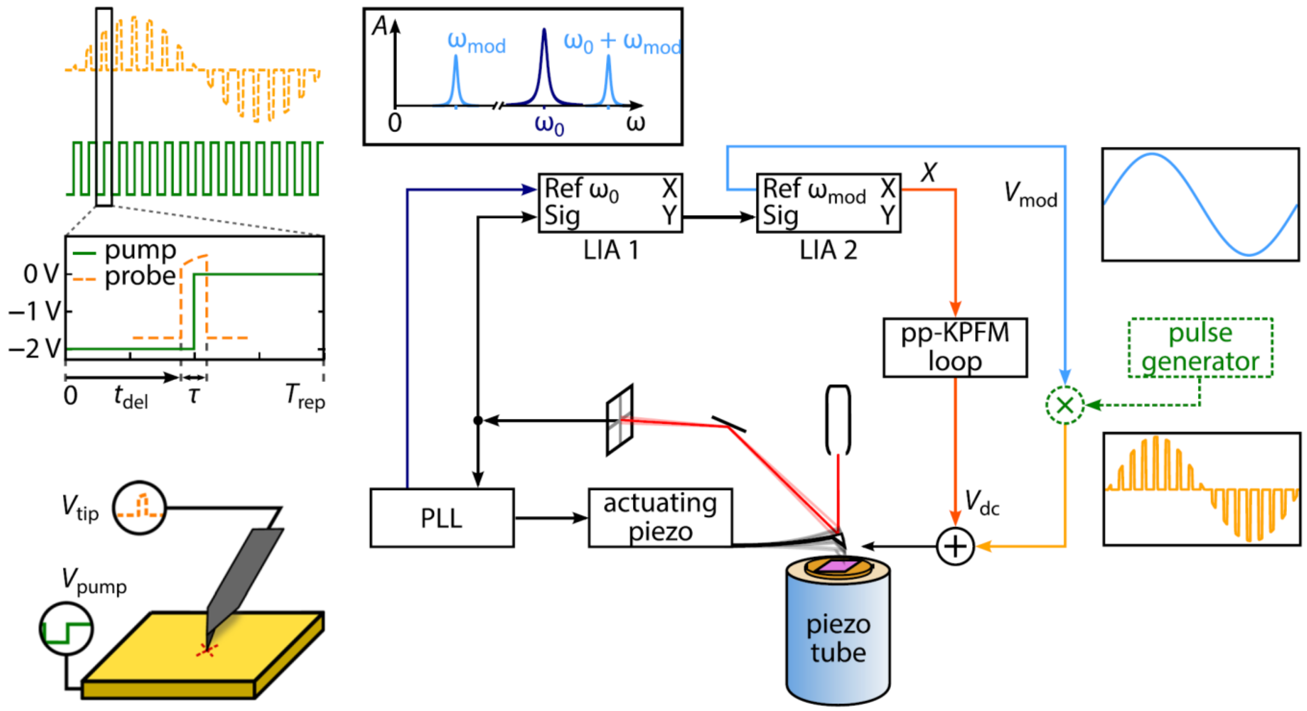
Pump

Square pulse with period 20 µs

Probe

Rectangular pulse modulated at kHz

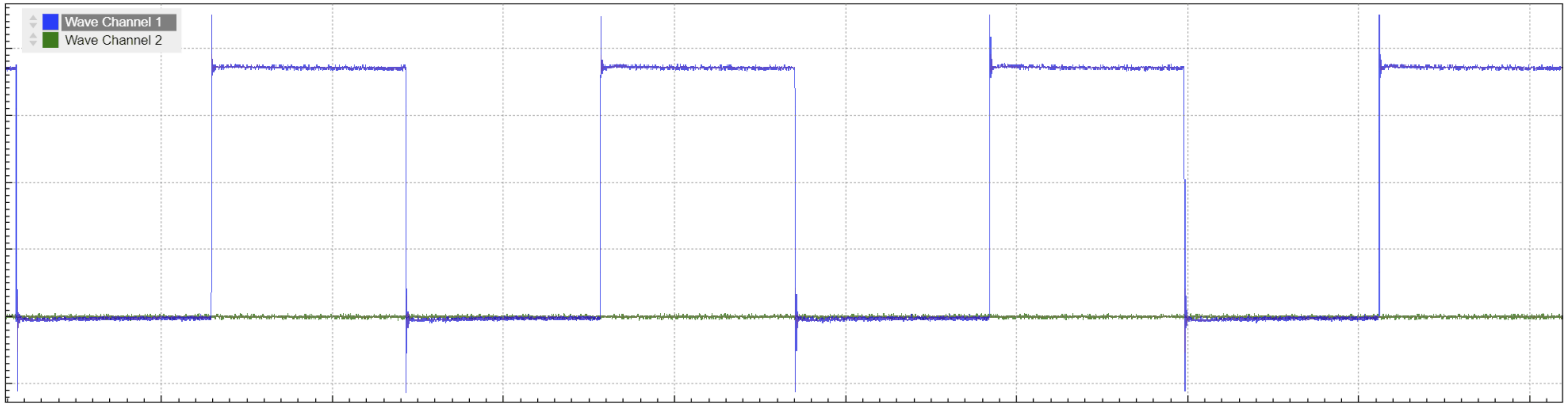




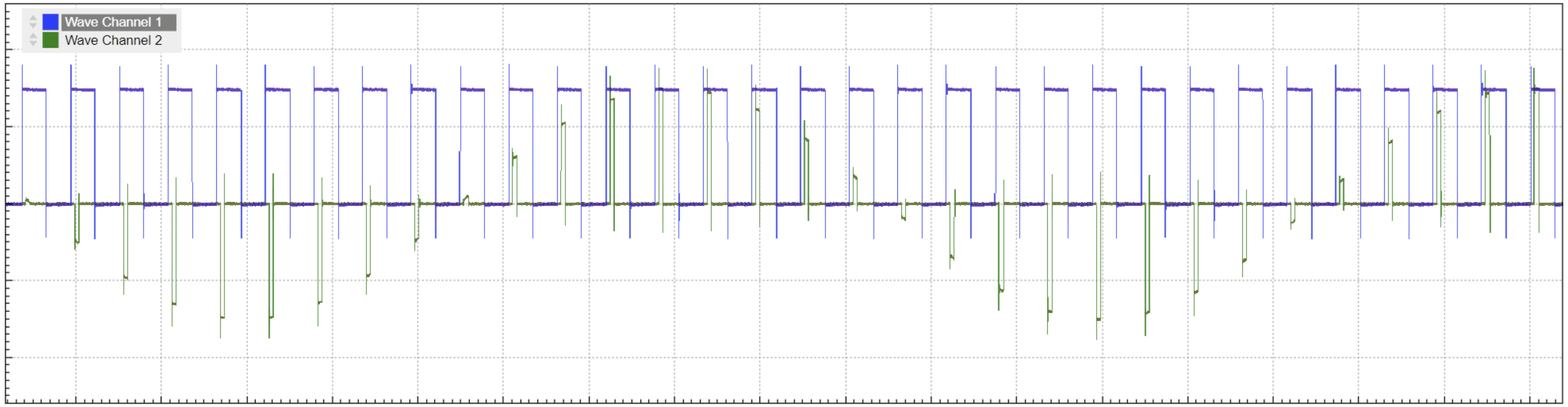
Pump-probe signal generation

AWG – Two-channel pulse trains

Sweeping the delay between pump and probe



Modulating the probe pulse train

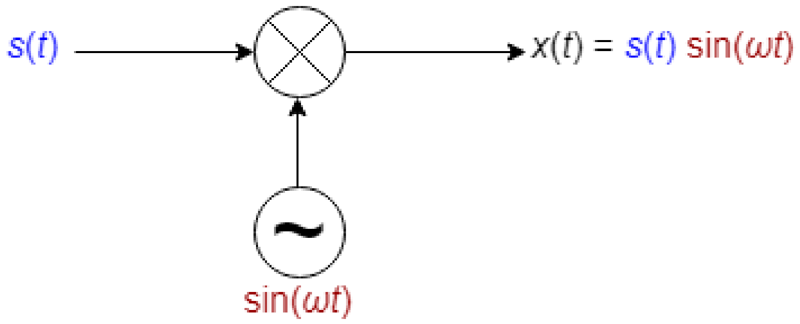


Pump-probe signal generation

AWG – Amplitude modulation

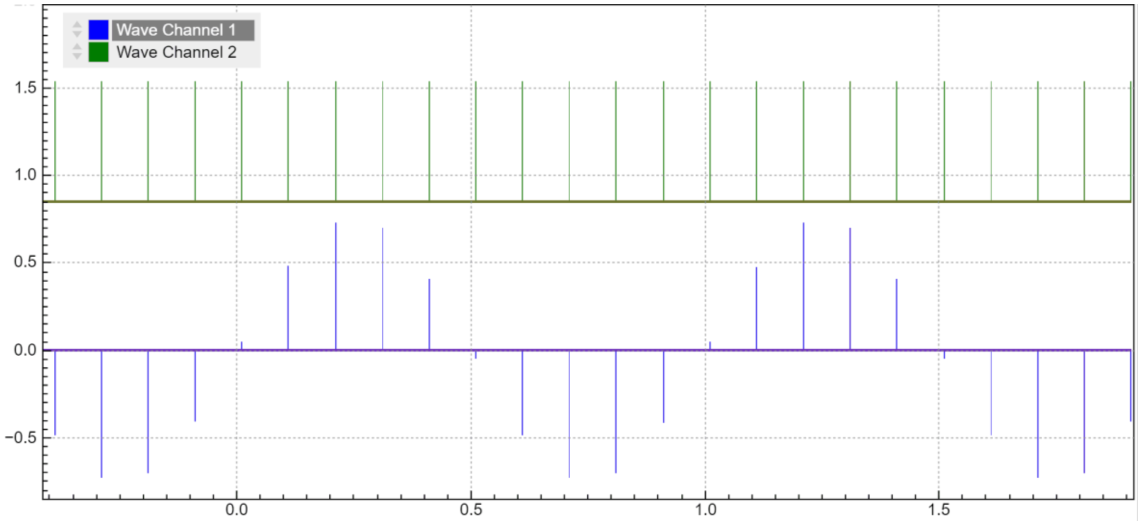
How does it work?

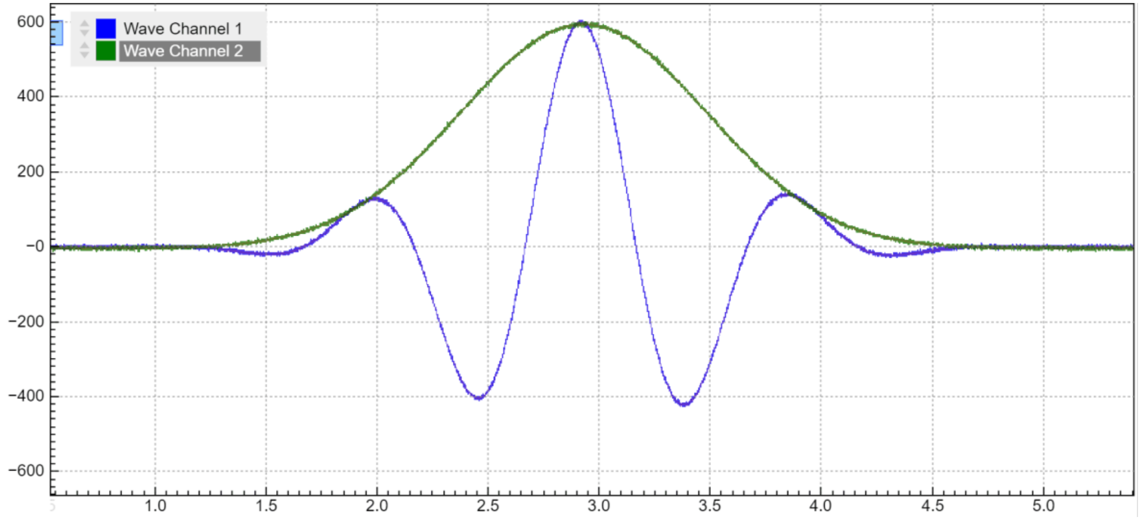
1. Waveform $s(t)$
2. Signal $\sin(\omega t)$
3. Modulation $s(t)\sin(\omega t)$



Independent control over

- Pulse width, repetition rate
- Carrier frequency, delay and phase





Signal detection and delay control

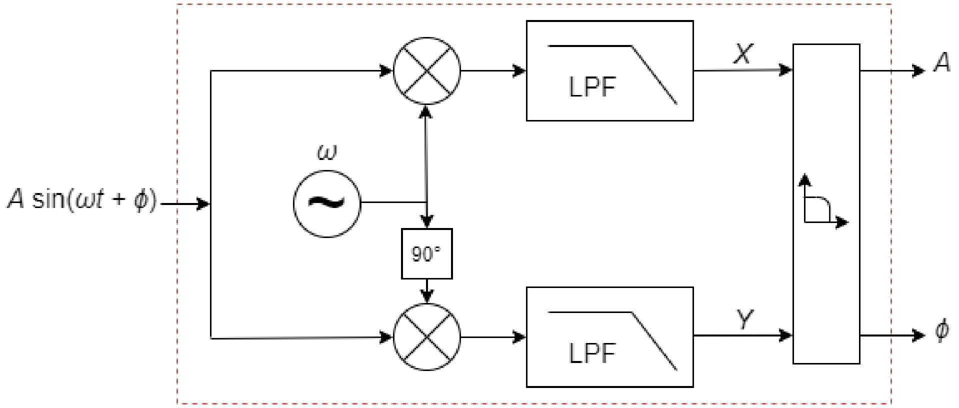
Measurement tools

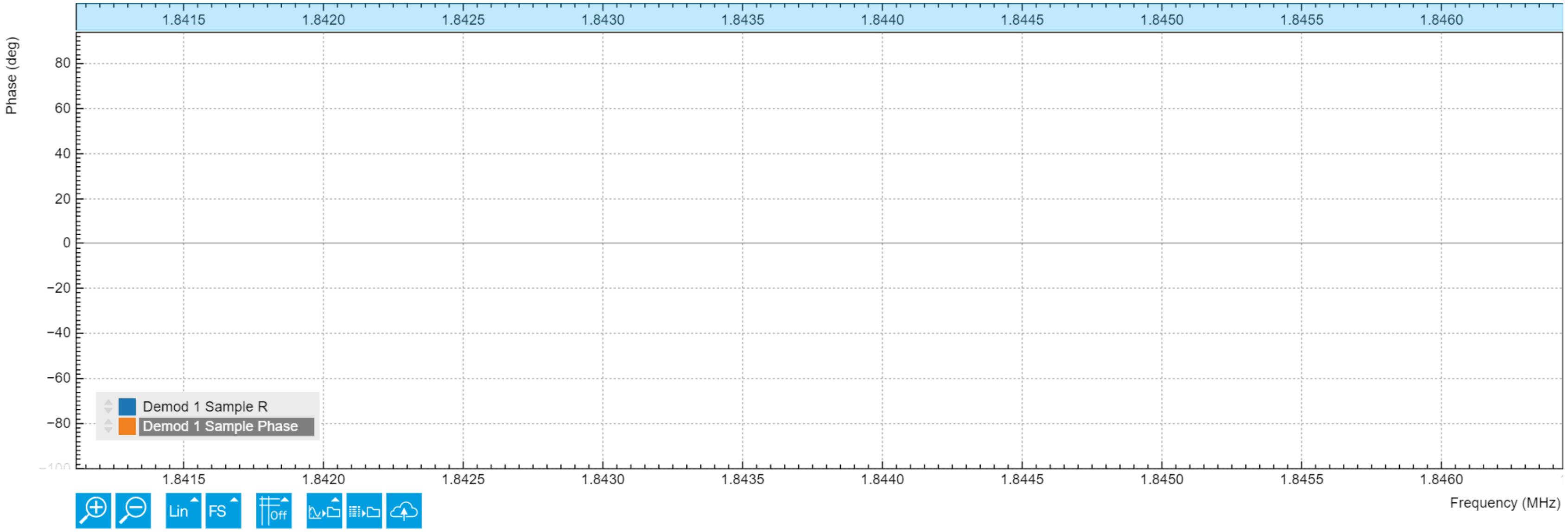
Lock-in amplifier

- Amplitude and phase
- Extremely low-noise

Sweeper

- Parametric measurement
- Synchronizing signal generation and detection





Demo: Electrical pump-probe method

Signal generation and detection

What UHFLI Lock-in Amplifier does?

- Locks to the modulation frequency
- Demodulates the signal
- Synchronizes detection and generation

What UHF-AWG does?

- Generates 2-channel pulse trains
- Adjusts the duty-cycle of pulses
- Sweeps the delay between 2 pulses

The cockpit of the instrument: LabOne

- Controls the AWG and Lock-in Amplifier
- Records and process measured data

Part 2: Time-resolved AFM methods

What we leaned so far?

- Time-resolved method provides high temporal resolution for AFM systems
- Electrical pump-probe technique can be used in KPFM
- AWGs and lock-in amplifiers are the tools to implement electrical pump-probe

→ **Lock-in and AWG in one box:** Synchronized signal generation and detection