Fast, Local, and In-situ Cure Rheology of Photo-processable Polymers using Atomic Force Microscopy

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contact: callie.higgins@nist.gov

Motivation: *Additive Manufacturing*

Rapid prototyping

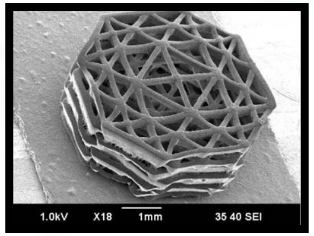
Professional

Personal

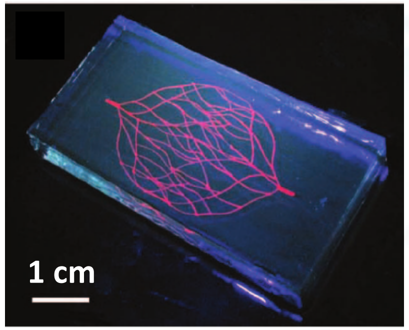
Personalized Medicine

Regenerative medicine

Bone-tissue scaffolds



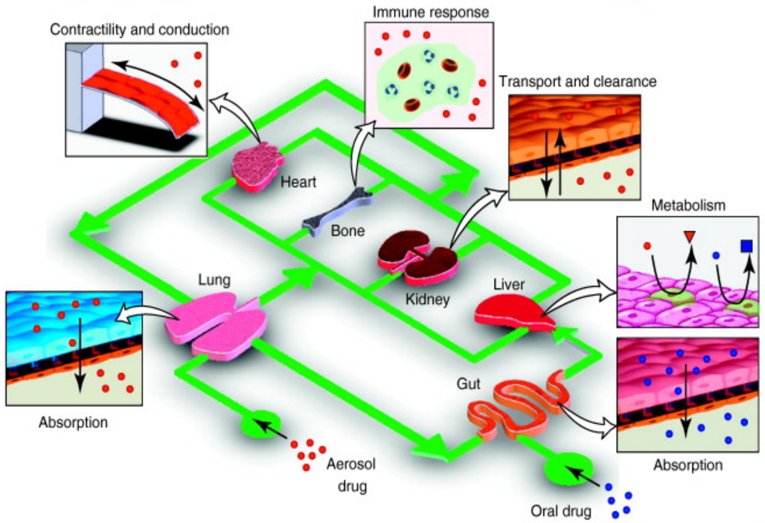
Printed vasculature



Pharmaceutical Testing

Body-on-a-chip

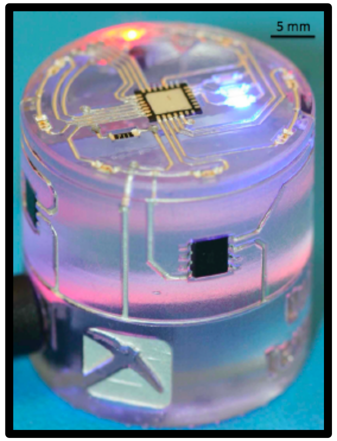
Patient-specific representative organoid structures in a microfluidic device for high-throughput drug screening



Industry

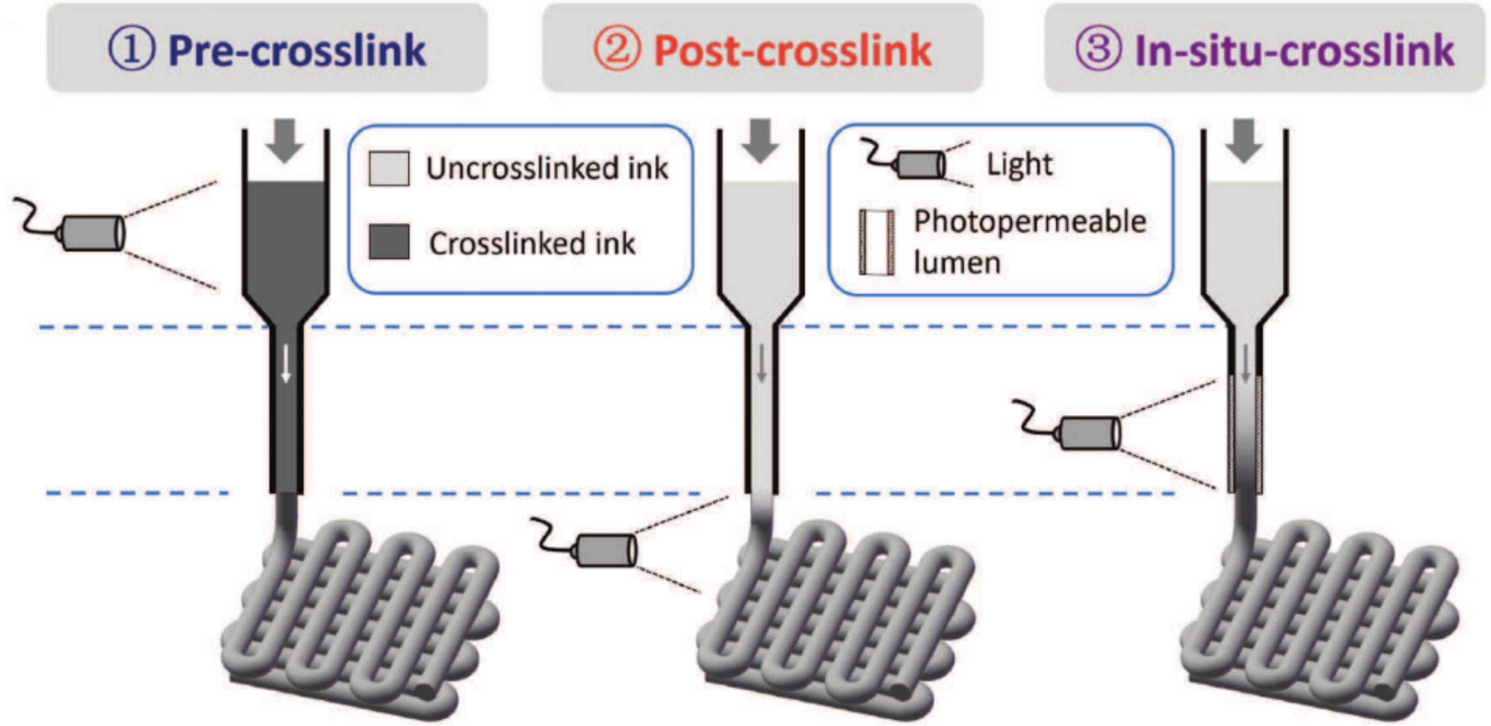
High-performance parts

Complex circuitry

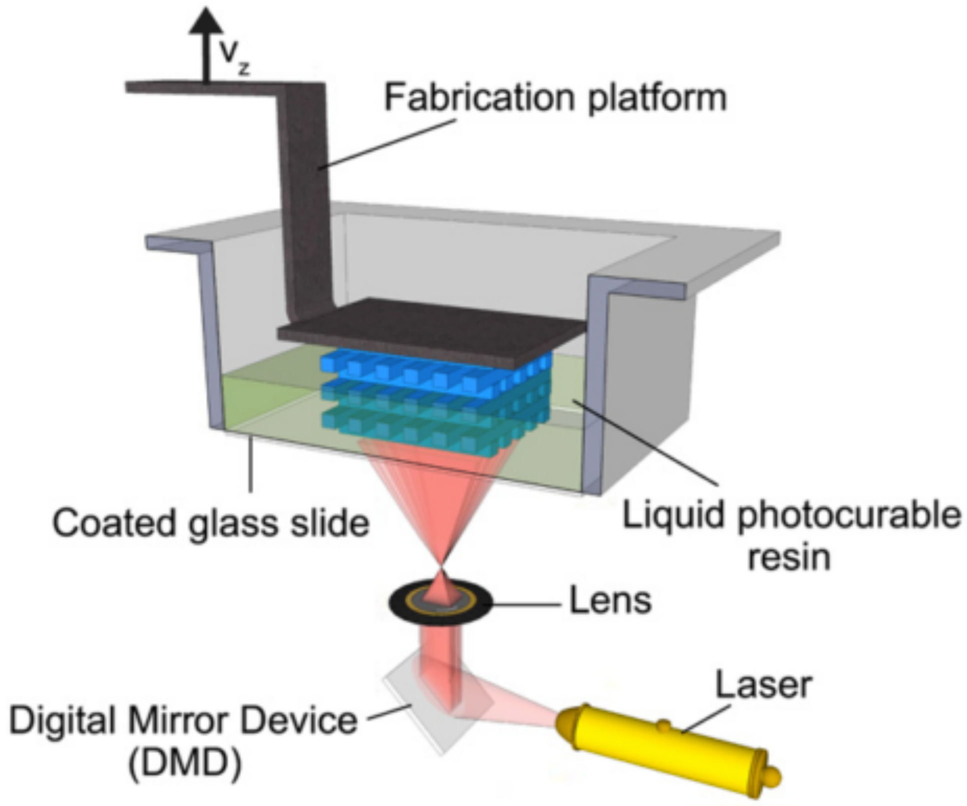


3D Printing: *Light-induced*

Nozzle-based printing



Stereolithography



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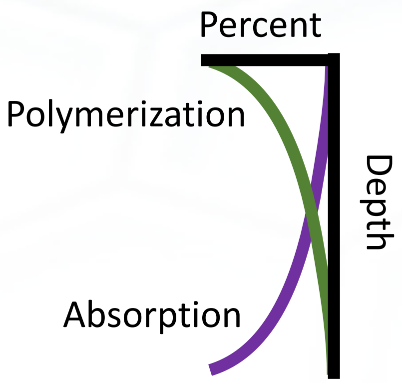

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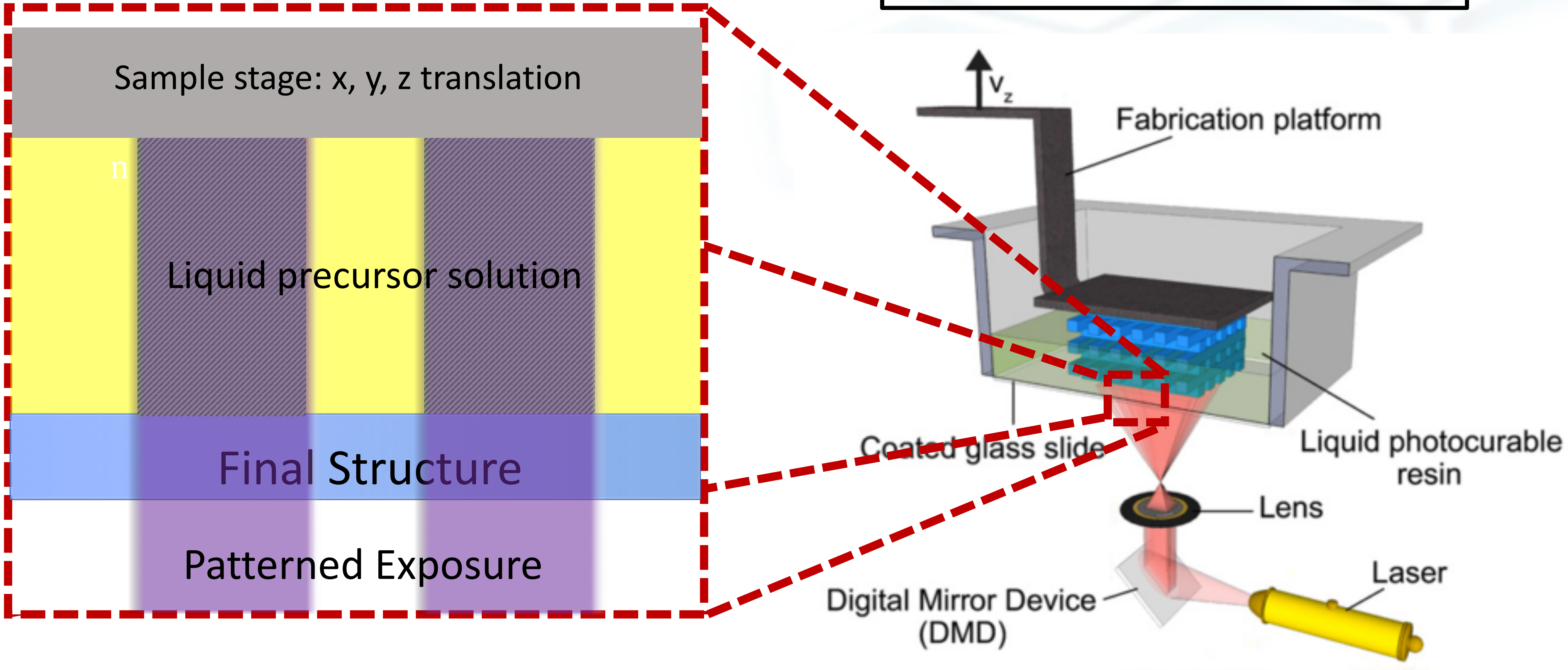
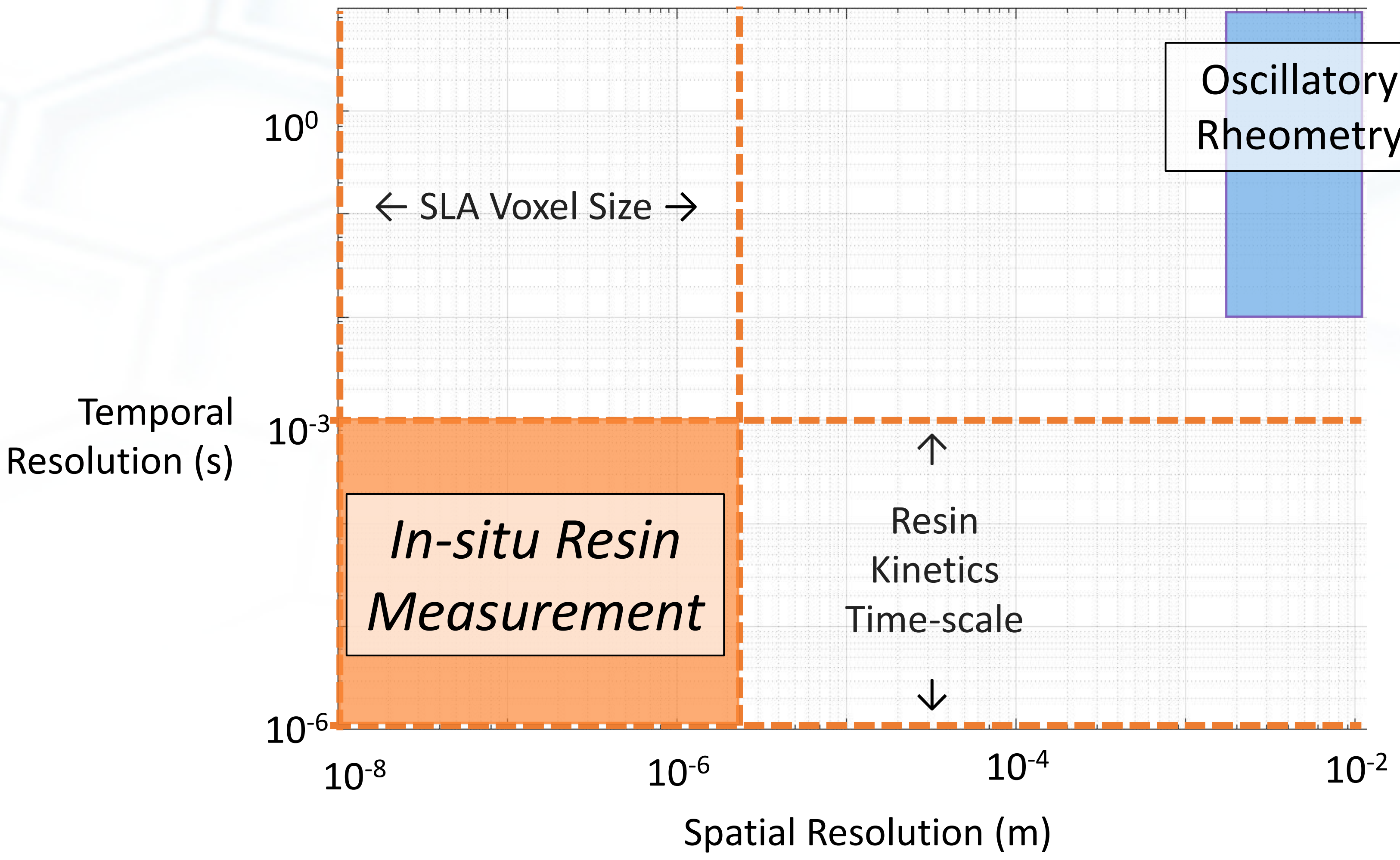
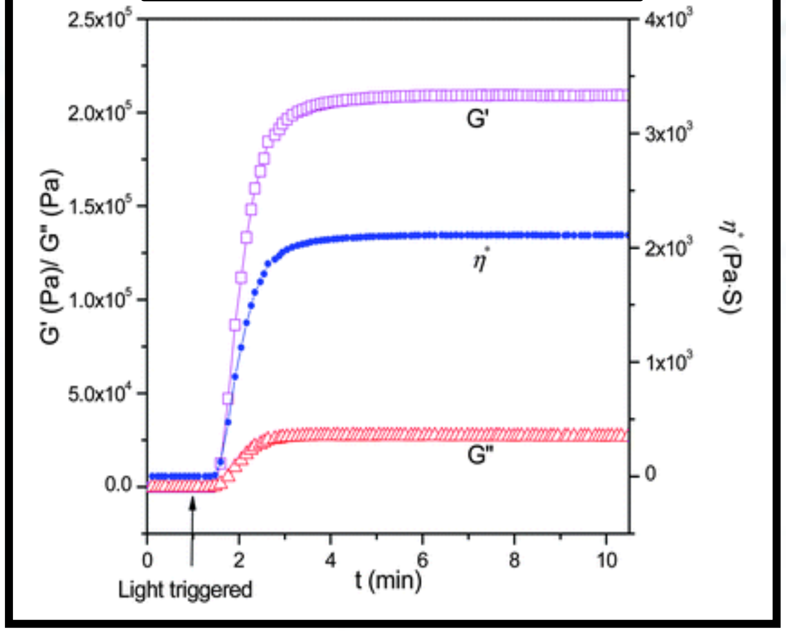


In-situ Characterization: *State-of-the-art*

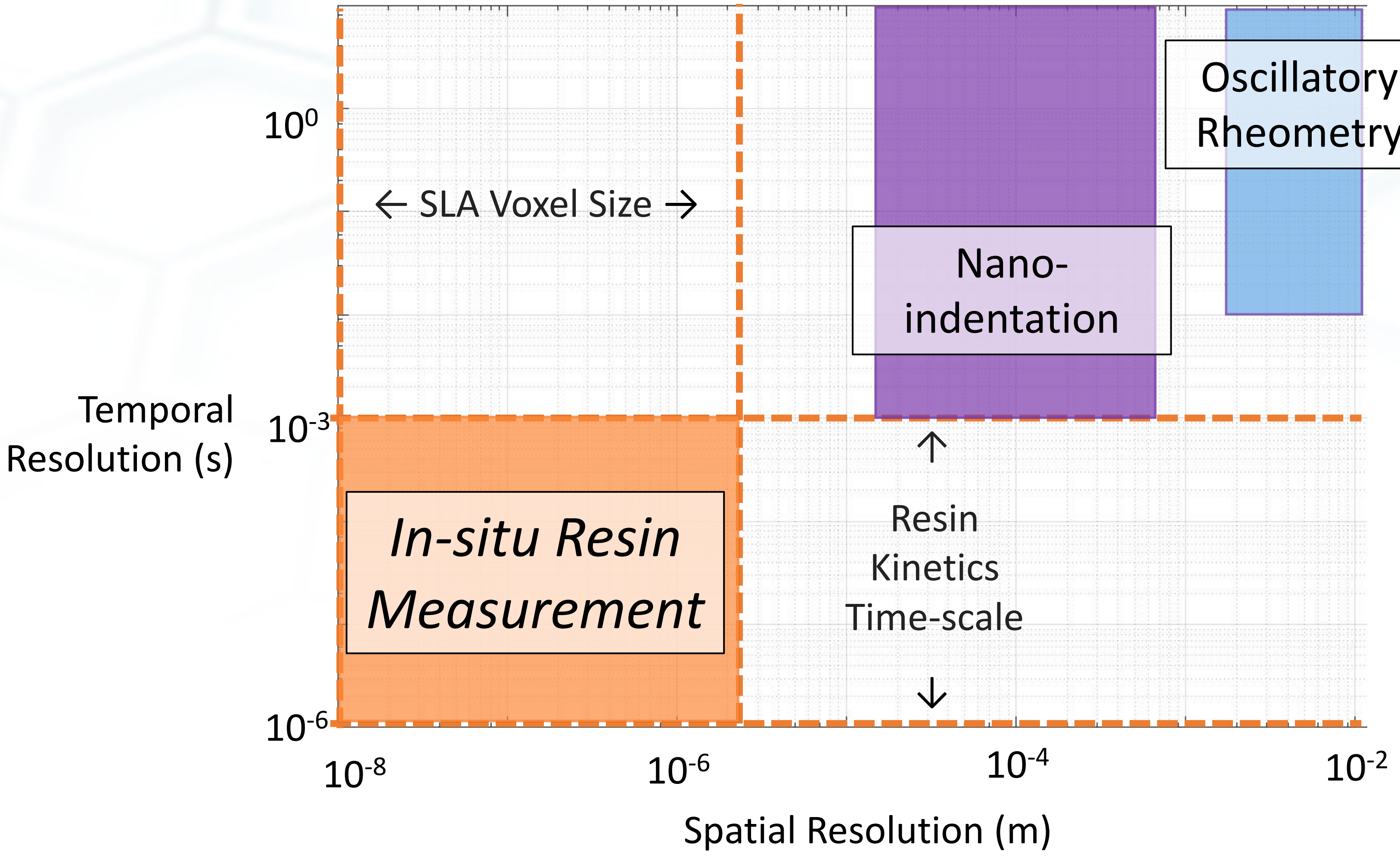


Oscillatory Rheology



Low resolution → Non-representative
Low Intensity

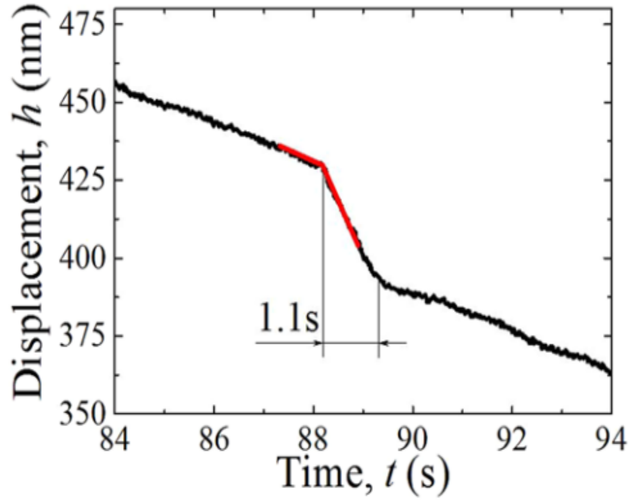
In-situ Characterization: *State-of-the-art*



Oscillatory Rheology

Low resolution → Non-representative
Large Sample Size Low Intensity

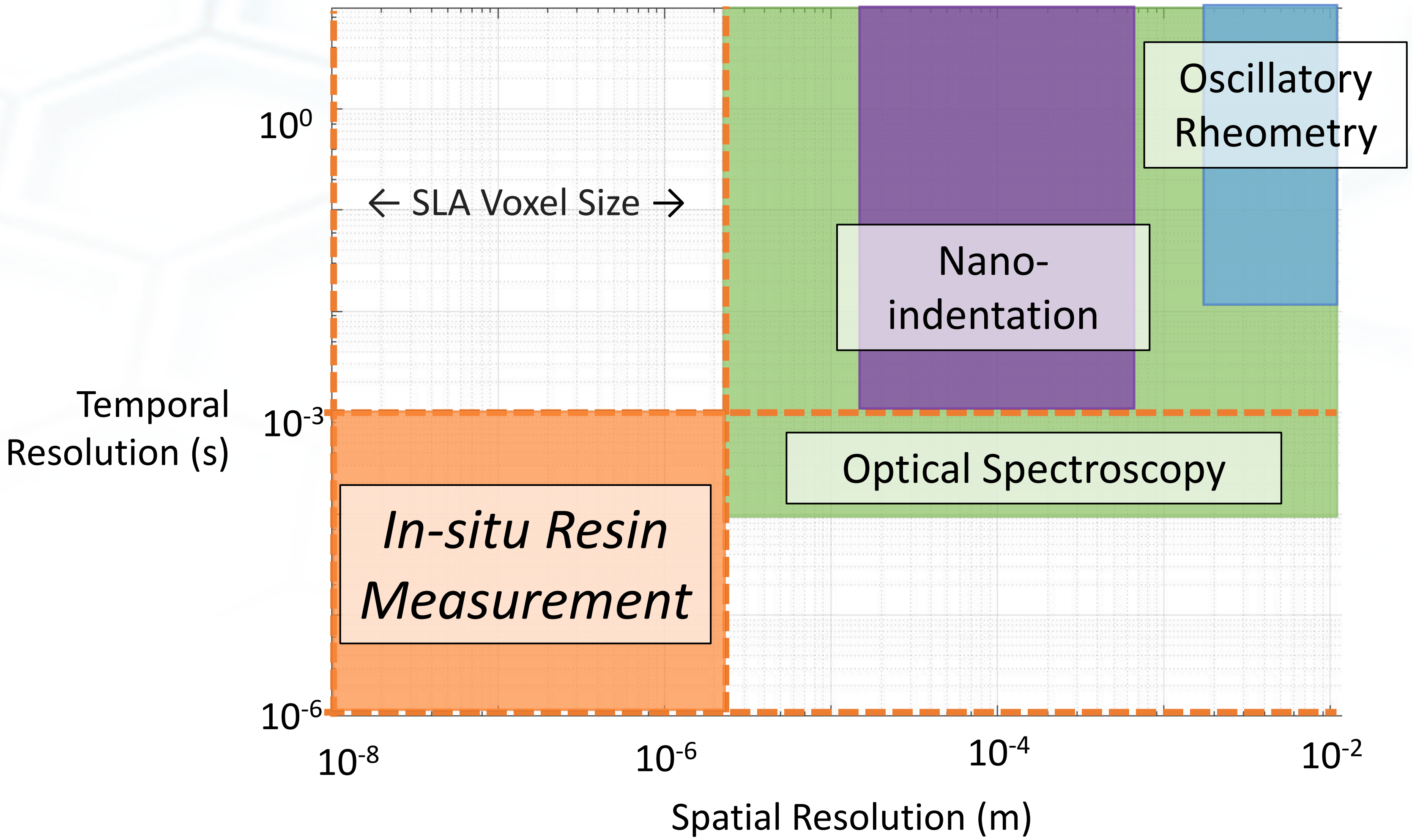
Nano-indentation



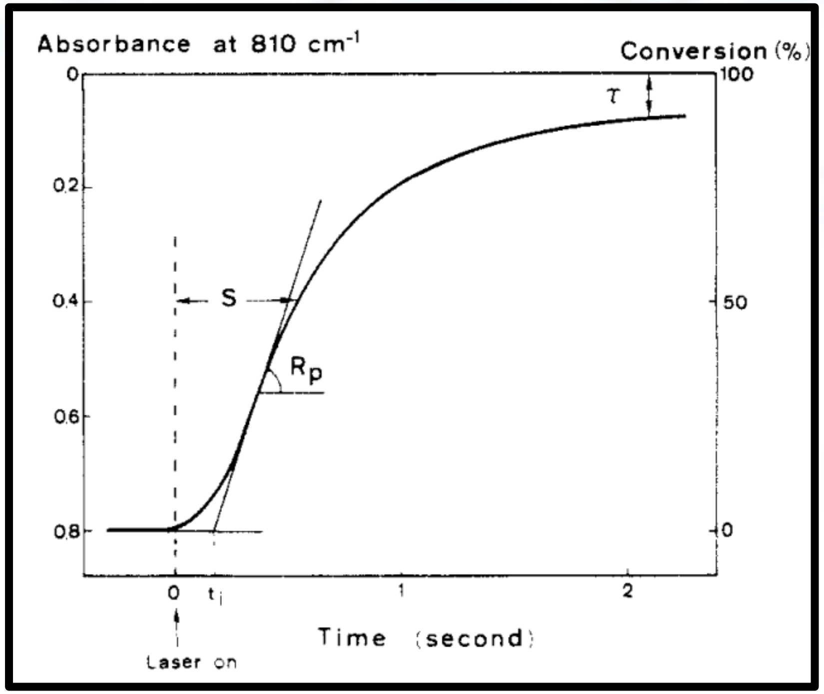
Medium Resolution - Medium Sample Size
No cure source

http://www.directindustry.com/prod/anton-paar/product-16352-1645542.html
Andrzejewska, E., & Andrzejewski, M. (1998). *Journal of Polymer Science Part A: Polymer Chemistry*, *36*(4), 665-673.

In-situ Characterization: *State-of-the-art*



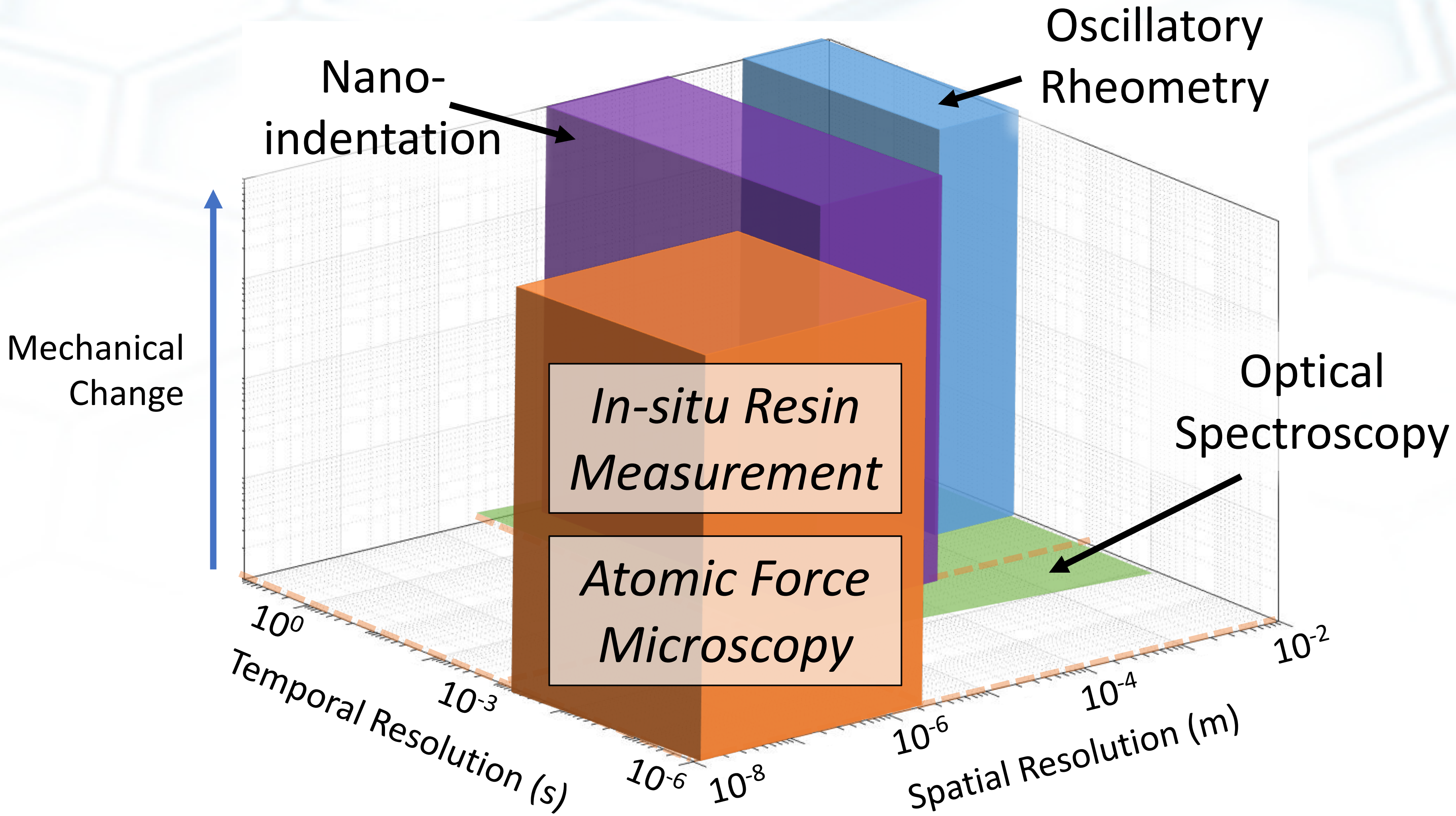
Optical Spectroscopy



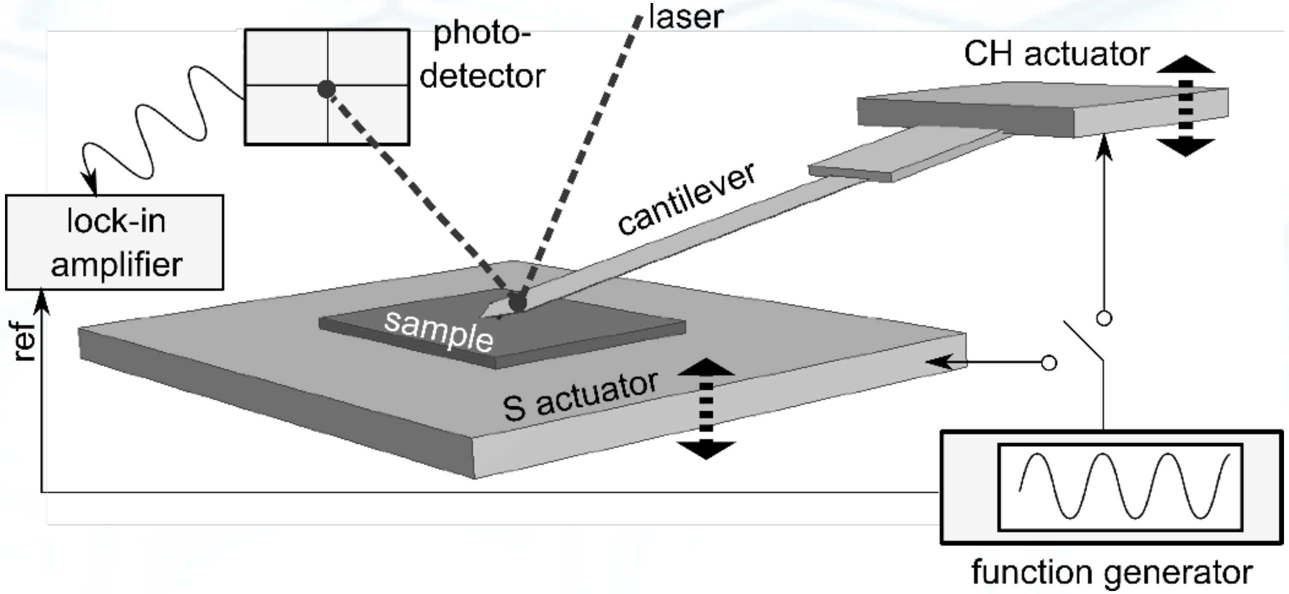
High Resolution – Small Sample Size

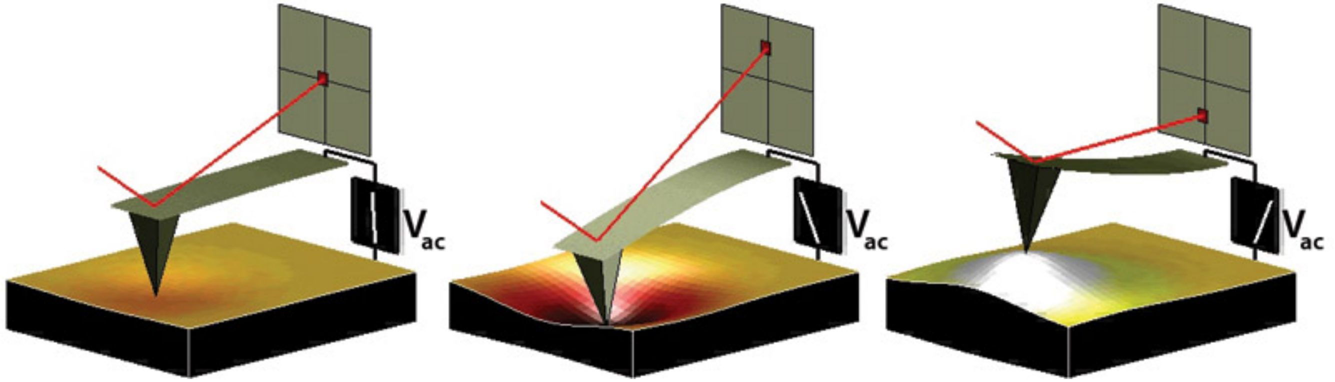
http://www.directindustry.com/prod/anton-paar/product-16352-1645542.html
Andrzejewska, E., & Andrzejewski, M. (1998). *Journal of Polymer Science Part A: Polymer Chemistry*, *36*(4), 665-673.

In-situ Characterization: *State-of-the-art*

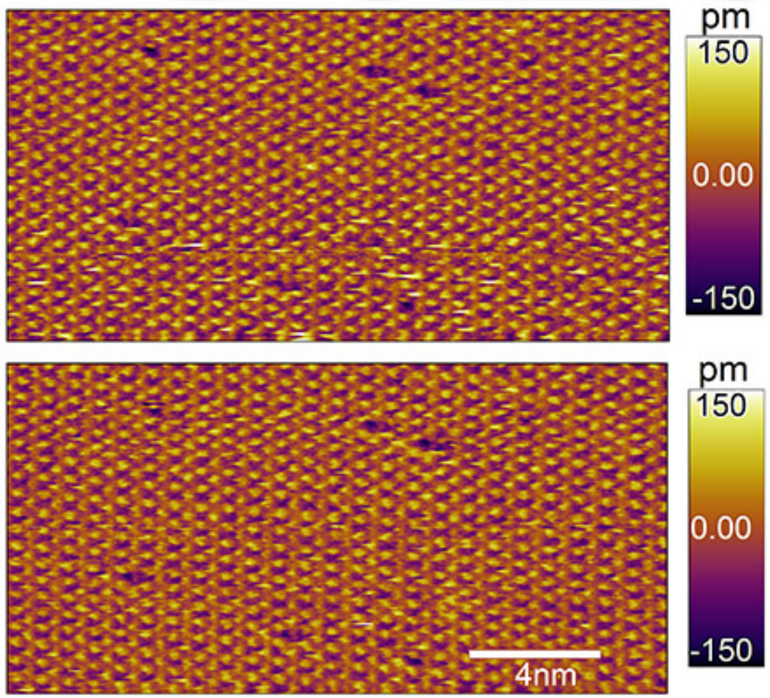


Atomic Force Microscopy





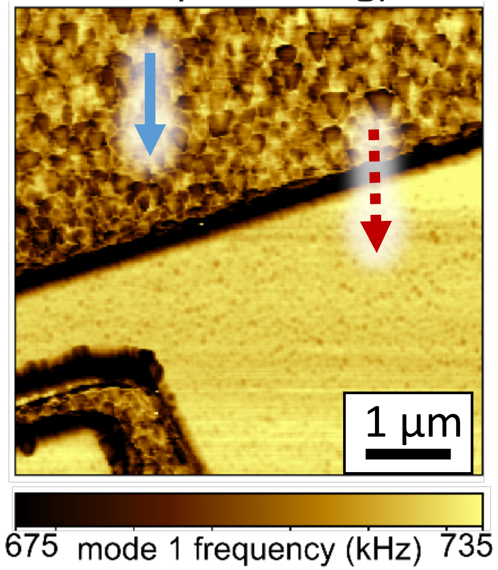
Single-atom calcite defects

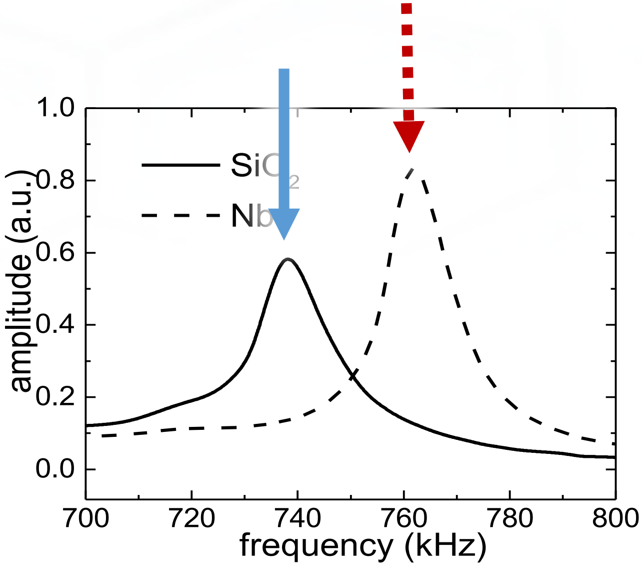


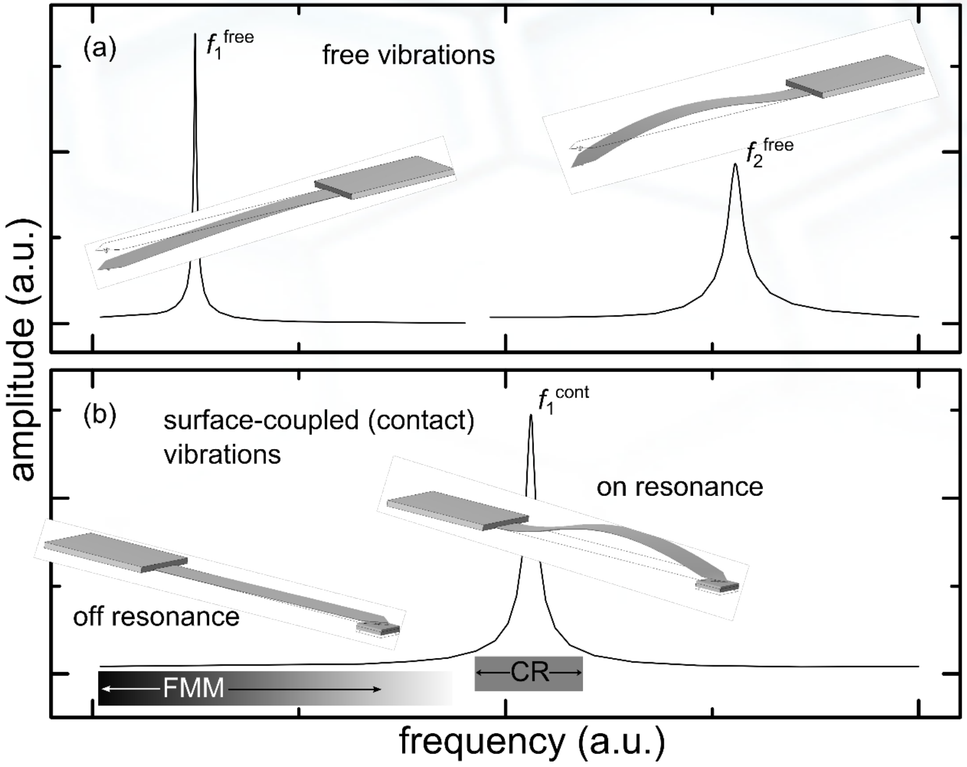
Atomic Force Microscopy

Dynamic Contact Sensing: Contact Resonance

- Shift in frequency proportional to stiffness
- Viscoelasticity obtained from Q-factor ($f_r/\Delta f_r$)
    - Quantitative evaluation of storage modulus, loss modulus, tan δ via Elastic Beam model
- Fast sensing capability (bandwidth (1 kHz -100 kHz -> 1 ms – 10 µs sensing)







Atomic Force Microscopy

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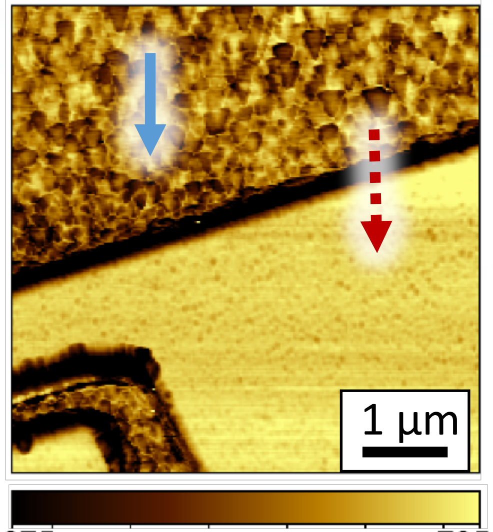
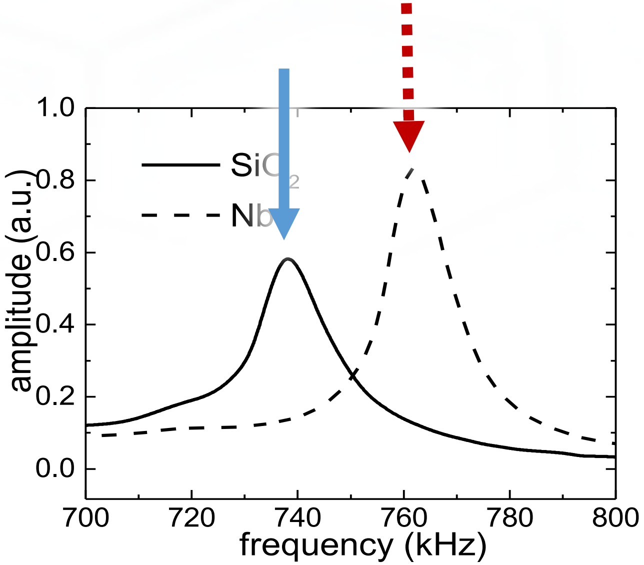
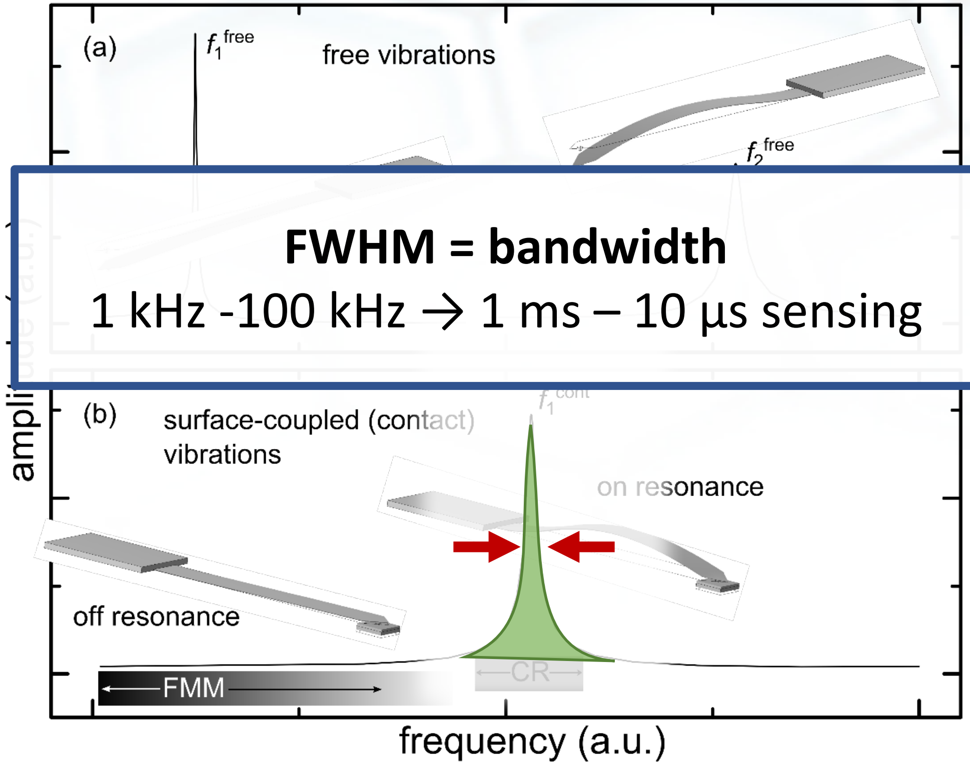




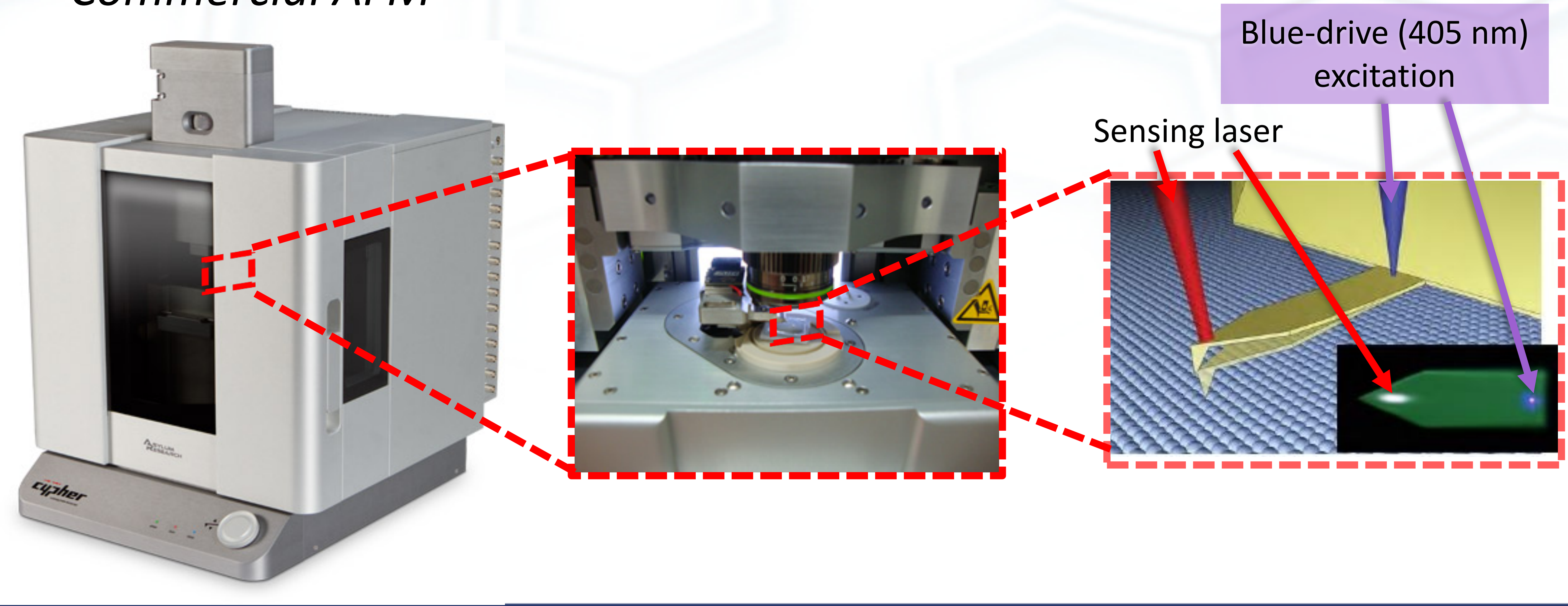


FWHM = bandwidth

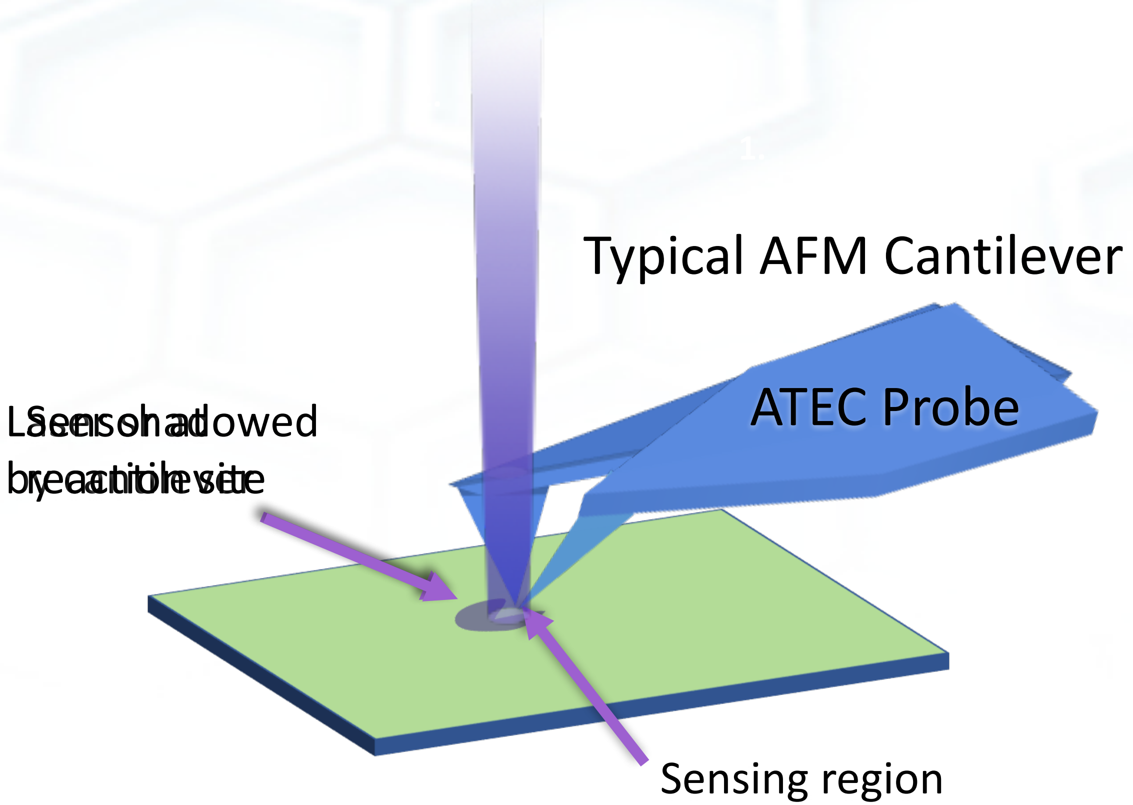
1 kHz -100 kHz → 1 ms – 10 µs sensing

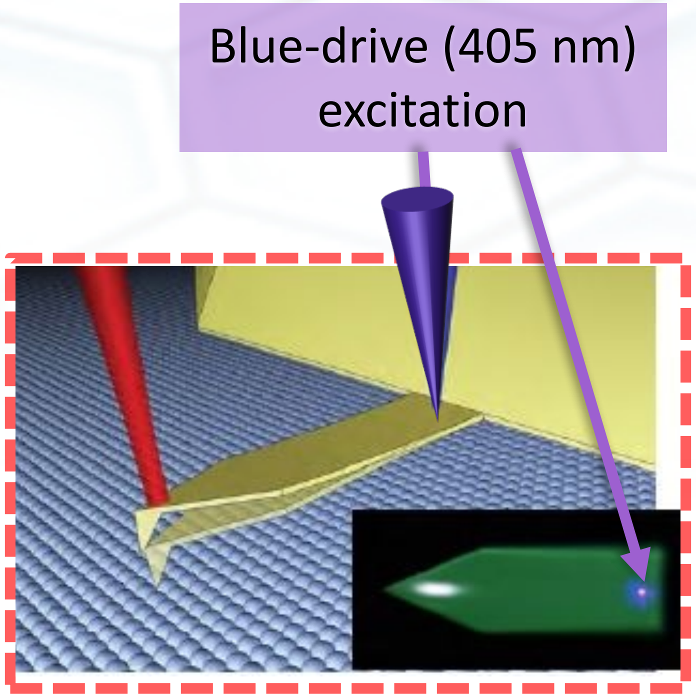
Adapting Current System

Commercial AFM

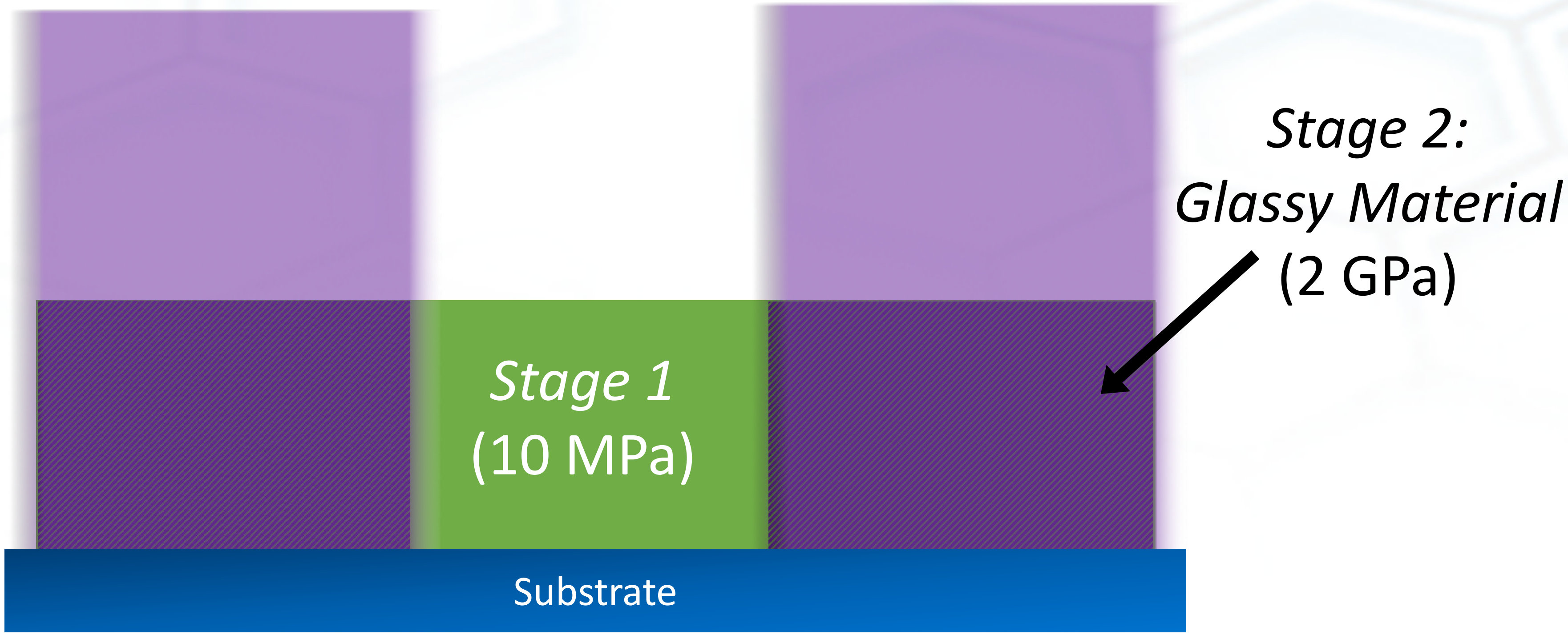


Adapting Current System





In-situ cure: *Dual-cure material*



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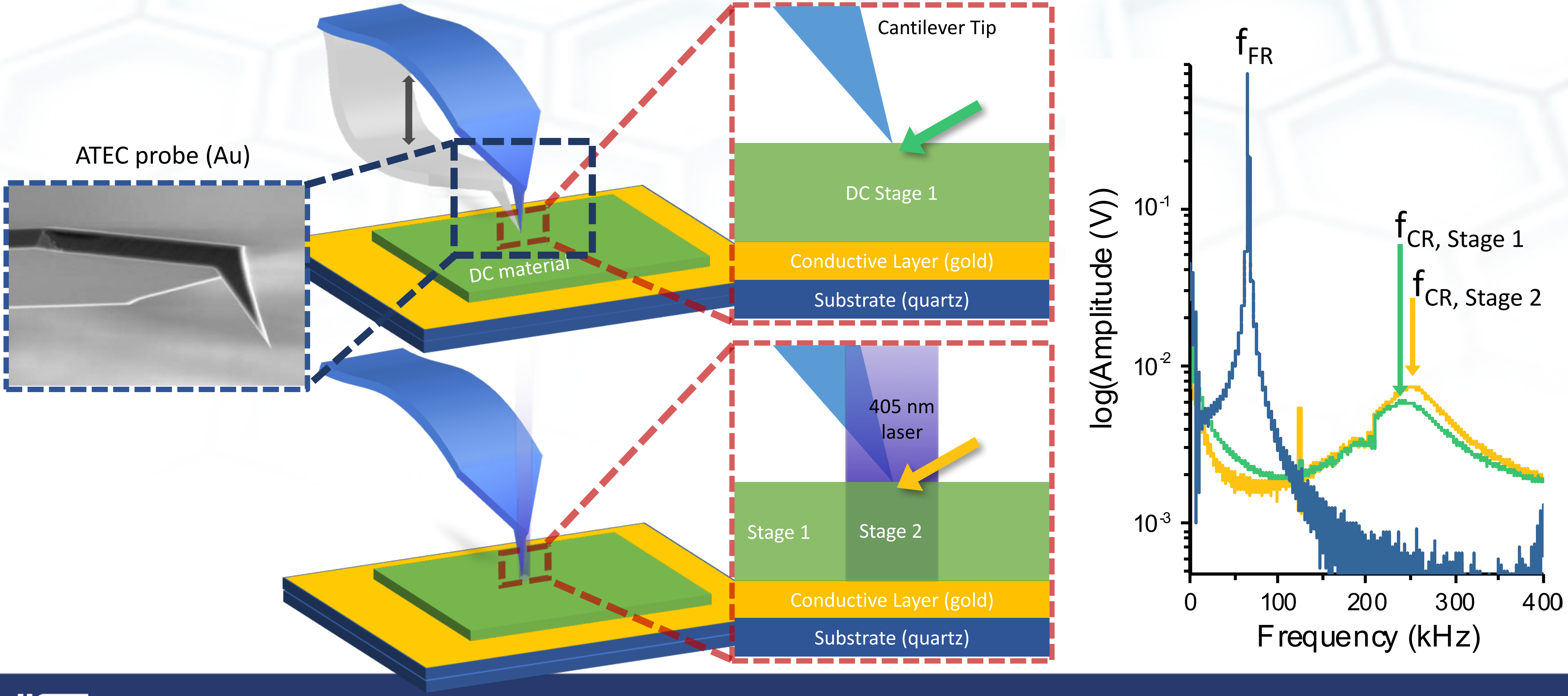

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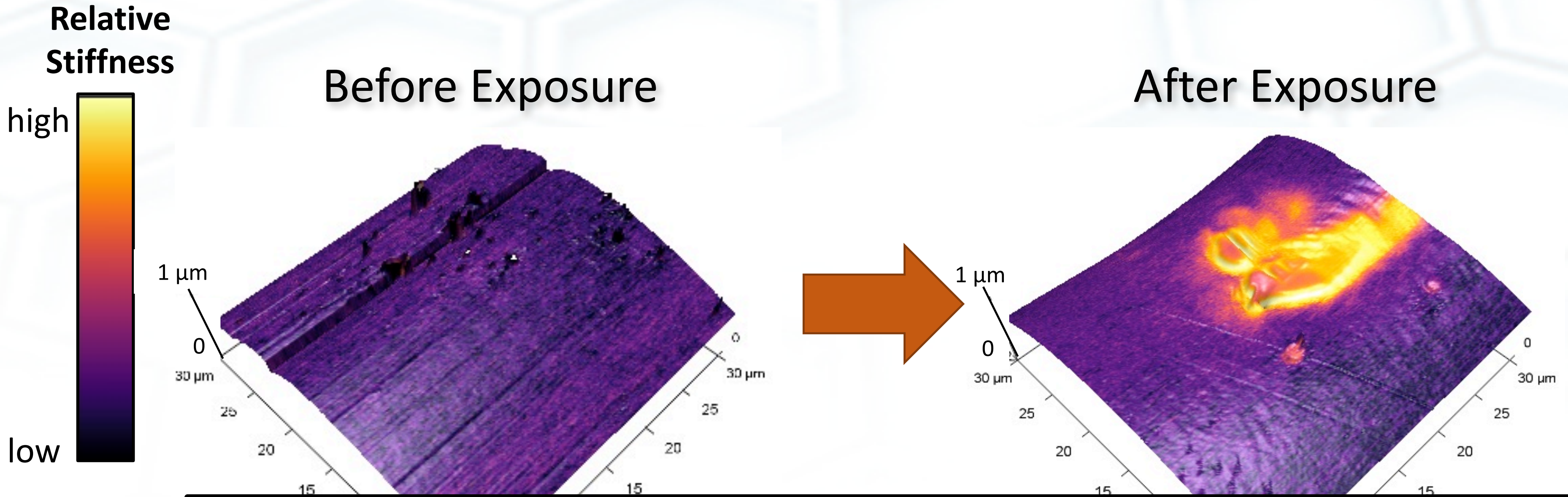
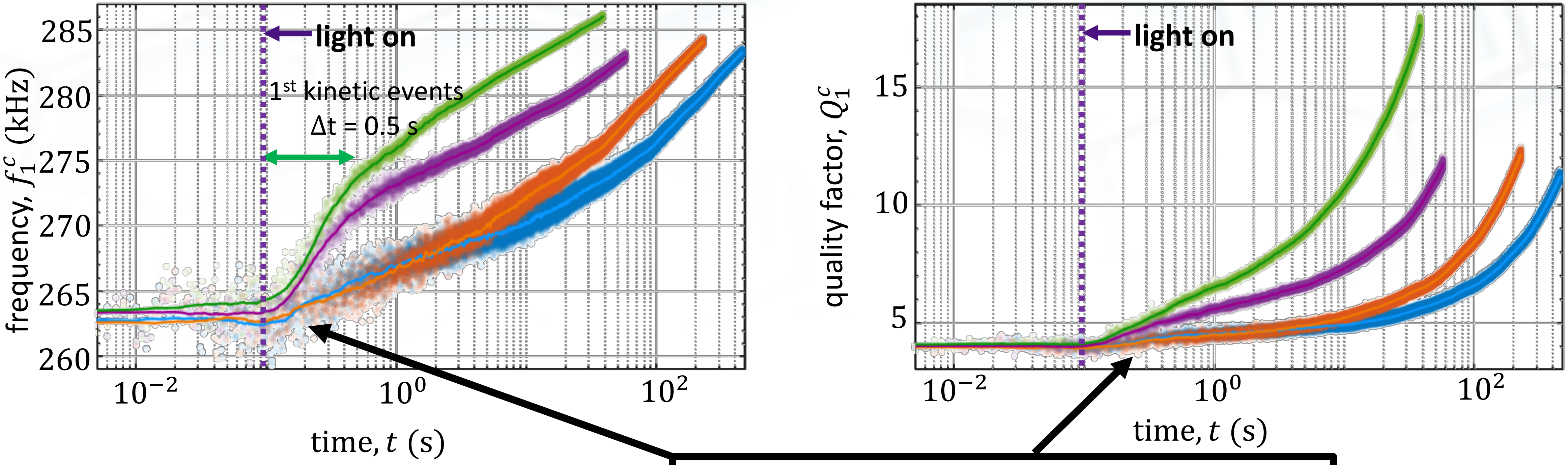


What* locally *occurs* during *exposure?

In-situ cure: *Dual-cure material*

Varied exposure power, constant dose

energy dose (mJ) = power (mW) x exposure time (s)



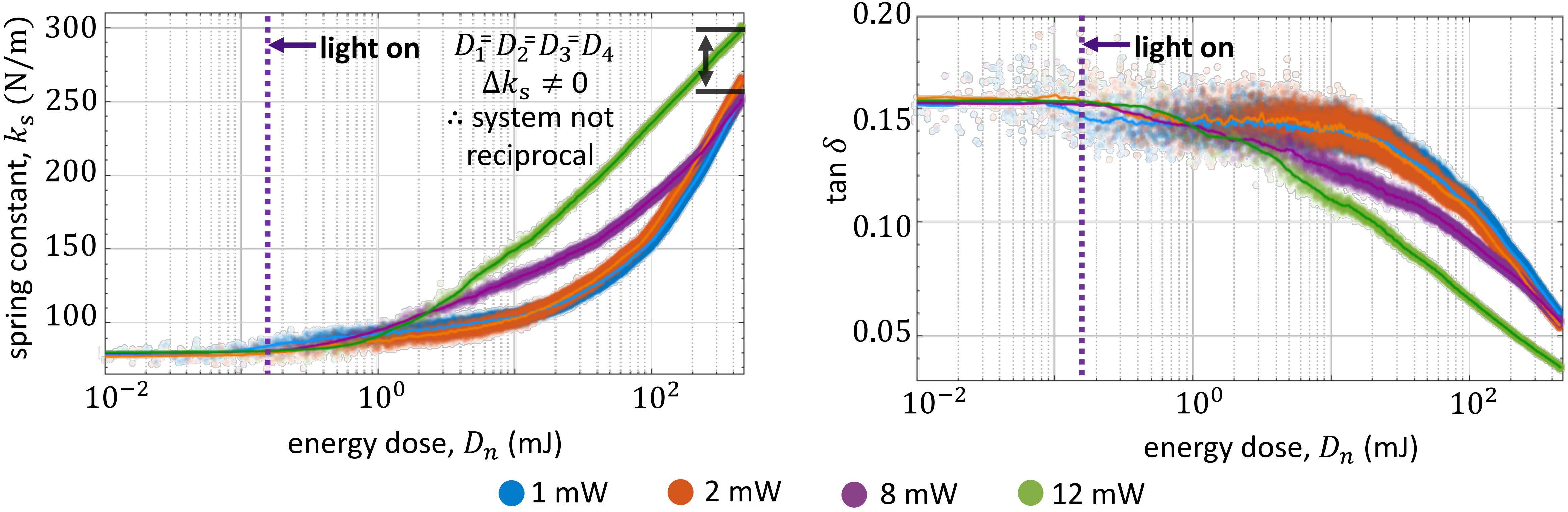
1 mW   2 mW   8 mW   12 mW

Capturing <1 ms kinetic events

In-situ cure: *Dual-cure material*

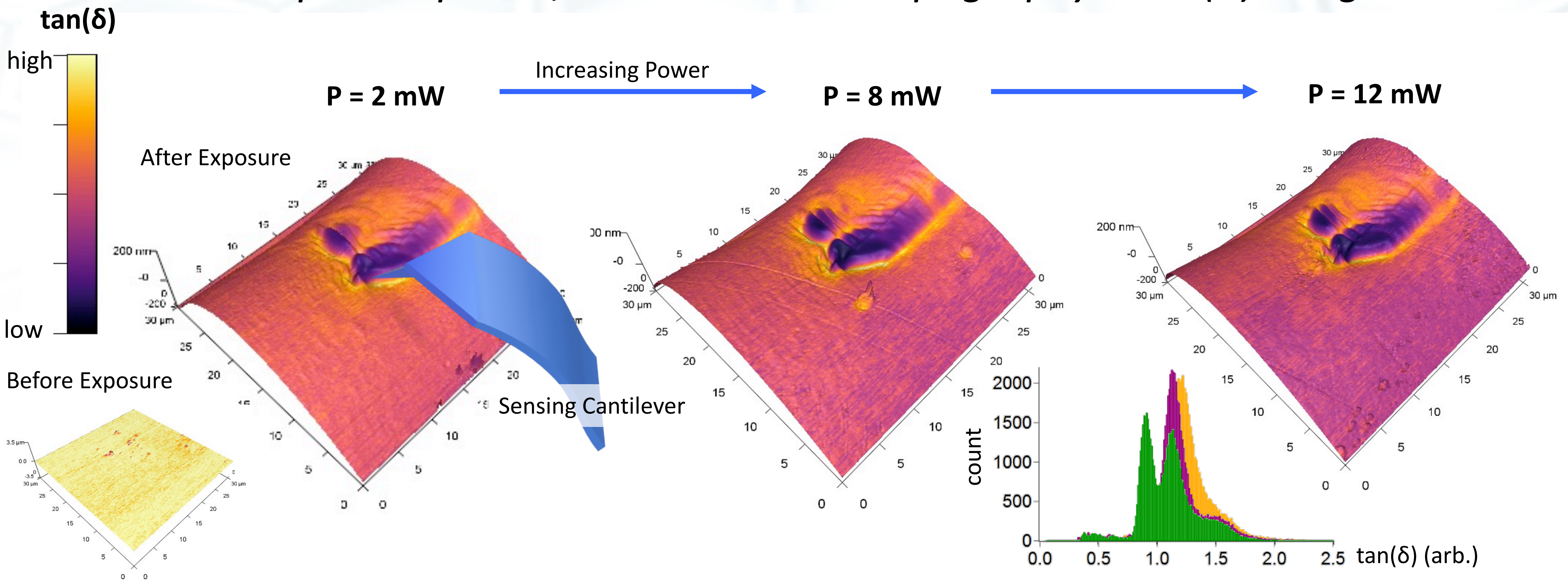
Varied exposure power, constant dose

energy dose (mJ) = power (mW) x exposure time (s)



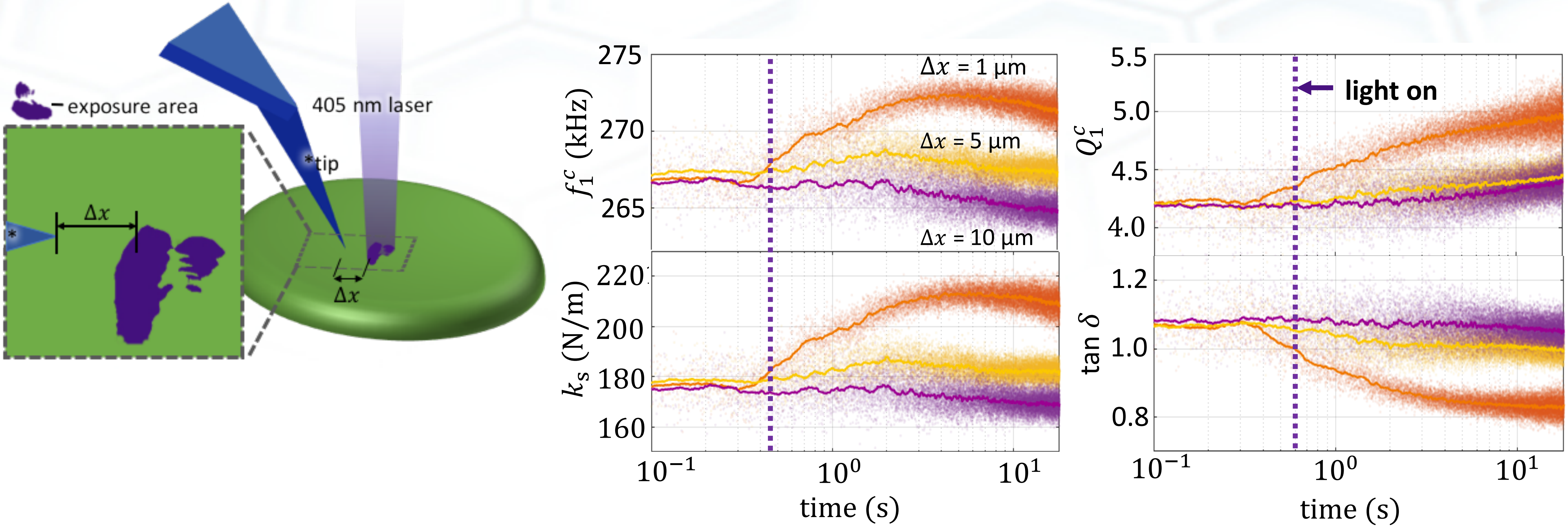
In-situ cure: *Dual-cure material*

Varied exposure power, constant dose : Topography + tan(δ) image

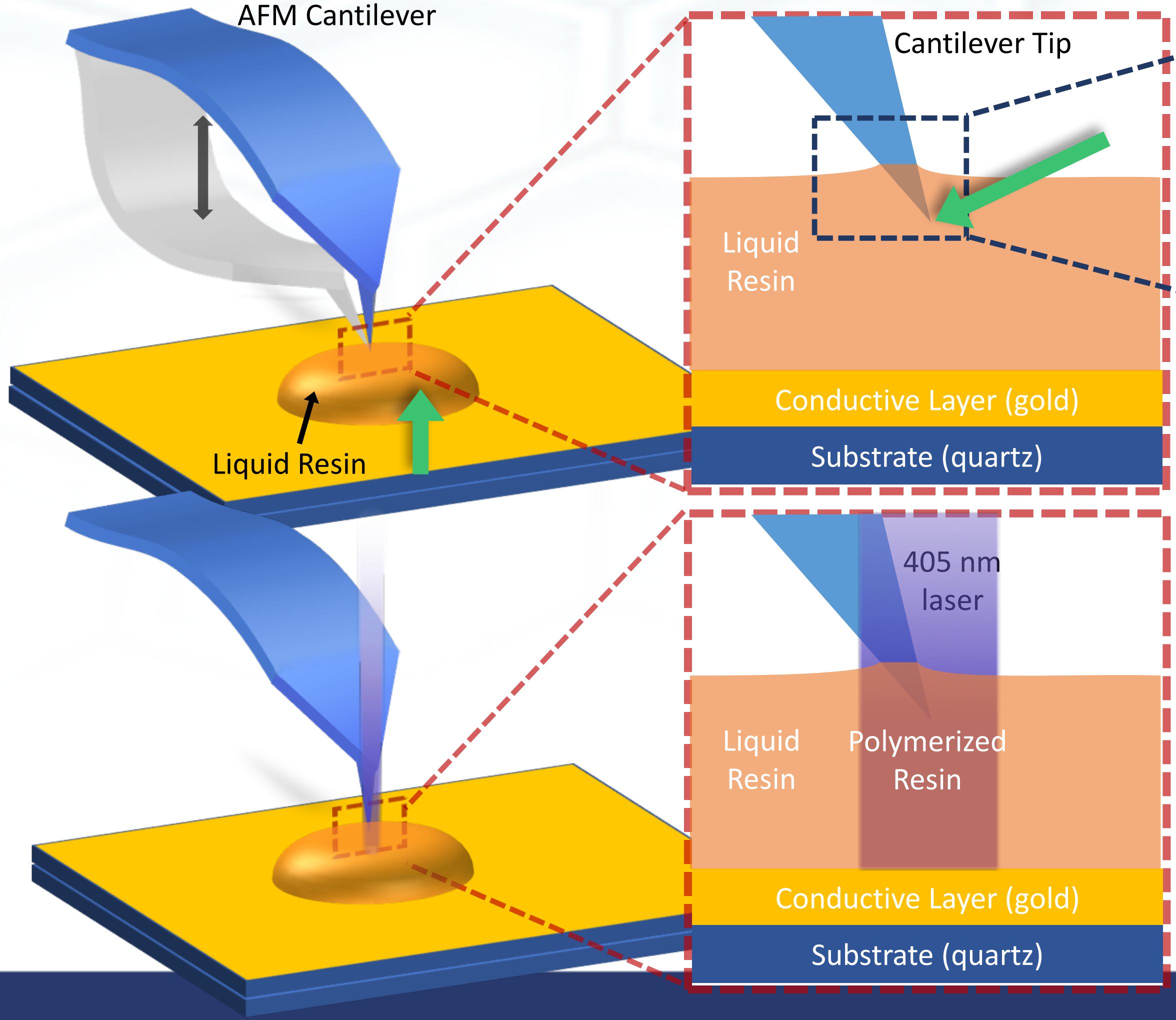


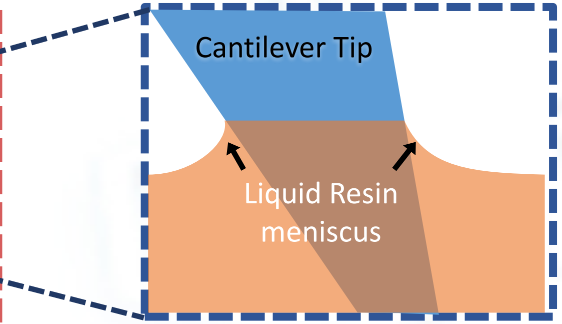
In-situ cure: *Dual-cure material*

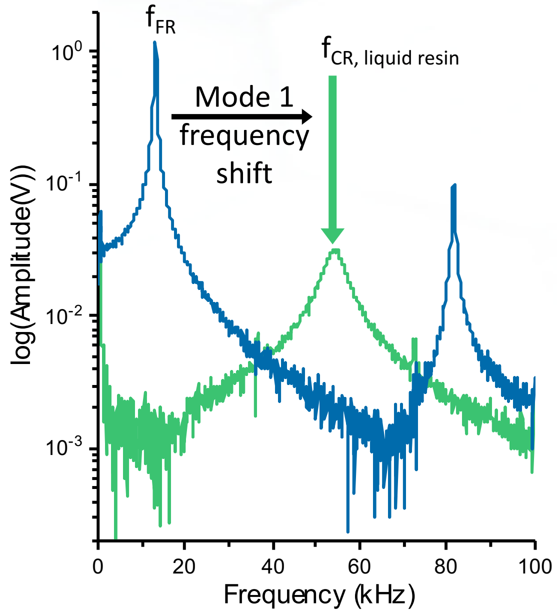
Exposure spatial dependence : damping (Q) and stiffness (f_0)



In-situ cure: *Commercial, liquid AM resin*

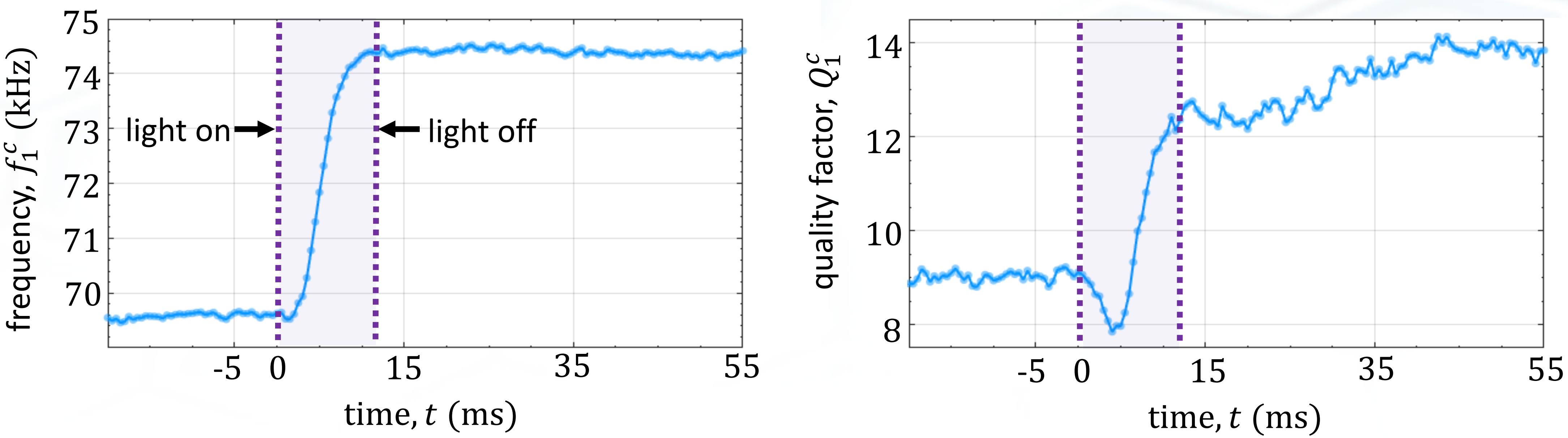






Commercial Resin

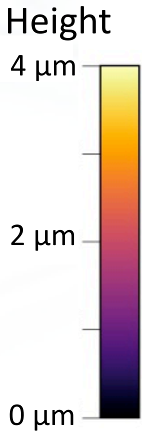
In-situ cure: *Commercial, liquid AM resin*

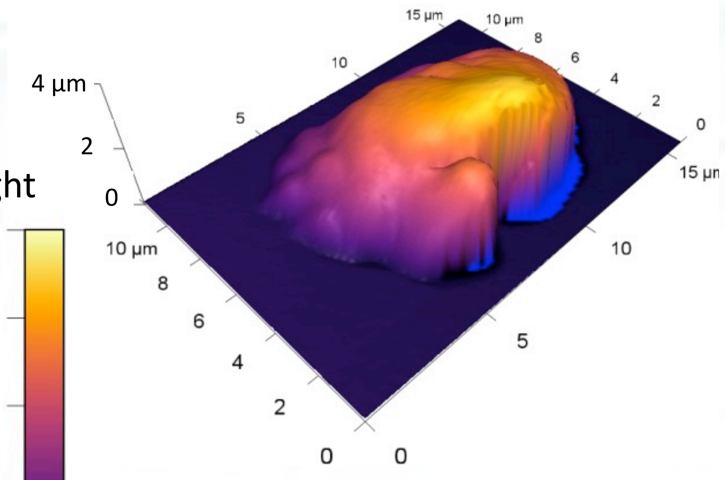


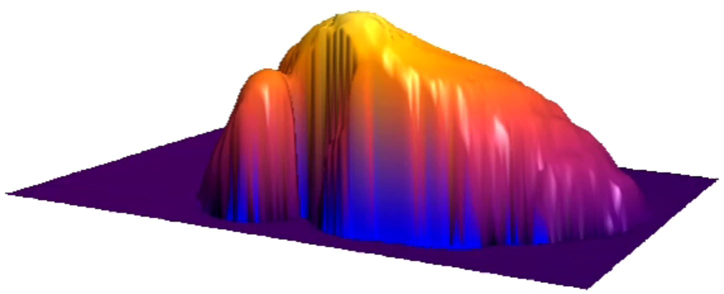
Capturing polymerization event of **commercially available** *resin at the* **relevant intensities** *and* **spatiotemporal resolution**

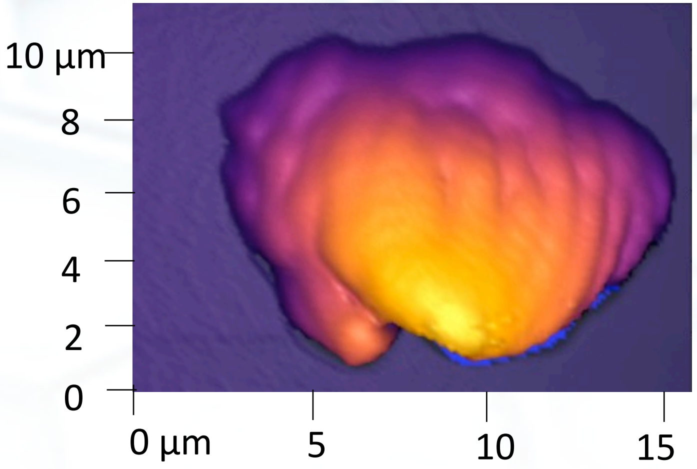
In-situ cure: *Commercial, liquid AM resin*

Topography:in resin

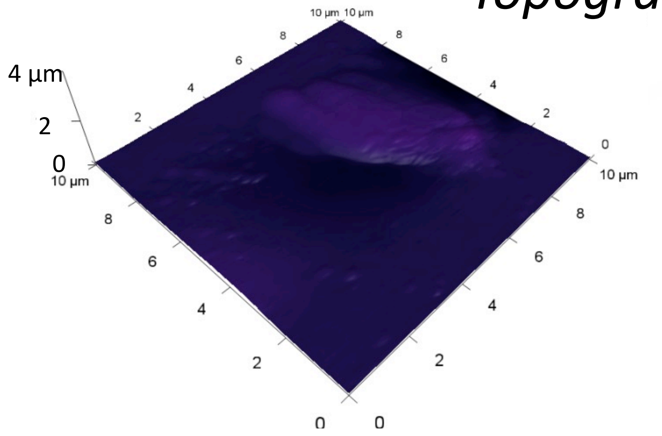


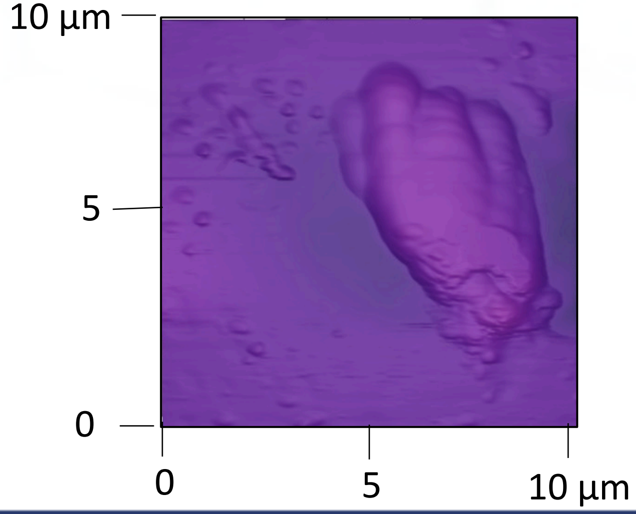






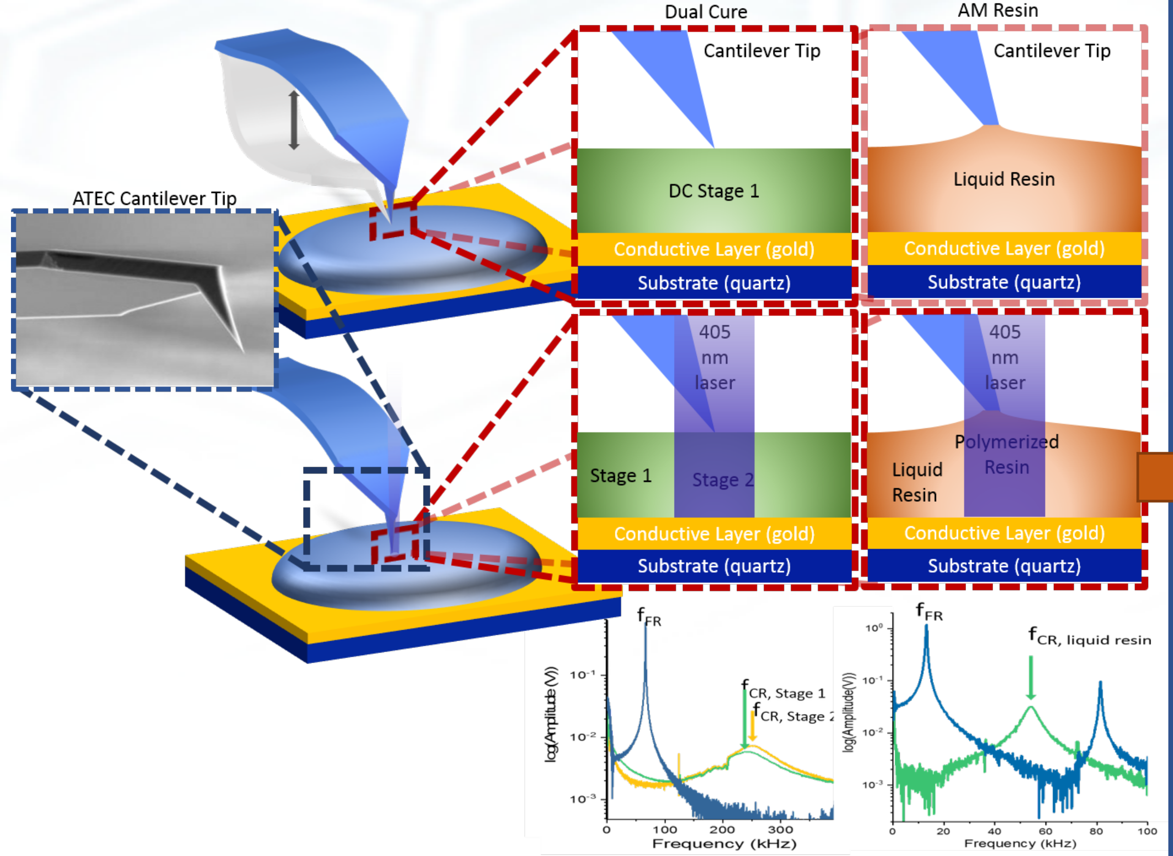
Topography: after rinse



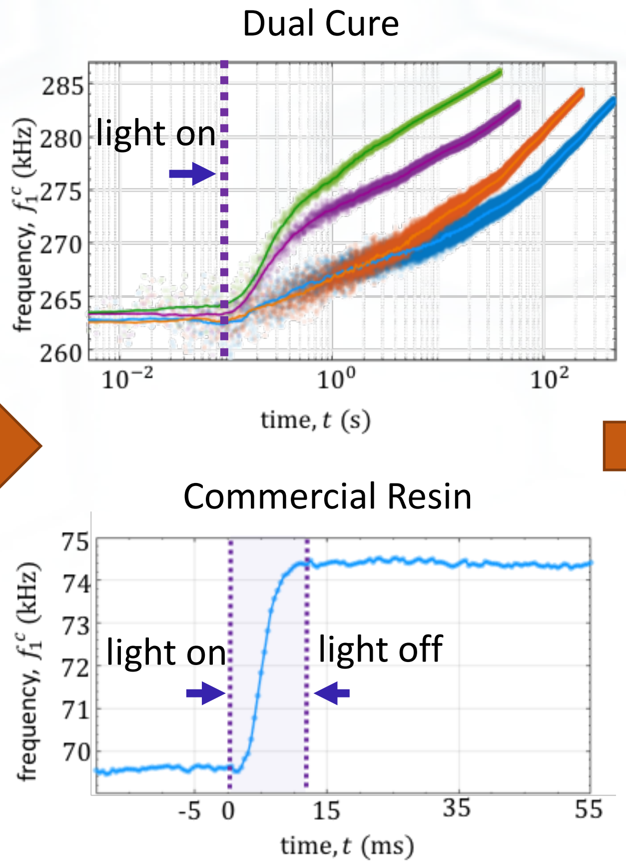


Summary

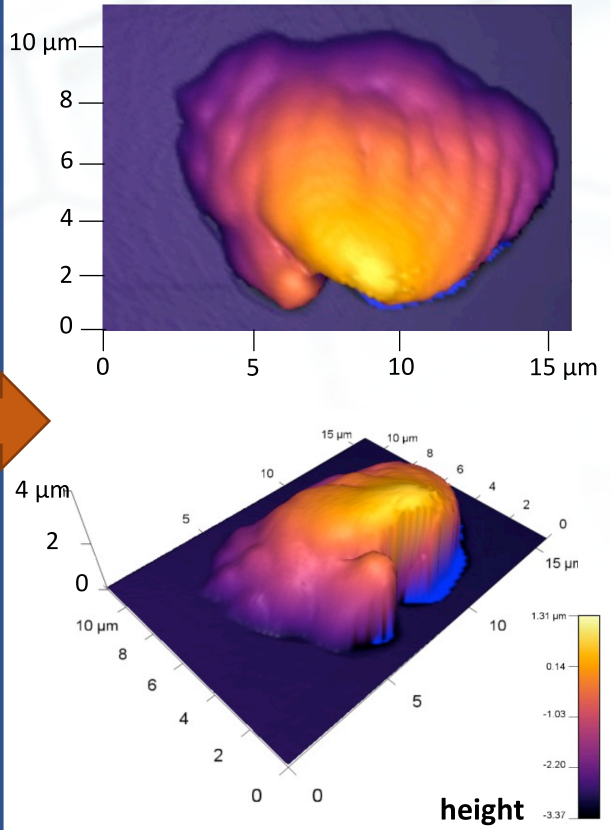
In-situ Contact Resonance



In-situ mechano-rheology



Voxel Imaging



Acknowledgements

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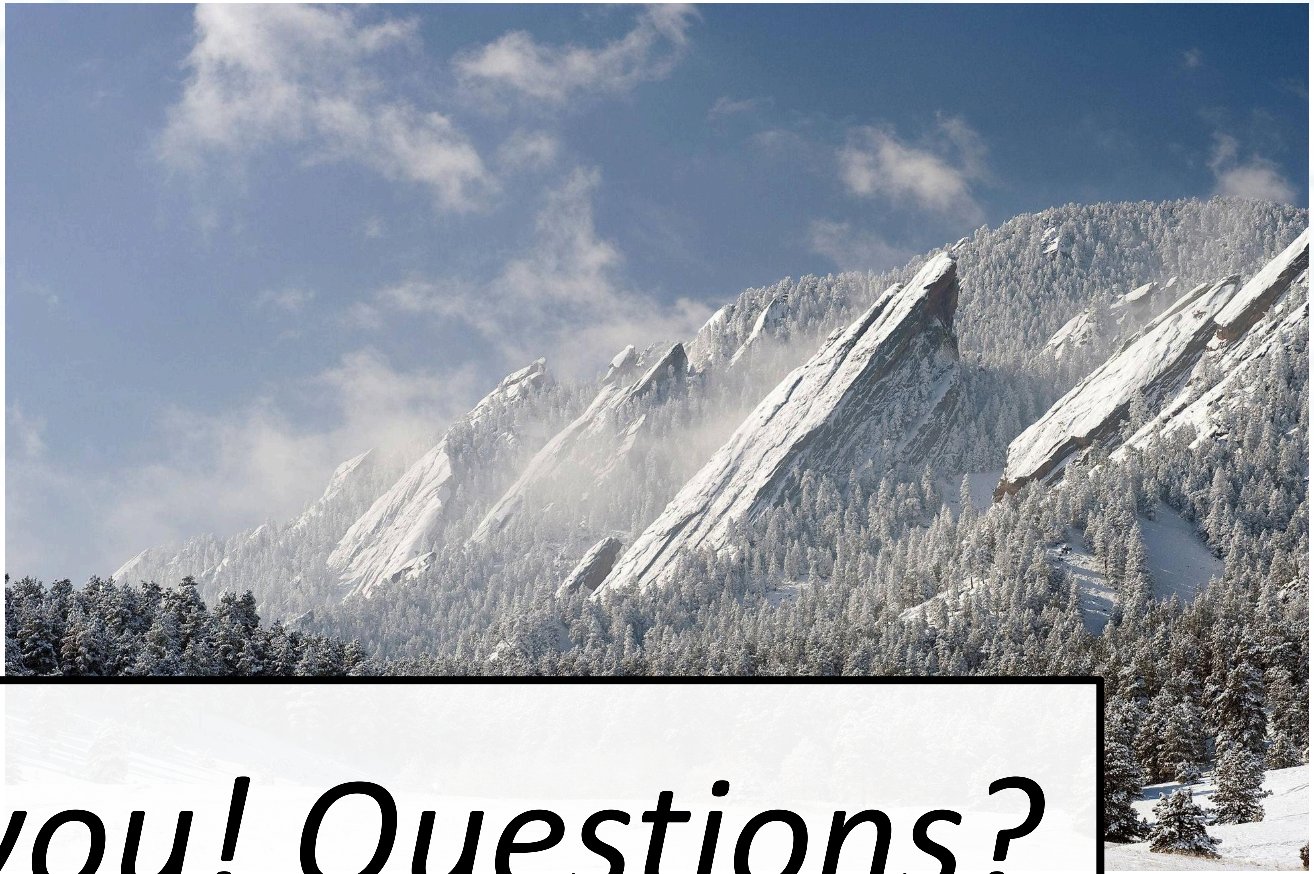
Dr. Jason Killgore

Dr. Lewis Cox

Dr. Ben Caplins



Thank you! Questions?