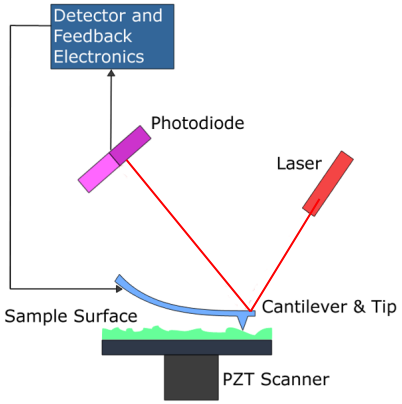
Hydrogel Probes for Atomic Force Microscopy

Melissa Cadena

Mentor: Tejank Shah

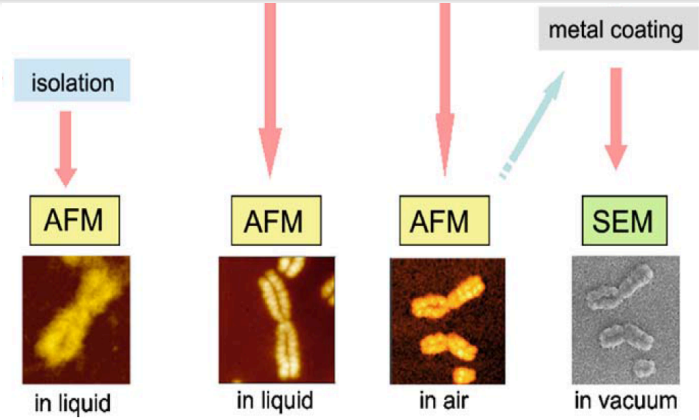
PI: Dr. Stefan Zauscher

Background



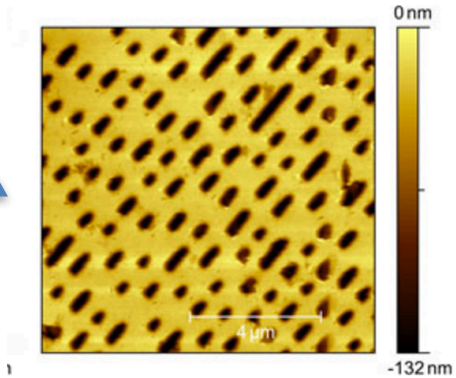
AFM measures a sample by detecting the movements of the cantilever across the sample surface

Advantages

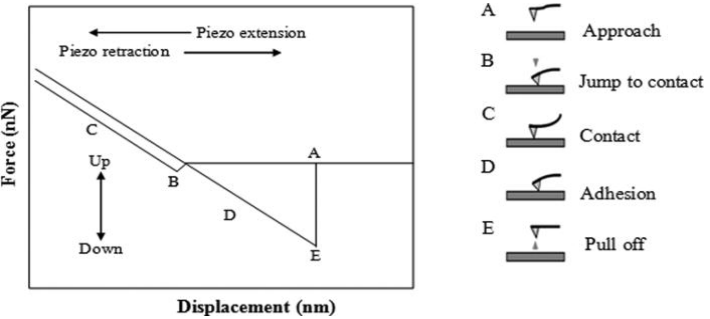


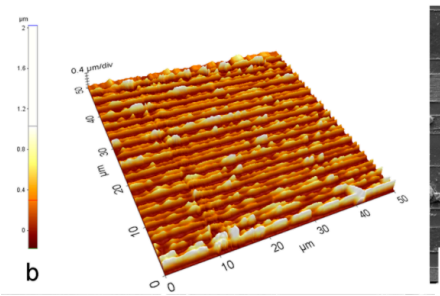
- Human Chromosomes imaged in ambient air and liquid by the AFM and in vacuum by SEM. AFM does not require metal coating of sample.

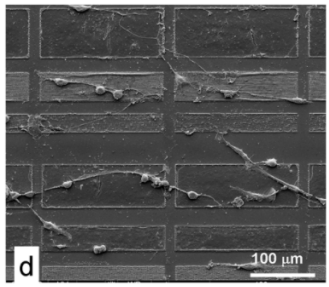
AFM image of DVD stamp imprinted into a PMMA layer



Detects forces the tip experiences as it comes to contact with the sample





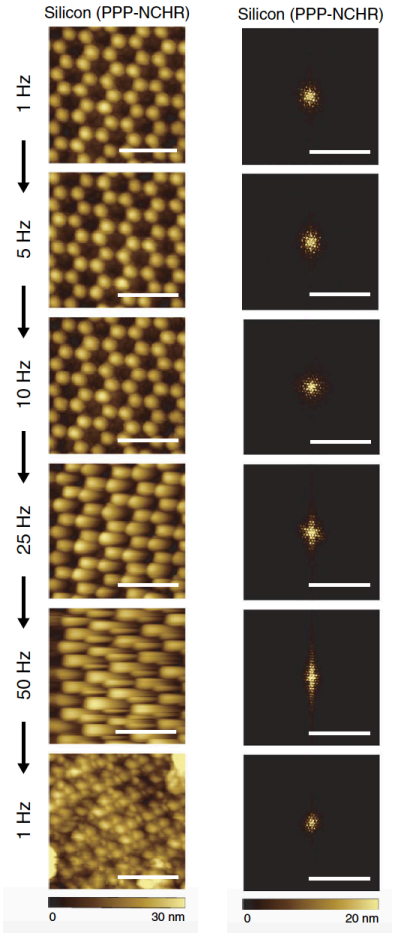


- OLN-93 cells distributed on lamin-coated line structured PMMA. The AFM image (b) provides surface topography and better 3D resolution than SEM (d).

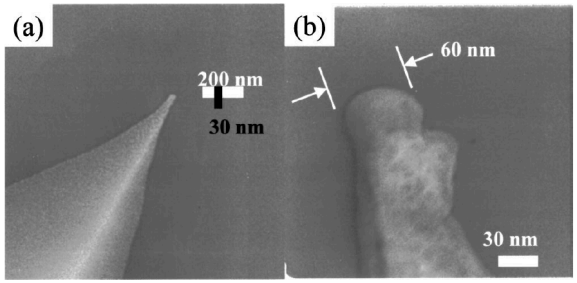
[1] "Atomic Force Microscopy", Wikipedia; [2] J. Frischeisen, *ENB*, 2012; [3] D. Yu, *Applied Physics*, 2014; [4] *Chromosome Research,* 2008; [5] V. Dinca, *Recent Advances in Biopolymers*, 2016

Disadvantages of Silicon Probes

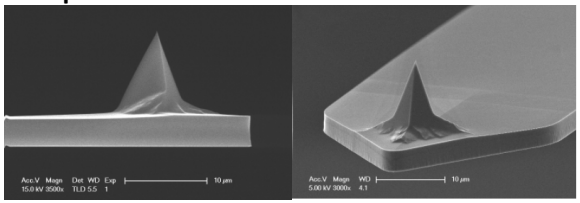
1. Unable to image at fast scan rates and observe increased artifacts



2. Short Tip Life



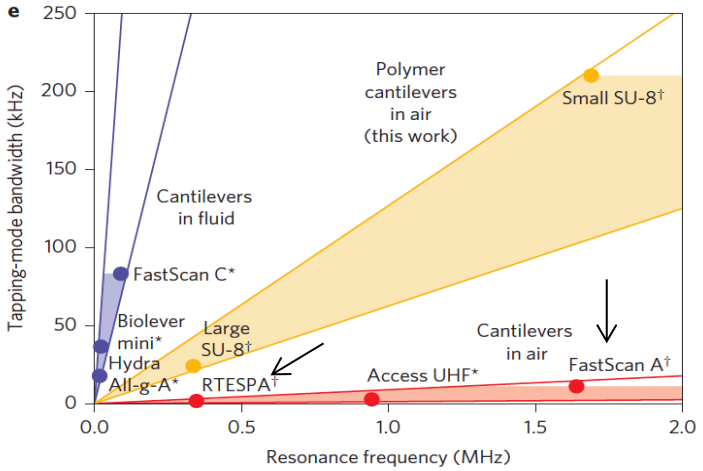
3. Limited tip geometries and aspect ratios



Ideal Probe Characteristics:

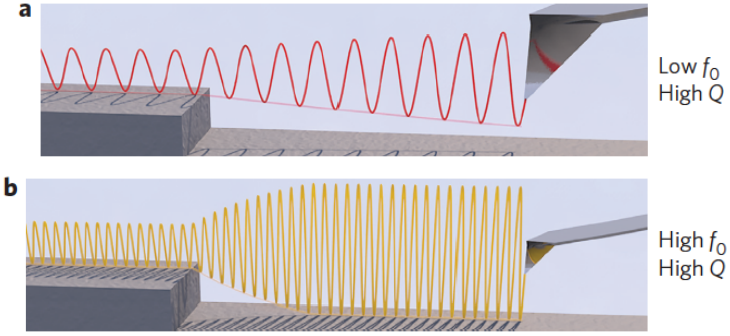
1. Image at fast scan rates
2. Reduce artifacts on sample
3. Minimal tip wear/be cheaper
4. Have different tip geometry and aspect ratios
5. Provide tunable mechanical properties

4. Limited Mechanical Tunability



RTESPA has a low f_o and high Q (350) and FastScanA has a high f_o and high Q (180)

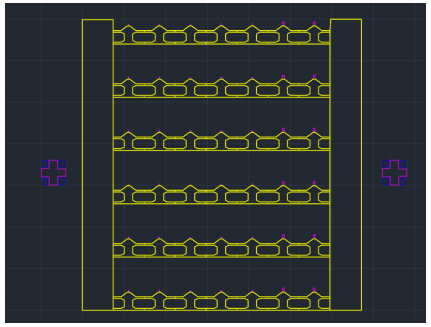
Q factor: Ratio of energy stored to energy lost per cycle at resonance



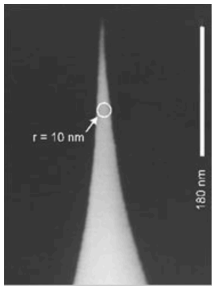
[1] J. Lee, *Nature Comm*, 2016; [2] T. Larsen, *Appl. Phys. Lett*, 2002; [3] Bruker AFM Probes; [4] J.D. Adams, *Natue Nanotech, 2015*

Project Goals

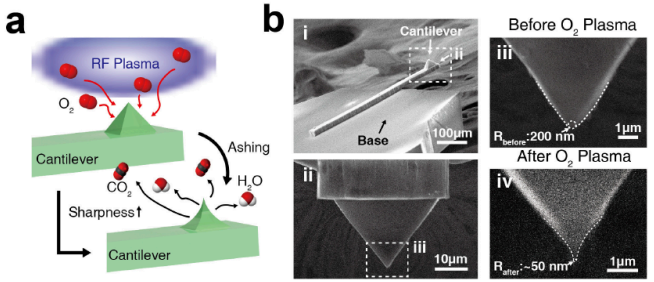
- Replicate Lee's procedure using Nicolas Wainwright's mask design and a modified mask



- Test the effect of oxygen plasma ashing on tip radius

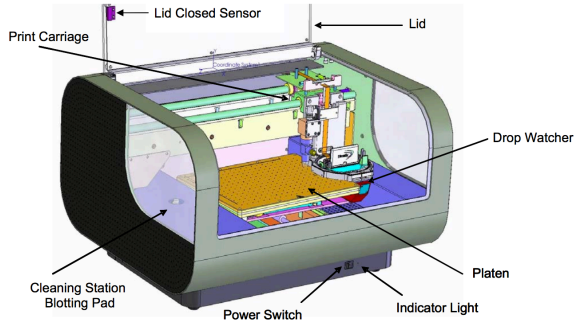


Silicon tip with 10 nm tip radius



Hydrogel tip with 50 nm tip radius

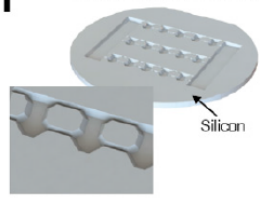
- Controllably print PEGDA on tipless cantilevers using an inkjet printer



[1]J. Lee, *JMEMS Letters*, 2017; [2]Nano and More AFM Probes, SSS-FM Probes; [3]Fuji Film, *Dimatix Material Printer User Manual*, 2007

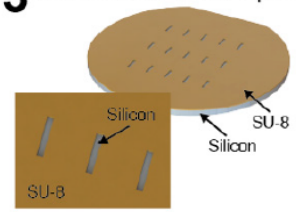
Batch Fabrication Approach

1 Silicon Base Mold



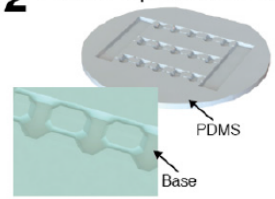
Photolithography and Deep Reactive Ion Etching on silicon wafer to achieve 150 µm deep chip mold

3 SU-8 Cantilever & Tip Mold



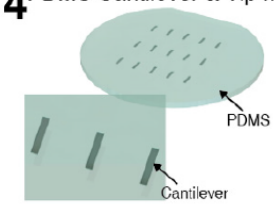
Photolithography and KOH etching of silicon wafer with 400nm silicon dioxide layer. Then pattern wafer with SU-8 to make aligned negative cantilever and tip mold

2 PDMS Replica Base Mold



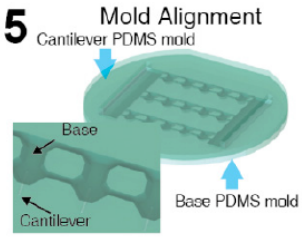
Treat with 1H, 1H, 2H, 2H-perfluorodecyltriethoxysilane (PFDTES) and replicate twice with PDMS to obtain 150 µm deep chip mold

4 PDMS Cantilever & Tip Mold



Treat with PFDTES and replicate twice with PDMS to obtain negative cantilever and tip mold

5 Mold Alignment



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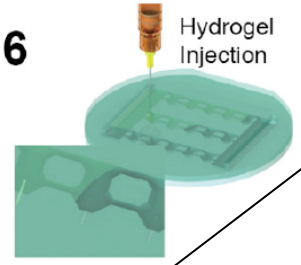

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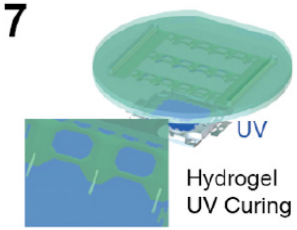

Demolding

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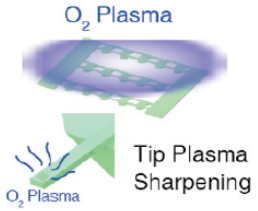

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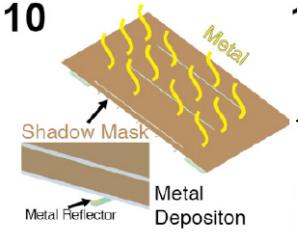
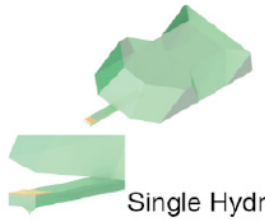


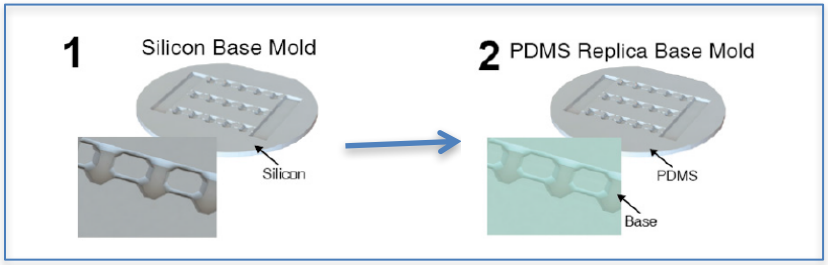
Cutting

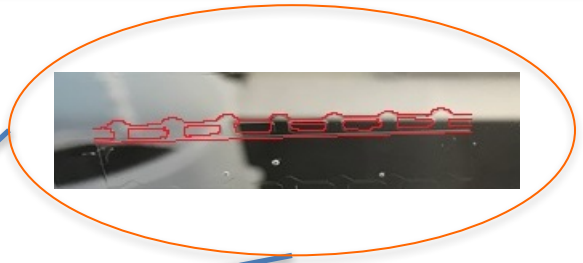
12

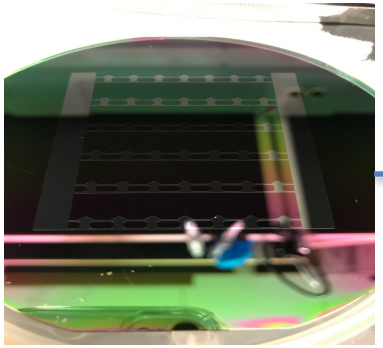


[1] J. Lee, *JMEMS Letters*, 2017

Results – Chip Mold

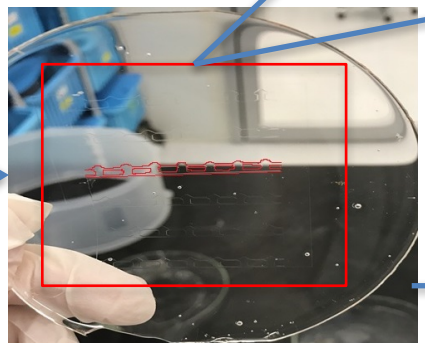






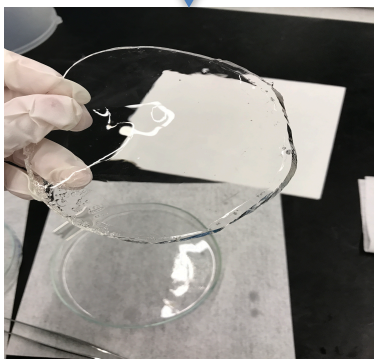
150 µm deep chip mold

Treat with PFDTES and replicate with PDMS



Positive 150 µm PDMS chip mold array

(1) Treat with PFDTES and replicate with PDMS
(2) Plasma ash, treat with PFDTES and replicate with PDMS



PDMS mold with no chip features

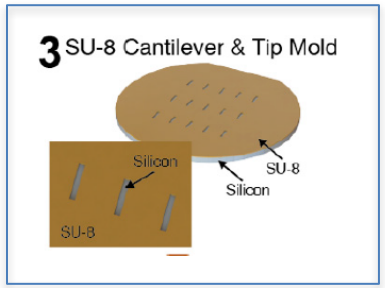
Proposed Solution:

(1) Plasma ash for longer and with more power, place in vacuum for longer

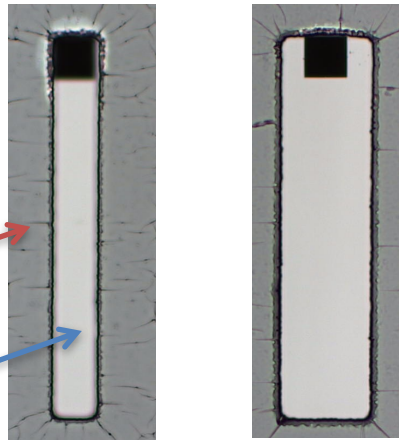
(2) Plasma ash for longer and with more power, place in vacuum oven

[1] J. Lee, *JMEMS Letters*, 2017

Results – Tipped and Tipless Cantilevers

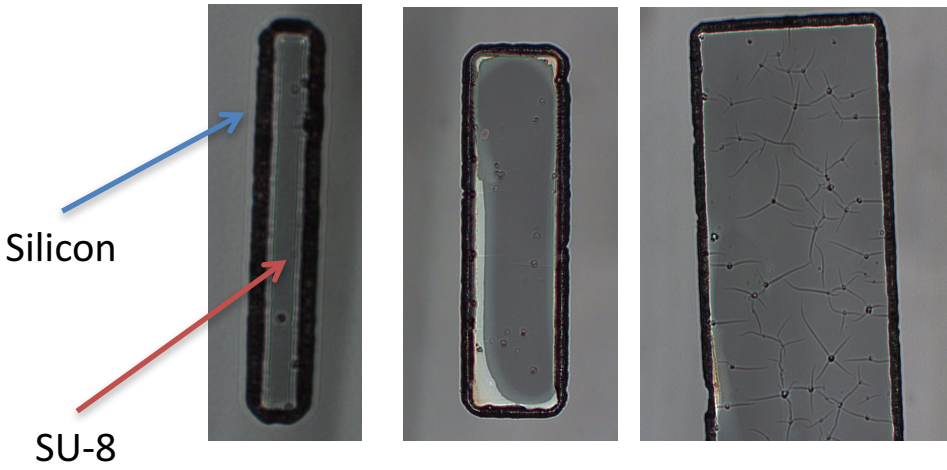


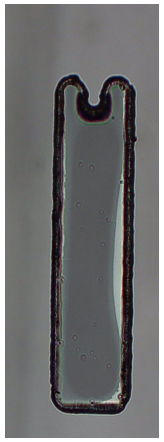
Tipped Cantilevers



- Three different dimensioned cantilevers are present
- No deformities present

Tipless Cantilevers



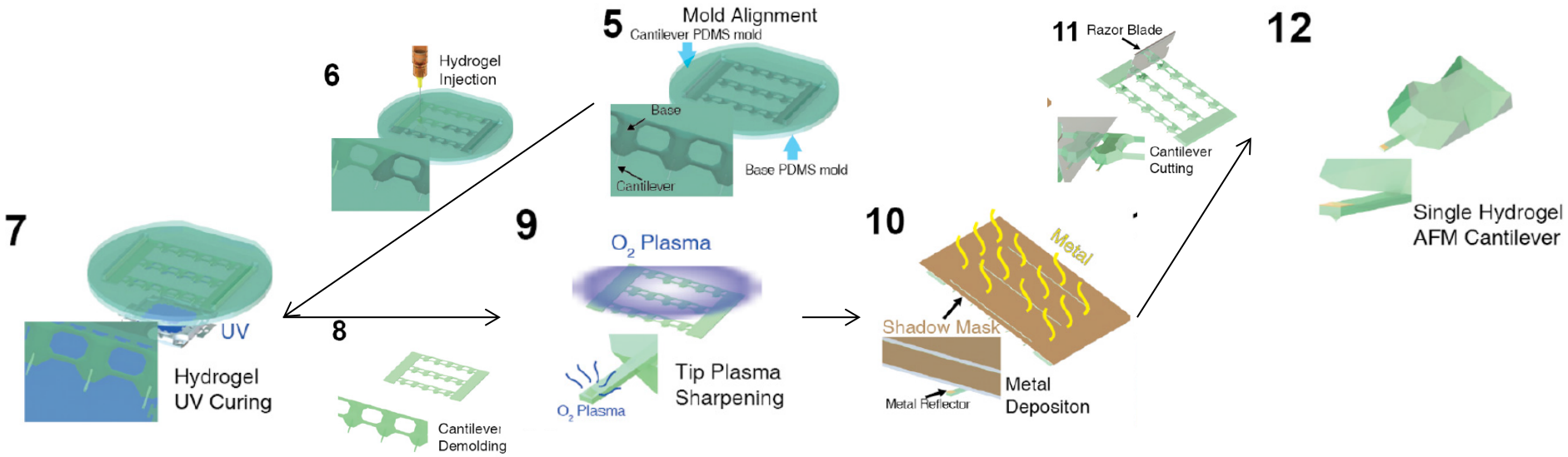


- Three different dimensioned cantilevers are present
- Deformed cantilevers present as a result of poor SU-8 adhesion
- 71% of cantilevers can be used

[1] J. Lee, *JMEMS Letters*, 2017

Next Steps

- Determine the best method for PDMS molding from PDMS master mold
- Continue batch fabrication approach to fabricate tip and tipless hydrogel probes:



- Quantify mechanical properties of hydrogel probe
- Assess the force-indentation curves on a standard sample with different types of probes

1. J. Lee, *JMEMS Letters*, 2017

Project Goals

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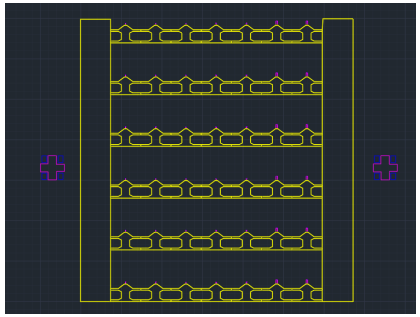

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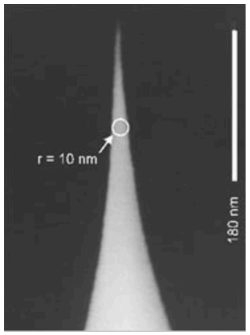

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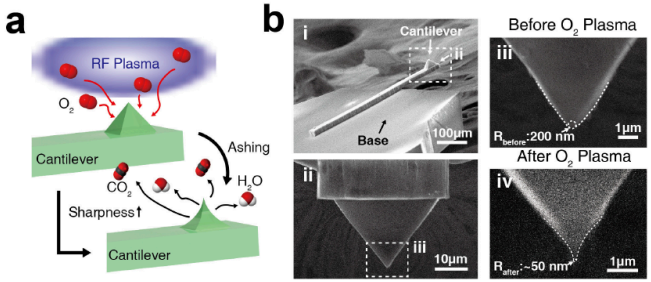

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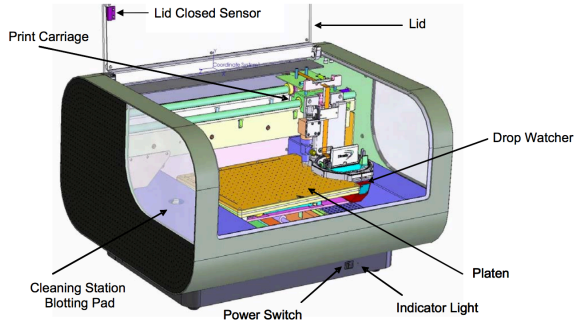

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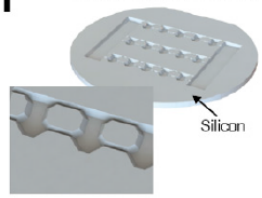

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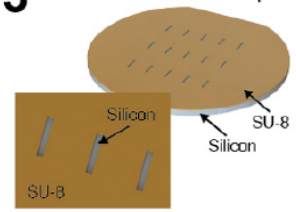

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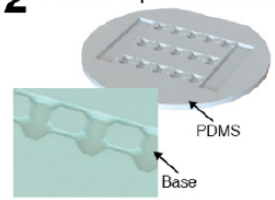

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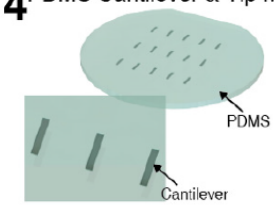

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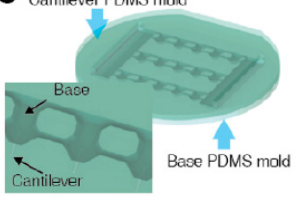
5 Mold Alignment



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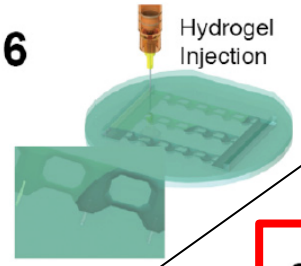

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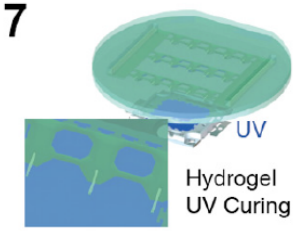
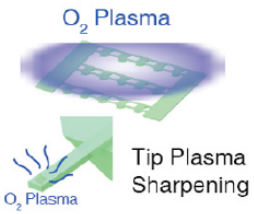


Demolding

9



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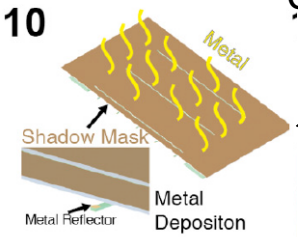

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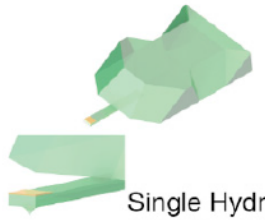

Time

[1] J. Lee, *JMEMS Letters*, 2017

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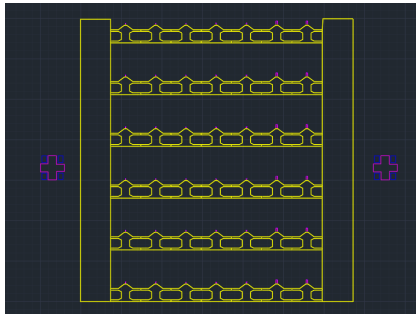

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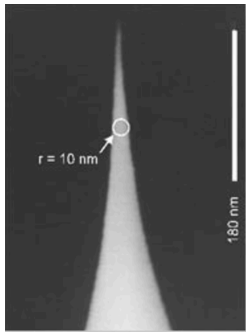
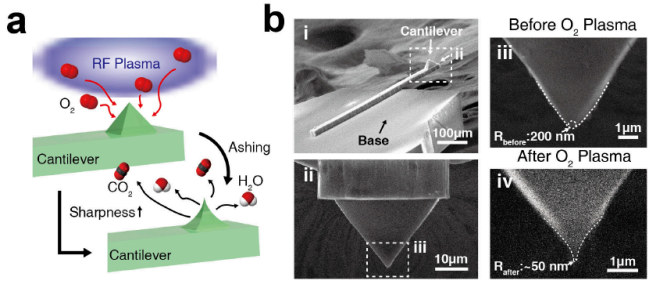
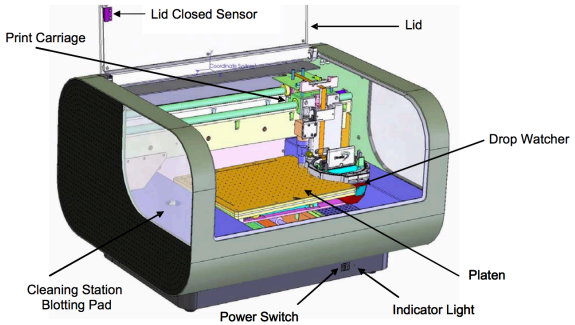


Silicon tip with 10 nm tip radius



Hydrogel tip with 50 nm tip radius

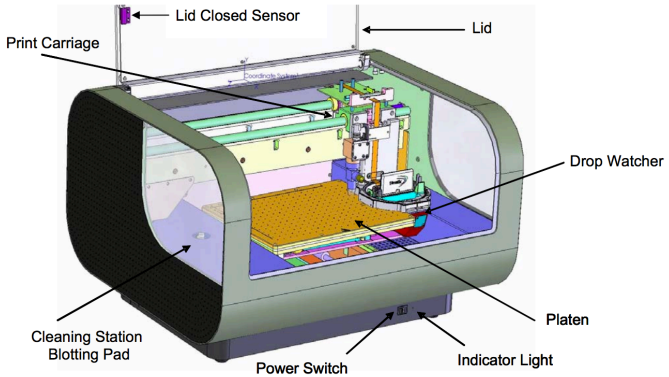
- Controllably print PEGDA on tipless cantilevers using an inkjet printer



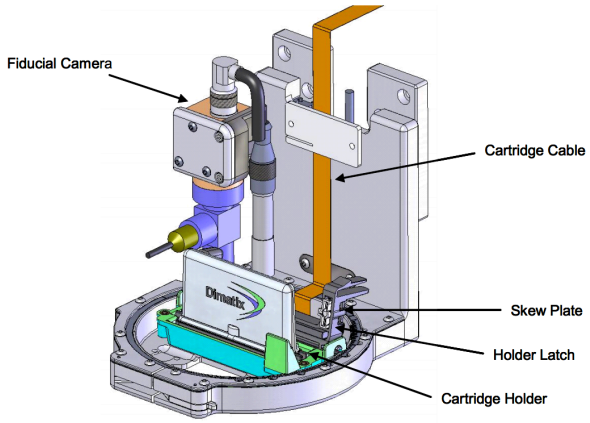
[1]J. Lee, *JMEMS Letters*, 2017; [2]Nano and More AFM Probes, SSS-FM Probes; [3]Fuji Film, *Dimatix Material Printer User Manual*, 2007

Can we control tip radii with batch fabrication?

Proposal: Controlling droplet volume through inkjet printing



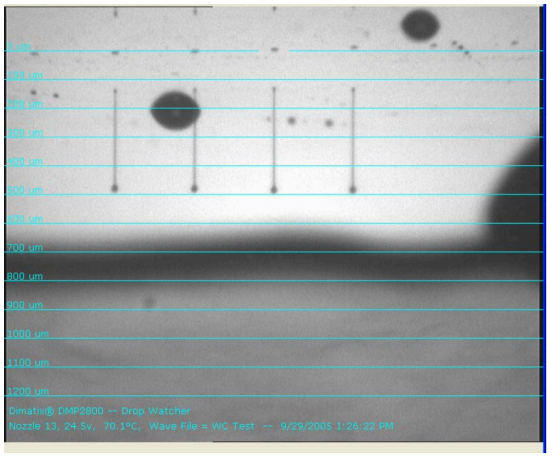
Dimatix Materials Printer, 2800 series



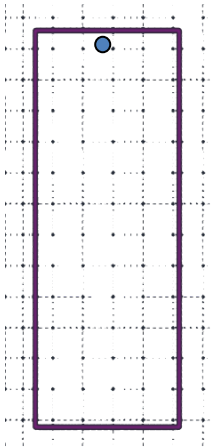
Long Term Goal: To controllably print PEGDA spherical tips on tip-less cantilevers

Thanks Dan Joh!

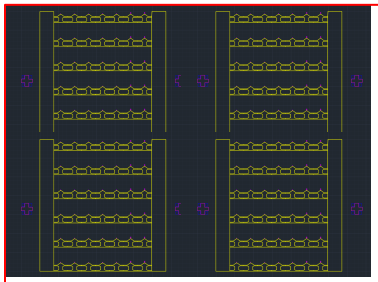
Can vary different parameters and observe changes through drop watcher



Can use a predefined design, create your own design, or import a specific pattern



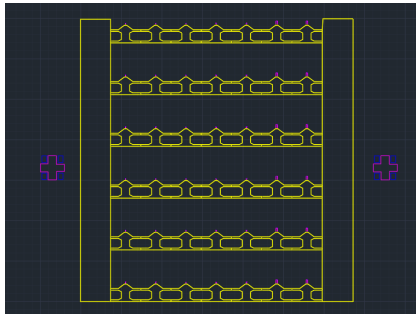
Can batch fabricate a pattern because of large printing area (8.5 inches x 11 inches)



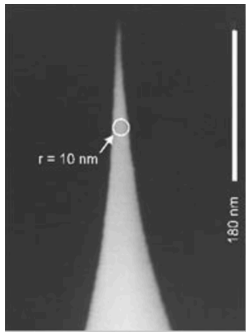
[1]Fuji Film, *Dimatix Material Printer User Manual*, 2007

Project Goals

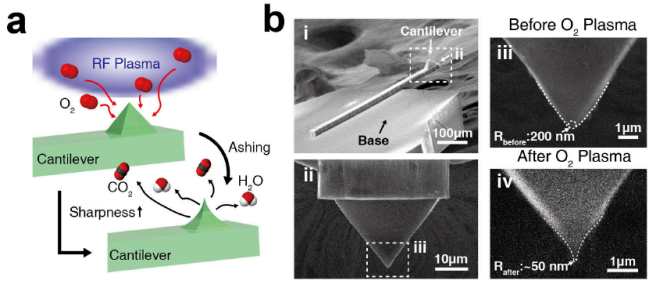
- Replicate Lee's procedure using Nicolas Wainwright's mask design and a modified mask



- Test the effect of oxygen plasma ashing on tip radius

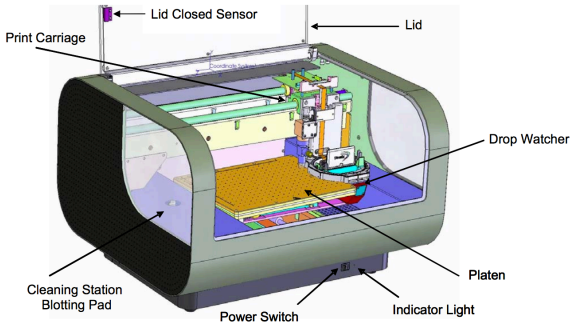


Silicon tip with 10 nm tip radius



Hydrogel tip with 50 nm tip radius

- Controllably print PEGDA on tipless cantilevers using an inkjet printer



[1]J. Lee, *JMEMS Letters*, 2017; [2]Nano and More AFM Probes, SSS-FM Probes; [3]Fuji Film, *Dimatix Material Printer User Manual*, 2007

Acknowledgements

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Chilkoti Lab
Dan Joh

SMIF
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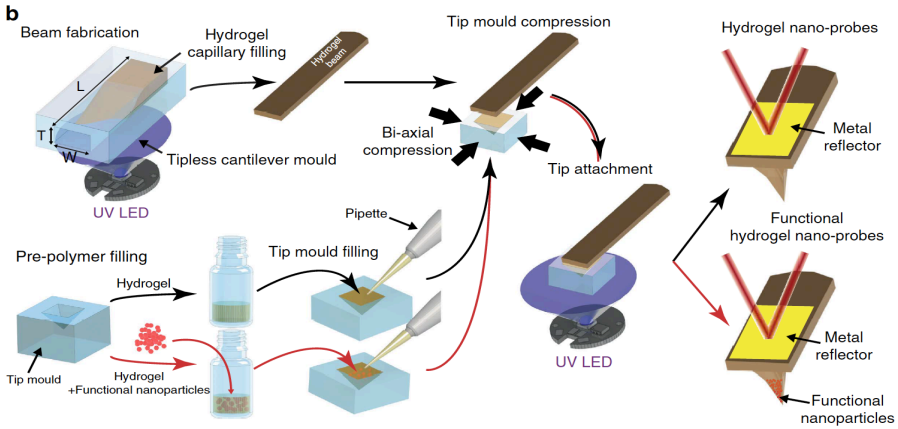
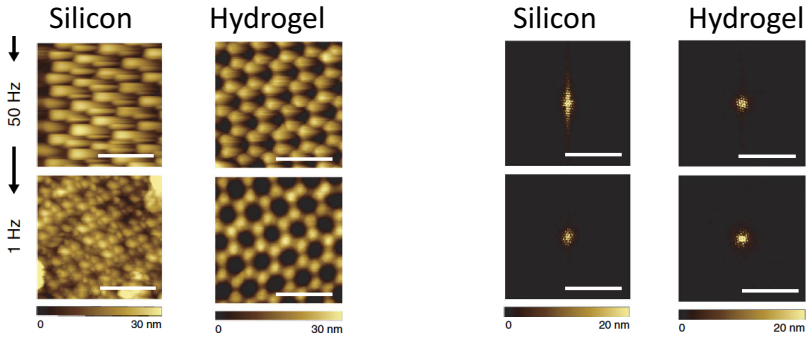
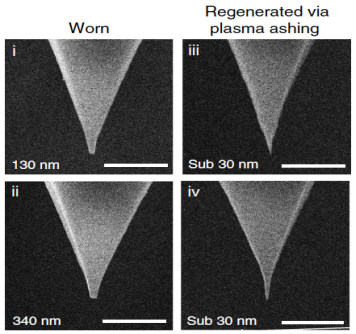
Extra Slides

Hydrogel Probe Fabrication and Results



Hydrogel nanoprobes image well at fast rates and minimize distortions on the sample

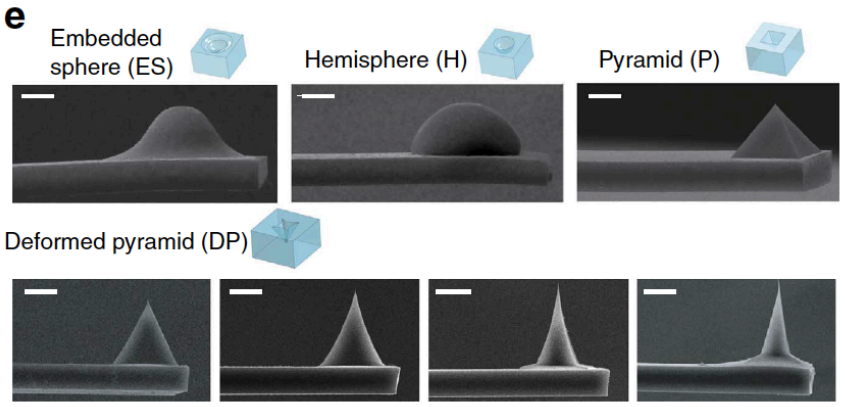




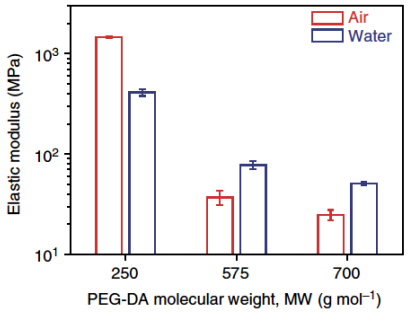
Can regenerate worn hydrogel tips through oxygen plasma ashing

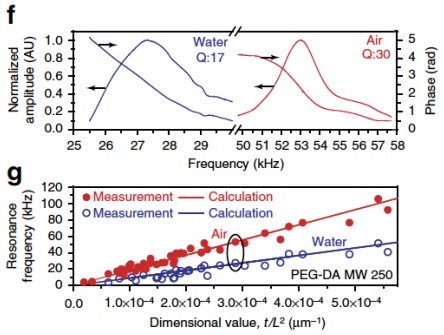
1. J. Lee, *Nature Comm*, 2016

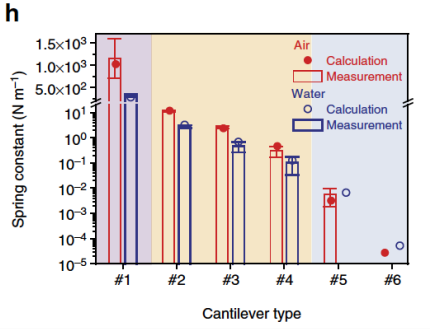
Different tip geometries and aspect ratios achievable



Can tune mechanical properties



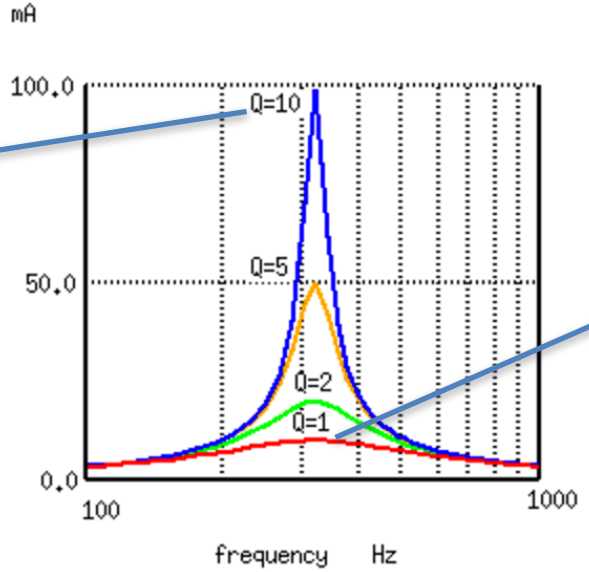




Limitation: not a scalable procedure

Q-Factor

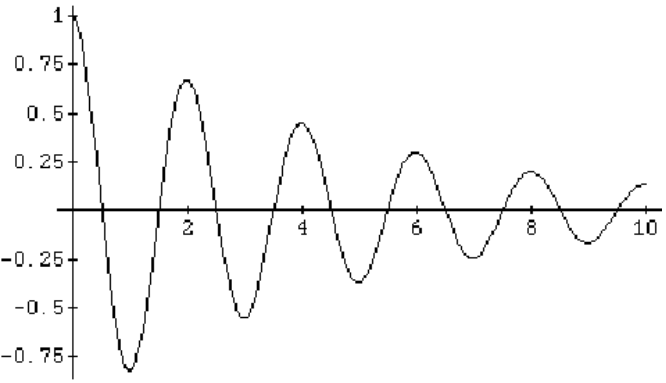
- Describes the ratio of energy stored to energy lost per cycle at resonance



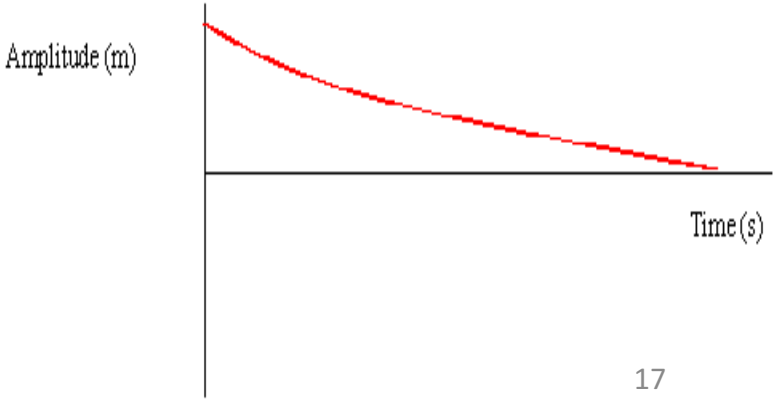
High Q : More energy stored than lost

Low Q : More energy lost than stored

High Q = Underdamped System



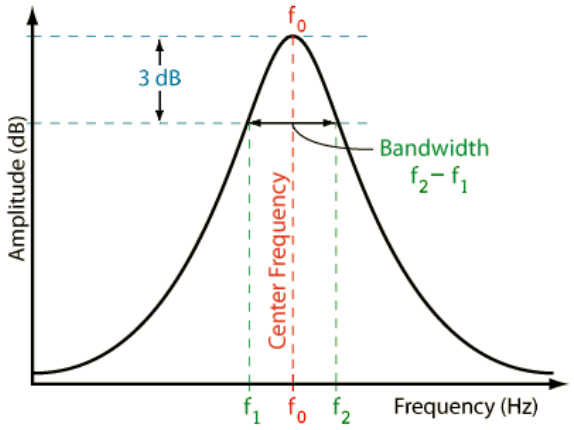
Low Q = Overdamped System



1. All About Circuits, Chap 6
2. University of Massachusetts
3. Antonine Education, Oscillati

Q-Factor

Proportional to: $Q = \frac{f_o}{B}$

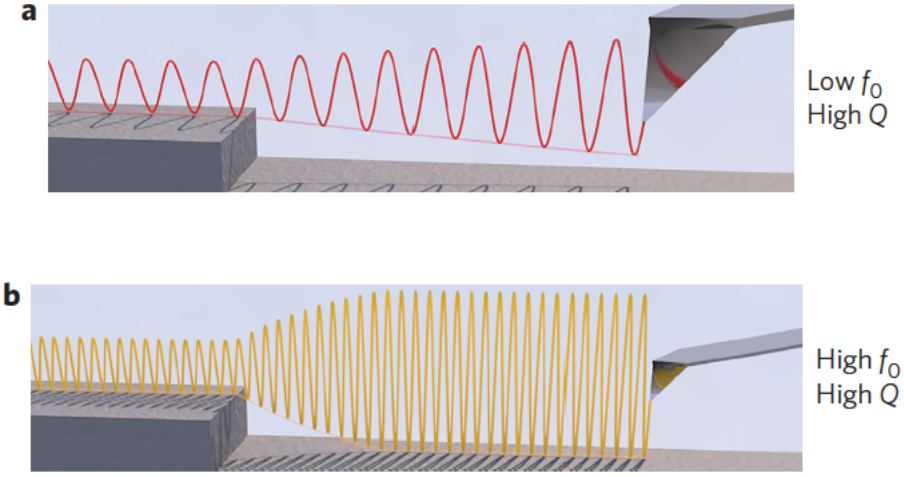


f_o **:** Frequency where maximum amplitude is observed

B: Bandwidth, range of frequencies for which the oscillator resonates

Why does this matter in AFM?

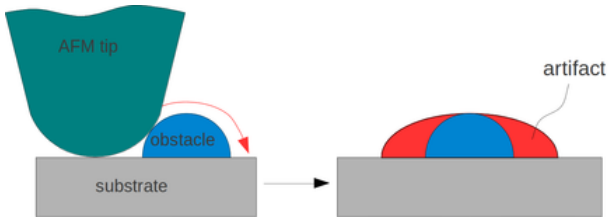
Q and f_o determine how quickly a topographical change is detected and subsequently measured



Want this

1. Tontechnik-Rechner-Sengpielaudio
2. J. Adams, *Nature Nanotech,* 2015

Shortcomings of AFM



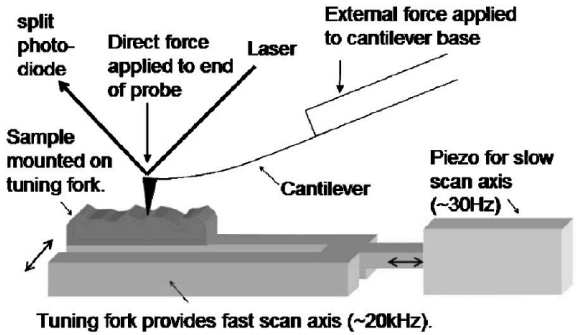
1. Artifacts on sample caused by worn AFM tip

Solution: Change mode (i.e. from contact to tapping mode) to minimize tip damage or change tip

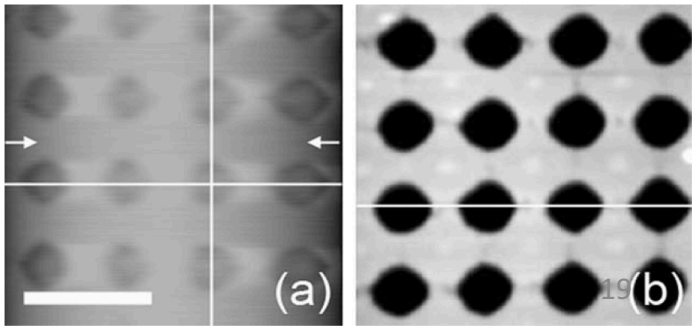
Pro: Minimizes sample damage, tip wear, and artifacts because reduced tip sample interaction time (1 µs vs 1 ms)

2. Slow scan rate (12 frames/s) and small image area (250 x 250 nm)

Solution: VideoAFM or High Speed AFM (HSAFM)



Con: Not good at imaging topography that changes rapidly



1. *Atomic Force Microscopy, Wikipedia, 2017*
2. A.D.L. Humphris, *Appl. Phys. Lett., 2005*
3. J.K. Hobbs, *The Analyst, 2005*