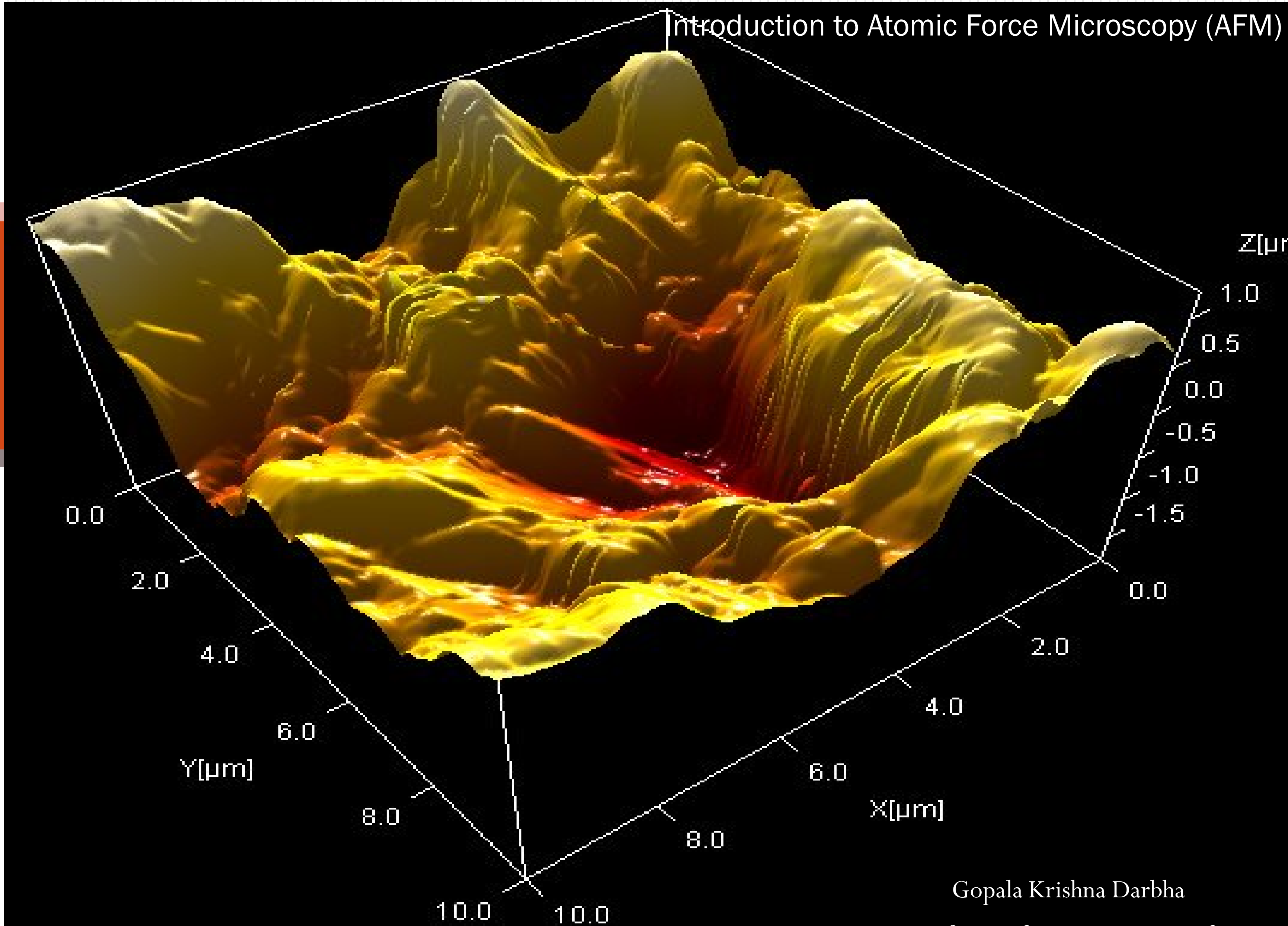
# Introduction to Atomic Force Microscopy (AFM)

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# Outline

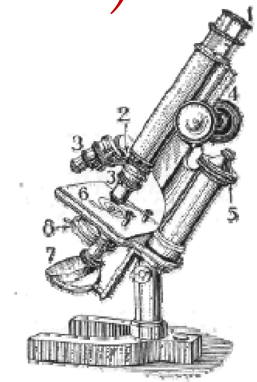
- Scanning Probe Microscopy
- Atomic Force Microscopy

  -General set-up & operation modes
- Applications

  -Imaging mode

  -Force-distance mode
- Sample preparations
- Conclusion

# Scanning probe microscopy (SPM)



~1600 Light Microscope

1938: Transmission Electron Microscope

1964: Scanning Electron Microscope

1982: Scanning Tunneling Microscope

1984: Scanning Near-field
Optical Microscope

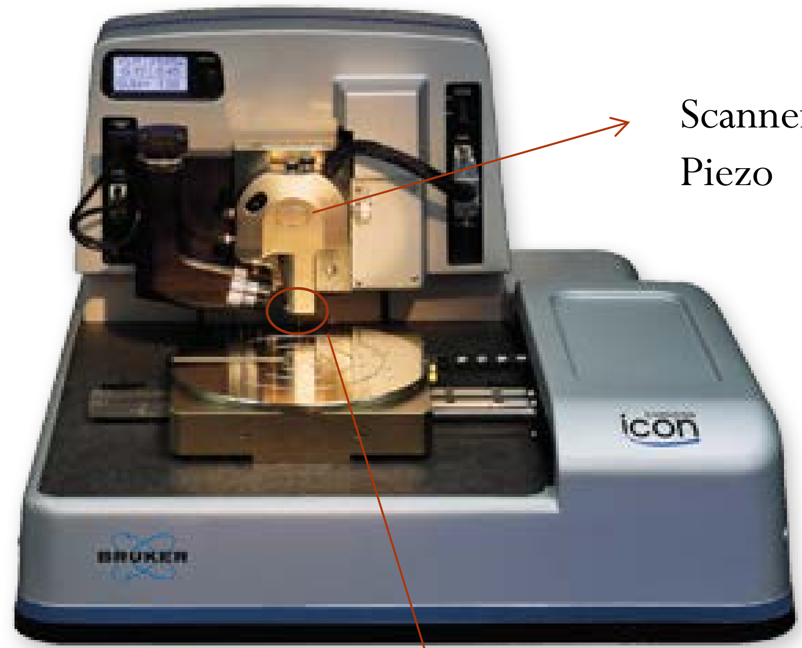
1986: Atomic Force Microscope
- magnetic force, lateral force, chemical force...

When a conducting tip is brought very near to the surface to be examined, a bias(voltage difference) applied between the two can allow electrons to tunnel through the vacuum between them. The resulting *tunneling current* is a function of tip position, applied voltage, and the local density of states (LDOS) of the sample (Resolution: x-y:0.1 nm)

Gerd Binning and Heinrich Rohrer (1981) Nobel prize

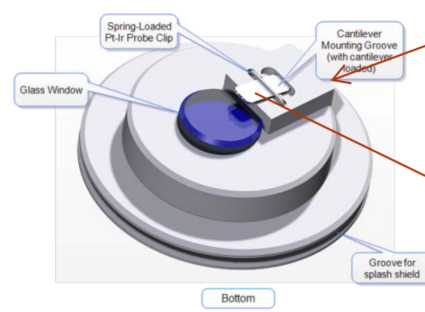
# Impression..

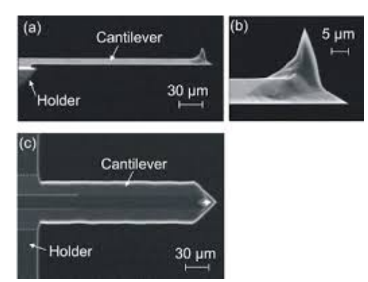

Typical resolution:
X-Y: 1 nm; Z: 0.1 nm




Scanner/
Piezo

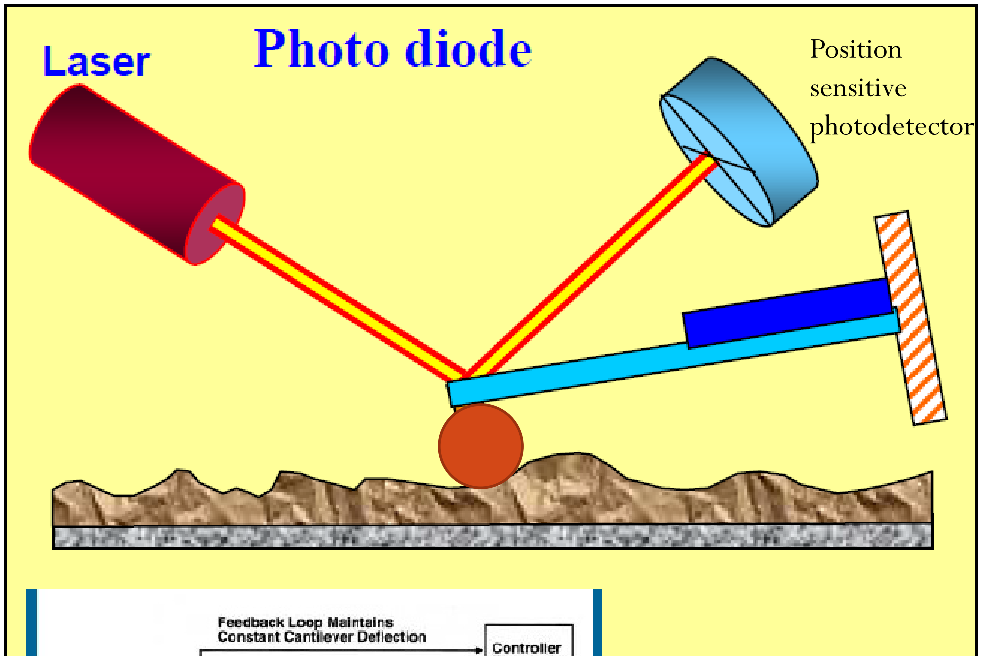
Cantilever holder

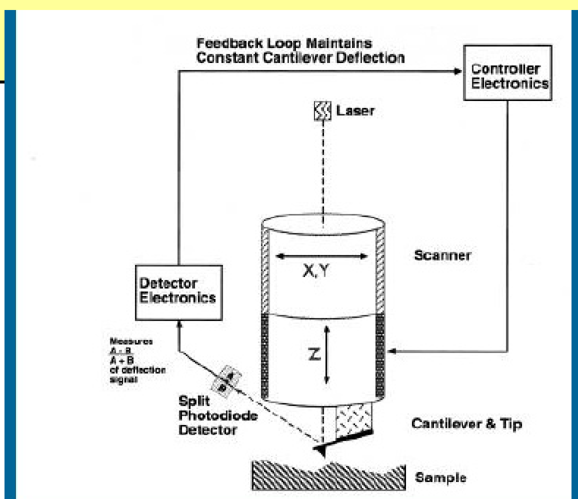


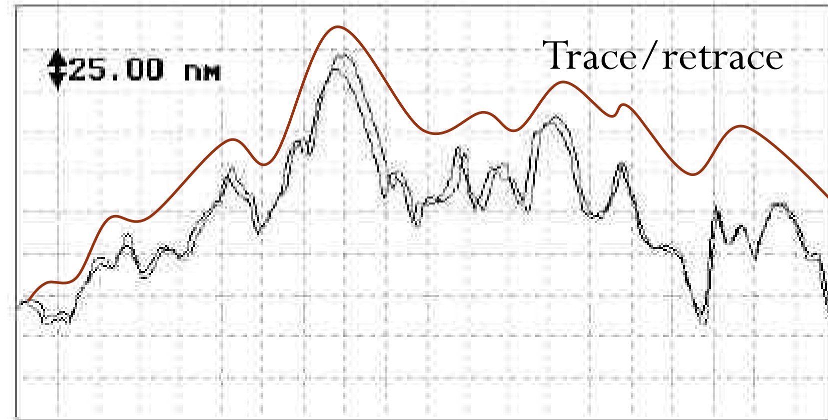



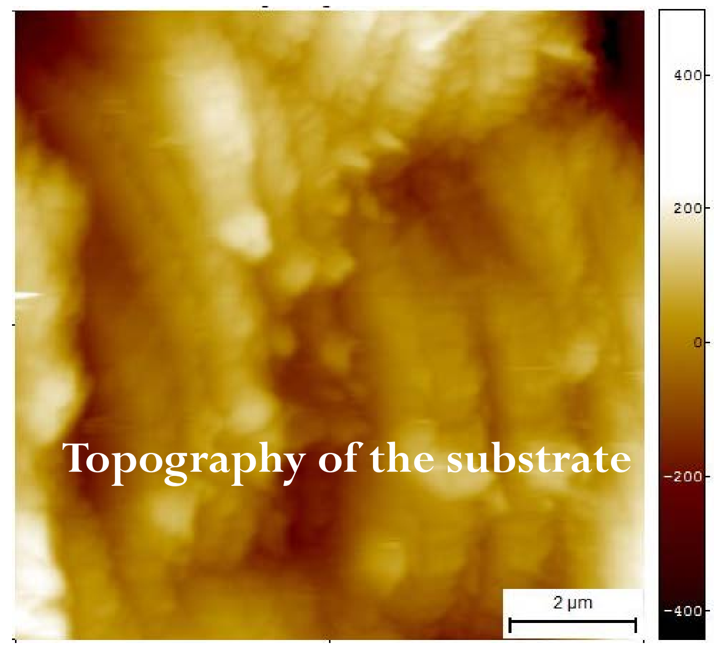

Using Dimension 3100 with Nanoscope IV controller (open-loop), possible scan size is 100 x 100 $\mu m^2$

# How AFM works?



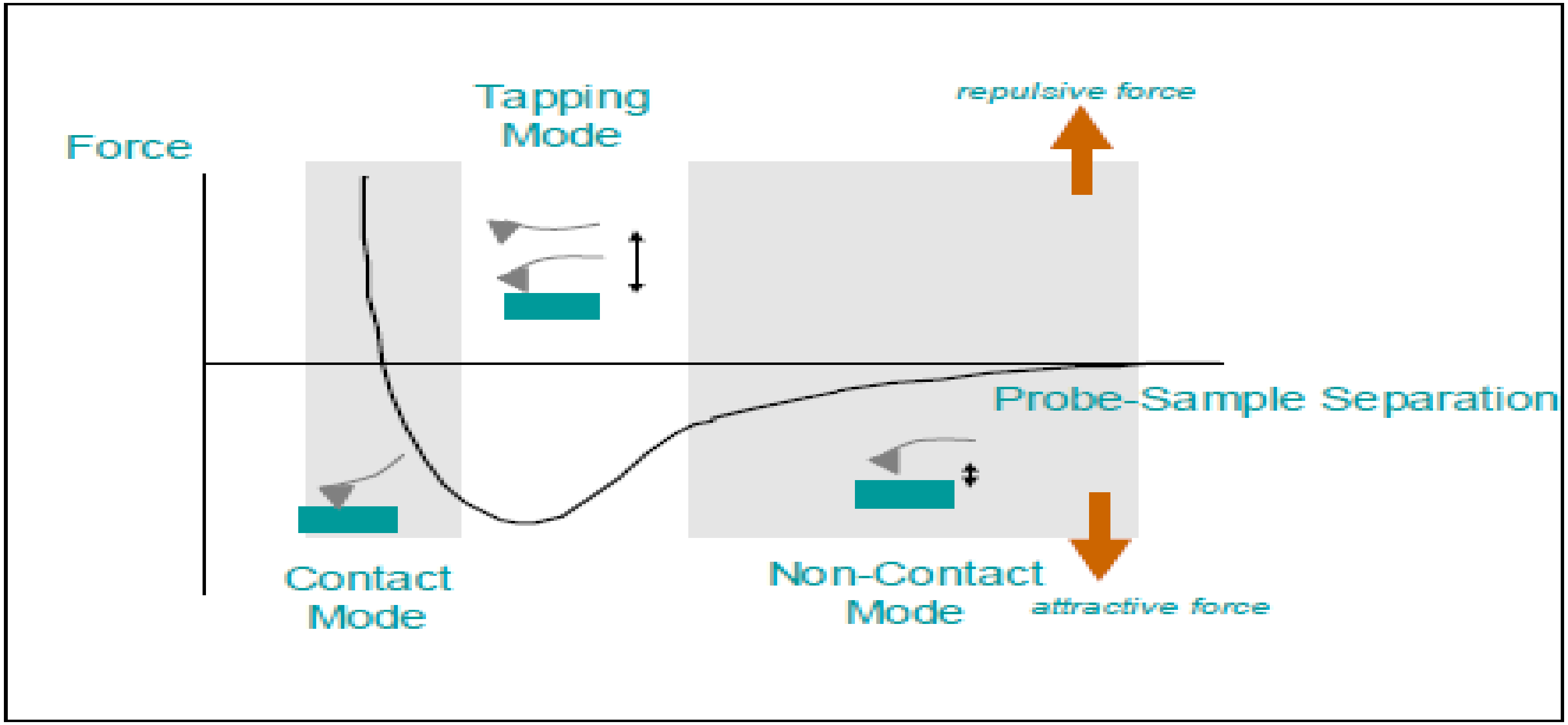




Hooke's law

F=-k.x

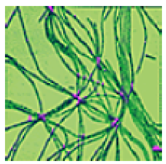
# AFM operating modes

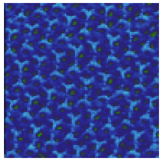
- Topographic modes:
  - Contact mode (Static mode) (<0.5 nm separation distance)
  - Tapping/intermittent mode (Amplitude modulation) (0,5-2 nm)
  - Noncontact mode (Frequency modulation) (0,1-10 nm)

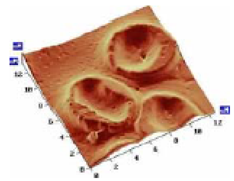


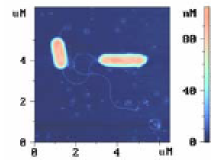
# Applications of AFM

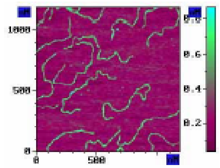
## Lifescience


Actin filaments

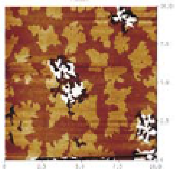

Proteins

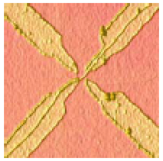

Erythrocytes

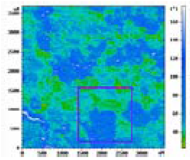

Bacteria

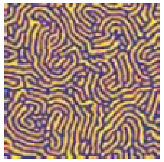

Linearised DNA

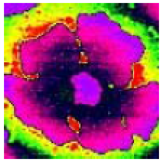
## Materials and Surface Science


Organic film

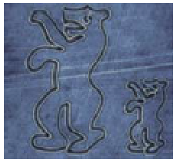

Transistor


Ferroelectric domains

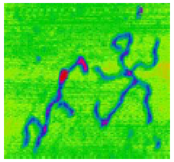

Triblock co-polymer film


Polymer

## Nanolithography & Nanomanipulation


Polymer film engraving


Anodic oxidation

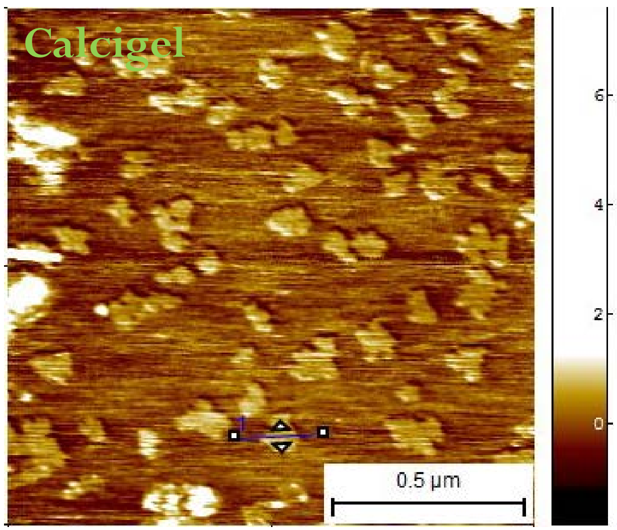

DNA on mica

http://www.jpk.com/
http://www.veeco.com/nanotheatre/
http://www.ntmdt.ru/Scan-gallery/

# Applications in our context

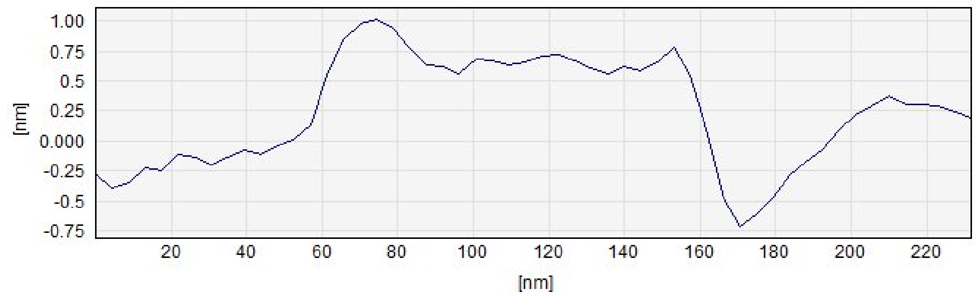
- Characterization of clay/nanoparticle size/morphology
- Topography of rock and mineral surfaces
  - Mineral dissolution, precipitation etc.
- Forces between surfaces using colloid probe technique
  - Mineral colloids
  - In the presence of electrolytic media

# Clay particle characterization

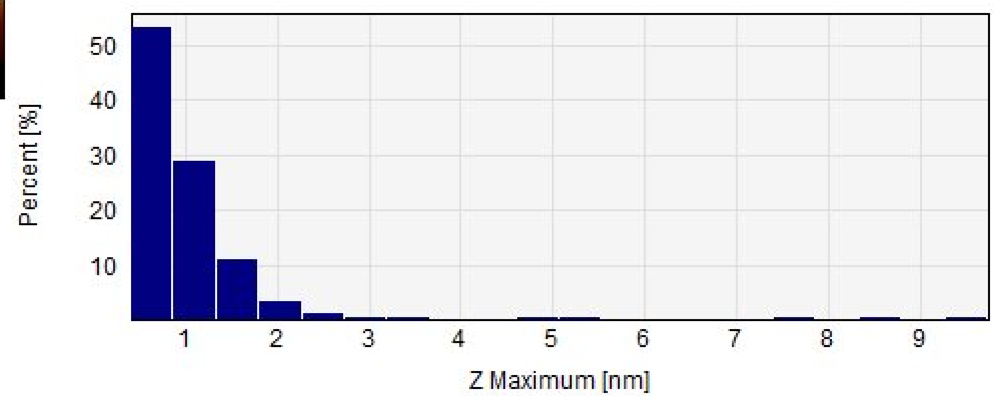



Topographic image

Parameters obtained:
Size, shape, height distribution




Height profile along the highlighted line

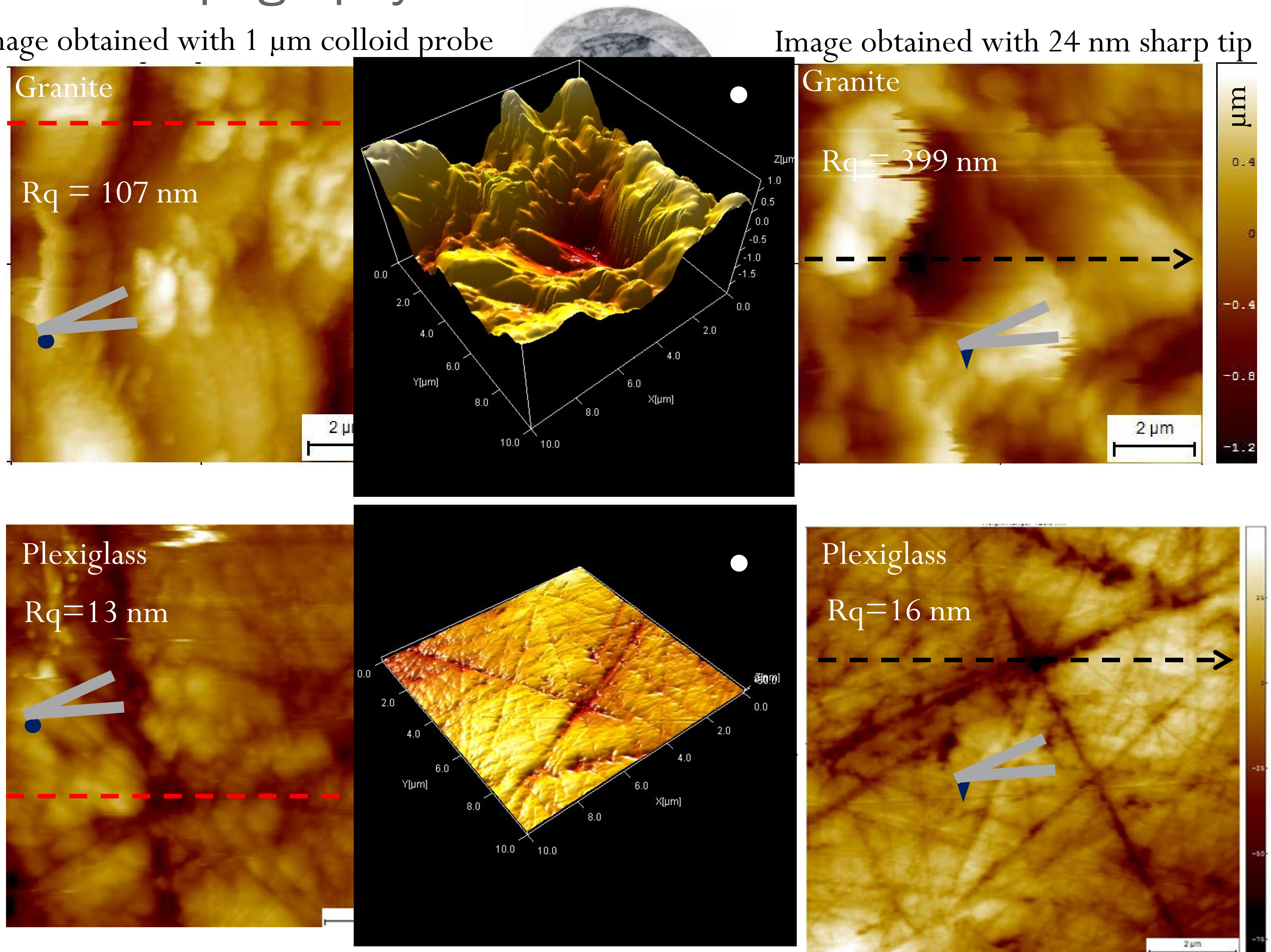



Particle height distribution histogram

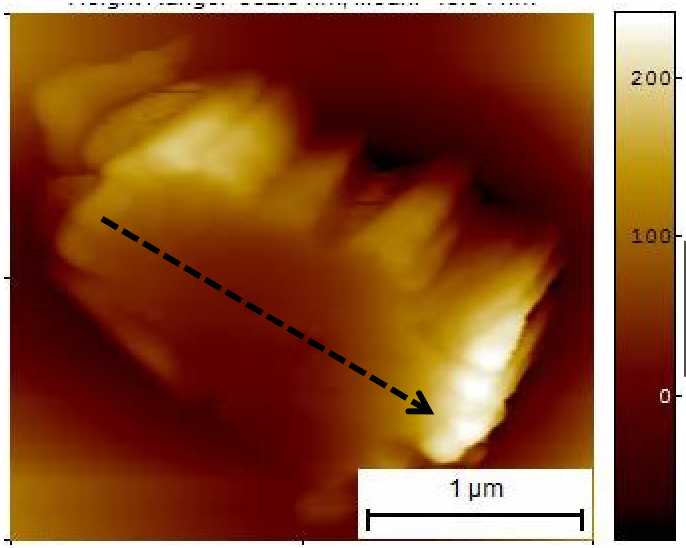
# Topography of rock and mineral surfaces

Image obtained with 1 μm colloid probe

Image obtained with 24 nm sharp tip

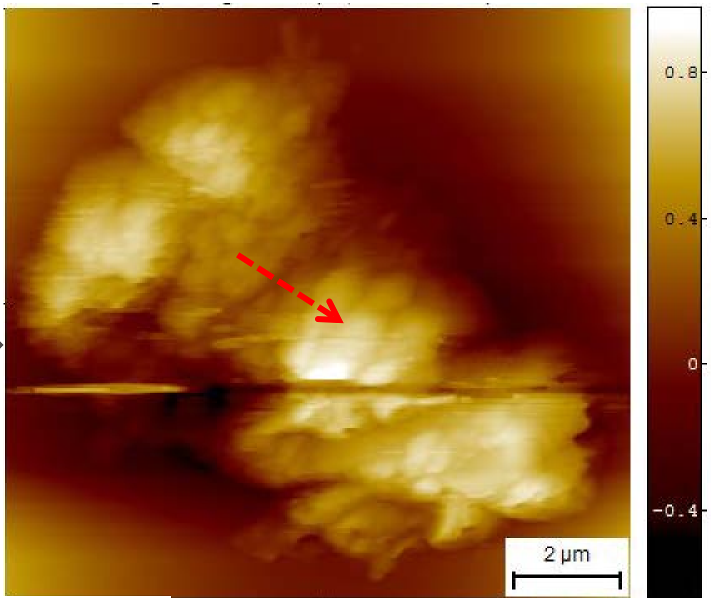


# Magnetite surface topography change with $NaBH_4$

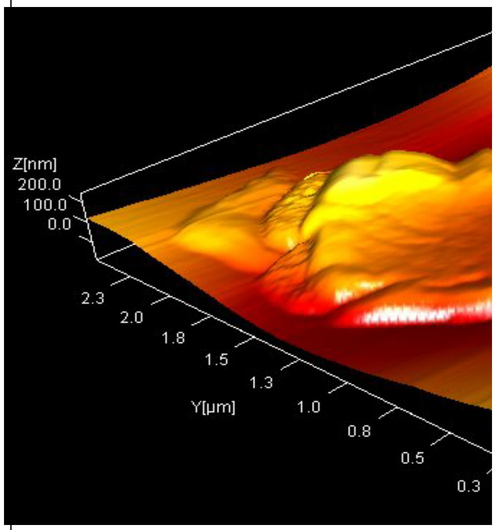


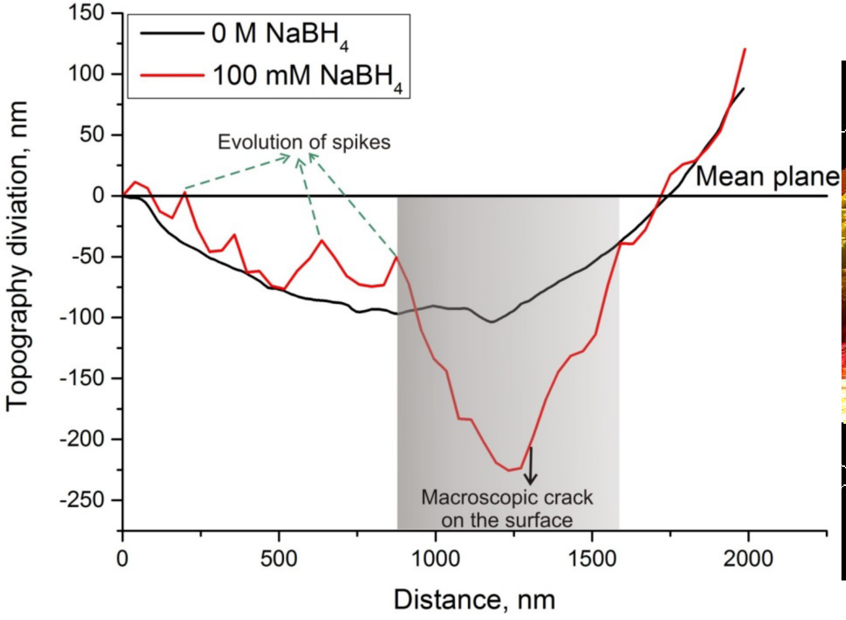

Magnetite in milliQ water

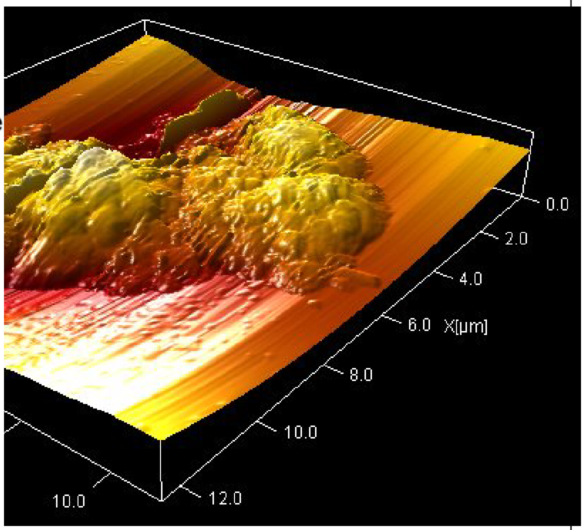
$NaBH_4$



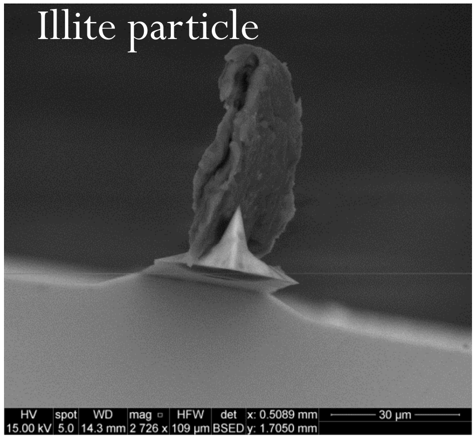

Magnetite in 100 mM $NaBH_4$

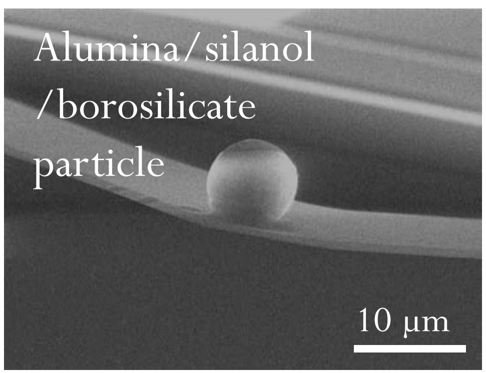


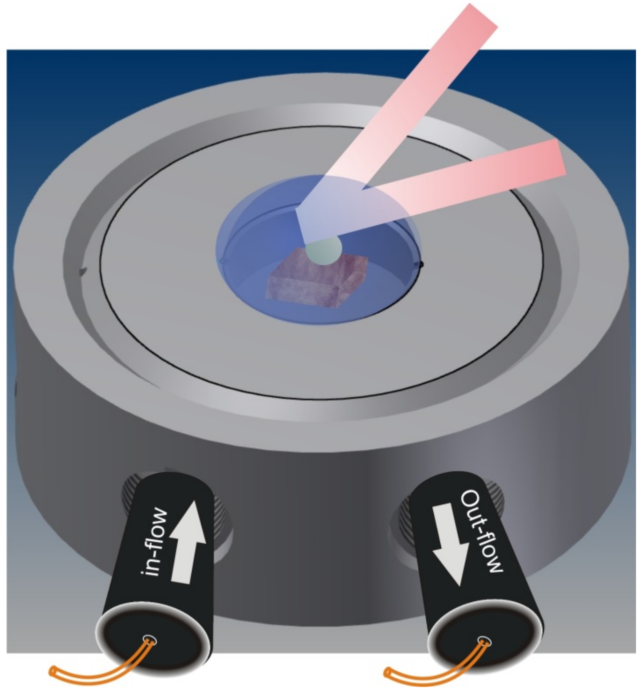



# Force measurements

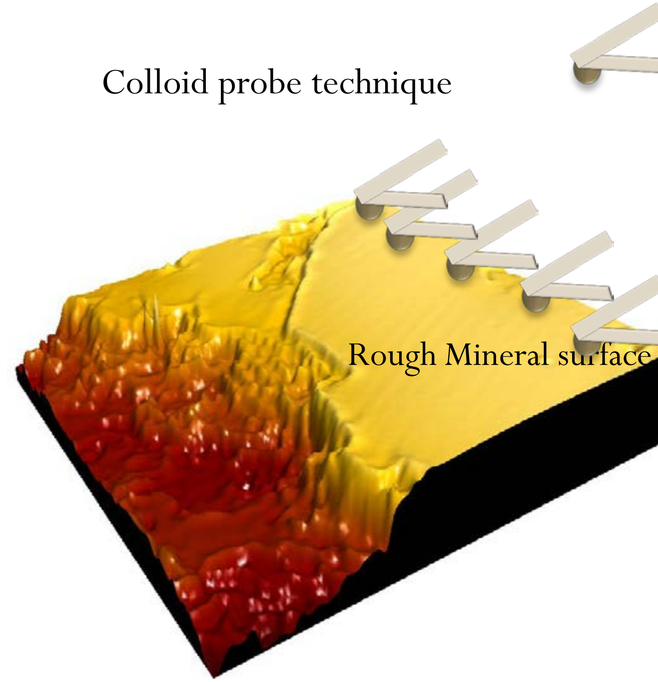



SEM of $Al_2O_3$ particle attached to cantilever




Home made continuous flow through-fluid cell for AFM experiments




Parameters measured:

1. $F_{Adhesion}$
2. Snap-in force
3. Force-volume measurements

**Applying colloid probe technique to measure the surface forces between colloid particles (polystyrene, $Al_2O_3$) and rough minerals**

# Colloid probe technique

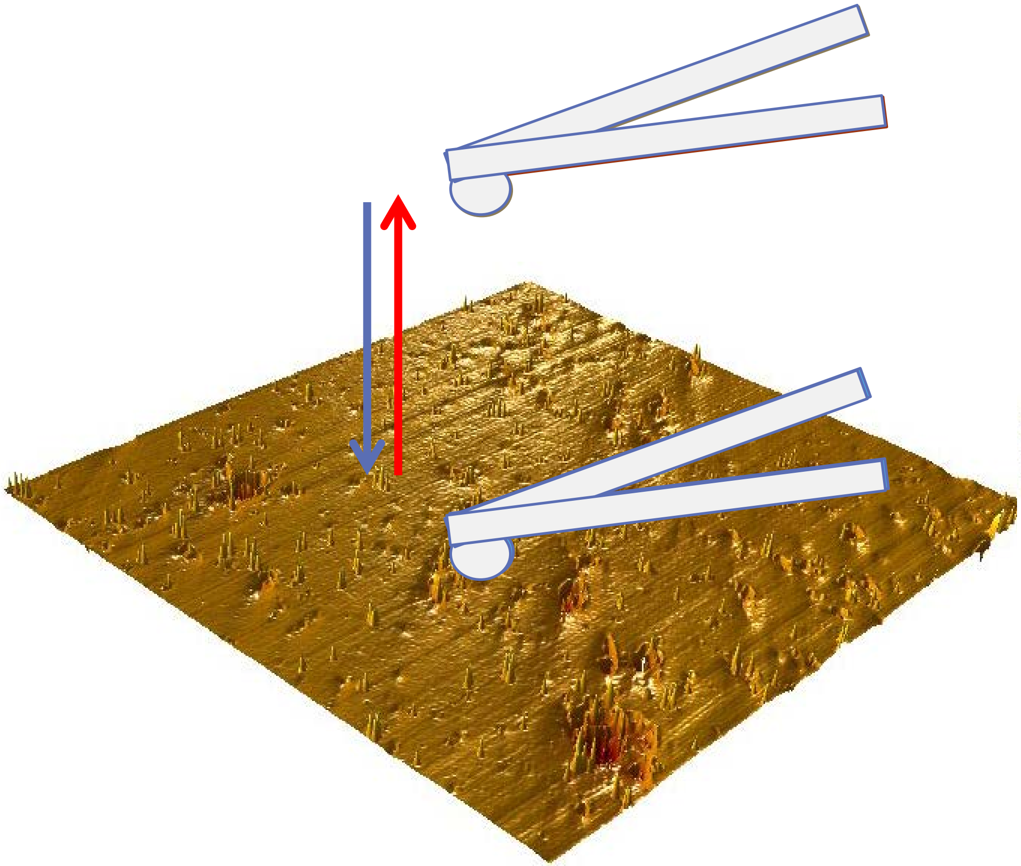
Parameters obtained:

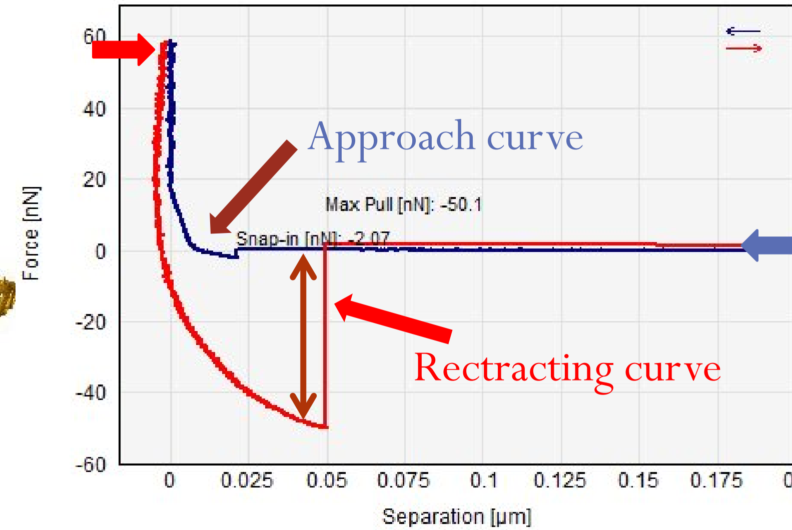
Surface potential

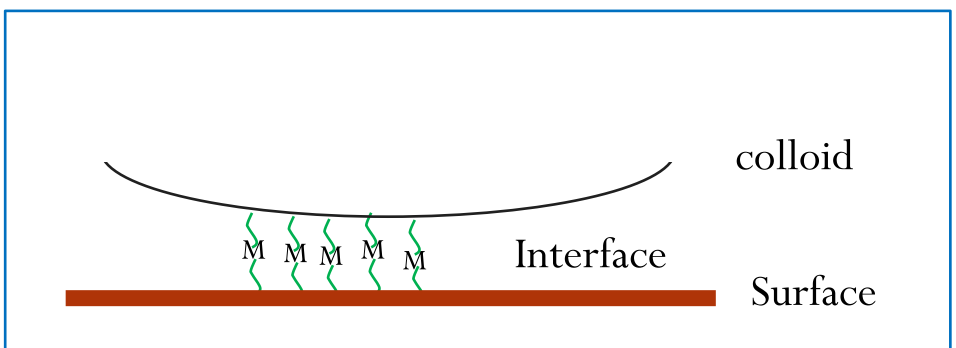
Young's modulus

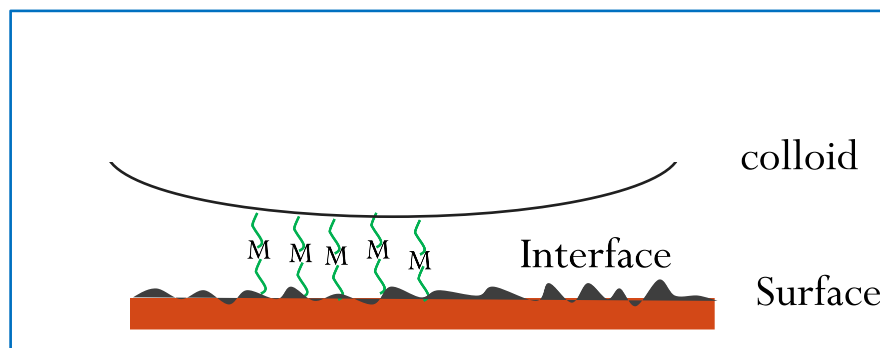
Hamaker's constant

Dissipated Energy etc..

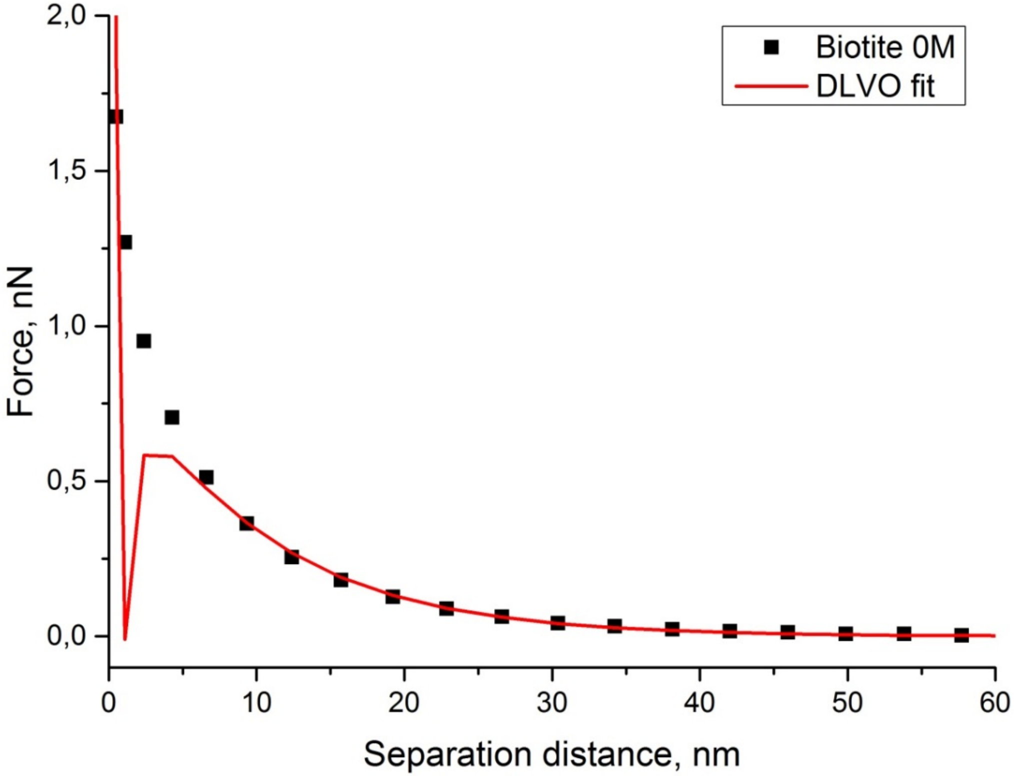


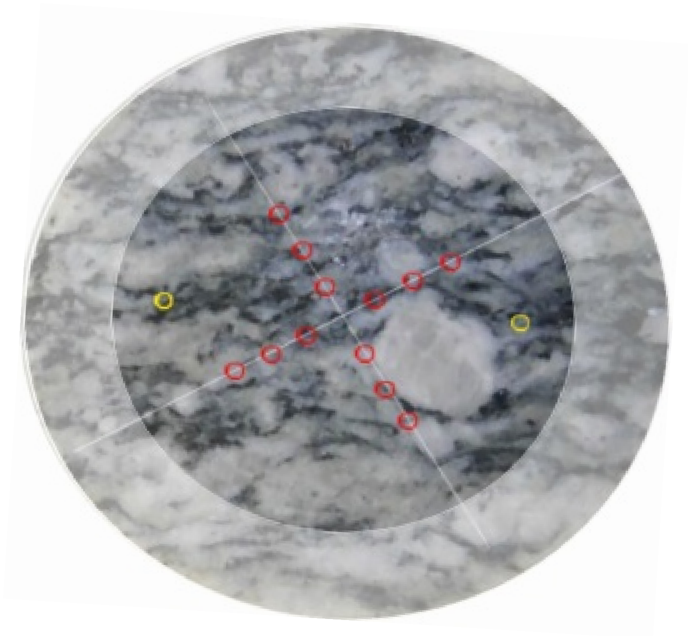


# Fitting of AFM force curve with DLVO equation to obtain unknown potential of substrate



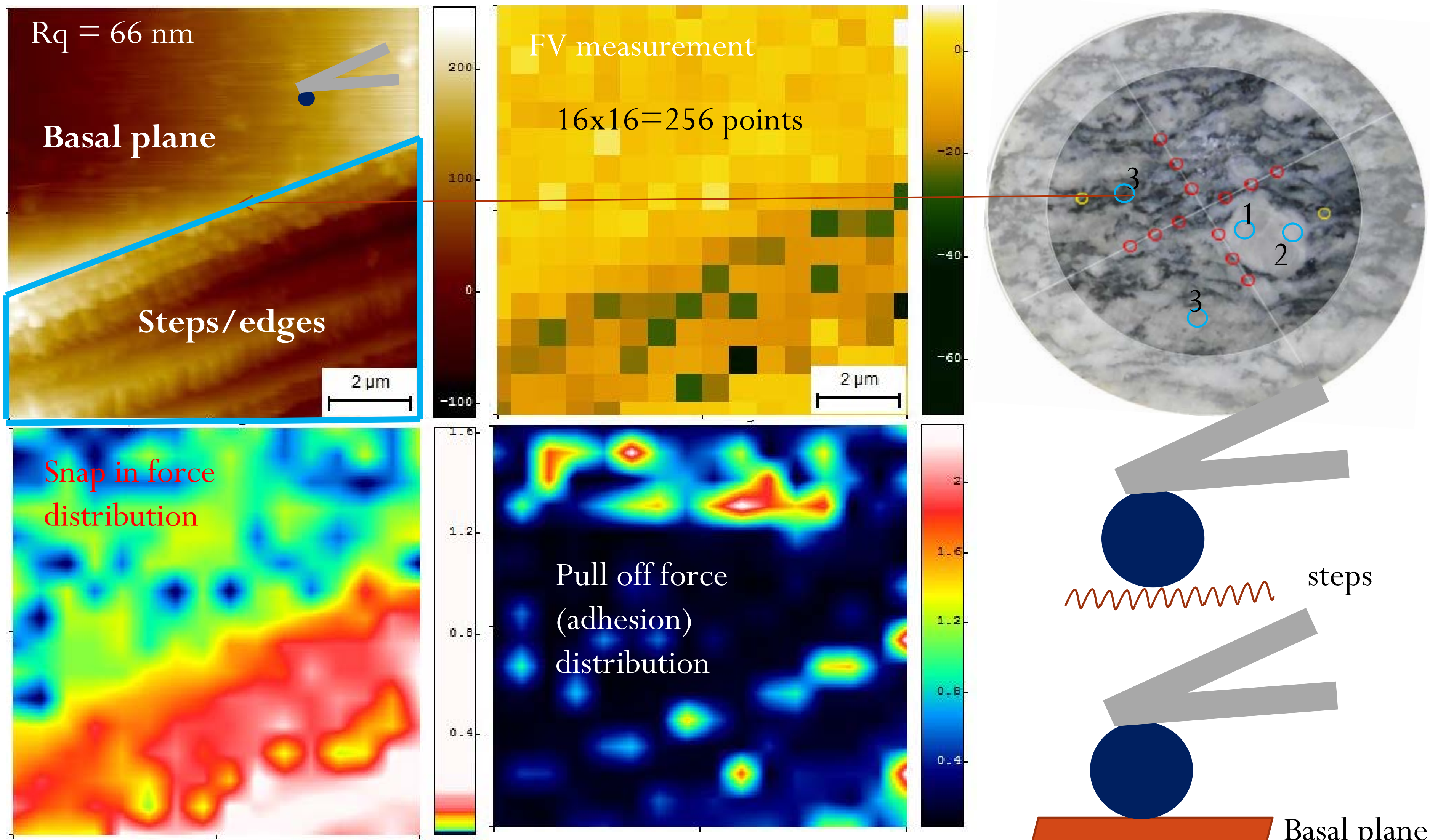



Zeta potential of colloid (measured using zeta sizer) = -32.4 mV

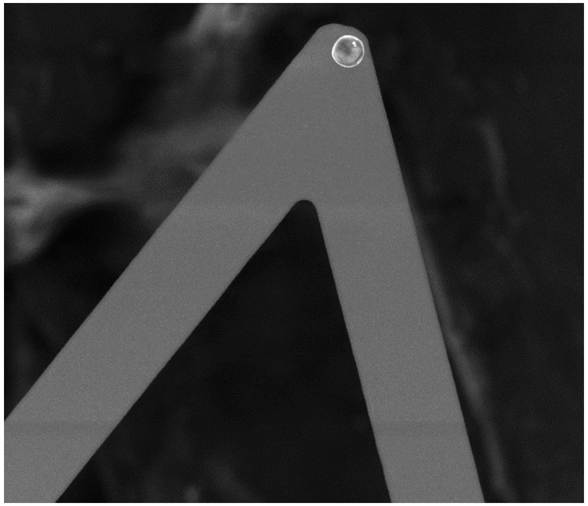
Zeta potential obtained after fitting the AFM approach force curve using DLVO equation = -74.2 mV

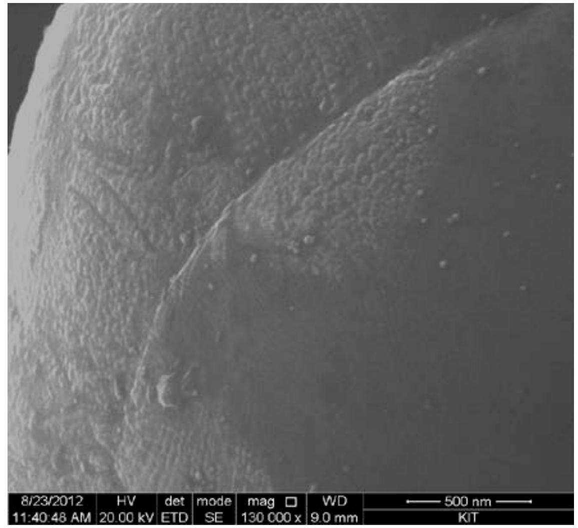
# Force-volume measurements (Force+imaging)

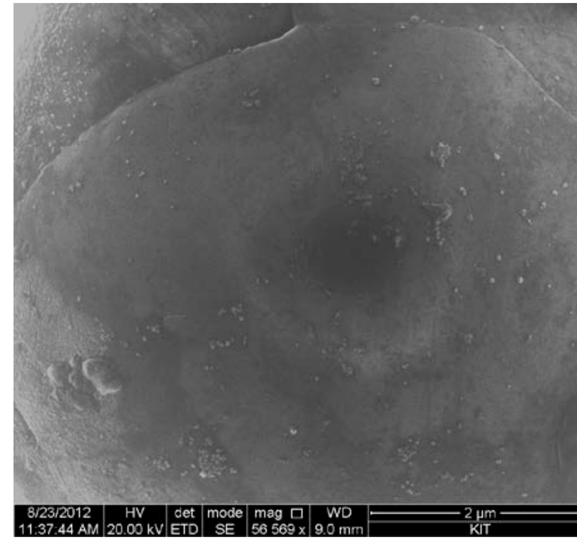
## Biotite/Dark minerals (mica) in 1 mM NaCl at pH 5

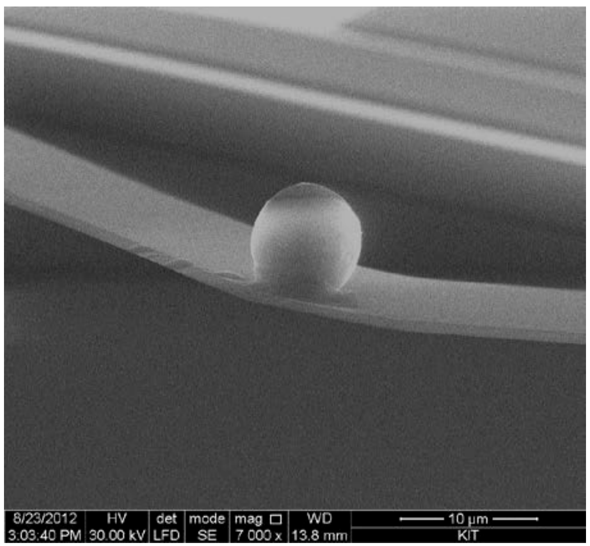


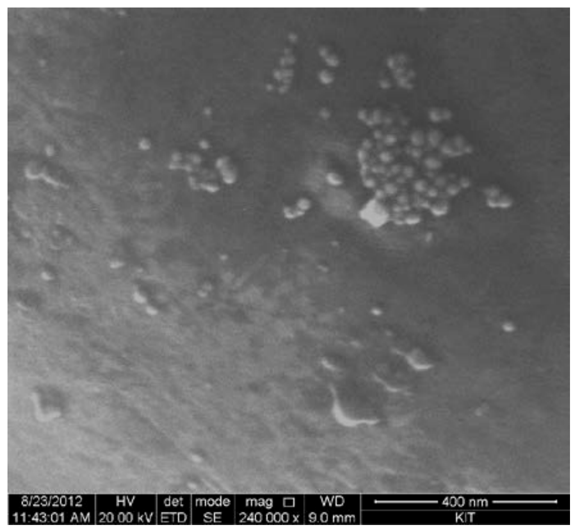
SEM of $Al_2O_3$ particles at high magnification

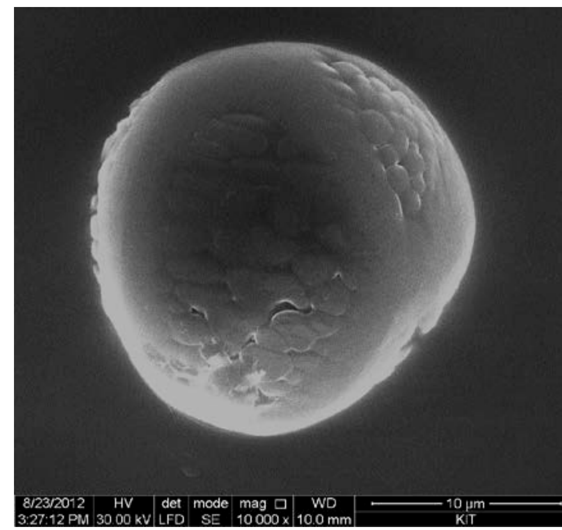




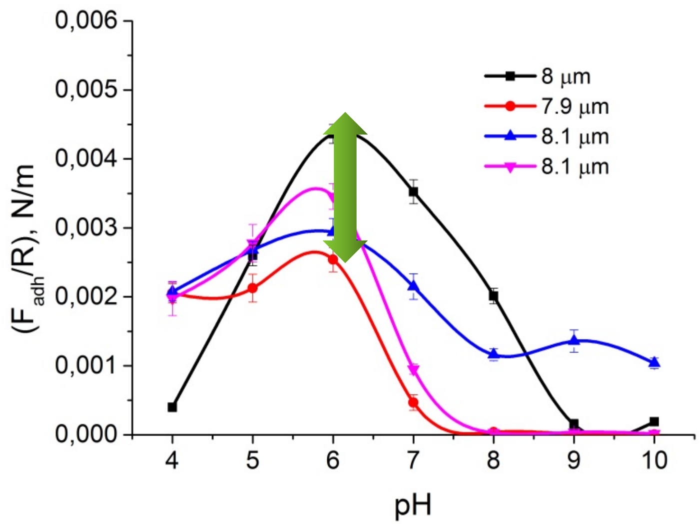



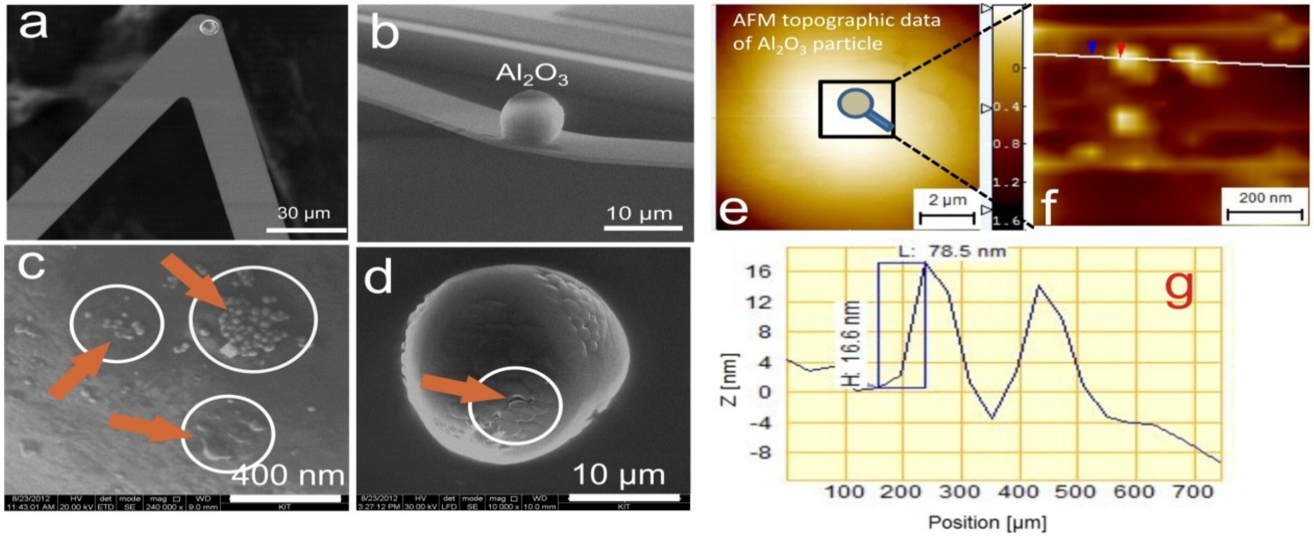




The SEM images clearly demonstrate: at higher magnification, there is a sub-micron scale roughness contribution from the colloids that could vary surface forces measured at the interface.

Different $Al_2O_3$ colloid probes Vs Biotite at varying pH



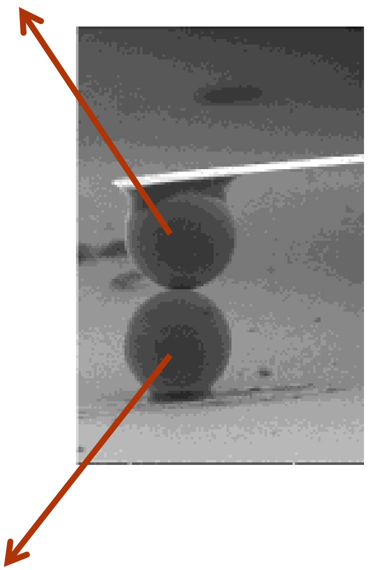

Adhesion forces between alumina particle and biotite



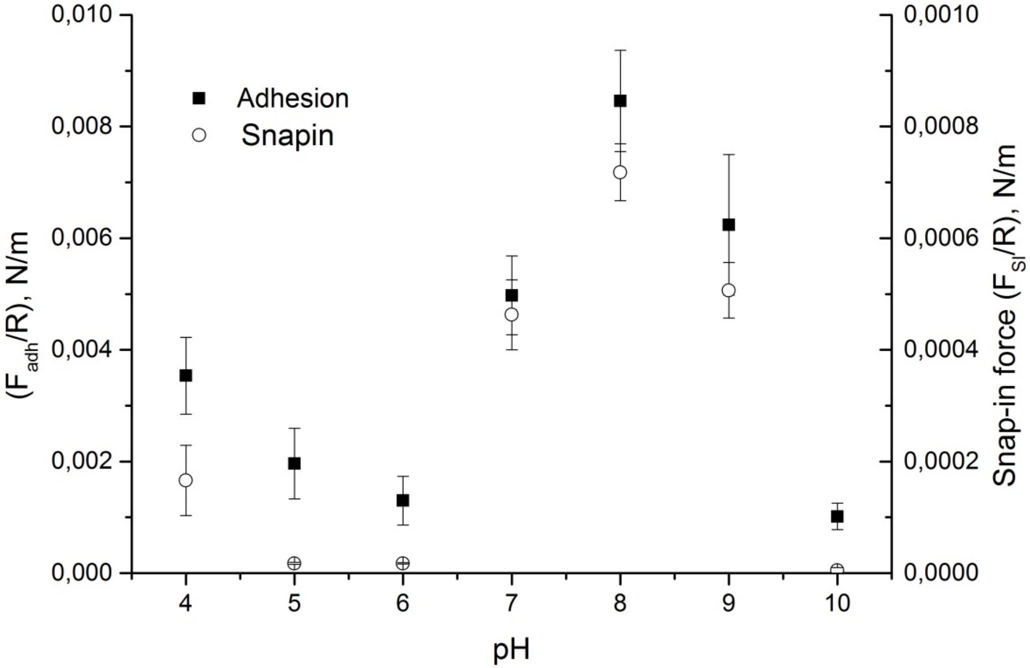

Roughness features on $Al_2O_3$ particle of <50 nm have pronounced effect on adhesion forces

# Interaction between $Al_2O_3$ colloid probe and $Al_2O_3$ particle as a function of pH at 1 mM NaCl

Particle on cantilever



Particle on silicon wafer or other immobile substrate




The point of zero charge of $Al_2O_3$ particles is at pH 8

# Sample preparation

- Clay colloid characterization:

0,01 g / 10 mL

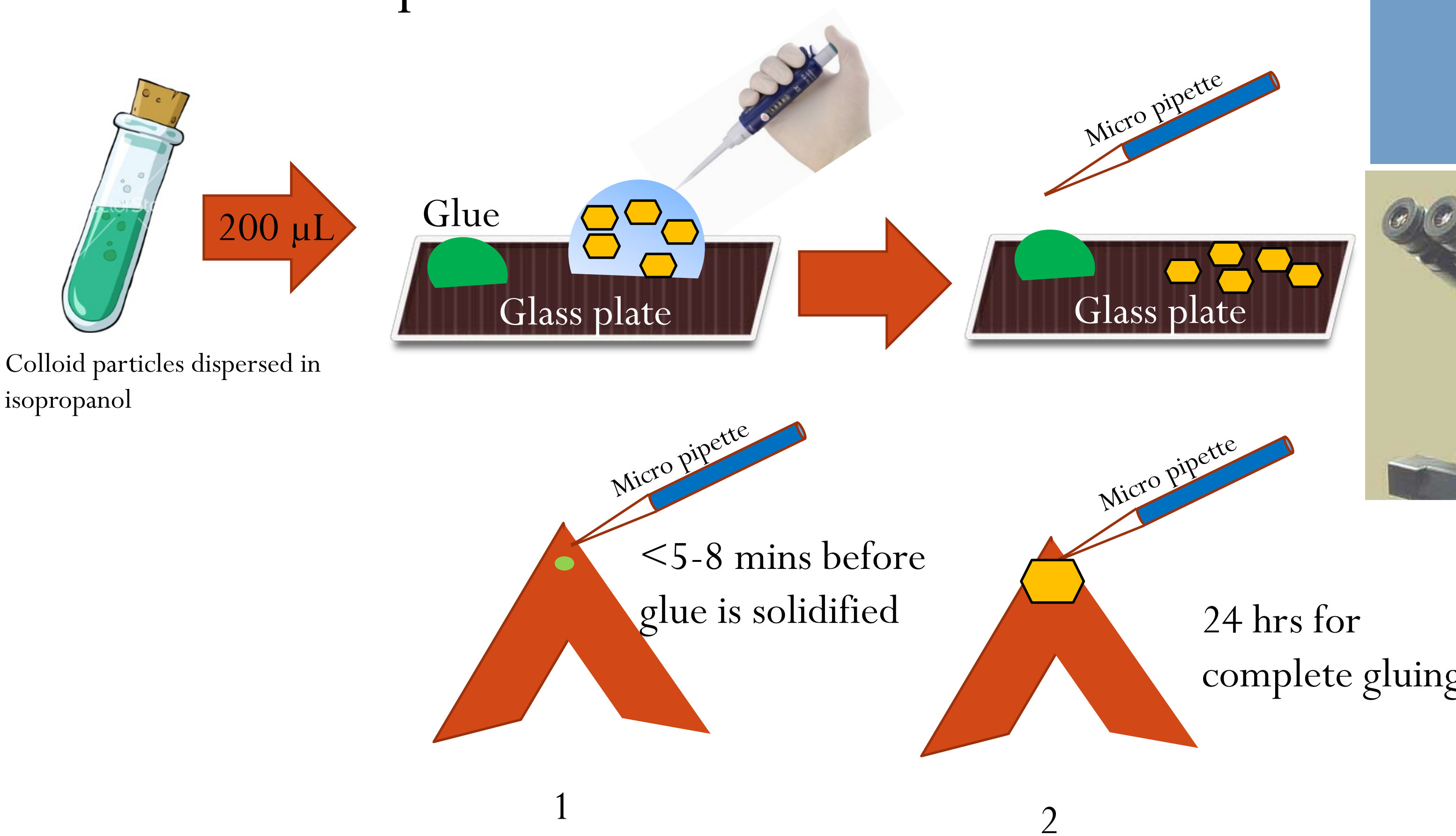
Clay suspension

50 µL


Allow it to dry for measurements

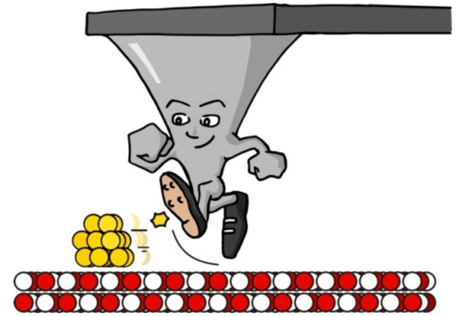
# Sample preparation

- Colloid probe for force measurements:

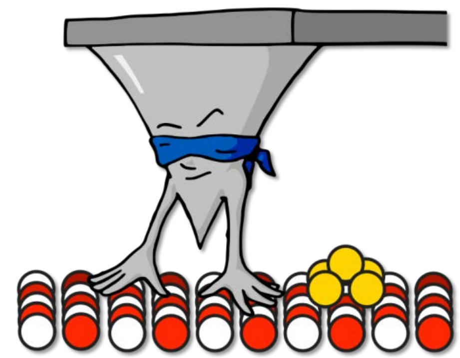



The samples are characterized in SEM before

# Conclusions….



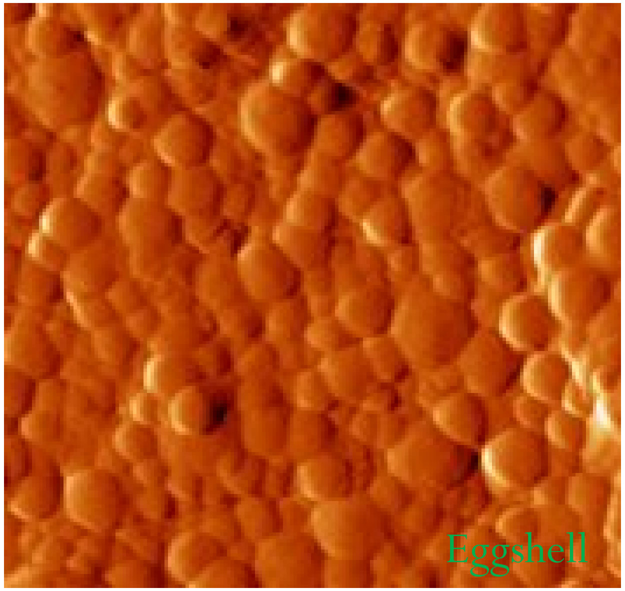
1. AFM provides nano and submicron scale topogrpahic information
2. It is versatile technique to measure the forces between surfaces
3. Finally…AFM is a blind technique



# Limitations…

1. Sample should be steady, e.g. swelling clays: possibility of contamination to the tip
2. Substrates should not be too rough
3. The suspensions should not be too viscous
4. Particles attached should be > 4 μm
5. Etc….


Human hair


Eggshell