Some Basic Principles of AFM Sample Preparation

Song Xu
Application Scientist
Agilent Technologies, Inc.
Basic principle of sample preparation I: separation

Snow flak and packed snow

5nm and 10nm gold nanoparticle on glass slide
Techniques of separation

• Lower concentration
• Charge based separation: a molecular level separation.
• Solvent dispersion: for nanoparticles, colloidal, nanotubes.
• Sonication: extra energy to help.
• Use of dry air jet stream.
Concentration: the math of monolayer coverage

Oxidized nanotubes on mica
Basic principle of sample preparation II: choose a clean and flat substrate
Gold nanoparticles on mica vs. Platinum nanoparticle on fuel cell membrane
An atomically flat substrate: Muscovite Mica

\[ \text{[KAl}_2\text{(OH)}_2\text{(AlSi}_3\text{O}_{10})] \]

- Layered structure of Al, Si, O, & K
- Mechanically cleaved to produce clean, atomically flat surfaces
- Negative surface charge in pure water – strong electrostatic interactions
- Model substrate for adsorption studies
HOPG: Atomically flat surface with conductivity

- Upper layer (Å)
- Lower layer (Å)

HOPG atomic resolution

HOPG 500 x 500nm
A versatile substrate: Au(111)

Thermo evaporated gold on mica
Cleanness of substrate

Au(111) after exposed in air for

After hydrogen flaming
Glass slides

5 nm and 10 nm gold on glass slides
Sample Preparation Principle III: immobilization
Functional Modification on Silica surface

- End group, e.g., $\text{CH}_3$
- $-(\text{CH}_2)-$

- Silicon substrate (100)
Functional modification on Au(111): *n*-alkanethiols/Au(111) self-assembled monolayer

Surface structure of alkanethiols/Au(111). Sulfur (gray circles) binds at the triple hollow sites of Au(111).
Thiol self-assembled monolayer on Au(111) Highly ordered functional surface with internal height reference
Surface functional group selected immobilization

Formation of Protein Nanosensors via Covalent Binding

1. Wash with water
2. Injection of Normal Rabbit IgG
3. Wash with Tween 20
Sample Preparation Principle IV: avoid use fixing material that cause drifting, creeping, contamination and peeling.
BCP Toluene exposure

Before

after
Example of Sample with unusual shape

Cleaved calcite dissolve in water
Conclusion

• Principle I: separate individual species and features from each other

• Principle II: choose a flat and clean substrate

• Principle III: immobilize the sample onto the substrate

• Principle IV: avoid any source of drifting, creeping and peeling.