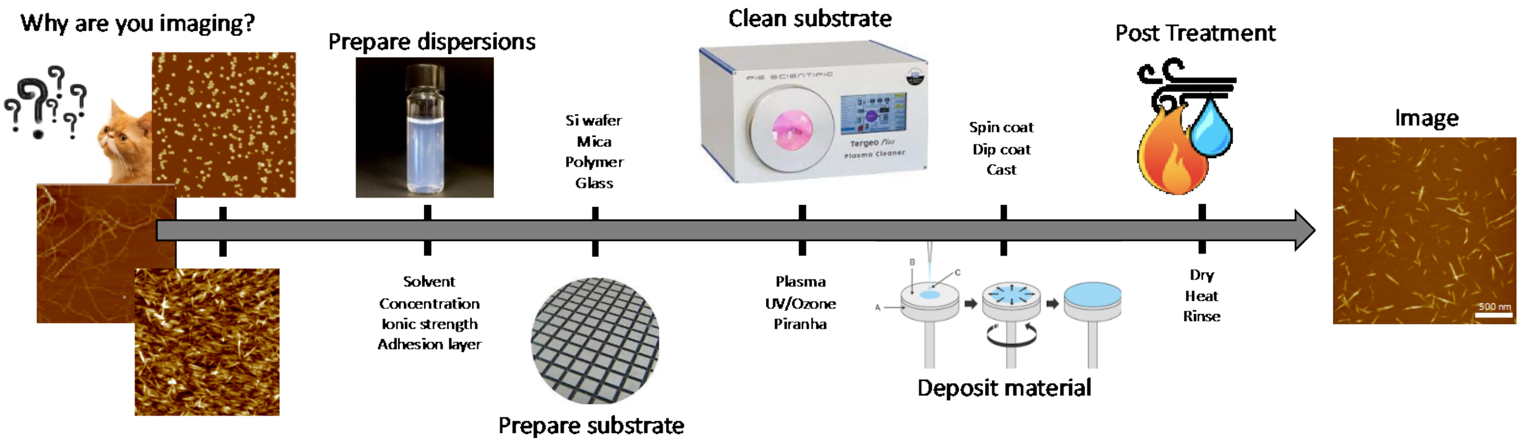
Treesearch AFM 2022 – Sample Preparation

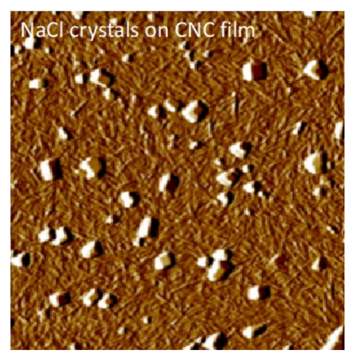


Why are you imaging?

Before you start preparing you samples, you need to decide the purpose of using AFM. Do you want to measure particle size and polydispersity? Are you interested in thin film morphology and thickness? Do you want to measure the length of fibrils? Are you preparing model films for force measurements? These are important questions that must be answered before you start, as they determine how/what needs to be prepared for accurate AFM measurements.

Prepare dispersions

In most cases, AFM samples are prepared from solutions, suspensions or dispersions. The solvent, concentration and ionic strength all play a role during material deposition. Ideally material should be well dispersed and stable in an appropriate solvent as aggregates can lead to non-uniform deposition, thus making imaging more difficult. Depending on your particular material, the ionic strength will play a role in stability and aggregate formation and must be accounted for. Moreover, in some deposition procedures evaporation leads to salt crystal formation (at high ionic strengths) that can make imaging more difficult.



For particle size analysis, particles should be individualized on the surface and thus dispersion concentrations should be very low (0.01 wt% or less). It is often useful to prepare a concentration series to determine the appropriate concentration for your material and substrate. Additionally, for charged materials adhesion layers (oppositely charged polymers) are deposited prior to material of interest and thus these solutions must also be prepared. For example, CNCs are negatively charged particles and thus do not readily adsorb to Si substrates. Therefore, cationic polymers (PEI, PAH, PDADMAC) are deposited as an adhesion layer to improve CNC adsorption. Some materials take time to completely disperse (i.e. overnight) so ensure dispersions are prepared well in advance.

Prepare Substrate

Regardless of the substrate selected it must be flat, uniform and clean. Common AFM substrates are Si wafers and mica, but many others are utilized. For example, Au or SiO_2 sensors for QCM-D or SPR can be imaged via AFM. Prior to material deposition mica and Si wafers must be cut/cleaved to appropriate size for material deposition and AFM imaging. Substrate size is dependent on the specific AFM, but remember images are collected at the micron scale so large substrates are not always needed. A good size to start with is 10 mm X 10 mm. This large enough to handle with tweezers but small enough to save material.

Clean Substrate

AFM has nanometer resolution and thus contamination can ruin your images. Substrates need to be free of dust, oils (finger prints) and any other artifacts. Three common cleaning methods are plasma cleaning, UV/Ozone cleaning and piranha (H_2SO_4:H_2O_2) cleaning. Plasma and UV/Ozone are air/vacuum methods whereas piranha is a solution-based method. **CAUTION** Piranha is very dangerous. Mixing H_2SO_4 and H_2O_2 is exothermic and creates gas. Piranha reacts violently with organic matter and should not be used without appropriate training, protective equipment and waste disposal. Piranha waste has the potential to explode as organic matter can enter bottles, produce gas and build pressure. Piranha will not be covered in this course. Both plasma and UV/Ozone create radicals that react with surface contaminates to create volatile organic compounds that are removed from the substrate surface. Both are generally fast and effective cleaning methods.

Deposit Material

There is a variety of ways to deposit materials of interest. These include spin coating, dip coating, solvent casting and many others. Spin coating rapidly evaporates solvent from a dispersion and “forces” material onto the substrate. Dispersion viscosity, substrate wettability, rotation speed and rotation stages, must be controlled for spin coating to be effective. Dip coating works by submerging a substrate into a dispersion of interest and allowing the material to adsorb via surface forces (van der Waals, electrostatic double layer). Solvent casting simply allows a dispersion to dry on a substrate. Solvent casting can lead to strong capillary forces, aggregation and the coffee ring effect. Generally, solvent casting is only used for volatile solvents which have low surface tension.

Post Treatment

Depending on what environment you will be imaging your material in (ambient, liquid, vacuum), post treatment may be necessary to improve surface stability. For example, after dip coating substrates need to be dried via air/N_2 gas to eliminate capillary forces. Additionally, annealing your substrates in an oven can improve the stability of the film in liquid environments. In some cases, re-wetting the surface may remove loosely bound material that would make AFM imaging more difficult.

<u>In this Lab</u>

We will be preparing nanocellulose surfaces for AFM imaging on Si wafers and mica.

Spin Coating on mica
-CNC thin film (1 wt%)
-PAH/CNC 0.1 wt%
-PAH/CNC 0.01 wt%
-PAH/CNC 0.001 wt%

Dip Coating
-NH_2 – Si adhesion layer
-0 mM CNF 0.1 g/L
-5 mM CNF 0.1 g/L
-10 mM CNF 0.1 g/L