Plasma Polymerized Hexafluoropropylene Process Validation Using Nano Thermal Analysis

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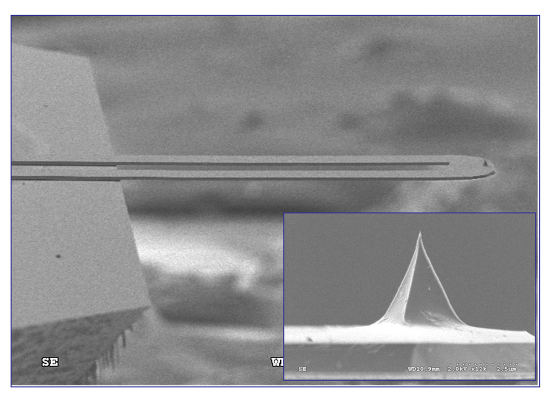
Introduction:

Plasma Enhanced Chemical Vapor Deposition (PECVD) is a rapidly growing technology providing unique thin films on a wide variety of substrates. As a plasma process the characteristics of the deposition product is influenced by the plasma parameters. In this study an unsaturated fluoroalkene, hexafluoropropylene, is deposited via PECVD on commercially available glass slides. The effect, due to the variation of the processing parameters, on the coating properties is qualified by nano thermal analysis (nanoTA). Two sets of process parameters A & B are repeated at three different deposition times 5,10, &15 minutes.



SEM image of a AFM cantilever with embedded resistive heating element

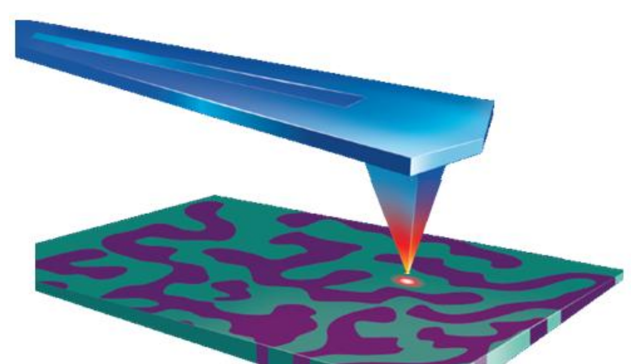
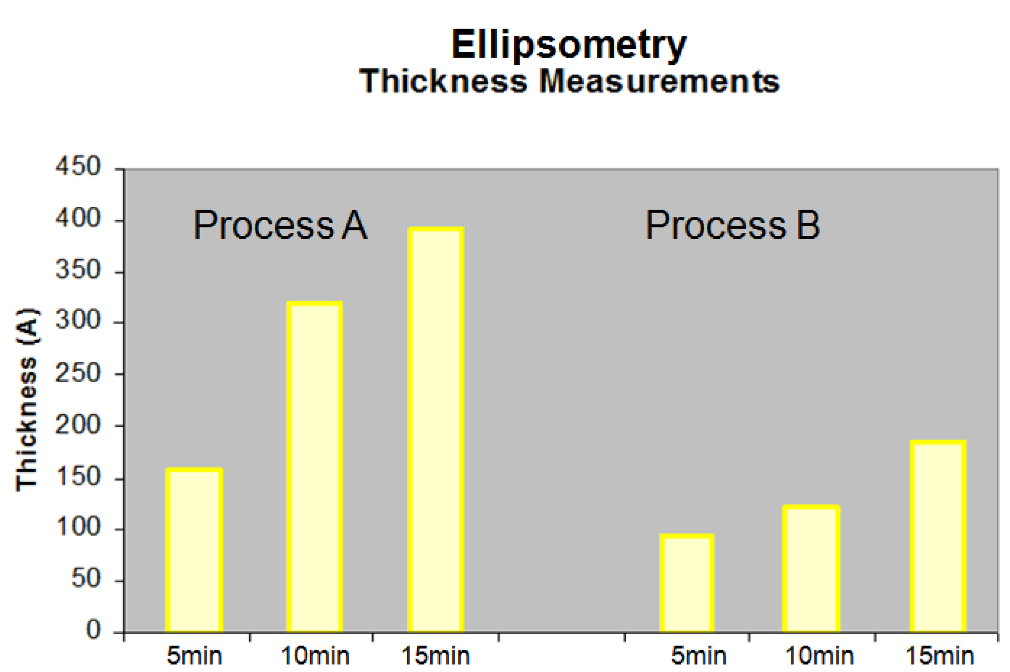
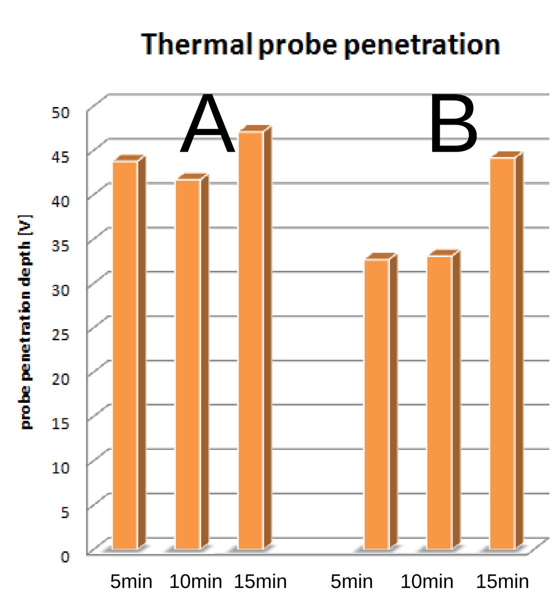


Diagram of a nanoTA measurement on a micro-scale surface feature. At softening the probe penetrates into the sample revealing a local transition temperature.

Background:

NanoTA makes use of a micro-fabricated silicon probe with an integrated heating element. This probe can be used in place of a standard Atomic Force Microscope (AFM) probe to scan a sample and generate an image of the topography. The probe then has the novel utility of probing softening points at select locations by increasing the temperature of the probe until the surface softens due to the local temperature increasing above a transition temperature. This allows nanoscale measurements of the transition temperature on the surface of the sample with good correlation to conventional bulk techniques.

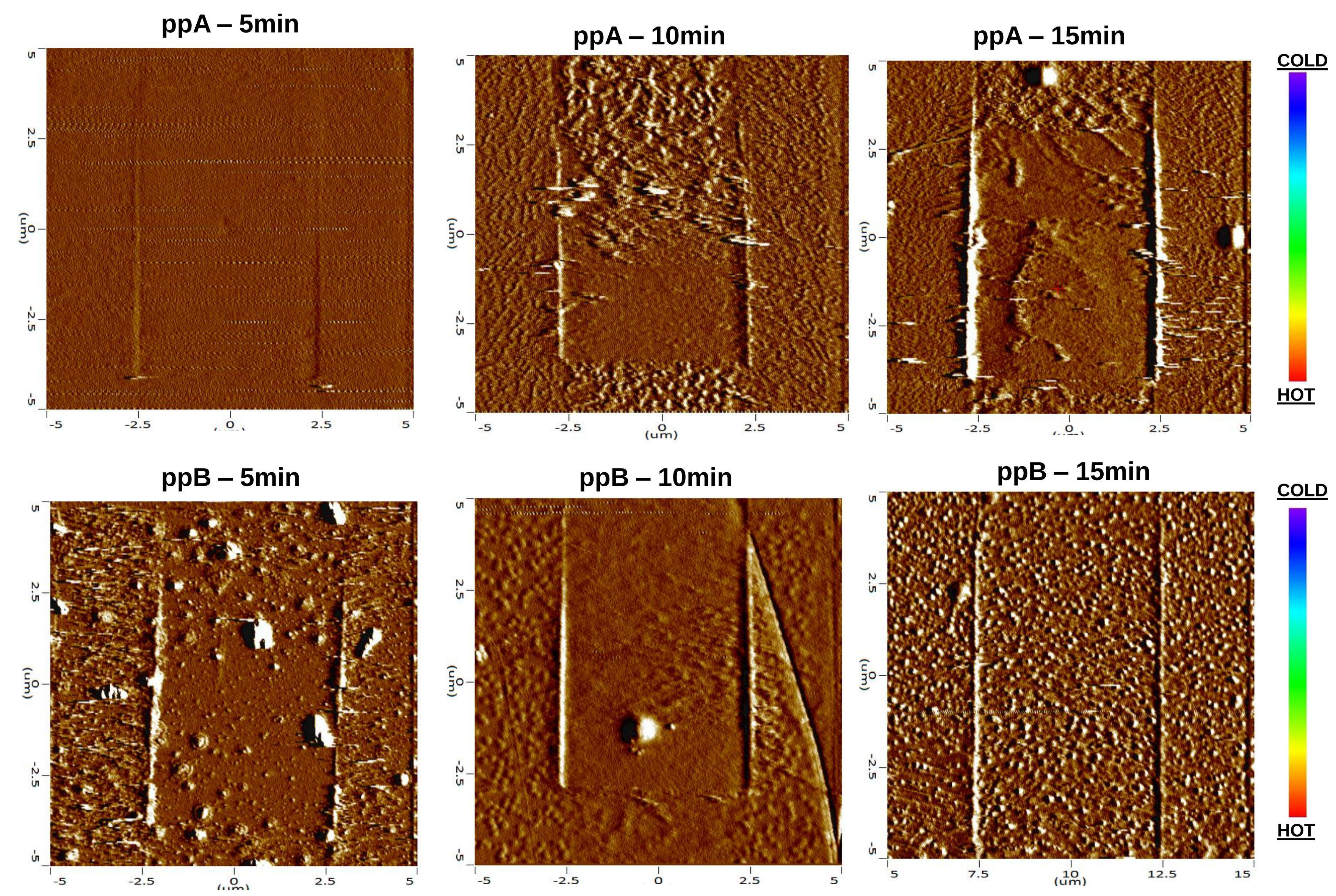




Experiment:

Film thickness is measured using ellipsometry. Process A yields faster film formation than process B. As deposition time is increased film thickness increases. The greatest delta is seen in process A with a 300Å increase from 5 to 15 min as compared to 75Å in process B for the same time period. In thin films 5um or less the thermal probe may be used to approximate film thickness. A heated probe pushes through the coating until making contact with the underlying glass substrate. The vertical displacement correlates with a film thickness and trends with the ellipsometry measurements in this study.

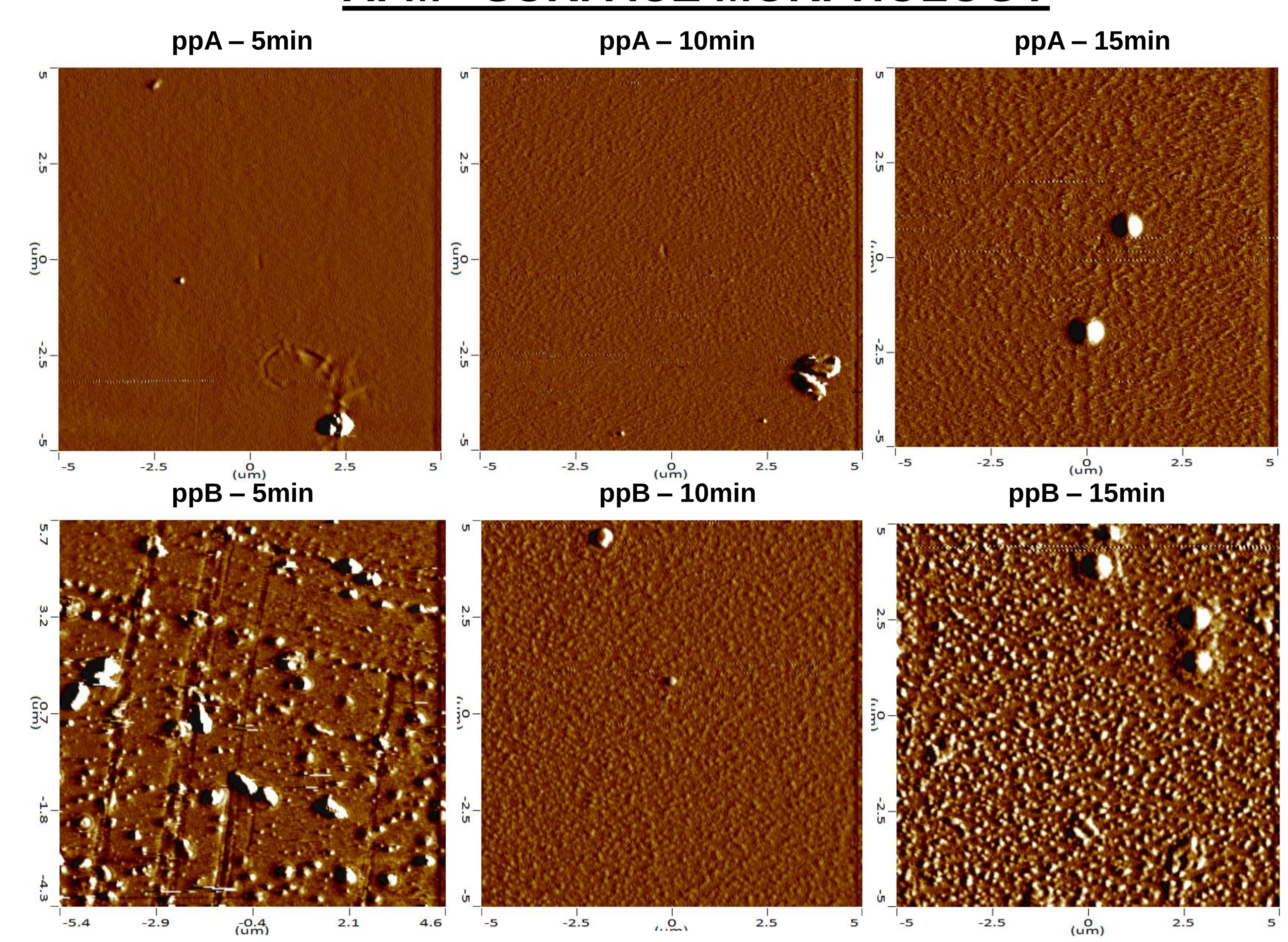
Heated tip AFM scans with Thermal Probe



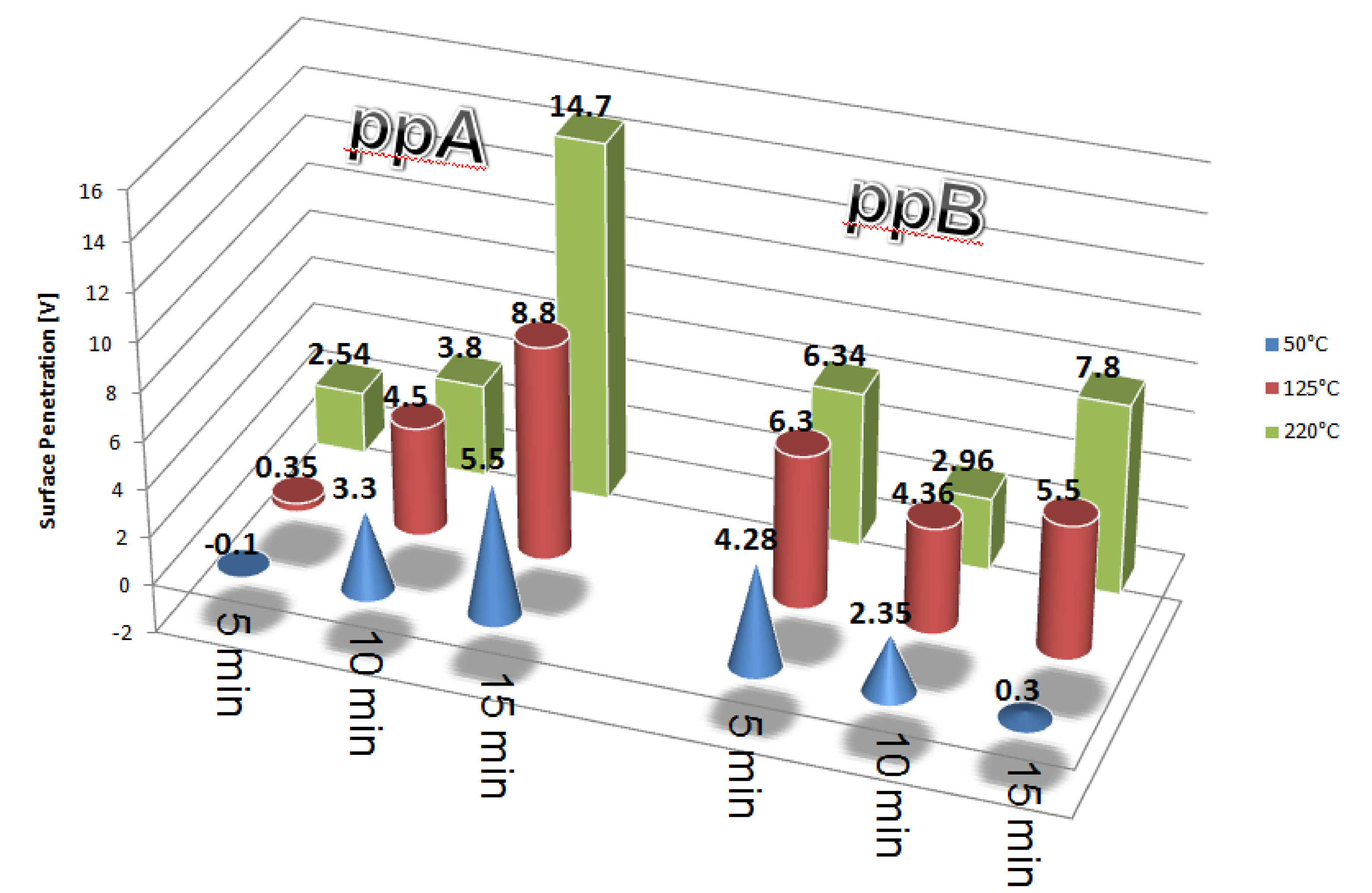
Analysis:

A thermal AFM probe is used to generate high profile images of topography. The deflection maps below visualize different morphologies developing between process A & B at increasing deposition times. In the maps above the thermal probe is incrementally heated while raster scanning a 5 um wide column from top to bottom. Next a complete 10 um wide scan captures an increasing step edge as a function of tip temperature. Surfaces which are softer or composed of mobile polymer experience greater wear due to the force of the thermal probe. Typically wear will increase with temperature and depending on a films thermo-mechanical response.

AFM - SURFACE MORPHOLOGY

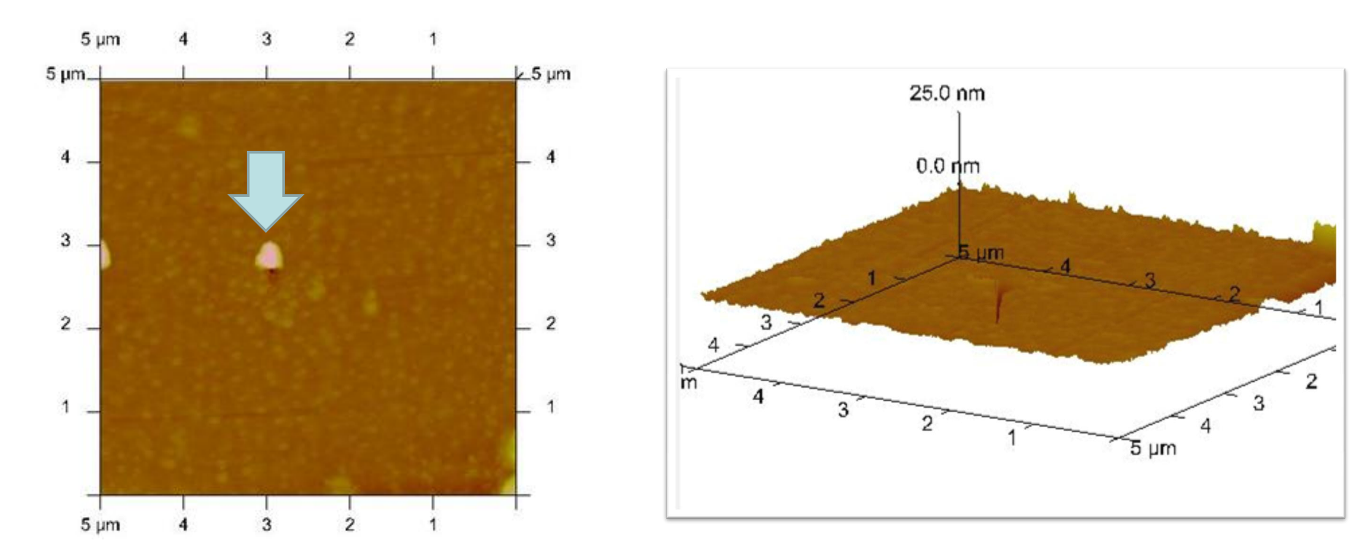


Surface Distortion as a Function of Temperature



Conclusion:

Process *A* yields the thicker films. This set of films exhibit sincreasing wear with increasing deposition time and with increasing tip temperature. Process *B* exhibits decreasing wear at the low tip temperatures and approximately no change in wear at the higher tip temperatures. Of the two processing parameters compared in this study process B deposits a harder film.



Aerial and 3D perspective of a nanoTA indentation on a thin film of plasma polymerized Hexafluoropropylene

Summary:

The structure and chemistry of a plasma polymerized deposition can vary greatly with processing condition & time. Nanometer length scales further challenge characterization of the deposited material . NanoTA is conventionally used to probe local softening points on thin films & other polymeric/organic materials. Films less than 100 nm complicate assignment of thermal transitions due to inherently low volumetric expansions vs interference effects of the AFM laser detection system. An alternative method is described which scans a locally heated thermal probe. Surface distortions provide evidence of polymer mobility with escalating tip temperature. Glass and melt transition are core indicators of micro-scale structure & chemistry.

Acknowledgments:

The team would like to thank Alan Antiporda for his reliable film depositions.

Equipment: PS0500