Keysight Technologies
Humidity-dependent AFM Nanolithography

Application Brief

Introduction

The Keysight Technologies, Inc. 7500 AFM system is comprised of a sealed environmental chamber with a built-in sensor to monitor the local relative humidity (RH) and temperature around the sample. In addition, the eight preset ports through to the chamber at the AFM base allow the easy incorporation of 7500 AFM with an external gas humidification system, thus allowing the control of the RH during AFM measurements.

Membranes have emerged as an attractive material for separating gases from liquid and gaseous streams due to their advantages of low energy requirements, simplicity of operation, and high specificity. They can be configured into hollow fiber tubes and assembled into a membrane gas humidifier. Either air or a gas stream enters from a port on one end and flows through the lumens of those hollow fibers. Meanwhile, water is filled into the shell side and it can permeate into the lumens thereby humidifying the gas stream that is finally connected to a closed AFM. For the Keysight 7500 atomic force microscope, the RH inside its environmental chamber can be effectively regulated using this method and a schematic of the setup is shown in Figure 1. In this application brief, the impact of RH on AFM-based nanolithography will be demonstrated.

Effect of RH on AFM-based Nanolithography

In AFM operation, water present on sample surfaces at nonzero RH could lead to the formation of meniscus around the tip when the cantilever is in contact with the sample. Since material transport through the liquid meniscus has been proposed as one of the possible mechanisms for probe mediated deposition (PMD), the effect of RH on this type of AFM-based nanolithography has been well reported. For instance, the size of the meniscus is proven to be critical for the results of dip-pen nanolithography (DPN).

The impact of RH on other types of AFM nanofabrication such as tip-directed electrochemical reactions is expected to follow a similar trend. The working principle of tip-directed electrochemical reactions or anodic oxidation is that a localized electrochemical cell around the tip can be formed once a conductive probe is used and a sufficient bias is applied to the probe while the sample is connected to a grounding cable. As a consequence, the existing water between the tip and sample can be electrolyzed to generate oxygen radicals that will subsequently lead to an oxidation of neighboring regions of the sample.

Humidity-dependent AFM Nanolithography via Tip-directed Electrochemical Reactions

Shown in Figure 2a is an example of AFM-based nanolithography using Keysight PicoLITH software, from which both the location and the geometry of targeted surface modification can be defined. In this case, an equally separated 3 x 3 array is chosen as the design pattern. The exact fabrication condi-

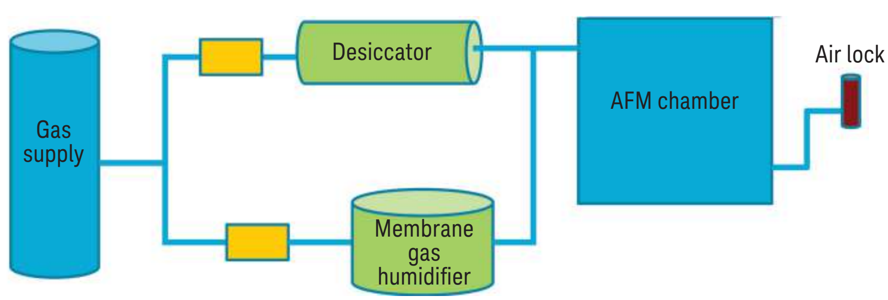

Figure 1. A setup schematic of the Keysight 7500 AFM with controlled humidity.

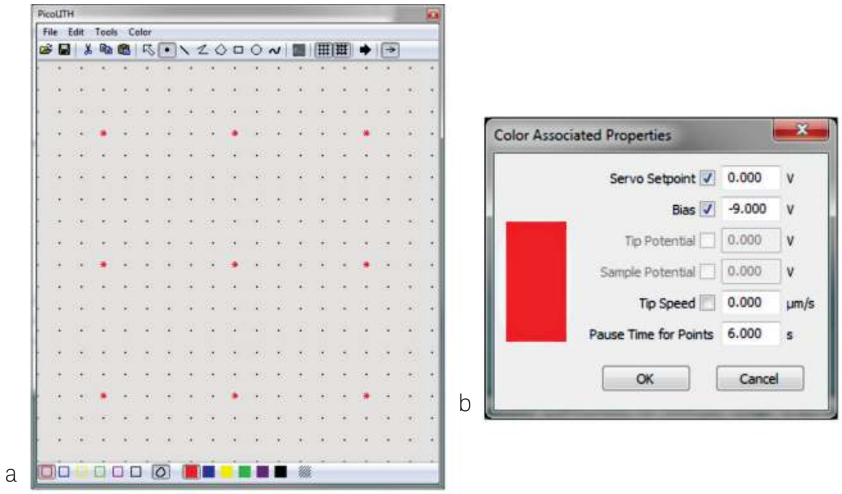

Figure 2. An example of designed AFM-based nanolithography via tip-directed electrochemical reactions. (a) A 3 x 3 array of nine fabrication locations was selected in the Keysight SPM lithographic package PicoLITH and (b) corresponding fabrication conditions defined in the PicoLITH.

tions are illustrated in Figure 2b. When the AFM probe was guided to each of those 9 spots, the tip will remain at that location for 6 seconds while a -9V bias will be simultaneously applied to the tip during that period. Figure 3 is side-by-side comparison of the resulting AFM topographic images of a silicon sample after the AFM nanolithography using the same tip and identical fabrication conditions. The only difference is the humidity level in the environmental chamber. The left picture is corresponding to the experiment performed at a RH of 20% while the right one is conducted at a much higher level (90%). As can be seen, patterned protrusion features on the surface are observed. They can be attributed to the formation of oxidized silica. While the spacing between the two neighboring features is the same because the two AFM image are with the same scan size and the nanolithography processes are following the same guide, the lateral size of the fabricated features is larger under high humidity conditions. This observation can be associated to the fact that larger meniscus are formed at a RH of 90%. It has been claimed by Weeks at al. that at high relative humidity, 70%–99%, the meniscus formed is 100 to 1200nm in height, orders of magnitude larger than predicted by the Kelvin equation.

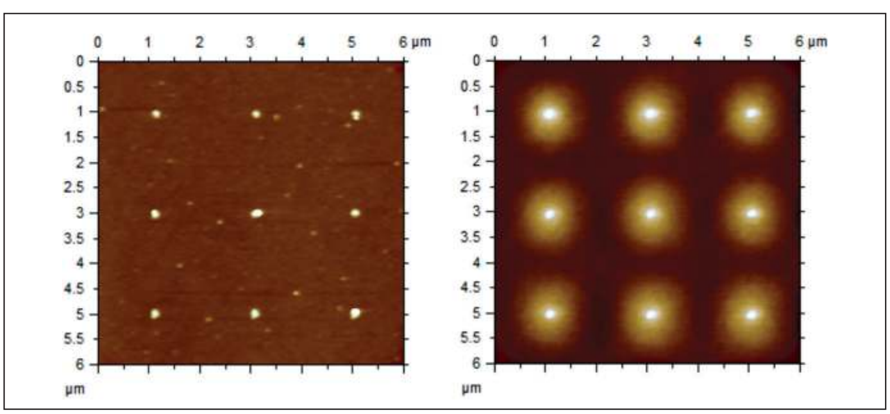

Figure 3. Humidity-dependent anodic oxidation of a silicon surface. Side-by-side AFM topographic images of the resulting surface after the tip-directed oxidation under a RH of 20% (left) and a RH of 90% (right), respectively.

Summary

Using investigations of the effect of RH on AFM-based nanolithography as an example, it is demonstrated that experiments under controlled humidity can be achieved readily with Keysight 7500 AFM.

AFM Instrumentation from Keysight

Keysight Technologies offers high-precision, modular AFM solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight's leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative and easy-to-use AFM technologies.
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