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 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.](image-url)
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 corre-
responding 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 forma-
tion of oxidized silica. While the spacing
between the two neighboring features
is the same because the two AFM im-
age are with the same scan size and the
nanolithography processes are follow-
ing the same guide, the lateral size of
the fabricated features is larger under
high humidity conditions. This observa-
tion 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 magni-
tude larger than predicted by the Kelvin
equation.

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-pre-
cision, modular AFM solutions for re-
search, industry, and education. Excep-
tional 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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