# Keysight Technologies
# High Resolution Imaging with Keysight 7500 AFM

Application Brief

## Introduction

Atomic force microscopy (AFM) was developed as a high resolution surface imaging technique, capable of imaging and probing material structures at the atomic or molecular level. The capability of obtaining high resolution images with ease on different materials, using various imaging methods such as contact and AC mode, has become expected for high performance scanning probe microscopy (SPM) systems. On the other hand, the multitude of SPM applications have also become more demanding in accurate positioning and correct dimensional measurement. Consequently, a large range scanner (greater than 90µm) and closed-loop control has also become a standard requirement for advanced SPM systems. As a result, the integration of a large scan area with accurate closed-loop control and the capability of high resolution imaging becomes the key design challenge. Here we present a couple of application examples that demonstrate the high resolution performance of the Keysight Technologies, Inc. 7500 AFM system.

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## Instrumentation

The Keysight 7500 AFM/SPM microscope is a high-performance instrument that delivers high resolution imaging with integrated environmental control functions. The standard Keysight 7500 includes contact mode, acoustic AC mode, and phase imaging that comes with one universal scanner operating in both open-loop and closed-loop mode. Keysight's patented magnetic AC mode (MAC Mode) is offered as a system option. Switching imaging modes with the Keysight 7500 AFM/SPM microscope is quick and convenient, a result from the scanner's interchangeable, easy-to-load nose cones. Every aspect of the Keysight 7500 AFM's design and construction are optimized to reduce mechanical noise, and deliver industry leading performance. The compact, completely encapsulated AFM Scanner, provides easy cantilever exchange, a slot for (optional) preamps for STM and CSAFM operation, as well as an integrated, high-reliability connector to interface with the control electronics. All 7500 AFM's come with the lowest noise closed loop position detectors to provide the ultimate convenience and performance in imaging, without sacrificing resolution and image quality.

The Keysight 7500 has built-in temperature and humidity sensors for monitoring/control of sample environments in the sample chamber. A control system offers a wide temperature range (-30°C to 250°C) that enables observation of structural and property changes induced by temperature and phase transitions.

## Examples of High-resolution Imaging

High-resolution visualization of the atomic/molecular structure of a sample is a key feature of the AFM instrument. Contact mode is typically used for high resolution imaging of samples that are least affected by the force applied during imaging, such as metal and inorganic solid crystals. Intermittent-contact and non-contact imaging in AC mode are often applied to more delicate/soft samples such as polymers and biological materials.

### Mica Surface Imaged in Contact Mode

Muscovite mica is widely used as a substrate for sample preparation in AFM imaging. It is a layered material that are loosely bound together electrostatically by interlayer potassium ions ($K^+$), and is easily cleaved to expose a fresh aluminosilicate layer by disrupting the $K^+$ layer. The aluminosilicate layer consists of regularly arranged hexagonal $SiO^4$ rings, as shown by the model in Fig.1. The mica surface is readily imaged by contact mode AFM in ambient environment to reveal the lattice structure, which is about 5.2Å between neighboring $SiO^4$ rings. Since the mica surface can show a perfect lattice in a very large scale without noticeable defects and is easy to image with AFM, it is often used as a calibration standard and performance testing sample for an AFM.

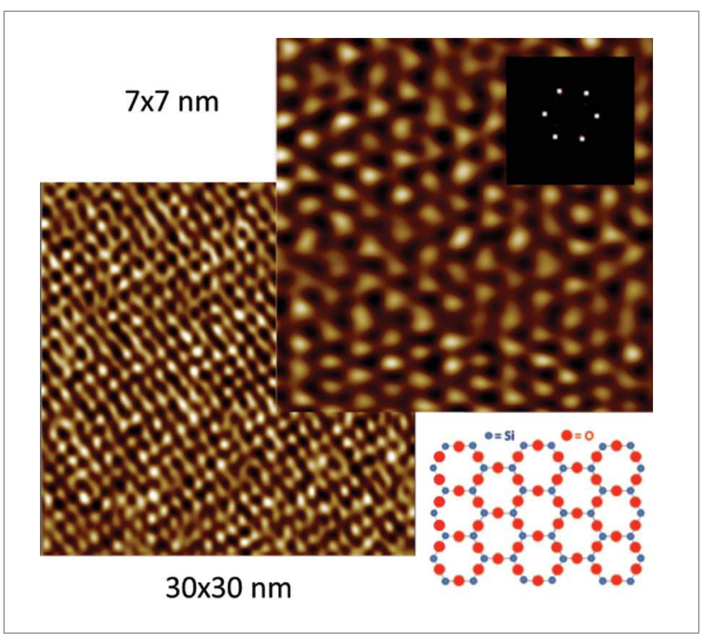


Figure 1. Closed-loop AFM images showing atomic lattice of mica. The insert shows the FFT spectrum of the pseudo-hexagonal unit cell. A model of the Si and O atoms on the (001) surface of a freshly cleaved muscovite mica crystal is shown at the bottom-right of the picture as well.

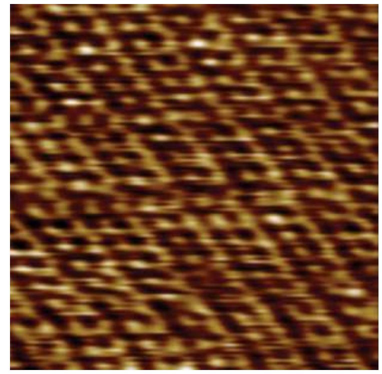

Figure 2. Closed-loop contact mode AFM topography image of barite in Millipore water. Scan size: 11 nm x 11 nm.

### Barite Imaged in Water in Contact Mode

Barite is a member of a large class of isomorphous crystals, consists of mainly barium sulfate, and has an orthorhombic crystal structure. On the (001) surface of barite, each unit cell consists of two $BaSO^4$ layers. Within each layer there are two different $Ba^{2+}$ sites, which are arranged at different levels causing a corrugation of about 1.3Å on the surface. Corrugations with different heights are arranged in rows. The lateral distance between the large corrugations is about 8.9Å. A topography image of barite surface in water is shown in Fig. 2.

### PTFE Thin Film Imaged in Contact Mode

Polytetrafluoroethylene (PTFE) is a synthetic polymer of tetrafluoroethylene that has one of the lowest coefficients of friction against any solid. PTFE thin films can be formed by sliding a block of PTFE over glass substrate at about 200°C. In the film, densely packed PTFE chains align themselves along the same direction of the sliding. The inter-chain spacing between PTFE chains is about 5.6Å, and can be visualized by contact mode AFM imaging (Fig. 3).

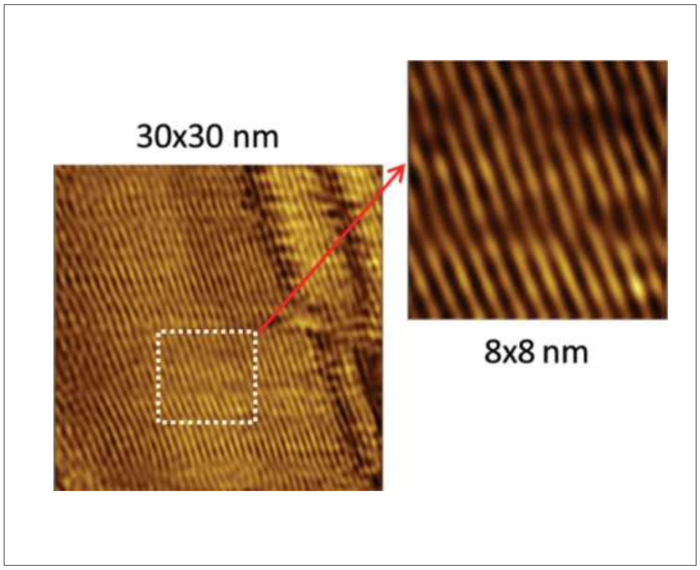


Figure 3. AFM friction closed-loop images of PTFE thin film on glass substrate showing tightly packed molecular chains oriented along the sliding direction. The spacing between PTEF chains is about 5.6Å.

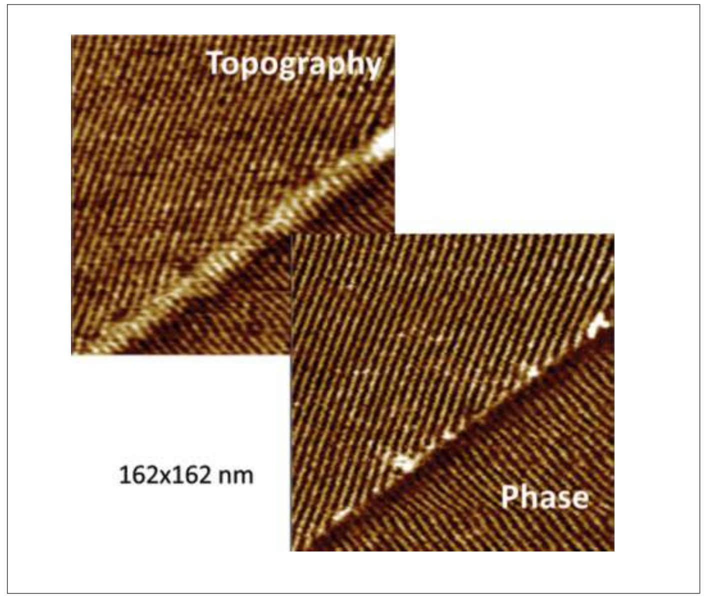


Figure 4. Closed-loop AAC mode imaging of n-$C_{36}H_{74}$ lamellar structures on HOPG.

## $nC^{36}H^{74}$ N-Alkane Lamellar on HOPG Imaged in AAC Mode

N-alkanes are known to form highly ordered lamellar structures on HOPG when crystallized from vapor. Lamellae are formed by molecules regularly arranged side by side and lying with their long axis parallel to the substrate surface. An image of n-$C^{36}H^{74}$ lamellae on HOPG obtained by AFM imaging in AAC mode is presented in Fig. 4. The two lamellae are oriented in an angle of about 120° against each other. The orientations of the individual molecules are most frequently perpendicular to the stripe direction.

The distance between stripes in the lamellae is about 5 nm (the theoretical length of the n-C36H74 chain is 4.38 nm, and the spacing between chains is about 0.5 nm).

## Purple Membrane on Mica Imaged in KCl Solution in MAC Mode

Bacteriorhodopsin is a light-driven proton pump in the cell membrane of Halobacterium salinarium. The bacteriorhodopsin molecules adsorb on freshly cleaved mica substrate to form a densely packed 2-dimensional membrane. The membrane has a distinguished purple color, thus often called purple membrane (PM) in the literature. PM is stable on mica in the pH range from 4 to 9, it forms patches of several hundred nm to several microns in size. The thickness of the membrane is about 5.5 nm in average and changes slightly with pH. Contact mode AFM has been used to image PM in KCl solutions of different pH level. Till today, most of the high resolution images of PM seen are achieved by contact mode AFM. However, contact-mode imaging is less suitable for weakly attached bio-sample, because bio-molecules are often disrupted by the AFM stylus during scanning. Dynamic force microscopy (DFM) methods like tapping mode and MAC mode are designed to reduce the lateral force exerted on the sample surface, thus better suited for imaging soft and weakly attached biological samples. However, tapping mode in solution is difficult to operate. Also for delicate samples like PM, the force for imaging has to be very small, otherwise the lattice structure of the PM would be easily destroyed. Fortunately with MAC mode, the oscillation of the cantilever is directly driven by an oscillating electromagnetic field, thus the interaction force between the tip and the sample can be more accurately controlled. Consequently, high resolution images of the lattice structure of PM can be achieved using MAC mode in solution.

The densely packed bacteriorhodopsin molecules form highly ordered 2-D trigonal lattices in the purple membrane. A to-

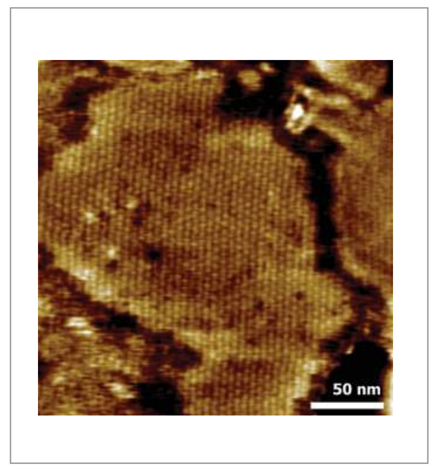


Figure 5. Topography image of PM obtained by MAC mode in solution. The hexagonal arrangement of the bacteriorhodopsin molecules with a repeating unit of about 6 nm and a corrugation of less than 0.8 nm is clearly revealed.

pography image of a patch of PM of about 200 nm in size, obtained by MAC mode, is presented in Fig. 5. The hexagonal arrangement of the bacteriorhodopsin molecules with a repeating unit of about 6 nm and a corrugation of less than 0.8 nm is clearly revealed by the MAC mode image.

At high magnification, the arrangement of bacteriorhodopsin trimers in each of the repeating unit in the membrane revealed a distinct donut-like shape, as seen in Fig. 5. Careful examination of Figure 5 also shows the donut like trimer structures are likely connected by fibrous arms. This demonstrates that MAC mode AFM is a favorable method in studying the topography of soft and weakly attached biological samples, capable of revealing submolecular structures with high resolution under physiological conditions.

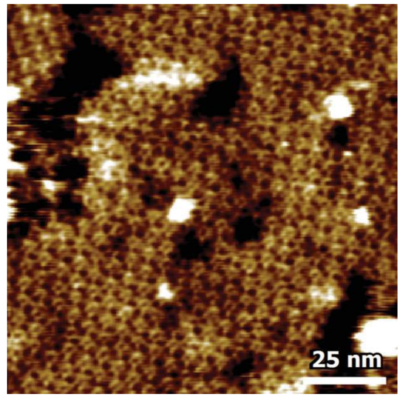


Figure 6. High resolution MAC mode image of PM, revealing the donut-like structure of bacteriorhodopsin trimers, and the connecting fibrous arms in between.

## Summary

Practical examples of high resolution imaging with the Keysight 7500 microscope demonstrate the capabilities for visualizing surface structures of different materials with atomic/molecular resolution. Combined with the outstanding environmental control capability, it enables the study of dynamic process and structural changes at the atomic/molecular level.

## AFM Instrumentation from Keysight Technologies

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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