Keysight Technologies

QuickScan Imaging of *in situ* Dynamical Processes Using Keysight 9500 AFM

Application Note

KEYSIGHT TECHNOLOGIES

Unlocking Measurement Insights

Introduction

Atomic force microscopy (AFM), with its sub-nanometer high spatial resolution has emerged as an increasingly important instrumentation for wide, diversified applications in both academia and industry in recent years. Since its first development in 1986, AFM has undergone many advancements, not only to extend its measurement capabilities, but also to make the instrument more robust and user-friendly. Despite these efforts, the slow image acquisition speed of AFM still remains a challenge to date. Typically, depending upon samples and imaging conditions including scan size and resolution, it can take up to several minutes to scan an AFM image, thereby limiting the AFM's throughput and its applicability to capture *in situ* dynamical processes. Therefore, the QuickScan technology developed by Keysight Technologies, Inc. addresses this issue through its ultrafast scanning capability of more than 100 lines/s or up to 2 s/frame (200 × 200 pixels) without sacrificing the high image quality. Other AFM measurement capabilities, such as but not limited to electrical characterization are also not compromised by the ultrafast acquisition speed. In this application note, examples showing the application of the QuickScan technology for capturing *in situ* dynamical processes are demonstrated using the Keysight 9500 AFM. Kelvin force microscopy (KFM) measurement using QuickScan is also illustrated here.

Instrumentation

The Keysight 9500 AFM/SPM microscope is a high-performance instrument that delivers high speed and high resolution imaging with integrated environmental control functions. The standard Keysight 9500 includes contact mode, acoustic AC mode, and phase imaging that comes with one universal scanner operating in both open-loop and closed-loop modes.

Keysight's QuickScan technology for ultrafast scanning is available as a system option, controlled through the easy-to-use Keysight's NanoNavigator imaging software that comes equipped with an Auto Drive feature that can automatically and optimally sets feedback loop parameters within seconds. Switching imaging modes with the Keysight 9500 AFM/SPM microscope is quick and convenient as a result from the scanner's interchangeable, easy-to-load nose cones. The QuickScan technology utilizes a specialized titanium QuickScan nose cone that contains a secondary Z-piezo separate from the primary Z-piezo in the scanner body. This secondary Z-piezo can react very quickly to changes in samples' topography during high speed scanning whereas the primary Z-piezo in the scanner body corresponds to slower changes in surface topography such as tilt. A crossover circuit is tuned to optimize the frequency at which both Z-piezos respond to. The titanium material of the QuickScan nose cone ensures a high resonant frequency and wide bandwidth required for high speed operation.

Every aspect of the Keysight 9500 AFM's design and construction are optimally designed and engineered to reduce mechanical noise and to deliver industry leading performance. All 9500 AFMs come with the lowest noise closed-loop position detectors that ensure accurate and repeatable scanning motion to provide the ultimate convenience and performance in imaging, without sacrificing image resolution and quality.

High Speed, High Resolution Imaging with QuickScan Technology

All examples presented here are performed with the acoustic AC (AAC) mode using Arrow UHF cantilevers from NanoWorld (Neuchâtel, Switzerland). However, it is noted that although commonly used in AAC mode, QuickScan technology may also be applied to contact mode.

Figure 1 is an AFM topography image of C_{36} alkanes on HOPG substrate, demonstrating the QuickScan capabilities of high resolution imaging at high speed, in this case at 100 lines/s. The orderly rows of C_{36} molecules, with a periodicity of 4 nm were clearly resolved even at a high scanning rate.

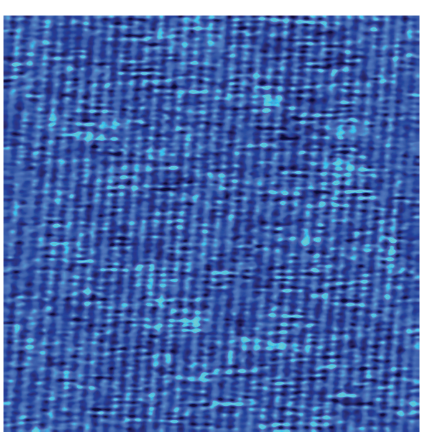

Figure 1. High resolution AFM topography image of C_{36} alkanes on HOPG substrate, captured at 100 lines/s. Image scan size is 150 nm × 150 nm.

Dynamic Capturing of Polymer Phase Transition

Poly (diethylsiloxane) (PDES) is a polymer that can exist in different phases depending upon temperature changes. At a temperature below 7 °C, PDES polymer exists in a crystalline form, whereas at a temperature higher than 47 °C, it is in an amorphous state. Within the intermediate temperature range, this polymer remains in a semi-crystalline phase. Nanoscale study of the thermal-dependent phase transitions of PDES was performed using the 9500 AFM QuickScan mode. Sample was prepared by depositing the PDES polymer on a Si wafer by means of rubbing. Using the temperature control system of the 9500 AFM, the sample was heated on a heating stage from room temperature to 48 °C before subsequently being cooled back down to room temperature. AFM imaging was performed throughout this heating-cooling process. Figure 2 shows AFM topography and phase images of the PDES polymer when subjected to different temperature changes, obtained at a rate of 0.1 frame/s. Patches of the polymer due to rubbing of PDES on Si substrate are observable in the images. The main polymer material is in stretched patches, as seen primarily in the center of the images. As expected, initially at room temperature, PDES occurs as a semi-crystalline structure. The crystalline structure is easily distinguishable from the phase image based on its rod-like structure oriented perpendicularly to the rubbing direction. During the heating process, the crystalline features melted and the polymer became amorphous, as observable from the disappearance of crystals. Subsequent cooling resulted in the recrystallization of the material.

(a)

25 °C

(b)

46 °C

(c)

28 °C

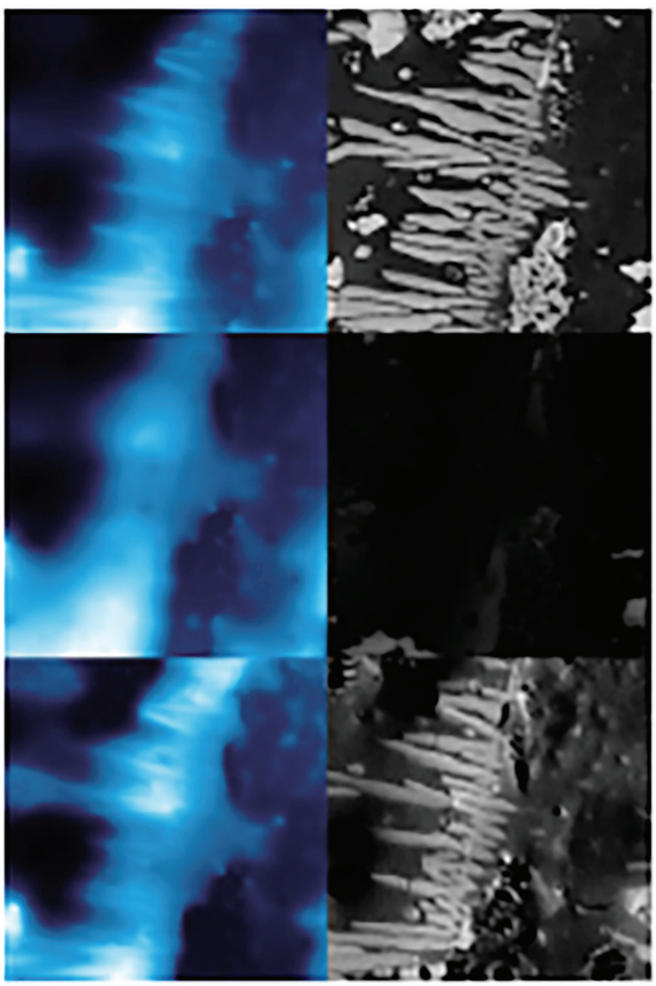

Figure 2. Dynamical AFM studies of PDES polymer's phase transitions during a heating-cooling process using QuickScan imaging under AAC mode. Topography (left columns) and phase (right columns) images showing the (a) initial semi-crystalline structures at room temperature, (b) melting of crystal structures when heated to 46 °C, and (c) recrystallization following subsequent cooling to room temperature. AFM imaging was performed at 0.1 frame/s. Image scan size is 4 µm × 4 µm.

Real Time Monitoring of Ice Crystallization

Understanding the dynamics of ice crystal formation, in particular from vapor phase is important and useful, such as for aviation applications where this information can be applied to develop materials that can assist in the prevention of undesirable ice crystal growth. Investigation of this phase transition process at a nanoscale is demonstrated here through high speed and high resolution scanning of the AFM QuickScan mode. A sample cooling stage and an environmental control chamber of the AFM system were utilized to cool a freshly cleaved mica sheet below the freezing point of water, and also at the same time, to generate environment with humidity levels (~27%) ideal for ice crystallization. AFM images were collected at a rate of 0.4 frame/s throughout the crystallization process. Figure 3 shows every fourth AFM topography image captured during this process. The progressive ice crystal growth from a nucleus to a structure with many branches is clearly observable from the images.

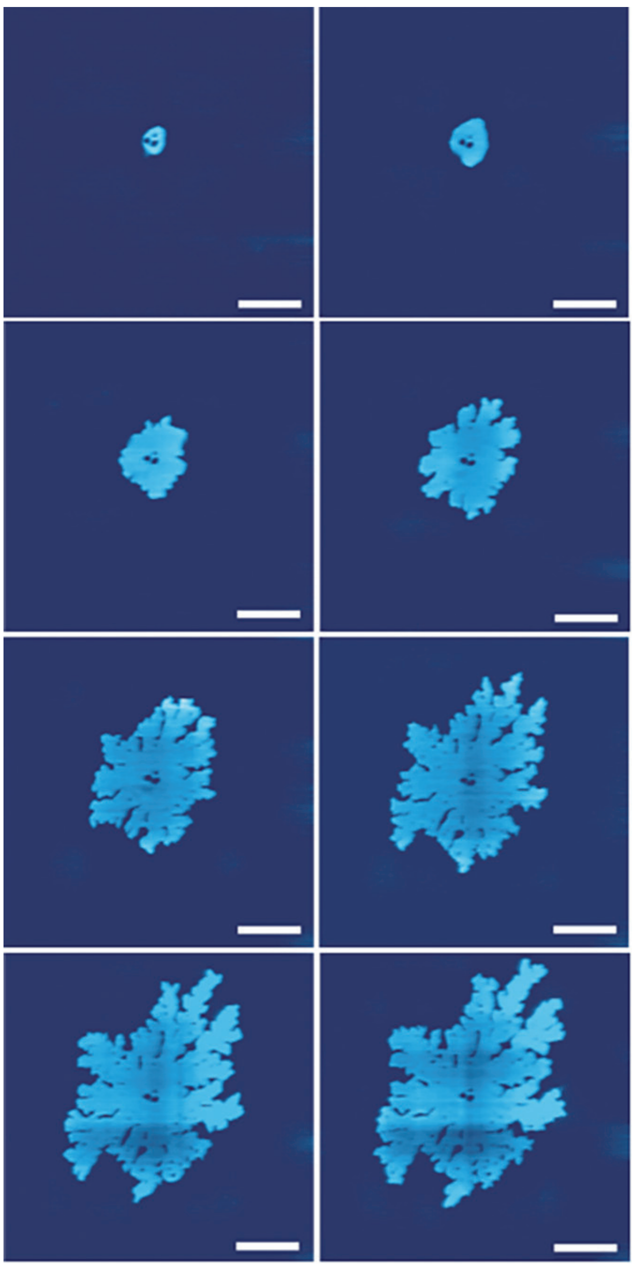

Figure 3. Every fourth AFM images obtained through AAC QuickScan mode illustrating the progressive growth of ice crystal formation, starting from a small nucleus into a structure with many branches. AFM imaging was performed at 0.4 frame/s. Scale bar is 1 µm.

Surface Potential Measurement of Self-Assembled Fluoroalkanes

Kelvin force microscopy (KFM) is used for direct and quantitative measurement of surface potential at the nanoscale, which may reveal important information such as surface charging, molecular dipole orientation in organic thin films, and also band bending and dopant concentrations in semiconductor materials. The basic principle of KFM is based on the measurement of electrostatic forces between the tip and the sample. For an applied DC bias (V_{CD}) with a small AC modulation signal ($V_{AC} sin(\omega_2 t)$), the induced capacitive force is given as

$$F(z) = -\frac{1}{2}\frac{\partial C}{\partial z}\left[(V_{DC} - \phi)^2 + \frac{1}{2}V_{AC}^2\right] - \frac{\partial C}{\partial z}(V_{DC} - \phi)\, V_{AC}\, sin(\omega_2 t) + \frac{1}{4}\frac{\partial C}{\partial z}V_{AC}^2\, cos(2\omega_2\, t)$$

where ϕ is the contact potential difference (V_{CPD}) between the tip and the sample, C is the capacitance and z is the height. From the equation, it can be seen that the first ω_2 dependent term becomes zero if $V_{CD} = \phi$. Therefore, surface potential can be measured by nullifying the ω_2 component. In addition, as the $cos(2\omega_2\, t)$ term is proportional to $\frac{\partial C}{\partial z}$, thus mapping the amplitude of the cantilever deflection signal at $2\omega_2$ yields an image of these variations, which can be attributed to the spatial variations of local dielectric properties.

Fluoroalkanes ($CF_3(CF_2)_n(CH_2)_mCH_3$) form self-assembled structures, usually toroids or ribbons, on Si substrate. These self-assembled structures typically exhibit strong surface potential due to the vertical orientation of the chains carrying the molecular dipole at the $-CH_2-CF_2-$ bond. Figure 4 shows the topography and surface potential of self-assembled $F_{14}H_{20}$ fluoroalkane molecules in toroid form on Si substrate, measured simultaneously using QuickScan and KFM, achievable by applying dual frequency excitation to the weakly conductive Arrow UHF probe. The fast scanning at 60 lines/s did not compromise the AFM high spatial resolution and surface potential measurement capabilities of KFM. The surface potential of these self-assembled structures is determined to have a value of approximately -0.75 V, consistent with predominantly vertical alignment of the molecular chains.

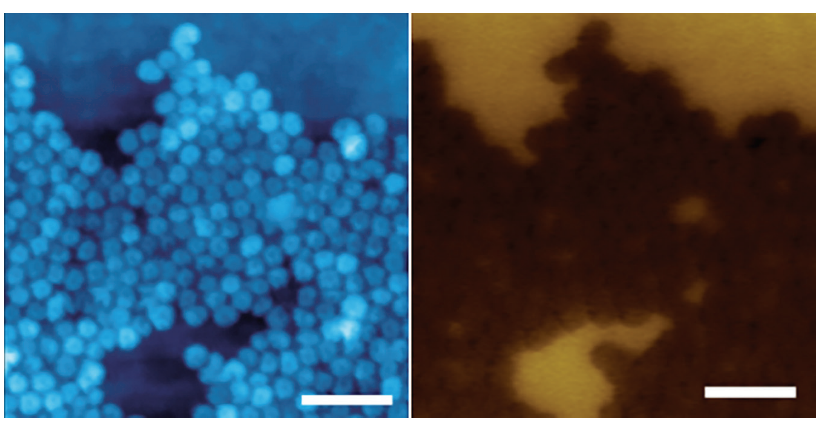

Figure 4. Topography (left) and surface potential (right) images of $F_{14}H_{20}$ fluoroalkane self-assembly on Si substrate, obtained at 60 lines/s. Scale bar is 200 nm.

Summary

The thermal-dependent phase transition of PDES polymer and ice crystallization using temperature and environmental control are two practical examples highlighting the impressive aspects and options of the 9500 AFM. They are evidences of the *in situ* applications of dynamic processes made possible as a result of the ultrafast image acquisition speed of QuickScan technology, without compromising the high spatial resolution of the AFM images. In addition, they demonstrated that AFM imaging with well-controlled temperature and environment are readily achievable with the 9500 AFM system. The functionalities of the 9500 AFM system, with QuickScan option, along with its environmental control chamber can also be extended to other applications, such as dynamic studies of live cells. In addition, the QuickScan-KFM measurement of the surface potential of self-assembled fluoroalkane structures shows that electrical characterization capability is not affected by the fast scanning ability of the AFM system.

AFM Instrumentation from Keysight Technologies

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