Keysight 5600LS AFM
Surface Potential Measurements Using Keysight 7500 AFM
Application Brief

Introduction

Scanning kelvin force microscopy (KFM) has been widely used in mapping surface potential (SP) distribution at the nanoscale. The principle of KFM is based on the measurement of electrostatic forces between the tip and the surface. When a dc bias (V_{dc}) and a small ac modulation signal $V_{ac}\sin\omega t$ are applied between the tip and the sample, the induced capacitive force is

$$\begin{aligned} F(z) &= F_{dc} + F_{\omega} + F_{2\omega} \\ &= -\frac{1}{2}\frac{\partial C}{\partial z}\left[(V_{dc}-\phi)^2 + \frac{1}{2}V_{ac}^2\right] \\ &\quad -\frac{\partial C}{\partial z}(V_{dc}-\phi)V_{ac}\sin\omega t \\ &\quad +\frac{1}{4}\frac{\partial C}{\partial z}V_{ac}^2\cos 2\omega t \end{aligned} \qquad (1)$$

where ϕ is the contact potential difference (CPD) between the tip and the sample. It is evident from the F_{ω} term in Equation (1) that F_{ω} depends linearly on V_{dc} and becomes zero when $V_{dc} = \phi$. Therefore, SP can be measured directly by nullifying F_{ω}. Since SP is measured here by nullifying the amplitude of the F_{ω}, it is named AM-KFM, meaning amplitude sensitive. Alternatively, SP can be measured by nullifying the resonance frequency shift, Δf_{ω}, caused by the ac modulation (FM-KFM),

$$\Delta f_{\omega} = -\frac{f_0}{2k}F'_{\omega} = -\frac{f_0}{2k}\frac{\partial^2 C}{\partial z^2}(V_{dc}-\phi)V_{ac}\sin\omega t, \qquad (2)$$

where f_0 and k are the resonance frequency and spring constant of the cantilever, respectively.

The force component at 2ω is proportional to dC/dZ. Therefore, by mapping the $F_{2\omega}$ response one can get spatial variations of local dielectric behavior. In other words, KFM can provide an advanced characterization of local electric and dielectric properties.

Instrumentation

The Keysight Technologies, Inc. 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. 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. 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 AFM is equipped with an AC Mode controller that has three dual phase lock-in amplifiers (LIA). These digitally-controlled analog LIA have a broad bandwidth up to 6MHz. This allows the Keysight 7500 AFM to perform single-pass KFM measurements by applying dual-frequency excitation signals to the AFM tip simultaneously. One excitation signal is used for modulating the mechanical oscillation of the AFM tip and for topography imaging. The second excitation signal is applied for the modulation of the AFM-based electrostatic tip-sample force, and is used for the measurement of sample surface potential. The 3rd LIA can be set for monitoring various signals, e.g., $F_{2\omega}$ for dC/dZ imaging. Detailed instrumental setup/operation are available in other Keysight documents. A number of practical examples are shown below to demonstrate the application of Keysight 7500 AFM in high resolution surface potential measurement and spatial mapping of dielectric properties over the sample surface.

Surface Potential of Self-assembled Fluoroalkanes

Fluoroalkanes F_nH_m [$F_nH_m = CF_3(CF_2)_n(CH_2)_mCH_3$] form self-assembled structures, usually toroids or ribbons, on Si substrate. Those structures exhibit strong surface potential due to the vertical orientation of the chains carrying the molecular dipole at the $-CH_2-CF_2-$ bond [4]. A set of $F_{14}H_{20}$ self-assembled structures (most are toroids) on Si substrate was examined in KFM (AM-AM) mode, the corresponding topography and SP images are shown in Figure 1. As it revealed that there are only a number of patches of self-assembled $F_{14}H_{20}$ molecules show strong dark contrast in the SP image. Other areas of the surface are covered with randomly adsorbed $F_{14}H_{20}$ molecules and show a relatively homogeneous potential background. The surface potential of these self-assembled structures has a value of around -0.7V, which is consistent with predominantly vertical alignment of the molecular chains.

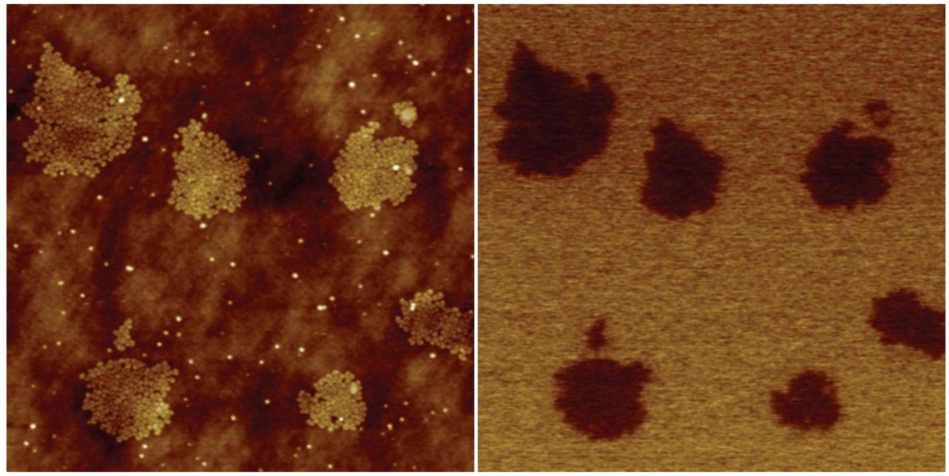

Figure 1. KFM topography (left) and surface potential (right) images of fluoroalkane $F_{14}H_{20}$ self-assembly on Si. Scan size: 4 µm x 4 µm.

KFM measurement can be done in either AM-AM mode or AM-FM mode with the Keysight 7500 AFM, depending on the input signal of the surface potential servo. In general the AM-FM mode has better sensitivity on the surface potential value. The surface potential values obtained in the AM-FM mode are higher than those obtained in the AM-AM mode. The better sensitivity of the AM-FM detection also leads to higher spatial resolution achieved in mapping of surface potential.

Differentiation of Heterogeneous Polymer Materials with dC/dZ

Phase imaging has often been used in mapping of heterogeneous polymer materials. The phase contrast is assigned to differences of local mechanical properties and variations of energy dissipated in the tip-sample interactions. Surface potential images can play a similar role in identifying the surface locations with different electric properties. In addition, dC/dZ imaging can be particularly useful in differentiating components of different dielectric properties. A range of polymer materials that might be sensed by KFM and dC/dZ can be expanded by including acrylic polymers. The KFM images of a 20-nm thick film of 3PS/7PMMA film (weight ratio of the components is 3:7), which was deposited on Si substrate by spin-casting, are shown in Figure 2. The phase image (Figure 2B) indicates the islands on the topography (Figure 2A) correspond to the 70% of PMMA that has a stiffer mechanical property. The SP images show little difference between the PMMA and PS domains, about 60 mV from a separate measurement. The dC/dZ image (Figure 2D), on the other hand, clearly revealed the difference in dielectric behavior between the two components.

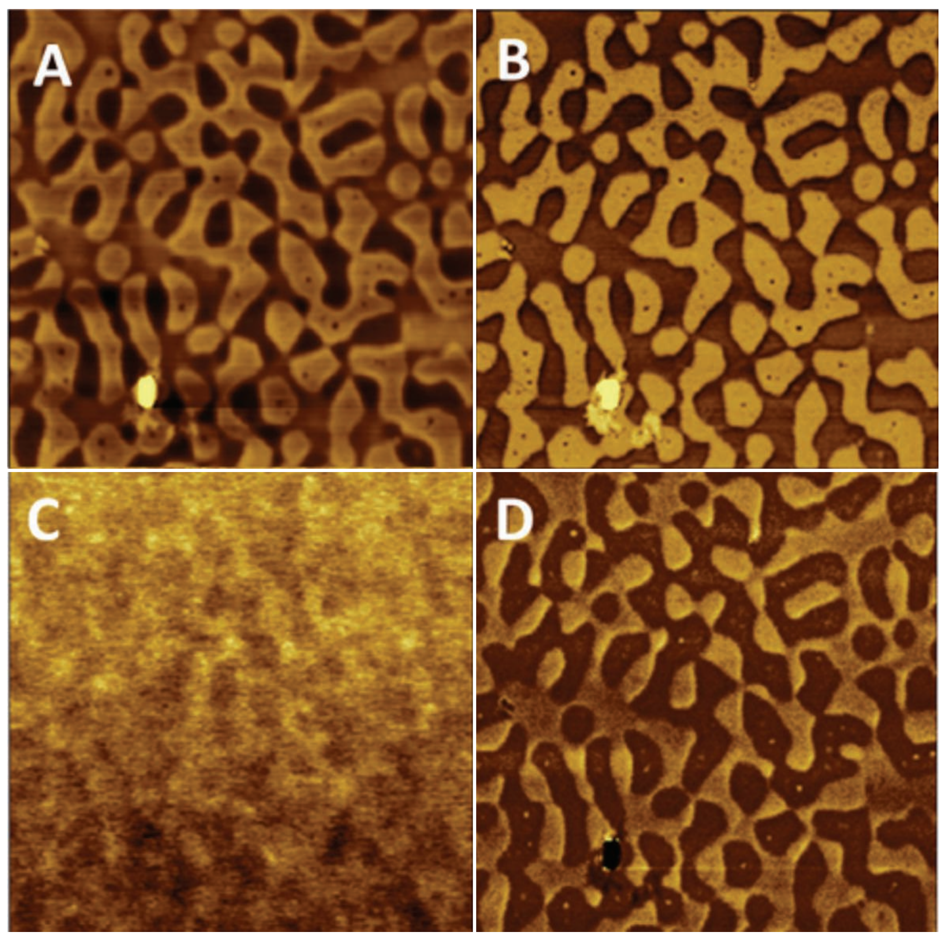

Figure 2. AFM topography (A) , phase (B), SP (C), and *d*C/*d*Z (D) images of PS(3)-PMMA(7). Scan size: 8 µm x 8 µm.

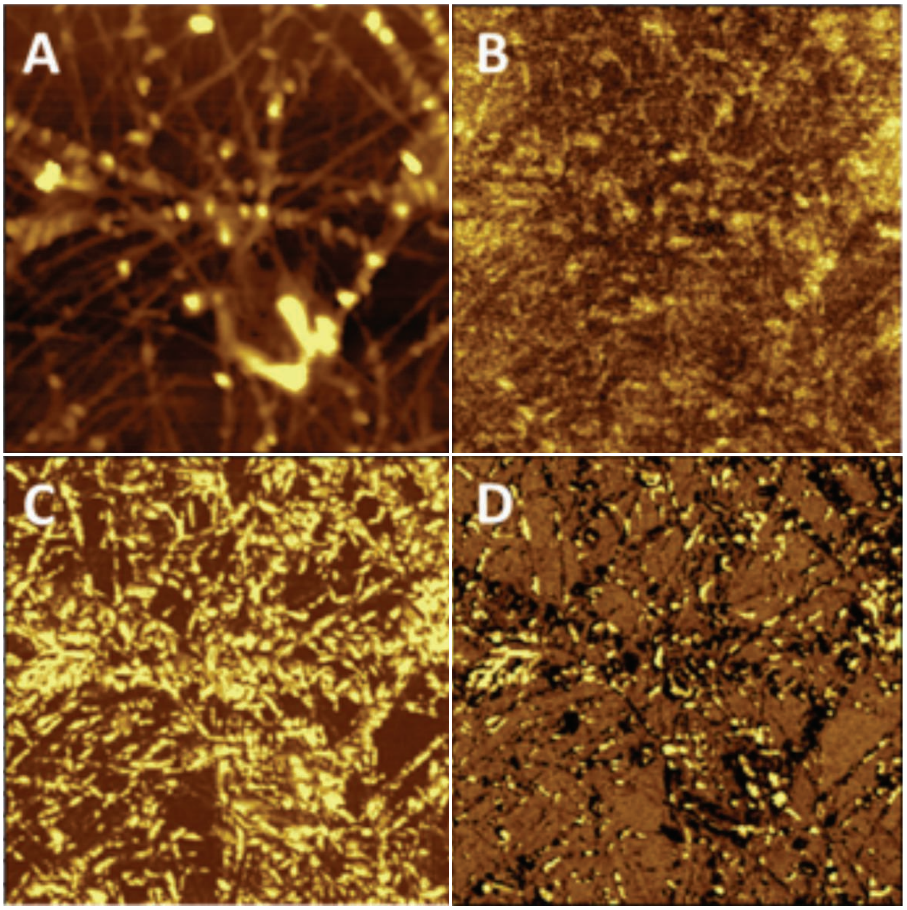

Figure 3. AFM topography (A), SP (B), dC/dZ amplitude (C) and $\partial C/\partial Z$ phase (D) images of Ti coated CNT deposited on ITO. Scan size: 2µm x 2µm.

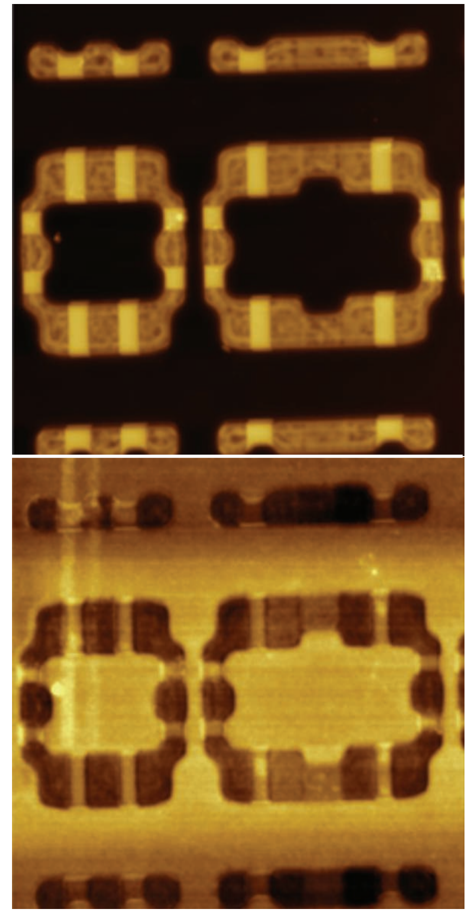

Figure 4. Topography (top) and surface potential (bottom) images SRAM. Scan size: 25µm x 25µm.

KFM Measurements on Metals and Semiconducting Materials

Surface potentials of metals are directly related to their work functions. KFM can be used to measure difference in work functions between different metals and semiconducting materials. In addition, dC/dZ imaging is also important in the characterization of semiconductor materials. One example shown in Figure 3 is the KFM and dC/dZ imaging of Ti coated carbon nanotubes deposited on ITO. Carbon nanotubes coated with Ti clusters are clearly seen in the topography image Figure 3A. They generally show a higher surface potential than the underlying ITO substrate, Figure 3B. The dC/dZ amplitude shows the difference between the ITO and the Ti coated nanotube as well, Figure 3C. A detailed examination of the dC/dZ phase image (Figure 3D) further revealed the difference between the carbon nanotube and Ti clusters on top of them, which also indicates not all the particles seen in the topography are Ti clusters, but carbon residues from the CNT preparation.

Another important KFM application is the characterization of semiconductor devices, in both fabrication and failure analysis. The surface potential measured using KFM is correlated to the local work function of the semiconductor sample, which in turn depends on the material and the dopant level near the surface. Through careful experiment and tip calibration, evaluation of localized Fermi level on semiconductor surface is possible using KFM. It is also interested in looking at the electric field distribution around certain elements in an IC device, particularly in the case of a hot circuit with currents flowing. As an example, a KFM image of a piece of SRAM de-processed to the bare silicon level, exposing the PMOS and NMOS structures is presented in Figure 4. The potential image clearly reveals the different potential levels correlating to the different doped regions on the surface.

KFM Measurement on Materials for Renewable Energy Research

KFM has been widely used in energy related research in battery, fuel cell and photovoltaic cells, to investigate the electronic properties of the candidate materials. KFM has been used to study the work function differences between different facets of crystal orientation on single grains of $CuGaSe_2$, to characterize the grain boundary structures within polycrystalline absorber materials, and to study the cross-section of the junction region through a complete solar cell device. KFM has also been used to study organic solar cells (OPV). The investigation of a classical organic solar cell system consisting of MDMO-PPV/PCBM is well suited to analysis using KFM, together with high resolution SEM data, resulting in identification of a barrier for electron transmission from the electron-rich PCBM nanoclusters toward the extracting cathode, and correlation of the power conversion efficiency to the nano-scale morphology in the bulk heterojunction. KFM was used to differentiate the work function of the dopants from the polymer matrix, which further helped in identifying the type of charge carrier in the system.

Figure 5 shows an example of KFM imaging of dual-patterned conducting polymer structure on Au substrate. The honeycomb frame of the structure is formed by conductive polymer, while the hole is backfilled with SAM layers of insulating organic molecules. The surface potential image clearly revealed difference between the conducting polymer frame and the SAM layer. It also shows the imperfection existing in the structure that gives rise to a slightly higher potential. The dC/dZ image also shows the different dielectric properties of the two materials.

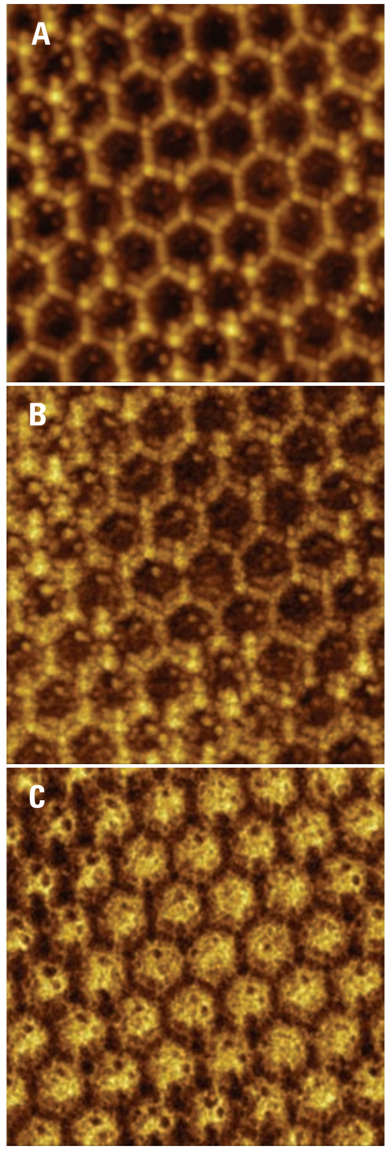

Figure 5. Topography (A), SP (B) and dC/dZ (C) images of conducting polymer patterned on Au substrate. Scan size: 3µm x 3µm.

Summary

Single-pass KFM mode offers high sensitivity and spatial resolution for surface potential measurement. KFM are applicable to a wide range of materials, for both surface potential measurement and dielectric characterization. It also complements phase imaging for compositional imaging by advancing to earlier not accessible areas such as metal alloys and semiconductors. KFM studies in different environments are also bringing new and valuable information about morphology of heterogeneous polymers. The area of environmental AFM will further benefit from local electric and mechanical measurements with multiple frequency detection. Finally, KFM is widely used in energy related research in battery, fuel cell and photovoltaic cells, to investigate the electronic properties of the candidate materials.

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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