Keysight Technologies
7500 AFM Applications in Polymer Materials

Application Note

Introduction

Atomic force microscopy (AFM) is a powerful characterization tool for polymer science, capable of revealing surface structures with superior spatial resolution. AFM is extremely useful for studying the local surface molecular composition and mechanical properties of a broad range of polymer materials, including block copolymers, bulk polymers, thin-film polymers, polymer composites, and polymer blends.

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. 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 control chamber. A control system offers a wide temperature range (-30°C to 250°C) that enables observation of structural and mechanical property changes of polymers induced by temperature and phase transitions. A number of examples for the application of Keysight 7500 in polymer materials are presented below. All images presented here are in closed-loop.

High-resolution Imaging of Polymer Structures

THigh-resolution visualization of a sample's morphology, which is a key feature of AFM, defines most of its applications. AAC mode is a good technique for imaging of polymer and rubbery samples. Shown below is the imaging of a microporous membrane, Celgard©, made of isotactic polypropylene that demonstrates the AFM capability of direct and high-resolution visualization of polymer surface structures. This material is prepared by alternative stretching and annealing procedures. As a result, the material exhibits surface features corresponding to fibrillar and lamellar regions. These features are clearly seen

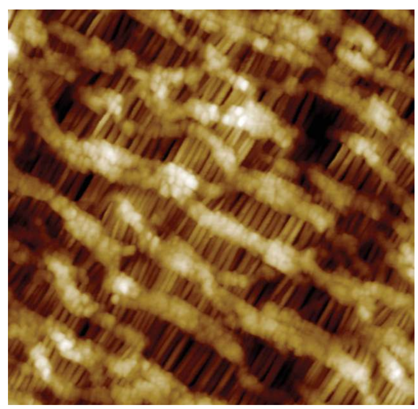

Figure 1. AFM topographic images of polymer isotactic polypropylene with a scan size of 2µm x 2µm in Closed-loop.

in topography image, in which stacks of lamellae are ~ 10-15 nm higher than the surrounding fibrillar regions (Figure 1, left). In addition, one can see individual fibers of ~ 20 nm in diameter, which are uniaxially oriented and separated by nanometer-scale gaps that serves as pores for filtration purposes.

Differentiation of Heterogeneous Polymer Systems Using Phase Imaging

In addition to the unprecedented high spatial resolution, the phase contrast in AAC mode is very sensitive to the differences in material properties such as variation of the mechanical (stiffness, friction) and adhesive properties. Visualization of different components of heterogeneous polymer materials in phase images has be demonstrated by examples taken from studies of block copolymers, semicrystalline and mesomorphic polymers. Shown here are the height and phase images of a triblock copolymer polystyrene-b-polybutadiene-b-polystyrene (PS-b-PB-b-PS) film. A microphase separation pattern of this material, which is mostly pronounced in the phase image, is characterized by structural parameter ~ 35 nm. The phase contrast is related to the fact that at room temperature, PS is in a glassy state while PB is in a rubber-like state. Consequently, the brighter areas in the phase image (corresponding higher areas in topographic image) can be attributed to stiff lamellae of PS.

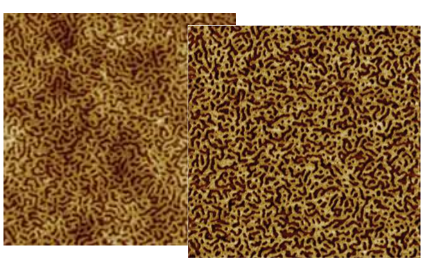

Figure 2. AFM (left) topography and (right) phase images of copolymer PS-b-PB-b-PS. Scan size: 1.5 µm x1.5 µm in Closed-loop.

Besides amorphous and crystalline forms, many liquid crystalline polymers such as poly(diethylsiloxane)(PDES) usually exist in a partially ordered or mesomorphic form, which can also be characterized by phase imaging. Rubbing of PDES on Si substrate leads to patches of the polymer as seen in height image in Figure 3. The stiffer lamellar-like aggregates, which are embedded in amorphous material, are visible due to its brighter contrast in the phase image.

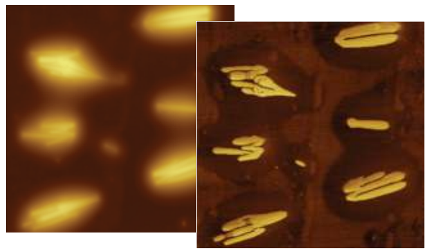

Figure 3. AFM topography (left) and phase (right) images of PDES at room temperatures. Scan size: 10 µm x 10 µm.

Examination of Polymer Thermal Transitions

Polymer or plastic materials can be divided into two major groups: thermoplastic ones or thermosetting ones based on their heat response. Therefore, knowledge of polymer behavior at different temperatures is essential for many practical applications. Direct and nanometer-scale visualization of polymers at different temperatures can provide unique microscopic insight on polymer thermal behavior. For example, cooling of PDES deposited on Si substrate will induce crystallization of material inside the lamellar-like aggregates. The topography and phase images showing different stages of crystallization of PDES at 5° C and -19° C are shown in Figure 4.

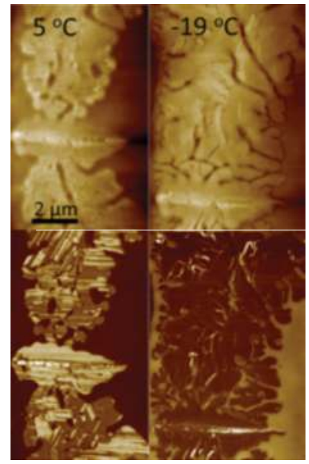

Figure 4. In situ AFM topography (top) and phase (bottom) images of PDES at 5° C (left) and -19° C (right).

Summary

Practical examples of imaging of different polymer samples with the Keysight 7500 microscope demonstrate the capabilities for visualizing important polymer nanostructures and monitoring of structural changes caused by thermal transitions. The outstanding environmental control capability is a unique feature for the microscope.

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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