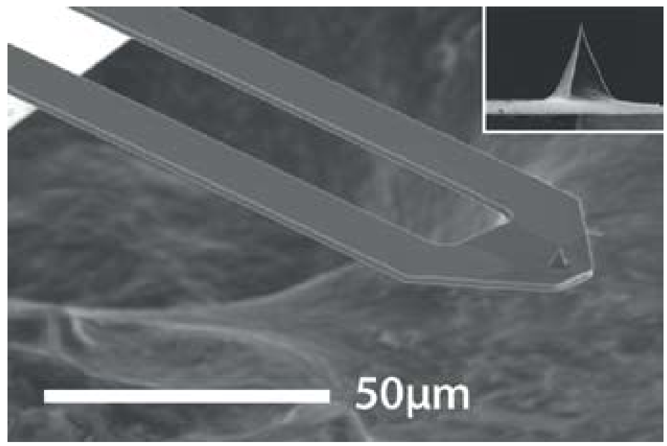
Ztherm™ Modulated Thermal Analysis Option for Asylum Research AFMs

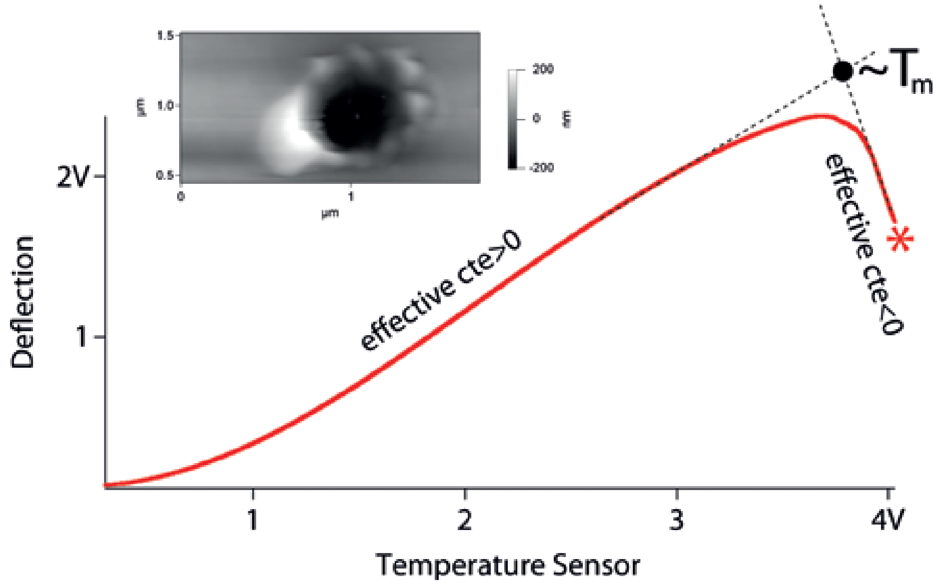
Ztherm combines powerful conventional and new advanced thermal analysis capabilities with the high resolution of AFM, and adds patent pending compensation for thermally-induced cantilever bending and measurement of contact stiffness and dissipation for the highest sensitivity and resolution available. The Ztherm Option provides highly localized heating with sensitivity to ≤10^{-22} liter (sub-zeptoliter) materials property changes, more than an order of magnitude improvement in volume over that previously available with commercial systems. The Ztherm Option is available for the Asylum Research MFP-3D™ and Cypher™ AFM Systems. The Ztherm option utilizes and includes Anasys ThermaLever™ probes.



ThermaLever probe showing cantilever structure and close-up of tip (upper right). The probes are mounted in a proprietary dual-contact cantilever holder (see below) with heating up to 350-400˚C (probe dependent). The probes also permit high-resolution AC imaging before and after heating.

Introduction

Traditional thermal analysis has been used for many years to examine changes in bulk properties of a variety of samples in response to heating. Micro thermal analysis used relatively small Wollaston-wire cantilevers to extend these techniques down to areas as small as a few microns on a side. These probes were typically used in an Atomic Force Microscope (AFM) to control the position of the Wollaston probes, both for probing and imaging applications. The advent of heated silicon probes improved thermal analysis resolution to well below a micron. Nanoscale thermal analysis, where the deflection of the cantilever is used to detect thermally induced changes in surface properties, is a relatively new capability that has been added to AFMs and applied to a variety of materials. For example, most polymers are mixtures of components whose mixing ratios, distribution and uniformity affect the properties of the material and its performance in products such as tires, glues, containers and food packaging. AFM is frequently applied to these polymer samples to reveal surface morphology and nanostructure. With nanoscale thermal analysis, these materials can also be examined for their response to thermally-induced morphological changes, such as melting and glass transitions, at scales not possible with traditional thermal analysis. In many cases, the components can be conclusively identified and mapped at the micro- and nanoscale, thus assisting the polymer formulator in optimizing the desired properties of the final product. Now, the scientists at Asylum Research have taken nanoscale thermal analysis to unprecedented resolution and sensitivity levels with improvements incorporated into the new Ztherm Option.



Conventional Local Thermal Analysis performed with Ztherm. This curve shows the cantilever deflection as a function of the temperature sensor. Initially, the thermal expansion of the polyethylterephthalate (PET) sample under the tip causes the deflection to increase. As the sample starts to soften under the tip, the deflection levels off and then decreases. The transition temperature can be estimated from the crossover between these two behaviors. The inset image shows the topography of the (slightly) sub-micron indent that was created during the measurement.

Cantilever Compensation and Control

A standing problem with existing AFM-based thermal analysis systems is thermally induced bending of the cantilever that results in spurious deflection signals and variable loads being applied during heating. Asylum has developed a patent pending cantilever compensation and control solution that corrects this problem, providing constant-load detection of thermally induced melting (Tm), phase transitions (Tg) and other morphological and compliance effects for materials studies and identification – for areas less than 20nm x 20nm.

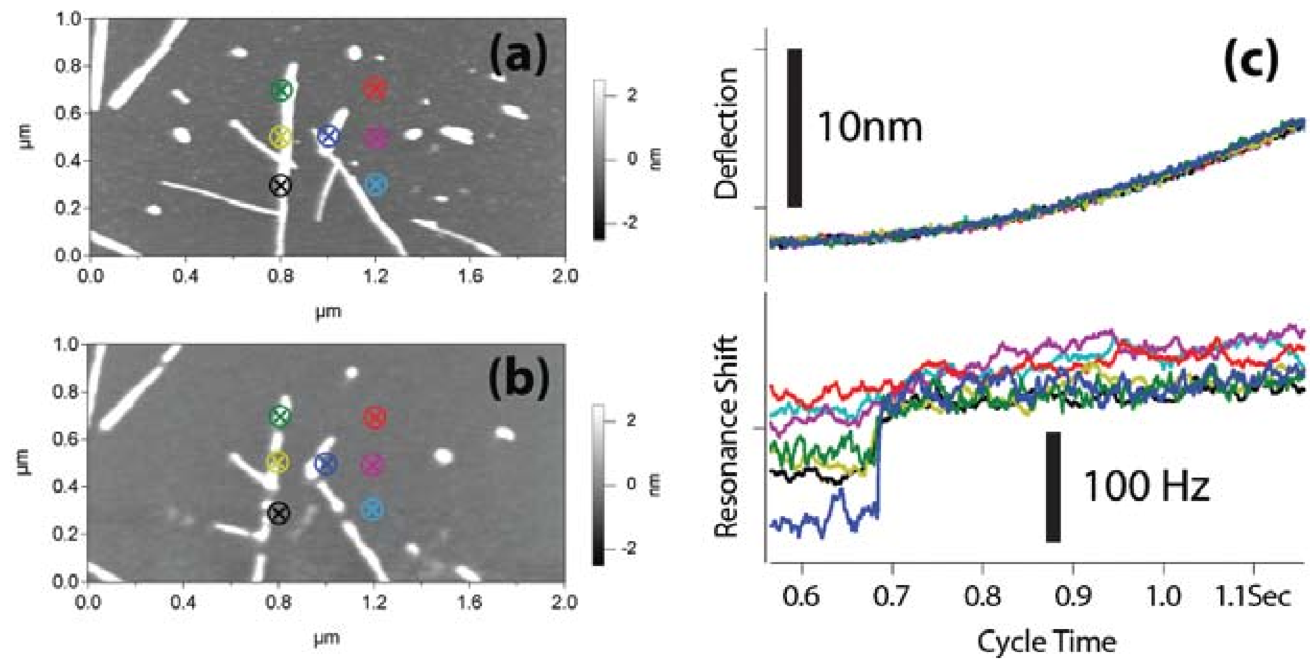
Contact Stiffness and Dissipation

In addition to standard thermal analysis capabilities, the Ztherm Option can also be used to evaluate contact stiffness and dissipation as a function of temperature with advanced techniques such as Dual AC Resonance Tracking (DART™). The contact stiffness and dissipation – measured at the cantilever resonance – are much more sensitive to temperature dependent properties, including surface melting and transition temperatures, than conventional deflection-based measurements. In addition, integrated piezo actuation allows high resolution AC imaging of samples for surface topographical mapping before and after thermal measurements.

Ztherm dual-contact, differential drive cantilever holder. Compatible with and allows easy exchange of Anasys ThermaLever probes.

Features/Benefits

- The Ztherm Option includes software, thermal calibration samples and ten (10) Anasys ThermaLever AN2-200 silicon probes.
- Probes are used in a proprietary dual-contact cantilever holder that allows easy exchange, high resolution AC AFM imaging, and heating up to 350°C (AN2-200 probes).
- Probe heating rates up to 600,000°C/min.
- Lateral resolution capabilities to <20nm



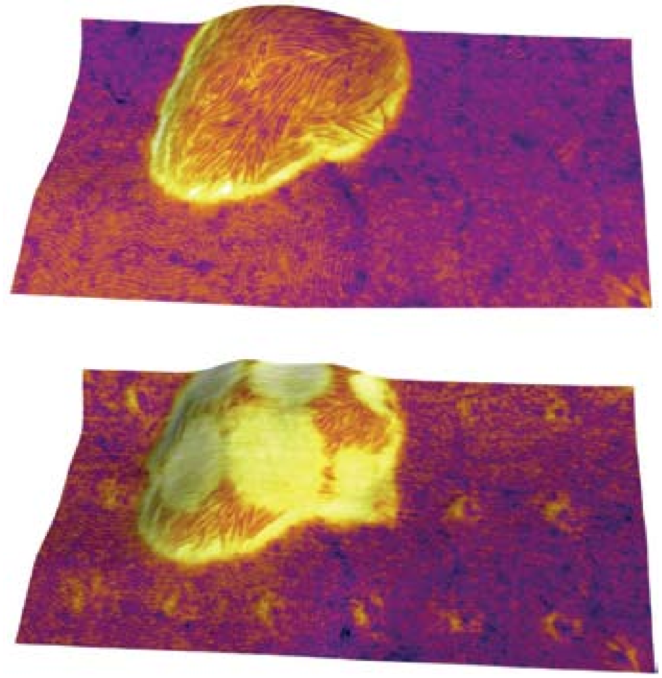
Sub-zeptoliter thermal decomposition of insulin fibers. (a) shows a 1x2µm AC (tapping) mode image of insulin fibers deposited on a mica surface. After imaging, a series of thermal-bending-compensated, low-temperature thermal cycles were performed in a 12x6 array of points. A small selection of those locations are indicated by the colored markers in both (a) and (b). (b) is an AC image of the same region after the thermal cycling was complete, showing numerous gaps in the fibers where thermomechanical decomposition has occurred. (c) shows the local thermal expansion (top deflection plots) and resonant frequency shifts (bottom plots) associated with the thermal cycles, color coded by location. Note the clear signal associated with thermal decomposition of the fibers visible in the resonant frequency shift curves. The deflection curves show no significant response at the same temperature. Note that some tip broadening has occurred during the thermal cycling that reduces the resolution between (a) and (b). Because the heating cycles were made at constant load, compensated for the thermally induced bending of the lever, the resonant shifts can be primarily attributed to thermal decomposition, rather than simple mechanical effects.

- Temperature control is fully integrated into the ARC2™ controller interface. No additional electronic boxes are required, providing natural and integrated communication between the controller and the thermal probe.
- Proprietary calibration and measurement routines improve sensitivity while reducing artifacts:
  - Integrated parasitic bending measurement and correction removes errors associated with residual stresses in the thermal cantilevers. As the probe is heated, stresses associated with manufacturing will cause some bending. *Z*therm corrects for this bending, preventing varying loads from being applied to the sample which limit the accuracy of measurements and can even cause unintended surface damage.
  - Tip-sample voltage compensation - a unique drive circuit allows the potential at the cantilever tip to be controlled separately from the heating current. This allows performance of a variety of voltage dependent techniques simultaneously with thermal measurements, for example, PFM.
  - Contact Resonance Sample Holder enables use of dynamic contact resonance modes, including DART, for sensitive probing of surface mechanical properties as a function of the local heating. Contact resonances are much more sensitive to surface properties than the deflection signal.

Specifications

- *Z*therm dual-contact, differential drive cantilever holder compatible with Anasys ThermaLever AN2-200 probes. Extra PEEK screws and 260 5x50 Wiha screw driver provided.
- Contact Resonance Sample Holder. Includes 50 epoxy packets for efficient acoustic coupling.
- 10 (ten) ThermaLever AN2-200 probes*
  - Length ~200µm, thickness ~2µm
  - Tip height 3-6µm, tip radius <30nm
  - Resonant frequency 55-80kHz
  - Maximum controllable temperature 350˚C
- License to purchase additional ThermaLever probes directly from Anasys
- Software capabilities:
  - Contact, AC, Nap, and the new DART resonance tracking imaging.
  - Point-to-point thermal mapping – Temperature, frequency, amplitude ramping at specific points on the sample surface. Can be determined with point-and-click after a reference scan or as a “force map” to create a map of thermal properties over the sample surface.
- DART specifications:
  - Frequency range 100Hz-2MHz standard
  - Frequency resolution less than 0.1Hz, sample dependent
  - Contact resonance drive amplitude variation <dB 100kHz-1MHz
- Thermal bending correction residual <5mV/V (spurious deflection/heater drive voltage).
- Differential drive allows separate heating and bias control.
  - Differential heater voltage 0V-20V combined AC and DC signals
  - Independent tip bias -10V – +10V combined AC and DC signals

**Other ThermaLever probes available on request*



Thermal data from a SEBS polymer sample. A 3x6 array of thermal probing events was performed over an area of the surface. Top image is the phase overlaid as color on topography before thermal measurements. Bottom image is after thermal measurements and shows induced local phase transitions in the SEBS materials. Phase boundaries "spreading out" in the raised feature can also be seen. 1µm x 2µm scans.

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