

nano-TA2

Sub-100nm Local Thermal Imaging and Analysis

Anasys Instruments introduces the second generation of its award winning nano thermal analysis product, the nano-TA2. This system extends the capabilities of the nano-TA system by the addition of local temperature mapping via the Scanning Thermal Microscopy (SThM) mode at a resolution of 0.1°C . This is achieved by a significant improvement in the noise levels as well as higher resolution electronics. The system also incorporates significantly more control due to the addition of a Digital signal processor (DSP), field programmable gate array (FPGA) and on board memory. This allows it to obtain heating rates up to $600,000^{\circ}\text{C}/\text{min}$ (100x higher than before) as well as improved control of the heating rate in slower temperature ramps.

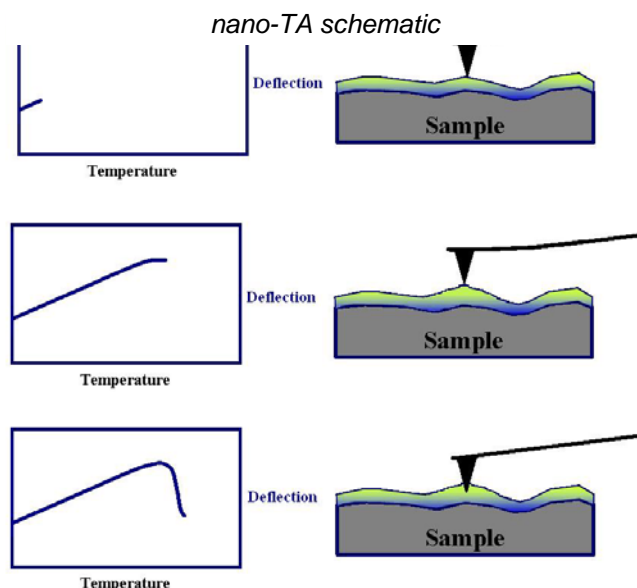
The nano-TA2 is an accessory (comprising hardware, software and probes) that enables a number of commercially available AFMs to perform nano Thermal Analysis (nanoTA), Heated Tip AFM (HT-AFM) and SThM.



The nano-TA2 electronics comprising the power supply, TMA controller and CAL box.

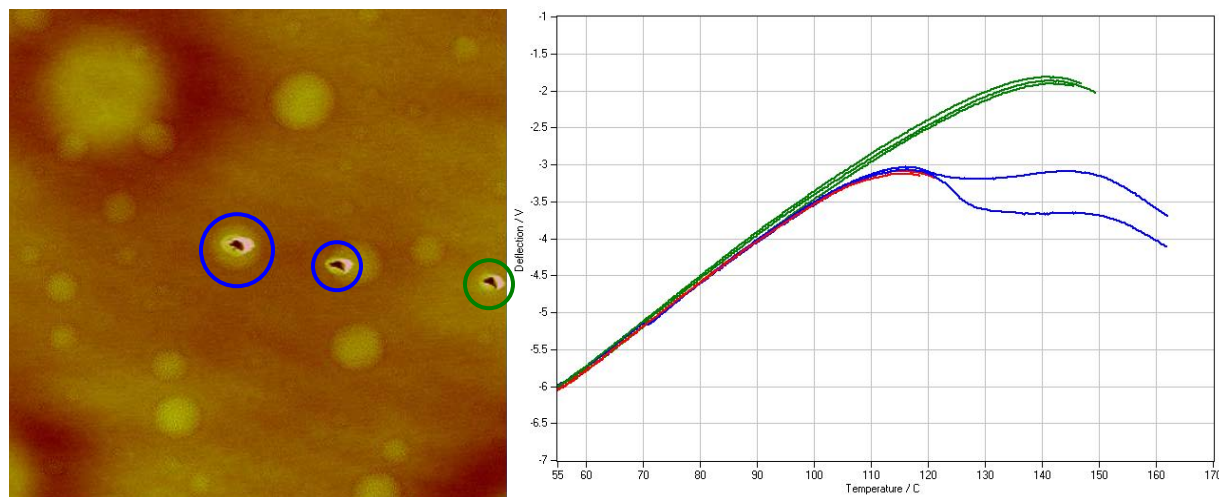
nano-TA

In the nano-TA mode, the user visualizes the sample at a typical resolution of better than 30 nm, identifies the regions of interest and then positions the probe using the AFM to subject



the region in contact with the probe to a thermal ramp. Shown to the left is a schematic of the nano Thermal Analysis mode showing the initial upwards deflection of the cantilever due to thermal expansion followed by the penetration of the probe due to softening or melting. This will typically happen at the glass transition or melt transition of the sample. The heating of the probe is due to a high resistance region incorporated into a silicon probe that is otherwise much like a standard silicon probe. This allows rapid, controllable heating up to by controlling the voltage applied to the probe using the nano-TA2 electronics. During the ramp, the operator can monitor, using the included software, up to four of a range of signals available including the

deflection of the cantilever, the resistance of the thermal probe or the power applied to the thermal probe. In addition, the power or resistance of the cantilever can be ramped to achieve a more controlled heating of the probe on rough or heterogeneous samples. The local heating means the system is ideally suited to make measurements of thin films, polymer blends and nanocomposites in the polymer market. In the pharmaceutical market, the system can be used to study spatial mapping of crystallinity, identification of amorphous / crystalline regions in a semi-crystalline sample, mapping of polymorphs, identification of components in compressed tablets and analysis of tablet coatings.

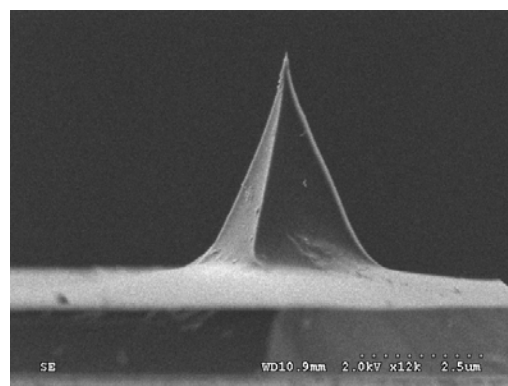


A scan of a PS-PMMA blend (left) showing the indents in the surface caused by the temperature ramps. The temperature ramps taken on the blend are shown on the right.

Local heating allows a number of measurements to be performed on each sample because the probe is only affecting a very local region allowing characterization of the uniformity of the sample. Shown above is a 5 μm intermittent contact mode image of a polymer blend composed of Polystyrene (PS) or Poly(methyl methacrylate) (PMMA) deposited on glass. The three features in the middle of the image highlighted by the circles are the indents caused by the probe after its temperature passed the glass transition temperature of the PS or PMMA.

The data shown above are the temperature ramps from this sample. The data from the PS (red) and PMMA (green) clearly differentiate the two materials. Also shown is data from a thin film of PS on PMMA (blue) showing the initial penetration of the PS followed by the melting of the PMMA.

In order to determine the temperature of the probe, each ThermoLever™ probe needs to be calibrated. This is accomplished using the semi crystalline polymeric calibration sample supplied with each system. These samples cover a range of melting temperatures, are very homogenous and exhibit a sharp melting transition. Once calibrated, the electrical characteristics of the silicon probes are such that the system can ramp the probe temperature from room temperature up to $\sim 400^\circ\text{C}$ repeatedly in a controlled fashion. The probe can also go above this temperature for brief periods to clean the probe. Due to the small size of the heater and the fast sampling of the nano-TA2 system, the temperature of the probe can be ramped rapidly, with rates up to $600,000^\circ\text{C/min}$. This allows accurate measurements of



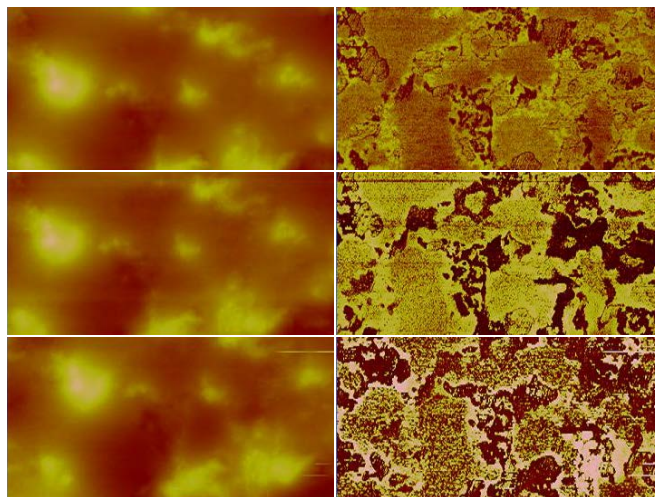
A SEM image of the AN style nano-TA probe, showing the high aspect ratio tip.

materials that may change form at slower heating rates such as polymorphs in drug compounds.

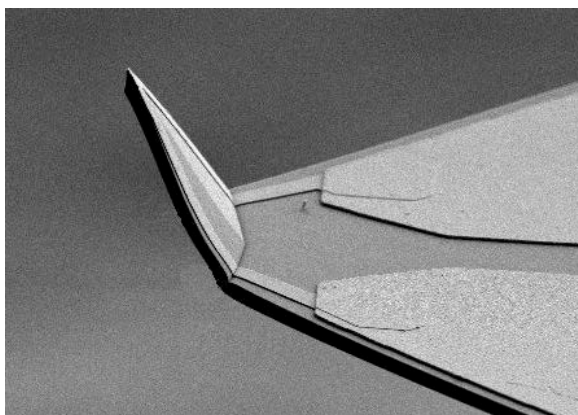
Heated Tip AFM (HT-AFM)

The ThermoLever™ probes can also be used to perform a variety of SPM measurements but with the addition of heating the probe. While this will not give the quantitative measurements obtainable with the nano-TA mode it can be used to differentiate the different materials at the sample surface. These probes have been used in the contact, intermittent contact, force curve, pulsed force mode and nanolithography while heated up to temperatures as high as 400 ° C. As opposed to sample heating stages, the temperature can be changed rapidly while inducing minimal thermal drift due to the small heated area.

Shown to the right are 3 μm images taken of a polystyrene(PS)-polypropylene(PP) blend in the intermittent contact mode showing topography (left) and phase (right) images. As the temperature is increased from room temperature (top), the PS becomes more visible in the phase image as the probe passes the glass transition temperature of the PS. At a higher temperature, the entire surface becomes soft as indicated by the decrease in contrast in the phase image.



3 μm x 1.5 μm intermittent contact images of a PS-PP Blend where the probe temperature was changed from room temperature (top) to 160 ° C (middle) to 230 ° C (bottom)



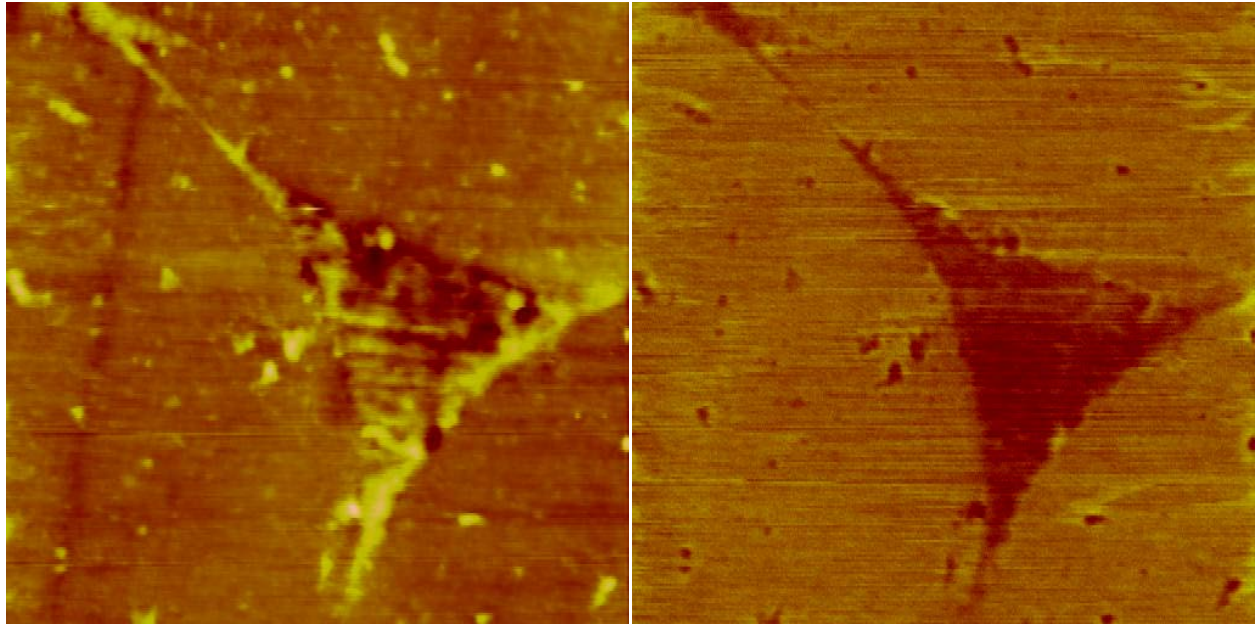
SEM image of the end of the SThM probe

Scanning Thermal Microscopy (SThM)

SThM uses specially designed probes that incorporate a thin metal film near the apex of the probe. The nano-TA2 system can very sensitively measure the resistance of this metal film which is an indication of its temperature. Shown to the left is the end of the cantilever and probe of one of the SThM probes. These probes are premounted onto a larger support to allow them to be easily mounted in your SPM as well as making good electrical contact to the metal film on the cantilever substrate.

The SThM mode works by applying a fixed voltage to the probe. This voltage can be varied which allows control of the temperature of the probe. When measuring active heated samples such as magnetic recording heads, electrical circuits or laser diodes, the voltage applied is kept low to reduce self heating. Alternately, when measuring qualitative variations in local thermal conductivity, the voltage can be increased to more easily sense the heat flow from the probe into the sample. The electronics measure the resistance of the probe using a Wheatstone bridge circuit with high gain, low noise electronics. The signal is output of the nano-TA2 electronics on a BNC connection which is connected to a spare input connection on your SPM. Thus the SThM image is generated and displayed by the SPM software.

Shown below is a 3 μm SThM image of glass fibers embedded in epoxy. The darker areas in the SThM image indicate the probe is at a higher temperature as expected for the lower thermal conductivity epoxy. These images demonstrate the <100 nm lateral resolution of this technique.



SThM image of a glass fiber/epoxy composite. The image on the left is topography and the image on the right is SThM.

Features:

- System includes TA Studio software, a power supply box, the TMA controller, a CAL box, bridge cable, calibration samples and 10 ThermoLever™ silicon probes.
- The probes come premounted for easy exchange and allow high resolution AFM imaging and heating up to 400° C for the ThermoLever™ probes. The SThM probes have a maximum temperature of 160° C.
- Rapid controllable Thermo-Mechanical Analysis with heating rates up to 600,000°C / min.
- Temperature mapping via Scanning Thermal Microscopy (SThM) with a probe temperature resolution of $<0.1^\circ\text{C}$ and a lateral resolution of <100 nm.
- Identify/characterize individual phases from their onset and peak temperatures and by measuring their thermal properties.
- Currently compatible with a number of commercially available AFMs, contact Anasys Instruments to see if your system will operate with the nano-TA2 system.

Requirements:

- AFM system must have available a spare input channel and one spare USB connection.
- The operating system for the AFM must be Windows 2000 or XP.

Controller Specifications:

Controller: 67XX DSP with FPGA
Connection to PC: USB or RS-232
Input Signals: 3 16 bit ADCs 100 ksps, 1 16 bit ADC 1.2 Msps
Input Gain (Vs-Vr): 10X, 100X or 1000X (software controlled)
SW Signals: SW can record up to 4 independent channels including the following options (Deflection, Resistance, Power, Vs, Vi, Vs-Vr, Delta Power, Vheat, and FbOut)
Output (to Probe): 0-10V or 0-40V (4 16 bit DACs, 100 ksps SW selectable range)
SThM Output: +/-4.2 V (equivalent to probe resistance)
Feedback: Digital feedback to ramp power, resistance or delta power
AC Input: BNC connector on the CAL box allowing up to 50 kHz sine waves to be summed onto the output to the probe

nano-TA/HT-AFM Specifications:

Measurement Mode: Single or Dual Probe (SW selectable)
Ramp Modes: Voltage, Power (single), Delta Power (dual)
Imaging Modes: Contact Mode / Intermittent Contact Mode (SPM Dependent)
Temp. Ramp Rate: Up to 600,000°C / min
Max. Temp. of Probe: 400°C (dependent on probe)
Probe Spring Const: ranges from 0.1 N/m to 5 N/m
Probe Res. Freq.: ranges from 20 to 80 kHz
Tip Radius: 10-30 nm
Tip Height: 3-6 microns
Cantilever Length: 200 - 350 µm

SThM Specifications:

Measurement Mode: Temperature contrast
Lateral Resolution: <100 nm (dependent on probe)
Temp. Resolution: <0.1 °C (dependent on probe)
Max. Temperature: 160°C (dependent on probe)
Spring Constant: ~0.5 N/m
Resonant Frequency: ~50 kHz
Tip Radius: <100 nm
Tip Height: ~10 microns
Cantilever Length: ~150 microns