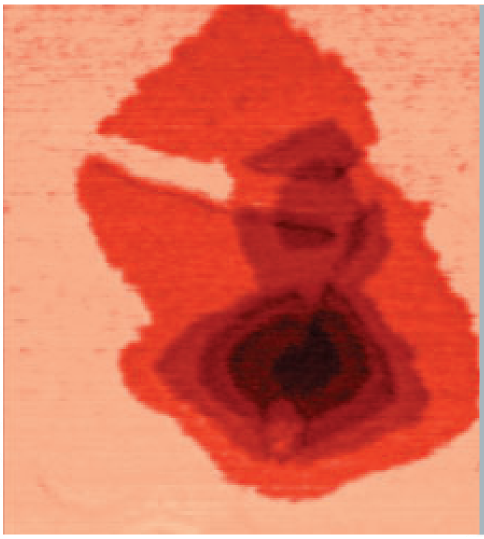
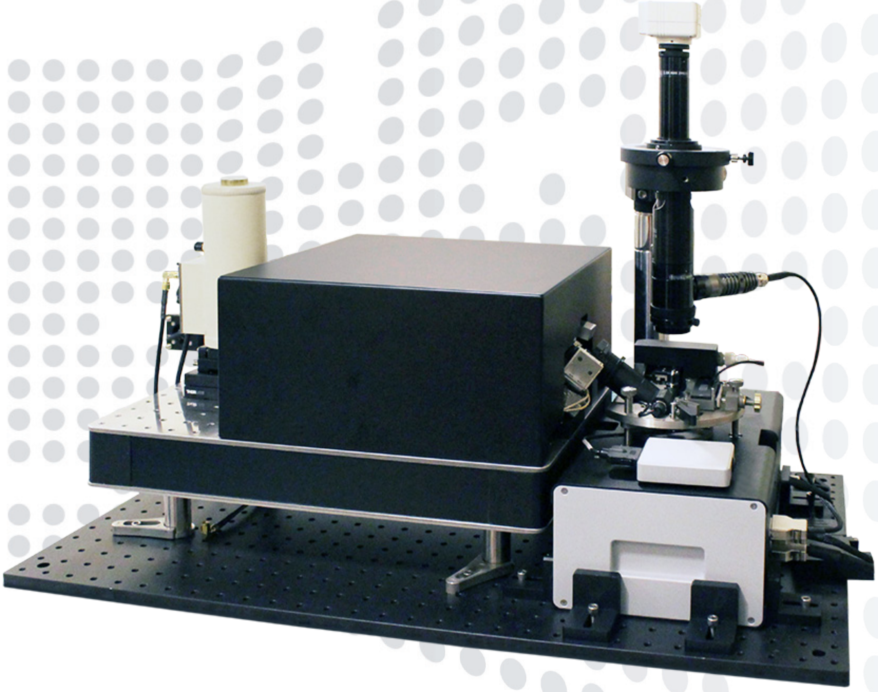
AFM — IR s-SNOM



Oligothiophene monolayers on Si, 1.5 x 1.5 µm



NTEGRA IR

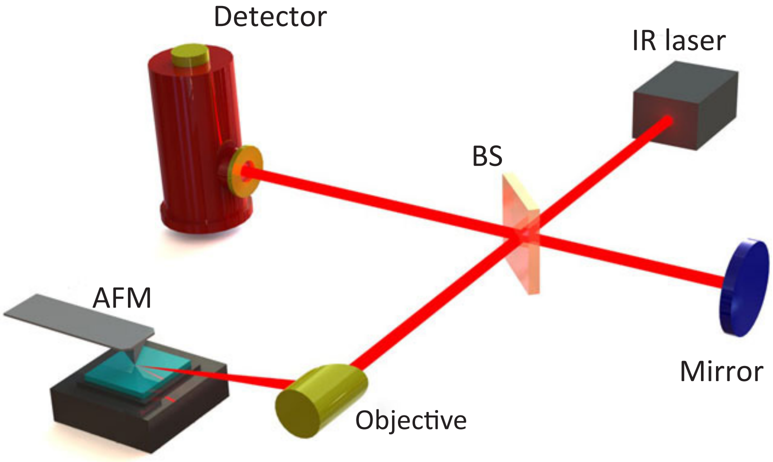
- IR s-SNOM microscopy and spectroscopy with 10 nm spatial resolution
- Wide spectral range of operation: 3-12 µm
- Incredibly low thermal drift and high signal stability
- Versatile AFM with advanced modes: SRI (conductivity), KPFM (surface potential), SCM (capacitance), MFM (magnetic properties), PFM (piezoelectric forces)
- HybriD Mode™ - quantitative nanomechanical mapping
- Integration with microRaman (optional)

OVERVIEW

NT-MDT Spectrum Instruments presents NTEGRA IR - scattering scanning near-field optical microscope (s-SNOM) designed for infrared (IR) spectral range.

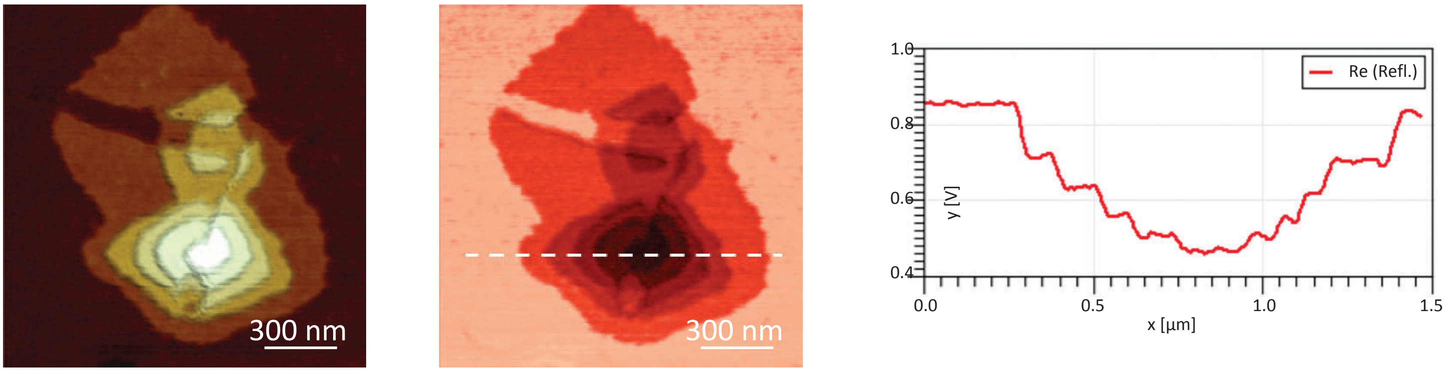
AFM probe is located in the focus of optical system which excites sample structure by IR laser and collects the optical response. Collected light is directed to Michelson interferometer for optical analysis.

Far-field component of the collected signal is suppressed by using lock-in techniques at cantilever oscillation frequencies. NTEGRA IR system allows detection of near-field signal amplitude and phase. Spatial resolution of IR s-SNOM is about 10 nm and defined only by tip size.



NTEGRA IR principal scheme

ULTRATHIN FILMS: OLIGOTHIOPHENE MONOLAYERS ON SILICON

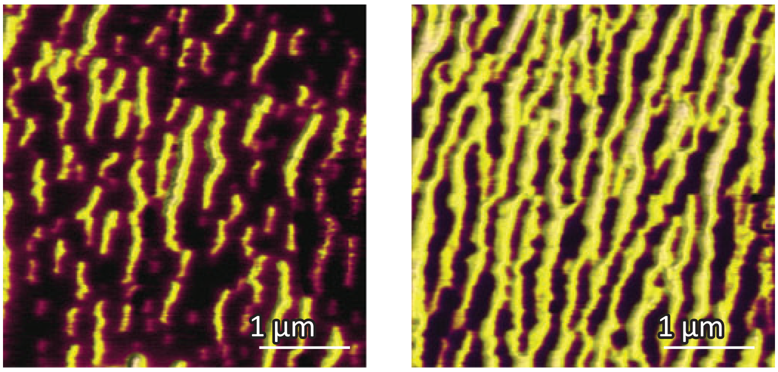


Topography (left), reflection for λ= 10.6 µm (center) and cross section of reflection signal (right).
Sample courtesy to Dr. A. Mourran (DWI, Aachen, Germany). Measured by Dr. G. Andreev (EVS Co)

IR reflection contrast of thin and soft structures easily detected. Each of five 3.4 nm steps is resolved. Spatial resolution is better than λ/1000.

PHASE TRANSITION BEHAVIOR OF VO_2 FILM

Under the heating VO_2 film demonstrates phase transition from insulator to conductor state. Same area was measured at different temperatures. Bright areas show conductive domains and dark areas correspond to dielectric domains. Above critical temperature conductive domains become connected to each other and VO_2 film demonstrates conducting properties in macroscale. Due to essential AFM parts are made of Ti system exhibit low drift and high signal stability: <1 um XY drift from 27 ^{0}C to 67 ^{0}C, no realignment of NTEGRA IR optics needed.



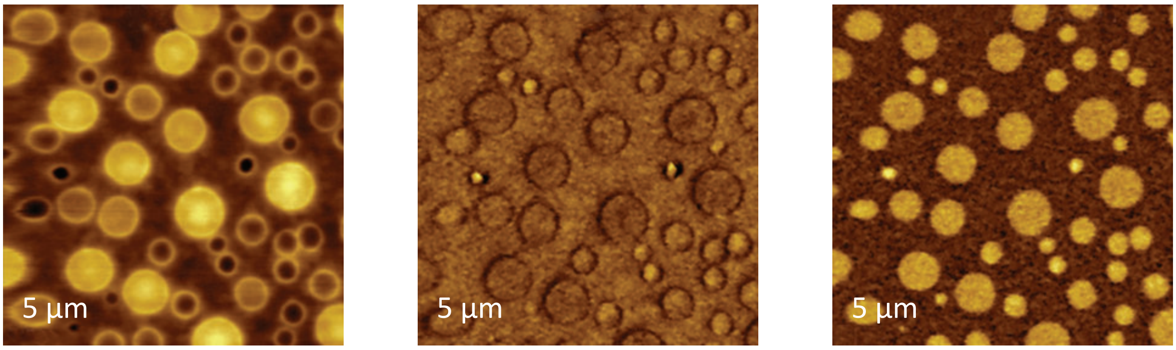
Overlay of IR reflection with topography at 55 °C (left)
and at 67 °C (right). λ= 10.6 µm.
Sample courtesy to prof. Liu (Stony Brook University, New York, USA)

Si-SiO$_2$ CALIBRATION GRATING

Height (left), IR reflection (center) images and dC/dZ map (right) of Si/SiO$_2$ grating

SiO$_2$ pads (1.5 x 1.5 µm) with ~20 nm height are grown on Si surface. Thanks to differences between the real components of dielectric permittivity (ε_r) of these materials at 10.6 µm wavelength the square pads are well distinguished in IR images. AFM-based electric measurements are applied for imaging the local variations of dielectric response of materials. The darker contrast of SiO$_2$ pads in dC/dZ map is consistent with lower value of its permittivity ($\varepsilon_r = 3.9$) compared to that of Si ($\varepsilon_r = 11$).

PS/PVAC FILM ON THE CONDUCTING ITO SUBSTRATE



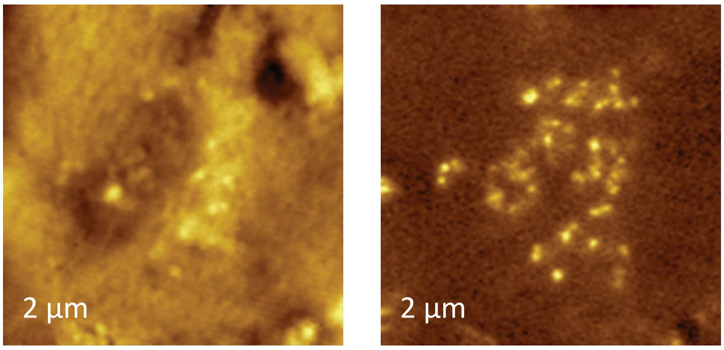
Height (left), reflection (center) and absorption (right) images of a PS/PVAC film on ITO substrate

The height image of PS/PVAC film on the ITO substrate shows the morphology in which the circular domains of different height are embedded into a relatively flat matrix. Only small contrast changes are noticed on the domains and matrix locations whereas the domains' edges are seen most pronounced. The situation is quite different in IR nano-adsorption image in which the high contrast of the circular domains allows their assignment to PVAC - the polymer that has adsorption band near 10.6 µm. These data demonstrate that the spatial resolution of IR reflection and absorption mapping is well beyond the diffraction limit of the applied IR light.

THERMOPLASTIC VULCANIZATE (TPV)

Thermoplastic vulcanizate (TPV) is a nanocomposite which is made of a blend of isotactic polypropylene and EPDM rubber that are mixed with carbon black particles. Such samples exhibit shiny black surface and, therefore, they are burned in Raman studies. The AFM/IR measurements of this sample are not only possible but they are very successful.

A number of bright nanoparticles seen in a central part of the height image can be assigned to carbon black fillers. The suggestion is strongly supported by the IR reflection image in which the carbon nanoparticles show extremely bright contrast compared to the polymer matrix.



Height (left) and IR reflection (right) images of TPV sample

SPECIFICATIONS

Lasers and light input system

Fiber coupling for easy laser source switching without realignment. Free space coupling module also available

CO_2 laser: TEC cooled, stabilized, software tunable CO_2 laser, λ = 10.3-10.8 µm with improved laser stability : less than +/-0.25 % variation in 30 min

Tunable midIR lasers in the range 4 µm - 11 µm with Mode-Hop-Free tuning range: 60 cm^{-1} typically

Displacement free attenuators. Transmission level of 0.05, 0.12, 0.2, 0.25, 0.45

s-SNOM imaging and spectroscopy system

Compact 45 cm by 30 cm footprint (45 cm by 75 cm including AFM basement)

Standard objective with aperture 0.35 NA. Broadband (3-12 µm) IR beamsplitter optimized for s-SNOM

Piezo-actuated reference mirror with closed-loop controller. Closed-loop interferometer reference arm alignment (tip/tilt). Enables remote optimization of the interferometer

Low noise LN_2 cooled MCT detector: <30 $nV/\sqrt{Hz}$ at tapping frequency harmonics (100 KHz – 1 MHz)

Open design: easy to open protective enclosure enables instant access to the interferometer. Possibility to add or replace optical elements

Modular design: all essential components are replaceable. Includes free space module, detector, beam splitter, focusing, collimating and detector lenses

HeNe the laser for tracking of IR beam and control of optical system

AFM

High-performance low noise AFM: Z-noise <0.05 nm (RMS in 10-1000 Hz bandwidth)

10 nm AFM and s-SNOM spatial resolution

Low system drift – titanium head design minimize phase drift between tip and interferometer reference mirror

Stable AFM performance at high temperatures: up to 150 ^{0}C with standard heating stage

XYZ closed-loop sample scanner 100 x 100 x 10 µm

Sample approach system. Different sample height does not require optical realignments after sample change

Focus track feature: sample always stays in focus due to sample Z-feedback

All standard SPM imaging modes are supported (>30 modes including KPFM, SRI, PFM, SCM) — combined with IR s-SNOM

Integration with HybriD Mode™ for nanoscale stiffness and adhesion properties investigation

Software

Hot-spot alignment assist: field maps enable finding the absolute maximum nanoReflection™ signal in minutes, with absolute certainty

Oscilloscope: real time observation of nanoReflection™ real, imaginary or amplitude and phase signals while aligning

Interferometer alignment: software adjustment of reference mirror tip/tilt. Better than 2 uRad precision for both tip and tilt.

Powerful export to other software (Excel, MatLab etc.)