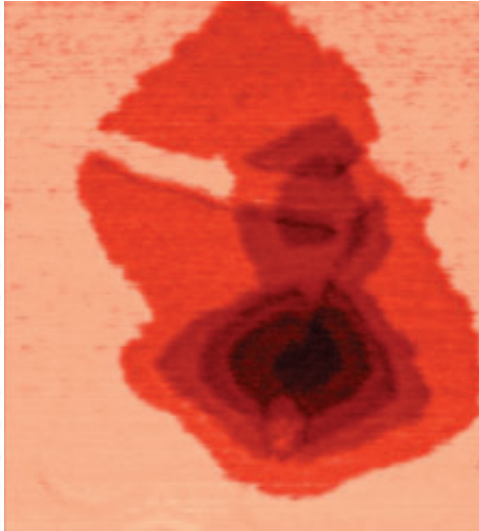
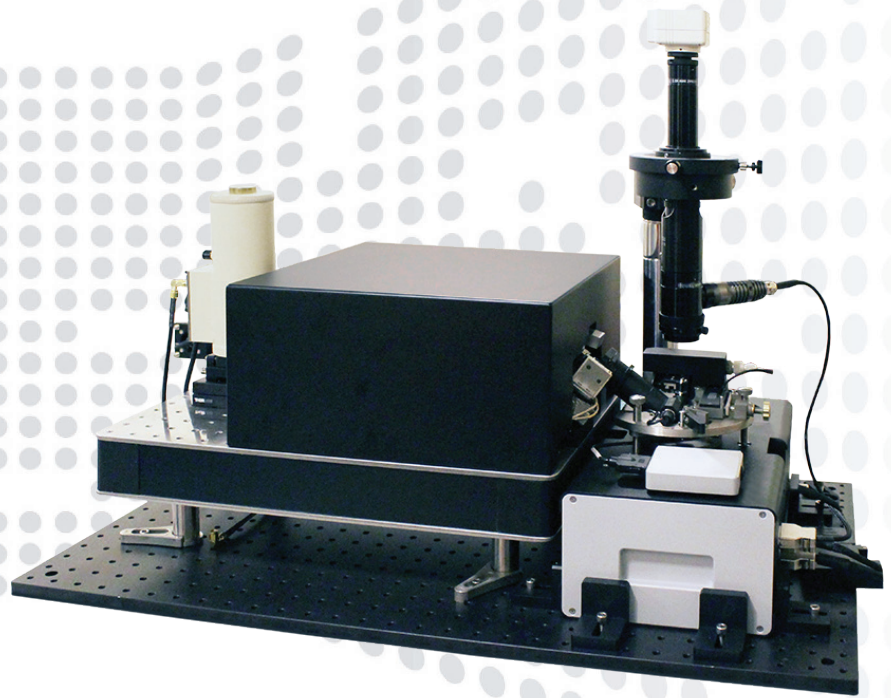


AFM — IR s-SNOM



Oligothiophene monolayers on Si, 1.5 x 1.5 μm



NTEGRA Nano IR

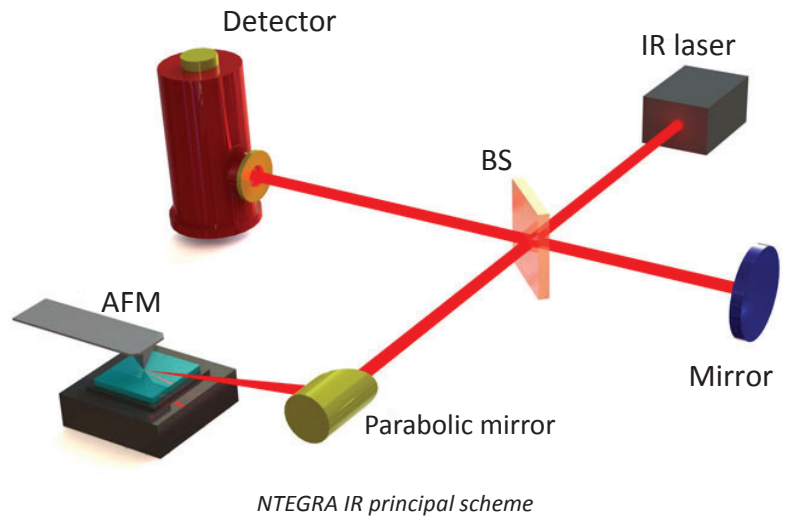
- IR s-SNOM microscopy and spectroscopy with 10 nm spatial resolution
- Wide spectral range of operation: 3-12 μm
- Incredibly low thermal drift and high signal stability
- Versatile AFM with advanced modes: SRI (conductivity), KPFM (surface potential), SCM (capacitance), MFM (magnetic properties), PFM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

OVERVIEW

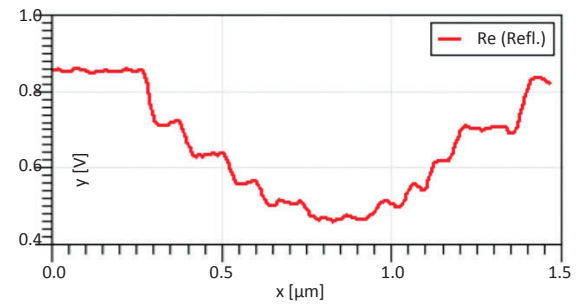
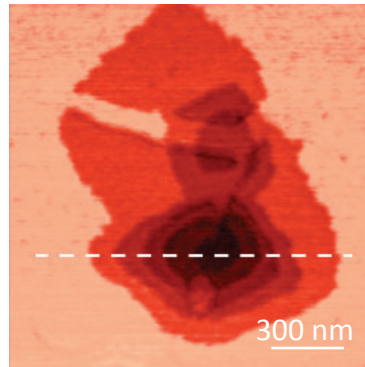
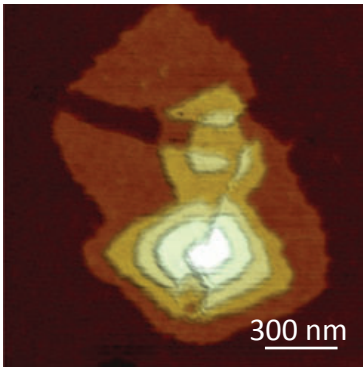
NT-MDT Spectrum Instruments presents NTEGRA IR - scattering scanning near-field optical microscope (s-SNOM) designed for infrared (IR) spectral range.

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.



ULTRATHIN FILMS: OLIGOTHIOPHENE MONOLAYERS ON SILICON



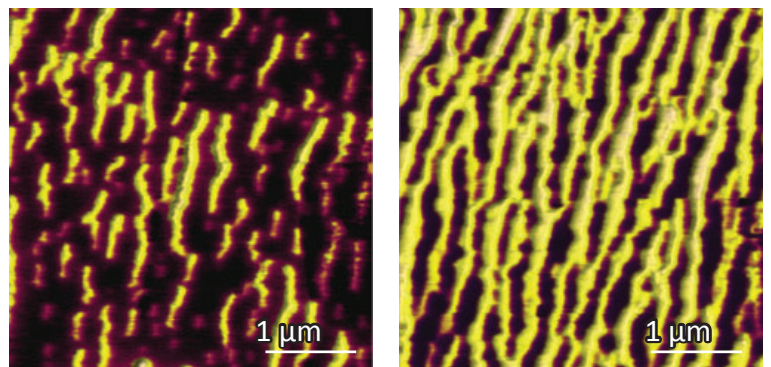
Topography (left), reflection for $\lambda = 10.6 \mu\text{m}$ (center) and cross section of reflection signal (right).
Sample courtesy to Dr. A. Mourran (DWI, Aachen, Germany). Measured by Dr. G. Andreev (EVS Co)

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

PHASE TRANSITION BEHAVIOR OF VO₂ FILM

Vanadium dioxide is a natural disordered metamaterial. VO₂ film grown on a TiO₂ substrate comprises nanoscale islands of metal- and dielectric-phase. Under the heating VO₂ film demonstrates phase insulator to metal transition. Same area was measured by s-SNOM 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 VO₂ film demonstrates conducting properties in macroscale. Due to essential parts of the AFM are made of Ti the instrument demonstrates high stability at temperatures up to 100 °C exhibits extremely low drift: <1 μm XY drift for temperature

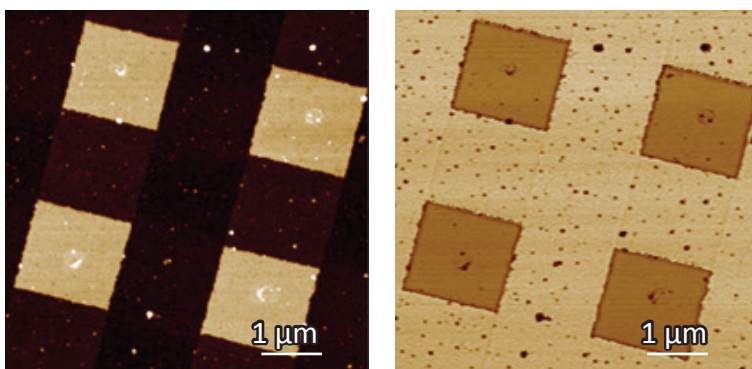
changes from 27 °C to 67 °C. No realignment of NTEGRA Nano IR optics needed.



s-SNOM reflection of VO₂ at 55 °C (left) and at 67 °C (right). $\lambda = 10.6 \mu\text{m}$.
Sample courtesy to prof. Liu (Stony Brook University, New York, USA)

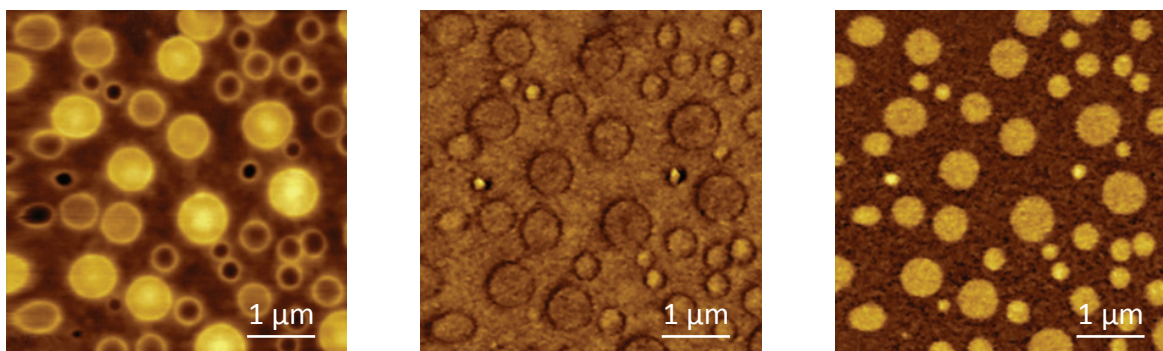
Si-SiO₂ CALIBRATION GRATING

Another example is a grating in which the square-shaped SiO₂ pads with lateral dimensions 1.5x1.5 μm and height of 20 nm are grown on Si surface. The square blocks are well distinguished in IR images by a darker contrast compared to the Si substrate. This contrast reflects the differences between the real components (ϵ') of dielectric permittivity of these materials at 10.6 μm wavelength. The darker contrast of SiO₂ pads in s-SNOM map is consistent with lower value of its permittivity ($\epsilon_r=3.9$) compared to that of Si ($\epsilon_r=11.7$).



Height (left), s-SNOM amplitude (right) of Si/SiO₂ grating

PS/PVAC FILM ON THE CONDUCTING ITO SUBSTRATE



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

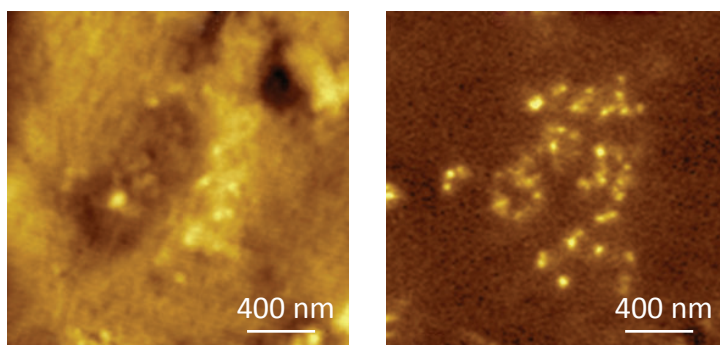
The height image of PS/PVAC film on the ITO substrate shows the morphology in which the circular domains of different height are embedded into a relatively flat matrix. Only small contrast changes are noticed on the domains and matrix locations whereas the domains' edges are seen most pronounced. The 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

Free space coupling module for easy laser source switching without realignment

CO₂ laser: TEC cooled, stabilized, software tunable CO₂ laser, $\lambda = 10.3\text{-}10.8\ \mu\text{m}$ with improved laser stability : less than +/-0.25 % variation in 30 min

Tunable midIR lasers in the range 4-11 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

High NA parabolic mirror or 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

XYZ micrometer linear translation stage and piezo-stage for hot-spot alignment

Low noise LN₂ cooled MCT detector: <30 nV/ $\sqrt{\text{Hz}}$ at tapping frequency harmonics (100 kHz – 1 MHz)

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

Controlled gas environment and temperature for highest stability and possibility to operate at IR atmospheric absorption bands

Visible laser for IR beam tracking and control of optical system alignment

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 °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

Top viewing microscope with 1 μm spatial resolution

Software

Hot-spot alignment assist: field maps enable finding the absolute maximum of s-SNOM amplitude signal in minutes, with absolute certainty

Oscilloscope: real time observation of amplitude and phase signals while aligning

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

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