

ULTRASTABLE THERMAL CABINET FOR NT-MDT MICROSCOPES

- Fan-free thermal cabinet with superb performance
- Sample temperature variations down to 5 millidegree °C
- Low thermal drift of SPM measurements of 0.2 nm/min
- Excellent acoustic and vibrational noise protection

OVERVIEW

Operating a scanning probe microscope, where the tip-sample force interactions are measured with high precision and at small scales, requires an environment that is free of the external perturbations caused by vibrational and acoustic noise. Just as important is the temperature stability of the microscope in order to ensure a low thermal drift of a sample during experiments.

A unique feature of Atomic Force Microscopy (AFM) and Scanning Tunneling Microscopy (STM) is a relatively slow feedback mechanism in most of their modes. Therefore, a large number of experiments such as imaging at the atomic-scale, profiling of corrugated surfaces, collecting of local force curves in the force volume operation, among others will benefit from low-thermal drift conditions.

OUTSTANDING PERFORMANCE

High performance of the thermal cabinet has been tested in respect to its damping of the external acoustic noise, vibrational protection, and temperature stability. A graph that shows the difference between the inside acoustic noise with the front door both opened and closed, is presented in Figure 1.

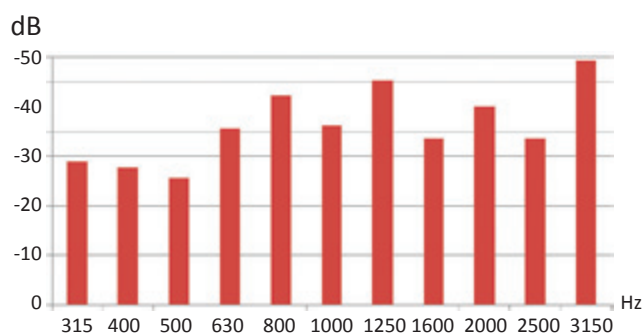


Figure 1. Demonstration of acoustic protection with sound pressure loss in a closed thermal cabinet.

The graph demonstrates a substantial reduction of the sensor signal in the broad frequency range after closing the cabinet door. In other tests the AFM deflection signal of a probe with a spring constant of 12 N/m, which stayed in the contact with a sample, was measured to detect the external mechanical vibrations.

The latter were caused either by human traffic near the cabinet or by operating a vacuum pump, which was placed at the same table as the cabinet. The deflection measurements were performed for the mi-

A stable position of the sample will also help to examine the same surface region with different AFM modes, making the surface analysis more comprehensive. In addressing the needs of temperature-stable and perturbation-free environment we introduce the microscope cabinet, in which temperature of the sample stage is electronically controlled at the set-point level with high precision.

For this purpose the cabinet temperature is slightly raised above room temperature and the embedded heaters support its set-point level using the fan-free heat convection mechanism. The brief description of the cabinet and its technical specifications are given below together with several practical examples of AFM imaging obtained with the microscopes operating inside the cabinet.

croscope placed on the bungee cords' support and on the active vibration protection tablet TS-150 in "off" and "on" conditions. The comparative deflection signal values, which were obtained in these experiments, are shown in Table 1.

Table 1. The AFM Probe Deflection at Different Noise Conditions and Various Microscope's Supports

| Noise Conditions | walking people | working pump on a table |
|---------------------|----------------|-------------------------|
| Protection | | |
| Bungee cords | 0.0880 | 0.1722 |
| Active table TS-150 | 0.4080 | 1.4350 |

* Numbers in the table are RMS in nm

The most important test is controlling for temperature stability and thermal drift of the microscope inside the cabinet. Typically, the set-point temperature at the sample stage of the microscope is chosen 3-4 degrees °C above the room temperature. After switching on the cabinet heaters, several hours are needed to bring the microscope to the set-point temperature. In an air-conditioned room, the temperature may oscillate as much as 3 °C.

Such fluctuations are on the extreme side, but they can be successfully dampened by the cabinet features so that the variations of the microscope temperature are only in the 8 millidegree °C range.

This situation is illustrated by the graphs in Figure 2.

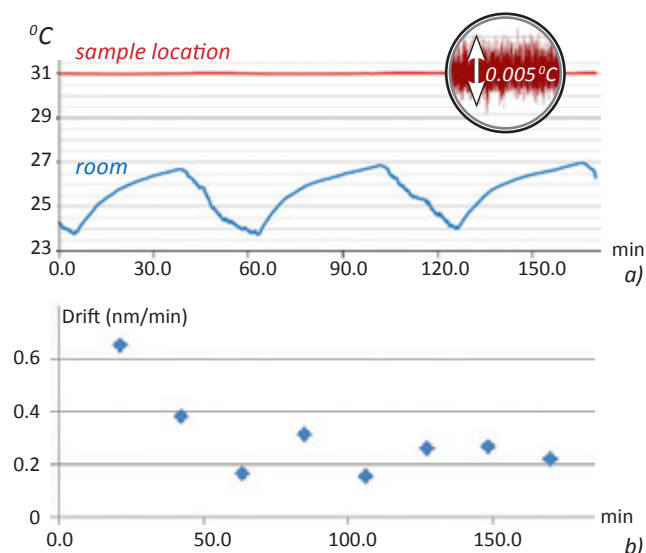
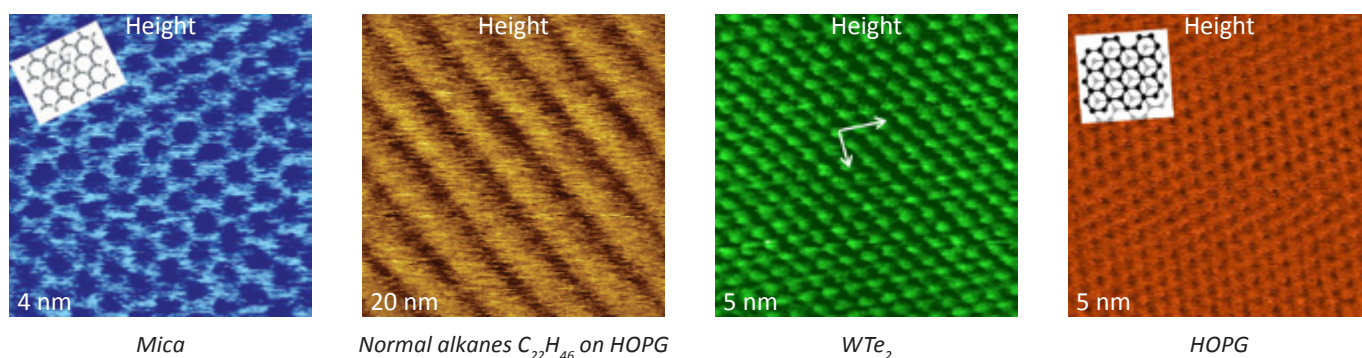
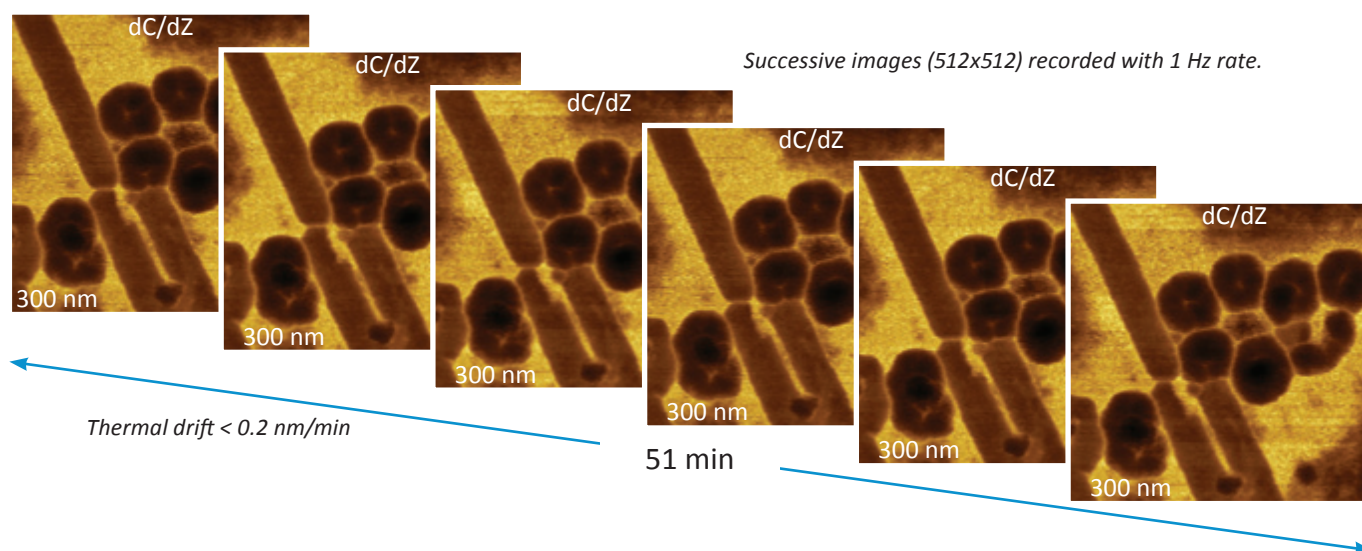


Figure 2. a) Temporal variations of room and sample location temperatures. b) A reduction of thermal drift after the cabinet door was shut.

The temperature variations proceed rather slowly with time constant around 50 minutes. Even more important, such temperature conditions lead to very small thermal drift (approx. 0.2 nm/min) in AFM images, which were obtained in the continuous scanning of the areas from 4 nm to 300 nm on side, as proven by the data below.



Figures 3. Height images (512x512) with atomic and molecular scale resolution of several compounds, which were recorded with scanning rates of 1.5 - 2 Hz in the contact and amplitude modulation mode. The studies were made with NEXT microscope inside the cabinet, and the bungee cords' damping was used.



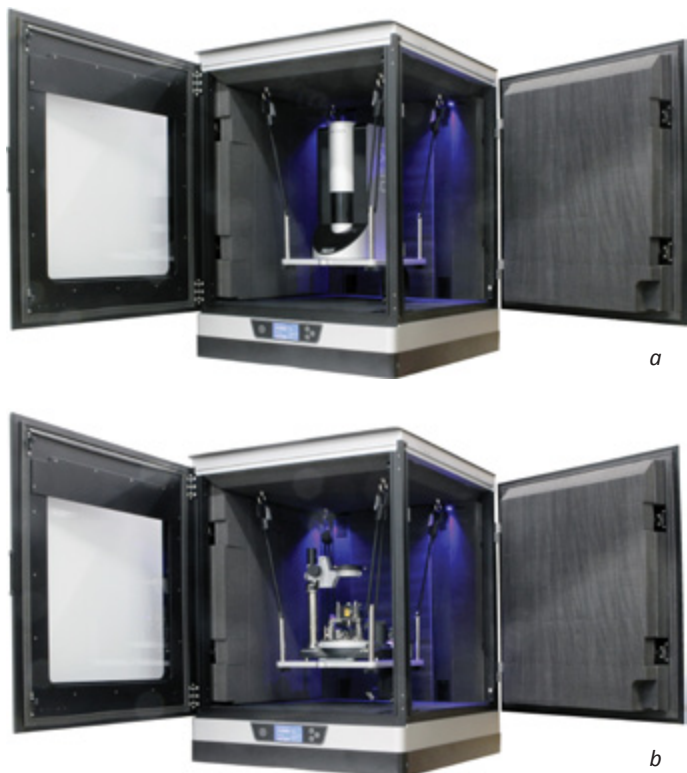
Figures 4. Demonstration of low thermal drift on the sample of self-assemblies of semifluorinated alkane on HOPG.

The conditions are further improved when the room temperature variations are smaller (down to 1 degree °C) and the temperature stability of 5 millidegree °C has been achieved in such cases. When the cabinet door is opened for a several minutes in order to change a probe or a sample, the microscope temperature drops. After the door is closed, the microscope temperature recovers to the set-point level within tens of minutes. This process proceeds gradually, and it has a minimal influence on thermal drift. Several AFM images were chosen to illustrate the microscope performance at the low thermal drift conditions. They include the atomic-scale images of mica, normal alkane $C_{22}H_{46}$ on graphite, WTe_2 , HOPG (Figure 3) and a series of the dC/dZ images of semifluorinated alkane on HOPG (Figure 4).

These dC/dZ images were recorded with the scanning rate in the 1-2 Hz range that allows precise recording of the surface corrugations reflecting atomic and molecular arrangement in these samples. The practically immobile positions of donut-shaped and ribbon-like self-assemblies of semifluorinated alkane (Figure 4) underline low thermal drift of the microscope.

HIGH-END DESIGN

The main frame of the cabinet is made of metal and it has enough room for the NT-MDT scanning probe microscopes NEXT, NTEGRA Prima and Nano. The cabinet walls incorporate foam, which suppresses any outside acoustic noise. The construction of the cabinet allows the wide opening of the front door and side doors (Figures 5a-b) for easy access to the cabinet inside during the installation of a microscope and cable connections.



Figures 5a-b. Photos of the thermal cabinet with NEXT (a) and NTEGRA Prima (b) microscopes.

The front door additionally includes a double-pane glass window. The cables, which connect the micro-

scope with the outside electronic units, are installed through a port on the back wall of the cabinet.

The port is equipped with a damping fixture that prevents the outside vibrations from being transmitting by the cables. Inside the cabinet, the microscope can be placed either on a metal plate supported by a four bungee cords, which are attached to the cabinet's ceiling, or on the passive or active vibration protection tablet (e.g. TS-150). The assembly of the bungee cords with the plate has means to regulate the bungee cord active length and to level the microscope sitting on the plate. Two heaters and vertical convection passes are embedded into the back wall. The electronic control of the heaters, which also includes the temperature sensors for room temperature and the sample location in the microscope, are performed with the control unit placed at the bottom part of the cabinet. The temperature readings are displayed on a small screen at the cabinet's front bottom.

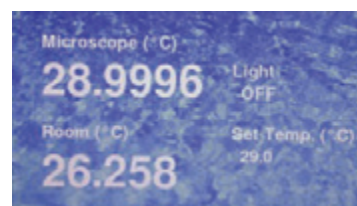


Figure 6. Screen of temperature readings.

The actual microscope temperature is shown with 6 digit precision (Figure 6) and the noise level is approximately at 0.25 millidegree °C. The control unit has a USB port for a possible communication with an external computer for recording the long-term temperature changes.

SPECIFICATIONS

Temperature Stability of Sample Location:

- for 3 degree RT variations - 1.3 millidegree °C RMS;
- for 1 degree RT variations - 0.6 millidegree °C RMS.

Temperature Recovery Time*: < 20 minutes.

*The cabinet was opened for 2 minutes.

Acoustic Protection: the averaged sound energy loss -30 dB for 300 Hz – 3 kHz bandwidth.

Vibration Protection: the noise level is characterized by 0.09 nm probe deflection for the passive and active damping

Dimensions (DWH, mm): 812 x 901 x 1079