Surface Analysis Technology Vacuum Components

SPECS<sup>®</sup>

Surface Analysis System Software

Computer Technology

# STM 150 Aarhus -High Stability Temperature Control

## **Application Note**

SPECS has developed a new temperature design for the original STM 150 Aarhus system. Excellent performance in terms of mechanical stability and thermal control could be demonstrated for  $LN_2$  temperatures and temperatures exceeding 1000°C.

## New features

- Extremely accurate sample temperature control and stability at liquid nitrogen (LN<sub>2</sub>) temperature by permanent cooling of the extra heavy mechanical scanner-platform during STM operation
- Very low drift rate in the complete temperature region due to high symmetric set-up and dedicated heat flow management.

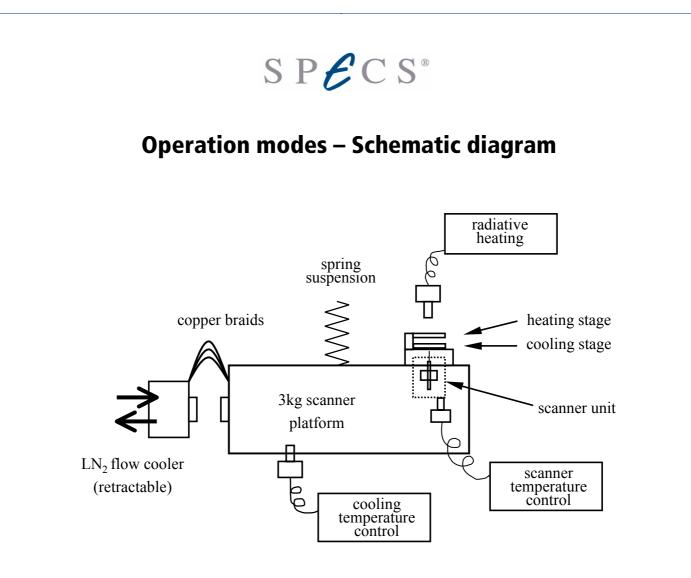


Figure 1: Schematic diagram of STM 150

#### Low temperature operation

- (1) Initial cool-down of the scanner-platform with LN<sub>2</sub> cooler-piston
- (2) Insertion of sample holder in the cooling position, release of scanner platform
- (3) STM operation copper braids maintain  $LN_2$  temperature during STM operation

#### High temperature operation

- (1) Insertion of sample holder in the heating position, release of sample stage
- (2) Heat STM scanner to 50°C, electronics takes control
- (3) Start STM operation, heat sample to desired temperature

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# Low temperature operation

## Initial cool-down of the scanner-platform

To operate the STM at low temperature the scanner-platform first has to be cooled down by a  $LN_2$  cooled piston.

The improved mechanical contact of the cooling piston allows reaching 100K within 60 minutes.

For STM measurements the piston is subsequently retracted.

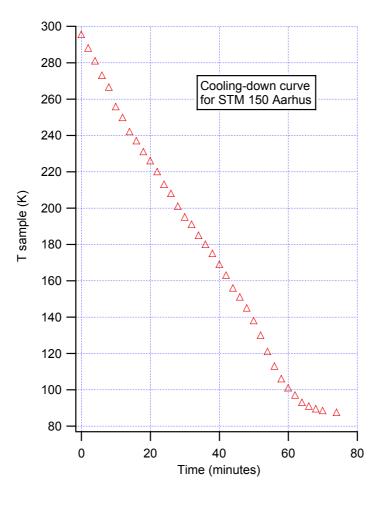


Figure 2: Initial cool-down of the scanner-platform

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### Heat flow at low temperatures

A thermal connection of the cooling piston to the sample stage by flexible copper braids was introduced. They compensate the small heat flow due to environmental radiation and scanner heating.

The cooling braids contact the extra-heavy scanner-platform and do not affect the excellent stability of the STM150 Aarhus.

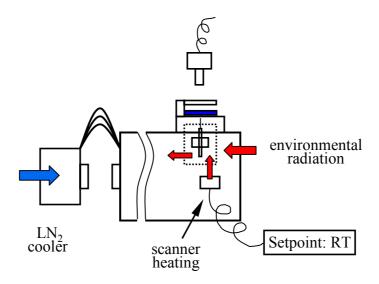


Figure 3: Heat flow at low temperature operation

### Stability of sample temperature

- Range: Stabilisation of any sample temperature higher than 115K
- Extremely high temperature accuracy of 0.1K
- Extended operation range down to 90K (with a warm-up rate of maximum 6K per hour)

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#### In situ temperature variation

Because of the permanent connection to the  $LN_2$  cooler a cycling of sample temperature during measurement is possible.

The maximum in-situ cooling rate is higher than 10 K/hour for temperatures higher than 150K. The maximum heating rate is 40K/hour.

A very fast cooling can be achieved ex-situ (interruption of tunnel-contact) by the use of the cooling piston (see also Figure 2).

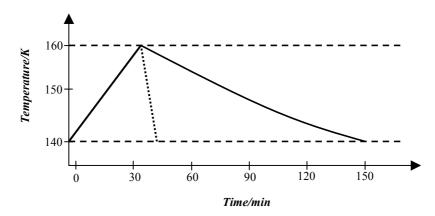


Figure 4: Example of heating ("flashing") and cooling of the sample during STM measurements. The dotted cooling curve represents the use of the cooling piston.

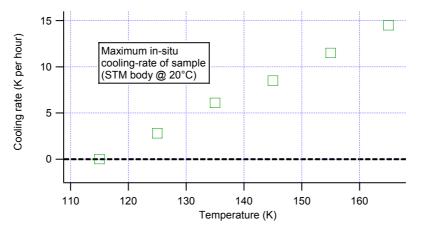
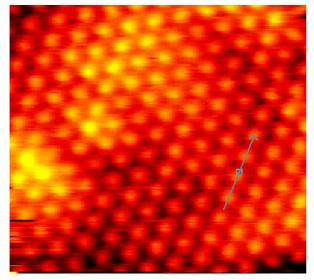


Figure 5: Maximum cooling rate during STM operation as a function of temperature

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### **Results at SPECS**



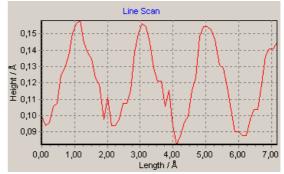
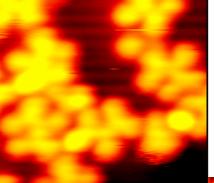


Figure 6: Herringbone reconstructed Au(111), scan size 2.5nm, T=115K; linescan  $LN_2$  cooling running



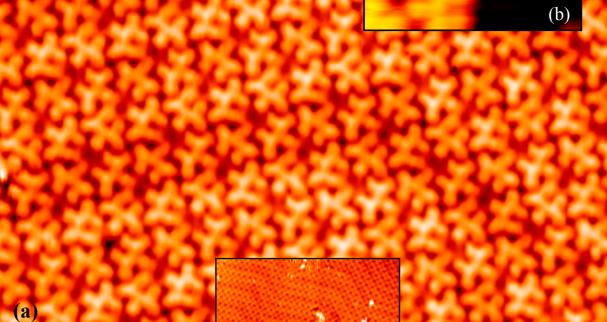


Figure 7: Oligopyridine/Au(111), (a) size 23x12nm, T=265K, 1.49V, 0.04nA, inset 60x22nm; (b) size 4nm, 1.32V, 0.05nA, T=163K Data courtesy of Harry Hoster, University of Ulm (Germany)

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# **High temperature operation**

A radiative heater heats up the sample and the  $LN_2$  cooler acts as a heat sink for the complete system. The STM can be run in thermal steady-state condition. The only object that changes it's temperature during operation is the sample. All other parts of the instrument stay at constant temperature. This way, thermal drift is minimized.

#### **Heat flow management**

The instrument uses a combination of permanent cooling and counter-heating of the scannerplatform and the scanner. Both heaters are regulated, compensating the changing power flow induced by changing the sample temperature from 1000°C to e.g. 500°C.

The scanner is set to a slightly higher temperature (50°C) and controlled. The scanner platform acts as a heat sink for the power flow from the hot sample to the scanner.

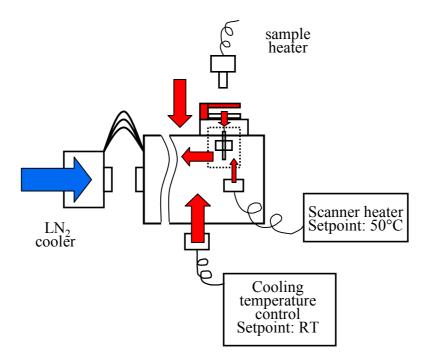


Figure 8: Heat flow at high temperature operation

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### **Results obtained at SPECS**

The following measurements are obtained with a Si(111) sample prepared (flashed) on the STM150 Aarhus.

The images are taken from a series cycling from room temperature up to around 1100°C and back again. The drift in z-direction was negligible during the full experiment. The lateral drift was only significant during temperature changes.

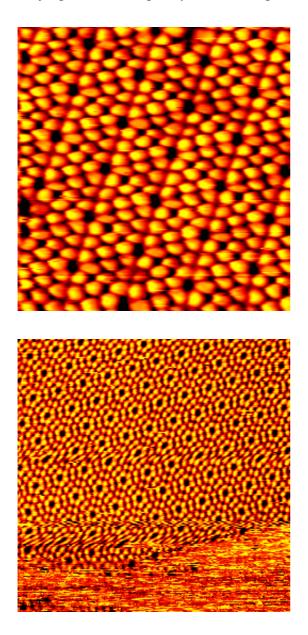


Figure 9: Si (111)-7X7 at 200°C, 0.19nA, 0.65V

Figure 10: Si(111)-7X7 at 950°C. Coexistence of 7x7 20x20nm, 0.73nA, 2.09V and 1x1 phase.

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At 1100°C, the lateral drift is only some nm per minute. The domination effect is the mobility of the steps and not the drift of the instrument.

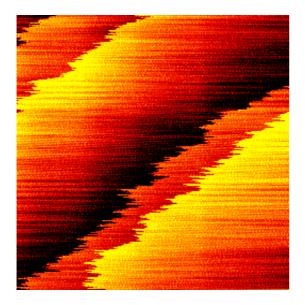
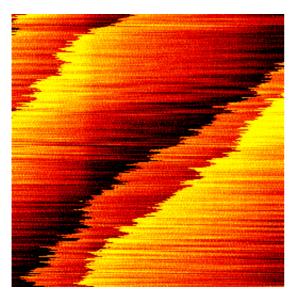


Figure 11: Si (111) at 1100°C. Only 1x1 phase is observed. 15x15 nm, -0.77 nA, - 1.74 V



#### Figure 12:

Si (111) at 1100 °C. Image was taken 30 seconds after the image in Figure 11. The steps do not move relative to the frame.

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