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High temperature surface imaging using atomic force microscopy

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Atomic force microscopy (AFM) is one of the most important tools in nanotechnology and surface science. Because of recent developments, nowadays, it is also used to study dynamic processes, such as thin film growth and surface reaction mechanisms. These processes often take place at high temperature and there is a clear need to extend the current operating temperature range of AFM. This letter describes a heating stage and a modified AFM that extends the maximum operating temperature to 750 °C. Atomic step resolution is obtained up to 500 °C in ambient and even up to 750 °C in vacuum. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2836943]

Atomic force microscope[1] (AFM) has become a well established technique to image surface properties in liquid, air and vacuum. This makes AFM a suitable research tool in several scientific fields such as biology, physics, and chemistry. In these fields, AFM is utilized to image, for example, surfaces, growth phenomena, phase transitions, and reaction mechanisms. Many of these phenomena take place at elevated temperature and are not accessible by AFM since the maximum operating temperature is often limited[2] due to the excessive heating of the heat sensitive elements in an AFM setup, such as the piezoelectric scanner. In many dynamic AFM setups, a second piezoelement is located directly below the cantilever body to excite the cantilever. The piezoelectric coefficients of both elements are temperature dependent and the maximum operating temperature must be well below the Curie temperature to avoid depolarization. Furthermore, temperature variations of the piezoscanner must be avoided. We have developed a small thermal mass heating stage to extend the maximum operating temperature.[3–9] This heating stage is suspended in such a way that the thermal conduction losses and heat transfer to the AFM head is minimized. Furthermore, we minimized heating of the piezoscanner by isolating it from the heat source. By decreasing the thermal load on the AFM, other issues are reduced as well, such as thermal drift due to lateral and vertical thermal expansion of the system components, contamination due to thermal evaporation of heat sensitive elements as well as the resonance frequency drift of the cantilever. This enables high temperature AFM measurements both in air and vacuum. In this letter, the developed hardware, i.e., the heating stage and extender tube to isolate the piezoscanner from the heat source, as well as their performance are described.

In our experiments, we used an inverted fiber AFM, based on a commercially available AFM (Ultraobjective from SIS GmbH, Herzogenrath, Germany). Only vacuum compatible components are used and, as mentioned above, the piezoscanner is thermally isolated from the heat source. This is done by placing a 20 mm long Macor tube on top of the scanner.[10] The geometry of the electrodes of the piezoscanner is such that, at the maximum applied voltage, no voltage breakdown is expected in the pressure range of interest.[11] Since this AFM is based on interferometry, a second piezoelectric element is located directly beneath the cantilever body (see Fig. 1). This element is used to actively tune the interferometer[12] and to excite the cantilever in dynamic mode. Especially at variable temperatures, this element is required to tune the interferometer. In contrast to the piezoscanner, this piezoelement is less sensitive to temperature variations.[13] Using the modified AFM, the maximum operating temperature merely depends on the thermal load. To minimize this load, we developed a low power heating stage. Rigid connection wires are used to suspend the heated area (2×2 mm^2) almost freely and, therefore, the heat losses are minimized (see Fig. 1). A heater filament (Pt200 resistor) embedded in a rigid ceramic plate ($2 \times 4 \times 1.2$ mm^3) is used to heat this area on which samples with dimensions up to 2×2 mm^2 are glued. Thermally conducting platinum paint is used to improve the temperature homogeneity and to minimize the temperature gradient between heater and sample surface.

The filament resistance is directly related to the filament temperature[14] and is reliable up to 850 °C (Ref. 15) with an accuracy of ±15 °C. Therefore, no additional thermocouple or pyrometer is required. The AFM performance has been tested at elevated temperatures in a homebuilt vacuum chamber.[16] We have used noncoated cantilevers[17] since at elevated temperatures coated cantilevers can bend due to a

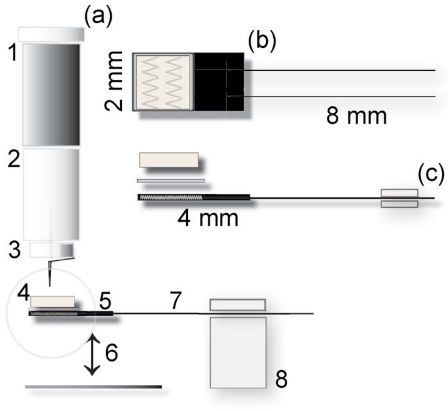

FIG. 1. (Color online) Side view (a) of AFM and heater configuration. 1: piezoscanner, 2: Macor tube, 3: second piezoelement, 4: sample, 5: ceramic plate (Al_2O_3) with embedded heater filament, 6: Gap, 7: connection wires, and 8: support block to clamp the wires, top (b) and side (c) views of the heater.

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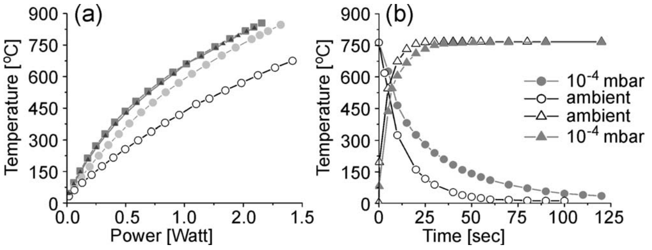

FIG. 2. (a) Sample temperature as a function of input power and pressure, ■ and ▲ 10^{-4} mbar, ● 10^{-1} mbar, and ○ atmospheric pressure. (b) Quenching and heating response of the heating stage in ambient (○, △) and 10^{-4} mbar (●, ▲).

difference in thermal expansion coefficients and contaminate the setup due to thermal evaporation. Vicinal $SrTiO_3$ substrates have been used to study thermal drift and demonstrate atomic step resolution at elevated temperatures.

The required input power to reach a stable heater temperature as a function of pressure is given in Fig. 2(a). This input power corresponds to the total heat loss. As expected, the heat loss increases with increasing pressure and temperature. Increased radiation losses appear above heater temperature of 300 °C and convection losses above 10^{-2} mbar, as observed from the nonlinear behavior and the clear drop in temperature, respectively. At atmospheric pressure, the heat loss is dominated by convection. Unfortunately, literature data on heating stages for SPM cannot be easily compared since power data is often not specified. Our low power heaters are easy to implement and inert to many environments, such as O_2. Since the heating stage has a small thermal mass it can be quickly quenched and heated [see Fig. 2(b)]. Quench and heating rates of 25 and 50 °C/s, respectively, have been measured. Furthermore, the heater temperature is quickly stabilized.[18]

Static mode and dynamic mode measurements have been performed at elevated temperatures to test the setup. Of these two modes, dynamic mode AFM at elevated temperature is the most challenging. The two detection schemes for dynamic mode, amplitude modulation[19] and frequency modulation,[20] are based on the feedback of the cantilever amplitude and frequency, respectively. In both schemes, the cantilever free resonance frequency and amplitude has to be constant. The resonance frequency is related to Young's modulus and the geometrical shape and, therefore, heating will shift the resonance frequency and amplitude since the cantilever will expand and Young's modulus will decrease with temperature. For a rectangular cantilever, the resonance frequency[21] is expressed by

$$f_0 = 0.162\frac{t}{l^2}\left(\frac{Y}{\rho}\right)^{1/2}.$$

In this expression, Y=Young's modulus, l=length, t =thickness, and ρ=mass density. For the used cantilevers a resonance frequency decrease of 5.2 Hz/K, mostly due to a change in Young's modulus, has been found by Ivanov *et al.*[6] To study the shift in resonance frequency due to heating, the cantilever is excited directly above the hot sample surface and the steady state free cantilever resonance frequency is measured (see Fig. 3). The measurement time for every data point has been set to half an hour to stabilize the cantilever temperature. Above a heater temperature of 300 °C, a clear decrease in resonance frequency is observed. This can be expected since the radiation increases above 300 °C, leading to an increased cantilever temperature. The total shift in the resonance frequency can be used to estimate the cantilever temperature. In our case, a decrease of 450 Hz is found for an increase of the heater temperature from RT to 750 °C. This frequency shift corresponds to an increase of the cantilever temperature of ~90 °C. This temperature increase is pressure dependent. At ambient conditions, a cantilever temperature increase of 90 °C is found at a heater temperature of ~500 °C.

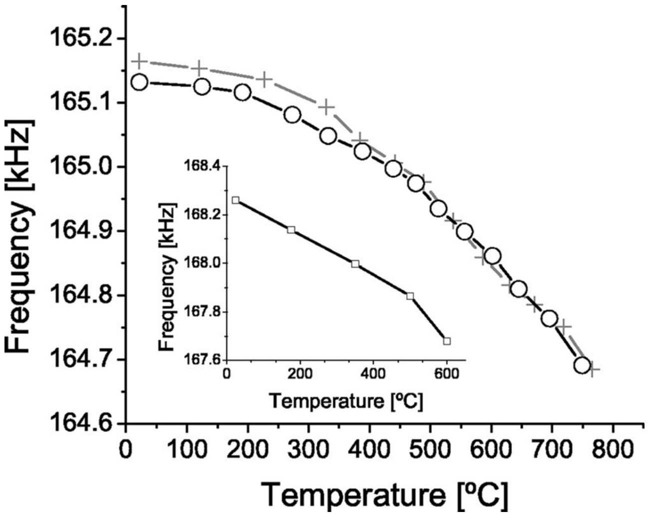

FIG. 3. Free cantilever resonance frequency measured twice in 10^{-4} mbar as a function of heater temperature. Inset: frequency decrease at atmospheric pressure.

Although the cantilever temperature increase is less in vacuum compared to ambient, AFM measurements in vacuum are more sensitive to temperature variations. Since the full width at half maximum (FWHM) of the resonance peak decreases with pressure, cantilever temperature variations should be limited to avoid unstable AFM performance at low operating pressure. Typically, a FWHM of ~50 Hz is found at a pressure of 10^{-1} mbar.

In a controlled vacuum environment (10^{-1}–10^{-5} mbar) dynamic mode AFM measurements up to 750 °C have been performed (see Fig. 4). The images, extracted from dynamic mode measurements carried out at 750 °C in 10^{-2} mbar O_2, show a vicinal $SrTiO_3$ surface with $SrTiO_3$ nanoclusters. The cluster and step heights correspond to the $SrTiO_3$ unit cell lattice constant (~0.4 nm). This indicates that silicon cantilevers are suitable to measure on hot surfaces with atomic step resolution. The temperature gradient across the extender tube is such that the piezoscanner is warmed up at most by a few degrees. The piezoscanner properties are therefore equal to those at room temperature. In these measurements the initial lateral drift is ~50 nm/min which reduces to 7 nm/min (comparable to the room temperature drift) after thermal stabilization.[22] At ambient conditions, AFM measurements up to 500 °C have been performed, the highest AFM operating temperature obtained in ambient.

In conclusion, we have demonstrated a simple setup to extend the maximum AFM operating range in ambient and vacuum in static as well as in dynamic mode by several hundreds of degrees. Our heater is designed such that the input power is minimized, and it can be operated in all kinds of environments such as ambient, oxygen, as well as UHV.

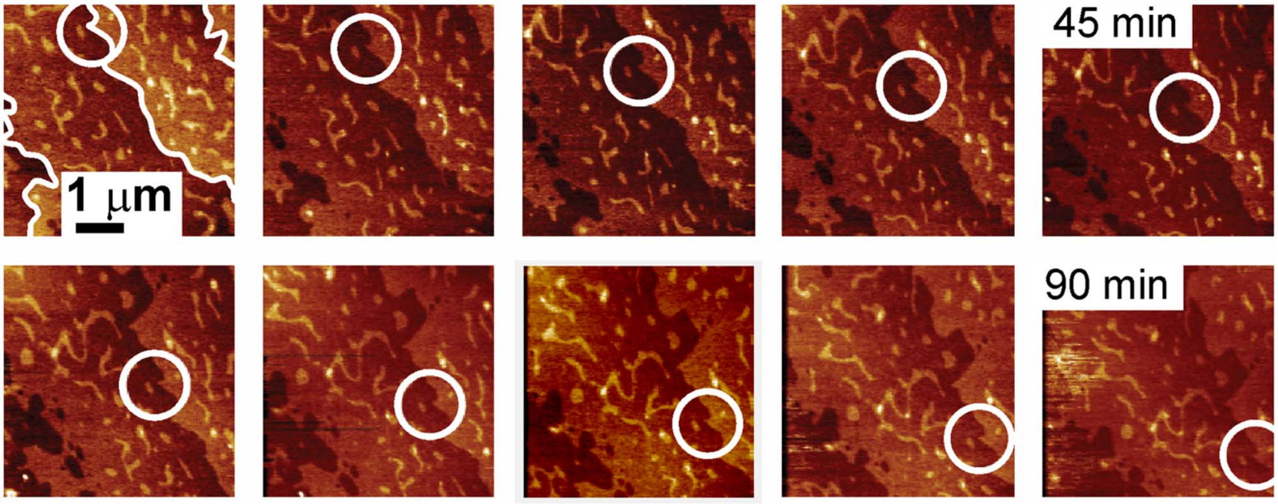

FIG. 4. (Color online) AFM topography images of a $SrTiO_3$ surface measured at 750 °C in 10^{-2} mbar O_2. In the first image, the substrate step ledge is colored white as a guide for the eye. Furthermore, markers are placed in the subsequent images to visualize the lateral drift for 90 min.

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[2] The temperature range for current designs is limited up to 250 °C in ambient and up to 500 °C in UHV.

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[10] As a piezoscanner we use the regularly used EBL2 from Staveley Sensors, Inc.

[11] The maximum applied potential (330 V) in combination with the electrode distance of ~1 mm prevents voltage breakdown in the pressure range of interest (10^{-6}–10^{3} mbar). See, for instance, *Breakdown of Gases*, edited by J. M. Meek and J. D. Craggs (Wiley, New York, 1978).

[12] Since we use an interferometer based AFM, the distance between the cleaved fiber-end and cantilever is tuned to the optimal working point, i.e., the maximum slope of the signal versus distance curve. The micron sized cavity is tuned with a piezoelement directly beneath the cantilever body. Since the cavity dimensions are temperature dependent due to thermal expansions of the AFM head, this element is required to preserve the optimal working point.

[13] For tuning of the interferometer, the piezoelement does not need to be calibrated, whereas to ensure accurate topography measurements the piezoscanner should be calibrated.

[14] The resistance contribution from the thick platinum wires (250 μm) can be neglected.

[15] This is the temperature for which the filament resistor is specified.

[16] In the vacuum chamber, different pressures ranging from ambient to UHV and other gas environments (such as O_2) can be used.

[17] Cantilevers are standard probes with a length of 225 μm and a free resonance frequency between 150 and 200 kHz (Nanoworld).

[18] After heating the sample from RT to 750 °C, the temperature is stabilized in less than 1 min.

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[22] The thermal stabilization time was 24 h.