Proximity heater for elevated temperature *in situ* vacuum scanning tunneling microscopy of metal surfaces

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The design and application of a radiant heater assembly for elevated temperature scanning tunneling microscopy (STM) in ultrahigh vacuum (UHV) is presented. The proximity heater is a noninvasive modification to an existing commercial room-temperature microscope and is capable of radiatively heating samples up to 650 K *in situ*. Imaging at higher temperatures should be readily accessible with other microscope construction designs. It is demonstrated that this heater is well suited for enabling an STM to capture surface morphological transformations such as the motion of atomic steps on metal surfaces at elevated temperature. Various design issues and solutions related to variable temperature UHV-STM are also discussed. We believe the approach described to be general in nature, offering a direct route to adapting UHV-STM designs for elevated temperature imaging. © *2000 American Institute of Physics.* [S0034-6748(00)04201-5]

I. INTRODUCTION

With the maturation of the scanning tunneling microscope (STM) into a stable and modular surface science tool, there is an ever-increasing opportunity to acquire real-space electronic and structural information to complement reciprocal space experiments. To this end, many commercial instruments, with minor modifications, are being installed into existing surface science experimental systems. The largest obstacles in the design and implementation of ultrahigh vacuum (UHV)-STM instrumentation are associated with managing some of the stringent stability requirements for making quantum mechanical tunneling measurable. In general, the ability of scanning probe microscopes like the STM to generate real-time local scale data comes at the expense of sensitivity to some environmental issues such as mechanical vibration and temperature fluctuations that other surface science techniques are less susceptible to. The challenge is to create ways for the STM to access key portions of experimental phase space without sacrificing microscope performance.

Substantial efforts have been made at designing microscopes capable of operating at variable temperatures since thermal energy plays such a vital role in determining surface and chemical behavior.[1–9] The focus has been on building low temperature STM systems since there are many advantages related to running experiments in cold environments. The overall stability of the microscope is enhanced and many atomic level surface phenomena become more accessible to imaging and tunneling spectroscopy measurements at colder temperatures. Many systems, particularly those associated with the study of adsorbate properties on surfaces, have been studied with superb results.[9–11] There is a wealth of interesting behavior however at elevated temperatures such as surface reconstruction, diffusion, adatom islanding, order-disorder transitions which occur on time scales that the STM is well suited to probe, as well as thermally activated interfacial chemistry. In this article, we will present a simple modification to an existing commercial UHV-STM that has enabled us to perform *in situ* imaging experiments on single-crystal metallic samples at elevated temperature.

II. DESIGN

A. General

The stainless-steel two-stage UHV chamber (Fig. 1) is supported on a steel frame that is bolted to pneumatic vibration isolators. The chamber is pumped by a 220 L/s D-I ion pump, cryoshroud and titanium sublimation pump as well as a 55 L/s turbomolecular drag pump used for pumping sputtering gases and chamber bakeouts. A base pressure of 6.5×10^{-11} Torr is routinely achieved after a standard baking cycle. During STM experiments, the turbomolecular drag pump is turned off along with its mechanical forepump to eliminate vibrational noise. The upper level of the chamber is equipped with standard tools for sample preparation and characterization (Fig. 2). A retractable four-grid low-energy electron diffraction (LEED) optics is used for surface diffraction, and surface cleanliness is checked by Auger electron spectroscopy utilizing the optics as a retarding field analyzer (RFA). A residual gas analyzer performs routine partial pressure analysis. Dosing of sputtering and reagent gases by chamber backfilling is done using high precision leak valves. A sample stage with two docking stations, one for electron bombardment heating and the other for resistive heating, is mounted on an XYZ translator and polar drive. Single-crystal metal samples are cleaned by Ar^+ sputtering cycles in combination with annealing by electron bombardment.

The lower level houses the STM and a load-lock assembly for transferring samples in and out of vacuum. Transfer of samples between the sample preparation stage and the STM or the load-lock magnetic linear drive are accom-

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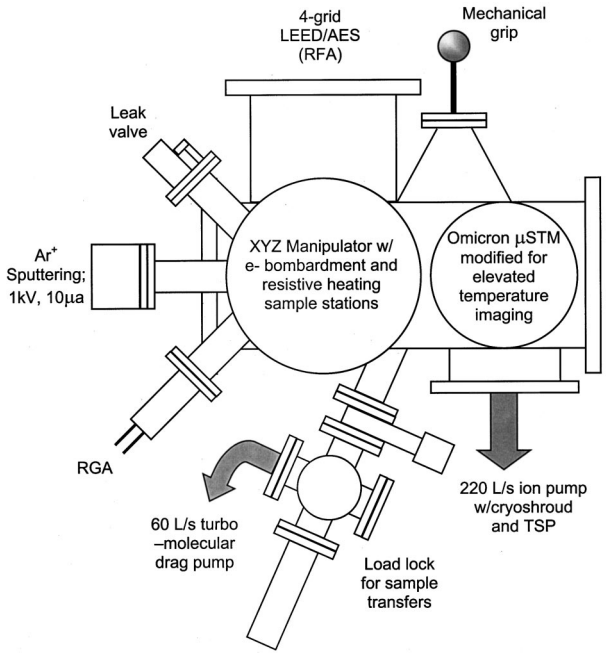


FIG. 1. Schematic top view of UHV-STM system.

plished using a versatile pincer grip with $+/-22.5°$ articulation tilt range, 360° rotation and 8 in. of linear travel. The microscope used in the system is an Omicron Micro-STM supported by a Viton® elastomer stack attached to a flange (see Fig. 3). Microscope control electronics are from Topometrix (model ECU+) which consist of a low electronic noise feedback control and scalable voltage power supply for piezo motion in conjunction with an adapter unit that operates the coarse positioning of the STM scanner and the sample in the STM. All of the modalities of the Omicron STM have been maintained with the Topometrix driving electronics. Samples are held upside down in a molybdenum holder that rests on three piezo inertia motors for coarse positioning. The tip approaches the sample from below and is held with a set screw at the top of a four quadrant piezo electric ceramic tube attached to a coarse z inertial motor. The single-crystal metal samples, typically 0.500 in. in diameter and 0.100 in. thick used in our experiments are mounted on molybdenum platens with small molybdenum clips. A

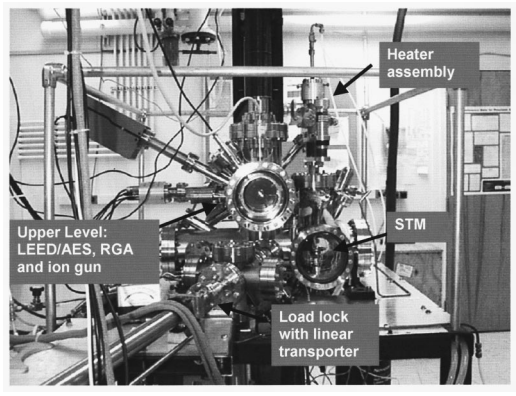


FIG. 2. Photograph of UHV-STM system from a side view which shows the two levels of the chamber.

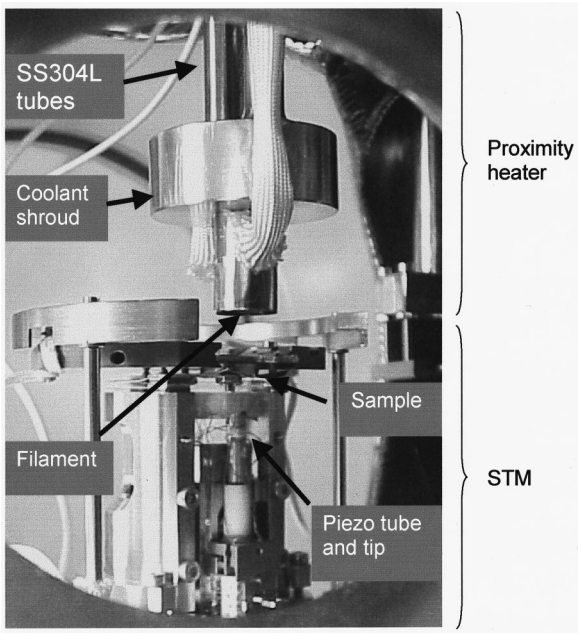


FIG. 3. Photograph of proximity heater and the Omicron Micro-STM. Note that the proximity heater assembly resides in a water or liquid cryogen cooled assembly to minimize radiative loading on nearby components; such cooling also mitigates vacuum contamination.

circular opening is machined in the platens for direct access to the rear of the sample for heating.

B. Heater construction

The key features of the proximity heater design are the complete mechanical isolation, i.e., there are no physical connections between the heater and the sample, and the management of heat from the filament. The radiant heater is a shaped tungsten filament held in a water-cooled stainless-steel shroud suspended from two 0.250 in. stainless-steel tubes directly above the STM (Fig. 4). The tubes are welded to a liquid delivery feedthrough mounted on a port aligner and a small bellows that are used for positioning the heater in close proximity to the rear of the sample in the STM. The aligner controls angular displacement of the feedthrough flange and the bellows allows for up to 1 in. of travel along the vertical centerline of the STM. Typically, the filament would be placed within 0.1 in. of the rear of the sample. With this design, there is no contact between the heater and the sample thus ensuring that the mechanical stability of the sample, i.e., the crucial tunneling junction for STM operation, is not compromised. Delivery of power to a heater mounted directly on the STM would involve lower gauge wires or braid that would need to be pinned appropriately as well as have the flexibility to move with the sample holder. The only additional connections made at the microscope are 0.003 in. diameter wires inserted in fiberglass insulation for a type K thermocouple that measures the temperature of the sample plate. The sample plate temperature and the heater power are calibrated to the temperature of the sample in the STM with a separate thermocouple attached to the sample that is removed before baking.

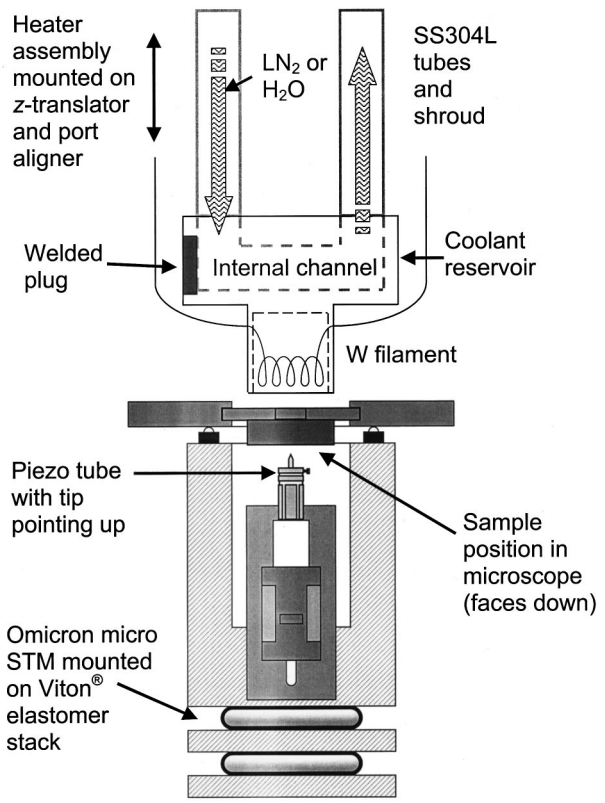


FIG. 4. Schematic drawing of the proximity heater and its relative position with respect to the Omicron Micro-STM.

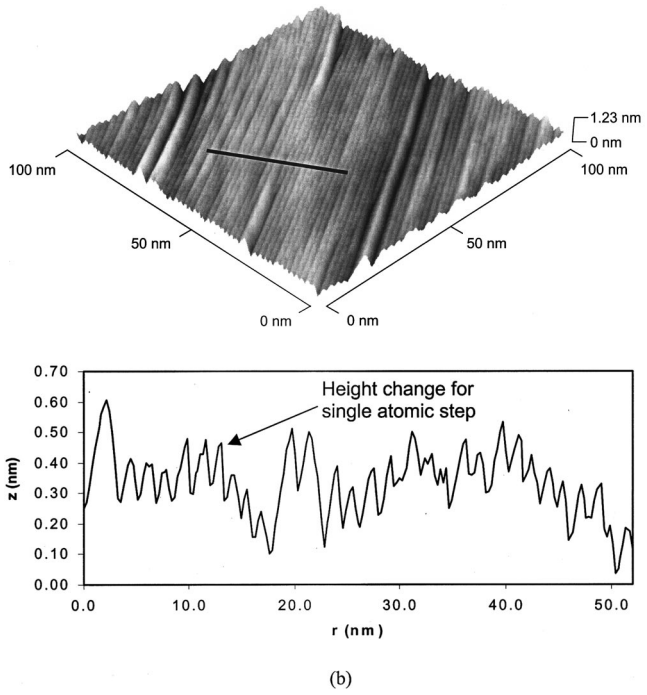
The second key aspect of the design was to have control over the distribution of heat by surrounding the filament in a cold environment. The cold shroud helps manage outgassing from the filament to maintain vacuum cleanliness as well as minimizes the heat load on nearby components. The shroud was machined from one solid piece of 304 low carbon stainless steel comprised of two concentric shapes. A cylindrical snoot with a wall thickness of 0.050 in. and a diameter of 0.500 in. houses the filament and it is attached to a 0.500 in. thick, 1 in. diameter disk shaped coolant reservoir. The snoot has two holes for holding ceramic spacers that support the tungsten filament on either side of the snoot and allow for incoming wires to deliver power to the filament. The filament is made from 0.012 in. diameter polycrystalline tungsten wire and is shaped in a coil to maximize the heating surface area. Water circulates through the coolant reservoir via the stainless-steel support tubes and an internal channel running along the diameter of the coolant disk. The internal channel was made by machining a blind hole through one side of the disk and then welding a thin stainless-steel plug into the opening. The two holes coming from the top of the disk to which the stainless-steel tubes are welded are connected by this channel.

III. OPERATION

Thermal stability plays a vital role in ensuring a stable tunneling junction. Generally, STM experiments are performed isothermally or with only minor temperature variations in order to avoid tunneling instability created by thermal expansions and contractions of both the sample and the scanning piezos. In many designs, the microscope is held at one temperature and the sample at another so that the STM maintains maximum thermal isolation.[5] In our design, we allow the microscope to equilibrate to its new, slightly warmer temperature following a sample temperature change, before imaging. This is done by placing a dummy sample in the STM and raising its temperature to the temperature at which an experiment will be performed. The dummy sample assembly is composed of the same materials and approximately the dimensions in order to simulate the real sample assembly used for experiments. Typically, the STM reaches thermal equilibrium within one hour as evidenced by no changes in heater power. After the real sample has been prepared for an experiment by sputtering and annealing (1100 K) and has cooled to approximately the temperature at which the STM experiment will be operated at, the heater is turned off and moved away from the STM sample holder. The dummy sample is removed and placed in a parking station located on the lower level of the chamber and the real sample is immediately placed into the STM. The heater is then repositioned directly above the real sample and the heater power is turned to the setting used during preheating. Imaging can usually begin within ten to fifteen minutes of the transfer of the sample to the STM. Experiments were customarily run using constant power to the heater as opposed to continual temperature control by feedback loop so as to avoid field effects on the piezos of the scanner.

Once the sample is at the experimental temperature range in the STM, the time for reequilibration due to small changes in temperature is relatively short. The STM remains stable as long as its operating components, i.e., piezos, preamplifier circuitry and microscope housing, remain in thermal equilibrium. Deviations in the sample temperature can occur and imaging can continue provided the small changes in sample temperature translate into even smaller changes within the STM. The magnitude of the thermal variations that a particular STM can tolerate will depend on the specific orientation of the different STM components with respect to the sample and the heater. In our setup, the sample temperature can slowly rise or fall (~ 1 K/min) with no effect on imaging. For more abrupt changes in temperature, faster cooling or heating rates can be used with the microscope out of tunneling feedback. The STM then needs only a few minutes to re-equilibrate and re-enter feedback provided the new temperature reached is approximately ± 20 K with respect to the preheat temperature. This flexibility allows for temperature dependent processes to be imaged such as crossing of overlayer structure phase boundaries and order-disorder transitions.

Figure 5 shows a constant current image taken of a Ni(977) surface at 550 K. The terraces and monatomic risers that make up this stepped surface are clearly resolved. The drift at elevated temperatures is approximately 50 Å /image (scan rate: 25 s/image) in both x and y directions within the first hour of having transferred the sample. While this drift rate may seem large, no attempts have yet been made to minimize it. Drift or change in imaging area can be lessened by invoking sample holding materials with low thermal ex-



(b)

FIG. 5. (a) Constant current image taken of Ni(977) at 550 K with 1 nA tunneling current and 100 mV bias to the sample. (b) Topography profile taken along the line marked in the STM image. Atomically resolved single and multiple steps are clearly resolved.

pansion coefficients and inserting judiciously placed thermal shields for the scanning piezos. The current work with this STM and heater is aimed at imaging surface reconstructions that occur on Ni(977).[12] The sequential images taken to capture the morphological transformations are large enough that the existing drift is easily managed by user repositioning. We are now able to carry out real-time imaging of the changes in the step configuration on a local scale that will provide us with detailed mechanistic steps to complement an existing macroscopic kinetics database (i.e., derived from reciprocal space He diffraction measurements).[13]

This heater was an inexpensive and simple addition to our room temperature microscope, and it has enabled us to further explore experimental systems that had previously only been studied using reciprocal space, scattering methods. The only way to use a room temperature microscope to study elevated temperature surface phenomena would be to carefully quench the surface back to room temperature after performing each experiment at elevated temperature. The great disadvantage here is the inability to conduct real-time imaging studies of microscopic surface dynamics processes. With our technique we are now able to access metallic surface morphology on a local scale at elevated temperature in real time.

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