An ultrahigh vacuum fast-scanning and variable temperature scanning tunneling microscope for large scale imaging

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We describe the design and performance of a fast-scanning, variable temperature scanning tunneling microscope (STM) operating from 80 to 700 K in ultrahigh vacuum (UHV), which routinely achieves large scale atomically resolved imaging of compact metallic surfaces. An efficient in-vacuum vibration isolation and cryogenic system allows for no external vibration isolation of the UHV chamber. The design of the sample holder and STM head permits imaging of the same nanometer-size area of the sample before and after sample preparation outside the STM base. Refractory metal samples are frequently annealed up to 2000 K and their cooldown time from room temperature to 80 K is 15 min. The vertical resolution of the instrument was found to be about 2 pm at room temperature. The coarse motor design allows both translation and rotation of the scanner tube. The total scanning area is about 8×8 μm^2. The sample temperature can be adjusted by a few tens of degrees while scanning over the same sample area. © *2007 American Institute of Physics.* [DOI: 10.1063/1.2789655]

I. INTRODUCTION

Scanning tunneling microscope[1] (STM) has proven to be a powerful tool for real space investigations of surfaces and surface hosted phenomena. Its high spatial resolution makes it an important technique for studying atomic configurations of surfaces and adsorbate molecular aggregates with picometer resolution as well as for studies of the local electronic properties. Dynamical processes such as nucleation and growth, self-assembly, surface diffusion, and chemical reactions can also be investigated. Both static and dynamical studies can hugely benefit from having a system able to operate in a large temperature range.

Our primary motivation in designing and building a variable temperature STM came from the need of having a versatile system aimed at large scale atomically resolved static and fast dynamical studies of strained metallic interfaces and self-assembly processes onto them.

II. INSTRUMENT DESIGN

Designing a STM able to achieve subangstrom resolutions in a large temperature range while having low thermal drifts and integrating it into a larger surface science UHV chamber are a challenging task. The subangstrom resolution requires a careful design of the vibration isolation system where the usual approach is a two stage damping for high and low frequencies.[2] Also, maintaining the ability to probe the same sample area after a large temperature change (few tens of kelvin) imposes hard restrictions on the thermally associated drifts. Other desired features are the ability to change sample temperature at a fast rate, the possibility of finding the same surface area after sample preparation, and also the design needed to accommodate a larger UHV chamber for surface science studies. To address all these restrictions, an in-vacuum damping-cooling system with a large bath cryostat[3] has been chosen. It has the advantage of being vibration-free and also, due to its large mass, it proved to be quite impervious to external vibrations, which makes it well suited for a chamber having no external vibration isolation. Another design direction was to build a very rigid STM head with low thermal expansion, axial symmetry, and a self aligning sample-holder/STM-head system, which allows for repeated investigations of the same sample area even after various sample preparation stages. The STM head is also thermally isolated from the sample holder, thus offering a large temperature range of the sample while operating the STM and a temperature independent resolution and range of the instrument.

A. Vibration isolation and cooling system

A three-dimensional on-scale rendering of the vibration isolation with the cold bath ensemble is presented in Fig. 1. It consists of a large Al-6061 cylinder block (~7 kg), which

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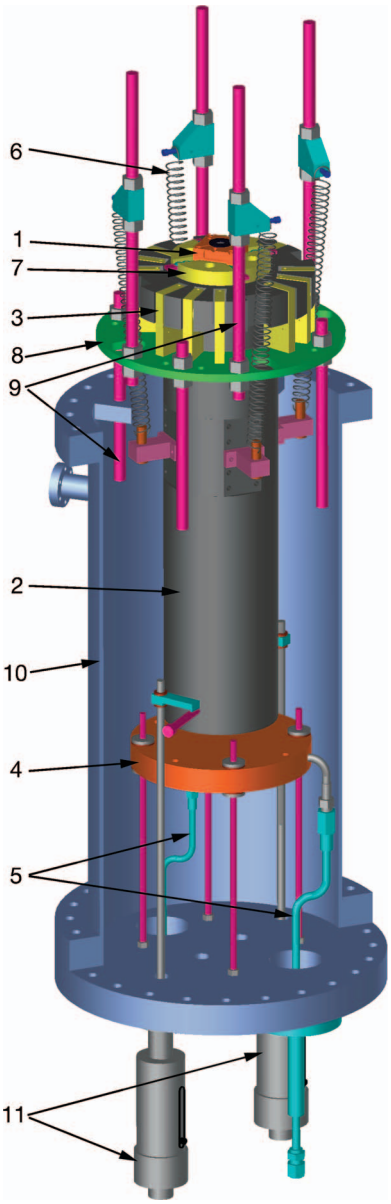

FIG. 1. (Color) Flange-on STM cryogenics and vibration isolation assembly: sample holder (1), Al cold reservoir (2), Sm-Co magnet assembly (3), cold finger (4), coolant feedthrough (5), springs (6), sample-holder Cu base (7), St-St magnet ring holder (8), threaded rods (9), UHV chamber (10), and linear motion manipulators (11).

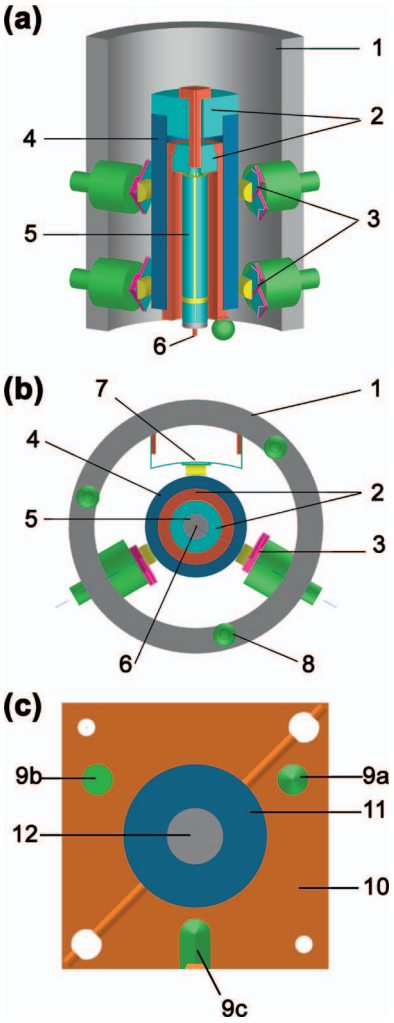

FIG. 2. (Color) STM cross section view with the spring removed for clarity (a), bottom view (b), and sample holder (c): STM Invar body (1), STM tube holder assembly (2), coarse motor piezostacks (3), sapphire tube (4), four-quadrant scanner tube (5), STM tip (6), spring (7), glass balls (8), self-alignment grooves for the STM glass balls with conical (9a), cylindrical (9b), and edge profile (9c), OHFC copper body (10), Ta washer (11), and sample crystal (12).

rests on four spring-steel springs. The elastic constant of the springs has been chosen such that the resonant frequency of this ensemble is close to 1.5 Hz. An eddy current damping system consisting of 12 Sm-Co magnets which are protruding between Al fins machined at the top side of the Al block are distributed in a circular fashion. An oxygen-free high conductivity (OFHC) copper disk carrying the electrical contacts for the filament and thermocouple acts as a support and thermal path for the sample holder. A polished sapphire disk[4] mounted between the top part of the Al block and the copper disk electrically isolates the sample from the large bath cryostat while providing good thermal conduction at low temperatures. The sample is mounted inside the OFHC copper sample holder, which contains a *C*-type thermocouple required for high temperature annealing needed to prepare refractory metal surfaces such as Ru(0001) and a filament. The heating of the sample could be done both by electron bombardment for high temperatures, larger than 2000 K, or radiatively when moderate temperatures are required. In order to reliably measure the temperature of the sample in the range between 70 and 2000 K, a full *C*-type thermocouple line is used. A second *K*-type thermocouple is mounted on the copper disk supporting the sample holder for the purpose of individual *C*-type thermocouple calibration in the cryogenic range while at thermal equilibrium.

The design of the sample holder which contains a W filament and a *C*-type thermocouple allows for cooling and heating of the sample in the STM scanning position as well as in the manipulator, thus preparation of the sample and investigation with other tools being possible under controlled conditions in the whole temperature range. The sample holder is held in place in the STM scanning position by a μ-metal disk bolted to the underside and a magnet mounted in the sample-holder Cu base (Fig. 1), which also carries the

electrical contacts to the filament and the *C*-type thermocouple mounted in the sample holder.

The cooling of the assembly is done by pulling down the bath cryostat in contact with the cold finger made out of OFHC copper which is cooled with liquid nitrogen. The liquid nitrogen flows through a zigzag channel machined in the copper body of the cold finger in order to maximize heat transfer and streamline the flow. In order to thermally isolate the bath cryostat, various Vespel® (Ref. 5) and Al_2O_3 breakers have been placed between the mounting points of the springs and the chamber in this way slowing down the warm-up speed to about 3 K/h around 100 K. While the cooldown of the bath cryostat from room temperature to 90 K is about 6 h when using liquid nitrogen as coolant, the sample-holder cooldown time from room temperature to 90 K is about 10 min.

When measurements are performed, the bath cryostat is released and is supported only by the springs, thus serving as a vibration-free cold reservoir for the sample holder, with the STM head sitting on the top of the sample holder. Any desirable sample temperature above the bath cryostat's temperature can be reached and maintained by radiatively heating the sample. Such a large bath cryostat needs a supplemental way of heating if fast warm-up speeds are needed. For this purpose, a set of four BZY93V75 Zener diodes have been mounted along the bottom circumference of the bath cryostat with other four diodes on the circumference of the cold finger.

B. STM head

A compact, rigid, axially symmetrical STM-head construction is desirable to achieve low noise and low thermal drifts. Figure 2 shows (a) a side cross section and (b) a bottom view of the STM head. The main body is machined out of gold-plated Invar,[6] which has a cylindrical shape in order to minimize lateral thermal drifts. The STM body houses the STM's four quadrant piezoscanner tube mounted axially in a sapphire cylinder[4] with a 5 μm polished outer surface. Various lengths of the scanner tube can be accommodated with a proper choice of the STM tube holder length. We are currently using EBL-1 piezoceramic with Ni electrodes of 12 mm length in order to achieve a large lateral scan range of 8×8 μm^2.[7] The coarse movement of the STM tip is a stick and slip motion[8] of the whole sapphire cylinder-scanner tube assembly done with the four coarse motors mounted in pairs of two at 120° around the vertical axis. The third 120° symmetrical position is taken by the stainless-steel (St-St) spring assembly [Fig. 2(b)] that has the role of pressing the sapphire tube against the four coarse motor pads. The course motors consist of a sandwich of two EBL-1 with Ni electrodes piezoceramic plates that are glued with conductive and low outgassing epoxy[9] in a right angle geometry of the shear directions. The side of the piezostack facing the sapphire cylinder has a semicylindrical Al_2O_3 pad glued on it in a right angle orientation with respect to the scanner axis such that it minimizes the contact area between the pad and the sapphire tube.

The four piezostacks are wired in a parallel configuration to obtain synchronous operation. For a given shear geometry of the piezostacks, the shear directions of the piezoceramic plates are composed vectorial to give up-down or rotational motion of the scanner tube assembly[10,11] while applying the sawtooth high voltage signals.[8] The STM head rests on the sample holder via three symmetrically bottom placed glass balls in order to decouple thermally and electrically the STM from the sample holder. When the sample holder is kept at 100 K, the temperature of the STM-head body thermalizes at about 0 °C. The coarse approach speed can be adjusted by both sawtooth signal amplitude and frequency in a wide range from fractions of mm/min to a few mm/min usually yielding about 3 min total STM tip approach time.

In order to be able to reach the same nanometer-size scanning area on the sample, (1) the position of the STM head with respect to the sample holder has to be fixed and (2) the azimuthal angle of the scanner tube assembly with respect to the STM body has to be controlled. The glass balls on the bottom of the STM fit in a self-aligning system of grooves [Fig. 2(c)] machined on the top of the sample holder fixing the STM-head/sample-holder position. The rotational degree of freedom of the scanner tube assembly with respect to the STM body is controlled via a goniometer mounted in a window machined into the STM body that monitors the position of a vertical thin mark on the outer surface of the sapphire cylinder. In terms of linear displacements of the tip with respect to the sample, the goniometer gives a precision of about 1 μm, which is a few times smaller than the total scanning area of 8×8 μm^2 for the EBL-1 scanner tube where finer tuning can be achieved by looking at recognizable features on the sample.

Access to the sample is done by raising the STM head in a vertical translation stage which also serves as a path for the STM wiring. In-vacuum wiring of the STM is done with braided pairs of 0.1 mm Kapton-insulated copper wires in order to minimize mechanical coupling to the chamber.

C. STM tip preparation

We use polycrystalline high purity tungsten etched tips.[12] After air exposure, each tip has to be conditioned in UHV in order to achieve clean, sharp, and stable tips needed for atomically resolved large scale imaging or for fast scanning. The procedure we use consists of initial Ar^+ sputtering under UHV condition, which generally yields clean and stable but not very sharp tips, followed by self-sputtering in field emission, which was found to systematically yield sharp and stable tips. Due to the overall column-type arrangement chosen for the STM head/sample holder/cooling assembly, axial sputtering of the STM tip with regular ion guns is not practical. We have therefore chosen to design a small ion source mounted internally in a specially designed sample holder, which yields a well collimated ion beam with energies up to 2 keV. During tip sputtering the STM head sits on the ion source, the grooves in the body providing a way to align the STM tip precisely in the ion beam. We found that a 500 eV Ar^+ beam at a current density of 20–30 nA/mm^2 yields consistently clean polycrystalline tungsten STM tips after a few minutes of sputtering. The second part of the conditioning method involves tip self-sputtering in field emission conditions. A good recipe is to apply up to 700 V between the tip

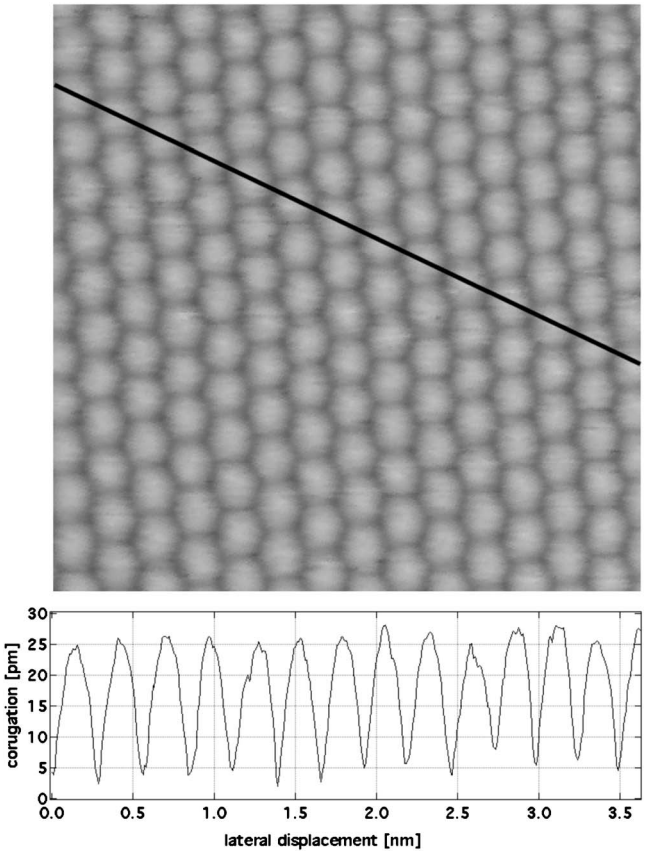

FIG. 3. Constant current image of Ru(0001) at 295 K (−10 mV sample bias, 6.6 nA tunneling current, and 33 Å scan range) and the line scan profile of the line marked on the image showing the apparent corrugation.

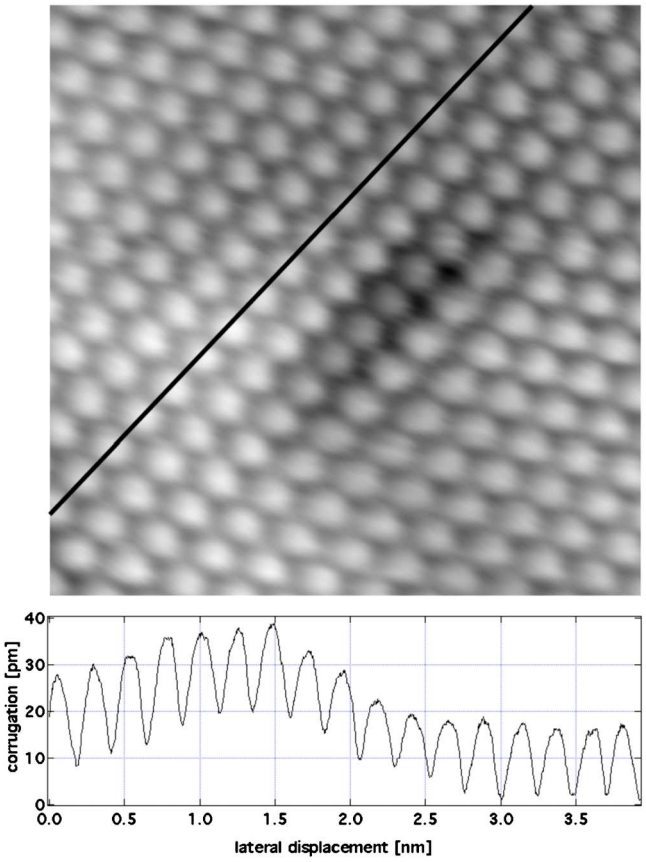

FIG. 4. Constant current image of the short herringbone reconstruction of one atomic layer Ag on Ru(0001) at 110 K (−30 mV sample bias, 6.7 nA tunneling current, and 33 Å scan range) and the line scan profile of the line marked on the image showing the apparent corrugation. The long range vertical modulation of the Ag film is due to the strain relaxation of the misfit dislocation network.

and a polycrystalline Ta foil while the distance between them being adjusted such that a field emission current of 150–200 nA is achieved. Short sputtering of 1–2 min at a partial pressure of 1×10^{-7} Torr Ar is then sufficient to sharpen the tip while maintaining it clean. A STM tip is usually considered ready for STM experiments when stable, few nanoampere field emission currents are obtained at bias voltages of about 100 V.

D. The UHV system

The sample-holder rests on the cold reservoir, which is mounted by the way of the vibration isolation stage in the UHV chamber. The chamber is also equipped with a linear motion manipulator, which allows one to move the sample to different positions in front of various instruments for preparation and characterization, ion gun, gas doser, various metal evaporators, low energy electron diffraction, Auger electron spectroscopy, and scanning tunneling microscope position. The chamber also has a load-lock mechanism for sample exchange and precleaning before introduction in the UHV chamber and a garage stage. The chamber is mounted on a rigid rectangular steel frame, which rests directly on the floor via 1 in. rubber dampers. Due to the excellent damping performance of the in-vacuum damping stage, there is no need for air-spring legs on the chamber. The UHV system is equipped with a 480 l/s ion pump and a 400 l/s N_2 turbopump. Even if the STM is able to achieve atomic resolution with the turbopump running, for best resolution during STM operation, the turbopump can be turned off after the gate valve is closed and the system is being pumped only with the ion/titanium sublimation pump (TSP). The roughing pumps have been placed in a nearby room in order to minimize acoustic noise.

III. EXPERIMENTAL RESULTS

The instrument performance was observed on various compact metallic surfaces and atomically flat thin films grown onto them. Initial measurements were done on a single crystal (111) surface of Au, followed by extensive variable temperature studies on the (0001) surface of Ru and strained monolayer thick Ag films grown on Ru(0001).[13] Atomically resolved terraces and surface atomic steps of Ru(0001) have been used for instrument calibration and testing of the noise level. The Au(111) and one atomic layer thick Ag films on Ru(0001) have been used to test the ability of large scale atomically resolved imaging and fast STM imaging.[13]

The surfaces of compact metallic interfaces were used as test systems for the STM's performance. In Fig. 3 we show a high resolution constant current image of Ru(0001) at 295 K and a line scan profile of the line marked in the image. The vertical resolution is about 2 pm peak to peak. Figure 4

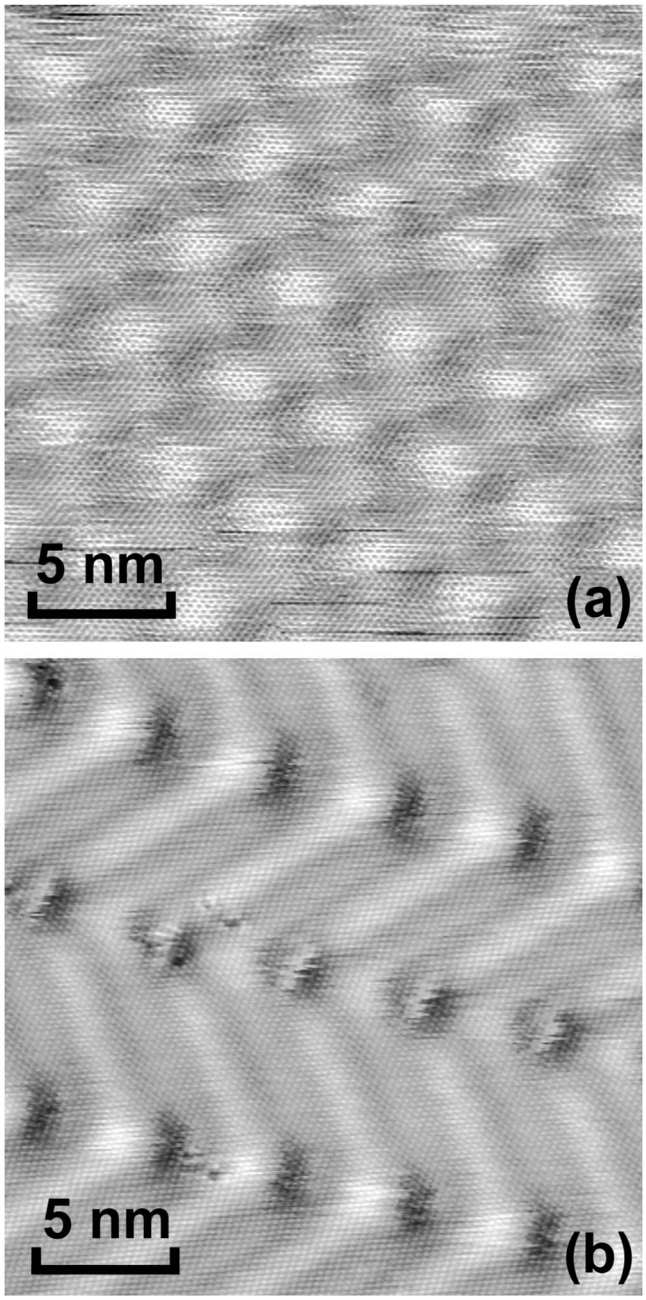

FIG. 5. Constant current image of one atomic layer thick Ag film on Ru(0001): (a) short herringbone reconstruction at 330 K (−20 mV sample bias, 4.6 nA, and the overall Ag coverage is lower than 1 ML normalized to Ru(0001) substrate); (b) long herringbone reconstruction at 280 K [−170 mV sample bias, 19.7 nA, and the overall Ag coverage is larger than 1 ML normalized to the Ru(0001) substrate].

shows a high resolution constant current image of one atomic layer thick Ag film grown onto Ru(0001) at 110 K. The line profile shows a vertical resolution lower than 2 pm peak to peak. Such values of the vertical resolution of the instrument are consistent with the electrical noise level of the piezodrive's high voltage amplifiers[14] for the sensitivity of the scanner tube used at room temperature. Consistently, this noise level is attainable on all other measured surfaces at low and room temperatures since the STM-head temperature is relatively independent of the sample temperature.

Strained metallic interfaces can form misfit dislocation networks with unit cell sizes in the range of tens to hundreds of angstroms.[15,16] Detailed analysis of such structures requires one to image with atomic resolution on the scale of the reconstruction. More important, the analysis of networks of misfit dislocation networks needs even larger scale imaging with atomic resolution. One of the goals of this instrument is to achieve such performance. We were able to investigate the misfit dislocation network of Au(111), the short herringbone (SHB) reconstruction and large herringbone reconstruction (LHB) of one atomic thick layer of Ag on Ru(0001),[17] and 2 ML (monolayer) Ag/Ru(0001) (Ref. 18) on the scale of few unit cell sizes. Figure 5(a) shows the SHB network of Ag/Ru(0001) at 330 K and Fig. 5(b) shows the LHB network at 280 K.

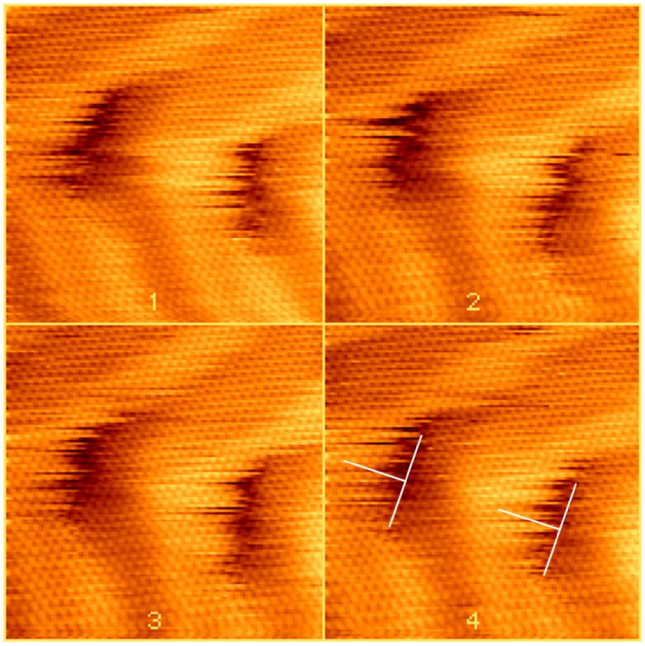

FIG. 6. Consecutive constant current images of one pair of threading dislocation cores—which are marked with white T's on the last image—of the long herringbone reconstruction of one atomic layer thick Ag film on Ru(0001) at 280 K (−170 mV sample bias, 19.6 nA). Scanning time for each image is 3 s.

The dynamical performance of this STM design is exemplified by the images in Fig. 6, which follow the time dependence of the positions of two threading dislocation cores like the ones seen in Fig. 5(b). Each image is about 90×90 Å^2 and was acquired in about 3 s. The Ag atom diffusion results in a change in the relative position of the threading dislocation cores. This vibration of the threading dislocation cores is clearly resolved by STM at the darker, linear region, which show changing positions from one scan line to the next.

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