AIP | Review of Scientific Instruments

Development of an ultrahigh vacuum scanning tunneling microscope cooled by superfluid 4 He

Y. Kondo, E. T. Foley, T. Amakusa, N. Shibata, S. Chiba, M. Iwatsuki, and H. Tokumoto

Citation: Review of Scientific Instruments **72**, 2977 (2001); doi: 10.1063/1.1379959
View online: http://dx.doi.org/10.1063/1.1379959
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/72/7?ver=pdfcov
Published by the AIP Publishing

Development of an ultrahigh vacuum scanning tunneling microscope cooled by superfluid ^{4}He

Y. Kondo[a] and E. T. Foley
Joint Research Center for Atom Technology (JRCAT), Angstrom Technology Partnership, AIST Central 4, Higashi 1-1-1, Tsukuba, Ibaraki 305-0046, Japan

T. Amakusa, N. Shibata, S. Chiba, and M. Iwatsuki
JEOL Limited, 1-2 Musashino 3-chome, Akishima, Tokyo 196-8558, Japan

H. Tokumoto
JRCAT, National Institute of Advanced Industrial Science and Technology (AIST), AIST Central 4, Higashi 1-1-1, Tsukuba, Ibaraki 305-8562, Japan

(Received 30 January 2001; accepted for publication 18 April 2001)

We have developed an ultrahigh vacuum (UHV) scanning tunneling microscope (STM) cooled by superfluid ^{4}He. This microscope is integrated with a solenoid and split-pair superconducting magnet. The STM can be operated at $300>T>4$ K in a rotatable magnetic field of up to 8 T perpendicular to, and 1.5 T parallel to the sample surface. Moreover, tips and samples may be changed without venting the UHV system. The performance of the STM was confirmed by obtaining atomic resolution images of Si reconstructed surfaces at low temperatures. The STM performance was unaffected by the application of high magnetic fields. [DOI: 10.1063/1.1379959]

I. INTRODUCTION

Temperature and magnetic field are important parameters to be controlled when materials are investigated. A low-temperature environment is necessary in order to reduce thermal vibration and to perform precise measurements. It is also essential because interesting phenomena occur at low temperatures, for example, superconducting transitions, Kondo effects, and various magnetic phase transitions. In order to study these phenomena with atomic scale resolution,[1–3] we required an ultrahigh vacuum (UHV)-compatible low-temperature scanning tunneling microscope (STM) capable of operating in a high magnetic field. Although many low-temperature STMs have been reported, few systems[4] satisfied these requirements. Some of these are UHV compatible, but may not be operated in a high magnetic field. Others are combined with a magnet, but are exposed to an exchange gas or cooling liquid. Therefore, we have developed a new low-temperature STM.

II. CONSTRUCTION

The system described here is based on a standard UHV-STM system consisting of a sample and tip treatment chamber and a main chamber for STM investigation. Samples and tips are loaded into the treatment chamber through a loadlock pumped by a turbomolecular pump so that tips and samples may be exchanged without venting the UHV system. The treatment chamber and loadlock are the standard commercial design,[5] while the main chamber and STM unit are new. Both chambers are equipped with a sputter ion pump and a titanium sublimation pump, and the base pressure of both chambers is below 1.5×10^{-8} Pa. For low-temperature STM investigation the main chamber has been modified to include a cryostat and vertical manipulator. The vertical manipulator is used to shuttle an STM unit between a room-temperature stage (RTS) and a low-temperature chamber attached to the bottom of the main chamber. The entire system is mounted on a pneumatic vibrations isolation table, which is resting on a massive concrete block weighing 30 tons. See the schematic drawing of Fig. 1(a).

A. Treatment chamber

Samples and tips are prepared in a treatment chamber equipped with a storage carousel for four samples and three tips. Samples may be resistively or indirectly heated, and tips may be indirectly heated or cleaned by electron bombardment in the carousel. The treatment chamber also includes a differentially pumped ion source for sample and tip sputtering, and a triple source evaporator. Samples and tips are prepared in the treatment chamber, and then transferred to the main chamber for STM investigation.

B. Main chamber and cryostat

Inside the main chamber there is a room-temperature stage where STM may be performed at room temperature, as shown in Fig. 1(b). The RTS is rotated by 90° to allow for sample and tip exchange, as seen in Fig. 1(c). A vertical manipulator mounted on top of the chamber [Fig. 1(a)] is used to transfer the STM unit from the RTS to the low-temperature stage (LTS). The LTS is located at the bottom of

[a]Present address: Dept. of Phys., Kinki Univ., Higashi-Osaka 577-8502, Japan; electronic mail: yasushi@mailaps.org

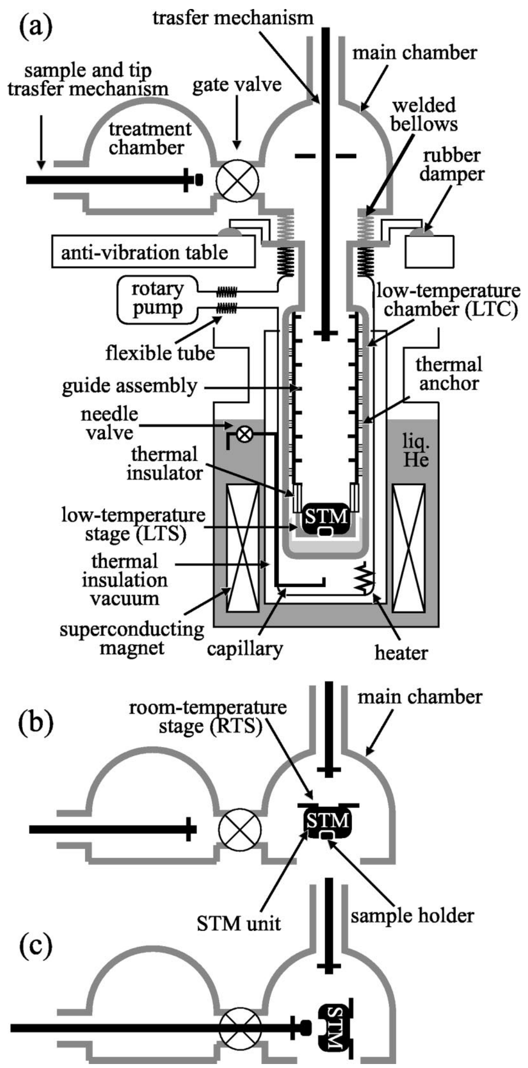

FIG. 1. Conceptual drawing of the low-temperature UHV-STM system. (a) A gas-flow type cryostat is equipped with a solenoid and a split-pair superconducting magnet. The dewar is also equipped with a needle valve for controlling ^{4}He flow and a heater. A portion of the UHV chamber called the low-temperature chamber is inserted into the dewar. The LTC is vibrationally isolated from the rest of the system by means of two welded bellows and three rubber dampers. The bottom of the LTC, where the STM unit is fixed for low-temperature operation, sits inside the bore of the superconducting magnets. There is a guide assembly inside the LTC which serves to guide the STM unit up and down the LTC, and also serves as a support structure for the electrical wiring and for the stainless steel gearing used in the *X* and *Z* coarse motion. (b) For operation at room temperature, the STM unit is fixed into a RTS in the main chamber. (c) For tip/sample exchange the RTS is rotated by 90° to allow access to the tip/sample holder at the bottom of the STM unit.

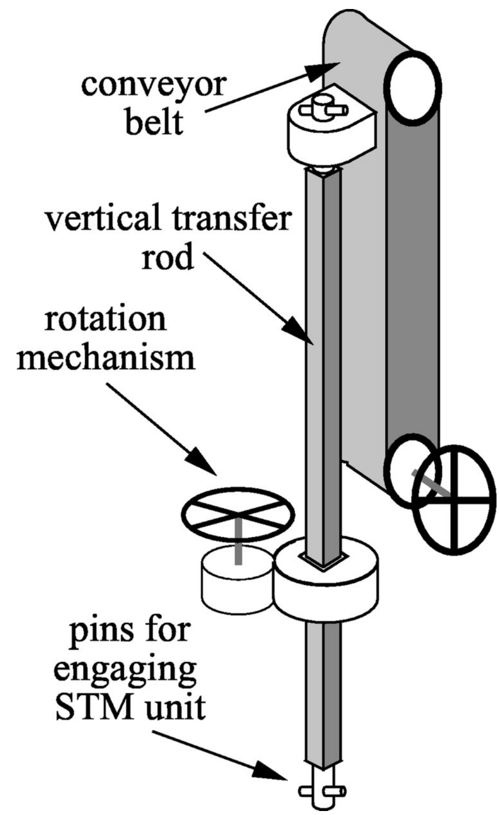

FIG. 2. Vertical manipulator for the STM unit. The end of a vertical rod is fixed to a *conveyor belt*. The rod can rotate in addition to moving vertically.

a long tube attached below the main chamber (the low-temperature chamber, LTC). The vertical manipulator is a long rod fixed to a stainless steel conveyor belt, as shown in Fig. 2. This rod slides through a rotational coupling so that pins on the end of the rod may be rotated to engage the STM unit and fix/release it into/out of both the RTS and LTS. Because the entire vertical manipulator mechanism is contained within the UHV, a welded bellows arrangement, which is fragile and susceptible to leaks, is avoided.

The LTC extends into a cryostat.[6] The LTC is itself surrounded by another double-walled tube called the variable temperature insert (VTI). The VTI is situated inside the bore of the superconducting magnets, and the space between the walls of the VTI serves as a thermal insulation vacuum. The VTI is equipped with a needle valve that is used to transfer cryogen from the Dewar to the space between the VTI and LTC. This space may be pumped to reduce the temperature of the cryogen. The LTC is vibrationally isolated from the rest of the system by means of two welded bellows and three rubber dampers as shown in Fig. 3. In order to ensure the effective isolation of the LTC, the bottom of the LTC must be prevented from making mechanical contact with the interior wall of the VTI. For this purpose the LTC is equipped with three touch sensors at its bottom.

A guide assembly for the STM unit is fitted inside the LTC, as shown in Fig. 1(a). This guide assembly serves to heat sink the STM unit to the bottom of the LTC, and also serves as a heat sink and the support structure for the electrical wiring and for the stainless steel gearing used in the *X* and *Z* coarse motion. In order to reduce the heat flow to the bottom of the guide, the assembly is divided into two parts separated by thermal insulators made of polyimide-based resin.[7] The upper part is a bird cage-like structure (BC) made of stainless steel tubes, and the lower part is a Cu cup (Cu block B) into which the STM unit is seated (see Fig. 3). The tubes of the BC are thermally anchored to the wall of the LTC by ten thermal anchor units (TAUs, three of them are shown in Fig. 4) and five smaller TAUs in the bore of the magnet. There are eight BeCu leaf springs in each TAU. Cu block B is thermally anchored inside the copper base (Cu block A) of the LTC. A heater wound around Cu block B is

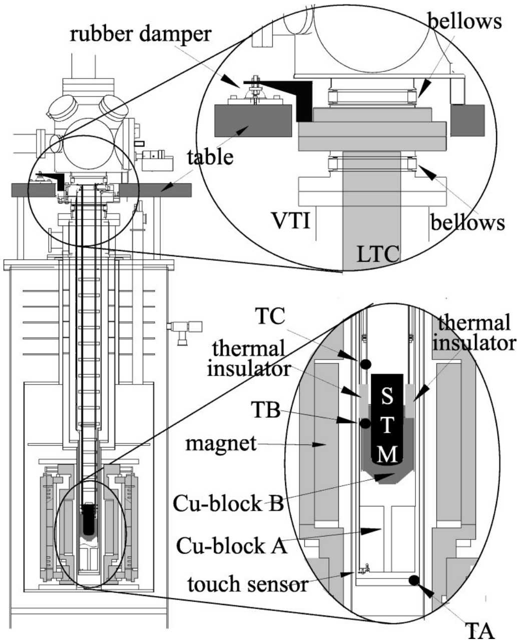

FIG. 3. Cryogenic portion of the system. The vibration isolation of the LTC, and parts surrounding the STM unit at the bottom of the LTC are shown in detail. The vibration isolation of the LTC consists of three rubber dampers and two welded bellows. Three thermometers surround the STM unit at the bottom of the LTC to check heat flow through the guide assembly.

used for variable temperature operation of the STM unit. The LTC is equipped with three thermometers positioned at the inner bottom of the VTI (TA), on Cu block B (TB), and on the BC just above the thermal insulators (TC).[8] TA is used to measure the cryogen temperature between the VTI and LTC.

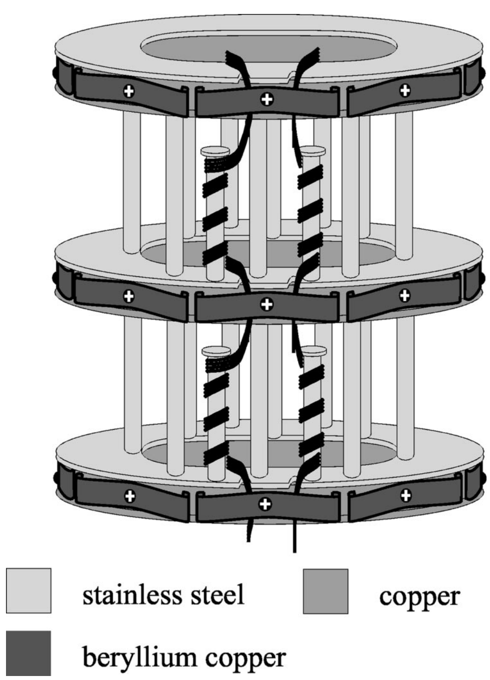

FIG. 4. BC of the guide assembly in the LTC, or thermal anchor units. The BeCu leaf springs bypass the heat from the room temperature portion to the wall of the LTC, otherwise the heat flows into the STM unit at the bottom. There are also posts for anchoring electric cables thermally. In the magnet bore, thermal anchor units are smaller because of the limited space, but the essential structure is the same as shown here.

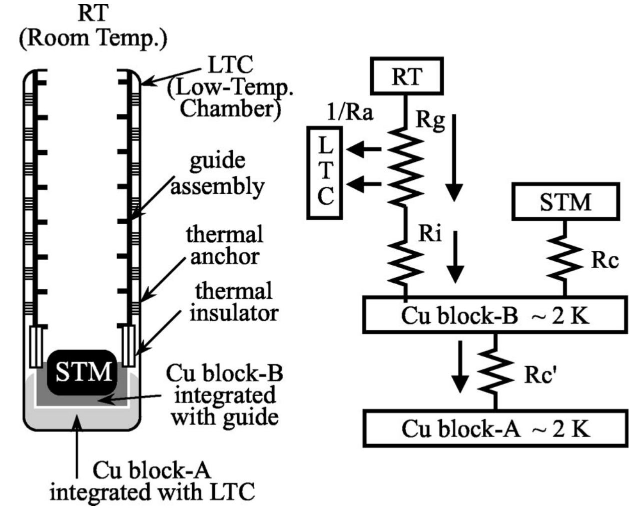

FIG. 5. Schematic diagram of the thermal design is shown on the left side, while the heat flow of this design is shown on the right side. The guide assembly for transferring the STM unit is set inside the LTC, and this conducts undesired heat to the STM unit at the bottom of the LTC. The main objective of this design is to reduce this heat flow. See the text for more details.

In order to check the sample cooling performance of the cryostat a special sample holder is used, where a Si diode sensor is set in place of a sample.

The heat flow through the guide assembly (its thermal resistance is Rg) from the room temperature portion to the bottom is diagrammed in Fig. 5. Most of the heat is bypassed by the TAUs (Fig. 4) and does not reach the bottom. The thermal conductance between the LTC inner wall and the guide assembly is represented by $1/Ra$. Note that about 1 W of heat flow is expected through the stainless steel tubes of the guide assembly without the TAUs. Cu block A is cooled to about 2 K by pumping the liquid He that surrounds the LTC. By using gold foil between Cu blocks A and B, the thermal resistance Rc between them is small, and Cu block B is also cooled to 2 K. Cu block B is fixed to the guide assembly with thermal insulators which significantly increases the total thermal resistance between the RTS and LTS by adding Ri. When the liquid He outside the LTC is superfluid, the measured temperatures at TA, the sample holder, and TC were 2 K, <4 K, and 18 K, respectively. The large temperature difference between TC and the others shows that the thermal insulators work well. The integration of Cu block B with the guide proved essential for ensuring a reliable tip approach, because this allowed for a good fit between the gears of the guide assembly and the STM unit.

The cryostat has a usable volume of 50 l, allowing for operation of the two NbTi superconducting magnets for up to two days without refilling. The magnets produce a magnetic field of up to 8 T perpendicular to, and 1.5 T parallel to the sample surface.[6] The magnets are operated in the persistent mode to avoid electrical noise generated by the power supplies. After switching to the persistent mode, 15 min are required to stabilize the magnetic field and obtain good thermal equilibrium.

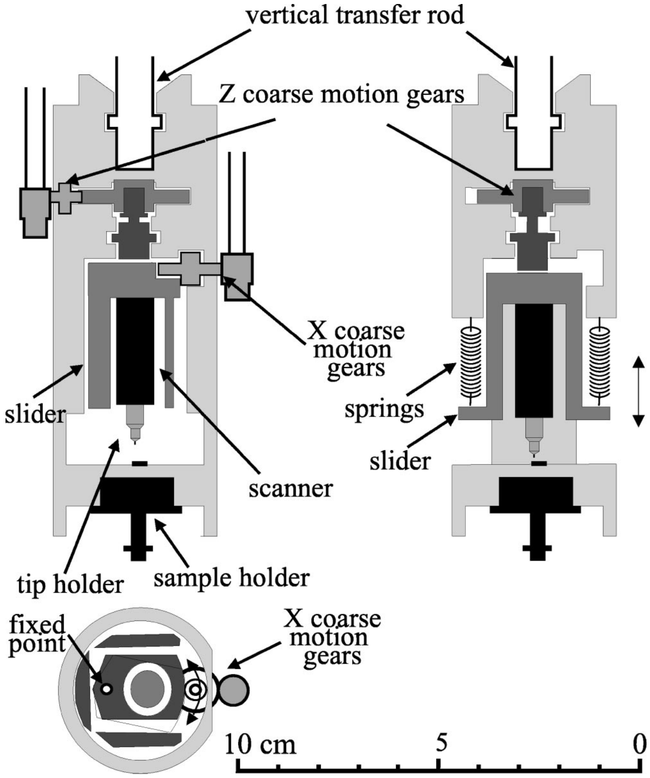

FIG. 6. Schematic view of the STM unit showing the STM scanner, tip holder, sample holder, and internal gearing for *X* and *Z* coarse motion. The figure at the top left shows the STM scanner in the retracted position, while the figure on the top right shows the scanner approaching the sample. The scanner is not fixed to the *Z* course motion gears, but is held against the gears by springs. The figure at the bottom left shows the mechanism for *X* course motion.

C. STM unit

A compact and rigid STM unit developed for this system is shown in Fig. 6. It consists of a sample holder stage, a tube-type scanner,[9] and gearing for both *X* and *Z* coarse motion. The unit is made entirely of nonmagnetic materials (stainless steel, beryllium copper, copper, Teflon, and ceramics) to avoid any influence of high magnetic fields. This unit is designed to use the same sample and tip holders as the commercial UHV-STM system.

Most low-temperature STMs employ piezo motors (e.g., piezo walkers and stick-slip motors) for coarse motion mechanisms because of limited space, difficulty with mechanical access, or problems with vibration and good thermal coupling to the cryogen. However, mechanical systems like the one we selected can offer greater reliability for low-temperature application despite their increased complexity. The coarse motion mechanisms of the STM unit are shown in Fig. 6. The rotational motion of *ex situ* stepper motors is transferred to the STM unit via rotational feedthroughs, several gears, and long rods in the BC. Gearing internal to the STM unit converts this rotational motion to *X* and *Z* coarse translation. The STM unit is fixed into Cu block B using a rotor at the top of the STM unit as shown in Fig. 7. Pins at the end of the vertical manipulator rod are used to turn this rotor and fix/release the STM unit into/out of Cu block B. Once the unit is fixed, the manipulator rod may be removed from the STM unit and raised to the room temperature portion of the main chamber.

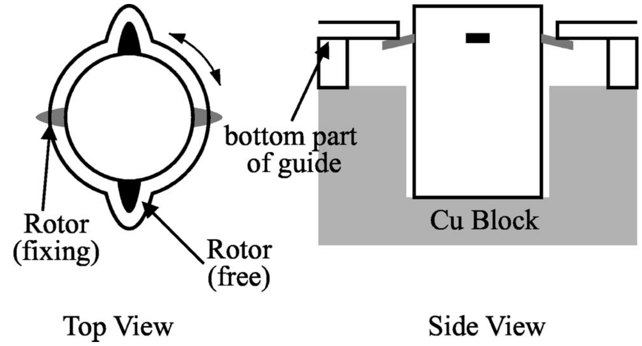

FIG. 7. Mechanism for fixing the STM unit into the bottom of the LTC. A rotor at the top of the STM unit is turned by pins at the end of the vertical manipulator. This rotor rotates from the free position to the fixed position. In the fixed position the bottom of the guide assembly presses against the rotor, and this fixes the STM unit into the bottom of the LTC.

The control electronics for the STM unit is the same standard type used for the commercial STM. STM control and image analysis software is run on a Windows95[10] computer, and is capable of performing conventional STM imaging, manual atom manipulation, as well as standard data processing such as filtering and fast Fourier transform.[5] A commercial tunneling current preamp is used.[11] Typical preamp settings used are 10^{-9} A/V and 0.1 ms time constant.

III. OPERATION

A. Baking

The treatment and the main chambers are equipped with heaters[5] for system bakeout. A ribbon heater is wound around the vertical manipulator casing, and a sheath heater compatible with low vacuum and low temperature is wound around the LTC.[12] The entire UHV system is heated up to about 150 °C during bakeout. Less than 40 W is required to heat the bottom of the LTC to 150 °C. Before bakeout the dewar is filled with liquid N_2 to protect the superconducting magnets from heat damage. The thermal insulation vacuum of the VTI is evacuated to minimize liquid N_2 consumption during bakeout to about 20 l/day. Without evacuating the thermal insulation vacuum the liquid N_2 consumption during bakeout is about 100 l/day.

B. Cooling

Three cooling methods are employed according to the desired temperature, economy, and magnetic field. In normal operation the STM unit shuttles up and down between the RTS of the main chamber and the LTS at the bottom of the LTC. It takes about 30 min to move the STM unit from the LTS to the RTS, to exchange the sample and tip, and to return it to the LTS. The STM unit cools from 300 to 4 K in about 10 h.

1. Without pumping

In this mode of cooling operation, we can employ liquid He or N_2 as cryogen. The Dewar is filled with liquid He

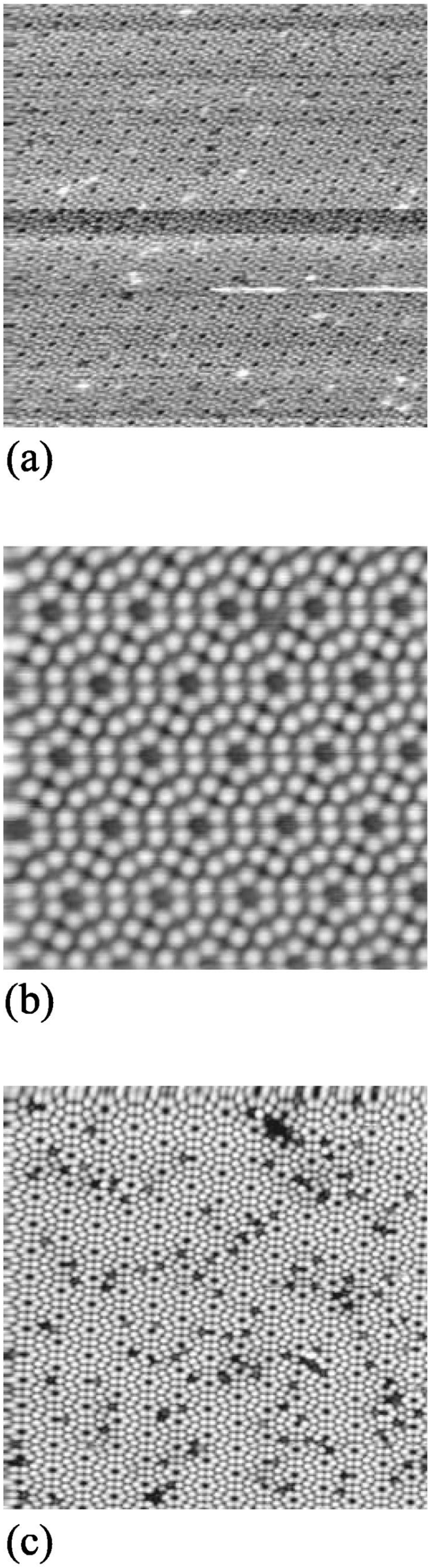

FIG. 8. STM images of Si(111)-7×7 (P-doped, 0.01 Ω cm) reconstructed surfaces. In all images sample bias (V_B) and tunneling current (I_t) are 2.0 V and 0.2 nA, respectively. (a) At 300 K. The scan area (A) is $50\times50\ \mathrm{nm}^2$. (b) At 10 K. $A=14\times14\ \mathrm{nm}^2$. (c) At 100 K and 8 T magnetic field perpendicular to the sample surface. $A=40\times40\ \mathrm{nm}^2$.

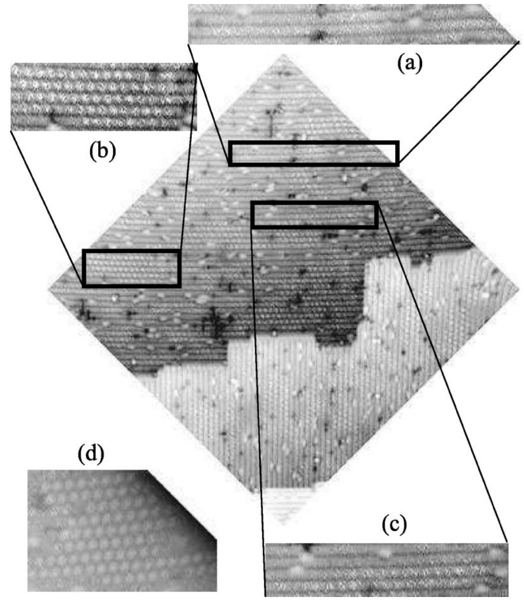

FIG. 9. STM image of adsorbed Xe atoms on Si(001) surface (P-doped, 0.01 Ω cm) at ~80 K. $V_B=2$ V, $I_t=0.05$ nA, and $A=35\times35\ \mathrm{nm}^2$. A one-dimensional gas (a), a two-dimensional solid (b), and the intermediate state between them (c) are visible. When Xe gas dosing is increased, a hard two-dimensional solid (d) can be observed.

(N_2). The thermal insulation vacuum of the VTI is broken by adding 1 (few) cm^3 of He exchange gas at atmospheric pressure. The resulting pressure is estimated to be on the order of 1 (10) Pa. The space between the LTC and the VTI is also filled with He gas of about 10^4 Pa, so that the three dampers work optimally. Note that the force applied to the dampers changes when the pressure of the space between the LTC and the VTI varies. The lowest sample temperature attained is about 7 (80) K, and liquid He (N_2) consumption is about 2 l/h at maximum (less than 10 l/day). The temperature is controlled primarily by changing the amount of exchange gas in the thermal insulation vacuum, but may also be controlled by using the heater wound around Cu block B. The usable temperature range is up to 150 (300) K.

The advantages with liquid He are (A1) the superconducting magnets can be employed, and (A2) the lowest temperature is 7 K instead of 80 K with liquid N_2. The disadvantages are (D1) 16 h of work is necessary for transferring liquid He after precooling with liquid N_2, and (D2) the running cost is much more than with liquid N_2.

2. Liquid He with pumping

When the lowest temperature is required, we need to operate a pump with liquid He. In this mode, the Dewar is filled with liquid He and the thermal insulation vacuum of the VTI is thoroughly evacuated. The volume between the LTC and the VTI is filled with liquid He by opening the needle valve of the VTI. Once filled, the needle valve is closed and a 200 l/s rotary pump pumps the liquid He. This pumping cools the liquid He below the superfluid transition temperature so that the lowest temperature is achieved. The bubbling of the liquid He, which affects STM operation, is also eliminated. When pumping the liquid He, careful adjust-

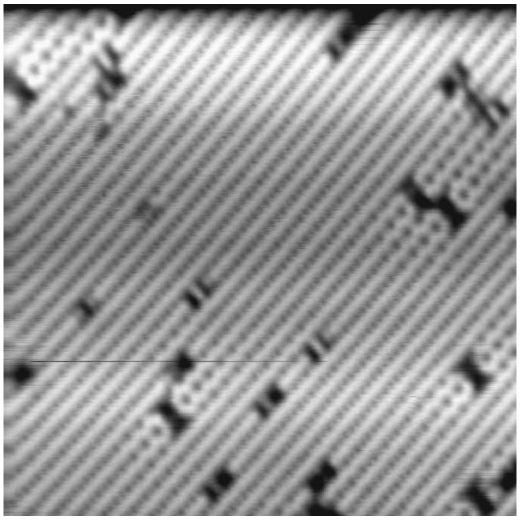

FIG. 10. STM image of Si(001) surface (*P*-doped, 0.01 Ω cm) at 20 K. $V_B = -2.5$ V, $I_t = 0.2$ nA, and $A = 23\times 23\ \mathrm{nm}^2$. Filled states are imaged. Symmetric dimers dominate the surface, but asymmetric (buckled) dimers are visible near defects.

ment of the vibration isolation dampers of the LTC is necessary, since the force applied to these dampers cannot be adjusted by controlling the He pressure in the space between the LTC and the VTI. The temperature of the superfluid ^{4}He is just below 2 K, and the liquid He lasts about 10 h. The lowest sample temperature in this mode is less than 4 K. The sample temperature changes by about 0.5 K during pumping before the liquid He is gone.

The Dewar manufacture[6] has suggested that the cryostat can be operated in the continuous flow mode, where liquid He flows into the space between the LTC and the VTI through the needle valve, as the liquid He is pumped by a rotary pump. However, when operated in this way, needle valve vibration produces too much mechanical noise for STM operation.

IV. STM PERFORMANCE

Si reconstructed surfaces were selected to evaluate the performance of the STM system. Xe adsorption on Si(001) was also investigated.

A. Si(111)

In order to evaluate STM performance, Si(111) surfaces were imaged under various conditions, as shown in Fig. 8. Images were obtained at 300 (a) and at 10 K (b) with no applied magnetic field. The piezo tube response decreases from 48 nm/V at 300 K to 7 nm/V at 10 K. An image was also obtained at 100 K in a magnetic field of 8 T perpendicular to the sample surface (c). STM performance was unaffected in a high magnetic field.

B. Xe atoms adsorbed on Si(001) surface

The adsorption of rare gas atoms has been extensively used as a model system for understanding essential physics such as first-order phase transitions.[13] Before the invention of STM, measurements of bulk properties such as heat capacity were the most important means of investigating these systems. With STM, direct observation of the liquid and solid phases is possible. Since the nucleation of a new phase can be initiated by atomically small irregularities,[14] information obtained on the atomic scale with STM can provide critical insight into the physics of these processes. We have begun investigation of the adsorption of Xe on Si(001). Three phases of adsorbed Xe atoms are visible in Fig. 9.

C. Si(001)

The Si(001) reconstructed surface mostly consists of symmetric dimers at room temperature. In variable-temperature STM experiments it has been observed that the population of asymmetric dimers increases at the expense of symmetric ones at about 100 K.[15,16] Current theories also conclude that asymmetric dimers are more stable than symmetric ones.[17,18] We have also investigated the temperature dependence of the surface reconstruction of Si(001) surfaces. We found that asymmetric dimers dominated the surface at 110 K as previously reported. However, we found by decreasing the temperature still further that asymmetric dimers became less stable, and that symmetric dimers once again dominated the surface at 20 K. Asymmetric dimers were only near defects in Fig. 10 at 20 K. These results indicate that the Si(001) surface at the zero temperature limit is not $c(4\times 2)$ but rather $p(2\times 1)$ symmetric, and suggest a reconsideration of the ground state.[19]

ACKNOWLEDGMENT

This work was supported by NEDO.

[1] J. Li, W. Schneider, R. Berndt, and B. Delley, Phys. Rev. Lett. **30**, 2893 (1998); V. Madhavan, W. Chen, T. Jamneala, M. F. Crommie, and N. S. Wingreen, Nature (London) **280**, 567 (1998).

[2] A. Yazdani, B. A. Jones, C. P. Lutz, M. F. Crommie, and D. M. Eigler, Science **275**, 1767 (1997).

[3] See the article by B. G. Levi, Phys. Today 17 (2000), and references therein.

[4] R. R. Schulz and C. Rossel, Rev. Sci. Instrum. **65**, 1918 (1994); S. H. Tessmer, D. J. van Harlingen, and J. W. Lyding, *ibid.* **65**, 2855 (1994); Chr. Witneven, R. Dombrowski, S. H. Pan, and R. Wiesendanger, *ibid.* **68**, 3806 (1997); O. Pietzsch, A. Kubetzka, D. Haude, M. Bode, and R. Wiesendanger, *ibid.* **71**, 424 (2000).

[5] JSTM-4500XT, JEOL Ltd., http://www.jeol.co.jp

[6] OXFORD Instrument Ltd., http://www.oxinst.com

[7] Produced by TORAY Ltd., http://www.ns.toray.co.jp/e/jigyou/index.html

[8] TA is a ceramic oxynitride sensor, and the others are silicon diode sensors (CX-1050 and DT-470 produced by Lake Shore Cryogenics, Inc., http://www.lakeshore.com).

[9] The scanner is a standard tube type with five electrodes (length=30 mm, diameter=10 mm, and wall thickness=0.5 mm) made of C-82 (Fuji ceramics, Fujinomiya, Japan, 418-0111).

[10] Microsoft Ltd., http://www.microsoft.com

[11] DL INSTRUMENTS, LLC., http://www.dlinstruments.com/

[12] Model 2M-1-300A, Sakaguchi E. H VOC Corp., http://www.jribc.co.jp/smc/sakaguchi/index.html

[13] Papers in *Colloque International du C. N. R. S., Two-dimensional Adsorbed Phases*, J. Phys. (Paris) **C-4**, 10 (1977).

[14] *Nucleation*, edited by A. C. Zettlemoyer (Dekker, New York, 1969).

[15] R. A. Wolkow, Phys. Rev. Lett. **68**, 2636 (1992).

[16] H. Tochihara, T. Amakusa, and M. Iwatsuki, Phys. Rev. B **50**, 12 262 (1994).

[17] K. Inoue, Y. Morikawa, K. Terakura, and M. Nakayama, Phys. Rev. B **49**, 14 774 (1994).
[18] A. Ramstad, G. Brocks, and P. J. Kelly, Phys. Rev. B **51**, 14 504 (1995); J. Fritsch and P. Pavone, Surf. Sci. **344**, 159 (1995), and references therein.
[19] Y. Kondo, T. Amakusa, M. Iwatsuki, and H. Tokumoto, Surf. Sci. **453**, L318 (2000); a similar observation, but with a different conclusion, was reported by Yokoyama and Takayanagi in Phys. Rev. B **61**, R5078 (2000).