Design and operation of a low-temperature scanning tunneling microscope suitable for operation below 1 K

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A scanning tunneling microscope suitable for very low temperatures has been designed, and preliminary testing has been carried out. In order to improve cooling and temperature uniformity the instrument is arranged for operation immersed in the ^{3}He–^{4}He mixture inside the mixing chamber of a small dilution refrigerator. A discussion of the specific problems present in the design of this kind of an instrument is given as well as a description of our design. Special attention is given to the vacuum sealing and vibration-damping solutions required.

1. Introduction

In addition to the well known stability, resonance frequency and vibration sensitivity considerations, special attention must, in many cases, be paid to specific environmental problems. So have scanning tunneling microscopes for operation in high and ultra-high vacuum been discussed extensively in the literature [1], and a number of systems where the tunneling site is placed in a liquid cell have been presented over the years [2]. Another important class of instruments deals with phenomena at extreme temperatures [3,4]. In that case special care must be exercised in at least two ways. Firstly, the thermal expansion or contraction of construction materials used may often shift the tip-to-sample position to a large extent, in severe cases even beyond the stroke of the approach mechanism. Secondly, a proper control of the actual tip and sample temperatures can be very hard to achieve in an instrument as delicate as a tunneling microscope. The two difficulties typically appear in different temperature regimes with thermal expansion being the major problem at higher temperatures, especially above room temperature [4], whereas temperature control must be considered in detail at the lowest tem-

peratures. At temperatures well below the boiling point of nitrogen (77 K) expansion coefficients tend to decrease to practically zero at low temperatures (< 4.2 K) [5]. The only expansion problem remaining in the very-low-temperature case is therefore shift during the cooling process. In that case a thermally compensated design will simplify sample installation and approach.

2. Temperature control

The temperature-control problem should be expected to be much more difficult in a vacuum instrument than in one surrounded by some fluid because thermal conduction through the solid structure is a very uncertain process when many materials and joints are involved. At the lowest temperatures, this problem will be accentuated by the Kapitza resistance phenomenon occurring at all interfaces. Low-temperature scanning tunneling microscopes have been built according to at least three different surrounding standards: vacuum, exchange gas, and direct immersion in a cryogenic liquid. Vacuum instruments have been described by several groups [3,6,7], exchange gas instruments by Renner et al. [8] and by Endo et

al. [9] and immersion systems by, for example, Smith and Binning [10].

A microscope intended for operation well below helium temperature (~ 1 K) has been designed by the Austin group [11]. Their instrument and our first low-temperature instrument (unpublished) are mounted inside the inner insulation vacuum of a dilution refrigerator capable of reaching the 10 mK temperature range. Both instruments rely upon heat conduction through metal from the tip and the sample to the heat sink which, in this case, is the refrigerator mixing chamber.

Cooling below – say – 100 mK must in such a case be regarded as most uncertain due to the difficulties associated with obtaining thermal contact between different solids (Kapitza resistance in a wide sense) [12]. A more reliable, although still not trivial, cooling of the system is achieved by arranging a small chamber around the microscope and by filling the chamber either with an exchange medium or by the coolant itself. For temperatures below 0.3 K, exchange gas cannot be used since the gas pressure of ^{3}He, the lowest boiling point gas, practically vanishes below that temperature. A liquid mixture of ^{3}He and ^{4}He should be chosen at lower temperatures. ^{4}He is an extremely good heat conductor below the λ-point (2.17 K). However, the conductivity decreases rapidly at the lowest temperatures. ^{3}He, on the other hand, shows an increasing conductivity at temperatures below 100 mK [13]. The thermal contact between liquid He and solids is limited by the Kapitza resistance in its strict meaning [14]. Operating the instrument directly in the cooling medium in this case is equivalent to installing the instrument inside the refrigerator mixing chamber, where the medium present already is a ^{3}He–^{4}He mixture.

3. Vibration insulation

The combined problem of cooling and vibration insulation has often been a hard one to solve. Vibration damping stages can be introduced either inside the cryostat next to the microscope frame [8–10] or outside the entire cryostat.
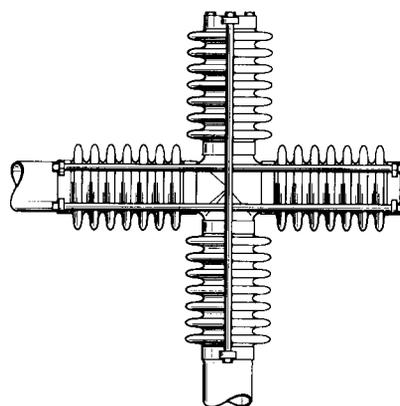

Fig. 1. A cross bellows link for vibration decoupling of vacuum lines.

In the latter case, efficient vibration decoupling must be arranged on all gas and vacuum lines required for the cryostat operation. The two methods can also be adopted simultaneously [6].

In combination with immersion microscopes, outside vibration damping is the only practical solution. An ordinary continous dilution refrigerator requires several gas and vacuum lines (usually two large pumping lines and, say, two smaller ones). All these connections have to be equipped with facilities for absorbing mechanical vibrations as well as acoustical noise from connected pumps. One or more pressure-compensated bellows systems, preferably in a cross arrangement, are usually installed for this purpose (fig. 1). A non-continuous refrigerator with internal charcoal pumps [15] might be an interesting device since it completely eliminates pump noise. Vibrations generated in the cryostat itself should be considered as well. A severe noise source is boiling cryogenic liquids, a super-insulated cryostat requiring no liquid-nitrogen shroud is most preferable despite a slightly larger helium consumption. Noise from boiling helium can be virtually eliminated by pumping the helium bath temperature below the λ-point.

4. The new instrument design

The instrument (fig. 2) is designed for mounting in the mixing chamber. It is built around a
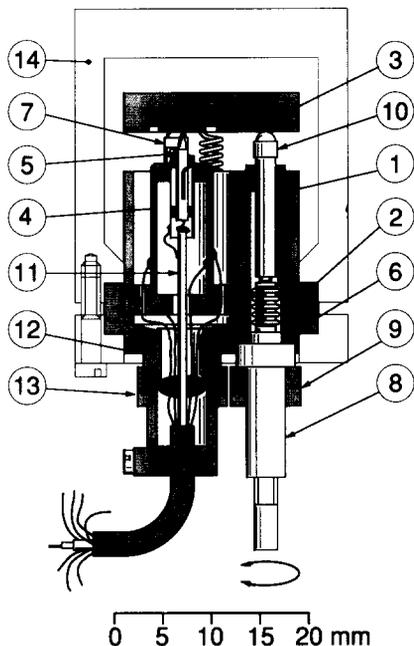
Fig. 2. The new instrument: (1) compensating cylinder, (2) insulating disc, (3) sample holder, (4) scanner tube, (5) tip holder, (6) vacuum flange, (7) pre-adjustable ball support, (8) differential screw assembly with soldered on miniature bellows, (9) clamping flange for (8) (the two bolts are of the drawing plane), (10) push rod extension with fine approach ball support, (11) tunnel current coaxial line, (12) feedthrough sleeve with "stycast" seal, (13) clamping flange for (11) with integral cable clamp, (14) mixing chamber.

tube scanner and a mechanical approach system similar to the one introduced by Drake et al. [2,16]. A vacuum-tight flange with electrical feedthroughs and a miniature bellows link for the fine-approach system is incorporated as an integral part of the instrument.

5. The central part

The tube scanner is fixed inside a solid instrument frame on top of which the sample holder is resting on three adjustable steel ball supports. In order to achieve the best possible thermal compensation the main frame consists of a nickel alloy cylinder that has the same active length as the scanner tube as well as a matching coefficient of thermal expansion. The material chosen is

NILO 36 from Wiggins [17]. In addition to the scanner tube and the compensating cylinder, the mechanical loop is completed by two end plates made out of MACOR machinable glass. The lower plate, called the insulation disc, is firmly clamped in between the compensating cylinder and the brass vacuum flange that acts as a mounting base for the entire instrument. Two long NILO anchor bolts give a more or less temperature-independent compressive force to this joint.

The top plate or sample holder is resting on the three steel ball supports, two of which are adjustable at room temperature only. The third fine-adjustment support is activated by a differential screw system arranged for manipulation from outside the cryostat. The geometry of the entire sample stage is such that the tunneling site will receive one tenth of the movement produced by the differential screw support. Adjusting the two other supports, with a tool like a needle, and with the instrument under an optical microscope, should be performed in order to bring the sample within reachable distance for the fine approach (100 μm). A self-aligning arrangement for the sample holder with one conical hole, one V-groove and one flat area is adopted rather then the more symmetric equivalent with three V-grooves [18]. The reason is that the differential screw support must be regarded as less accurately guided and should therefore be coupled in via a flat contact region.

Due to the small overall dimensions of the instrument, gravitation alone is not sufficient for keeping a reliable contact between the steel balls and the sample holder. Two small helical springs are installed between hooks on both sides of the two main parts. An additional advantage with this arrangement is that the instrument is virtually orientation independent.

As the entire sample holder is fabricated out of MACOR glass, the electrical connections to the sample are automatically insulated from the instrument frame. Two independent connections can be arranged via screw-mounted combined fastening and contact leaf springs and the two main springs. No special sample leads need, therefore, to be connected when installing an ordinary sample. We experienced that substrate

contacts may occasionally cause problems when cooling a sample; the arrangement with two independent leads provide a simple way of characterizing the connection quality by a circuit measurement after the cooling procedure but before any tunneling attempts are made. The tip holder consists of a high-quality female connector element of the kind normally used in multi-pole LEMO connectors [19]. A short flexible wire is used as a link to the inner lead of a 0.5 mm outer diameter micro miniature semi-rigid coaxial cable.

This kind of coaxial line will serve as an excellent current line but has the additional advantage of providing a microwave path for frequencies up to 18 GHz. A small-diameter shield cylinder, that is overlapping the coaxial line outer lead, is installed between the tip holder and the scanner tube in order to improve the microwave transmission.

6. Feedthroughs and mechanical transmission

The rim of the vacuum flange is shaped to accommodate the indium seal required for a mixing or exchange medium chamber lid.

All electrical leads, including the micro coaxial line, are passed through one single removable sleeve. Sealing is done with STYCAST 2850 FT epoxy resin from Emerson & Cuming Inc. on a "housekeepers seal"-type thinned edge as described by Lounasmaa [20]. A second similar seal is needed between the inner and outer leads of the coaxial line, a method employed several times in our laboratory. The sleeve is inserted into the vacuum flange with a small indium o-ring and a separate clamping flange.

Fine-approach movement is transferred via a non-rotating push rod from the differential screw assembly on the vacuum side. A small nickel bellows is soldered to the push rod and to a small indium seal flange that is installed in a similar way as the electrical feedthrough. The extension of the push rod into the microscope chamber is made out of NILO 36 to a length corresponding to the scanner tube.

The cryostat insert is equipped with a rotary shaft from a feedthrough at the pump head to the
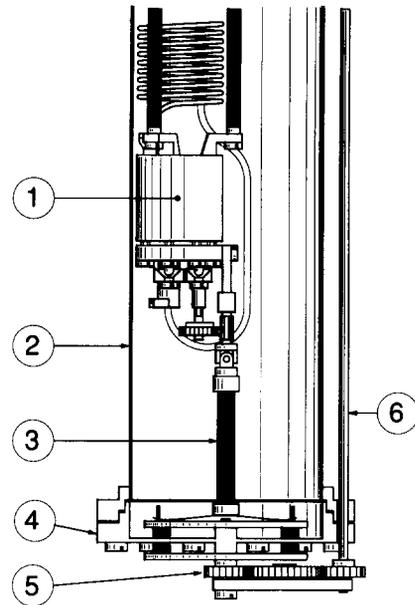

Fig. 3. The lower part of the cryostat insert with the instrument installed: (1) mixing chamber with STM insert, (2) vacuum can, (3) "vespel" thermally insulating drive shaft, (4) bottom flange with built in magnetic coupling, (5) gear train, (6) shaft from room temperature.

lower end of the insulating vacuum can. Transmission to the vacuum is done by means of an axial air gap magnetic coupling incorporated into the vacuum can bottom flange [21]. A thin tubular shaft made from graphite-filled polyimide (VESPEL from Du Pont) serves as a low-thermal-conductivity link between the inner magnet wheel and the microscope insert. A final 1:5 reduction gear has the double duty to give a more smooth screw action and to align the drive shaft to the cryostat centre line (fig. 3).

7. Preliminary results

Instrument performance has been tested in air at ambient temperature and immersed in liquid nitrogen as well as liquid helium. In the room-temperature and nitrogen cases a marble plate suspended by rubber straps from the ceiling was used as a vibration-damped table. For the helium measurements a simple 25 mm diameter "dip stick" was designed for insertion in a storage
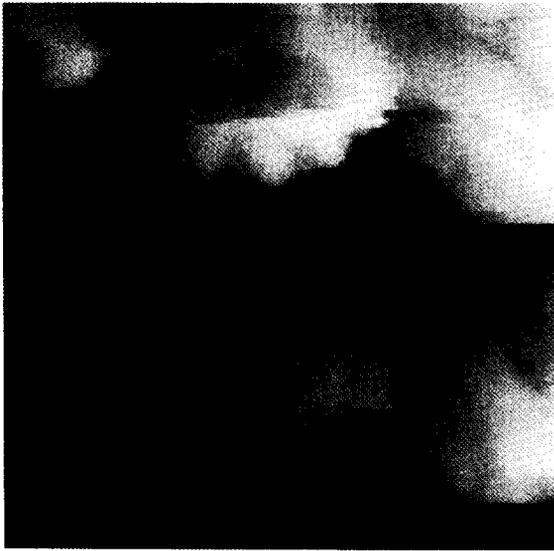
Fig. 4. Electron-beam-lithography-fabricated periodic structure (period 2000 Å, corrugation 500 Å) in gold-plated silicon imaged for piezo tube calibration. Maximum voltage swing gives a scanning range of 4000 Å at 1.2 K.

realistic but not final conditions. This novel design provides a very reliable cooling of the active parts at temperatures below 1 K.

Dewar or a pumped standard cryostat; no special vibration damping was provided. Measurement was made on fabricated periodic structures (fig. 4) [22]. In another set of measurements evaporated gold films was investigated at different temperatures (fig. 5).

A tunneling stability test with the instrument mounted on its final place in the mixing chamber (fig. 3) and with the cryostat cooled to helium temperature has also been done in order to find out whether the vibration level in the dilution refrigerator is low enough; all pumps and auxiliary equipment were operated as in normal service. The z-axis noise level seems to be identical to the one derived in the 1.2 K dip-stick measurements.

8. Conclusions

The capability of a scanning tunneling microscope for operation immersed in the ^{3}He–^{4}He mixture in a dilution refrigerator is proven under
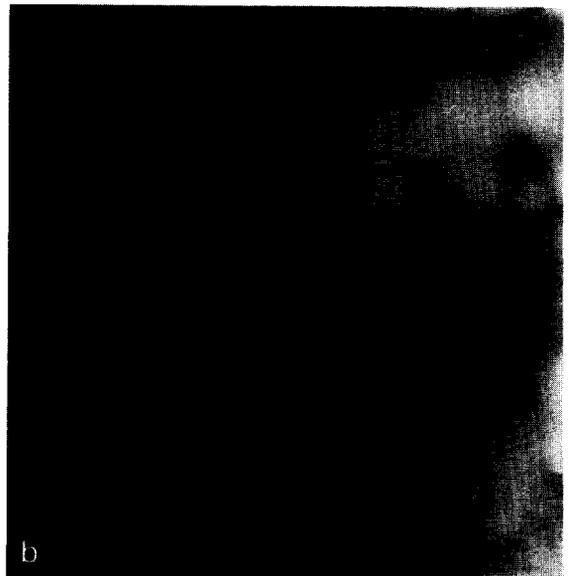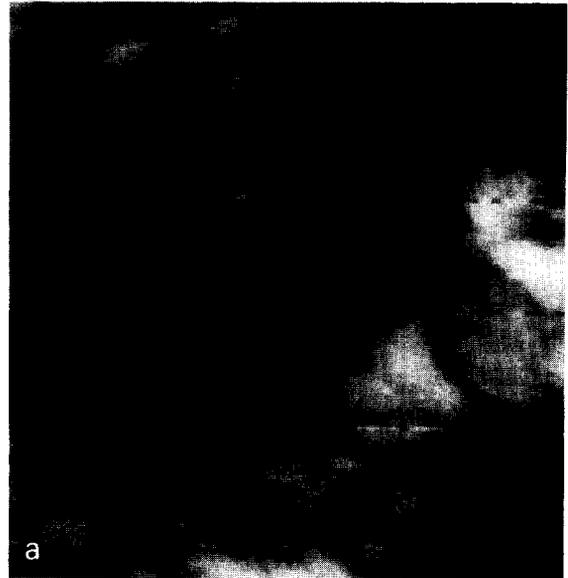
Fig. 5. Evaporated gold film imaged at (a) 77 K and (b) 1.2 K, respectively. In both cases the maximum scanning length was used corresponding to an image side of 2.8 μm at room temperature. The maximum z deflection is in both cases approximately 200 Å. The periodic disturbed lines in the 77 K image come from large gas bubbles escaping from the instrument case.

Acknowledgements

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