Design of an ultrahigh vacuum direct-drive, cryogenic sample manipulator providing two degrees of rotational freedom

Robert M. Braun and Nicholas Winograd
Department of Chemistry, The Pennsylvania State University, 152 Davey Laboratory, University Park, Pennsylvania 16802

(Received 12 February 1993; accepted 19 June 1993)

An ultrahigh vacuum direct-drive, cryogenic sample manipulator is described. It is fully bakeable in UHV, uses no lubricants, and does not require differentially pumped seals. It provides independent polar and azimuthal rotation of a sample, three degrees of translational freedom, electron-beam heating, as well as cooling to 100 K using liquid nitrogen.

I. INTRODUCTION

Although there are many ultrahigh vacuum (UHV) sample manipulators described in the literature, there is still a need for further development in this field. The lack of a universally accepted design is a testament to the number of complexities involved in incorporating sample cooling and heating into a manipulator that also permits many degrees of rotational and translational freedom. In addition, cooling continues to be the major obstacle when designing a sample manipulator with a very large temperature range. However, this barrier is often surmounted by sacrificing one or more degrees of freedom, limiting the precision or decreasing the range of motion of the manipulator.[1–5]

For our experiments, we have rather strict requirements for the accuracy, precision, and range of the azimuthal (ϕ) and polar (θ) rotations of the manipulator, as well as the x, y, and z translations (Fig. 1) which are not met by present designs. A new manipulator, described below, was necessary for the development and extension of adsorption experiments on single crystals using shadow-cone enhanced secondary ion mass spectrometry.[6,7]

II. DESIGN AND CONSTRUCTION

One of the first points to consider, when planning a manipulator, is the number of degrees of freedom required to perform an experiment, followed by the range of motion and resolutions associated with those degrees of freedom. The choice of materials is another concern in UHV systems. Outgassing, galling, vibrational isolation, electrical and thermal conductivity are just a few of the properties that influence the final characteristics of a manipulator. In particular, cooling and heating efficiencies are usually the most difficult attributes to maximize because of contradicting requirements that enable a sample to be cooled to very low temperatures, as well as to be heated to very high temperatures. The former requires excellent thermal contact and minimal thermal mass in order to maximize sample cooling and minimize cooling time while the latter requires poor thermal contact in order to minimize heat transfer to the rest of the manipulator and sample heating time.

Sample positioning is a vital part of shadow-cone enhanced secondary ion mass spectrometry (SIMS) experiments, which require two degrees of rotational and three degrees of translational freedom. The manipulator allows us to rotate a sample on two independent, orthogonal axes. The first, polar (θ) rotation, is described as rotation about the centerline of the drive shaft along the sample face (Fig. 1) and is the axis about which the experiments are performed. The second, azimuthal (ϕ) rotation, is simply defined as rotation about the surface normal at the center of the crystal face which allows us to align a single crystal along a particular crystallographic direction. Both rotations are driven by stepper motors[8] yielding a stepping precision of $\leqslant$0.02° about each axis. The ranges are limited to ±100° and ±120°, respectively. An IBMPS/2 computer is used to interface the motors for stepping control during data acquisition. In addition to rotation, the manipulator can be translated along three cartesian axes using two independent, vertically stacked welded bellows. The lower bellows (not shown) lets us translate the manipulator up to 15-in. along the z axis between various levels in the vacuum chamber for analysis and/or dosing,[9] while the upper bellows (Fig. 2) enables us to manually fine tune the x, y, and z displacements to ±0.001 in. in the range of ±0.5 in. The incorporation of two bellows into our system gives us a significant amount of translational and rotational freedom without the use of differential pumping.[10–12]

As outlined above, motion precision constraints, material properties, and vacuum cleanliness requirements prompted us to design a direct-drive sample manipulator made of 304 stainless steel and oxygen free high conductivity (OFHC) copper. The manipulator and components are shown in Fig. 1. The support tube (A) is a seamless 1.25-in. o.d. stainless steel tube with several holes along its major axis to prevent gases from being trapped inside the tube. An unlubricated stainless steel ball bearing was pressed into the end of this tube to hold the drive shaft running through its center on axis.[13] This tube damps low frequency vibrations and supports coiled 0.125-in. o.d. copper cooling lines. An OFHC copper dewar is attached to the lower end of the cooling lines (Figs. 1 and 3) while 1.33-in. o.d. knife-edge feedthroughs[13] provide the external connections at the upper end (Figs. 2 and 3). Hex-head nuts are used on the threaded connectors to seat the internal copper gaskets (Fig. 3). These external connections allow the coolant delivery lines to be completely removed

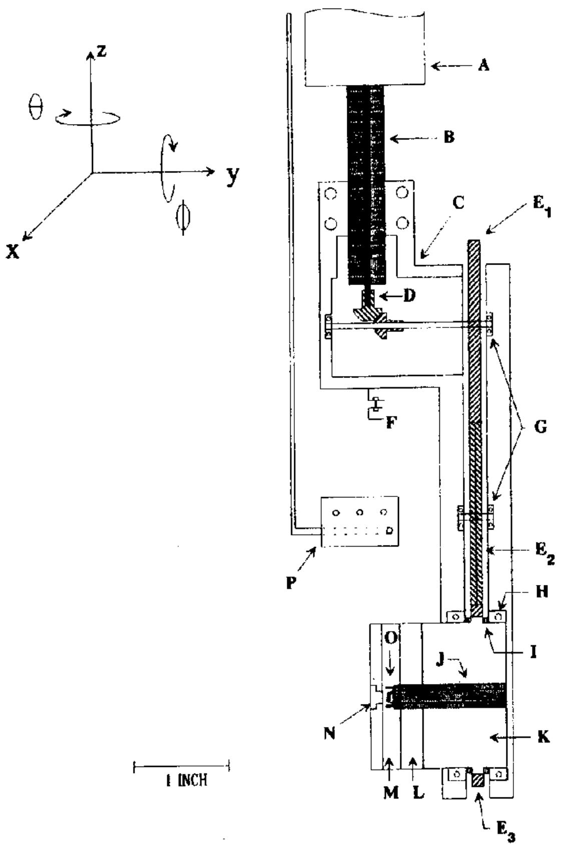

FIG. 1. Cross section of manipulator body: (A) 1.25-in. o.d. support tube; (B) coaxial, rotary-feedthrough (outer and inner shafts are for polar and azimuthal rotations respectively); (C) 304 stainless steel manipulator body; (D) bevel gears, upper is antibacklash; (E_{1-3}) 1.875-in. spur gears, (E_2) is antibacklash; (F) electrically isolated stainless steel current plate (lower), grounded stainless steel plate with P1 phosphor (upper); (G) stainless steel ball bearings for gear train support; (H) slim line ball bearings to hold (K); (I) 0.050 in. of stainless steel spacers on each side to align sample face on center line of (B); (J) Macor filament support, rotates with sample (clamps and wires not shown for clarity); (K) 304 stainless steel hub, supports (E_3) and (J); (L) Macor disk for thermal isolation of (M and N); (M) OFHC copper braid mount used to attach OFHC copper braid to (P) (braid not shown for clarity); (N) OFHC copper sample mount with counter bore for sample; (O) tantalum, electron-reflector cup surrounding a 0.25-mm tantalum filament; (P) OFHC copper dewar and cooling lines from continuous flow LN2 system.

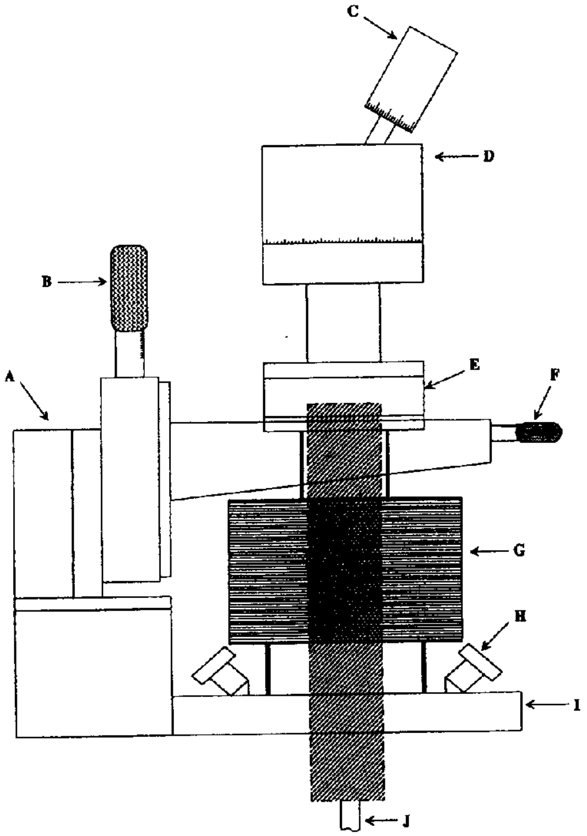

FIG. 2. Side view of upper XYZ translation stage: (A) Stainless steel stage support; (B) z-vernier (± 0.5-in.); (C) azimuthal drive unit of manipulator; (D) polar drive unit of manipulator; (E) 2.75-in. knife-edge spacer welded to 1.25-in. o.d. support tube in Fig. 1; (F) y-vernier (x-vernier not shown) (±0.5-in.); (G) 2.64-in. i.d. 346 stainless steel welded bellows; (H) 1.33-in. o.d. feedthroughs for electrical and cooling lines; (I) 6-in. o.d. knife-edge flange that attaches to lower bellows assembly (not shown); (J) coaxial rotary feedthrough shaft in Fig. 1. X, Y, and Z axes shown at top of Fig. 1. Θ=polar rotation axis, ϕ=azimuthal rotation axis.

from our UHV system or easily modified for other experiments.

Sample rotation is performed by drive shaft (B) which is part of a Thermionics model No. FRRC-275 coaxial rotary feedthrough.[14] This enables us to remotely control polar (θ) and azimuthal (ϕ) rotation of our sample. Polar rotation is established by clamping the stainless steel manipulator body (C)) to the outer drive shaft, while the azimuthal drive-train (D–E_{1-3}) couples to the inner drive shaft allowing the sample to be rotated about an orthogonal axis. The manipulator body uses two sizes of high precision stainless steel ball bearings (G,H), which support three stainless steel spur gears (E_{1-3}), in the azimuthal drive assembly. In addition, two anti-backlash gears are used to minimize hysteresis that would ultimately decrease our resolution and reproducibility. The first is a spring loaded bevel gear[15] (D) and the second is a spring-loaded, split spur-gear[15] located in the center of the drive assembly (E_2). The last spur gear in the assembly (E_3) was bored so it could be pressed around the stainless steel cylindrical hub (K). The protruding rim of hub (K) is separated from slim-line ball bearings (H) by 0.050 in. of thin stainless steel spacers (I) on each side. These spacers allow the sample face to be positioned close to the centerline of the drive shaft by repositioning them on either side of the hub. The sample is precisely placed on the center line of the drive shaft using sample mount (N) and is held in place by tantalum clips made from 2.0 mm wire. This allows individual sample mounts to be fabricated, at relatively low cost, in order to accommodate a diverse range of sample diameters and thicknesses. Positioning the sample on the centerline of the drive shaft minimizes the off axis transition or round-out of the sample face as it is rotated about the polar axis.

The cooling and heating efficiencies of the manipulator are noticeably increased by thermally isolating stainless steel hub (K) from the OFHC copper mounts (M,N) using a Macor[16] disk (L). Sample cooling is achieved by

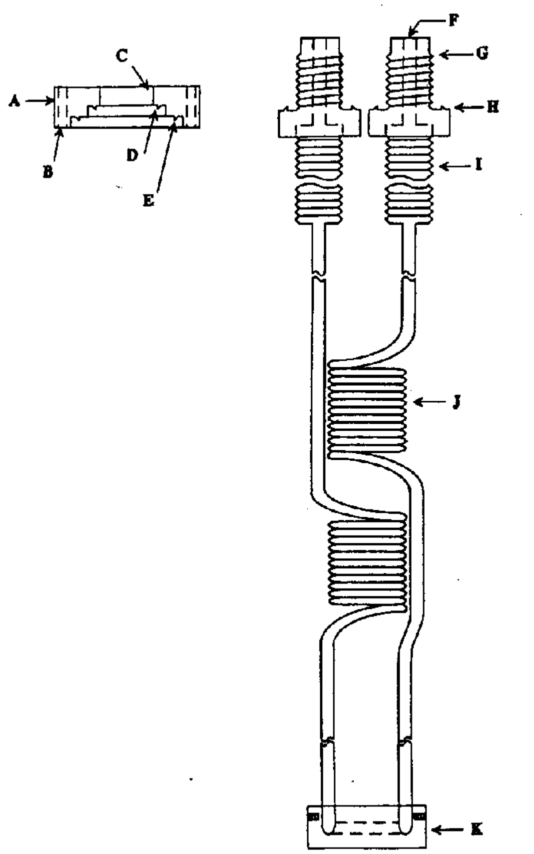

FIG. 3. End view of cooling lines and dewar assembly. (A) Modified 1.33-in. o.d. knife-edge flange; (B) bolt holes; (C) clearance hole for cooling line connector (G); (D) knife-edge to mate with (H) on cooling line connector; (E) standard 1.33-in. o.d. flange knife-edge; (F) liquid nitrogen inlet/outlet hole; (G) liquid nitrogen cooling line connector; (H) knife-edge for UHV seal with (A); (I) 0.325-in. o.d. flexible, stainless steel conduit; (J) 0.125-in. o.d. coiled copper cooling lines, supported by (A) in Fig. 1; (K) OFHC copper dewar with threaded holes to attach copper braid.

thermally connecting OFHC copper braid, which offers significant flexibility, from braid mount (M) to copper dewar (P) (braid not shown for clarity). The copper braid is crimped into the ends of 0.187-in. long pieces of 0.125-in. o.d. copper tubing using a machinists vise.[17] Small machine screws are used to attach the braid at each end. Besides providing a means of establishing good thermal contact between the dewar and sample mounts, the tubing also helps to reduce wear and fraying of the braid. To date, we have not tried gold plating the copper parts in the cooling assembly. Ultimately, this would reduce the temperature drop across the contact junctions which should augment the cooling process.[18,19] Heating, on the other hand, is promoted by an electron beam from the rear of the sample. The main support for the heater assembly (J) comes from a cylindrical Macor rod that is clamped at the rear of hub (K) (Clamp and wires not shown for clarity). Fiberglass insulated copper braid is used to connect the filament wires to the electrical feedthroughs (Fig. 2) in order to minimize strain upon rotation. Two holes running the length of the Macor rod hold 1.00 mm tantalum posts that are spot welded to a coiled length of 0.25 mm tantalum wire. This coiled filament positioned directly behind the sample, sits inside of a tantalum cup (O) which acts as an electron reflector. Two 0.010-in. chromel/alumel prewelded thermocouples are spot welded to the sample face for temperature monitoring and also rotate with the sample.

Finally, the ion beam, which passes between the two copper cooling lines (Fig. 3), is detected and measured on two stainless steel plates (F) (Fig. 1). The upper, grounded plate is coated with a thin film of P1 phosphor[20] in order to visually focus the ion beam while the lower, electrically isolated plate is connected to a picoammeter for current measurement.

III. TEMPERATURE CONTROL

A. Cooling

Sample cooling is accomplished by a continuous flow liquid nitrogen delivery system consisting of 0.125-in. o.d. copper tubing, 0.325-in. o.d. flexible stainless steel conduit,[21] and an OFHC copper dewar that are all silver soldered together (Fig. 3). With this coolant delivery system in place, the sample can be cooled from room temperature to 100 K with liquid nitrogen in ~1 h. Subsequent flashing to elevated temperatures followed by cooling takes significantly less time. This is important when one wishes to dose a sample with overlayer species since it is undesirable to have background contaminants present on the surface.

B. Heating

Electron-beam heating is performed by running 4–5 A of ac current through the filament (Fig. 1) which is biased to 1000 V dc in order to heat the grounded sample using $\leqslant 100$ mA of emission current. There are two heating configurations that will be discussed below. The first involves direct electron-beam heating to the back of the crystal while it is positioned in the counter bore of sample mount (N) (Fig. 1). In this configuration it is possible to heat the sample to ~1000 K whether the continuous-flow liquid nitrogen system is on or off. Heating the sample beyond the limit increases the temperature of the copper mounts until they ultimately reach the same temperature as the sample. Since many transition metals require annealing temperatures that exceed 1000 K, a second configuration has been implemented which extends the manipulators heating range by ~500 K while abating heat transfer to the sample mounts. This is accomplished by placing a 0.010-in. thick tantalum washer between the sample and copper mount (N) (Fig. 1). We still use electron beam heating to the back of the crystal and are able to obtain temperatures in excess of 1450 K without significantly increasing the temperature of the copper sample mounts.

ACKNOWLEDGMENTS

This work was supported by the National Science Foundation, the Office of Naval Research, the Department of Energy and the National Institute of Health. We would like to thank E. E. Chaban for his assistance and technical expertise during the production of this manipulator. Finally, we thank J. S. Burnham for many fruitful conversations during the development process.

[1] P. G. Strupp and P. C. Stair, J. Vac. Sci. Techol. A **9**, 2410 (1991).
[2] H. Dürr, Th. Fauster and R. Schneider, J. Vac. Sci. Technol. A **8**, 145 (1990).
[3] D. Venus, Rev. Sci. Instrum. **62**, 1361 (1991).
[4] N. J. Wu and A. Ignatiev, Rev. Sci. Instrum. **56**, 752 (1985).
[5] D. J. D. Sullivan and A. C. Kummel, Rev. Sci. Instrum. **63**, 4285 (1992).
[6] C.-C. Chang and N. Winograd, Phys. Rev. B **39**, 3467 (1989).
[7] C.-C. Chang and N. Winograd, Surf. Sci. **230**, 27 (1990).
[8] Superior Electric, 383 Middle Street, Bristol, CT 06010-7488.
[9] R. A. Gibbs and N. Winograd, Rev. Sci. Instrum. **52**, 1148 (1981).
[10] A. Pararas, S. T. Ceyer, and J. T. Yates, Jr., J. Vac. Sci. Technol. **21**, 1031 (1982).
[11] S. M. George, J. Vac. Sci. Technol. A **4**, 2394 (1986).
[12] J. A. Stroscio and W. Ho, Rev. Sci. Instrum. **55**, 1672 (1984).
[13] MDC Vacuum Products Corporation, 23842 Cabot Boulevard, Hayward, CA 94545-1651.
[14] Thermionics Laboratory, 22815 Sutro Street, Hayward, CA 94541.
[15] Winfred M. Berg, Inc., 499 Ocean Avenue, East Rockaway, NY 11518.
[16] Registered Trademark, Corning Glass Works, Corning, NY 14831.
[17] Private communication with E. E. Chaban, AT&T Bell Laboratory.
[18] R. E. Palmer, P. V. Head, and R. F. Willis, Rev. Sci. Instrum. **58**, 1118 (1987).
[19] V. K. F. Chia, D. K. Viers, and G. M. Rosenblatt, J. Vac. Sci. Technol. A **7**, 108 (1989).
[20] USR Optonix, Box 409, Hackettstown, NJ 07840.
[21] Simmonds Precision, Co-Operative Industries, 100 Oakdale Road, Chester, NJ 07930.