Inexpensive ultrahigh vacuum heatable/coolable *xyz*-rotary motion sample manipulator

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A simple design for a heatable, coolable, rotatable sample manipulator, suitable for ultrahigh vacuum (UHV) applications, is described. Highlights of the design include using a combination power/thermocouple feedthrough for heating, cooling, and temperature measurement; and the use of primarily "off-the-shelf" components available from most UHV components vendors. The described manipulator is capable of sample cooling to ~100 K, sample heating to above 900 K, while maintaining 360° of rotary motion, ~1 in. of *x* and *y* motion, and 2 in. of *z* motion. The apparatus can be assembled for approximately $5500 (all new parts) and uses about 3 ℓ of liquid N_2 per day. © *1995 American Institute of Physics.*

I. INTRODUCTION

Various designs of sample manipulators for use in ultrahigh vacuum chambers have been proposed in the past decade. These designs range from simplistic[1–4] to complex.[5–7] The appropriate choice of a sample manipulator depends on its intended use and the complexity of sample orientation necessary to perform experiments. This paper details an inexpensive, easily assembled, heatable/coolable top-down sample manipulator with *xyz* and rotational motion for use in a single chamber ultrahigh vacuum (UHV) system.

This design has several distinct advantages. First, most of the necessary components for the manipulator assembly are purchased "off the shelf;" our design requires only one custom-built part. Second, the apparatus is inexpensive to assemble and operate because of the simplicity of the design. Third, the connection between the liquid nitrogen well and the sample support hardware ensures a rapid return to low sample temperatures (~100 K) after heating the sample in excess of 900 K. Fourth, relatively small amounts of liquid nitrogen are used in sample cooling, making this manipulator configuration economical to operate. Finally, the suggested design incorporates a combination thermocouple-power feedthrough, which allows straightforward sample heating and cooling with convenient temperature measurement.

The sample manipulator reported here is mounted in an UHV system outfitted with Auger electron spectroscopy, thermal programmed desorption (TPD), low-energy electron diffraction, a platinum filament for gas dissociation, and a four-point surface conductivity probe. Because the experimental equipment for these techniques occupies different coplanar ports on the vacuum chamber, it is necessary to orient the sample in the *x*,*y*,*z* directions and rotationally about the system's vertical axis.

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II. DESIGN

This design incorporates a liquid nitrogen reservoir in thermal contact with the sample to facilitate cooling, employs a resistive sample heating, and uses a type K thermocouple feedthrough for temperature measurement. The apparatus described is a modification of a previously reported liquid nitrogen cooled cryostat with rotary motion.[1] The thermocouple and resistive heating wires are run inside the tube that serves as the liquid nitrogen reservoir. This arrangement, coupled with an *xyz* precision manipulator and a differentially pumped rotary seal, provides ~1.0 in. movement in the *x* and *y* directions, a 2 in. range in the vertical direction, and full 360° of rotational motion.

Figure 1 shows a schematic of the sample manipulator system. The major components are all illustrated: the precision *xyz* sample manipulator; the stainless steel liquid N_2 reservoir tube; and the combination thermocouple and power feedthrough. Table I details the approximate cost and component manufacturer for the major items needed to assemble the sample manipulator.

The *xyz* manipulator is the key component in the assembly. Several manufacturers make *xyz* manipulators that would work with our design. We chose a manipulator with *x* and *y* movements of 1 (±0.5) in., and a *z* (vertical) movement of 2 in. The only constraint on the *xyz* stage is that the hole in the sample bellows had to be large enough to allow for the passage of a miniflange (1.33 in. diam). The size of the base flange of the *xyz* stage was chosen to match the top port on our UHV chamber.

In order to allow sample rotation to access different coplanar ports, a differentially pumped rotary seal was added to the *xyz* sample stage. There are several manufacturers of differentially pumped 2.75 in. rotary seals, but only the Thermionics RNN-150 had a large enough hole (1.53 in.)[8] to allow a miniflange to easily pass through. The rotary seal was attached to the top of the *xyz* stage, and was connected

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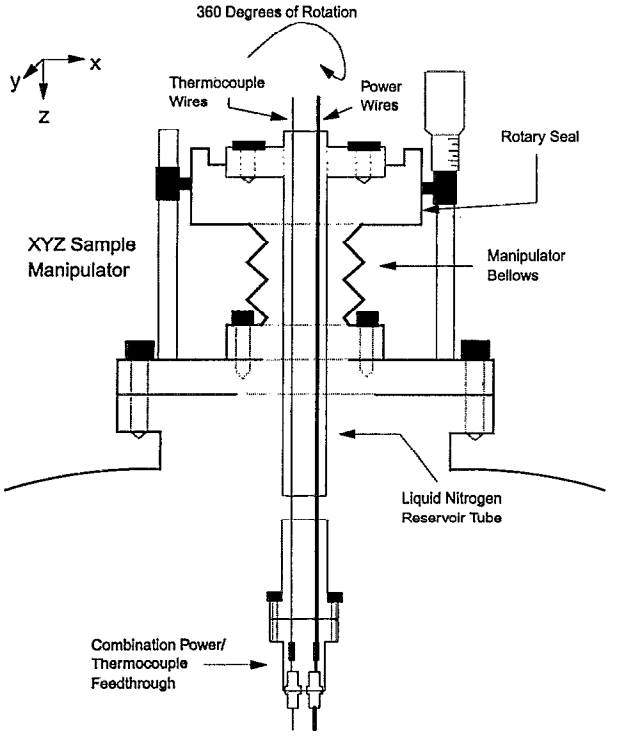

FIG. 1. A schematic diagram of the entire manipulator assembly including the *xyz* manipulator, the rotary seal, the liquid nitrogen reservoir tube, and the power/thermocouple feedthrough. The diagram is not drawn to scale.

to a mechanical roughing pump (first stage) and a UHV pump (second stage). The advantage of the differentially pumped seal is that there is little change in system pressure during sample rotation.

The only required part of the entire manipulator assembly that is not commercially available off the shelf is the liquid nitrogen reservoir tube. This piece is simply a stainless steel nipple (11/32 in. i.d., 0.75 in. o.d.) with a miniflange at one end and a 2.75 in. flange at the other. The length of the tube is dictated by the design of the UHV chamber and the *xyz* manipulator selected. However, careful measurements of the overall length must be made to ensure that the sample is in a suitable position for experiments while allowing some possible adjustment of the sample position. A schematic diagram of the tube is shown in Fig. 2. The liquid nitrogen

TABLE I. The major components, manufacturer, and approximate cost necessary to construct the manipulator assembly.

Component	Model number	Approximate cost
XYZ sample manipulator	Thermionics *xyz* manipulator, EC-1275-1-1-2	$2900
Differentially pumped rotary seal	Thermionics rotary seal, RNN-150	$2200
Stainless steel LN_2 reservoir tube	Custom manufactured	$100
Power thermocouple feedthrough	Insulator seal incorporated 9392015	$210

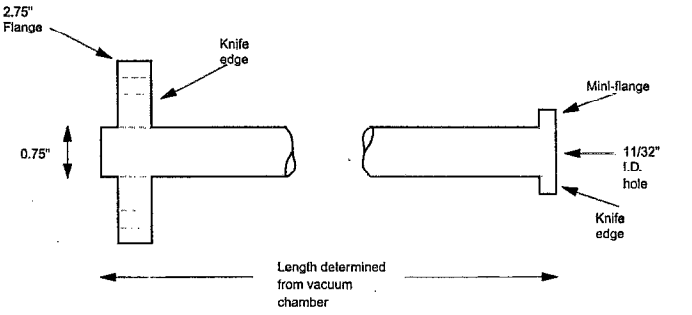

FIG. 2. A schematic diagram of the stainless steel tube used as the liquid nitrogen reservoir.

reservoir tube has two purposes in addition to providing sample cooling. It positions the sample in the center of the UHV chamber, and it houses the thermocouple and heating wires. It should be noted that the addition of the tube to the manipulator assembly decreases the full range of x and y motion from 1 ± 0.5 in. to $\sim0.5\pm0.5$ in. due to the tube coming in contact with the side of the manipulator bellows at the extremes of the x and y motion.

The final critical component of the manipulator assembly is the combination power/thermocouple feedthrough. A variety of these are available[9] with various power ratings and thermocouple types. We chose a feedthrough that had the highest power rating (5 kV–30 A) with a type K thermocouple mounted on a miniflange.[10] Wires for heating (10 gauge) were fastened to the power feedthroughs using crimp connectors, and were then encased using shrink wrap plastic tubing. Thermocouple extension wires were also connected to the thermocouple feedthrough wires. The feedthrough was then mounted on the miniflange at the end of the liquid nitrogen reservoir, with the wires fed through the stainless steel tube (see Fig. 1). The entire apparatus was then mounted on the rotary seal using the 2.75 in. flange at the opposite end of the tube.

There are a number of ways to mount samples to the power leads on the feedthrough. We constructed copper blocks, approximately 1/8 in. thick and 3 in. long, which were attached to the power leads. The copper blocks are both electrically and thermally conductive and act as a cold sink when cooled with liquid N_2. A schematic diagram of the copper blocks is shown in Fig. 3. The sample is clamped onto a thin tantalum plate (0.25 mm×12.5 mm×17.5 mm)

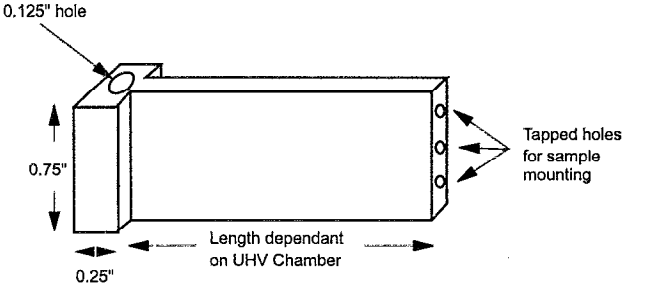

FIG. 3. A schematic diagram of the thermally and electrically conductive copper blocks used for sample mounting and positioning.

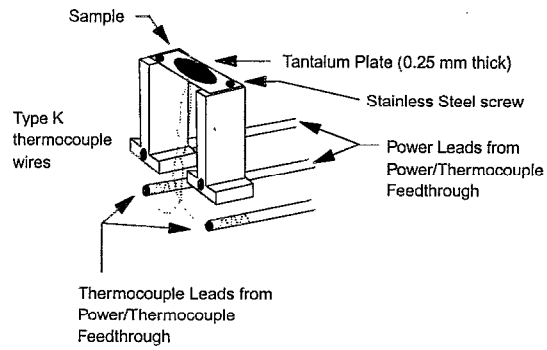

FIG. 4. A schematic diagram showing an enlarged view of the sample mounting blocks, the tantalum mounting plate, the sample, and the thermocouple wires.

which is attached to the ends of the copper blocks using stainless steel screws. A small hole in the tantalum plate allows thermocouple wires to be cemented to the back of the sample. A diagram illustrating the sample mounting to the copper blocks is shown in Fig. 4. Our samples (2 mm×10 mm×10 mm) are fragile ceramic materials (normally metal oxide or metal sulfide single crystals), and are mechanically attached to the tantalum plate using tantalum shims held in place with stainless steel screws.

III. PERFORMANCE

Sample cooling is accomplished by pouring liquid nitrogen (using a funnel) into the liquid N_2 reservoir. Initial cooling from room temperature to 100 K may take 10–30 min. The heater and thermocouple wires can be connected to a dc power source for heating and a conventional type K thermocouple readout device, respectively. Sample heating is accomplished by passing current through the thin tantalum plate, thus heating the sample by conduction. Heating rates of ~4 K/s were attained with this apparatus. Higher heating rates may be possible, but because of the ceramic nature of our samples, no larger heating rates have been attempted. We have found that a 0–50 A, 0–5 V power supply is sufficient to heat our samples in excess of 900 K. Following heating, the power supply is turned off, after which the sample immediately begins to cool. It is advised to add liquid N_2 during the cooldown period to keep the reservoir "topped off." This cycle is repeated as many times as necessary.

One of the advantages of this design is the capability to allow numerous heating and cooling cycles over the course of one day of experiments. The rate of sample cooling following an experiment is ~30 K/min. With this apparatus, TPD trial runs were performed every 45–55 min in our laboratory. The limiting factor in the number of executed experiments per hour was the ability of our ion pump to maintain low chamber pressures following large gas doses.

The liquid nitrogen requirements for this apparatus are modest. We typically consume 1 ℓ of liquid N_2 per 10 cooling/heating cycles, with an additional 2 ℓ of liquid N_2 for initial system cooldown. Thus, this design requires relatively little liquid nitrogen for continuous operation.

Because of the vertical position of the liquid nitrogen reservoir, water condenses in the stainless steel tube following experimentation (after liquid nitrogen boils off). Significant problems occur if this condensate is not removed prior to subsequent experimentation. Reintroduction of liquid N_2 into the reservoir freezes the condensate, thereby cracking the ceramic parts of the power/thermocouple feedthrough. An effective method to remove the condensed water involves flushing the liquid nitrogen reservoir with dry nitrogen gas following experimentation. A thin 1/8-in.-diam copper tube is inserted into the liquid N_2 reservoir tube, running along next to the insulated thermocouple and power wires and serves to convey dry nitrogen gas to the bottom of the tube. A 5–10 min period of flushing with nitrogen has proven to be sufficient to evaporate the condensate.

It should be noted that as the sample support hardware warms to room temperature following an experimental session, the chamber pressure will increase substantially. This is caused by the desorption of adsorbed molecules from the sample support hardware.

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[10] The combination power/thermocouple feedthrough used is not rated by the manufacturer for LN_2 immersion applications. We obtain a life of approximately 12–15 months for the specified feedthrough.