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Precision Internal Temperature Control in a High Pressure Apparatus

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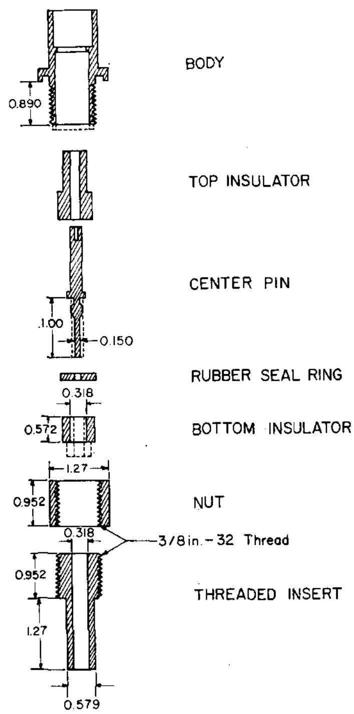

FIG. 2. Connector parts. Dashed lines indicate material removed from the original UG-657/U connector. (All dimensions in centimeters.)

liquid helium bath because the breakdown potential of helium vapor is very low. Here, we describe a coaxial connector (not constant impedance) which avoids this source of electrical breakdown for rf voltages up to at least 2.5 kV. It is based on a simple modification of a standard hermetic seal BNC connector. The breakdown is prevented by covering the center conductor, which carries the large rf voltage, with a high dielectric strength material that is unbroken throughout the entire helium vapor region.

The high dielectric strength material we have used is Teflon tubing. The modifications to the BNC connector permit the tubing to pass through a mounting plate into the laboratory atmosphere with a vacuum tight seal.

Now consider the configuration of the modified connector in its assembled form, as shown in Fig. 1. Details of the modifications are given in the next paragraph. The body of the connector is held in place on the mounting plate with the nut. An hermetic seal between the outer surface of the body and the mounting plate is effected with the O-ring. The top insulator, center pin, rubber seal ring, and bottom insulator are secured by the threaded insert. It is tightened enough to compress the rubber seal ring against the inner surface of the body and the Teflon tubing, thereby extending the hermetic seal to these parts. The final seal is obtained by the tight fit of the Teflon tubing over the small shoulder which is left after a machining operation on the lower part of the center pin. With these arrangements, the connector is vacuum tight and the Teflon insulation is brought out of the helium vapor without a break.

The modifications to the connector parts are shown in Fig. 2. The body, O-ring, top insulator, center pin, rubber seal ring, and bottom insulator are all obtained from a standard UG-657/U connector. Modifications to these parts, in the form of material removed, are indicated by dashed lines. The first operation is to remove the material on the body with a lathe; then the connector is easily disassembled. The nut and threaded insert are extra parts, made from brass. A tight fit between the shoulder left on the lower part of the center pin and the Teflon tubing is obtained by using AWG size 14 standard wall tubing (1.68 mm i.d., 0.41 mm wall thickness).

We have found it convenient to use the connector in coaxial setups as follows. First, the body is attached to the mounting plate. Then, the center conductor is soldered to the bottom of the center pin. A length of Teflon is pushed over the conductor and pin as shown in Fig. 1. The rubber seal ring is slipped into place over the Teflon. Internal parts are put into place and secured with the threaded insert. Our outer conductor is a piece of 6.35 mm o.d. by 0.25 mm wall thickness stainless steel tube. It is slipped over the small end of the threaded insert and secured with a clamp. The Teflon tubing is held in the center of the outer conductor by thin strips of masking tape wound around the Teflon at 2 in. intervals.

The arrangement used here has been employed many times in pulsed NMR experiments. It is easy to construct and assemble. Although the maximum gas lead rates and breakdown potential of this connector have not been measured, it is known that the gas leakage is small enough to be no problem with the usual pumped liquid helium systems, and rf voltages of 2.5 kV peak can be sustained without breakdown.

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Precision Internal Temperature Control in a High Pressure Apparatus

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IN attempting to do annealing and diffusion experiments at high pressures[1] the following technique was devised for temperature control of a furnace within the high pressure chamber, which has proved to be extremely useful. These experiments required a rapid temperature rise to the desired value and a closely controlled temperature over long periods of time. The first of these requirements

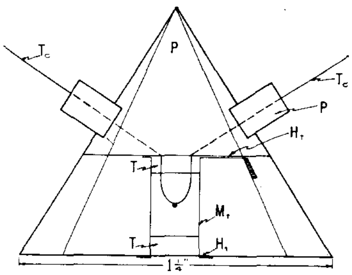

FIG. 1. Crossection of the furnace within the high pressure cell. The tetrahedron is made of pyrophyllite P; T_c's are the thermocouple leads; H_t's are metal tabs to serve as electrical leads to the heater; M_t is a thin wall monel tube; and T's are Teflon plugs to seal the liquid in the tube.

is very simple in a high pressure cell because the furnace volume is necessarily very small, with a small heat capacity, and the heater is unavoidably in good thermal contact with its surroundings, making temperature changes very rapid. The thermal response time of our system is less than 1 sec in contrast to several minutes or hours for large volume furnaces outside the press. The large heat loss to the surroundings and the small volume of the furnace require large currents at low voltage to attain the desired temperatures. Recently this technique has also been applied to controlling the temperature of the specimen over long periods of time in high temperature, high pressure x-ray measurements.

The scheme presented here used a furnace similar to that discussed by Curtin, Decker, and Vanfleet[2] and is shown in Fig. 1. The furnace is a Monel tube 0.47 cm diam with a 0.010 cm wall thickness. This metal tube, which was filled with petroleum ether, required 175 A at 1.8 V to heat to 300°C at 25 kilobars. The 0.25 mm diam Chromel–Alumel thermocouple passed out of the high pressure region through one of the pyrophyllite gaskets. A reference junction at 0°C was used.

Figure 2 gives a schematic diagram of the control circuit. The voltage signal from the thermocouple junctions is bucked against a reference voltage, which is set to give the desired temperature. The sense of the respective voltages is indicated in the figure. The nature of the reference voltage is not vital to the operation of the control circuit. We used the simple scheme shown in the figure with a mercury battery for long term stability. A lower impedance reference emf would help reduce noise and pickup. The difference voltage is integrated by an operational amplifier, the output of which replaces the control voltage in the control bridge of a programmable power supply. The basic requirement of the operational amplifier is that it have low zero drift to insure long term temperature stability. We used a Dymec 2460A. Any power supply which can be programmed by an external voltage can be used. We controlled the current from Kepco KS 8–100 supplies, either singly or in parallel operation, because of the high currents required in our application. The high degree of regulation for these supplies is probably not necessary for this control system.

In operation, the emf of the reference is set to give the desired temperature using thermocouple tables. The power to the heater will change at a rate which is proportional to the difference between the furnace temperature and the set temperature. When the furnace temperature matches the set value the input to the integrator is zero and the output remains constant. Since there is a maximum allowable current in the power supply control bridge, R_2 must be chosen so that one does not exceed this value when the amplifier is driven to saturation. In our application the

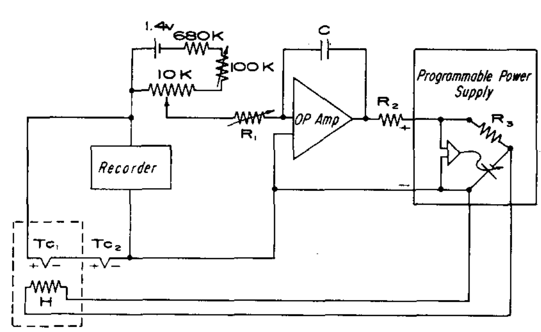

FIG. 2. Schematic of the circuit. T_{c1} is the sensing thermocouple in the heater, H, within the high pressure cell. T_{c2} is a reference junction at 0°C. The connection to the control bridge within the power supply is also indicated. The remainder of the circuit is discussed in the text.

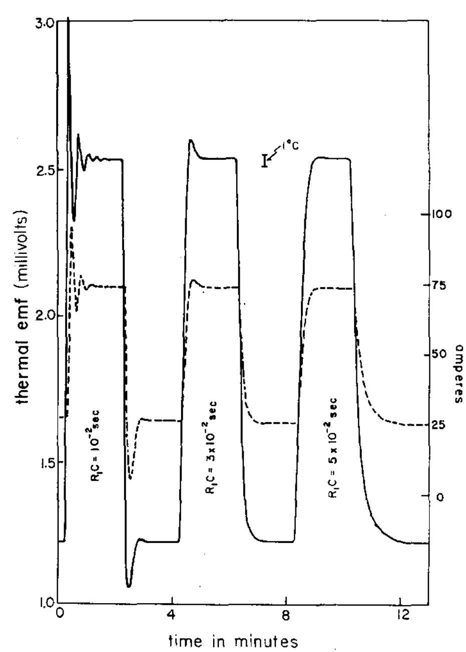

FIG. 3. Chromel–Alumel thermocouple emf (solid line) and output current from the power supply (dashed line) vs time. The reference emf was switched between 1.2 and 2.55 mV for 3 different values of the integrating time constant R_1C.

rated bridge current is 10 mA so R_2 was 1100 Ω. Since the output of the integrator is bipolar we would recommend passing the operational amplifier output through a diode (not shown) to prevent a reversal of current in the control bridge circuit. The gain of the circuit can be adjusted by varying the output control resistor R_3 within the power supply. The feedback capacitor C and input resistor R_1 control the integration rate of the feedback loop. If the product R_1C is too large, the temperature response is sluggish, and if R_1C is much smaller than the thermal response time of the furnace the temperature will oscillate. The recorder is only to monitor the temperature vs time; however, it must have a high input impedance so as not to disturb the circuit.

The temperature within the furnace as well as the current output of the power supply are shown as functions of time in Fig. 3 for a series of step changes from 30 to 62°C. The integrating time constant can be adjusted as demonstrated in the figure such that the new temperature is attained without overshoot and controlled to within ± 0.05C° in about 1 min. The thermal response time of the furnace increases with temperature, so in practice R_1 is made variable, allowing one to adjust the product R_1C during operation. We chose C equal to 0.1 μF and R_1 could be stepped from 5 kΩ to 5 MΩ with a rotary switch. Monitoring the internal temperature with a strip chart recorder revealed a long term stability better than ± 0.03C° for periods of several hours. We have also checked this stability by a 4 lead measurement of the resistance of a gold wire in the pressure cell while controlling at 100 and 200°C. The resistance varied by less than $+0.02\%$ even upon returning to a given set point after leaving it, corresponding to a temperature variation of less than ± 0.05C°. Since one is controlling on the emf of a thermocouple under pressure there will be a problem at high temperatures where degradation of the thermocouple takes place. All the results reported here are at relatively low temperatures where this will not be a problem. A problem which does arise in the high pressure system is electrical shorting of the thermocouple to the heater within the pressure cell. This will reduce the maximum output of the supply and not allow the circuit to function correctly.

The application of this technique to control other than temperature is obvious.

I wish to thank H. L. Laquer, H. B. Vanfleet, J. L. Peel, Jack Weyland, and J. D. Barnett for their help in developing this system.

[1] J. L. Peel, "Annealing of Vacancies in Gold under High Pressure," unpublished thesis, Brigham Young University, 1967.

[2] H. R. Curtin, D. L. Decker, and H. B. Vanfleet, Phys. Rev. **139**, A1522 (1965).

EPR Sample Orientation Servo

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IN many electron paramagnetic resonance (EPR) experiments, it is frequently desirable to change the orientation of the sample with respect to the cavity without removing the cavity from the spectrometer. This is the case when one is empirically searching for the orientation of the principal axis of the crystalline field interaction with respect to the crystal morphology. An-

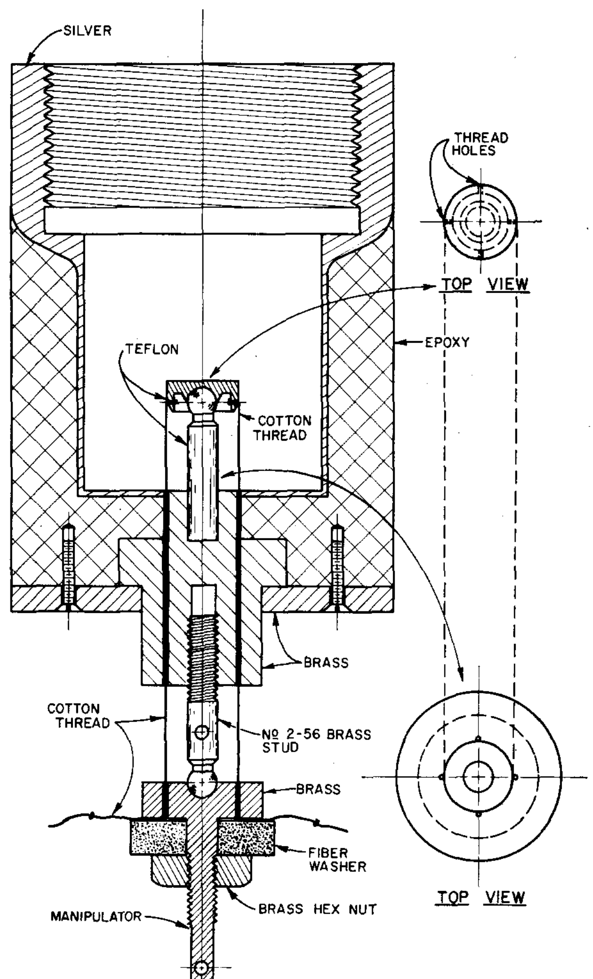

FIG. 1. Sketch of a cross section through the center of sample orienter in a 16.8 mm diam K band microwave cavity which indicates relative sizes. The orienter is held in place by a brass (or other nonmagnetic material) washer which is screwed to the epoxy cavity.