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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 pressures¹ 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 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 Vanfleet² 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

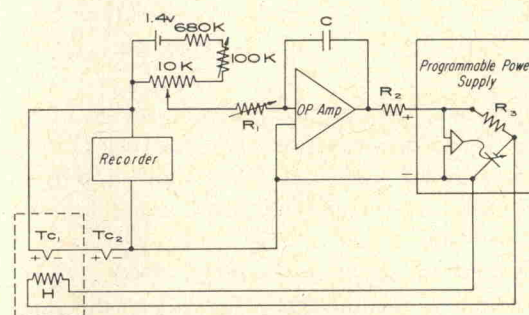
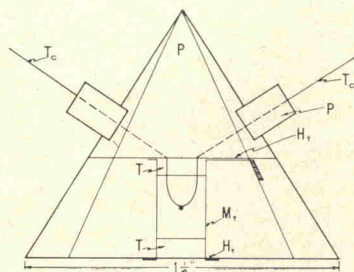


FIG. 2. Schematic of the circuit. T_{c1} is the sensing thermocouple in the heater, H , within the high pressure cell. T_{c2} is a reference junction at 0°C. The connection to the control bridge within the power supply is also indicated. The remainder of the circuit is discussed in the text.

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

FIG. 1. Cross-section of the furnace within the high pressure cell. The tetrahedron is made of pyrophyllite P ; T_c 's are the thermocouple leads; H '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.



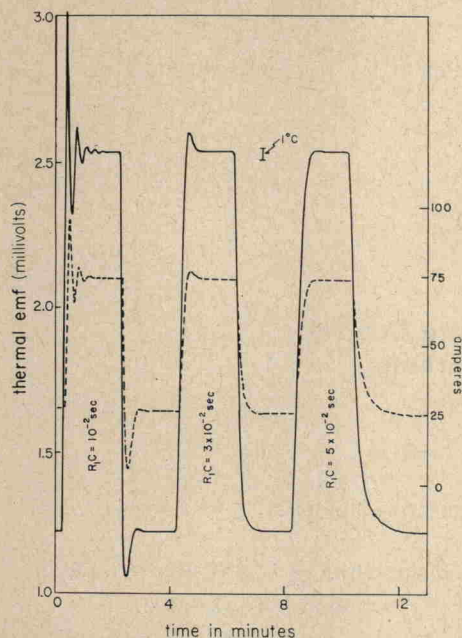


FIG. 3. Chromel-Alumel thermocouple emf (solid line) and output current from the power supply (dashed line) vs time. The reference emf was switched between 1.2 and 2.55 mV for 3 different values of the integrating time constant R_1C .

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 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 $\pm 0.05^\circ$ 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 k Ω to 5 M Ω with a rotary switch. Monitoring the internal temperature with a strip chart recorder revealed a long term stability better than $\pm 0.03^\circ$ 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 $\pm 0.05^\circ$. 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).