Bakeout controller for the use of helium closed-cycle refrigerators in ultrahigh vacuum applications[a]

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Simple control electronics are described which provide automated bakeout of an ultrahigh vacuum (UHV) chamber equipped with a helium closed-cycle refrigerator. By preventing the refrigerator from reaching elevated temperatures ($T > 50$ °C) during bakeout, these devices can be utilized in the ultrahigh vacuum environment.

I. INTRODUCTION

The helium closed-cycle refrigerator provides a convenient way to achieve ultralow temperature without the daily consumption of cryogenic liquids. Matrix isolation spectroscopists today utilize these as cryostats almost exclusively,[1] and they are widely used as the heart of cryopumps.[2] There have been a number of recent crystal manipulator designs based on the use of flowing cryogens.[3–7] Devices such as the CTI Model 350 refrigerator,[8] would be useful as an alternative to cooling crystal samples for surface science applications—the 350 provides 2 W of cooling power at 15 K (second stage) with 20 W of heat load at 77 K (first stage)—but they are not UHV compatible. Joints that are normally soft soldered can be replaced with silver-soldered joints,[9] but lead shot in the displacer as well as the piston assembly in the head which is not rated above 150 °F preclude the practice of a 150–200 °C bakeout. We have found, however, that with careful control of the refrigerator temperature during bakeout, these devices can remain within a vacuum system that is baked above 150 °C. This note describes the temperature sensing and control electronics we have employed for this purpose.

II. BAKEOUT CONTROL SYSTEM

As implemented in our system, the bakeout control system is configured in three subunits, as illustrated in Fig. 1:

(a) The *bakeout controller* houses the thermocouple amplifier, which senses the temperature of the refrigerator's second cold stage. An additional amplifier stage provides a convenient monitor voltage, and comparators establish under- and over-temperature set points.

(b) The *refrigerator pulser* responds at the first (lower) set point of the bakeout controller to activate a cooling cycle of the refrigerator.

(c) The *oven interlock* severs power to the bakeout ovens in the event of an over-temperature condition (second set point).

The heart of the bakeout controller (Fig. 2) is the AD595, a thermocouple amplifier with internal ice-point reference.[10] A particularly convenient feature of this amplifier is that the sensing thermocouple junction can be grounded—in the case of a differential amplifier, one would need to electrically isolate the junction while maintaining good thermal contact. The standard 10 mV/°C output of the AD595 is additionally

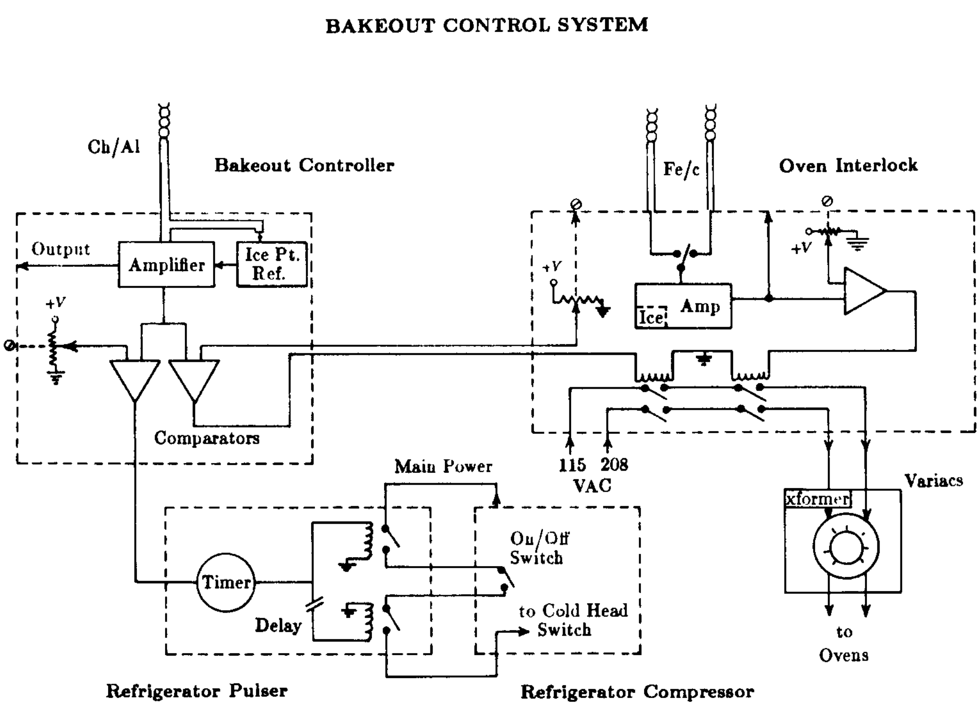

FIG. 1. Schematic of the bakeout controller system.

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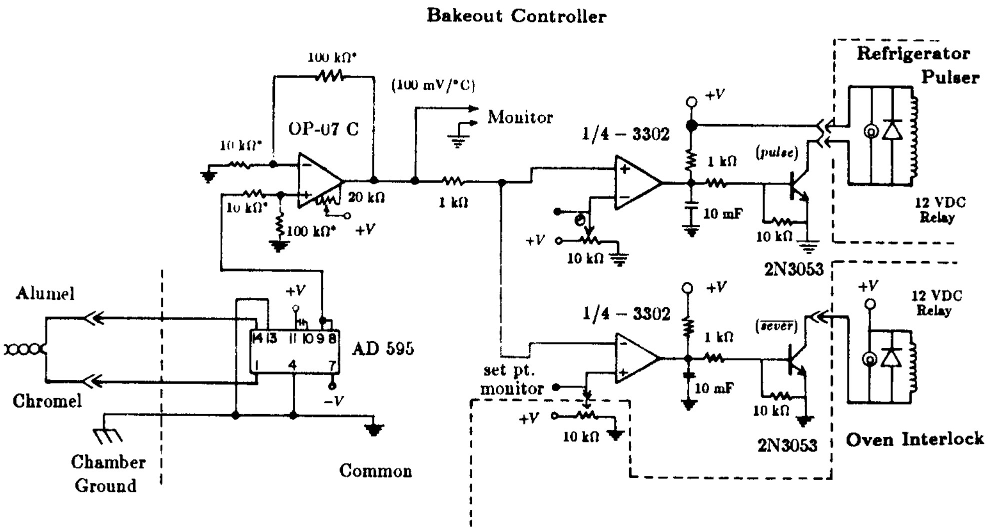

FIG. 2. Diagram of the bakeout controller.

amplified by a second state (× 10), providing a monitor output as well as input to the comparators. The comparators (LM321) provide the levels to open as close relays in the refrigerator pulser and oven interlock.

The refrigerator pulser (Fig. 3) provides automated control of both the refrigerator compressor unit and the cold head piston. A pair of 3-conductor 14-gauge cables are interposed behind the two switches on the front panel of the compressor unit. This is the only modification made to the original compressor unit and is accomplished so that the cover can still be conveniently removed. The signal from the bakeout controller, through a low-current relay, initiates a Cycl-Flex 0→10 min timer[11] which in turn activates the compressor. A time-delay relay allows the compressor to stabilize (~ 10 s) before providing power to the cold head piston. A manual override provides constant operation for crystal cooling purposes.

The oven interlock (not shown) provides power to the bakeout ovens through a latched relay. Should the refrigerator not remain sufficiently cool, the bakeout would be stopped. Provision could be made here for maintaining the bakeout temperature below a maximum value, but this is not a problem in our system.

III. OPERATION

A sample ion-gauge trace during a bakeout using this controller system is shown in Fig. 4. The gradual decrease in chamber pressure is modulated by the refrigerator cooling cycles. Gases desorb as the refrigerator surface warms to 35 °C, the first set point. Chamber pressures in the low 10^{-7} Torr range are achieved during bakeout, and titanium pumping after cooldown routinely results in pressures of 1–2×10^{-10} Torr. Refrigerator pulses of 5–7 min are required every 20–30 min during the 200 °C bakeouts. The piston assembly in the cold head is located outside the ovens, and remains relatively cool with the use of a fan.

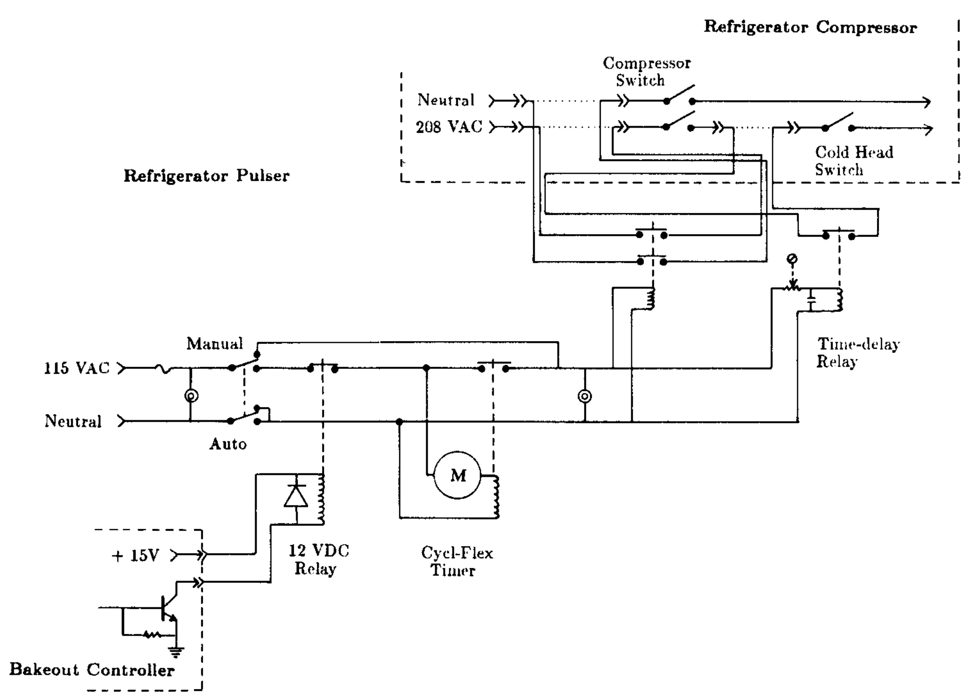

FIG. 3. Diagram of the refrigerator pulser.

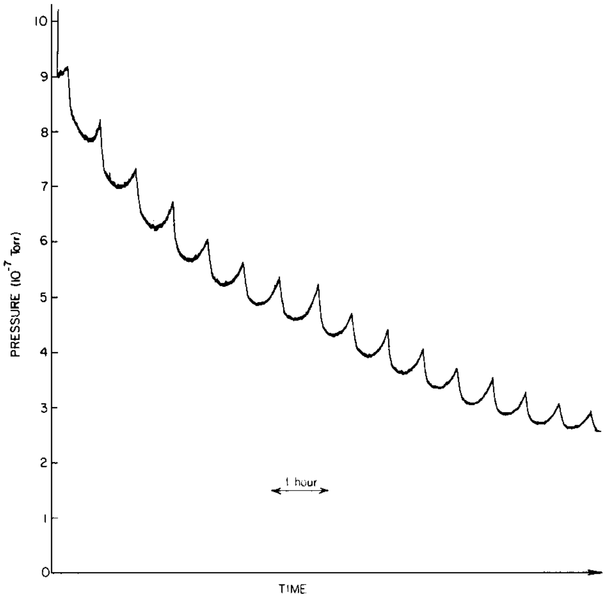

FIG. 4. Ion-gauge trace obtained during bakeout of UHV chamber. The chamber pressure is modulated by on–off cycles of the refrigerator.

Using the two-stage cooling provided by the CTI 350, we have achieved crystal temperatures as low as ~35 K (using a calibrated Chromel vs gold–5% iron thermocouple pair) with heat conduction through a high-grade (99.999% or 5N grade) flexible Cu braid,[12] to allow crystal rotation on a manipulator with 2.5 in. offset, and without extensive crystal shielding. Presumably the design of this low-temperature sample mount could be improved. We have also used a high-temperature mount, which is equipped to heat samples to 1500 K, and with this configuration the crystal cools to 120 K. In addition to providing the two cold stations, the refrigerator acts as an integral cryopump during operation. A multichannel array gas doser is therefore desirable so that samples may be preferentially adsorbed on the crystal sample rather than the refrigerator. The refrigerator being pulsed on–off during bakeout prevents H_2O and other condensibles from freezing onto the cold refrigerator surfaces and remaining in the chamber.

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[1] A. J. Barnes and H. E. Hallam, in *Vibrational Spectroscopy—Modern Trends*, edited by A. J. Barnes and W. J. Orville-Thomas (Elsevier, New York, 1977).
[2] M. Michaud and L. Sanche, J. Vac. Sci. Technol. **17**, 274 (1980); R. A. A. Kubiak, W. Y. Leong, R. M. King, and E. H. C. Parker, J. Vac. Sci. Technol. A **1**, 1872 (1983).
[3] J. A. Stroscio and W. Ho, Rev. Sci. Instrum. **55**, 1672 (1984).
[4] P. A. Thiel and J. W. Anderegg, Rev. Sci. Instrum. **55**, 1669 (1984).
[5] N. J. DiNardo, J. E. Demuth, W. A. Thompson, and P. G. Ledermann, Rev. Sci. Instrum. **55**, 1492 (1984).
[6] E. E. Chaban and Y. J. Chabal, Rev. Sci. Instrum. **54**, 1031 (1983).
[7] I. Arakawa and Y. Tuzi, Rev. Sci. Instrum. **55**, 617 (1984).
[8] CTI Cryogenics, Kelvin Park, Waltham, MA 02154; the same model refrigerator can be purchased mounted on a conflat flange from: Cryosystems, Inc., 190 Heatherdown Dr., Westerville, OH 43081.
[9] G. L. Fowler and J. A. Panitz, Rev. Sci. Instrum. **51**, 1730 (1980).
[10] Analog Device AD595 (pretrimmed for Chromel–Alumel) or AD594 (iron–constantan).
[11] Cycl-Flex HP54, Eagle Signal, 736 Federal St., Davenport, IA 52803.
[12] S. Samson, E. Goldish, and C. J. Dick, J. Appl. Crystallogr. **13**, 425 (1980).