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HIGHLY STABLE TEMPERATURE SYSTEM FOR ANGULAR CORRELATION EXPERIMENTS

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The design and construction of a highly stabilized variable-temperature system for Differential Perturbed Angular Correlation measurements is described. In the temperature range from room temperature up to 300 °C a stability of 0.001 °C is achieved for short periods (a few hours) and a stability of better than 0.01 °C for prolonged (typically 30 h) measurements. The temperature gradient over the sample is less than 0.001 °C.

1. Introduction

We describe here the design and performance of a relatively inexpensive, highly stabilized variable-temperature system for the above room-temperature region (up to about 300 °C). The present system was designed specifically for use in a Perturbed Angular Correlation[1]) experiment for the study of ferroelectric phase transitions[2]) but it can be easily adapted for different experimental setups.

The design of this system and its ultimate performance were accomplished as an optimum compromise between two sets of partly contradicting criteria. One set of criteria is determined by the conditions of the angular correlation experiment:

a) the short half-life (≈ 44 days) of the radioactive source used in the present experiment and the long measuring periods (≈ 2 days at each temperature) dictate the need for rapid approach to a preset temperature;

b) the requirement to minimize the attenuation of the radioactive radiation dictates the choice of thin, low atomic number material to be used in the construction of the system. These requirements lead to a system having a relatively small thermal inertia and a short thermal time-constant. This evidently opposes the conditions posed by the other set of criteria regarding the temperature characteristic of the system:

a) the necessity of maintaining the temperature of the sample constant within $\lesssim 0.01$ °C over the prolonged measuring periods;

b) minimizing the temperature gradient along the sample (≤ 0.01 °C). These requirements for homogeneous temperature both in time and space are essential for an accurate investigation of the highly temperature-sensitive ferroelectric properties of the sample in the close proximity of the critical temperature. To satisfy these requirements, a long thermal time-constant is necessary.

In the following we present a system which satisfies all of the above-listed requirements to a satisfactory degree. In section 2 the details of this system are described. The arguments leading to the determination of the various details are also outlined. In section 3 we describe the temperature control and measurement system. We discuss the various problems encountered in the design of this system and their effects on the stability and accuracy of the temperature measurements. Finally, in section 4, the performance data and characteristics of the system are presented.

2. Experimental apparatus

The constant temperature oven is described schematically in fig. 1. The outer cylindrical container (a) serves essentially as a vacuum chamber. A vacuum of less than 10^{-3} torr is maintained in order to minimize convection losses to the environment. The vertical radiation shields (c) minimize the heat radiation losses. The three stainless steel supporting tubes (b) of 1 mm diameter, 0.1 mm wall thickness and 8.5 cm length reduce the heat losses due to conduction while providing the necessary mechanical rigidity for positioning of the heater (f). The overall reduction of heat losses increases the thermal resistance between the stabilized temperature region and the outer environment, rendering the system's insensitivity to fluctuations in ambient temperature.

The vacuum chamber was made of 2.5 mm thick aluminium. The central part of the chamber was reduced by lathe to 1 mm to serve as the exit window (d) for the radioactive radiation. The resulting attenuation of the radiation for the two γ-

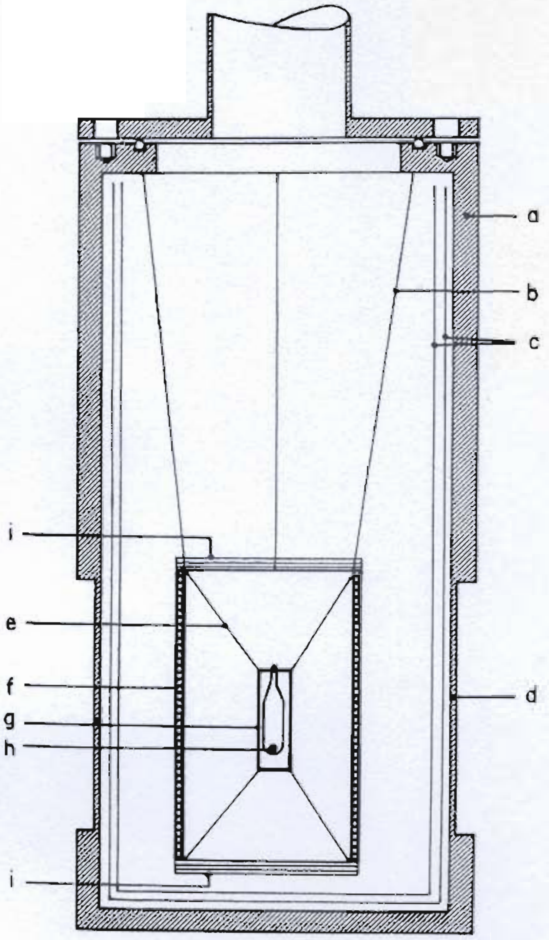

Fig. 1. Schematic of the furnace: (a) vacuum chamber, (b) suspending stainless steel tubes, (c) radiation shields, (d) radiation-exit window, (e) suspending Chromel wires, (f) heater, (g) sample holder, (h) radioactive sample, (i) radiation shields.

rays in the present experiment (130 and 480 KeV) is 4% and 2% respectively.

An improvement in temperature homogeneity can be obtained by increasing the dimensions of the chamber. However, a diameter of 10 cm was chosen on the basis of optimal distance between the sample and the photomultipliers. The height of the chamber was dictated by the length of the heater plus its supporting tubes.

The heater (f) is made of 40 mm diameter, 70 mm high and 0.2 mm thick aluminium tube. This choice of the material and wall thickness was made to minimize the heat capacity of the heater to enable a rapid response to controlled changes of its temperature. Furthermore, this choice also minimizes the attenuation of the radioactive radiation. The diameter of the heater was chosen to be one half of the vacuum chamber diameter. This provides the temperature gradient compensation along the heater via multiple reflections of heat radiation between the heater and the vertical radiation shields. Moreover, this also ensures that each point of the sample holder (g) views a large enough portion of the heater, which is essential for obtaining a good temperature homogeneity along the sample holder.

On the outer circumference of the heater, 15 m of 5 Ω/m Nichrom resistance wire were wound uniformly and bifilarly upon a thin layer of Aremco Cermabond 503. This alumina bond serves both as electric insulation and thermal contact. A second thick layer of alumina was applied upon the heater wire. Owing to the thickness of this layer, most of the heat produced is radiated inwards, towards the sample. Multilayer horizontal radiation shields (i) are fitted next to the top and bottom of the heater cylinder, so that the sample holder is heated by reflections from these sides. This serves mainly to improve the temperature homogeneity along the sample holder.

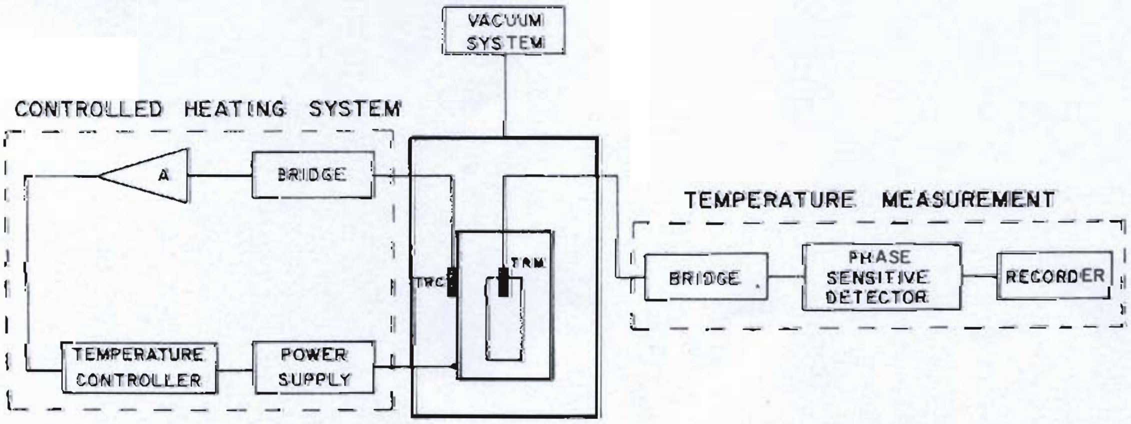

Fig. 2. Schematic of the electronic system. TRM is the sample temperature measuring thermistor. TRC is the thermistor controlling the temperature of the heater.

The sample holder (g) is made of 10 mm diameter, 25 mm long and 0.2 mm thick aluminium tube. Here again the material and dimensions were determined by arguments similar to those explained in the preceding paragraph. To reduce as much as possible the heat conduction between the sample holder and the heater, the sample holder is suspended and held in position by means of four 0.075 mm thick Chromel. wires (e) of low thermal conductivity. The thermal contact between the sample holder and the radioactive sample (h) (in powder form, enclosed in a thin-walled quartz-glass ampoule) is provided by silicone grease filling out entirely the space between the ampule and the sample holder.

3. The control and temperature measurement circuitry

The schematic diagram of the temperature control and measurement units is shown in fig. 2. The temperature control sensor, a Fenwal Electronics Iso-Curve GA51MM322, is glued upon the outer alumina layer on the heater. This thermistor is part of a standard d.c. bridge providing the feedback voltage controlling the power supplied to the heater. The response of the temperature controller is determined both by the magnitude of the feedback voltage and by its rate of change. The sample temperature measuring element is a miniature Iso-Curve GA51JM71, inserted in the grease filling the sample holder, thereby reducing the effect of self-heating. This sensor is fitted with four very thin leads (0.001 mm diameter) to reduce a degradation in its performance due to the effect of self-cooling, thus improving both the relative as well as the absolute reading of temperature. The resistance of this sensor was measured by an improved ac bridge[3]) to eliminate thermal emf effects which may cause significant errors.

4. Discussion and performance

The temperature gradient over the heater was established using differential thermocouples and was found to be less than 0.07 °C/cm. This gives rise to a corresponding, but much smaller, gradient along the sample holder. (Averaging is achieved, because each point of holder absorbs heat via radiation from a large portion of the heater). In the steady state, however, this gradient is balanced by the heat conduction current along the sample holder. The gradient along the sample is even more reduced owing to the temperature homogenizing action of the silicon grease. Thus the overall temperature difference along the sample is less than 0.001 °C.

The fluctuations of the sample temperature were measured and found to be less than 0.001 °C for a few hours and less than 0.01 °C for prolonged periods. Typical temperature-versus-time data are shown in the insert of fig. 3. A 15 °C change of the ambient temperature results in a corresponding change of 0.01 °C of the sample temperature. Hence, it may be concluded that the main cause for the instability of the temperature is due to the temperature vulnerability of the electronic control system.

The thermal inertia of the system can be deduced from the observed rate of approach of the

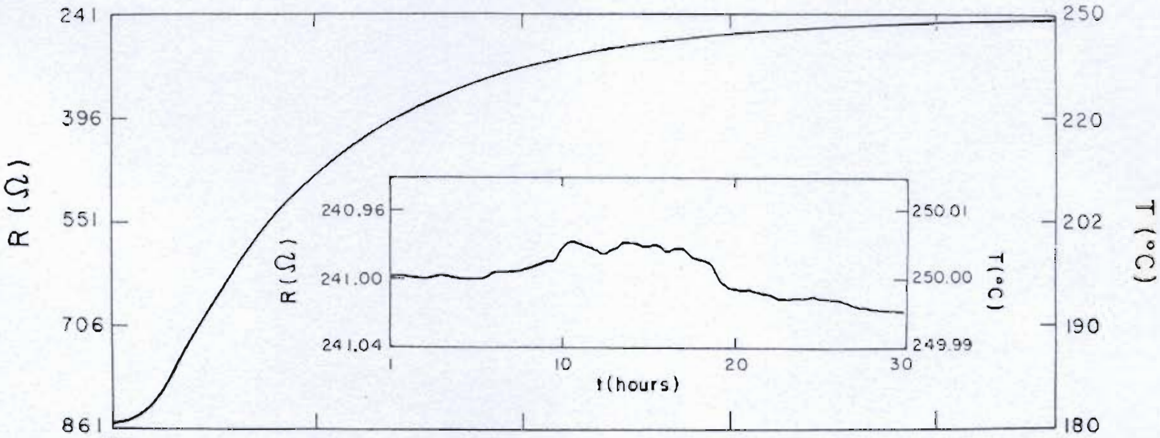

Fig. 3. The time-response of the system for a 70 °C implied change of temperature. The insert shows typical temperature-versus-time variations during prolonged measuring time.

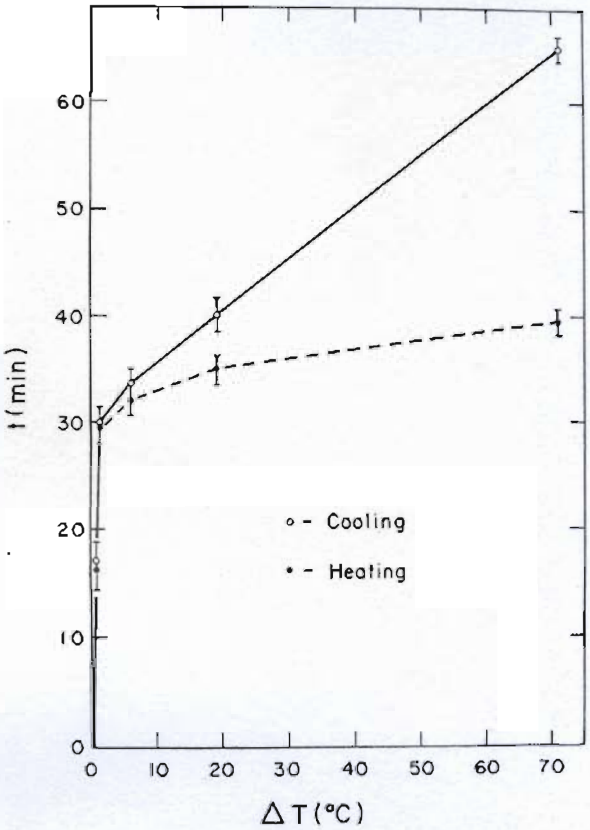

Fig. 4. Temperature relaxation times of the sample (see text) as a function of the temperature change.

sample temperature to a preset value. A typical example of the systems time-response to an implied temperature change of 70 °C is shown in fig. 3. In this case, the time required to achieve the preset temperature to within 0.01 °C ("relaxation time") is about 40 min. Fig. 4 shows the "relaxation time" as a function of the implied change of temperature ΔT, both for heating and for cooling.

It should be noted that the sample follows the heater temperature via the heat radiation process. The rate of heating of the sample depends on the difference between the temperatures of the sample and the heater. Hence, owing to the rapid warm-up of the heater, the rate of heating of the sample is large at the beginning, slowing down when the temperature difference decreases. The corresponding process of cooling-down is slower because of the slow heat loss rate of the heater via radiation. This explains the shorter relaxation times for heating. Typical relaxation times are shown in fig. 4. As indicated by this figure, the main part of the heating relaxation time is due to the time required to overcome the final ~2 °C temperature difference, this being of the order of 30 min. This explains the change of the slope of the corresponding curve. On the other hand, the cooling curve in fig. 4 exhibits the slower heat exchange process as explained above.

5. Conclusion

The apparatus described here offers a number of characteristics which are necessary for critical phase transition experiments, namely: high temperature stability; highly homogeneous sample temperature; relatively short temperature relaxation times; negligible attenuation of the radioactive radiation and convenient sample handling. The system can be easily adapted for a wide range of different experiments and experimental conditions.

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