

Relaxation calorimetry technique for measuring low temperature specific heat

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A rudimentary calorimeter was constructed to measure the temperature dependence of the specific heat of a 10 μ thick (0.62 mg) copolymer film of vinylidene fluoride and trifluoroethylene in the previously unexplored vicinity of 6 K. The data were processed with the algorithm developed by Hwang *et al.* [Rev. Sci. Instrum. **68**, 94 (1997)] as modified for our configuration in which a Cernox thermometer also served as a heater. The measurements extended from 3.75 to 8.5 K, with a temperature resolution of 0.2 K, and the ratios of the statistical uncertainties to the mean values deduced for c_p are $\pm 3\%$ over most of this temperature range. The data taken with the intermediate resolution of this calorimeter provides evidence for the absence of any slowly varying anomaly in the temperature dependence of c_p . Such rudimentary systems provide a useful option to gather exploratory information at a comparatively low cost for components. © 2004 American Institute of Physics. [DOI: 10.1063/1.1633989]

I. INTRODUCTION

Design improvements over recent years enable some calorimeters to precisely measure the specific heat of very small samples at low temperatures.^{1–7} Sophisticated material processing techniques have, for example, reduced background contributions from addenda by several orders of magnitude, in systems which typically use ac or semiadiabatic relaxation modes for heating.^{2–7} Unfortunately, the implementation of such advanced technologies requires use of special equipment that is often expensive and not readily available. Restrictions due to cost and limited access, have historically motivated users with limited resources to develop simplified systems with intermediate resolution, that can nevertheless provide useful data.^{7–9} Information surveyed by such systems may also facilitate efficient use of more sensitive calorimeters.

The rudimentary calorimeter described here, was developed in response to the limited objective of making a low cost measurement of the temperature dependence of the specific heat of a 10 μ thick copolymer film in the vicinity of 6 K, to search for an anomaly that might be related to that observed in the pyroelectric coefficient.^{10–12}

Several alternative options^{8,9} were examined before a decision was made to construct a simplified calorimeter that utilizes the algorithm recently developed by Hwang *et al.*¹³ It utilizes a realistic model of heat flow with an algorithm that fits the transient thermal response of a calorimeter with basis functions derived from an integral solution to the heat equation. An appropriate calorimeter, with a Cernox thermometer that also served as a heater, was easily constructed at a low cost. Preliminary tests also indicated that the analytical procedures in the Hwang algorithm (HA) are robust. For example, numerical simulations and subsequent measurements

confirmed that the nonlinear temperature dependence of our heater power did not degrade the output.

We were also motivated to use the HA¹³ in order to learn more about the operational characteristics of its potentially useful methodology for processing calorimetry data. Although a recent review¹⁴ thoroughly evaluates the output performance of a commercial product¹⁵ which utilizes this algorithm, that report contains little information about details of the design parameters and their operational impact. The data in the temperature range of our measurements are comparable with the accuracy reported for the commercial device at liquid helium temperatures.^{14,15} There are, however, several significant differences in design which may be useful in resolving the bounds for useful operation of the algorithm. For example, the thermal time constant of our system was several orders of magnitude larger, and the data sampling rates were correspondingly smaller,¹⁶ than in the commercial system. In addition, our Cernox thermometer was also used as a heater.

The HA assumes that the specific heat and the thermal conductance are constant over the integration windows used to process the data. It is therefore not surprising that the recent evaluation of the commercial implementation of the algorithm reported a degraded sensitivity to the rapid change in specific heat that is associated with a first-order phase transition.¹⁴ Such changes are fueled by latent heat requirements, whose presence is often indicated by hysteresis in the measured specific heat, depending upon whether the transitions are traversed while heating or cooling the sample.^{14,17,18} The data from our copolymer sample show no evidence for such hysteresis in the vicinity of 6 K. In addition, the anomaly in the temperature dependence of the pyroelectric coefficient which motivated this study, exhibits a discontinuity only in the first derivative of its temperature dependence.

In contrast to high resolution systems that have negligible background from addenda, our calorimeter exhibits a

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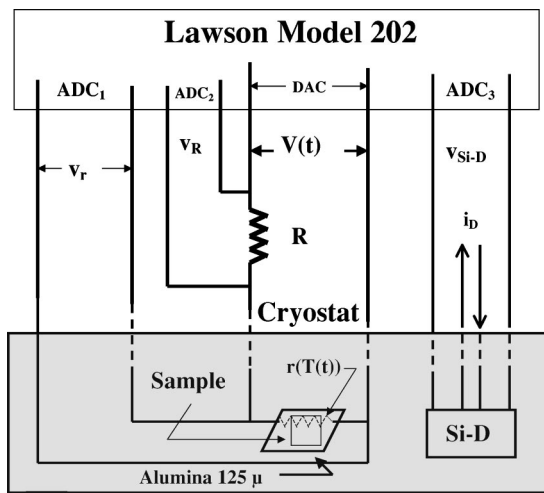


FIG. 1. A schematic of the data acquisition¹⁶ and cryostat sensor configurations. The shaded box encompasses the relevant components of the cryostat. The time-dependent temperature of the Cernox thermometer and its alumina support platform were deduced by four-wire measurements of its resistance, $r[T(t)]$. Readings from the silicon diode were used only to monitor the temperature stability of the cryostat housing and were not used for analysis of the data.

contribution from addenda that is comparable to that from the sample. The statistical errors associated with subtraction of addenda background for our system, were reduced to acceptable levels by averaging the data over many runs. The comparable contributions from addenda and sample had an unexpected advantage in our configuration, because the background subtraction process removed the sensitivity of results to the location of the data analysis window relative to occurrence of the heater pulse. This sensitivity is apparently due to power developed in the long leads,¹⁹ which were used to create large thermal time constants to take advantage of the reduced noise when the data acquisition system was operated at low sampling rates.

The results obtained with our simplified system are noteworthy because the temperature dependence of the specific heat of the copolymer sample was measured well enough to show a dominant cubic dependence, with no indication of any anomaly in the vicinity of 6 K. The cost for calorimeter components was comparatively low, and the information obtained should be useful in setting priorities for future measurements.

II. MEASUREMENT AND CALIBRATION PROCEDURES

A. Sample and apparatus

A minute layer of either N-Apiezon or silicon vacuum grease was used to attach the sample to one side of a 125 μ thick wafer of alumina. The oblong shaped wafer was approximately 4 mm wide and ~ 6.2 mm long. A resistive thermometer, which also served as a heater, was attached to the other side of the wafer, with a thin layer of GE 7031 varnish.

The thermometer was a thin film, Cernox-1050, resistor mounted on a bare sapphire chip, whose mass was ~ 3 mg.²⁰ As indicated in Fig. 1, two of the four leads to the Cernox thermometer were used to monitor the voltage across it. The other two leads were used to connect the resistance of the

thermometer $r[T(t)]$ in series with a large load resistor R of ~ 33 k Ω , which was kept at room temperature and shielded in a metallic enclosure. The resistance of the thermometer at liquid helium temperatures is approximately 3 k Ω . The load resistance was carefully measured before and after each complete set of data runs. Records of the voltage across it could therefore be used to deduce the current flowing through the thermometer $r[T(t)]$.

The calibrated thermometer resistance $r[T(t)]$ was used to deduce the temporal dependence of the temperature via a calibration curve represented by a continuous least-squares fit to the calibration data for the thermometer. The master reference was provided by a calibrated rhodium-iron resistive thermometer obtained from Lake Shore.²⁰ The Cernox thermometer was then calibrated in its operational environment via use of helium exchange gas to establish thermal equilibrium between it and a reference thermometer thermally grounded to the interior housing of the cryostat. The accuracy of this calibration was checked at two points by the *in situ* response of the thermometer to thermal transients at the boiling point and the lambda point of liquid helium under standard conditions.

Two pairs of coiled (0.064 mm diameter) manganin wire, were soldered to each of the short (~ 6 mm long, 0.064 mm diameter) copper leads attached to the Cernox film. Each of these four coils were approximately one meter long, in order to achieve the desired thermal time constants. The coils were counter wound in order to minimize their inductance. The bottom ends of the coils were thermally grounded on the inside of the copper cylinder which housed the wafer assembly comprising the calorimeter. A silicon diode thermometer was also mounted on the inside of this copper housing, in order to provide a reference check on the temperature stability of the thermal ground (i.e., bath temperature). The temperature measurements from the silicon diode were only used to independently check the thermal stability of the housing for the calorimeter. Temperature readings used in data analyses were deduced from the calibrated resistance of the Cernox thermometer. The top ends of the four coiled leads used to monitor the resistance of the Cernox thermometer, also provided the primary support for the alumina wafer that supported the sample. These leads were mechanically stabilized with nylon strands that were 25 μ in diameter.

The entire assembly for the calorimeter was suspended within a stainless steel cylinder whose inside diameter was ~ 24 mm. This cylinder was evacuated in order to remove all traces of helium gas, which would otherwise provide a thermal short to the calorimeter. Therefore it was pumped to a pressure of less than 10^{-5} mbar for at least 24 h while the calorimeter was at room temperature. Pumping was continued throughout all subsequent measurements at low temperature. The calorimeter was cooled by submerging it in liquid helium contained in a glass dewar with a liquid nitrogen jacket. This dewar system performed well enough to facilitate automated collection of data on a 24 h basis. It also provided a stable thermal environment, with drifts as low as ~ 10 mK per hour at ~ 6 K.

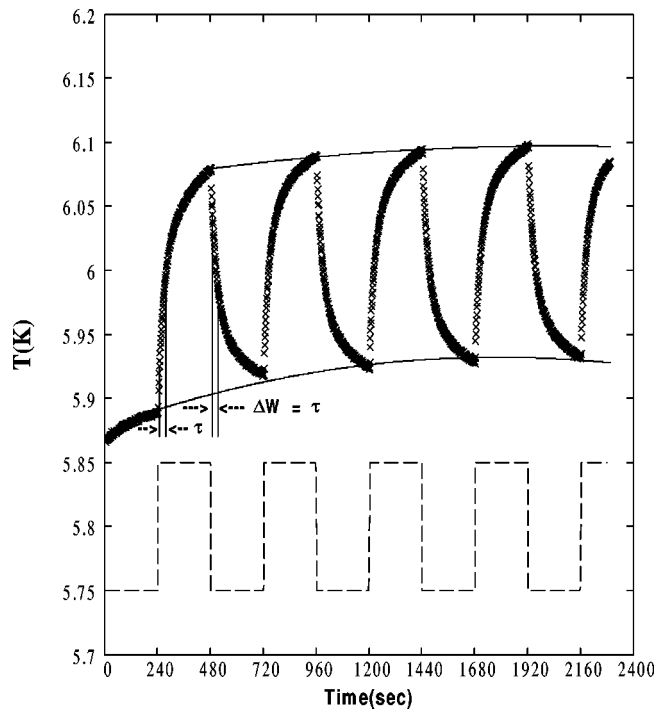


FIG. 2. The temperature response of the Cernox thermometer/heater to a sequence of square-wave driver voltages, indicated by the dashed line. The solid curves provide estimates of the drift in the background temperature. The pairs of vertical lines show the positions and width of the temporal windows ΔW used to select data for analyses. The thermal time constant, τ , is approximately 28 s in this temperature range.

B. Measurements, analyses, and calibrations

A pivotally important aspect of the specific heat measurements presented here, is the ability to accurately monitor temporal changes in the temperature. Thermal transients were induced in the sample by applying a voltage $V(t)$ directly across the series combination of the load resistor R and thermometer resistor, $r[T(t)]$, as shown in Fig. 1. The driver voltage $V(t)$ depicted by the dashed lines in Fig. 2, was generated with a digital-to-analog (DAC) converter in a Lawson Model 202 data acquisition system.¹⁶ The overall noise level was $\sim \pm 1$ mK, when quiescent temperature readings at ~ 6 K were sampled at 30 Hz. Analog representations of driver wave forms were created with a personal computer, and stored in digitized data files used to drive the output of the DAC. A brief survey confirmed that square-wave driver voltages yielded the best signal-to-noise ratio for the measured temperature response. The square voltage pulses for the heater always had a dc bias level which determined the quiescent temperature of the thermometer and alumina support platform.

Separate analogue-to-digital (ADC) channels in the Lawson unit were used to monitor voltages across the Cernox thermometer and the load resistor connected in series with it. These data were processed at selected settings of t_0 , the temporal offset from changes in the heater voltage, and ΔW , the width of the acceptance window used to select the data for analysis. Two representative windows for data analysis are indicated by the pairs of vertical lines in Fig. 2. Here, $\Delta W = \tau$, where τ is the thermal time constant. The average value of τ , for all rising and falling temperature profiles in

Fig. 2, is ~ 29 s. The corresponding changes in the temperature and power of the thermometer/heater system, averaged over the data selection windows of all the thermal peaks shown in Fig. 2, are respectively, 64 mK, and 2.19×10^{-9} W.

The data displayed in Fig. 2 were taken with a copolymer sample attached to the substrate.¹⁰ Data analyses were always restricted to records of the temporal changes in temperature and power within the selection windows. The selected data were then fit to a fourth-order polynomial in order to avoid the biased assumption that they might be adequately represented by a pure exponential.

The solid curves in Fig. 2, represent least-squares fits through ten data points at the extremal of the temperature response pulses. These curves provide a quantitative estimate of the drift in the background temperature of the thermometer. A similar procedure was used to estimate the background drift in the power. These corrections were usually very small compared to the primary signal.

The essence of the modified HA used for this work, is presented in the following simplified example. Consider the equation for energy conservation when power $P(t)$ is applied to a substrate with a specific heat c . The substrate is also assumed to have a thermal leak of conductivity λ to a thermal bath at constant temperature T_B :

$$P[T(t)] = c \frac{dT(t)}{dt} + \lambda [T(t) - T_B]. \quad (1)$$

Since this must hold at any *initial* time t_0 the resulting equation may be subtracted from the original formulation at time t , which eliminates the bath temperature T_B . When the results of this subtraction are integrated over time intervals small enough that the temperature dependence of c and λ may be neglected, then one obtains the following relation:

$$T(t) - T(t_0) = \frac{1}{c} \int_{t_0}^t \{P[T(t)] - P(t_0)\} dt, \\ - \frac{\lambda}{c} \int_{t_0}^t [T(t) - T(t_0)] dt. \quad (2)$$

The algorithm treats the two integrals on the right side of this equation as basis functions, whose multiplicative factors are adjusted via standard least-squares procedures to provide a fit to the measured temperature profile, as represented on the left-hand side. These multiplicative factors contain the desired parameters in the form $1/c$ and λ/c . In our analysis, the offset time t_0 equals the delay from any step change in the driver voltage, depicted in Fig. 2, to the onset of the analysis window ΔW .

Prior to the formulation of the HA, an integral solution of the heat equation, in the form of Eq. (2), was the basis of an alternative algorithm which iteratively extracted the specific heat over analysis intervals in which the temperature dependence of λ was not assumed to be constant.⁸ It is a semiadiabatic approach which is apparently sound,¹⁷ but is more cumbersome than the HA.

Although our use of the thermometer as a heater has the advantage of reducing the addenda, this configuration also

has the disadvantage of producing a nonlinear temperature dependence in the power delivered to the heater/thermometer:

$$P[T(t)] = \frac{V(t)^2}{(R + r[T(t)])^2} r[T(t)], \quad (3)$$

where the parameters are defined in Eq. (1) and Fig. 1.

A numerical simulation was developed to test the robustness of the HA when it is revised to accommodate this nonlinear temperature dependence of delivered power. Our test was to generate a solution for $T(t)$ in Eq. (1), using reasonably selected values for c and λ . This was then used in Eq. (2) to test the accuracy of the HA. Numerical solutions to Eq. (1) were readily obtained via the Bulirsch-Stoer method for stiff systems of differential equations.²¹ The numerically extracted values for c and λ usually agreed with the assigned values to better than four significant places. These results were insensitive to the shape of the driver voltage pulse, and to the choices of t_0 and ΔW . This agreement also held when the simplified formulation in Eq. (2) was extended to include a thermal link between the sample and the substrate.¹³

C. Validation measurements of the specific heat of copper

The specific heat of copper was measured as an additional test of the revised analysis algorithm, and of our rudimentary calorimeter, and the results were compared with the 1972 reference data published by Holste, Cetas, and Swenson.²² The copper sample weighed 0.0228 g, and was rated as oxygen-free, high-conductivity (OFHC). It was lightly etched in nitric acid and then washed in hydrochloric acid and deionized water, as described in Ref. 23, before placing it in the calorimeter. It is of interest to note that specific heat measurements of copper by Buchanan²³ are within $\sim 1\%$ of those by Holste, Cetas, and Swenson, from 4 to 9 K, which is the temperature range of interest here. The sample was then attached to the alumina substrate with a minute layer of Apiezon-N grease, because the residual layer of silicon grease did not provide adequate adhesion. The addenda measurements did not include this additional layer of Apiezon-N grease, but a subsequent correction based on its weight, was made by using data for its specific heat.^{24,25} This correction was negligible over most of the measured temperatures. The comparatively recent measurements by Swenson²⁵ show significant differences below ~ 4 K, in specific heats for bulk compared to thin films of Apiezon-N grease. The new data are, however, reported²⁵ to be consistent with earlier results²⁴ over the temperature range of interest here.

Data from the first phase of the measurements are presented in Fig. 3. for the copper sample plus addenda, and for the addenda alone. Data are shown for two sets of the parameters t_0 and ΔW , which, respectively, represent the temporal offset and width of the selection windows. The thermal time constant varied from approximately 13 to 25 s over the measured range of temperatures from 3.7 to 8.5 K. The widths of ΔW were automatically adjusted during the data

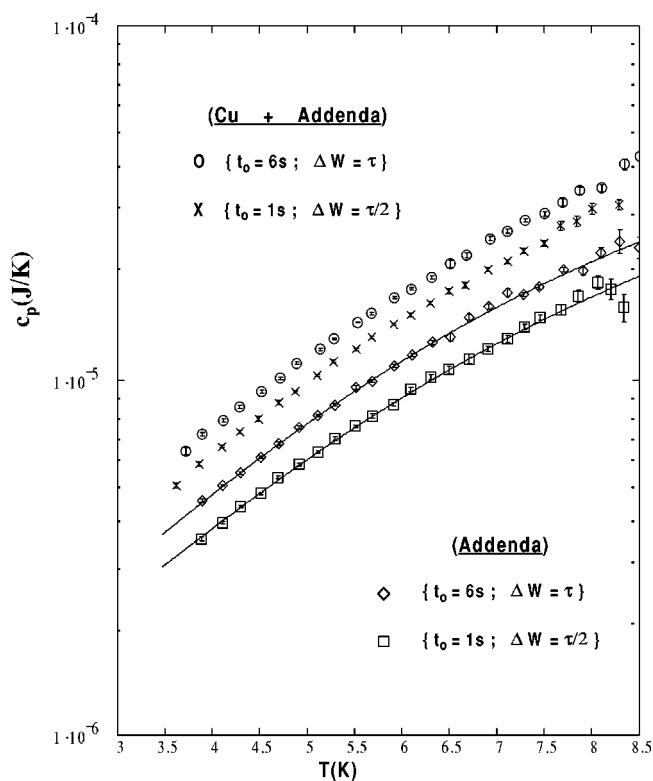


FIG. 3. Data for the specific heat of a copper sample plus addenda, and for the addenda alone, which were processed for two settings of the temporal offset t_0 and width ΔW of the analysis windows. The solid curves through the addenda data represent least-squares fits to a fourth-order polynomial, and the vertical bars denote the $\pm \sigma$ range of statistical errors about the mean.

reduction process to correspond to a specified fraction of a thermal time constant over the entire temperature range of the measurements.

The temperature dependence of τ was routinely established by fitting a smooth curve through survey data for the time constant, gleaned from least-squares fits of the thermal response curves to a pure exponential function. The results were invariably a smooth curve with a slowly varying temperature dependence, that was consistent with subsequent averages for the ratio of c/λ deduced from final output of the analysis algorithm.

Note that the temporal dependence of all processed data, for temperature as well as power, were generated by least-squares fits to a fourth-order polynomial over the range of the data selection windows. Such fits to the data were superior to what could be attained with a pure exponential function.

The data plotted in Fig. 3, show that the specific heat is systematically larger for data sets with larger values for either ΔW or t_0 . These differences are emphasized in that figure by pairing the smallest value of the offset (i.e., $t_0 = 1$ s), with a 50% reduction in ΔW . The resultant shifts in the raw values deduced for the specific heat are probably due to power dissipation in the long manganin leads,¹⁹ which is estimated to be less than $\sim 10\%$ of that produced in the thermometer at 4 K. The processed data in Fig. 4 show that a simple subtraction of the addenda measurements provides an adequate correction for our system. This is probably a con-

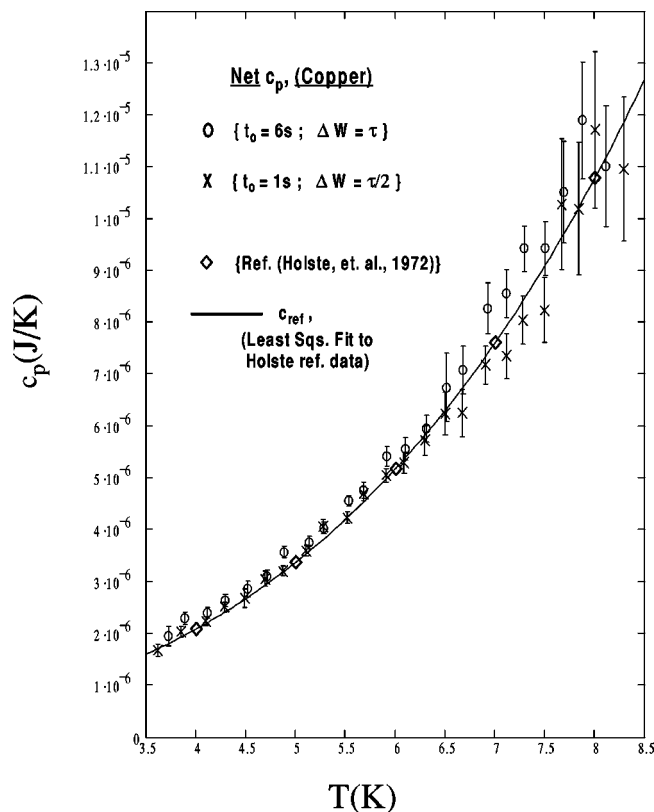


FIG. 4. The net specific heat for a 0.0228 g copper sample, compared with reference measurements by Holste *et al.*²² Our data are shown for two settings of the temporal offset t_0 and width ΔW of the analysis windows. The vertical bars denote the $\pm \sigma$ range of statistical errors about the mean.

sequence of the fact that the specific heat for the composite configuration is comparable to that for the addenda alone. It is therefore plausible that effects due to the flow of heat produced in the leads would be comparable, and therefore cancel to first order, when the data for the addenda are subtracted from that for the composite system.

The self consistency of the addenda correction procedure is indicated by the overlap of both sets of data for the net specific heat in Fig. 4. The temperature dependence exhibited by our data, from 3 to 9 K, is in good agreement with the least-squares fit of a fourth order polynomial to the reference data from Holste, Cetas, and Swenson.²²

A more systematic comparison of the deviations of our specific heat measurements for copper compared with those from the reference source,²² is presented in Fig. 5. Although our data for both sets of these analysis options exhibit comparable scatter, the smaller deviations from the reference line occur for the option in which the selected data were closer to the onset of the heater pulse. This indicates that the corrections which the addenda subtractions provide for the systematic shifts exhibited in Fig. 3, becomes less accurate with increasing elapsed time from the onset of the heater pulse. That is consistent with our previous conjecture that the shifts of the data for the two options in Fig. 3 are due to the flow of power generated in the leads. All subsequent analyses use data processed from selection windows defined by parameters associated with the smaller deviations, although that choice does not significantly affect the final results.

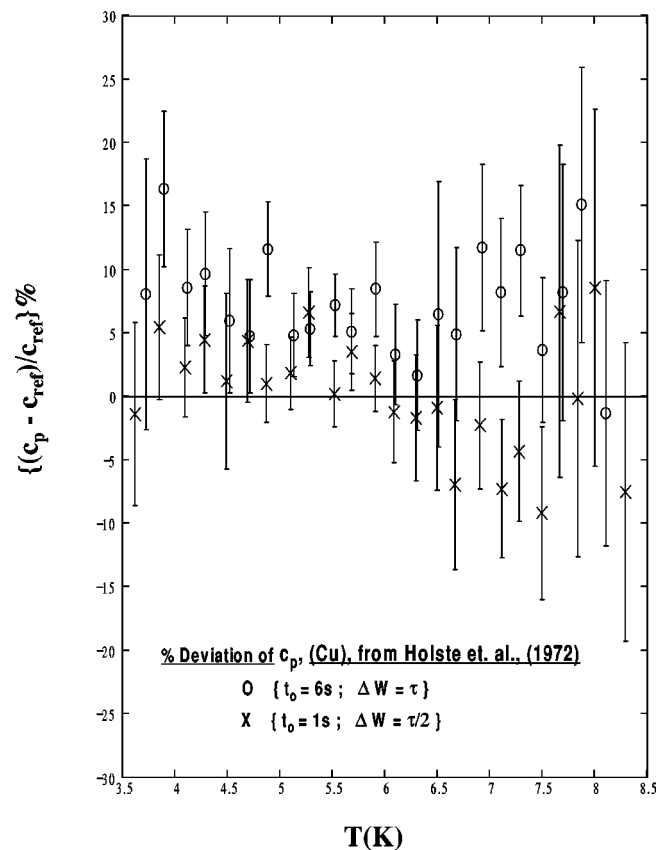


FIG. 5. A comparison of the fractional percent deviation of our measurements for an OFHC copper sample with the 1972 reference measurements of Holste *et al.*²² The plots show our measurements for two settings of the data analysis window. The vertical bars denote the $\pm \sigma$ range of statistical errors about the mean.

Figure 5 shows that the percentage deviations of the averages of our best set of measurements are within approximately $\pm 6\%$ of the accepted reference. That is significantly larger than the $\sim \pm 1\%$ deviations customarily reported for such measurements, but it is deemed acceptable for the intermediate resolution of the present study. This scatter is indicative of the absolute accuracy of our measurements of the specific heat of copper. There are, however, no reference comparisons for our copolymer measurements, whose statistical uncertainty is $\pm 3\%$ over most of the measured range of temperatures. The latter estimate is an indicator of the relative accuracy of c_p for the copolymer as a function of temperature, which was the primary objective of our measurements.

A thorough analysis of the thermal response of a sample on a substrate should in principle include the effect of a nonideal thermal link between the two. This typically results in an additional, comparatively fast time constant during the initial phase of the thermal response function. This second time constant is known as the τ_2 effect,²⁶ and it is resolved in the original presentation of the HA.¹³ We elected not to use it because our sampling rate was not high enough to consistently yield convergent solutions. The simplified version appeared adequate because our values for the specific heat of copper are consistent with temperature dependence of the reference measurements.²² Limited use of the extended ver-

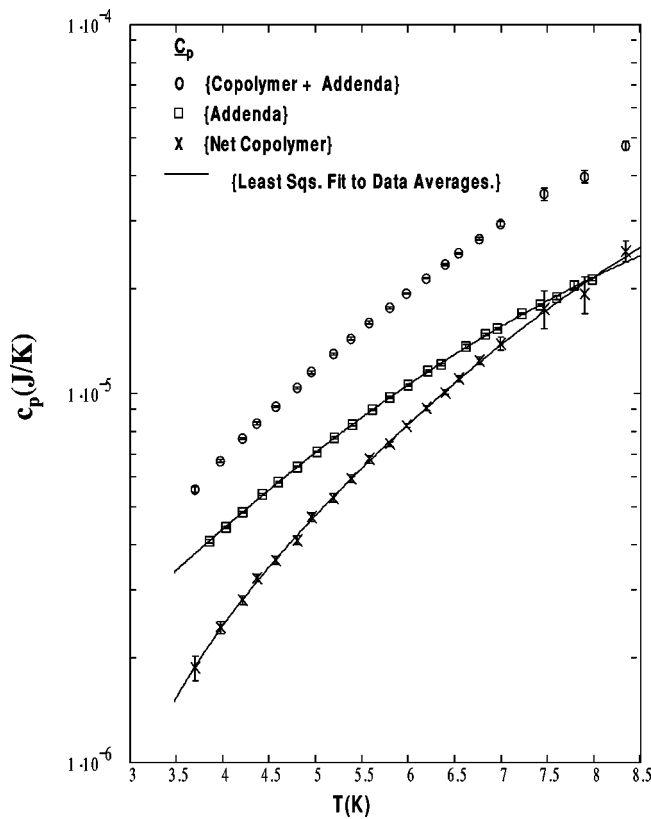


FIG. 6. A comparison of the measured components used to deduce the specific heat of a 0.62 mg sample of a copolymer of vinylidene fluoride and trifluoroethylene.¹⁰ The plots show the contributions from the sample plus the addenda, and the addenda, and the difference of the two. The vertical bars denote the $\pm \sigma$ range of statistical errors about the mean.

sion also indicated that the thermal conductance between our sample and the substrate was typically five times or more greater than between the substrate and the bath, which gave additional justification for our use of the simplified version.

In order to obtain the satisfactory signal-to-noise ratios indicated by the vertical error bars in Figs. 3 and 4, data runs were accumulated for ~ 96 h and then averaged. The vertical error bars, which bracket the mean values plotted in Figs. 3 and 4, represent only statistical uncertainties deduced from the data, and do not include systematic errors. Two possible sources of such errors involve the temperature calibration and the weight of the sample.

The temperature calibration was rechecked after completion of all the measurements presented here. This was performed with the Cernox thermometer mounted on the alumina backing in the same configuration used to make all the reported measurements. The leads from the thermometer were thermally grounded on the inside of the copper housing for the calorimeter. Rapid thermal equilibrium was facilitated by a small amount of helium exchange gas. Two independent thermal markers were provided by the boiling point of liquid helium under standard conditions, at ~ 4.2 K, and the lambda point of superfluid helium at ~ 2.172 K. The results of the recheck showed that our previous calibration was offset by 200 mK over the temperature range of interest. This correction was applied to all the data presented here. This first-order correction for scale effects, discussed in Refs. 14 and

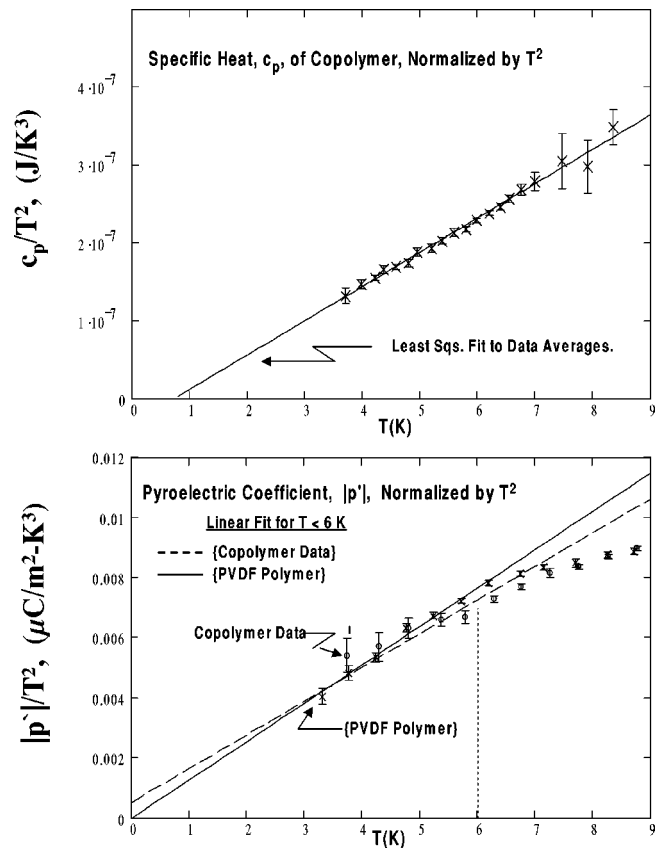


FIG. 7. Plots with a T^2 normalization, of the specific heat and the pyroelectric coefficient^{10,11} for a copolymer sample of vinylidene fluoride and trifluoroethylene. The pyroelectric coefficient was also measured for a pure polymer sample of polyvinylidene fluoride (PVDF).¹²

22, appears adequate for the intermediate resolution of our measurements.

The other source of systematic errors is in the weight of the sample. The mechanical and electronic balances used for weighing the samples, had a stated accuracy of ten micrograms, but deviations in results separated over intervals of several months, indicated that inaccuracies ranged up to 100 micrograms. Estimates of the impacts of the estimated uncertainties in weight and temperature, indicate that they are both within the stated uncertainties.

D. Measurements of the specific heat of the copolymer sample

The principal components of data from our measurements of the specific heat of the copolymer sample,¹⁰ which motivated this study, are summarized in Fig. 6. These data were processed with the analysis parameters t_0 and ΔW , respectively set at 1 and τ seconds. The statistical uncertainties, represented by the barely discernible vertical error bars, are less than $\sim \pm 3\%$ of the corresponding average values over most of the measured range of temperatures. These percent errors are small enough so that subtraction of addenda contributions do not cause excessive errors in the net results for the sample, in spite of their comparable strengths. The large symbols in Fig. 6 represent data averages over temperature intervals of 200 mK. The interval was doubled for temperatures above 7 K due to poorer statistics for those data.

The graphs in Fig. 7 compare plots of the net specific heat¹⁰ with previous measurements of the pyroelectric coefficient, after these data are normalized by T^2 . A straight line gives an excellent fit to the specific heat data over the entire range of measured temperatures, with no indication of any anomaly similar to that exhibited by the pyroelectric coefficient in the vicinity of 6 K. Possible implications of this difference are discussed in Ref. 10.

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