

# The $3\omega$ technique for measuring dynamic specific heat and thermal conductivity of a liquid or solid

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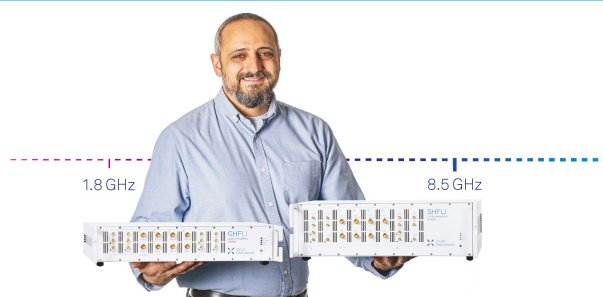
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# The $3\omega$ technique for measuring dynamic specific heat and thermal conductivity of a liquid or solid

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We show how to measure dynamic specific heat and thermal conductivity of a solid or liquid sample using the  $3\omega$  technique, which is an ac-modulation method where we use a heater simultaneously as the sensor. By varying the width of the heater relative to the thermal decay length, one can choose the proper regime to measure thermal conductivity or specific heat. The technique is applied to window glass and the results confirm the validity of the method. Experimental results for potassium dihydrogen phosphate crystal demonstrate the first-order transition at the Curie point, and the dynamic specific heat of supercooled liquid potassium–calcium nitrate is shown. © 1996 American Institute of Physics. [S0034-6748(96)03301-8]

## I. INTRODUCTION

Measurements of thermal properties, specific heat, and thermal conductivity of condensed matter constitute one of the major characterizations of physical properties of condensed matter. Measurement of thermal conductivity of a solid, for example, offers a very nice way to investigate the elementary excitations, which participate as heat carriers or limit their mean free path in a given system. It also allows one to study mechanisms, such as defects in a crystal, which limit the mean free path of the heat carrying excitations. On the other hand, specific heat measurement allows one to directly monitor the free energy change of a given system as the external parameter such as temperature varies. This unique ability of specific heat measurements follows from the fact that specific heat per unit volume  $C_p$  is defined thermodynamically as

$$C_p = \frac{1}{V} \frac{dH}{dT} = \frac{T}{V} \frac{dS}{dT}, \quad (1)$$

where  $T$ ,  $H$ ,  $S$ ,  $V$  are the temperature, the enthalpy, the entropy, and the volume of a given system, respectively.

In addition to the above, more traditional usage of the thermal measurements, one can use the specific heat measurements as a probe to the dynamics of a system, since specific heat is expressed as the enthalpy fluctuation of the system from the statistical mechanical point of view. Using the linear response theory,<sup>1</sup> it is fairly straightforward to define the dynamic, i.e., frequency-dependent specific heat  $C_p(\omega)$  in terms of the time correlation function of the enthalpy of the system, namely,

$$C_p(\omega) = C_p^0 + \frac{i\omega}{k_B T^2 V} \int_0^\infty dt e^{i\omega t} \langle \delta H(0) \delta H(t) \rangle, \quad (2)$$

where  $k_B$  is the Boltzmann constant,  $C_p^0$  is the usual static specific heat, and  $\delta H(t) = H(t) - \langle H \rangle$  is the enthalpy fluctuation of the system. Although it is not common to actually observe the frequency-dependent specific heat, there are experimental situations where these dynamic specific heats were indeed measured. For example, Smith<sup>2</sup> observed the frequency-dependent specific heat in germanium and Smith and Holland<sup>3</sup> discussed it in terms of a delay in equilibrium. Frequency-dependent specific heat was also measured near the glass transition of glycerol<sup>4</sup> and potassium–calcium nitrate mixture.<sup>5,6</sup>

We have adopted the third harmonic detection method, an ac modulation method using the heater as a sensor simultaneously. The third harmonic detection method was originally discovered by Corbino long ago<sup>7</sup> and exploited extensively by Smith *et al.*<sup>8</sup> More recently Birge and Nagel<sup>4</sup> and Jung *et al.*<sup>5</sup> used a *planar* heater in applying the same method to probe the slow dynamics associated with the glass transition, while Cahill *et al.*<sup>9</sup> utilized a thin *line* heater to measure thermal conductivity of solids. In this article we shall call the calorimetric technique based on the third harmonic detection method a  $3\omega$  technique. We report our theoretical and experimental investigations of the  $3\omega$  technique applied to the general heat diffusion problem from a heater of arbitrary width. We show how the two regimes of planar and line types follow from the more general case and present experimental examples to display the validity and power of the technique.

## II. THEORETICAL BASIS OF THE $3\omega$ TECHNIQUE

In this section we lay down the theoretical basis of the  $3\omega$  method. We solve the problem for diffusion of heat into a medium from an oscillating heater located on the surface of the medium. We show that one can measure the specific heat and thermal conductivity of the medium, since the amplitude of the temperature oscillation of the heater itself contains the thermal information of the medium.

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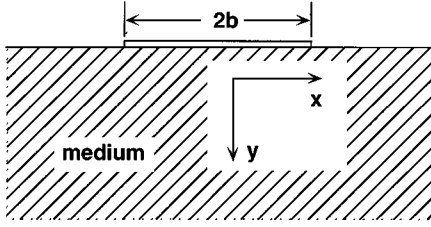


FIG. 1. Geometry of the heater and the medium. The medium is considered semi-infinite, and the heater of width  $2b$  is regarded as a superposition of infinitely thin line heaters located between  $x = -b$  and  $b$ . The heater is assumed to have a negligible mass and consequently no effect on the temperature profile of the medium.

### A. Heat diffusion into a semi-infinite medium

Let us suppose that an infinitely long heater with a width  $2b$  is placed on the surface of a semi-infinite medium as shown in Fig. 1, and it generates an oscillating power per unit length,  $P_0 \exp(i2\omega t)$ .<sup>10</sup> Using the result for an infinitely thin heater due to Carslaw and Jaeger<sup>11</sup> and remembering that the heater of width  $2b$  can be regarded as a superposition of such thin heaters located between  $-b$  and  $b$ ,<sup>9</sup> we arrive at the expression for the complex amplitude of the temperature oscillation at the surface:

$$\delta T(x, y=0) = \frac{P_0}{\pi \kappa} \int_0^\infty \frac{\cos(kx) \sin(kb)}{kb(k^2 + q^2)^{1/2}} dk, \quad (3)$$

where  $\kappa$  is the thermal conductivity and  $q$  is the complex thermal wave number defined as

$$q = \sqrt{\frac{i2\omega C_p}{\kappa}}. \quad (4)$$

It is noted here that the thermal oscillation decays exponentially in the  $y$  direction with a characteristic length, defined by various authors as the diffusion length,  $\lambda = |q|^{-1} = \sqrt{\kappa/2\omega C_p} = \sqrt{D/2\pi/\sqrt{2f}}$ , in which  $D$  is the thermal diffusivity. Because we shall use the heater as a sensor simultaneously (see Sec. III A), we calculate the temperature variation of the heater itself,  $\delta T_h$ , by taking the average of Eq. (3) from  $-b$  to  $b$ , that is,

$$\begin{aligned} \delta T_h &= \left( \frac{1}{2b} \right) \int_{-b}^b \delta T(x, y=0) dx \\ &= \frac{P_0}{\pi \kappa} \int_0^\infty \frac{\sin^2(kb)}{(kb)^2(k^2 + q^2)^{1/2}} dk. \end{aligned} \quad (5)$$

Since the analytic expression of Eq. (5) is not known, one has to resort to the numerical calculation to find out the frequency dependence of  $\delta T_h$ . As an example we chose the medium to be window glass. Using the thermal diffusivity,  $D = 0.3 \text{ mm}^2/\text{s}$ , typical for window glass, we obtain  $\sqrt{D/2\pi} = 0.22 \text{ mm s}^{-1/2}$ , and  $\lambda(\mu\text{m}) = 220 \mu\text{m s}^{-1/2}/\sqrt{2f}$  where  $f = \omega/2\pi$ . Figure 2 is the calculated results for the heater of width  $2b = 440 \mu\text{m}$ . (This width was chosen somewhat arbitrarily for the sake of calculation.) Figure 2(a) shows that the magnitude  $|\delta T_h|$  (in units of  $P/\pi\kappa$ ) is linearly proportional to  $\log 2f$  below  $0.01 \text{ Hz}$ , i.e.,  $\lambda > 10b$ . On the other hand, when  $\lambda < b/10$ ,  $\log |\delta T_h|$  is proportional to  $\log 2f$  with the

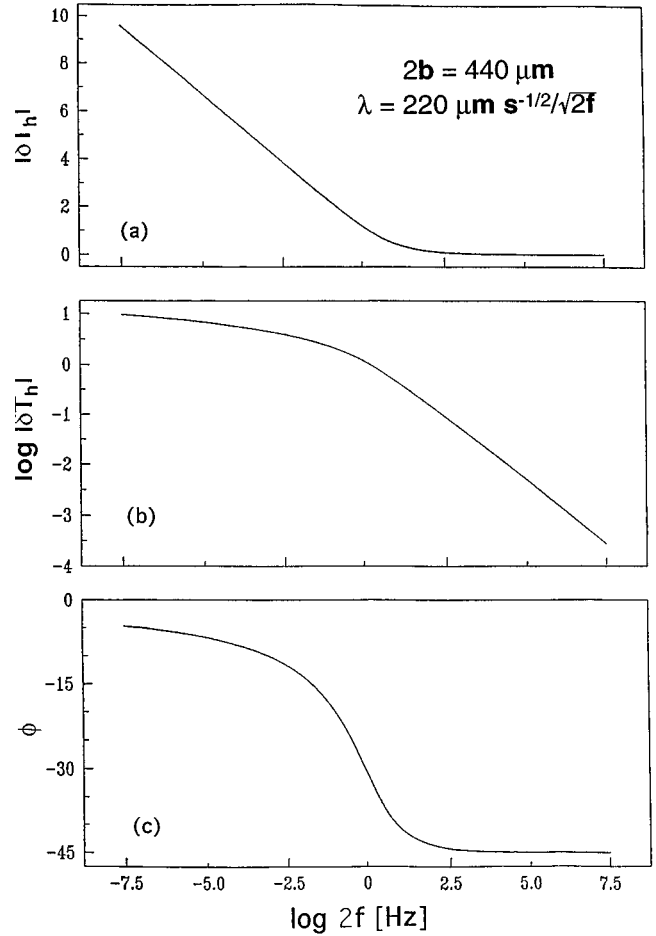


FIG. 2. The numerically calculated results for the heater of width  $2b = 440 \mu\text{m}$ . The thermal decay length was taken to be  $\lambda(\mu\text{m}) = 220 \mu\text{m s}^{-1/2}/\sqrt{2f}$ , which is a typical value for window glass. Magnitude  $|\delta T_h|$  of the heater oscillation is given in units of  $P/\pi\kappa$ , while the phase is in degrees.

slope of  $-1/2$  as is illustrated in Fig. 2(b). Figure 2(c) displays the phase as a function of frequency. It should be noted that the phase has a constant value of  $-45$  deg at high frequencies.

It is easy to see how these numerical results follow from Eq. (5). If  $b \ll \lambda$ , the integral can be carried out by first setting  $\sin(kb)/(kb) = 1$ . The result of integration is cast as

$$\begin{aligned} \delta T_h &= \frac{P_0}{\pi \kappa} \left[ -\ln \left( \frac{i2\omega b^2}{D} \right)^{1/2} + \eta \right] \\ &= -\frac{P_0}{2\pi \kappa} \ln 2\omega - \frac{P_0}{2\pi \kappa} \ln \left( b^2 \frac{C_p}{\kappa} \right) \\ &\quad -i \frac{P_0}{4\kappa} + \frac{\eta P_0}{\pi \kappa}, \end{aligned} \quad (6)$$

where  $\eta$  is a constant having the value of  $0.922\dots$ . Note that  $\delta T_h$ , more accurately its real part,  $\text{Re}[\delta T_h]$ , is indeed linearly proportional to the logarithm of the measuring frequency. Since the coefficient of this linear term is inversely proportional to the thermal conductivity, one can obtain the thermal conductivity from the slope if one measures  $\text{Re}[\delta T_h]$  as a function of frequency in this regime. Thus it

offers a way to measure thermal conductivity of a medium with a relatively small temperature gradient by taking advantage of the well-established power of the ac modulation technique.

For the opposite case of  $b \gg \lambda$ , one can use a mathematical identity for the Dirac delta function<sup>12</sup>

$$\lim_{b \rightarrow \infty} \frac{1}{\pi b} \left[ \frac{\sin bx}{\sin x} \right]^2 = \delta(x) \quad (7)$$

to obtain from Eq. (5),

$$\delta T_h = \frac{P_0/2b}{\kappa q} = \frac{P_0/2b}{\sqrt{2\omega C_p \kappa}} e^{-i\pi/4}, \quad (8)$$

where  $P_0/2b$  is now the power per unit area (or one can easily solve the one-dimensional heat diffusion problem to obtain the same result). By working in this regime, one can measure the product of specific heat and thermal conductivity,  $C_p \kappa$ , of the medium. Therefore, in principle by changing the measuring frequency to cover both regimes, one can obtain the thermal conductivity as well as the specific heat of the medium separately. In addition, if the specific heat becomes frequency dependent, the magnitude and phase of  $\delta T_h$  would follow Eq. (8) with the complex, instead of real,  $C_p(2\omega)$  as defined in Eq. (2). Thus the dynamic specific heat can be obtained by measuring  $\delta T_h$  as a function of frequency.

### B. Heat diffusion into both sides

When one deals with a liquid sample, one needs to evaporate a heater on a thick glass substrate. In this case the thickness of the substrate (like a solid sample) should be much greater than  $\lambda$  to avoid boundary effects from the back. A liquid sample is then put on top of the surface of the substrate. Now that there exist two different media on opposite sides of the heater, i.e., a glass substrate and a liquid sample, we have to extend the previous results. For the planar heater, it is straightforward, since the symmetry of the boundary matches that of the one-dimensional heat diffusion, to show that Eq. (8) becomes

$$\delta T_h = \frac{P_0/2b}{\sqrt{2\omega C_{pg} \kappa_g} + \sqrt{2\omega C_{pl} \kappa_l}} e^{-i\pi/4}, \quad (9)$$

where the subscripts  $g$  and  $l$  stand for glass and liquid, respectively.

In case of a line heater, on the other hand, it may not be possible to solve the heat diffusion equation exactly. However, if we neglect the boundary mismatch,<sup>13</sup> the problem can be written as a combination of two separate heat diffusions and, by defining  $F(x) \equiv -\ln x + \eta$ ,  $\delta T_h$  is given as

$$\delta T_h = \frac{P_0}{\pi(\kappa_g + \kappa_l)} F(q_l b) \times \left[ \frac{1}{1 + [\kappa_g/(\kappa_l + \kappa_g)](F(q_l b)/F(q_g b) - 1)} \right], \quad (10)$$

where  $q_l$  and  $q_g$  denote the complex wave numbers for the liquid and the glass, respectively. Since the quantity in the

bracket is very slowly varying with temperature, one can still obtain the sum of thermal conductivities from the slope of  $\delta T_h$  versus  $\log 2f$  (see Sec. IV). Thus, the glass contribution plays the role of background in both cases. To obtain the background information, one must carry out the measurements for the empty cell before taking data with samples.

## III. EXPERIMENT

To implement above ideas into experimental reality, a method must be devised to detect the temperature variation of the heater itself. Also it becomes necessary to vary the width of the heater to cover the necessary range of  $b/\lambda$ , since the dynamic range needed to cover both regimes for a heater with a given width is far greater (14 decades) than is available experimentally. Finally, the experimental situation is expected to be different from the one where the heat diffusion equation was solved. For example, we assumed that the metallic heater on top of the sample would not affect the temperature profile in the sample. Also the length of the heater will be finite instead of infinite as assumed. The ultimate justification of these assumptions can only be made through experimental test. The first and second points are dealt with in this section, while the last point is discussed in Sec. IV.

### A. Third harmonic detection

In order to measure the temperature oscillation of a heater itself we use the aforementioned third harmonic detection method, which is briefly described here.<sup>8</sup> If one drives the heater with a current at frequency  $\omega$ ,  $I(t) = I_0 \cos \omega t$ , then one gets Joule heating at frequency  $2\omega$  at the heater<sup>14</sup> and thus the temperature of the heater oscillates at the same frequency with the complex amplitude  $\delta T_h$  as given in the previous section. Since the heater is made of a metal such as gold or silver, its temperature coefficient of resistivity,  $\alpha \equiv (1/R)(dR/dT)$ , is not zero and as a result its resistance also oscillates at frequency  $2\omega$ . Thus,

$$R(t) = R_0[1 + \alpha |\delta T_h| \cos(2\omega t + \phi)], \quad (11)$$

where  $\phi$  represents the phase shift of  $\delta T_h$  with respect to the power oscillation.

Due to the driving current  $I(t)$  through the heater, the voltage across the heater appears as

$$\begin{aligned} V(t) &= I(t)R(t) = I_0 \cos \omega t \cdot R_0[1 + \alpha |\delta T_h| \cos(2\omega t + \phi)] \\ &= I_0 R_0 \cos \omega t + \frac{1}{2} I_0 R_0 \alpha |\delta T_h| \cos(\omega t + \phi) \\ &\quad + \frac{1}{2} I_0 R_0 \alpha |\delta T_h| \cos(3\omega t + \phi). \end{aligned} \quad (12)$$

It is the second and third terms that contain the thermal information of the sample we want to measure. Note, however, that they are relatively small compared to the first term since  $\alpha \approx 0.004 \text{ K}^{-1}$  and  $|\delta T_h|$  is usually kept less than 20 mK to stay in the linear regime.

However, the last term, while small, appears at the third harmonic of the driving frequency and one can take advantage of this fact to measure the magnitude and the phase of the small signal in the presence of much larger unwanted signal using the Wheatstone bridge. The actual experimental





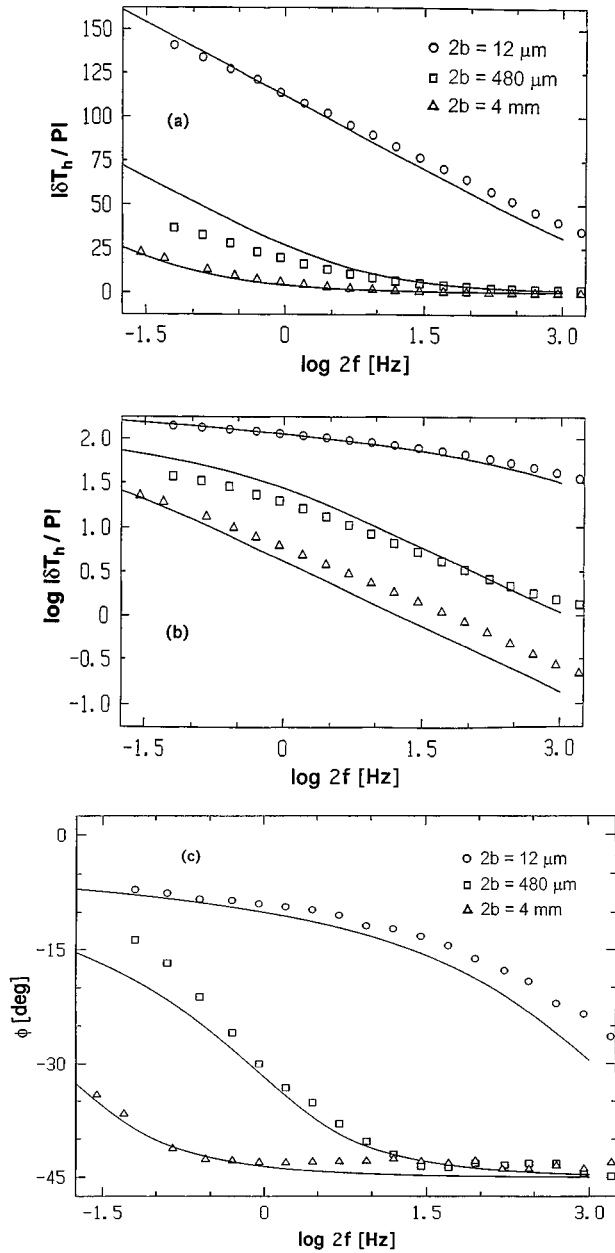


FIG. 5. Amplitude of temperature oscillation,  $\delta T_h$ , of the heaters of various widths on thick window glass vs the logarithm of measuring frequency  $2f$ : (a)  $|\delta T_h/P|$  vs  $\log 2f$ , (b) logarithm of  $|\delta T_h/P|$  vs  $\log 2f$ , (c) the absolute phase of  $\delta T_h$  vs  $\log 2f$ . The widths ( $=2b$ ) of the heaters are indicated in the figure. In (a) and (b),  $|\delta T_h/P|$  (in units mK and mW, respectively) is plotted since the power level was changed as the measuring frequency varied.

## B. Results for a solid: KDP

$\text{KH}_2\text{PO}_4$  (KDP) crystals were grown from an aqueous solution kept at  $35^\circ\text{C}$ . The samples used in the measurements had typical dimensions of  $10 \times 10 \times 10$  mm. The thickness was much larger than the thermal decay length,  $\lambda$ , in the frequency range used in the experiment and this fact ensured that the boundary did not perturb the thermal diffusion. Figure 6 is the plot of the thermal conductivity  $\kappa$  of KDP as a function of temperature measured by the  $3\omega$  method with a line heater deposited on the (010) surface. The width of the line heater was  $80 \mu\text{m}$  and measuring frequencies were be-

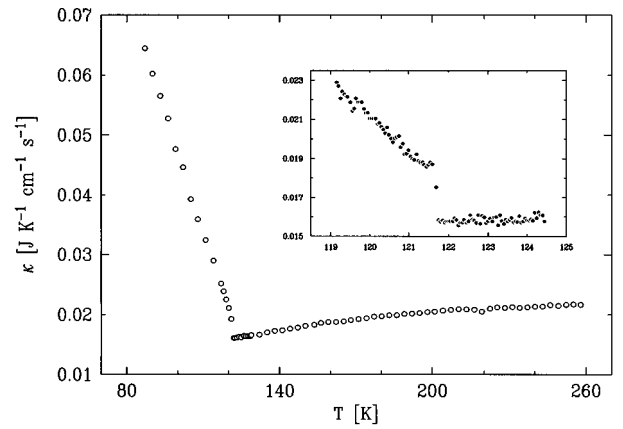


FIG. 6. The thermal conductivity of KDP as a function of temperature measured by the  $3\omega$  method.  $\kappa$  of KDP displays a discontinuity at the Curie point  $T_c$  as the inset illustrates.

low 12 Hz.  $\kappa$  was obtained from the slope in the graph of  $\text{Re}[\delta T_h]$  versus logarithm of measuring frequency at each temperature. In the ferroelectric phase,  $\kappa$  decreases sharply with increasing temperature, while in the paraelectric phase  $\kappa$  increases slowly as the temperature is raised. The inset displays a small but abrupt jump in  $\kappa$  at the Curie point ( $T_c$ ) 居里点 indicating the first-order nature of the transition. The detailed behavior of the thermal conductivity of KDP and the related physics are discussed in Ref. 15. Measurements with a planar heater were also performed and the data from the planar configuration were used to calculate  $C_p\kappa$  according to Eq. (8).  $C_p\kappa$  so obtained was divided by  $\kappa$  to yield the specific heat,  $C_p$ . Figure 7 is the plot of  $C_p\kappa$  and  $C_p$  of KDP measured at 0.2 Hz of temperature oscillation.  $C_p$  shows a familiar peak associated with the ferroelectric phase transition and this result agrees fairly well with the previous measurements.<sup>16</sup>

## C. Results for a liquid: CKN

$(\text{Ca}(\text{NO}_3)_2)_{0.4}(\text{KNO}_3)_{0.6}$  (CKN) is an ionic liquid which can be easily supercooled. When a liquid is supercooled without crystallization, a slow relaxation phenomenon occurs and the dynamic specific heat can be observed.<sup>4,6</sup> We used

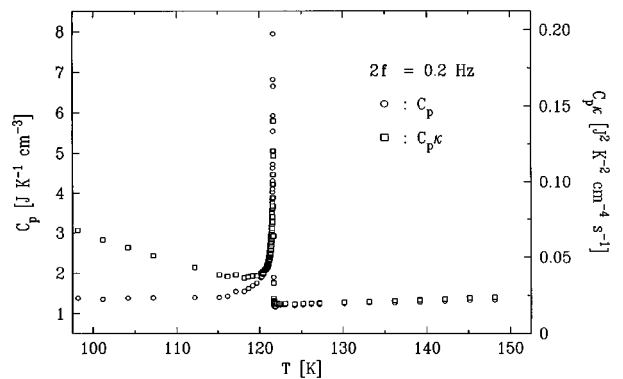


FIG. 7.  $C_p\kappa$  (square) and  $C_p$  (circle) of KDP as a function of temperature measured at 0.2 Hz (twice heating-current frequency) by the  $3\omega$  method with a planar heater. The decrease of  $C_p\kappa$  in the ferroelectric phase, as  $T$  increases, is due to the decrease of  $\kappa$ .

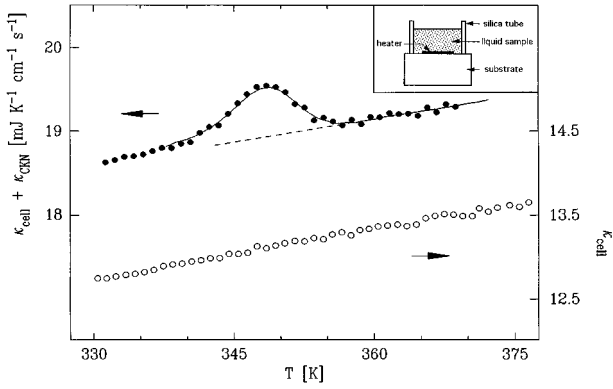


FIG. 8. Thermal conductivity of the cell,  $\kappa_{\text{cell}}$ , and the total conductivity,  $\kappa_{\text{cell}} + \kappa_{\text{CKN}}$ , obtained from the slope of  $\text{Re}[\delta T_h]$  of the line heater against  $\log 2f$  are plotted as a function of temperature. The peak in  $\kappa_{\text{cell}} + \kappa_{\text{CKN}}$  is solely due to the frequency dependence of  $C_p$  of CKN, while the linear part is due to the cell contribution. The solid line represents the calculated values according to Eq. (10) assuming that the thermal conductivity of CKN,  $\kappa_{\text{CKN}}$ , is constant. Thus,  $\kappa_{\text{CKN}}$  is essentially constant and the true total conductivity is indicated by the broken line. Inset shows the liquid sample cell.

the  $3\omega$  method to measure the thermal conductivity and the dynamic specific heat in the supercooled state of CKN. Since the sample is liquid, we evaporated a heater on a glass substrate and put a short silica glass tube with the diameter slightly larger than the size of the heater as shown in the inset of Fig. 8. A small amount of CKN was put into the tube and melted.

Figure 8 displays the thermal conductivity data of the empty sample cell and CKN as a function of temperature. They were obtained from the slope of  $\text{Re}[\delta T_h]$  against  $\log 2f$  for the empty and liquid-filled cell with a line heater of width  $60 \mu\text{m}$  according to Eqs. (6) and (10). While  $\kappa_{\text{cell}}$  shows a linear behavior in temperature,  $\kappa_{\text{CKN}}$  displays a peculiar peak around 350 K. This behavior can be understood if we take into consideration the frequency dependence of  $C_p$  of CKN. When  $C_p$  is a frequency-independent, real quantity at high and low temperatures, the term in the bracket of Eq. (10) shows little frequency dependence and its effect on the coefficient of  $\log 2f$  is negligible ( $\sim 0.05\%$ ). Thus, the slope gives rise to correct, total thermal conductivity of the cell and the sample. However, when  $C_p$  of CKN becomes frequency dependent, the coefficient of  $\log 2f$  obtained from the blind linear fitting of the  $\delta T_h$  data yields an erroneous value for the thermal conductivity and one has to use the full expression of Eq. (10). In other words, the frequency dependence of  $C_p$ , which in turn makes the term in the bracket of Eq. (10) frequency dependent, does not allow the simple interpretation. If one insists the linear relationship between  $\text{Re}[\delta T_h]$  and  $\log 2f$ , then the coefficient is no longer inversely proportional to thermal conductivity.

To account quantitatively for the peak around 350 K, we calculated  $\text{Re}[\delta T_h]$ , as a function of frequency, according to Eq. (10) by assuming  $\kappa$  of CKN is constant and using the  $C_p(2\omega)$  values of CKN in Fig. 9. Then we obtained the coefficient of  $\log 2f$  by linear fitting the calculated  $\text{Re}[\delta T_h]$  values against  $\log 2f$ . The solid line of Fig. 8 represents the results and it is in full agreement with the data. This fact

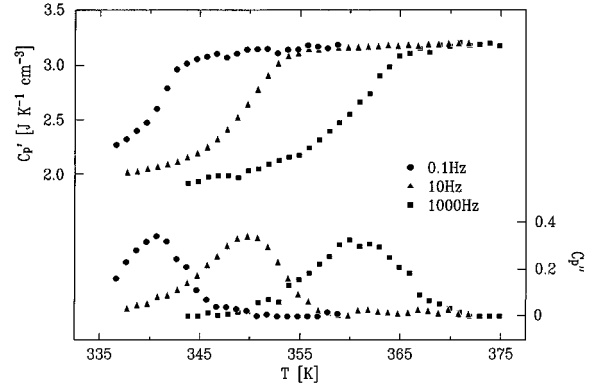


FIG. 9. Real and imaginary parts of the dynamic specific heat of CKN at various frequencies are displayed as a function of temperature. The frequencies indicated in the figure are those of the temperature oscillation, i.e., twice the heating frequencies. The frequency-dependent behaviors of  $C_p$  of CKN are those seen in typical relaxations.

clearly demonstrates that  $\kappa$  of CKN is essentially temperature independent.

Figure 9 shows the dynamic specific heat,  $C_p(2\omega)$ , of CKN as a function of temperature at various frequencies. These values of  $C_p(2\omega)$  were obtained by dividing the planar heater data,  $C_p(2\omega)\kappa$ , with the constant value of  $\kappa$  of CKN from Fig. 8.  $C_p(2\omega)$  shows a typical relaxation behavior of a supercooled liquid as is often seen in dielectric measurements. In other words, the real part shows dispersion and the imaginary part a peak as a result of the characteristic time of the system matching the probing time (=inverse frequency). The physics related to this behavior is discussed elsewhere.<sup>6</sup>

## ACKNOWLEDGMENTS

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<sup>10</sup>The angular frequency  $2\omega$  is due to the fact that a current at  $\omega$  through the heater produces Joule heating at  $2\omega$ . We take  $\exp(i2\omega t)$ , unlike Ref. 5, as a time-varying factor following Ref. 11.

<sup>11</sup>H. S. Carslaw and J. C. Jaeger, *Conduction of Heat in Solids* (Clarendon, Oxford, 1959), p. 193.

<sup>12</sup>G. Arfken, *Mathematical Methods for Physicists* (Academic, Orlando, 1985), p. 488.

<sup>13</sup>This is, of course, a crude approximation neglecting the heat transfer between the two media. In an experimental situation dealing with a liquid, however, this approximation is not as bad as it may appear. For example, the thermal decay lengths of substrate glass and potassium–calcium nitrate liquid in the last example of Sec. IV differ by only 20%.

<sup>14</sup>There also exists, due to the dc component of power, a constant shift of

the sample temperature with respect to the bath temperature, but it can be easily calibrated away.

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