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Study of thermal conductivity close to the melting point

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Abstract. Methods of measuring the thermal conductivity of materials in solid and liquid state at high temperatures are reviewed. Factors influencing the accuracy of the obtained results are examined. Special attention is paid to the role of contact resistance. Results for the thermal conductivity of sodium chloride, bismuth germanate, and germanium over a wide temperature range are presented. The behaviour of thermal conductivity during melting of these materials is analysed.

1 Introduction

For modeling of many technological processes and, in particular, for simulation of crystal growth from the melt, it is necessary to know the data on thermal conductivity for the solid and liquid states over a wide range of temperatures—from nearly room temperature up to temperatures exceeding the melting point by 100-200 K. The importance of these data has essentially increased in connection with necessity to solve the problem of verification of numerical models of heat transfer and generation of benchmark data. To obtain high-temperature data for semitransparent materials, it is recommended to use absolute stationary methods of measurement, these being the most precise and universal ones in this case (Golyshev and Gonik 2001). The method of coaxial cylinders made it possible to obtain data on the thermal conductivity of a wide class of semitransparent melts in the temperature range 500-2500 K (Golyshev and Gonik 1992a). Golyshev and Gonik (1994) described a setup for measuring the thermal conductivity of the solid phase by the absolute stationary method on two identical samples, modified for semitransparent materials.

However, data close to the melting point are often required. In particular, it is often necessary to measure in situ the supercooling of the melt-crystal interface (Golyshev et al 2000). To solve this problem by the absolute steady-state technique it is necessary to overcome many problems with respect to the solid phase. As regards studying melts, this technique is rather time-consuming. Golyshev and Gonik (1994) described a relative method of measurement based on the comparison of thermal conductivities of the solid and liquid phases of the same semitransparent material. This method makes it possible to obtain data close to the melting point, and in combination with methods mentioned above to obtain a complete set of data in the required temperature range.

In the present work, the experience of applying these methods for thermal conductivity measurements close to the melting point, of semitransparent materials and materials opaque to thermal radiation is described. Special attention is paid to the effect of thermal contact resistance on measurements as a main error component and to ways of reducing and eliminating it.

2 Experimental technique

2.1 Measurement of thermal conductivity of single crystals

The thermal conductivity, λ^{cryst} , of Bi₄Ge₃O₁₂ single crystals in air in the temperature range 750–950 K was measured on two identical samples by the absolute stationary method (Golyshev and Gonik 1994). A scheme of the setup used for the measurements is shown in figure 1a. The samples were made from a single crystal in the form of disks with diameter D = 80 mm and thickness H = 7.75 mm, and placed between 0.3 mm thick platinum plates. A plane two-sectional heater was located between samples. Its external section served for the compensation of radial heat leakage. The temperature of the surface of the platinum plates was measured with Chromel–Copel thermocouples with electrodes 0.3 mm in diameter mounted in BeO ceramics with a diameter of 1.5 mm. Channels for the thermocouples were located in disks made of heat-resistant steel.

The effective thermal conductivity was calculated by the equation:



Figure 1. Scheme of experimental setups for the study of thermal conductivity in solid (a), liquid (b), and both solid and liquid states (c).

where q is the axial flux through samples of thickness H_1 and H_2 . ΔT_1 and ΔT_2 are temperature drops across them. To determine the real thermal conductivity of a material, the radiative component, $q_{\rm rad}$, should be excluded from the total flux, q. With sufficient accuracy, $q_{\rm rad}$ for highly transparent materials (optic density $\tau \ll 1$) can be calculated in an optically thin-layer approximation (Filippov 1954):

$$q_{\rm rad} = 4n^2 \sigma \left[\frac{1-R}{1+R} - \frac{2}{3} \tau \frac{1-4R+R^2}{\left(1+R\right)^2} \right] T^3 \Delta T , \qquad (2)$$

where *n* is the refractive index of the medium, σ is the Stefan–Boltzmann constant, *R* is the reflectivity of platinum, ΔT is the temperature drop, and *T* is the average temperature across the sample.

Taking into account that thicknesses of samples and temperature drops across them are close to each other, it is possible to enter into the equations their average values: $H = \frac{1}{2}(H_1 + H_2), \Delta T = \frac{1}{2}(\Delta T_1 + \Delta T_2)$. We then finally obtain:

$$\lambda_{\rm cond}^{\rm cryst} = (q - q_{\rm rad}) \frac{H}{\Delta T} \ . \tag{3}$$

The main instrumental error in the determination of λ^{cryst} is due to the error in measuring the temperature drop ΔT and to the error in calculation of the heat flux. The latter is connected with a possible deviation from one-dimensionality of the temperature field. To reduce it, thin enough samples were taken $(D/H \approx 7)$. Furthermore, radial heat leakages were compensated for by a guard heater. The basic error in measuring ΔT is due to the thermal contact resistance between the sample and the platinum plate, as well as between the platinum plate and the disk for mounting the thermocouples.

To analyse the contribution of the thermal contact resistance to the overall temperature drop (measured by thermocouples), as well as to estimate the influence of other factors on its value, additional experiments were performed. Experiments were conducted with the same measuring cell. In the first series of experiments, measurements were carried out on a material with well-known thermal conductivity (fused quartz in the form of disks 65 mm in diameter and 10 mm high). The temperature drop at thermal contacts was calculated from the measured data, and was subtracted later from the overall temperature drop. In the second series of experiments, the thermal resistance of the assembly itself was measured. A thin, 0.1-0.3 mm, molybdenum or platinum plate (in air atmosphere) replaced the sample (to imitate the contact resistance between the sample and platinum disks), and it was then possible to neglect the temperature drop across this plate. Similarly, corrections to the measured value of the temperature drop were entered.

To find the sources of systematic errors, measurements of thermal conductivity of crystalline NaCl were made in the range 600-950 K. Single crystals, 70 mm in diameter and 13 mm thick, were studied. The total error in the determination of the thermal conductivity by this technique is estimated at 10%-20%, depending on the measurement temperature.

2.2 Measurement of the thermal conductivity of the melt

The thermal conductivity of the $Bi_4Ge_3O_{12}$ (BGO) melt was measured by the relative method described by Golyshev and Gonik (1994) and tested previously on the NaCl melt (Golyshev and Gonik 2001). The BGO single crystal was used as a sample with a known thermal conductivity. A scheme of the measurement setup is shown in figure 1b. A specimen of monocrystalline BGO in the form of a disk, 60 mm in diameter and 15 mm high, was placed between the main heater in the platinum casing and a platinum support cooler. The temperatures on the support surface T_{cool} and the main heater surface T_{hot} were measured by thermocouples T_3 and T_1 , respectively. A portion of the sample of thickness $h \simeq 1-2$ mm was melted, with the melt suspended on the heater casing by surface tension forces (Golyshev et al 1999).

The exact values of the melt layer height, h, and the rest of the crystal $H \simeq 12-13$ mm were determined on a crystal cut out of a quickly solidified specimen after finishing the measurements. Convection in the melt layer was found to be negligible (Rayleigh numbers, $\text{Ra}_{\text{axial}} \sim 50-400$, depending on thickness; $\text{Ra}_{\text{radial}} < 1$); therefore it does not make a contribution to the heat transfer. As the ratio d/D = 15/60 is small and the hot surface is isothermal (owing to compensation for radial leakages by the guard heater), the heat flux may be considered as one-dimensional. In that case, we have

$$\lambda_{\rm eff}^{\rm melt} = \lambda_{\rm eff}^{\rm cryst} \frac{T_{\rm m} - T_{\rm cool}}{T_{\rm hot} - T_{\rm m}} \frac{h}{H} , \qquad (4)$$

where λ_{eff}^{melt} is the effective coefficient of melt thermal conductivity ($\lambda_{eff}^{melt} = \lambda_{cond}^{melt} + \lambda_{rad}^{melt}$), and T_m is the melting point. We use the effective value λ_{eff}^{melt} , because the BGO melt is almost opaque to thermal radiation (Golyshev et al 2002). It is possible to determine λ_{eff}^{melt} because of the known value of λ_{eff}^{cryst} and to find finally the thermal conductivity of the melt from equation (5):

$$\lambda_{\rm cond}^{\rm melt} = \lambda_{\rm eff}^{\rm melt} - q_{\rm rad} \frac{h}{T_{\rm hot} - T_{\rm m}} .$$
⁽⁵⁾

The value of the radiation flux in the crystal, q_{rad} , can also be calculated from equation (2), taking into account that the absorptivity of the crystal is rather small, $k = 0.03 - 0.04 \text{ cm}^{-1}$ (Golyshev and Gonik 1994) and $\tau \ll 1$.

The instrumental error in the determination of λ_{eff} equals to 30%. Here the contribution of contact resistance to this value is insignificant. This is due to the fact that junctions of the thermocouples were welded directly to the platinum support and the casing of the heater. There is no thermal resistance at the boundary between the melt and the heater casing because of wetting of platinum by the melt. As the temperature of the cold boundary is lower than the melting point by only 10-20 K, the contact resistance between the crystal and the support will also vanish after a while as a result of the process of diffusion welding. Because of this effect, it was even difficult to detach the crystal from the support when disassembling the cell.

2.3 Measurement of the thermal conductivity of the crystal and the melt in one experiment

The possibility of reducing the value of contact resistance or of getting rid of it altogether essentially improves the accuracy in the measurements of thermal conductivity. A scheme of the device for measuring the thermal conductivity of a specimen in the liquid and solid state by the absolute stationary method of the plane layer is shown in figure 1c.

The disk-shaped specimen of germanium with a diameter of 67.6 mm and thickness $\Delta = 16$ mm was placed between two graphite blocks serving for mounting of thermocouples (in drilled holes) and for calculation of the heat flux passing through the studied sample. Both end faces of the disk were well machined (to within ± 0.03 mm). The graphite parts were polished with micro-powder. A heating element consisting of a central (main) and a ring (guard) sections was installed on top, above the sample.

The temperature difference between graphite blocks, ΔT_{total} , is

$$\Delta T_{\text{total}} = \Delta T_{\text{Ge}} + (q_{\text{up}}R + q_{\text{low}}R) \quad , \tag{6}$$

where ΔT_{Ge} is the temperature drop across the sample, and q_{up} and q_{low} are the heat fluxes at the top and bottom boundaries between germanium and graphite; *R* is the contact resistance which was assumed to be the same at both boundaries during the measurements.

The value of ΔT_{total} was determined from the measured temperature difference ΔT_{meas} between thermocouples T₂ and T₃, corrected for the temperature drop across the graphite layers, δ_{up} and δ_{low} , are the distances from the place where these thermocouples were embedded to the surface of the graphite block:

$$\Delta T_{\text{total}} = \Delta T_{\text{meas}} - \left(\frac{q_{\text{up}}\delta_{\text{up}}}{\lambda_{\text{graph}}} + \frac{q_{\text{low}}\delta_{\text{low}}}{\lambda_{\text{graph}}}\right) , \qquad (7)$$

with heat fluxes $q_{\rm up}$ and $q_{\rm low}$ determined from temperature drops between thermocouples T_1 and $T_2 (\Delta T_{\rm up})$, and T_3 and $T_4 (\Delta T_{\rm low})$ embedded in graphite blocks at the known distances $l_{\rm up}$ and $l_{\rm low}$:

$$q_{\rm up, \, low} = \lambda_{\rm graph} \frac{\Delta T_{\rm up, \, low}}{l_{\rm up, \, low}} \ . \tag{8}$$

Using the equation for the thermal conductivity for germanium

$$\frac{1}{2}(q_{\rm up} + q_{\rm low}) = \frac{\lambda_{\rm Ge} \Delta T_{\rm Ge}}{\Delta_{\rm Ge}}$$
(9)

and equations (7) and (8) we can calculate the required value of contact resistance:

$$R = \frac{\Delta T_{\text{total}}}{\frac{1}{2}(q_{\text{up}} + q_{\text{low}})} - \frac{\Delta_{\text{Ge}}}{\lambda_{\text{Ge}}} ;$$
(10)

or determine thermal conductivity of the sample itself:

$$\lambda_{\rm Ge} = \frac{\frac{1}{2}(q_{\rm up} + q_{\rm low})}{\Delta T_{\rm Ge}} \Delta_{\rm Ge} \quad . \tag{11}$$

The contact resistance between germanium and graphite vanishes on melting of the specimen. This allows us to exclude it from consideration in measurements of the thermal conductivity of germanium, at first in the liquid, and then in the solid state. A gap with predetermined height (see figure 1c) was provided in the construction of the measuring cell for implementation of this scheme. Then, upon melting (density of the melt is more than density of the crystal), the melt entirely fills the whole volume in graphite. The top graphite block comes down onto the skirting of the bottom one, and the height of the sample, Δ , becomes exactly known. The surplus of germanium flows out of the graphite reservoir during solidification; the final height of the sample is measured after cooling. The main thing is that the contact resistance across boundaries does not occur in the measurements of thermal conductivity at high temperatures.

In later experiments, with the value of contact resistance being now known, it is possible not to melt the sample and to carry out measurements making corresponding corrections. The accuracy of such measurements, however, is lower, since the value of R is individual for each specific case of surface processing. The instrumental error in the measurements of contact resistance and thermal conductivity is 7% and 6.5%, respectively. In spite of rather large thickness of the melt layer (about 12 mm), the Rayleigh number is small: $Ra_{axial} \sim 1000$ at the steady-state temperature drop across the layer of about 15 K. As the heating is applied from above, convection is practically suppressed (the radial temperature gradient does not exceed 0.2 K cm⁻¹; consequently $Ra_{radial} \sim 10$) and its contribution to heat transfer has been neglected.

3 Discussion

3.1 Role of contact resistance

In figure 2, it is shown that the value of the contact resistance depends essentially on the quality of assembly of the component parts. The better are the surfaces of disks and plates machined, the better they match each other, and the lower is the temperature



Figure 2. Influence of the load P (a) and surface treatment of squeezing disks (b) on the temperature drop across the contact resistance: • measurements on a quartz glass sample 10 mm thick; • measurements without a sample; I, after additional polishing of stainless steel and platinum plates; II, before additional surface treatment.



Figure 3. Variation of the temperature drop with temperature: I, the measured total value with thermocouples T_2 and T_3 ; II, calculated value across germanium specimen.

drop across the contact resistance. Therefore, all measurements (after additional machining of the surfaces of the disks) were conducted later under a load of 25 kg.

The experimental data for the contact resistance (figure 2b) in the whole range of imposed thermal loads lie on a straight line, which indicates that the guard heating system functions effectively, and the character of the temperature field in the measuring cell does not vary with the value of the heat flux from the main heater.

The contact resistance between the germanium sample and graphite measured in this work (according to the scheme in figure 1c) is about 3×10^{-4} kW⁻¹ m². With the average heat flux through the sample equal to $(3.5-4) \times 10^4$ W m⁻², this corresponds to a temperature drop of 10-12 K at the contact, which is well correlated with the data obtained for the other measuring cell (according to the scheme in figure 1a). Thus, the order of this value should be considered typical for contacts between materials with sufficiently well machined surfaces. The temperature dependence of R found in the experiment is well described by the formula $R = R(T_m) + (\delta R/\delta T) (T_m - T)$, where $R(T_m) = 2.57 \times 10^{-4}$ and $\delta R/\delta T = -0.53 \times 10^{-6}$ kW⁻¹ m². Reduction of R with temperature is connected with increase of thermal conductivity of the gas and contribution of radiation to heat transfer.

Figure 3 illustrates the drastic change in the contact resistance at the moment of melting of the material. One can see that, when the specimen of germanium is solid, the temperature drop ΔT_{meas} (curve I in figure 3) measured across the whole assembly (see figure 1c) appreciably exceeds the estimated value of temperature drop across the specimen (curve II in figure3) because of the temperature drop across contact resistances. As soon as the contact resistance vanishes on melting, the values of ΔT_{meas} and ΔT_{Ge} become equal.

Table 1. Corrections to the measured temperature drop, Δt_{meas} , across the thermal contact resistance: δt_1 —estimates based on measurements of thermal conductivity of fused quartz; δt_2 —measurements depending on the axial heat flux.

	Temperature drop												
$\Delta t_{\rm meas}/{\rm K}$	10.7	13.8	14.6	15.6	18	20.1	21.8	23.9	26.3	28.8	30.2	33.5	44.3
$\delta t_1/\mathbf{K}$	2.3	2.3	2.4	2.5	2.9	3.1	3.2	3.6	3.8	4.1	4.5	4.7	6.6
$\delta t_2/\mathbf{K}$	1.2	1.2	1.3	1.4	1.9	2.2	2.3	2.8	3.0	3.4	3.9	4.2	6.5

3.2 Data on thermal conductivity

3.2.1 Sodium chloride. The corrected data for the temperature drop across the samples have been used in the calculations of the thermal conductivity of NaCl. The values of amendments to the thermal contact resistance based on two methods of their calculation (section 2.1) are presented in table 1. The second method is more precise as corrections δt_2 are calculated depending on the heat flux with the use of nomogram I in figure 2b, and are not rigidly related to the temperature drop determined in measurements of the thermal conductivity of fused quartz.

The data (obtained in this work) for the thermal conductivity of single crystals of NaCl in the range 550–950 K and measured by Petrov et al (1974) in the range 80–450 K are presented in figure 4a. It is obvious that these data are in good agreement with the theoretical results (Missenard 1966). The same figure shows also the temperature dependence $\lambda(\theta^3/T^m)$, where θ is the Debye characteristic temperature and m = 1 for pure crystals. The scatter of data is well within the error of the measurement methods.

Figure 4b illustrates the need to take into account the radiant component of heat transfer in the measurements of thermal conductivity of semitransparent materials at high temperatures. The data on the effective thermal conductivity differ substantially not only from the true thermal conductivity even at temperatures above 700 K, but they have a radically different character of temperature dependence, namely an increase of thermal conductivity with temperature.

The data on thermal conductivity of molten NaCl reported by Golyshev and Gonik (2001) are also shown in figure 4b. Thermal conductivity of NaCl is seen to decrease stepwise approximately by a factor of 1.8 during melting. Such behaviour of thermal conductivity of NaCl would be expected. According to the Debye theory (Missenard 1966), thermal conductivity is proportional to specific heat, sound wave velocity, and



Figure 4. Thermal conductivity of NaCl in the solid state over a wide temperature range (a) and for both solid and liquid states near the melting point (b): \bullet Petrov et al (1974), \bullet this work for solid and Golyshev and Gonik (2001) for liquid phases, \blacksquare effective values; θ is the Debye temperature.

mean value of free path length *l*. While dielectric crystal is melting, *l* decreases and λ decreases accordingly.

3.2.2 *Germanium*. After complete melting of the sample, the contact resistance vanishes at both surfaces, and the temperature drop across the sample is fully determined by the thermal conductivity of the material. This allows measurement of the thermal conductivity of both molten germanium close to the melting point and crystalline one after solidification. From equation (11) we obtain, taking into account equations (6), (7), and (9):

$$\lambda_{\rm Ge} = \lambda_{\rm graph} \frac{\Delta_{\rm Ge}}{2} \left(\frac{\Delta T_{\rm up}}{l_{\rm up}} + \frac{\Delta T_{\rm low}}{l_{\rm low}} \right) \left/ \left[\Delta T_{\rm meas} - \left(\Delta T_{\rm up} \frac{\delta_{\rm up}}{l_{\rm up}} + \Delta T_{\rm low} \frac{\delta_{\rm low}}{l_{\rm low}} \right) \right]$$
(12)

Data on the thermal conductivity of germanium are given in figure 5a. According to our data, the value of thermal conductivity of the melt at the melting point is $\lambda_m = 35.7 \pm 2.3 \text{ W m}^{-1} \text{ K}^{-1}$. Thermal conductivity of solid germanium close to melting point is well described by the straight line (figure 5b):

$$\lambda^{\text{cryst}} = [21.36 - 0.28 \times (1210 - T/\text{K})] \text{ W m}^{-1} \text{ K}^{-1} .$$
(13)

Thus, thermal conductivity of germanium increases stepwise by the factor of 1.7 during melting. Such behaviour reflects cardinal changes in the mechanism of heat transfer. A sharp increase in the concentration of electrons, due to the break of spatial system of sp^3 hybrid covalent bonds when the specific resistance drops more than by two orders of magnitude (Regel' and Glazov 1980), results in growth of their contribution to the total heat transfer.

Though data on the thermal conductivity of germanium are available over a wide temperature range in different scientific publications (Pavlov 1967; Okhotin et al 1972; Motakeff 1990; Grigorev and Meilikhov 1991), they are somewhat contradictory. However, if they are averaged with the data presented here (figure 5a), thermal conductivity of the solid phase above 400 K is quite satisfactorily described by the curve $\lambda/W \text{ m}^{-1} \text{ K}^{-1} \approx 1.9 \times 10^4/(T/\text{K})$. According to the estimates of Okhotin et al (1972), thermal conductivity of germanium changes only slightly with temperature of the melt.



Figure 5. Thermal conductivity of germanium over a wide temperature range (a) and near the melting point (b): \Box Pavlov (1967); \blacklozenge Okhotin et al (1972); \triangle Motakeff (1990); \blacktriangle Grigorev and Meilikhov (1991); \bullet this work; — averaged curves.



Figure 6. Thermal conductivity of Bi₄Ge₃O₁₂ single crystal and its melt.

3.2.3 Bismuth germanate. Data on the thermal conductivity of $Bi_4Ge_3O_{12}$ (BGO) at high temperature (close to the melting point) are presented in figure 6. As for other crystals of dielectrics, thermal conductivity of BGO decreases on melting. Owing to the high density of the material and large differences of atomic radii and weights in the molecule (Golyshev and Gonik 1992b), thermal conductivity of the melt would be expected to be low. The more the atoms in the molecule differ from each other in their dimensions and masses, the lower the value of thermal conductivity as calculated by Golyshev and Gonik (1992b). For instance, calculated thermal conductivity values for CsI and RbI are 0.096 and 0.119 W m⁻¹ K⁻¹, in good agreement with the data obtained a year later experimentally by Nakazawa et al (1992): 0.119 and 0.136 W m⁻¹ K⁻¹, respectively. It should be noted that a large ratio of thermal conductivity in solid and liquid states (by several times) is found also for other crystals. For instance, the thermal conductivity of the barium fluoride crystal close to the melting point is 0.45-0.8 W m⁻¹ K⁻¹ (Sergeev and Men' 1977; Vasil'chenko et al 1983) and in the molten state it is 0.22 W m⁻¹ K⁻¹ (Golyshev and Gonik 1992b); similarly, the thermal conductivity of the yttrium aluminium garnet is $3.5-4 \text{ W m}^{-1} \text{ K}^{-1}$ (Dambev 1981) and 1.1 W m⁻¹ K⁻¹ (Golyshev and Gonik 1992b) in the solid and the molten state, respectively.

On the other hand, it is necessary to note that the accuracy of the data essentially depends on the accuracy of the value of the melting point, which enters into equation (4). Kaplun and Meshalkin (1998) indicated that data on the crystallisation temperature need additional verification in connection with the observed large supercooling of the BGO melt. Accordingly, data on the thermal conductivity of the $Bi_4Ge_3O_{12}$ melt will also be affected.

Data on the thermal conductivity of all materials studied in this work close to the melting point are given in table 2.

Material	$T_{\rm m}/{ m K}$	$\lambda/\mathrm{W}~\mathrm{m}^{-1}~\mathrm{K}^{-1}$					
		single crystal	melt				
NaCl	1073	1.2 (this work)	0.41 (Golyshev andGonik 2001)				
$Bi_4Ge_3O_{12}$	1317	1.15 (Golyshev and Gonik 1994)	0.2 (this work)				
Ge	1210	17 (this work)	37 (this work)				

Table 2. Thermal conductivity, λ , of materials close to their melting point.

4 Conclusion

Measurements of thermal conductivity of materials at high temperatures by the absolute (relative) stationary methods are affected by the presence of thermal contact resistances. In spite of special preparation of contacting surfaces and increased compression, the temperature drop caused by the contact resistance, as measurements have shown, remains high and may reach 10% - 15% of the temperature drop across the specimen itself. Taking into account the effect of contact resistances or their elimination by the techniques described in this paper leads to an increase in the accuracy of measurements, with data on the thermal conductivity of NaCl and germanium crystals being in good agreement with the data reported by other authors. The data on the thermal conductivity of these materials, as well as of Bi₄Ge₃O₁₂, close to the melting point, have confirmed the general rule that thermal conductivity decreases during the melting of dielectrics and increases in the case of semiconductors.

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