

Spectral Absorptivity and Thermal Conductivity of BGO and BSO Melts and Single Crystals

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Received: 7 November 2007 / Accepted: 22 July 2008 / Published online: 15 August 2008
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Abstract The present work describes the results of spectral absorptivity, α , and thermal conductivity, λ , studies for compound oxides $\text{Bi}_4\text{Ge}_3\text{O}_{12}$, $\text{Bi}_{12}\text{GeO}_{20}$, $\text{Bi}_4\text{Si}_3\text{O}_{12}$, and $\text{Bi}_{12}\text{SiO}_{20}$ in molten and monocrystalline states. The data for the spectral absorptivity were obtained by placing the sample onto a mirror and using the transmission method. To obtain the data on the thermal conductivity of crystals, the stationary method of two identical samples was used. The data for the thermal conductivity of melts were obtained by a new stationary relative method in which the thermal conductivity of the crystal is used as a reference. Special attention is focused on numerical and experimental error analysis at high temperature. The studies have shown that α in the range of a transmission band strongly depends on crystal purity. It varies from 0.0005 cm^{-1} to 0.03 cm^{-1} for $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ and reaches 0.15 cm^{-1} for $\text{Bi}_4\text{Si}_3\text{O}_{12}$. It was found that α is significantly greater for melts than for crystals, reaching (150 to 200) cm^{-1} for the $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ melt. The thermal conductivity of the melts under investigation was found to be much smaller than that of the corresponding crystals.

Keywords Crystals · High temperatures · Melts · Spectral absorptivity · Thermal conductivity

1 Introduction

High-temperature data on thermal conductivity, λ , and spectral absorptivity, α , of dielectric single crystals and their melts are of great importance for the technology of crystal growth from the melt. These properties determine the conditions of heat

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transfer and the features of interphase kinetics that govern crystal quality. The influence of λ and α on crystal quality is greater for larger crystals and for crystals with a higher number of components. In such cases numerical methods are used, as the cut-and-try method becomes too expensive.

In this work, we investigate the influence of thermophysical and optical properties of crystals on heat and mass transfer for several compound oxides. These crystals have a great number of technical applications. In addition, compound oxides, e.g., $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ [1], were found to have a great advantage of supercooling (several degrees and more) on the growing faceted interface during crystallization. The above features make compound oxides attractive for studying fundamental regularities of interfacial kinetics. Compound oxides are also ideal for verification of modern numerical models, which contain descriptions of both radiative-conductive heat transfer (RCT) in two-phase media and interfacial kinetics processes [2].

The spectral absorptivity and thermal conductivity of some melts and crystals at elevated temperatures are available [3–6]. However, these properties are not available for compound oxides. Therefore, the previously developed numerical models and codes for RCT cannot, at present, be applied to compound oxides. This problem is mainly due to compound oxides having a high refractive index n . It should be noted that the method of placing the sample (a melt) on a mirror (“the mirror method”), used for high-temperature measurements of absorptivity, has been developed mainly for low refractive index materials, while the compound oxides under examination have a high refractive index, n , e.g., $n = 2.15$ for $\text{Bi}_4\text{Ge}_3\text{O}$ (BGO) at room temperature. Therefore, the use of the mirror method for high refractive index materials might result in significant errors in the measured absorptivity values.

The objective of the present paper is to analyze experimental errors in the measurement of the spectral absorptivity and thermal conductivity of dielectrics at high temperatures, and to measure the thermal conductivity and spectral absorptivity of compound oxides in the molten and single crystal states. In this work the method for measuring the thermal conductivity of matter close to the melting point, proposed earlier by the authors [6,7], will be used.

2 Experimental Setup and Research Methods

2.1 Spectral Absorptivity

The absorptivity coefficient α was determined by the following two techniques. The first one is a conventional method of transmission measurement for samples having different thicknesses. This method was used at temperatures (100 to 200) K less than the melting point. α was also measured by the method of reflection from a mirror on which the sample is located (MR method [3]).

A schematic of the MR method is shown in Fig. 1. This method was used to determine α of a crystal from room temperature up to the melting point. In the MR method, modulated infrared radiation directed on the crystal or on the melt sample goes through it, reflects from the mirror, then again goes through the sample, and, finally, goes to the monochromator and the detector. In order to measure the intensity of radiation passing

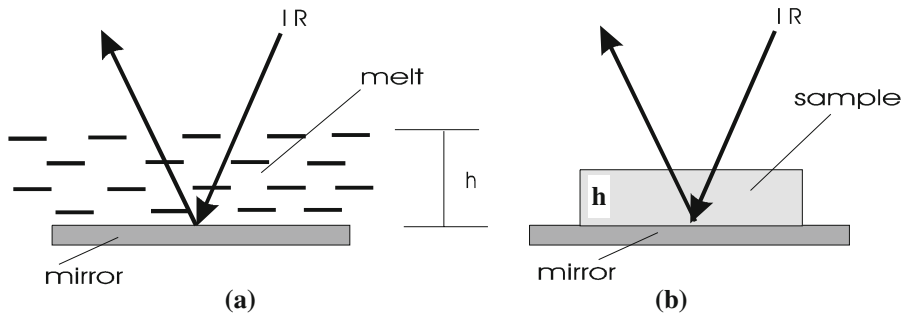


Fig. 1 Schematics of the method of reflection from the mirror for (a) melt and (b) solid samples

through the sample, it is necessary to use optical construction for measurement of the reflection coefficient. As a result, the reflective capacity of the sample-mirror device is actually measured, and in contrast to the transmission method, the radiation reflected from the sample surface contributes to the signal.

The intensity of the radiation reflected from the sample-mirror system, $I = RI_0$, is described for crystals by the formula presented in [6], and for melts by the following equation given in [4]:

$$R = r + [(1 - r)^2 r_1 \tau] / [1 - r_1 r \tau^2] \quad (1)$$

where I_0 is the intensity of the radiation passing through the sample, R is the effective reflective capacity of the sample-mirror layer, $r = (n - 1)^2 / (n + 1)^2$ is the sample reflection coefficient, r_1 is the mirror reflection coefficient, $\tau^2 = \exp(-2\alpha h)$, and h is the sample thickness (Fig. 1).

If multiple reflections are eliminated, then using the transmission method, the absorptivity is calculated as

$$\alpha = \frac{\ln(I_1/I_2)}{(h_2 - h_1)} \quad (2)$$

where I_1 and I_2 are the intensities of the radiation passing through samples of thicknesses h_1 and h_2 , respectively.

Using the MR method for small r , e.g., without taking into consideration the first and multiple reflections, a similar equation may be used to find α ;

$$\alpha = \frac{\ln(I_1/I_2)}{2(h_2 - h_1)} \quad (3)$$

where the factor of two illustrates the fact that the radiation goes through the sample twice. However, for samples with a high refractive index, the error caused by eliminating reflections must be analyzed. The error analysis is given in Sect. 3.1 below.

2.2 Measurement of Thermal Conductivity of Single Crystals

The effective thermal conductivity λ_{eff} of crystals was determined by the absolute stationary method of two identical plane samples. A schematic of the measurements is presented in Fig. 2a.

The single-crystal samples were machined as disks with diameters from 30 mm to 70 mm, and thickness-to-diameter ratios from 0.07 to 0.19. To reduce the contribution of heat radiation from the flat disk boundaries to heat transfer, each sample was placed between platinum plates 0.3 mm thick. The samples together with the platinum disks were placed between steel plates (T-disks) to which thermocouples were attached. The temperature was measured with chromel-copel thermocouples having electrodes 0.3 mm in diameter in BeO ceramics 1.5 mm in diameter. The main and guard heaters were placed between the internal T-disks (Fig. 2a). Finally, the assembly was held between two plates used to minimize temperature gradients. In order to reduce the effect of the contact resistance, these plates were pressed together with an external load.

The effective thermal conductivity was calculated by the following equation:

$$\lambda_{\text{eff}}^{\text{cryst}} = \frac{q}{\Delta T_1/H_1 + \Delta T_2/H_2}, \tag{4}$$

where q is the axial flux through samples of thicknesses H_1 and H_2 , and ΔT_1 and ΔT_2 are the temperature drops on the samples.

To determine a real thermal conductivity, the radiative component q_{rad} should be excluded from the total flux q . The radiative heat flux was determined as a result of solving the one-dimensional problem on radiative-conductive heat transfer [8]. For that purpose, the data on spectral absorptivity measured by the techniques described above were used. Taking into account that the temperature drop is linearly proportional to the sample thickness, the following equation was used to measure the real thermal conductivity:

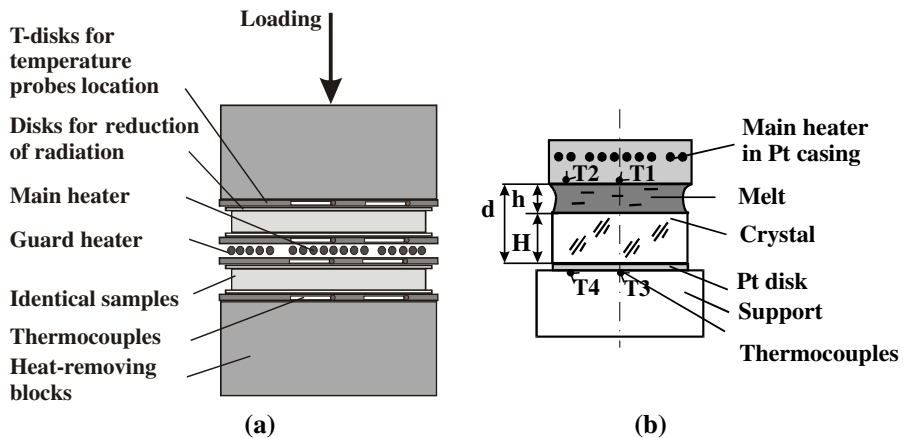


Fig. 2 Schematics of the measuring cell for thermal conductivity of (a) the single crystal and (b) the melt

$$\lambda_{\text{cond}}^{\text{cryst}} = (q - q_{\text{rad}}) \frac{H}{\Delta T}, \quad (5)$$

where $H = (H_1 + H_2)/2$ and $\Delta T = (\Delta T_1 + \Delta T_2)/2$ are the average values of the sample thickness and the temperature drop, respectively. One should take into consideration that q_{rad} is rather high due to very high values of the refractive index, which is in the range of 2.05–2.6 in these crystals [9], and due to high temperatures used in this work. This affected the accuracy of the measured thermal conductivity.

2.3 Measurement of Thermal Conductivity of Melts

Measurements of the thermal conductivity, λ^{m} , of BGO and BSO melts were carried out by the relative stationary method of two samples. The single crystal was used as a reference sample. The measurement method is shown schematically in Fig. 2b.

The specimens of BGO (in the form of a disk of 60 mm diameter and 15 mm thickness) and BSO (in the form of a rectangle with dimensions of 80 mm \times 55 mm and 5.8 mm thick) single crystals were placed between the main heater encased into the platinum casing and the platinum disk on the support (Fig. 2b). The temperatures of the disk surface $T_{\text{cool}} = T_3$ and of the casing surface of the main heater $T_{\text{hot}} = T_1$ were measured with thermocouples. A portion of the sample of thickness $h \simeq (1 \text{ to } 2)$ mm was melted, with the melt being suspended on the heater casing by the forces of the surface tension. Due to the small thickness of the melt layer, heating from above, and the small radial temperature gradient, thermal convection is suppressed. Rayleigh numbers calculated for the axial temperature drop are in the range of $\text{Ra}_{\text{axial}} \sim 50$ to 400 depending on the thickness of the melt layer; the Rayleigh number calculated for the radial temperature drop is $\text{Ra}_{\text{radial}} < 1$. Due to a small value of the ratio $d/D = 0.1$ to 0.25, as well as to a small h , and because the hot surface is isothermal, the heat flux may be considered one-dimensional. Therefore, an effective thermal conductivity of the melt $\lambda_{\text{eff}}^{\text{melt}} = \lambda_{\text{cond}}^{\text{melt}} + \lambda_{\text{rad}}^{\text{melt}}$ may be calculated as follows:

$$\lambda_{\text{eff}}^{\text{melt}} = \lambda_{\text{eff}}^{\text{cryst}} \frac{T_{\text{m}} - T_{\text{cool}}}{T_{\text{hot}} - T_{\text{m}}} \frac{h}{H}, \quad (6)$$

where T_{m} is the melting point.

Following a quick crystallization, the exact heights of the liquid and solid parts of the sample are determined by sectioning the samples with a longitudinal cut. Thus, the thermal conductivity of the melt was determined as follows:

$$\lambda_{\text{cond}}^{\text{melt}} = \lambda_{\text{eff}}^{\text{melt}} - q_{\text{rad}} \frac{h}{T_{\text{hot}} - T_{\text{melt}}}. \quad (7)$$

3 Discussion

3.1 Spectral Absorptivity

3.1.1 Analysis of Systematic Experimental Errors in Measurements for Crystals with High Refractive Index

For compound oxides the major source of error for the measured α is due to a high refractive index. If we denote the value of the absorption coefficient determined in Eqs. 2, 3, and 8 as α^* and the real value of the absorption coefficient resulting from Eqs. 1 to 3 as α , then the relative error of measurement would be: $\delta(\alpha) = (\alpha^* - \alpha)/\alpha$. Figure 3 shows how this error depends on the values of r and τ for different methods of absorption coefficient measurements.

One can see that for the transmission method, a large reflection coefficient for absorptivity results in errors $<4\%$. For the case of the MR method, when the first reflection is not taken into account, the error is approximately 30%. To improve the MR method accuracy, the intensity of the radiation reflected from the sample surface, i.e., the first reflection, I_r , was measured as well. This value is measured at wavelengths where the sample absorbs the falling radiation completely. The absorptivity is calculated by the following equation:

$$\alpha = \frac{\ln \left[\frac{I_2 - I_r}{I_1 - I_r} \right]}{2(h_2 - h_1)} \tag{8}$$

As shown by Fig. 1, the use of Eq. 8 reduces the error in the determination of absorptivity by up to 10%.

The accuracy of the experiment was improved further by adding calculated corrections $\Delta\alpha$ to the data obtained by Eqs. 2, 3, and 8. The resulting expressions are given in Eq. 9 for the transmission method and in Eq. 10 for the MR method, and measurements according to Eq. 8 require Eq. 11.

$$\Delta\alpha = \frac{\ln \left[\frac{1 - r^2 e^{-2\alpha d_2}}{1 - r^2 e^{-2\alpha d_1}} \right]}{h_2 - h_1} \tag{9}$$

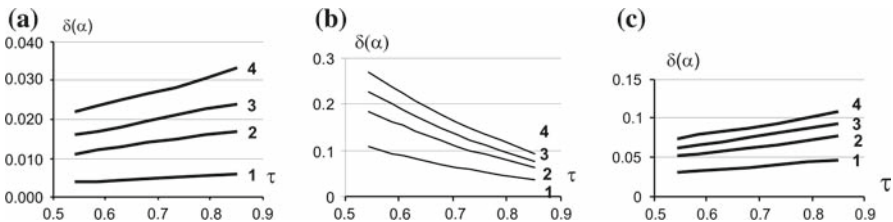


Fig. 3 Dependence of the relative error caused by neglect of multiple reflections $\delta(\alpha)$ on the optical thickness $\tau^* = 2\alpha h$ of the sample for reflection coefficients $r = 0.06-1$, $r = 0.1-2$, $r = 0.12-3$, and $r = 0.14-4$: (a) transmission method, (b) MR method with the first reflection not taken into consideration, and (c) MR method with the first reflection taken into consideration

$$\Delta\alpha = \frac{\ln \left[\frac{\left[r_2 + \frac{(1-r_2)^2 r_1 (\tau_1)^2}{1-r_2 r_1 (\tau_1)^2} \right] (\tau_2)^2}{\left[r_2 + (1-r_2)^2 r_1 \frac{(\tau_2)^2}{1-r_2 r_1 (\tau_2)^2} \right] (\tau_1)^2} \right]}{2(h_2 - h_1)} \quad (10)$$

$$\Delta\alpha = \frac{\ln \left[\frac{1-r_2 r_1 (\tau_2)^2}{1-r_2 r_1 (\tau_1)^2} \right]}{2(h_2 - h_1)} \quad (11)$$

Absorptivity measurements at high temperature involve a very complicated and expensive procedure. The possibility of obtaining the data with only a single sample was investigated in this work. In this case the absorptivity was determined through the ratio I_1/I_2 , where $I_1 = RI_0$ and $I_2 = r_1 I_0$ with the use of Eq. 3 and the addition of the corresponding correction $\Delta\alpha$.

3.1.2 Absorption Coefficient of Single Crystals

Figure 4 presents spectral absorption coefficients of $\text{Bi}_4\text{Si}_3\text{O}_{12}$, $\text{Bi}_{12}\text{SiO}_{20}$, $\text{Bi}_4\text{Ge}_3\text{O}_{12}$, and $\text{Bi}_{12}\text{GeO}_{20}$ at room temperature. The study was carried out by using the transmission method with two samples. It is obvious that the location and width of the transmission band depend on the type of crystal. The α value in the region of the transmission band depends on the quality of a crystal. For example, α may change from 0.005 cm^{-1} to 0.03 cm^{-1} for $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ and reaches 0.15 cm^{-1} for $\text{Bi}_4\text{Si}_3\text{O}_{12}$ and 0.59 cm^{-1} for $\text{Bi}_{12}\text{SiO}_{20}$. The authors believe that much higher values for the latter two oxides are due to a much higher number of crystal imperfections. This, in turn, is based on the fact that the growth technology for the latter two oxides is less advanced compared to $\text{Bi}_4\text{Ge}_3\text{O}_{12}$.

In Fig. 5 the temperature dependence of the $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ absorption coefficient in the near-infrared region of the spectrum is shown. The measurements were carried out in an air atmosphere by the MR method. Two samples of different thicknesses were placed on the platinum mirror. The samples were subjected to IR radiation. The

Fig. 4 Absorption coefficient of the single crystals $\text{Bi}_4\text{Si}_3\text{O}_{12}$, $\text{Bi}_{12}\text{SiO}_{20}$, and $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ at room temperature

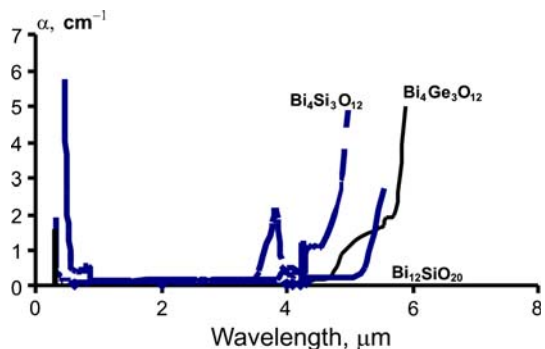
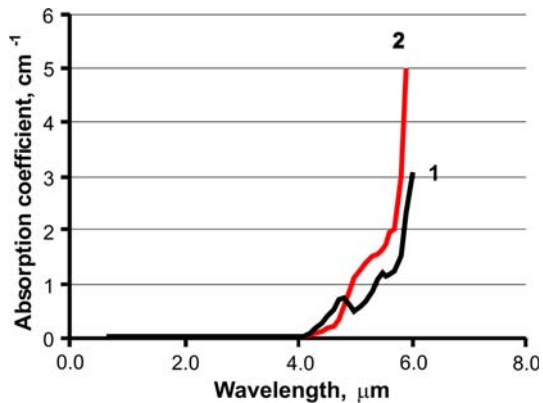


Fig. 5 Temperature dependence of a high-frequency edge of the lattice transmission band of $\text{Bi}_4\text{Ge}_3\text{O}_{12}$: 1—room temperature and 2—900 °C



temperature on the mirror surface in the area of the beam was measured with the platinum thermocouple. The measurements were carried out for the type of crystal for which the absorption coefficient in the transmission band at room temperature is very small and equal to 0.03 cm^{-1} .

The types of crystals with smaller absorptivity require longer samples. However, the optical setup for measurements of absorptivity of converging rays (Cassegrain' scheme) [3] gives large errors for longer samples. That is why these types of crystals have not been studied. Figure 5 demonstrates that with a temperature increase a high-frequency edge of the lattice transmission band shifts to the short wavelength area. However, this shift is not significant and should not be taken into account for RCT calculations. Within the measurement accuracy (optical thickness value of the sample $\tau^* = 2\alpha h = 0.06$), the absorptivity of $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ in the transmission region does not depend upon temperature. The value of α remains equal to 0.03 cm^{-1} in the spectrum range from $1 \mu\text{m}$ to $4 \mu\text{m}$ for temperatures up to the melting point. In order to measure the absorption coefficient close to the melting point, the MR method was used. The wavelength for the study was chosen as $2 \mu\text{m}$, and the change of the sample transmission under heating variation was measured. Melting of the sample was observed by eye, with large values of the melt absorptivity causing considerable reduction in the signal. Thus, the main contribution to the absorptivity in the transparency region is determined not by the temperature change, but by the quality of the crystal. In view of this, we can preliminarily conclude that until the crystal growth technology is improved, it is not necessary to measure the temperature dependence of crystal absorptivity in the transmission region because one can use data at room temperature.

The total instrumental error in the measurement of α (or more precisely of τ^*) is estimated as (12 to 20) %, depending on the wavelength. Unaccounted errors of the technique are caused by two factors. The first one is connected with the changing reflection coefficient of the platinum mirror with time because of the interaction between the mirror surface, the sample, and the air atmosphere of the furnace. This error was kept under control through measuring the 100 % line at the beginning and at the end of the experiment at a given temperature level. The second source of error

was due to the changing reflection coefficient of the sample surfaces because of the interaction with the platinum mirror and the air atmosphere of the furnace. It was controlled by comparing the sample transmission at room temperature before and after the high-temperature measurements.

3.1.3 Absorptivity of the BGO Melt

During transition of the single crystal $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ (BGO) into the liquid state, the α value increases considerably from 0.03 cm^{-1} , and reaches, in the range of (1–4) μm , values of more than (150 to 200) cm^{-1} . The measurements were carried out by the MR method. While studying the BGO melt, we could not use the method of reflection from the mirror in the melt in its conventional form [3], because to obtain several values of melt transmission, it is necessary to have several thicknesses of the melt layer on the mirror, which was not achievable. The most appropriate conditions of the experiment require a melt layer of such a thickness that its absorption would be in the range $0.3 < \tau^* < 0.7$. Because of the large absorption coefficient, the layer of the melt on the mirror should be very thin. But attempts to achieve thin melt layers by changing the position of the crucible with the melt relative to the mirror gave no result, because a melt of about 1 mm thickness escaped from the mirror. We failed to get a melt of less than 1 mm thickness and therefore, had to use another measurement method. A plate of 0.1 mm thickness was cut from the BGO single crystal. The plate was polished on both sides (it should be mentioned that it was impossible to make a polished plate of less than 0.1 mm thickness). The plate was placed onto the platinum mirror. Heating of the plate caused its adhesion to the polished mirror. Due to this behavior, when the plate melted, the melt remains a thin layer on the mirror. The exact value of the melt layer thickness was determined after its solidification as a thickness of the polycrystalline layer. Measurements of the intensity of the radiation reflected from the mirror with a thin layer of the melt showed that, even at 0.1 mm thickness, the melt absorbs radiation completely. The signal registered by the sensor did not differ (within a noise level of (0.5 to 1) %) from the signal for a thick layer of the melt. It means that it was determined only by reflection from the melt surface. Thus, one can make the following conclusion: melt transmission contributed a maximum of 1 % of the total signal; consequently, $e^{-2\alpha d} < 0.01$ and finally $\alpha > (\ln 100)/2d = 200\text{ cm}^{-1}$.

3.2 Thermal Conductivity

3.2.1 Role of Contact Resistance

The basic error in measurements of λ^{cryst} by the method described in Sect. 2.1 arises due to the presence of a thermal contact resistance, R , between the thermocouples and the samples and, consequently, is caused by the error in the measured temperature drop, ΔT , on the sample. Research carried out in [7] showed that the error of ΔT may reach (15 to 20) %, and it strongly depends on the value of the load on the samples. A load of more than 25 kg was found not to lead to a change in the temperature drop; therefore, all further measurements were carried out with such a load.

Table 1 Measured values of contact resistance

T (K)	$R \times 10^4$ ($\text{K} \cdot \text{m}^2 \cdot \text{W}^{-1}$)
304.4	9.81
324.8	9.52
369.5	8.48
545.6	7.44
720.5	6.84
724.6	6.76
869.3	5.73
872.1	5.61
994.5	5.02
997.5	4.70

Since disk processing has a significant influence on the results [7], the measuring and platinum disks were polished. For measurements of the thermal conductivity of sillenites $\text{Bi}_{12}\text{SiO}_{20}$ and $\text{Bi}_{12}\text{GeO}_{20}$ (bismuth silicate and germanate), platinum disks were replaced by nickel disks 0.5 mm thick. These disks did not stick to the samples during multiple heating and cooling and, unlike platinum disks, did not deform during assembly/disassembly. The emissivity of nickel in air is much higher than that of platinum. Therefore, the radiative flux into $\text{Bi}_{12}\text{SiO}_{20}$ and $\text{Bi}_{12}\text{GeO}_{20}$ samples grew in comparison to that with $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ samples, increasing the error in λ^{crist} . Nevertheless, the total error of the measurements was successfully reduced because the contact resistance of the assembly with the nickel disks appeared to be 40% less than that with the platinum disks. To improve the accuracy of the determination of the thermal conductivity of the samples, measurements of the contact resistance of the assembly itself [7] were carried out, when ~ 0.2 mm thick disks of Ni were used in place of the studied crystals. Data obtained for R are given in Table 1.

A decrease of the contact thermal resistance with temperature is related to an increase of the heat transfer in air gaps of the assembly by both convection and radiation. To reduce the radiation in the samples further, a 50 μm thick Pt layer was sputtered onto the sample surfaces while measuring the thermal conductivity of $\text{Bi}_{12}\text{GeO}_{20}$.

3.2.2 Thermal Conductivity of Crystals and Melts

The methods for measurement of the thermal conductivity of crystals and melts, described in Sect. 2, were tested with the use of materials which are considered to be reference standards for measuring the thermal conductivity under RCT conditions. Data for NaCl [7] show that 10% uncertainty is achievable with the current setup. The results of analyzing thermal conductivity of bismuth germanate and bismuth silicates are presented in Fig. 6.

As for all dielectrics, the thermal conductivity of $\text{Bi}_{12}\text{SiO}_{20}$ and $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ drops stepwise while melting. A peculiar property of the $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ melt is that the value of its thermal conductivity is abnormally low and is close to that of thermal insulation.

It should be noted that the uncertainty of measurements of the thermal conductivity of melts by the above described technique is rather high ($\sim (40$ to $45)\%$). On the one hand, it is connected with the error of calculation of radiation in the crystal that is

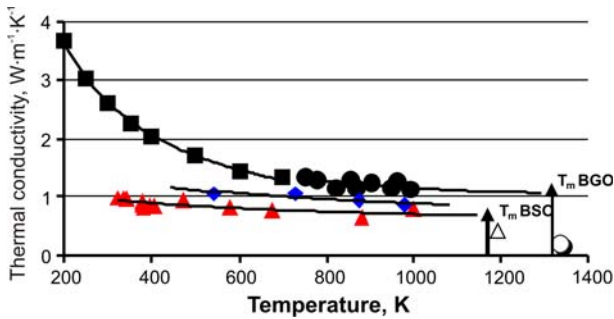


Fig. 6 Thermal conductivity of the single crystals $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ (■ — [10], ● — [6]), $\text{Bi}_{12}\text{GeO}_{20}$ (◆), $\text{Bi}_{12}\text{SiO}_{20}$ (▲) and of the melts $\text{Bi}_4\text{Ge}_3\text{O}_{12}$ (○) and $\text{Bi}_{12}\text{SiO}_{20}$ (△)

no less than (18 to 20)% according to our estimates. On the other hand, the applied method is very sensitive to the temperature uncertainty. Thus, in accordance with Eq. 6, an error of 1 K, for instance, in the melting temperature ($T = 910^\circ\text{C}$ [9]) results in an almost 20% error in the λ^{cryst} value.

4 Conclusions

This work shows that compounds of bismuth oxides with oxides of germanium and of silicon in the solid state have relatively low thermal conductivity values at high temperatures (about $1\text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$) and relatively low absorption coefficients. At the same time, the melt has very high values of absorption coefficient and abnormally low values of thermal conductivity.

Acknowledgments The research was done within the projects 1NO RE1-2233 of the U.S. Civilian Research & Development Foundation for the Independent States of the Former Soviet Union (CRDF), INTAS Project No. 2000-263, and by RFBR (Project No. 02-02-17128a). We are very thankful to Dr. A. Marin and Dr. E. Kozhbahteev who have grown crystals for the studies.

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