Estimating the uncertainty of measurements of thermal conductivity of thin films of thermoelectrics with the 3-omega method

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Abstract. Using the method of computer simulation, the uncertainty of measurements of the thermal conductivity of silicon, which is often used as substrates, and also thin films based on bismuth, is estimated. The influence of the application of an additional dielectric layer between the thermoelectric film and the resistive heater on the measurement results is shown.

1. Introduction
An intense development of methods for measuring the thermal conductivity of thin films began in the end of the 20th century. Cahill et al [1, 2] in 1987 proposed to measure thermal conductivity using the 3-omega method. The principle of this method is that the sample is heated using an alternating current that passes through a specially applied heater (Fig. 1.a). The main advantage of heating with alternating current is that the frequency can be chosen to localize the oscillating temperature field inside the film and substrate [1]. In addition, unlike other measurement methods, the 3-omega method allows directly measuring thermal conductivity rather than thermal diffusivity. Another advantage of the method is low losses for heat exchange with the medium [3]. In the case of direct current heating, all heat passes through the substrate to the environment. At the moment, the 3-omega method is used to measure the thermal conductivity in the longitudinal and transverse directions of the sample [4, 5], and to study the thermal properties of thin films.

To measure the thermal properties of thermoelectric films, an additional dielectric layer is applied [6–8]. When preparing samples, it is necessary to select the optimal frequency range and the width of the resistive heater.

2. Modeling
A computer model for analyzing the measuring the thermal conductivity of samples by the 3-omega method was developed in the Comsol Multiphysics software environment. The geometry of the two-dimensional model is shown in Figure 1, b.

In the model, the solution of differential equations of heat conduction was carried out by the finite element method.
Figure 1. Principle of the method: a) photograph of a resistive heater applied to the sample, and b) 2D model of the 3-omega method.

Model equations:

\[ \nabla \cdot \vec{q} = LQ \quad (1) \]
\[ \vec{q} = -Lk\nabla T \quad (2) \]

Border conditions:

\[ -\vec{n} \cdot \vec{q} = LQ_0 \quad (3) \]
\[ -\vec{n} \cdot \vec{q} = 0 \quad (4) \]

The ambient temperature and the initial temperature of the sample were 293.15 K. Resistive heater length \( L = 3.5 \) cm.

3. Results

To save time for calculating the model, the heating frequency is selected. The heating frequency is usually chosen so that the thermal wavelength in the sample is several times less than the sample thickness [3]:

\[ \frac{d}{\lambda_s} > 2 \quad (5) \]
\[ \lambda_s = \frac{\sqrt{\kappa}}{\sqrt{\omega}} \quad (6) \]
where \( d \) is the thickness of the sample, \( \lambda_s \) is the thermal wavelength in the substrate, \( \kappa \) is the thermal conductivity of the sample, and \( \omega \) is the heating frequency. An example of the result for silicon is shown in Figure 2. Red shading marks the frequency range where the condition of semi-infinity of the substrate is not satisfied, for example, silicon.

Using the Cahill approximation (formula 7), the thermal conductivity of the sample was determined, and then the measurement uncertainty was calculated based on the results obtained and the initial data that were specified in the model.

\[
\kappa = \frac{\Delta q_s \ln(\omega_2/\omega_1)}{2\pi L(\Delta T_1 - \Delta T_2)},
\]

where \( L \) is the length of the resistive heater, \( \omega_i \) is the angular frequency, \( \Delta q_s \) is the amplitude of the heating rate fluctuations and \( \Delta T_i \) is the averaged amplitude of the temperature fluctuations.

The calculation of the measurement uncertainty was carried out according to formula 8:

\[
\delta = \frac{S}{\kappa} \cdot 100\%,
\]

where \( S \) is the sample standard deviation, and \( \kappa \) is the arithmetic mean of the true and calculated thermal conductivity coefficients. The effect of sample thickness on measurement uncertainty was studied (Figure 3). As can be seen from the figure, the uncertainty of the values does not exceed 1% for silicon thicknesses over 300 \( \mu m \). When silicon thickness is less than 200 \( \mu m \), the measurement uncertainty increases and can reach 7%. This may be due to the fact that when the sample thickness is less than 200 \( \mu m \), the condition of semi-infinity of the sample is violated for silicon, or the possibility of using the Cahill approximation (formula 7) is violated due to the shift towards high frequencies.
It is possible to vary the width of the resistive heater, and in various works the width of the heater 2b ranges from 4 to 70 µm [9]. For measuring thinner samples, thinner heaters with a less than 20 µm width, are preferable. The results of measurement uncertainty for a silicon wafer with a thickness of 300, 500 and 700 µm are shown in Figure 4.

![Figure 3](image1.png)

**Figure 3.** Dependence of measurement uncertainty on sample thickness.

Very often, to study the thermal properties of thermoelectric films (due to the fact that they have less resistance than a resistive heater) it is necessary to additionally apply a dielectric layer, the thickness of this layer ranging from 500 to 800 nm [6–8, 10–11]. Si₃N₄ or SiO₂ is often used as a dielectric layer. The results of studying the thermal conductivity of a thin dielectric layer deposited on a substrate have shown that the measurement uncertainty is about 4% (Fig. 5).

![Figure 4](image2.png)

**Figure 4.** Dependence of measurement uncertainty on heater width for silicon thicknesses of 300, 500 and 700 µm.
Figure 5. Dependence of the uncertainty of thermal conductivity measurement in a dielectric layer on frequency.

If we investigate the thermal conductivity of a thermoelectric film in a three-layer structure: substrate-thermoelectric film-dielectric layer, then the measurement uncertainty increases to 30% (Fig. 6). It is possible that reducing the thickness of the dielectric layer can reduce the uncertainty in measuring the thermal conductivity of the thermoelectric film, but then there is a possibility that the dielectric film will not protect against the flow of electric current.

Figure 6. Determination of the measurement uncertainty for a 900 nm thick bismuth film.

Conclusions
The operator error does not exceed 7%, when measuring bulk samples using silicon as an example with the 3-omega method. Most often, the greatest deviation is associated with the choice of a straight section. For measurements, it is better to use a thicker substrate, since the uncertainty in measuring thermal conductivity is reduced to 1%.

The measurement uncertainty of a thin film deposited on a substrate can reach 4%; with the application of an additional coating in the form of a dielectric layer with a thickness of 500–900 nm, it can lead to an increase in uncertainty and exceed 30%.
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