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ABSTRACT

This article describes an improvement of the 3-omega thermal conductivity measurement system facilitating automatic mode measurement. The use of the electrical circuit with negative feedback to compensate the first harmonic in the voltage signal on the heater-thermometer eliminates manual resistance tuning. To test the procedure, measurements of the thermal conductivity of single crystal sapphire Al_2O_3 were made. The experimental thermal conductivity value is in a good agreement with the reference data.

I. INTRODUCTION

The thermal conductivity value is of interest both from fundamental and applied points of view. In the past two decades, the calculations from first principles of thermal conductivity of crystals have been actively developing,[1] and the progress in nanotechnology has led to the necessity of the revision of the size effects on thermal conductivity.[2,3] Besides in modern microelectronics, the problem of efficient heat removal from the "hot" points of electronic structures remains highly relevant since overheating of the elements negatively affects reliability and lifetime.[4]

A number of different methods for thermal conductivity measurements exist, all of them with their limits of applicability.[5,6] The most popular are the stationary heat flux methods, the flash, the thermal reflectance, and the 3-omega methods. The latter approach is suitable for measuring both bulk samples and thin films. It is based on the analysis of the temperature response at the sample surface on a variable input heat flux. The temperature response occurs at the third frequency harmonic of the alternating electric current flowing through a resistive heater-thermometer (hence the name of the method). For these measurements, a thin (about 100 nm thick) and a narrow (several tens of μm wide) metal strip about 3–5 mm long with current and potential contacts is deposited on the flat polished sample surface. The strip serves as both a heater and a low-inertial thermometer to measure the temperature on the sample surface; therefore, it is generally made of a metal with a relatively high temperature coefficient of resistance, i.e., Au, Al, or Pt.

The 3-omega method allows measuring the thermal conductivity of samples in a wide range of sizes while being a direct method (thermal conductivity, not thermal diffusivity, is measured)[7] with the complexity of sample preparation comparable to other methods. The most favorable feature of the 3-omega method is a significantly reduced parasitic heat loss (as compared with the longitudinal heat flux method), which allows measurements with a low error at high temperatures.

During the measurement with the 3-omega method, it is helpful to compensate three to four orders of magnitude larger first harmonic to determine the magnitude of the third harmonic from the total signal. There are implementations that rely only on a lock-in amplifier,[8] so to some extent limiting the measurement dynamic range. For compensation, a differential scheme is used. Since the resistance of a metal strip on a sample can vary with changing external conditions, it becomes necessary to adjust the resistance of a matching resistor. So far as we know, previously this was mostly achieved with non-automatic techniques.[7,9] Existing automatic balancing techniques require use of additional instruments and devices.[10] This paper describes the approach to solve this problem by using a system with negative feedback based on an optocoupler that regulates the transmission coefficient of the first harmonic amplifier, providing automation of the thermal conductivity measurement.

II. METHODS

The 3-omega method analyzes the frequency dependence of temperature fluctuations on the sample surface. The variable heat flux from the sample surface into its thickness is formed on a heater, a long narrow metal strip through which alternating current flows. At the same time, the strip serves as a thermometer, measuring the temperature on the surface of the sample. Usually, four terminals are connected to the strip for supplying current (two external ones) and for measuring the voltage from a heater-thermometer (two internal ones). Application of the alternating current $I = I_0 \cos(\omega t)$ to the strip leads to its heating according to the Joule law. In turn, the alternating heat creates fluctuations in the temperature $\Delta T(\omega)$ of the strip at a frequency of 2ω. They also lead to linearly dependent on the temperature resistance oscillations. The total voltage on the thermometer is the function of the first and third current harmonics. Moreover, it is the third harmonic which carries information on temperature fluctuations of the thermometer ΔT and thermal conductivity of the sample itself. The temperature fluctuations are given by[7]

$$\Delta T = \frac{2V_{3\omega}}{\alpha V_1}, \tag{1}$$

$$k = -\frac{P}{2\pi l S}, \qquad S = \frac{dRe(\Delta T(\omega))}{dln(\omega)}, \tag{2}$$

where $V_{3\omega}$ is the amplitude of the third harmonic, α is the temperature coefficient of resistance of the heater-thermometer, V_1 is the amplitude of the first harmonic, P is the average power on the heater-thermometer, l is the length of the strip, and ω is the frequency of the current flowing through the heater.

It is worth noting that Eqs. (1) and (2) may be used in a certain range of frequencies, limited by the adopted mathematical model.[11,12] On the one hand, in order to consider the sample as half-infinite, the measurement result should not be influenced by the thermal wave reflection from the back surface, so the thermal penetration depth $\lambda = \sqrt{D/2\omega}$, where D is the thermal diffusivity of the sample, should not exceed five widths of the sample. On the other hand, in order to consider the heat flow one-dimensional, the heater width should be more than five times smaller than the thermal penetration depth.

Heat exchange with the environment and the finite thermal resistance between the heater and the sample have a rather weak influence on the measurement accuracy. It is known that the amount of heat losses in the 3-omega method is less than 2% even at the temperature of 1000 K, while we conduct our measurement at room temperature, where this value is less than 2%.[11] The value of temperature rise on the metal-nonmetal junction is around 0.05 K,[13] which could influence the measurement accuracy, but in the 3-omega method, Kapitza resistance is a frequency independent constant; therefore, it does not change the thermal conductivity value.

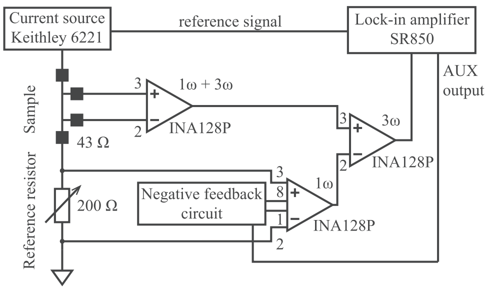

FIG. 1. Block diagram of the measurement fixture.

III. INSTRUMENTATION

Figure 1 shows a schematic used to process the voltage on the heater-thermometer. The sinusoidal current is excited by the Keithley 6221 precise current source. The initial compensation of the first voltage harmonic is achieved by using the 200 Ω multi-turn potentiometer featuring a 100 ppm K^{-1} temperature coefficient (Bourns 3009P-1-201). Resistance of heater-thermometers normally amounts to 20–200 Ω. The set of operational amplifiers facilitates the measurement of the full voltage and its first harmonic on the heater-thermometer and the reference resistor, and after compensation, third harmonic is measured by the lock-in amplifier (SR 850). The measurement is controlled by the personal computer (PC) running the LabView program.

The electric circuit with negative feedback is shown in Fig. 2. Possible part replacements are 2N4416 for JFET KP303 and BC546 for BJT KT3102. The PerkinElmer VTL5C3 optocoupler, utilized for the half-bridge balancing, has an advantage over similar devices: specifically very low temperature coefficient of resistance and provision of galvanic isolation. The resistance of the optocoupler is adjusted by the light-emitting diode (LED) bias current set by DC voltage of auxiliary analog output (AUX Out). This DC voltage is set proportional with a factor of 20 to the in-phase first voltage harmonic by the negative feedback subroutine of the LabView program. The outputs of the negative feedback circuit are used for reference resistor operational amplifier external gain G setting according to the formula $G = 1 + 50\ \mathrm{k}\Omega/R_{18}$, where R_{18} is the resistance between pins 1 and 8. With the help of this system, tuning of the reference resistor signal amplification is automated to cope with external condition changes (like temperature or pressure) or the sample replacement.

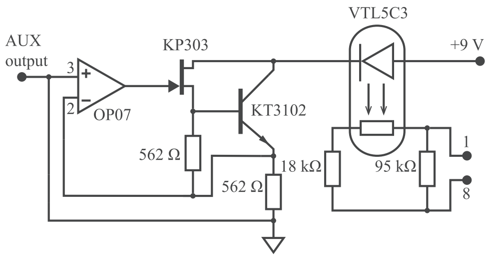

FIG. 2. Negative feedback circuit schematics.

The provided range of the amplification factor in our configuration is from 1 to 4, which allows conducting the measurement for the change of heater-thermometer resistance from 3 Ω to 800 Ω. We chose resistances to set this range of amplification in order to compensate the first harmonic with an error of 0.1% and less. The amplification range could be extended by choosing different resistance values, but it would lead to a less accurate compensation of the first harmonic.

The initial balancing takes about 100 s and comprises matching of reference resistor value to heater-thermometer resistance. Then the automatic balancing takes about 20 s and precedes each measurement cycle, which improves measurement accuracy and eliminates the need for manual adjustment.

IV. EXPERIMENTAL TESTS

The system is evaluated by the measurement of thermal conductivity of the sample with the length of 10 mm, width 6 mm, and thickness 0.42 mm, made of monocrystal sapphire wafer epi-ready on the one side ($R_a < 0.2$ nm) and finely ground on another ($R_a < 1\ \mu$m). The wafer plane is perpendicular to the c axis within 0.2°. The wafer was grown with the Kyropoulos process ("Monocrystall," Stavropol, Russia). According to the manufacturer data, the material purity exceeds 99.997%, impurity content of Ti is less than 1 ppm, and dislocation concentration is less than 10^{-3} cm^{-2}. The gold strip with the length of 3.4 mm, width of 20 μm, and 200 nm thick with four contact pads 2×2 mm^2 was deposited on the sample epi-ready surface with a standard photolithography process. The adhesion is improved by a 30 nm Cr buffer layer. From the measured dependence of strip resistance vs temperature, $\alpha = 0.0028 \pm 0.0001$ K^{-1} was determined at the room temperature. This value corresponds well to the reference one in Ref. 14. 3-omega and α measurements were performed within the evacuated experimental cell in a cryostat. The temperature was measured and controlled by using the LakeShore 336 controller with the Cernox resistance thermometer and the resistive heater.

Figure 3 shows the in-phase part of the temperature oscillation versus the current frequency on the heater-thermometer in the range from 1 to 30 000 Hz. The value of the temperature oscillations amplitude was determined directly from the measured voltage harmonics with Eq. (1). The experimental data are approximated over the linear dependent domain with the frequency natural logarithm as a variable in the range from about 20 to 1700 Hz by the first order polynomial. This frequency range is set by the described restrictions on the thermal penetration depths. The curve slope from the fitting is $S = 0.0073 \pm 0.0003$ K. The error indicated is caused mainly by the errors in α determination and voltage measurement.

Thermal conductivity of the sample is calculated from Eq. (2). The obtained value of $k_{\mathrm{eff}} = 40.5 \pm 1.6$ Wm^{-1} K^{-1} corresponds to the geometric mean of the thermal conductivities along the hexagonal axis c (k_c) and parallel to the basal plane (k_b)[12,15] and is in a good agreement as with the tabulated k_b value of sapphire thermal conductivity of 40 Wm^{-1} K^{-1}[16] and

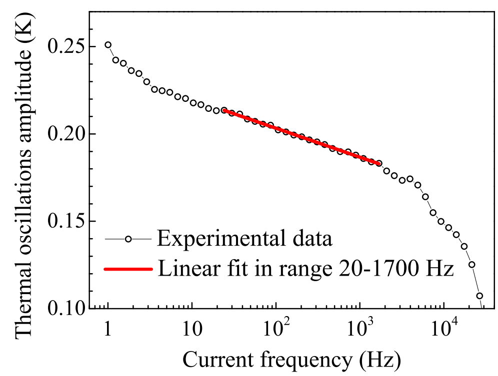

FIG. 3. Dependence of temperature oscillations amplitude in-phase part on the current frequency on the heater-thermometer for Al_2O_3 sample. Current amplitude is 18 mA, and temperature is 296 K.

with the results of the previous measurements for the bulk single-crystal sapphire, e.g., $k_b = 40$ Wm^{-1} K^{-1} obtained by the steady state method[17] and $k_{\mathrm{eff}} = 35$ Wm^{-1} K^{-1} by the 3-omega method.[18] According to the experimental data, k for sapphire is anisotropic: it is about 10% lower in the direction of the c axis than in the basal plane (perpendicular to the c axis).[17]

Note that the total error in thermal conductivity determination consists of the following measurement errors: heater-thermometer length determination, its resistance, voltage harmonics amplitude, α, and slope S. Also, there is a contribution from heat losses. The relative sizing error due to the photolithography process is about 0.06% for our lengths. The heater resistance is measured with a 0.2% error. The error in determining α is about 3.5%. The error in determining S is about 1.4%, excluding the error in α. The amount of heat losses is approximately 1%. So the total amount of the thermal conductivity measurement error is about 4% and maximal contribution is made by errors of α and thermal losses.

To prove reliability of our system, the thermal conductivity of the same Al_2O_3 substrate was measured with steady state heat flow and 3-omega methods at temperatures from

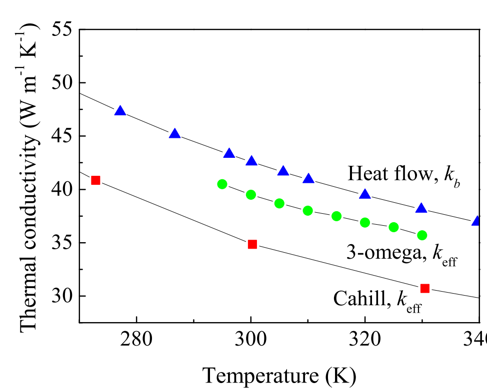

FIG. 4. Data for Al_2O_3 taken with the improved 3-omega system and heat flow method, compared to k_{eff} data obtained by Cahill.[18] The error bars are lower than the symbols size.

290 K to 330 K. In 3-omega measurements, the reference resistor was not manually adjusted. The obtained data are shown in Fig. 4. The 3-omega method provides with thermal conductivity k_{eff} data, while the steady heat flow method gives purely k_b. The "3-omega" data are about 8% less than the data from the steady-heat-flow measurements. Taking into account the anisotropy of k for the Al_2O_3 crystal, it is evident that the data in Fig. 4 are in good agreement.

V. SUMMARY

In conclusion, the negative feedback automatic first harmonic compensation system is proposed for usage in the 3-omega system for thermal conductivity measurements. The measurements of thermal conductivity of single-crystal sapphire were made in the range from 290 K to 330 K. The measured value of thermal conductivity agrees well with the previous data obtained by the competing techniques. So the efficiency of the method for thermal conductivity measurement was confirmed. Compared to the other existing installations for measurements with the 3-omega method, the system described here facilitates measurements in a wide temperature range in automatic mode without adjusting the reference resistor and need for additional devices.

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