Measurement of in-plane thermal and electrical conductivities of thin film using a micro-beam sensor: A feasibility study using gold film

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Kazuhiro Nishimura, Haidong Wang, Takanobu Fukunaga, Kosaku Kurata, Hiroshi Takamatsu

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Department of Mechanical Engineering, Kyushu University, 744 Motoooka, Nishi-ku, Fukuoka, Japan

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Abstract

A new method is proposed for measuring the in-plane thermal conductivity of thin films using a free-standing micro-beam metallic sensor. The sensor is heated in a vacuum by DC to induce a temperature rise, which is determined from the electrical resistance of the sensor. The method consists of two protocols: measurement of a bare sensor before and after deposition of a sample film on its top surface. The thermal conductivity of the sample film is determined by comparing the measured temperature rise with that obtained through numerical analysis. This is based on the principle that the temperature rises of the sensor with and without a deposited film are different because of a difference in in-plane thermal conductance. In this study, measurement of a 20-nm-thick gold film was demonstrated by fabricating two platinum sensors of different widths. The measured thermal conductivities of the platinum sensors and gold film were significantly smaller than those of bulk materials. The relationship between the thermal and electrical conductivities was also discussed.

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Keywords

Thermal conductivity

Measurement technique

Steady-state measurement

MEMS sensor

Thin film

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Nomenclature

D	thickness [m]
H_t	depth of trench [m]
L	length [m]
L_o	Lorentz number [$\text{W}\Omega\text{K}^{-2}$]
L_r	length of overhang [m]
q_v	rate of heat generation per unit volume [W/m^3]
Q	heating power [W]
R	electrical resistance [Ω]
T	temperature [$^{\circ}\text{C}$]
W	width [m]

Greek symbols

α	voltage drop at the electrode pad relative to that at the whole region [-]
β	temperature coefficient [K^{-1}]
ε_R	rate of electrical resistance per heating power [ΩW^{-1}]
Φ	nondimensional electric potential [-]
λ	thermal conductivity [$\text{Wm}^{-1}\text{K}^{-1}$]
Θ	nondimensional thermal conductivity [-]
σ	electrical conductivity [$\text{W}^{-1}\text{m}^{-1}$]

Subscripts

0	zero degrees
1D	one-dimensional
A	middle of sensor
ap	apparent
B	end of sensor
C	electrode
f	film
i	initial condition
s	sensor
sf	sensor with film

1. Introduction

Thermal management is essential for the design and operation of electronic devices and recording media because of the concentrated heat generation resulting from significantly miniaturized and integrated elements [1-4]. It is therefore important to precisely analyze heat generation and heat dissipation in devices that consist of multilayered patterns of thin films. However, the electrical and thermal transport properties of thin films are different from those of bulk materials due to the effects of electron scattering at the surfaces and grain boundaries, which are not significant in bulk materials. These properties also depend on the film thickness, grain size, and fabrication process. For example, the electrical resistances of single-crystal gold films that are fabricated by different processes or have different thicknesses are different from one another [5]. The electrical and thermal transport properties of platinum and gold films depend on the grain size and film thickness [6, 7]. The anisotropic nature of thin films has also been demonstrated. For example, the in-plane thermal conductivity of deposited TbFeCoZr film is an order of magnitude smaller than the cross-plane thermal conductivity [8].

Various methods have been proposed for measuring the thermal conductivity in each direction. The 3ω and thermoreflectance methods are commonly used for measuring the cross-plane thermal conductivity and interfacial thermal resistance [9-11]. A common feature of these methods is that the signals associated with the temperature change after heating the surface of a film on a substrate are detected. Materials with higher thermal conductivities than the film are used as substrates when preparing a system where the heat flow across the film in a vertical direction is dominant. With the 3ω method, the metallic line heater/sensor that is fabricated on the film is electrically heated at an angular frequency ω followed by the detection of a voltage oscillation at frequency 3ω [12]. With the thermoreflectance method, the change in reflectivity is optically detected using a probe laser after heating the surface with a different laser beam [13].

The in-plane thermal conductivity can be measured using these methods, but the principles are quite complicated [14-16]. Other methods for measuring the in-plane thermal conductivity and/or thermal diffusivity use a thin film in a membrane structure [17, 18]. A typical method is to use temperature detectors and heaters that are fabricated on the free-standing membrane [19, 20]. The thermal conductivity is then determined by comparing the measured temperature with the numerical solution to the 3D heat conduction model. Hence, the configuration of the system, i.e., sizes and locations of the sensors and heaters, must be precisely measured because it is critically important for the accuracy of the measurement. Another method is to use the sample film both as a heater and a temperature detector [21-25]. With this method, the sample is prepared in a bridge-like structure suspended from the substrate. Although the in-plane thermal conductivity of thin films is successfully measured using these methods, it would not be feasible for a variety of samples because all of these methods need special fabrication procedures after preparation of the thin film.

Fabrication of heaters and sensors or a bridge-like structure is needed as well as preparation of a free-standing structure. Therefore, these methods are not easy to use for the on-site measurement of thin films, although the properties significantly depend on the film size as well as fabrication conditions.

In this paper, we propose a new method for measuring the in-plane thermal conductivity of deposited thin films using a micro-beam sensor. The sensor, which was originally developed for measuring the thermal conductivity of small samples of gases and liquids [26], is made of a beam-type thin metallic film suspended across a trench on the silicon substrate, which is used as a self-heating thermometer. The protocol consisted of measuring the temperature of the sensor, which was heated in a vacuum, and repeating the same procedure after the deposition of a sample film on the sensor. A numerical analysis of the heat conduction was also performed to obtain the relationship between the thermal conductance and temperature rise. This method has several advantages over the others. One is that the measurement principle is simple and robust with adequate accuracy for the measurement of thin film samples. Another advantage is that electrical conductivity is measured at the same time. However, the greatest advantage is that thermal conductivity can be measured after deposition of a sample film on the sensor with no additional fabrication procedures. Assessing the effects of post-deposition processes such as annealing is also possible with the same specimen.

The objective of the present study is to demonstrate the feasibility of the method using prototype micro-beam sensors. We measured gold film samples of the same thickness deposited on two platinum micro-beam sensors of different widths. The measurement uncertainty was carefully checked and analyzed in detail.

2. Principles of Measurement

The sensor is made of an approximately 10- μm -long freestanding metallic film suspended from a silicon substrate across a trench on the substrate (Fig. 1). It is a self-heating resistance thermometer that is heated by DC in a vacuum. Although the temperature increases by uniform heat generation in the sensor, the temperature at both ends does not change from the constant substrate temperature owing to the large heat capacity of the substrate. By heating at a constant power, the sensor eventually reaches a steady state with a parabolic temperature distribution along the sensor, as shown in Fig. 2 (a). The temperature distribution, which depends on the thermal conductance of the sensor, is estimated by solving the heat conduction equation. After a sample film is deposited on the surface of the sensor, the temperature rise is less than the original because of the increased thermal conductance of the deposited layer (Fig. 2 (b)). The thermal conductivity of the sample film is thus calculated from the difference in the measured average temperature rises before and after deposition of the sample film.

Fig. 1 Schematic of a micro-beam sensor.

Fig. 2 Schematic of temperature distribution in the sensor: (a) bare sensor, (b) sensor with a sample film deposited on top.

When a sensor of uniform cross section is uniformly heated while keeping both ends at the initial temperature T_i , the average temperature rise of the sensor is calculated from the analytical solution to the one-dimensional heat conduction equation and expressed by a unique value in the nondimensional form.

$$\Theta \equiv \frac{T - T_i}{(q_v L^2 / \lambda)} = \Theta_{1D} = \frac{1}{12} = 0.0833 \quad (1)$$

where Θ is the nondimensional temperature rise, T is the average temperature of the sensor, q_v is the rate of heat generation per unit volume, L is the length of the sensor, and λ is the thermal conductivity of the sensor. We can therefore determine the thermal conductivity by measuring the average temperature rise of the sensor at a given heating power Q using the relationship

$$q_v = \frac{Q}{LWD}, \quad (2)$$

where W and D are the width and thickness of the sensor, respectively.

In the experiment, we measured the electrical resistance of the sensor at several given current intensities while keeping the chip at controlled temperatures in a vacuum chamber. The results indicated that the electrical resistance R increased linearly with the heating power Q :

$$R = R_i + \varepsilon_R Q, \quad (3)$$

where R_i is the electrical resistance at a given chip temperature T_i . By repeating the experiment at different chip temperatures, we determined the electrical resistance R_i as a function of temperature and obtained the relationship

$$R = R_0 \{1 + \beta(T - T_0)\}, \quad (4)$$

where R_0 is the electrical resistance at $T_0 = 0^\circ\text{C}$ and β is the temperature coefficient. Substituting Eqs. (2)–(4) in Eq. (1), we obtain

$$\lambda = \frac{R_0 \beta}{\varepsilon_R} \frac{L}{WD} \Theta. \quad (5)$$

After determining the thermal conductivity of the sensor, a sample film was deposited on the top surface of the sensor. The thermal conductivity of the two-layered sensor was then obtained using the same procedure. In this case, the rates of heat generation in the sensor and deposited film were different from one another because of different electrical resistances. However, the temperature in the cross section at any longitudinal position was uniform because all of the surfaces were adiabatic. The longitudinal temperature distribution was therefore the same as that of the sensor, which had a uniform apparent thermal conductivity λ_{ap} and heated uniformly at the same average rate of heat generation.

Considering that the total thermal conductance is the sum of those of the sensor and deposited layer, we can assume the following relationship:

$$\lambda_{ap} = \frac{D_s \lambda_s + D_f \lambda_f}{D_s + D_f}, \quad (6)$$

where the subscripts s and f represent the sensor and sample film, respectively. Consequently, the

thermal conductivity of the sample film is determined from Eq. (6) using the measured values of thermal conductivity λ_f and λ_{ap} and the thicknesses of the sensor D_s and sample film D_f .

3. Experimental Setup and Methods

The sensor and electrode pads were fabricated on a 10 mm $\times$ 10 mm silicon substrate. First, a 320-nm-thick electron beam (EB) resist layer was spin-coated onto a Si (1 0 0) substrate that had a SiO₂ layer on its surface. After the pattern in Fig. 1 was drawn on the EB resist layer using EB lithography, a platinum film was deposited using an electron beam physical vapor deposition method following the deposition of an 8-nm titanium adhesion layer. Then by dipping the chip in a liquid resist-remover followed by a wet-etching process using buffered hydrofluoric acid, only the platinum film remained on the drawn pattern. The titanium layer was also removed in this process. Finally, fabrication of a trench for suspending the platinum film was completed using a dry-etching process with XeF₂ gas. The process was finished by exposing the chip to oxygen plasma to clean the sensor surface and form an oxidized insulation layer on the silicon surface. Before measurement, the sensor was annealed in a vacuum at 220°C for 30 h to stabilize the sensor properties.

Figure 3 shows scanning electron microscope (SEM) images of sensors. It is clear from the images that the sensors had small variations in width with some burrs at the edges. To examine these effects, two sensors of different widths were prepared for measurement. The other significant difference from the model assumed in the previous section was that the bright part of the silicon substrate under the platinum film was removed by the dry-etching process. An overhung film was thus formed at both sides of the trench, which is distinguished by broken lines in Fig. 3. The dimensions of the sensors, i.e., length L , width W , thickness D , width of the overhang L_r , and depth of the trench H_t , which were measured by SEM and a confocal microscope, are summarized in Table 1. The thickness of the sensor was determined by measuring that of the deposited film at an electrode pad.

Table 1 Dimensions of sensors.

Parameter	Narrow sensor	Wide sensor
Length L (μm)	9.2	9.3
Width W (μm)	0.58	1.2
Thickness D (nm)	40	40
Overhang L_r (μm)	3.9	5.3
Depth of trench H_t (μm)	6.9	8.8

Fig. 3 SEM image of sensors: (a) narrow sensor and (b) wide sensor.

The experimental setup and electric circuit are shown in Fig. 4. The chip was fixed in a ceramic holder by pressing four electrode rods onto the electrode pads using springs. The sensor was thus electrically connected to the measurement system. The ceramic holder was mounted on a stage in a vacuum chamber, where temperature was controlled by adjusting the balance between the electrical heating of the stage and cooling with liquid nitrogen. During measurements, the chamber was kept in a vacuum at $\sim 10^{-3} \text{ Pa}$. The temperature of the substrate was measured using a resistance thermometer placed near the sensor.

Fig. 4 Experimental setup.

During the measurements, the sensor was used as a self-heating resistance thermometer. A constant voltage was applied to the sensor using a constant current DC supply (Keithley 2601A).

The electrical resistance of the sensor was measured using a four-wire method: a pair of external electrodes was used to apply a voltage to the sensor, whereas internal electrodes were used to measure the voltage drop across the sensor. The voltage drop across a 100- Ω standard resistor connected in the power line was also measured for calculation of the electric current. The measurements were carried out at 660-ms intervals using digital voltmeters (Keithley 2002).

After determining the thermal and electrical properties of the sensors, a gold film layer was formed on the top surface of the sensors by physical vapor deposition. The thickness of the gold film was 20 nm.

4. Analysis

Measurements were based on the fact that the nondimensional temperature rise is unique to a sensor with specific dimensions. However, Eq. (1), which was derived from the one-dimensional heat conduction equation, did not work for the fabricated sensors because of the overhang at their base. The numerical solution for two-dimensional heat conduction in the straight sensor with an overhang L_r was therefore obtained for the model shown in Fig. 5. We assumed an H-shaped sensor that is uniformly heated only at the narrow part (D , W , and L). A constant temperature was assumed at the boundary between the overhang and substrate. Taking symmetries in the x - and y -directions into account, we obtained the temperature distribution for one-fourth of the whole area. We used finite-element-based software, MSC Marc/Mentat, on the computing system at Kyushu University.

Fig. 5 Physical model for heat conduction analysis.

Calculating the average temperature at the straight part (length L), we obtained $\Theta_{\text{ave}} = \Theta_{\text{narrow}} = 0.1194$ for the narrow sensor and $\Theta_{\text{ave}} = \Theta_{\text{wide}} = 0.1486$ for the wide sensor. These values are higher than $\Theta_{\text{1D}} (= 0.0833)$ in Eq. (1) due to the fact that the temperature at the base of the straight part becomes higher than the substrate temperature due to the presence of the overhang. The reason for $\Theta_{\text{wide}} > \Theta_{\text{narrow}}$ is that the total amount of heat generation is larger in the wide sensor, resulting in a

larger temperature drop at the overhang.

Even though the voltage drop across the sensor was measured using the four-terminal method, the measured data possibly included the voltage drop at the outside of the sensor. A two-dimensional simulation was therefore conducted to obtain the distribution of electric potential in the whole region of the sensor and electrode pad. Giving a constant electric potential in nondimensional form, $\Phi = 1$, at the square part of the external electrode and $\Phi = 0$ at the middle of the sensor (Fig. 6), we solved the Laplace equation using the MSC Marc/Mentat software.

Fig. 6 Nondimensional electric potential distribution in wide sensor.

The electric potential at the boundary between the sensor and overhang was significantly different from that at the electrode used for measurement, as shown in Fig. 6. The voltage drop across the sensor was therefore determined by subtracting the voltage at the electrode pad from the total voltage drop. To this end, the voltage drop at the electrode pad relative to that at the whole region

$$\alpha = \frac{\Phi_C - \Phi_B}{\Phi_C - \Phi_A} \quad (7)$$

was determined for each sensor pattern, where the subscripts A, B, and C represent the middle of the sensor, end of the sensor, and electrode, respectively (Fig. 6). We obtained $\alpha = 0.2722$ for the narrow sensor and $\alpha = 0.3478$ for the wide sensor. The voltage drop at the electrode pad was greater for the

wide sensor because of the smaller electrical resistance of the sensor.

5. Results and Discussion

The electrical resistance of the sensor was measured as a function of the heating power Q at given chip temperatures. At any chip temperature, the electrical resistance increased linearly with Q , as shown in Fig. 7, due to the temperature rise resulting from Joule heating. A linear regression line expressed by Eq. (3) was obtained to determine the electrical resistance R_i at a given chip temperature. From the same experiments at different chip temperatures, we determined electrical resistance as a function of temperature (Fig. 8) and obtained the linear calibration correlation, Eq. (4), for calculating temperature from a measured electrical resistance.

Fig. 7 Measured electrical resistance of Pt sensor as a function of heating power (wide sensor).

Fig. 8 Relationship between electrical resistance and temperature (wide sensor).

Fig. 9 Temperature rise of the sensor as a function of heating power (wide sensor).

Fig. 10 Measured thermal conductivity as a function of temperature.

The constants and coefficients in Eqs. (3) and (4) for the narrow and wide sensors are listed in Table 2. The table also includes the electrical conductivity calculated from measured dimensions and the thermal conductivity that was determined from Eq. (5) with listed R_0 , β , and ε_R . Both the electrical and thermal conductivities of the two sensors agreed quite well with only a 3-4% difference. However, the values were considerably smaller than those of bulk platinum, which is a phenomenon peculiar to thin films. The temperature coefficient of the electrical resistance was also smaller than that of bulk platinum.

Table 2 Electrical and thermal properties of platinum sensors.

Parameters	Narrow sensor	Wide sensor
Variation of electrical resistance with input power at 20°C	2.610×10^5	7.268×10^4
$\varepsilon_R (\Omega W^{-1})$		

Electrical resistance at 0°C, R_0 (Ω)	93.55	42.83
Temperature coefficient β (K^{-1})	0.002045 (0.43)	0.002148 (0.46)
Electrical conductivity σ at 20°C ($\Omega^{-1}m^{-1}$)	4.11×10^6 (0.42)	4.28×10^6 (0.44)
Thermal conductivity λ at 20°C ($Wm^{-1}K^{-1}$)	35.0 (0.49)	36.0 (0.50)

Values in parentheses are those divided by bulk values.

We repeated the same procedure after deposition of a gold film. In Fig. 8, the relationship between the measured electrical resistance and temperature is compared between the sensors with and without a gold film. The electrical resistance decreased by ~65% with deposition of the gold film. The gradient of the line ε_R after deposition of the film was also smaller than that of the bare sensor owing to the different temperature coefficient of the film.

Figure 9 shows the temperature rise of the sensor as a function of heating power. It is clear that the deposition of gold film significantly decreased the temperature rise (by ~60%,) at the same heating power, as a result of increased apparent thermal conductivity.

Figure 10 shows the thermal conductivity of the sensor and apparent thermal conductivity of the film-deposited sensor, which were calculated with R_0 , β , and ε_R determined for each case. The thermal conductivity of the gold film, which was calculated from Eq. (6), is also shown in the figure. It turned out that the apparent thermal conductivity of the film-deposited sensor was 55% higher than that of the bare sensor. This resulted from the gold layer, which has a thermal conductivity that is 2.7 times that of the platinum sensor.

The effect of an annealing process on the thermal conductivity is shown in Fig. 11. Following the initial measurement immediately after deposition of the gold film, measurements were repeated several times after annealing for two hours at 127°C and an additional 3 × three hours at 197°C. The thermal conductivity increased with annealing time, probably due to the growth of grain size or reduction of defects, and reached a nearly constant value that was ~20% higher than the initial state.

Fig. 11 Measured thermal conductivity of gold film as a function of annealing time.

Fig. 12 Measured electrical and thermal properties of sensors: (a) Pt sensor and (b) gold film.

The measured properties of the platinum sensor and gold film are summarized in Fig. 12(a) and 12(b), respectively, where the electrical conductivity σ , thermal conductivity λ , temperature

coefficient β for the electrical resistance, and Lorentz number

$$Lo = \frac{\lambda}{\sigma T} \quad (8)$$

are shown as values relative to those of the bulk materials. The thermal and electrical conductivities of the gold film, which were measured for sensors with different widths agreed well with each other, although the effects of burrs at the edge and nonuniformity in the width are possibly greater in the narrow sensor. This suggests that the measurement and model were practically sound and reasonable. The thermal and electrical conductivities of thin films were considerably smaller than those of the bulk materials: 40–50% of bulk platinum and ~35% of bulk gold. The results were different from the available data [27], which showed that the in-plane thermal conductivity of a ~100-nm-thick gold film measured using the AC method was closer to that of bulk gold. Although a direct comparison might be avoided because the fabrication process was different from the present study, the results suggest that the effects of grain boundaries on electrical and heat conduction may be revealed when the gold film becomes considerably smaller than 100 nm. The larger reduction in the thermal conductivity of gold film versus bulk gold compared to that of platinum may be attributed to the fact that the thickness of the gold film was much smaller, only a half the thickness of the platinum film.

The thermal conductivity of metal is proportional to the electrical conductivity because electrons play the main role for energy transport. According to the Wiedemann–Franz law, $Lo = 2.44 \times 10^{-8} \text{ W}\Omega\text{K}^{-2}$ regardless of the type of metal. The measured value agreed fairly well with this value within +15% for the platinum film and $\pm 10\%$ for the gold film. The result is reasonable from the perspective that the thermal energy in thin films is also carried by free electrons. However, due to scattering at the grain boundaries, the temperature coefficient of thermal conductivity of thin films was approximately half of the bulk values.

The uncertainty in the measured thermal conductivity was evaluated by estimating the random and systematic errors [28] in the dimensions of the sensor, voltage drop across the sensor, heating power, linear regression relationship between the electrical resistance and heating power, temperature dependence of the electrical resistance, temperature rise of the sensor, substrate temperature, temperature rise determined by the numerical analysis, and thermal conductivity of the sensor. Among them, the errors in thickness and width were significantly larger than the others. The estimated uncertainty in the thermal conductivity was $\pm 6\%$. In the present experiment, we defined the width of the sensor by taking the presence of burrs into account. If we neglected the effects of the burrs, however, the thermal conductivity of the gold film would be higher by 6%.

The present paper demonstrated the feasibility of our method, which uses a micro-beam sensor. One of the unique features of this method is that the electrical conductivity is determined together

with the thermal conductivity. This is a great advantage because electrical conductivity is a key property for the estimation of heat generation and, consequently, the power consumption of devices. In the measurement of thin films, it is important to measure samples that were fabricated using the same process and conditions as those used for the real devices because the thermal and electrical properties of thin films strongly depend on the fabrication process and thickness. The greatest advantage of the proposed method is its potential to be used for this purpose. Once we have sensors with calibrated electrical and thermal properties, we can prepare a sample simply by depositing a film on the sensor. No additional procedures are needed for the preparation of samples. With such samples, measurement can be done without using expensive instruments, which is usually the case with more sophisticated methods.

6. Conclusions

Measurement of the in-plane thermal conductivity of a thin film was demonstrated with a new method using a free-standing micro-beam sensor, which was used as a self-heating thermometer. The thermal conductivity of a film deposited on the surface of the sensor was measured by comparing the temperature rise of the sensor before and after deposition of the film. The thermal conductivity of a 20-nm-thick gold film was ~65% smaller than that of bulk gold, indicating a size effect. The electrical conductivity of the gold film was also smaller than the bulk value by ~65%, which suggests that the relationship between the electrical and thermal conductivities follows the Wiedemann–Franz law. Both the electrical and thermal conductivities increased by ~20% after an annealing process.

An error analysis showed that the thermal conductivity of thin film was measured within a $\pm 6\%$ uncertainty. An advantage of the proposed method is that it measures electrical conductivity together with thermal conductivity. A more significant difference from other methods is its potential use for benchside measurement. The preparation of samples is possible simply by depositing a film on a calibrated sensor. Expensive instruments are not needed for the measurement, which is the case with ultra-fast optical or transient measurements.

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