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Thermal Conductivity Measurement of Thermally-Oxidized SiO_2 Films on a Silicon Wafer Using a Thermo-Reflectance Technique[1]

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This paper describes the development of an advanced method to measure the normal-to-plane thermal conductivity of very-thin insulating films. In this method the metal film layer, which is deposited on thin insulating films, is Joule heated periodically and the ac-temperature response at the center of the metal film surface is measured by a thermo-reflectance technique. The one-dimensional thermal conduction equation of the three-layered system was solved analytically, and a quite simple and accurate approximate equation was derived. In this method, calibration factors of the thermo-reflectance coefficient were determined using the known thermal effusivity of the substrate. The present method was examined for thermally-oxidized SiO_2 films (1000–20 nm thick) on a silicon wafer. The present results of the thermal conductivity agree with those of VAMAS TWA23 within ±10%.

KEY WORDS: thermal conductivity; thin film; thermo-reflectance; periodic method; silicon dioxide.

1. INTRODUCTION

The microelectronics industry requires techniques for measuring the thermal conductivity of electrically insulating materials in the form of very-thin films to evaluate heat dissipation across the thin films to a silicon wafer.

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A method has been developed by Taketoshi et al. [1] to measure the normal-to-plane thermal diffusivity of very-thin films deposited on a substrate using a picosecond thermo-reflectance technique. In this method the bottom surface of the thin film is heated by the laser-flash light, which is transferred through the substrate, and the temperature response of the top surface is measured. This method has successfully been applied to determine the normal-to-plane thermal diffusivity of very-thin metal films deposited on a glass substrate. However, the method has a disadvantage in that it cannot be applied to low-thermal-conductivity thin films deposited on a high-thermal-conductivity substrate, such as thermally-oxidized SiO_2 films on a silicon wafer.

The three-omega method has been developed by Lee and Cahill [2] to measure the normal-to-plane thermal conductivity of very-thin electrically-insulating films deposited on a substrate. Characteristic of the three-omega method is that the in-phase amplitude of the ac-temperature response is measured in the low-frequency range. This method has successfully been applied to various thin electrically-insulating films on a substrate including thermally-oxidized SiO_2 films on a silicon wafer. However, the three-omega method has a disadvantage in that a fine pattern of the metal film must be deposited on the specimen using a lithography technique, because the method is based on a two-dimensional thermal conductivity measurement system.

This paper describes the development of an advanced method to measure the normal-to-plane thermal conductivity of very-thin insulating films. In the present method, a wide metal film, which is deposited on the thin insulating films, is Joule heated periodically and the ac-temperature response at the center of the metal film surface is measured by a thermo-reflectance technique. Thus, the one-dimensional model of the thermal conduction equation can be applied for the three-layered system. The one-dimensional equation was solved analytically, and a quite simple and accurate approximate equation was derived. The present method was applied to thermally-oxidized SiO_2 films (1000–20 nm thick) on a silicon wafer.

2. THEORETICAL CONSIDERATIONS

In Fig. 1 the thermal conductivity measurement system of the present study is shown schematically. As shown in Fig. 1, it consists of three layers, the metal film layer, the thin-film layer, and the substrate layer. In the system, the metal film is Joule heated periodically and the ac-temperature response at the center of the metal-film surface is measured by a thermo-reflectance technique. Although the metal film is ac-heated uniformly, an ac-temperature gradient along the thickness of the metal film layer may

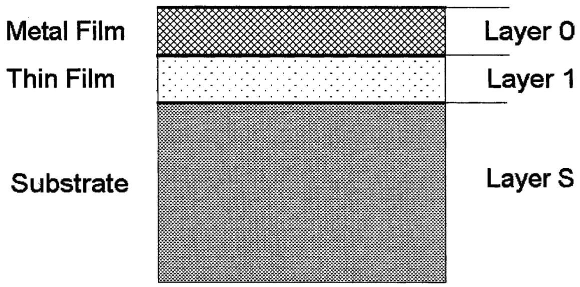

Fig. 1. Thermal-conductivity measurement system for the present method. System consists of three layers, the metal film layer, the thin film layer, and the substrate layer. In the system the metal film is Joule-heated periodically and the ac-temperature response of the surface of the metal film is measured by a thermo-reflectance technique.

take place. It is assumed in the system that the substrate layer has infinite thickness, but the metal film layer and the thin film layer have finite thicknesses. The one-dimensional thermal conduction equation of the system in the normal-to-plane direction was solved analytically. The solution of the thermal conduction equation is given as the following equation, where q denotes the heat per unit volume, and d_0 and d_1 denote thicknesses of the metal film layer and the thin film layer, respectively.

$$T(0) = \frac{q}{i\omega C_0}\left\{1+\left(\frac{\left(\frac{\lambda_0 k_0}{\lambda_S k_S}-\frac{\lambda_0 k_0}{\lambda_1 k_1}\right)\exp[-(1+i)k_1 d_1]\sinh[(1+i)k_0 d_0]}{\frac{\lambda_1 k_1}{\lambda_S k_S}\sinh[-(1+i)k_1 d_1]-\cosh[-(1+i)k_1 d_1]} - \cosh[(1+i)k_0 d_0] - \frac{\lambda_0 k_0}{\lambda_1 k_1}\sinh[(1+i)k_0 d_0]\right)^{-1}\right\} \quad (1)$$

When the following equations are valid,

$$k_0 d_0 << 1 \quad (2a)$$

$$k_1 d_1 << 1 \quad (2b)$$

Equation (1) can be simplified to the following equation:

$$\frac{T(0)}{q d_0} = \frac{\exp\left(-\frac{\pi}{4}i\right)}{\sqrt{\lambda_S C_S \omega}} + \left(1-\sqrt{\frac{\lambda_1 C_1}{\lambda_S C_S}}\right)\frac{d_1}{\lambda_1} + \left(\frac{1}{2}-\frac{\lambda_0 C_0}{\lambda_S C_S}\right)\frac{d_0}{\lambda_0} \quad (3)$$

The first term on the right-hand side of Eq. (3) is proportional to $\omega^{-1/2}$. On the other hand, the second and third terms of Eq. (3) are real constants independent of $\omega^{-1/2}$. The plot of the "In-phase Amplitude of $T(0)/qd_0$ vs. $\omega^{-1/2}$" gives a slope–intercept form. So the thermal effusivity of the substrate $\lambda_S C_S$ can be determined from the slope, and the sum of the second and third terms can be determined from the intercept. To determine an absolute value of the thermal conductivity of a thin film, the calibration factor of the thermo-reflectance coefficient must be known. However, it is very difficult to make a calibration using conventional temperature standards, because the thermo-reflectance coefficients of metals are generally very low. In this method, calibration factors of the thermo-reflectance coefficient are determined by adjusting the thermo-reflectance coefficients for the slope of the plot to fit with the value of the factor of proportionality, which can be calculated theoretically using the known thermal effusivity of the substrate. It should be noted here that the factor of proportionality is only a function of the thermal effusivity of the substrate $\lambda_S C_S$, but is not a function of the thermophysical properties of the other layers. Actually the calibration factor includes the error correction of the measurement of the ac-power and the dimension of the metal-film heater making the method easier to apply.

Assuming that the thermal effusivity of the metal film $\lambda_0 C_0$ is much smaller than that of the substrate $\lambda_S C_S$, the third term of Eq. (3) is reduced to half of the thermal resistance of the metal film. Assuming that the thermal effusivity of the thin film $\lambda_1 C_1$ is much smaller than that of the substrate $\lambda_S C_S$, the second term of Eq. (3) is reduced to the thermal resistance of the thin film.

The value of the third term of Eq. (3) can be determined theoretically using the known values of d_0, λ_0, C_0, λ_S, and C_S. The value of the second term of Eq. (3) can be determined by subtracting the theoretically-obtained value of the third term from the experimentally-obtained value of the sum of the second and third terms.

The following equation, which is a quadratic equation of $\lambda^{-1/2}$, is given by defining the second term of Eq. (3) as R_1^*;

$$R_1^* = \left(1 - \sqrt{\frac{\lambda_1 C_1}{\lambda_S C_S}}\right)\frac{d_1}{\lambda_1} \tag{4}$$

The solution of Eq. (4) is given by the following equation:

$$\sqrt{\frac{1}{\lambda_1}} = \sqrt{\frac{R_1^*}{d_1} + \frac{C_1}{4\lambda_S C_S}} + \sqrt{\frac{C_1}{4\lambda_S C_S}} \tag{5}$$

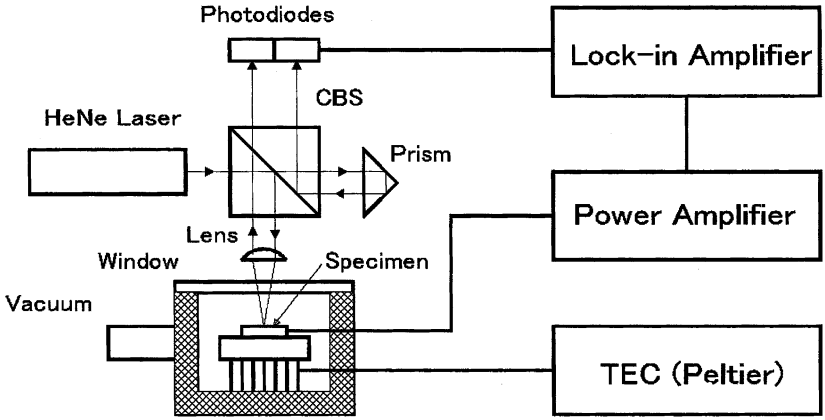

Fig. 2. Block diagram of the experimental setup. The specimen is set horizontally on the sample stage. A TEC system keeps the sample stage at a constant temperature (25°C).

Finally, by substituting R_1^* of Eq. (5) by the experimentally-obtained value and using the known values of d_1, C_1, λ_S, and C_S, the thermal conductivity of the thin film λ_1 can be determined.

3. EXPERIMENTAL

A block diagram of the experimental setup is shown in Fig. 2. The specimen is set horizontally on the sample stage. The thermoelectric cooler (TEC) system keeps the sample stage at a constant temperature (25°C). The whole sample assembly is installed in a vacuum chamber. At the top of the chamber above the sample assembly, an optical window is provided to enable thermo-reflectance measurements. The size of the specimen is 25 mm × 12.5 mm. The metal film is stripe-shaped with dimensions of 1.7 mm width, 10 mm length, and 100–200 nm thickness. The metal film is deposited on the specimen by evaporation or sputtering, using a mask made of stainless steel with a thickness of 0.1 mm. The thickness of the metal film is determined by a surface profiler (Sloan, Dektak 3030). At both ends of the metal film, a pair of electrodes made of silver paste are printed to make good electrical contact with the four-pin spring contactors for supplying ac-current and detecting ac-voltage.

As the thermo-reflectance coefficient is very low, a differential optical system has been developed for the thermo-reflectance measurement to minimize the common-mode noise of the HeNe laser [3]. The noise of the differential optical system is less than 0.01 ppm (at 500 Hz). It has been found that the bismuth film has quite a large temperature coefficient of thermo-reflectance (about 900 ppm·K^{-1}), although the thermo-reflectance

Table I. Thicknesses and Thermophysical Properties of Samples Used in the Present Study

Samples	Thickness (mm, nm)	Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Specific heat capacity per unit volume ($J \cdot cm^{-3} \cdot K^{-1}$)
Silicon	0.6 mm	148 [5]	1.66 [5]
Sapphire	0.2 mm	46 [5]	3.20 [5]
Bismuth (bulk)		8.5 [5]	1.19 [5]
Bismuth (film)	150 nm[a]	1.46[a]	
Gold (bulk)		315 [5]	2.47 [5]
Gold (film)	100 nm[a]	178[a]	
Thermally-oxidized SiO_2 films (NIST RR samples)	493.5 ± 1.4 nm 200.4 ± 0.9 nm		1.63 [5]
Thermally oxidized SiO_2 films (KST samples)	96.8 nm ($\pm 5\%$) 51.0 nm ($\pm 10\%$) 19.9 nm ($\pm 10\%$)		1.63 [5]

[a]These data were obtained in the present study.

coefficient of the gold film is only about 30 $ppm \cdot K^{-1}$ [4]. In the case of the bismuth sensor the temperature-equivalent noise of the system is less than 1.1×10^{-5} K (at 500 Hz). On the other hand, in the case of the gold film sensor, the temperature-equivalent noise of the system is about 3×10^{-4} K (at 500 Hz). As the amplitude of the ac-temperature response is about 0.3 K, the signal-to-noise ratios are sufficient in both cases.

The sine output signal from the built-in sine-wave generator of the lock-in amplifier (Stanford, SR8300), is amplified by the power amplifier to energize the metal film deposited on the specimen. The differential-photodiode sensor is plugged into the current input port of the lock-in amplifier to measure the second harmonic of the photocurrent signal.

4. RESULTS AND DISCUSSION

The condition of the specimen and the values of thermophysical properties used in the present study are listed in Table I [5]. Figure 3 shows the results of the measurement of a sapphire substrate with a bismuth-film sensor deposited on it. The plot shows good linearity in the frequency range from 500 to 8000 Hz. In this case calibration factors of the thermoreflectance were determined using the known thermal effusivity of the sapphire substrate. The thickness of the metal film was determined to be about 150 nm by a surface profiler (Sloan, Dektak 3030). From the experimental results of the intercept, according to the third term of Eq. (3), the

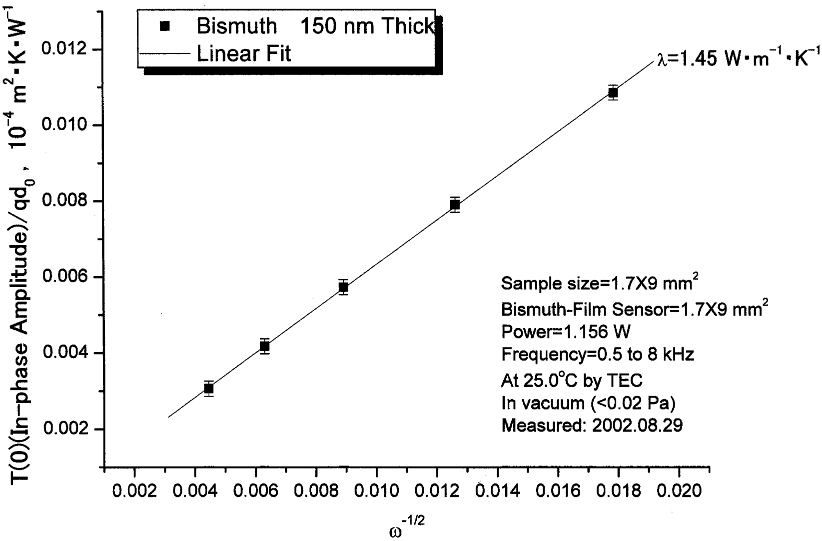

Fig. 3. $T(0)$ (In-phase Amplitude)/qd_0 vs. $\omega^{-1/2}$ plot for the measurement of the sapphire substrate using a bismuth-film sensor.

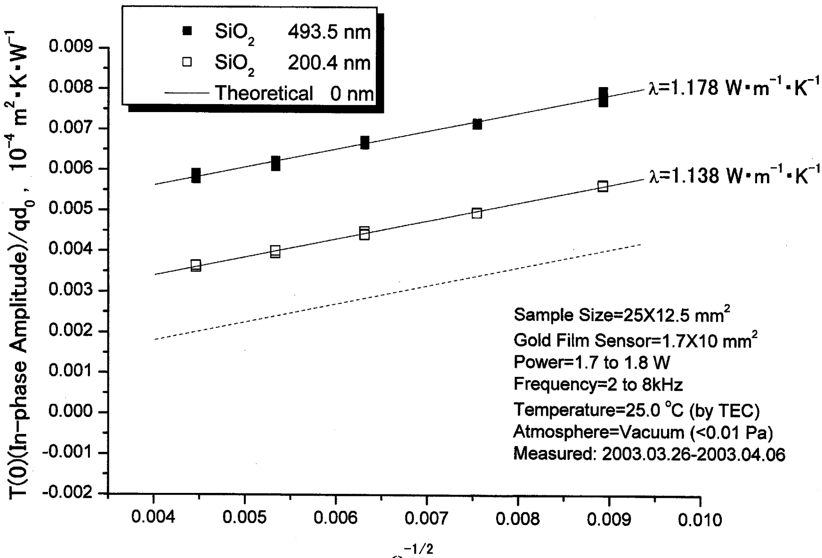

Fig. 4. $T(0)$ (In-phase Amplitude)/qd_0 vs. $\omega^{-1/2}$ plot for the measurement of the thermally-oxidized SiO_2 films (NIST RR samples) on a silicon wafer using a gold-film sensor.

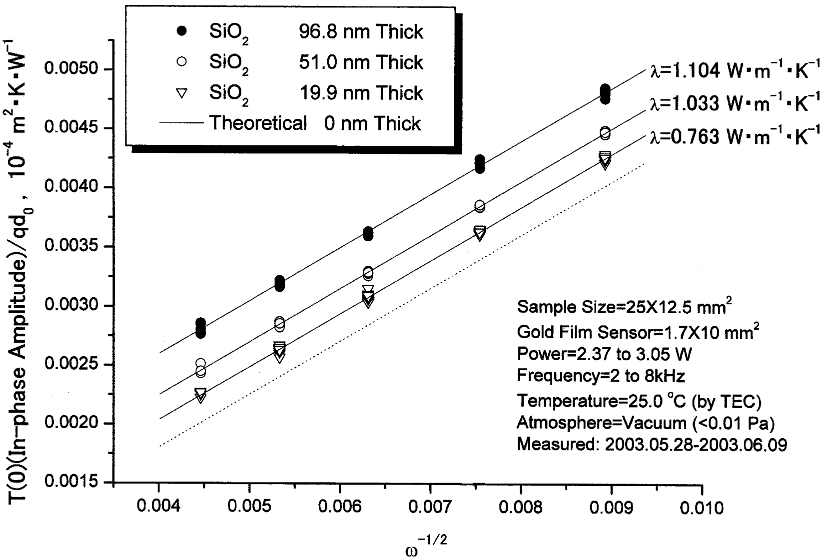

Fig. 5. $T(0)$ (In-phase Amplitude)/qd_0 vs. $\omega^{-1/2}$ plot for the measurement of the thermally-oxidized SiO_2 films (KST samples) on a silicon wafer by using the gold-film sensor.

thermal conductivity of the bismuth film was determined to be about 15 to 20% of that of the bulk material.

Figure 4 shows the results of the measurement of the thermally-oxidized SiO_2 films on a silicon wafer. These samples have been used in the NIST Round Robin (1998) [6,7]. In this case a gold film is deposited on the specimen by sputtering. The plot showed good linearity in the frequency range from 2000 to 8000 Hz. Calibration factors of thermoreflectance were determined using the known thermal effusivity of the silicon wafer. The thickness of the gold film was determined to be about 100 nm by a surface profiler (Sloan, Dektak 3030). The thermal conductivity of the gold film was determined to be about 56% of that of the bulk material by the measurement with the ac-calorimetric method [8]. By subtracting the theoretically-obtained value of the third term of Eq. (3) from the experimentally-obtained value of the intercept, the apparent thermal resistance of the thermally-oxidized SiO_2 films, R_1^* was determined. Then according to Eq. (5), the true thermal conductivity of the thermally-oxidized SiO_2 films was determined. Figure 5 shows the results of the thermal conductivity measurement of similar samples of the thermally oxidized SiO_2 films with smaller thicknesses on a silicon wafer. These samples were supplied by KST Corp. In Figs. 4 and 5, many dataset are plotted

Table II. Results of Thermal Conductivity Measurements

Thickness (nm)	VAMAS TWA23 [7, 8] Three-omega data ($W \cdot m^{-1} \cdot K^{-1}$)	VAMAS TWA23 [7, 8] All data ($W \cdot m^{-1} \cdot K^{-1}$)	Present results ($W \cdot m^{-1} \cdot K^{-1}$)
1000	–	–	1.23 ± 0.01
500	1.26 ± 0.07	1.20 ± 0.15	1.18 ± 0.03
200	1.27 ± 0.21	1.19 ± 0.22	1.14 ± 0.03
100	1.10 ± 0.19	1.15 ± 0.18	1.10 ± 0.03
50	0.80 ± 0.29	1.01 ± 0.63	1.03 ± 0.05
20	–	–	0.77 ± 0.05

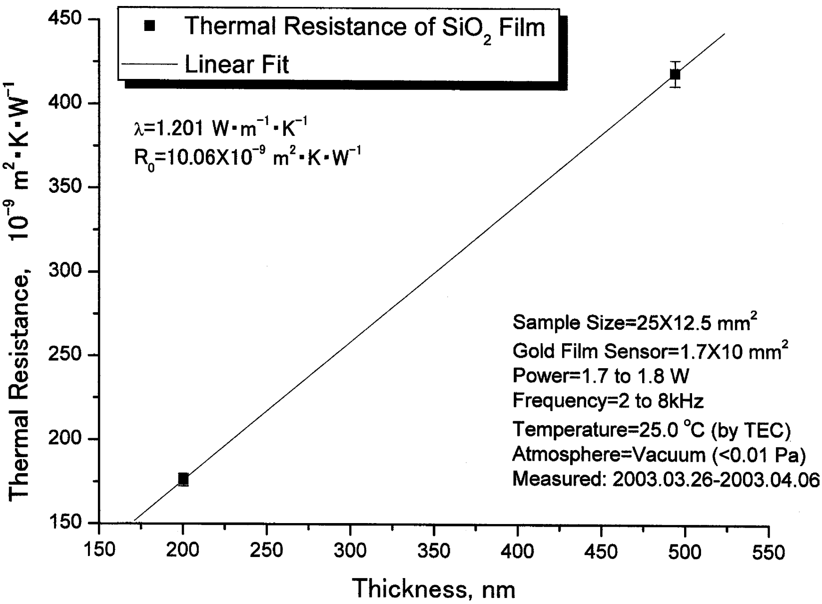

Fig. 6. Coefficients of the straight-line fit for the thermally-oxidized SiO_2 films (NIST RR samples) on a silicon wafer using a gold-film sensor.

together showing the reproducibility of the measurements for the same piece of the specimen at each frequency. Results of the thermal conductivity measurements are shown in Table II together with literature values. In Table II, the uncertainties of the present data were estimated from the standard deviations of the intercept of the straight-line fits shown in Figs. 4 and 5. Figure 6 shows the coefficient of the straight-line fit for the results of the NIST Round Robin samples. Figure 7 shows the coefficient of the straight-line fit for the results of the KST samples. Results for the

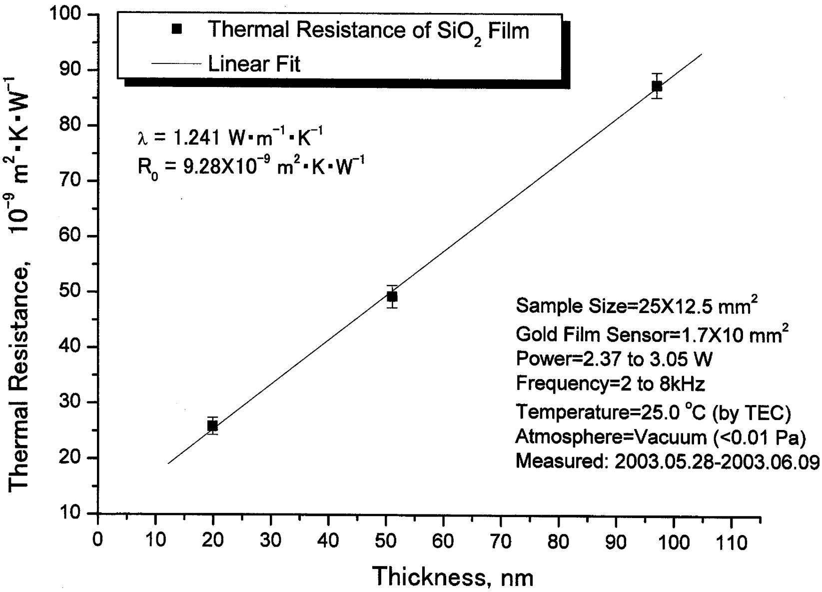

Fig. 7. Coefficients of the straight-line fit for the thermally-oxidized SiO_2 films (KST samples) on a silicon wafer using a gold-film sensor.

Table III. Coefficients of Straight-Line Fits.

	VAMAS TWA23 Three-omega data of NIST RR samples	Present data of NIST RR samples (500–200 nm)	Present data of KST samples (100–20 nm)
λ ($W \cdot m^{-1} \cdot K^{-1}$)	1.38 ± 0.05	1.21 ($\pm$ 10%)	1.24 ± 0.04 ($\pm$ 10 %)
R_0 ($10^{-9}\,m^2 \cdot K \cdot W^{-1}$)	22.90 ± 8.82	10.06	9.28 ± 1.44

Results for λ obtained from the slope and R_0 from the intercept of the straight-line fit of present data, listed in Table II, are shown.

coefficients, λ and R_0, are listed in Table III together with literature values. In Table III, the uncertainties of the present data were estimated from the standard deviations of the intercept of the straight-line fits shown in Figs. 6 and 7. The present results of the thermal conductivity measurements showed good agreement with literature values (VAMAS TWA23) within $\pm$10% [6,7].

5. SUMMARY

(1) In the present method, the metal film layer, which is deposited on the thin insulating films, is Joule-heated periodically and the

ac-temperature response at the center of the metal film surface is measured by a thermo-reflectance technique.

(2) The one-dimensional thermal conduction equation of the three-layered thermal-conductivity measurement system, including the metal-film layer, was solved analytically.

(3) In the present method, the calibration factors of the thermo-reflectance are determined using the known thermal effusivity of the substrate.

(4) The present method was verified with measurements on thermally-oxidized SiO_2 films (500 and 200 nm thick) on a silicon wafer, which have been used in the NIST Round Robin study of 1998.

(5) The present results of the thermal conductivity measurement show good agreement with literature values (VAMAS TWA23) within ±10%.

(6) The present method can provide more accurate values with much simple preparation of the specimen in comparison with the three-omega method.

NOMENCLATURE

Notation

C: specific heat capacity per unit volume ($J \cdot m^{-3} \cdot K^{-1}$)
d: thickness (m)
k: reciprocal of the thermal diffusion length (m^{-1})
λ: thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)
ω: angular frequency (s^{-1})
q: Power per unit volume ($W \cdot m^{-3}$)
R: thermal resistance ($m^2 \cdot K \cdot W^{-1}$)
T(0): ac-temperature of the surface of the metal film (K)

Subscripts

0: metal film layer
1: thin film layer
S: substrate layer

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