Thermal Conductivity Measurement of Fused-Silica Films Deposited on the Silicon Wafer by Using a Thermo-Reflectance Technique

R. Kato[2, 4] and I. Hatta[3]

[1] Paper presented at the Fifteenth Symposium on Thermophysical Properties, June 22-27, 2003, Boulder, Colorado, U.S.A.

[2] Ulvac-Riko Inc, 1-9-19 Hakusan, Midoriku, Yokohama 226, Japan

[3] Fukui University of Technology, 3-6-1 Gakuen, Fukui 910-0028, Japan

[4] To whom correspondence should be addressed.

ABSTRACT

Technology in microelectronics requires the development of methods of measuring normal-to-plane thermal conductivities of materials in the form of very-thin films deposited on the substrate. 3-omega method has been developed and shown to provide accurate values of thermal resistance and derived thermal conductivity of very thin films deposited on the substrate. However this method is not yet standardized, due to the practical difficulties in experimental setup. So some new techniques to improve the measurement method may be needed. This paper describes development of a new and improved method to measure normal-to-plane thermal conductivity of very thin films deposited on the substrate. In this method the metal film is used not only for periodic Joule's heating, but also for thermo-reflectance sensor for measuring ac temperature response. One dimensional thermal conduction equation of the three layered thermal conductivity measurement system, including the metal film layer, was solved analytically. Calibration factors of the thermo-reflectance were determined by using the value of the known thermal effusivity of the substrates. The present method was verified by using the specimen of the fused silica films (500 nm and 200 nm thick) deposited on the silicon wafer, which have been used in the NIST Round Robin (1998). The present results showed good agreement with the results of the 3-omega method within ±3-4 %.

1. INTRODUCTION

Technology in microelectronics requires the development of methods of measuring thermal conductivities of materials in the form of very-thin films deposited on the substrate. 3-omega method has been developed by D.G. Cahill et.al and shown to provide accurate values of thermal resistance and derived thermal conductivity of very thin films deposited on the substrate in normal-to-plane direction. [1] However this method is not yet standardized, probably due to the practical difficulties in experimental setup. So some new techniques to improve the measurement method may be needed. This paper describes development of a new and improved method to measure normal-to-plane thermal conductivity of very thin films deposited on the substrate. Characteristic of the method is that a metal film deposited on the specimen is used not only for periodic Joule's heating, but also for the thermo-reflectance sensor to measure ac-temperature response. [2] One dimensional thermal conduction equation of the three layered thermal conductivity measurement system, including the metal film layer, was solved analytically. In this method, calibration factors of the thermo-reflectance can be determined by using known thermal effusivity of the substrates. The present method requires much easier operation in preparation of the specimen in comparison with the 3-omega method. The present method was verified by using fused silica films (500 nm and 200 nm thick) deposited on the silicon wafer, which have been used in the NIST Round Robin (1998). [3, 4]

2. THEORETICAL CONSIDERARTIONS

In Fig.1 thermal conductivity measurement system of the present method is shown schematically. As shown in Fig.1, it consists of three layers, which are the metal film layer, the thin film layer, and the substrate layer. In the system, the metal film is Joule's heated periodically and ac-temperature response of the surface of the metal film is measured by a thermo-reflectance technique. Although the metal film is ac-heated uniformly, some ac-temperature gradient along the thickness of the metal film layer may take place. In the system, the substrate layer has infinite thickness, but the metal film layer and the thin film layer have finite thicknesses respectively. One dimensional thermal conduction equation of the system in normal-to-plane direction was solved analytically. The solution of the thermal conduction equation is given as Eq. (1). Where, q denotes the heat per unit volume (Wcm^{-3}), and d_0 and d_1 denotes thickness of metal film layer and the thin film layer respectively. When Eq. (2a) and Eq. (2b) are valid, Eq. (1) can be simplified as Eq. (3). The first term of Eq. (3) is proportional to the $\omega^{-1/2}$ and the factor of proportionality is a function of the thermal effusivity of the substrate $\lambda_S C_S$, but is not of the thermophysical properties of any other layers. On the other hand, the second term and the third term of Eq. (3) are real constants independent of $\omega^{-1/2}$.

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

$$-cosh[(1+i)k_0 d_0]-\frac{\lambda_0 k_0}{\lambda_1 k_1}sinh[(1+i)k_0 d_0])^{-1}\} \qquad (1)$$

$$k_0\, d_0 \ll 1 \qquad (2a)$$

$$k_1\, d_1 \ll 1 \qquad (2b)$$

$$\frac{T(0)}{qd_0}=\frac{exp(-\frac{\pi}{4}i)}{\sqrt{\lambda_S C_S \omega}}+(1-\sqrt{\frac{\lambda_1 C_1}{\lambda_S C_S}})\frac{d_1}{\lambda_1}+(\frac{1}{2}-\frac{\lambda_0 C_0}{\lambda_S C_S})\frac{d_0}{\lambda_0} \qquad (3)$$

$$\sqrt{\frac{1}{\lambda_1}}=\sqrt{\frac{R_1\times}{d_1}+\frac{C_1}{4\lambda_s C_s}}+\sqrt{\frac{C_1}{4\lambda_s C_s}} \qquad (4)$$

The plot of the "*In-phase Amplitude of* $T(0)/qd_0$ *vs.* $\omega^{-1/2}$" gives a slope-intercept form. Thermal effusivity of the substrate $\lambda_S C_S$ can be determined from the gradient of the form, and the sum of the second term and the third term can be determined from the intercept of the form. To obtain absolute values, calibration of the thermo-reflectance should be made using some temperature-reference at lower frequencies. In many cases, the calibration factors of the thermo-reflectance can be determined using known thermal effusivity of the substrate.

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

The value of the third term of Eq. (3) can be determined theoretically using known thermophysical properties of the substrate and the metal film. The value of 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 term and the third term. The expression of the second term of Eq. (3) is a quadratic of $\lambda^{-1/2}$. Defining the second term of Eq. (3) as R_1^*, the quadratic formula of $\lambda^{-1/2}$ is given as Eq. (4). Finally by substituting R_1^* of Eq. (4) by the experimentally-obtained value the second term of Eq. (3), thermal conductivity of the thin film can be determined.

3. EXPERIMENTAL

In Fig.2 block diagram of the experimental setup is shown. The specimen is set horizontally on the sample stage. The TEC system keeps the sample stage at constant temperature (at 25 °C). Whole sample assembly is installed in the vacuum chamber. At the top of the chamber above the sample assembly an optical window is provided to enable thermo-reflectance measurement. [5] The size of the specimen is 25×12.5 mm. The metal film is stripe-shaped with a dimension of 1.5 mm wide, 10 mm long and 100–200 nm thick. The metal film is deposited on the specimen by evaporation method or sputtering method, using a mask made of stainless steel with a thickness of 0.1 mm. Thickness of the metal film is determined by the surface profiler (Sloan, Dektak3030). At both ends of the metal film, a pare of electrodes of silver paste are printed to make good electrical contact with the four-pins spring contactors for supplying ac-current and detecting ac-voltage. It was found that of the bismuth film has quite big value (about 900 ppmK^{-1}) of the temperature coefficient of the reflectivity. As the noise of the thermo-reflectance measurement system is less than 0.01ppm (at 500 Hz), so the temperature-equivalent-noise of the bismuth-based system is less than 1.1×10^{-5}K (at 500 Hz). 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 photodiodes sensor is plugged-in to the high-impedance-port of the lock-in amplifier to measure the second harmonics of the signal.

4. RESULTS AND DESCUSSION

Condition of the specimen and the results of the present study are shown together in Table.1. [6] Fig.3 shows the results of the measurement of sapphire substrate with the bismuth-film sensor deposited on it. The plot showed good linearity in the frequency range from 500 to 8000 Hz. In this case calibration factors of the thermo-reflectance were determined using known thermal effusivity of the sapphire substrate. Thickness of the metal film was determined to be about 150 nm by the surface profiler (Sloan, Dektak3030). Thermal conductivity of the bismuth film was estimated to be about 25-50 % of that of the bulk material by the measurement electrical conductivity. The experimental results of the intercept coincided with the theoretically-obtained value of the third term of Eq. (4).

Fig.4 shows the results of the measurement of the fused silica film deposited on the silicon wafer. The specimens have been used in the NIST Round Robin (1998). In this case gold film is deposited on the specimen by using sputtering method. The plot showed good linearity in the frequency range from 2000 to 8000 Hz. In this case calibration factors of thermo-reflectance were determined using known thermal effusivity of the silicon substrate. Thickness of the gold film was determined to be about 100 nm by the surface profiler (Sloan, Dektak3030). Thermal conductivity of the gold film was determined to be about 56 % of that of the bulk material by the measurement of the ac-calorimetric method. By subtracting the theoretically-obtained value of the third term of Eq. (3) from the experimentally-obtained value of the intercept, apparent thermal resistance of the fused silica film, R_1^* was determined. Then according to Eq.

(4), true thermal conductivities of the fused silica film were determined. The results of the thermal conductivity of the fused silica film coincide with the results of the 3-omega method within 3-4 %.

5. SUMMARY

(1) In the present method the metal film is used not only for periodic Joule's heating, but also for thermo-reflectance sensor to measure ac temperature response.

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

(3) In many cases the calibration factors of the thermo-reflectance can be determined using known thermal effusivity of the substrates.

(4) The present method was verified by using the fused silica films (500 nm and 200 nm thick) deposited on the silicon wafer, which have been used in NIST Round Robin (1998).

(5) The present results showed good agreement with the results of 3-omega method within ±3-4 %.

NOMENCLATURE

q: Power per unit volume

k: Reciprocal of the thermal diffusion length

C: Specific heat capacity per unit volume

λ: Thermal conductivity

ω: Angular frequency

Suffix $_{0,1,S}$: denotes metal film, thin film, and, substrate layer respectively

REFERENCES

1. S.M.Lee, D.G.Cahill, *J. Appl. Phys.* **81**: 2950 (1997).
2. R. Kato, and I. Hatta, *Proc. 23st Jpn. Symp. Thermophysic. Prop.* : 265-267 (2002).
3. T.Yamane, S.Katayama, M.Todoki, *Proc. 21st Jpn. Symp. Thermophysic. Prop.* :16-18 (2000).
4. A.Feldman: "Results of a Round Robin on Thin Film Thermal Conductivity", *Proc. 14th STPP, Boulder, CO, June* (2000).
5. R. Kato, A. Maesono, and I. Hatta, *Proc. 21st Jpn. Symp. Thermophysic. Prop.* 10-12 (2000).
6. *"Thermophysical Properties Handbook", (Japanese Thermophysical Society), Yokendo, Ltd.* (1990).

(Figure captions)

Fig.1 Thermal conductivity measurement system of the present method

The system consists of three layers, which are the metal film layer, the thin film layer, and the substrate layer. In the system, the metal film is Joule's heated periodically and ac-temperature response of the surface of the metal film is measured by a thermo-reflectance technique.

Fig. 2 Block diagram of the experimental setup

The specimen is set horizontally on the stage. The TEC system keeps the specimen stage at constant temperature (at 25 °C).

Fig. 3 *In-phase Amplitude / Power vs.* $\omega^{-1/2}$ plot of the measurement of sapphire substrate with the bismuth-film sensor

Fig.4 *In-phase Amplitude / Power vs.* $\omega^{-1/2}$ plot of the measurement of the fused silica films deposited on the silicon wafer with the gold-film sensor

Table 1 Condition of the specimen and the results of the present study

Table 1 Condition of the specimen and the results of the present study

(*1) These samples were used in the NIST Round Robin (1998).

(*2) Present results

Sample	Specific Heat Capacity per unit Volume ($Jcm^{-3}K^{-1}$)	Thickness (mm, nm)	Thermal Conductivity ($Wcm^{-1}K^{-1}$)
Silicon	1.66	0.6 mm	1.48 [6]
Sapphire	3.20	0.2 mm	0.46 [6]
Bismuth (Bulk)	1.19 [6]		0.08 [6]
Bismuth film		150 nm	0.02-0.04 (*2)
Gold (Bulk)	2.47 [6]		3.15 [6]
Gold film		100 nm	1.78 (*2)
Fused Silica Film(*1)	1.63 [6]	200.4 nm	0.0141(*2)
		493.5 nm	0.0137(*2)

Fig.1 Thermal conductivity measurement system of the present method

The system consists of three layers, which are the metal film layer, the thin film layer, and the substrate layer. In the system, the metal film is Joule's heated periodically and ac-temperature response of the surface of the metal film is measured by a thermo-reflectance technique.

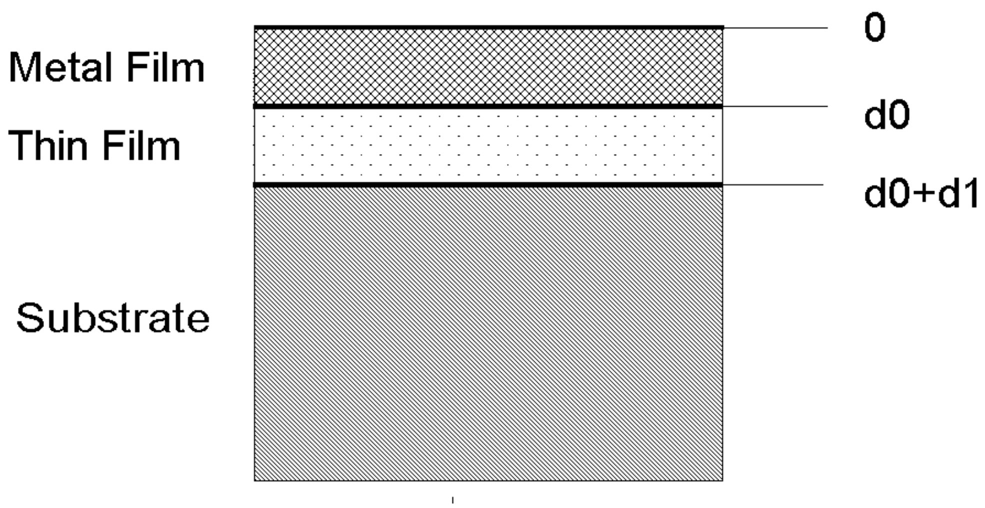
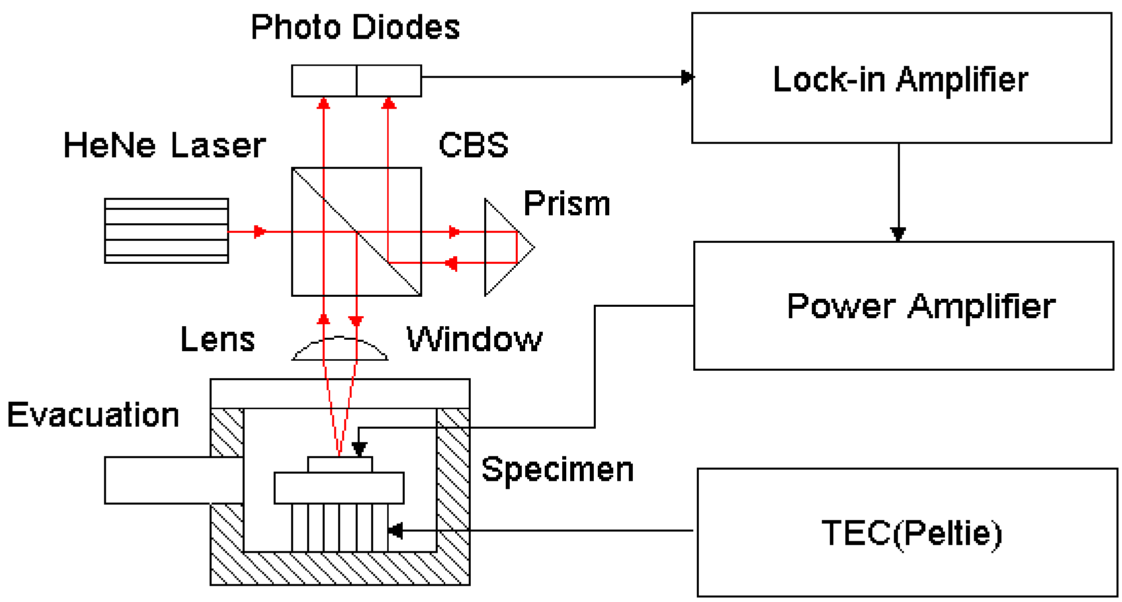


Fig. 2 Block diagram of the experimental setup. The specimen is set horizontally on the stage. The TEC system keeps the specimen stage at constant temperature (at 25 °C).



.

Fig. 3 *In-phase Amplitude / Power vs.* $\omega^{-1/2}$ plot of the measurement of sapphire substrate with the bismuth-film sensor.

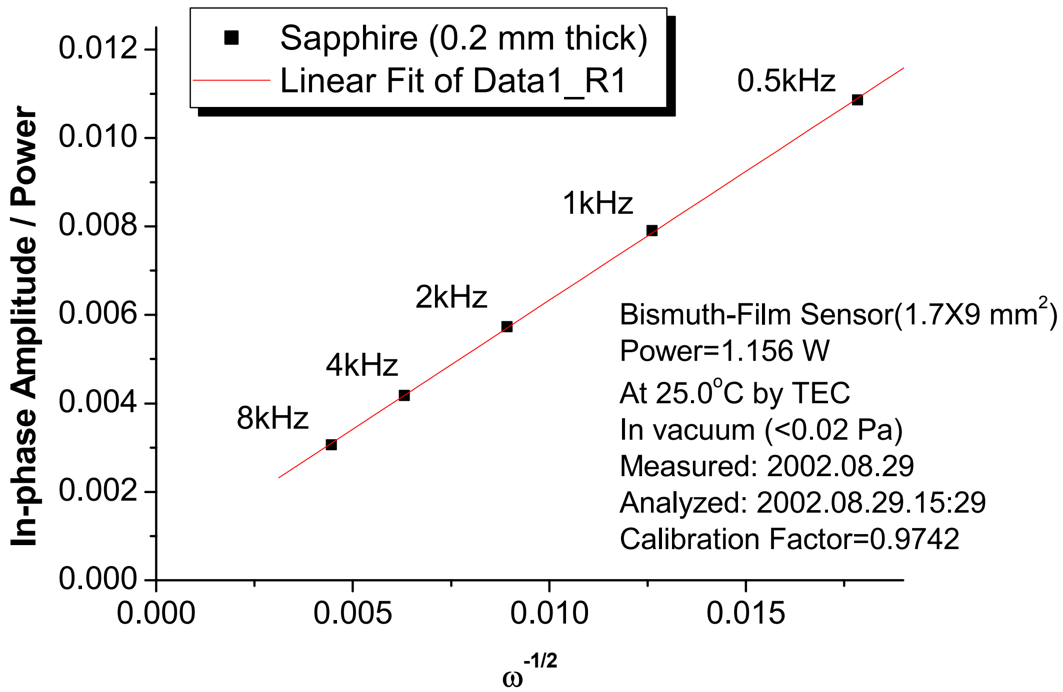


Fig.4 *In-phase Amplitude / Power vs.* $\omega^{-1/2}$ plot of the measurement of the fused silica films deposited on the silicon wafer with the gold-film sensor