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Thermal Conductivity Measurement of Submicron-Thick Films Deposited on Substrates by Modified ac Calorimetry (Laser-Heating Ångstrom Method)[1]

R. Kato,[2] A. Maesono,[2] and R. P. Tye[3,4]

Technological development, especially in microelectronics, necessitates the development of new and improved methods for measuring the thermal properties of materials, especially in the form of ultrathin films. Previously, modified ac calorimetry (laser-heating Ångstrom method) using a scanning laser as the energy source was developed and shown to provide accurate values of thermal diffusivity and derived thermal conductivity for a broad range of materials in the form of free-standing thin sheets or films, wires including fiber bundles, and some films on substrates. This paper describes further applications of the modified ac-calorimetry technique for measurements of the thermal conductivity of thin films deposited on substrates. It was used to measure successfully the thermal conductivities of 1000- to 3000-Å-thick aluminum nitride films, aluminum oxide films, etc., which were deposited on a glass substrate. It was also shown to be suitable for developmental measurements on submicron-thick chromatic films deposited on a PET substrate, which are photothermal recording layers, used in the media of CD-R drives of computer systems.

KEY WORDS: ac calorimetry; aluminum nitride; aluminum oxide; Ångstrom method; CD-R; thermal conductivity; thermal diffusivity; thin film.

1. INTRODUCTION

In many high-power density electronic components, many materials are used in the form of single and composite thin films, many of which may

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[2] Research and Development, Ulvac Sinku-Riko, Inc., 1-9-19 Hakusan, Midoriku, Yokohama 226, Japan.

[3] Hunters Lodge, Wells Road, Draycott, Cheddar, Somerset BS27 3ST, United Kingdom.

[4] To whom correspondence should be addressed. E-mail: kato@ulvac-riko.co.jp

have thermal conductivities that differ from those of bulk materials due to their reduced thickness. These issues require that thermal properties be measured on the particular form and under conditions in which the material is applied. This requires the use of newer measurement methods than those that have been used conventionally for bulk materials.

Modified ac calorimetry (laser-heating Ångstrom method) using a scanning laser as the energy source has been shown to provide accurate values of thermal diffusivity and derived thermal conductivity for a broad range of materials in the form of free-standing thin sheets or films, wires including fiber bundles, and some films on substrates [1, 2]. The method has been verified by using standard reference materials of 500- to 700-μm thickness, which are also measurable by other traditional methods such as the laser flash method and the guarded-hot-plate method [3, 4].

This technique has now been applied to measurements of the thermal conductivity of submicron-thick films deposited on a substrate from the measurement of the apparent thermal diffusivity of the composite and of the thermal diffusivity of the substrate alone. The major advantages of this technique are as follows.

(a) The sensitivity of the measurements of thermal conductivity of deposited films becomes higher as both the thermal conductivity and the thickness of the substrate are reduced. The method is applicable for any thermal conductivity and film thickness, and measurements can be made without error due to heat loss from the surface of the specimen.

(b) The precision of the thermal diffusivity of the free-standing specimen has to be much higher than that of the required precision for the thin film. This method is capable of measurements on a free-standing specimen with a precision of $\pm 0.3\%$.

(c) As there are no standard reference materials for submicron films, the method must be applicable not only to thin films, but also to thick films. The method is applicable for a broad range of thicknesses, from 1 to 700 μm.

It has been found that commercially available glass sheets do not have overall homogeneity throughout a whole sheet or lot, and as a result, the thermal conductivity can differ by 3%. This uncertainty of the thermal conductivity can cause large errors in the measurement of composite films. To solve this problem, a differential method has been developed in which the thin film is deposited on a half-surface of the rectangular substrate to ensure the homogeneity of the substrate material between the deposited region and the nondeposited region [5].

The method has two major applications for thermal conductivity measurement of thin films deposited on substrates. The first case is that of the thermal conductivity measurement of electrical-insulating layers in electronic devices, such as AlN films and Al_2O_3 films used in the MR or GMR sensor head of the hard disk drive of a computer system. In this case, the normal-to-plane thermal conductivity is essentially important and its thermal conductivity is required to be as high as possible to obtain good heat dissipation. The second case is that of the thermal conductivity measurement of thin films, which are photothermal recording layers, used in the media of CD-R, CD-RW, DVD, and MO drives of computer systems. In this case, the in-plane thermal conductivity is essentially important and its thermal conductivity is required to be as low as possible to obtain a higher density of memory. The present method is most suitable for the latter case, because it can be used to measure the in-plane thermal diffusivity and/or thermal conductivity. Furthermore, in the latter case, plastic substrates are commonly used, and these have a much lower thermal conductivity than glass, providing a potentially higher sensitivity of thermal conductivity measurement of thin films deposited on the substrates.

2. THEORETICAL CONSIDERATIONS

The principle of the measurement method is shown schematically in Fig. 1. A portion of a rectangular film specimen is heated by a modulated laser beam, which is a line heat source, made by scanning the laser spot rapidly at a constant speed in the width direction of the specimen [6, 7]. Temperature waves, which propagate one-dimensionally in the long direction of the specimen, are produced. As the irradiated portion of the specimen

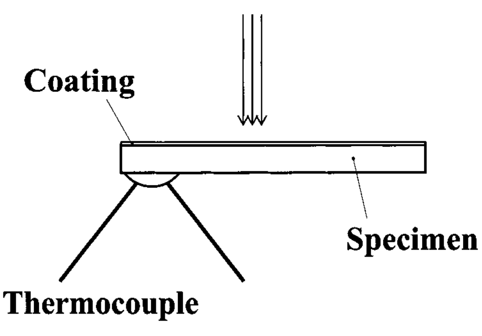

Fig. 1. Schematic view of the setup of a free-standing specimen.

x changes, the ac temperature response is measured by a small thermocouple attached at the opposite face of the specimen [8, 9]. By analyzing the amplitude decay and the phase shift of the ac temperature as a function of the spatial distance between the irradiated portion and the temperature sensor, the logarithmic decrement of the amplitude, k_{a} (reciprocal of the diffusion length), and phase shift increment, k_{p} (wavelength), can be obtained.

In the special case when no heat loss from the surfaces exists, k_{a} and k_{p} coincide with each other and are equal to k. However, in the general case when heat loss from the surfaces exists, k_{a} and k_{p} do not coincide with each other and are not equal to k, but the square root of the product of both values is equal to k as shown in Eq. (2). By using k_{a}, k_{p}, and the frequency, f, according to Eqs. (1) and (2), it is possible to determine the true thermal diffusivity, D, of the specimen [10, 11]. By selecting the appropriate frequency, the thermal diffusivity of a wide variety of film materials, from CVD diamond to polymers, with a thickness of 1 to 700 μm can be obtained.

$$k = \sqrt{\frac{\pi f}{D}} \tag{1}$$

$$k = \sqrt{k_{\mathrm{a}} k_{\mathrm{p}}} \tag{2}$$

A schematic of the setup for a thin film deposited on a half-surface of the rectangular substrate is shown in Fig. 2. The apparent thermal diffusivities of the deposited region (thin film + substrate), D_{012}, and the non-deposited region (substrate), D_{01}, are given by Eqs. (3) and (4), respectively.

$$D_{012} = \frac{D_0 C_0 d_0 + D_1 C_1 d_1 + D_2 C_2 d_2}{C_0 d_0 + C_1 d_1 + C_2 d_2} \tag{3}$$

$$D_{01} = \frac{D_0 C_0 d_0 + D_1 C_1 d_1}{C_0 d_0 + C_1 d_1} \tag{4}$$

where suffixes 0, 1, and 2 denote the coating layer, substrate layer, and thin-film layer, respectively. By subtracting Eq. (4) from Eq. (3), Eq. (5) is obtained. Then the thermal conductivity of the thin film, λ_2, is given by Eq. (6).

$$(D_{012} - D_{01})(C_0 d_0 + C_1 d_1) + D_{012} C_2 d_2 = D_2 C_2 d_2 \tag{5}$$

$$\lambda_2 = D_2 C_2 = D_{01} C_1 \left\{ \frac{C_2}{C_1} + \left(\frac{C_2}{C_1} + \frac{C_0 d_0}{C_1 d_2} + \frac{d_1}{d_2} \right) \left(\frac{D_{012}}{D_{01}} - 1 \right) \right\} \tag{6}$$

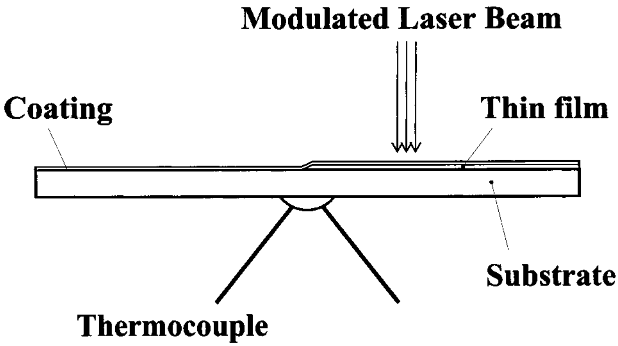

Fig. 2. Schematic view of the setup of the differential method.

In general, the specific heat capacities per unit volume of solids do not differ significantly from each other and Eqs. (7a) and (7b) are valid.

$$0.7 < \frac{C_2}{C_1} < 1.3 \tag{7a}$$

$$0.7 < \frac{C_0}{C_1} < 1.3 \tag{7b}$$

Assuming that the thicknesses of the layers are related as shown in Eqs. (8a) and (8b) (true for most practical cases), Eq. (6) can be reduced to Eq. (9).

$$\frac{d_0}{d_2} < 1 \tag{8a}$$

$$\frac{d_1}{d_2} > 100 \tag{8b}$$

$$\lambda_2 = D_{01} C_1 \left\{ 1 + \frac{d_1}{d_2} \left(\frac{D_{012}}{D_{01}} - 1 \right) \right\} \tag{9}$$

The error in λ_2 due to the approximation is estimated to be less than $\pm 1\,\%$. It should also be noted here that the coating layer has a negligible effect.

The thermal diffusivities of the deposited region (thin film + substrate), D_{012}, and the nondeposited region (substrate), D_{01}, can be determined by the measurement of a free-standing specimen. The thicknesses of the substrate and the thin film, d_1 and d_2, can also be determined by other means.

If the specific heat capacity per unit volume, C_1, of the substrate and the ratio of the specific heat capacity per unit volume, C_2/C_1, are known, then the thermal conductivity of the thin film, λ_2, can be determined. It should be noted here that the required accuracy for the determination of the thicknesses of the substrate and the thin film, d_1, and d_2, and the specific heat capacity per unit volume, C_1, is the same as that required for the determination of the thermal conductivity of the thin film.

The specific heat capacity per unit mass of a solid is the same regardless of its crystalline or other state, but the densities can differ depending on the amorphous or crystalline state and other factors. Assuming that there is no difference of chemical composition between the bulk material and the thin film and that there are no significant vacancies in the thin film, the specific heat capacity per unit mass of the thin film will be same as that of the bulk material. Provided that the density of the thin film can be determined by some technique, then its specific heat capacity per unit volume can be determined as the product of the measured density and the specific heat capacity per unit mass of the bulk material.

As mentioned earlier, specific heat capacities per unit volume of solids do not differ significantly and C_2/C_1 is equal to 1 with an uncertainty less than $\pm 30\%$. When the thermal conductivity, λ_2, of a thin film is much higher than that of the substrate, it can be determined with its specific heat capacity per unit volume unknown.

On the other hand, the sensitivity of the measurement of thermal conductivity, λ_s depends on the thermal conductivity of the substrate, the thickness ratio, d_1/d_2, and the precision of the thermal diffusivity measurement of a free-standing specimen. At present, the precision of the thermal diffusivity measurement is $\pm 0.3\%$. Thus, the sensitivity of the measurement of λ_s can be expressed by Eq. (10).

$$\lambda_s = D_{01} C_1 \left\{ 0.3 + \frac{d_1}{d_2} 0.003 \right\} \tag{10}$$

When the thickness ratio, d_1/d_2, is 100, the sensitivity of the thermal conductivity of a thin film is comparable with the expected maximum uncertainty of the specific heat capacity per unit volume of the thin film, which is $\pm 30\%$. When a borosilicate glass substrate is chosen, which has a specific heat capacity per unit volume of $1.80 \times 10^6\ \mathrm{J \cdot m^{-3} \cdot K^{-1}}$ and a thermal diffusivity of $5.6 \times 10^{-7}\ \mathrm{m^2 \cdot s^{-1}}$, it can be calculated as $\pm 0.3\ \mathrm{W \cdot m^{-1} \cdot K^{-1}}$. The sensitivity can also be expressed as the thermal conductance ($\mathrm{W \cdot K^{-1}}$) of the thin film, when the substrate material and its thickness are constant. When a borosilicate glass substrate with a thickness of 30 μm is selected, the sensitivity of the thermal conductance of a thin film is $\pm 90\ \mathrm{nW \cdot K^{-1}}$.

3. EXPERIMENTAL SYSTEM

The basic system and specifications have been described in detail previously, and only salient features are included here [3, 12]. The blackened surface (by using 1000-Å-thick bismuth or 1-μm-thick dry graphite film) of a test specimen of the order of 12.5 to 30 mm long and 2.5 to 5 mm wide with a thickness of 1 to 700 μm, with a thermocouple (0.1 mm diameter) fixed on the opposite surface, is irradiated by thermal energy from a laser diode source.

As shown schematically in Fig. 3 the modulated laser beam is focused on a spot on a 24-face polygonal mirror rotating at 6000 rpm and is concentrated into a 6- to 9-mm-long, 0.5-mm-wide strip using a scan lens. The resultant temperature signal is amplified using an input transformer and a low noise amplifier. The system is operated by a microcomputer connected to a personal computer with an RS232C interface. A firmware program performs a discrete Fourier transformation and averages the signal in the frequency range 0.01 to 10 Hz to provide the amplitude and phase data together with the spatial distance and temperature data to the personal computer. Analysis of the resultant data, delivered at a rate of 1 data set per 10 s, is carried out using firmware. The software, which runs on Windows 95/98/NT/2000, gives separate values of D_a^*, D_p^*, and D. For a measurement the thermal diffusion length is selected in the range of 1 to 3 mm according to the length of the specimen and frequency, f, set to this length by trial measurements. Actual measurements are made at frequencies of $f/2$, f, and $2f$ with data collected and averaged for a large number of data points

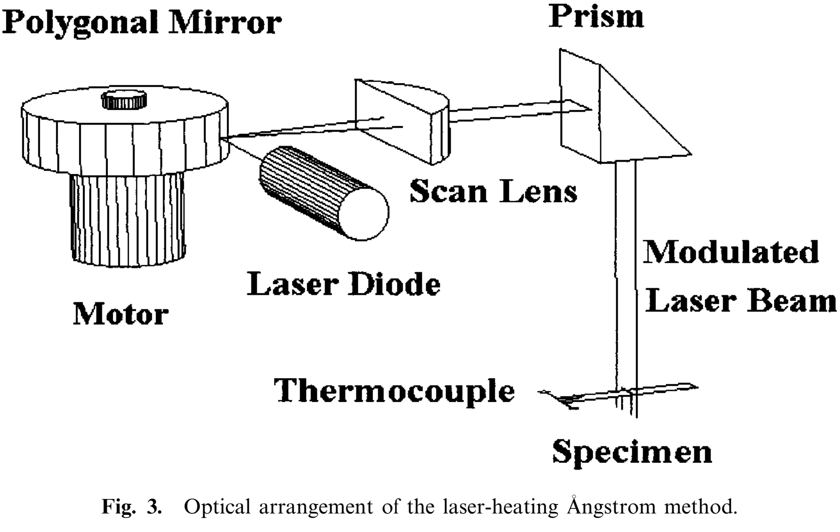

Fig. 3. Optical arrangement of the laser-heating Ångstrom method.

(more than 60) operating at a slow ramp rate. However, in the case when the frequency is lower than 0.05 Hz (for materials having very low thermal diffusivities and/or large thicknesses), measurements are undertaken at two discrete positions over long time intervals. In both cases the total measuring time is of the order of 10 to 20 min. For the highest precision it is preferable to use vacuum conditions and these are necessary where materials have thermal diffusivity values below approximately $5 \times 10^{-6}\ \mathrm{m}^2 \cdot \mathrm{s}^{-1}$ [13].

3. SPECIMEN PREPARATION

For the submicron-thick film of AlN and Al_2O_3, a specially designed specimen-holder frame, shown in Fig. 4, is used to keep the very thin glass substrate from being damaged. The substrate and frame materials are borosilicate glass of D263 having a thickness of 30 and 200 μm, respectively, supplied by DESAG (Schott Group). The substrate was machined to a width of 2.5 mm with a nonuniformity less than ±0.3% and it has a length of 12.5 mm. It is attached to the frame with a polyimide adhesive, and the whole specimen-holder frame is heat-treated up to 340°C in air. The aluminum nitride films and aluminum oxide films are then deposited on a half-surface of the subtrate. The conditions of the composite specimen are listed in Table I.

The specimen-holder frame was coated uniformly over the whole surface with 1000-Å-thick bismuth by evaporation. Every measurement was carried out at room temperature in a vacuum of less than 0.01 Pa, produced using a turbo-molecular pump, to eliminate the effect of air layers. Frequencies of ac-calorimetric measurements were selected in the range of 0.1 Hz, so that the diffusion length is much longer than twice the thickness of the specimen and shorter than 0.3 times the half-length of the specimen [14, 15].

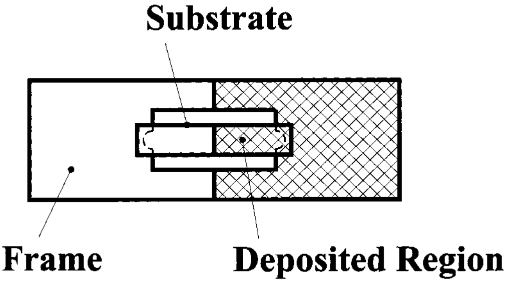

Fig. 4. The specimen-holder frame for a thin film deposited on the half-surface of a rectangular substrate.

Table I. Conditions of the Composite Specimen of AlN and Al_2O_3 Thin Films

Sample	Size (mm)	Thickness (μm)	Specific heat capacity ($J \cdot g^{-1} \cdot K^{-1}$)	Density ($g \cdot cm^{-3}$)	Supplier
Borosilicate glass (D 263)	2.5 × 10	30 ± 5	0.72 [19]	2.51	DESAG (Schott Group)
Aluminum nitride	2.5 × 10	0.1, 0.2, 0.3	0.71[a] [19]	3.26[a]	T. Morita et al. of Ulvac Japan Ltd.
Aluminum oxide	2.5 × 10	0.1, 0.2, 0.3	0.79[a] [19]	3.99[a]	

[a] These values assume that the materials are not highly porous or impure.

4. PROPERTIES OF THE FILM

Deposition of the AlN and Al_2O_3 films was accomplished by means of magnetron sputtering with an inductively coupled rf plasma with a sputtering target of aluminum (99.999%) and a gas partial-pressure ratio, N_2/Ar, of 1/33. The properties of the aluminum nitride (AlN) films have been investigated by T. Morita et al. [16] using film deposited on a silicon substrate by means of ellipsometry, RBS (Rutherford backward scattering), AES (Auger electron spectroscopy), XRD (X-ray diffraction), HRTEM (high-resolution transmission electron microscope), (I–V) electrical resistance, etc. [16].

The results of the ellipsometry indicated that the refractive index, n, was 2.05. The chemical compositions of the films determined by RBS are listed in Table II. In addition, the element ratio, N/Al, by AES was shown to be almost constant independent of the depth of the film. The (002) lattice plane spacing of AlN crystal was determined to be 0.2498 nm, which is very close to the ASTM value of 0.2490 nm by XRD.

5. RESULTS AND DISCUSSION

Experimental data are plotted in Fig. 5 illustrating the expected linear relationship for the logarithmic amplitude decay and phase shift as a function of the spatial distance. The homogeneity of the substrate was confirmed to be within ±0.3% by the measurement of specimen-holder frames on both

Table II. Chemical Composition of Aluminum Nitride (AlN) Films Determined by RBS

Substrate temperature	Element ratio N/Al	Yield Al at 200°C/yield Al at RT
RT	0.969	1.04 to 1.05
200°C	0.883	

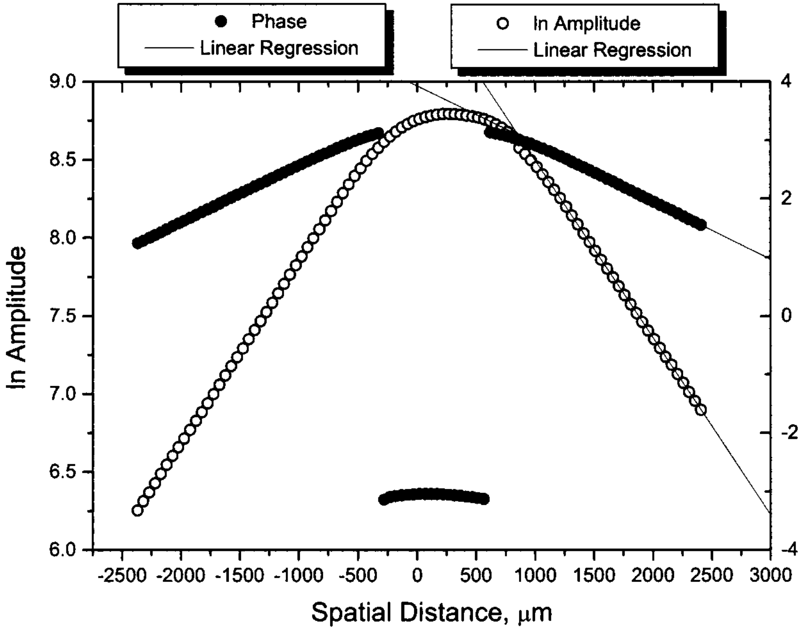

Fig. 5. Experimental data illustrating the expected linear relationship for logarithmic amplitude decay and phase shift, which are denoted as open and filled circles, respectively, as a function of the spatial distance.

regions. Results of the apparent thermal diffusivities of the deposited region (thin film + substrate), D_{012}, and the nondeposited region (substrate), D_{01}, at room temperature are listed in Table III. Derived thermal conductivities of the aluminum nitride (AlN) film and aluminum oxide film (Al_2O_3) are listed in Table IV. The results clearly indicate that the thermal conductivity of materials in the form of very thin (submicron) films is significantly lower than that of the bulk materials. However, there was very little difference

Table III. Results of the Apparent Thermal Diffusivities of the Deposited Region (Thin Film + Substrate), D_{012}, and the Nondeposited Region (Substrate), D_{01}, at Room Temperature

Thickness (Å)	D_{012} ($10^{-7}\,m^2 \cdot s^{-1}$)	D_{01} ($10^{-7}\,m^2 \cdot s^{-1}$)	Literature value of the substrate ($10^{-7}\,m^2 \cdot s^{-1}$) by TPRC [20]
1000	5.62 ± 0.017	5.54 ± 0.004	5.5 (reported error, ~15%)
2000	5.85 ± 0.010	5.64 ± 0.010	
3000	6.08 ± 0.015	5.67 ± 0.030	

Table IV. Derived Thermal Conductivity of Aluminum Nitride (AlN) and Aluminum Oxide (Al_2O_3) Thin Films Deposited on a Borosilicate Glass Substrate

Sample name	Thickness (Å)	Thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	Uncertainty ($W \cdot m^{-1} \cdot K^{-1}$)
Aluminum nitride	1000	5.6	±0.8
	2000	6.6	±0.6
	3000	8.4	±0.35
Aluminum oxide	1000	1.0	±1.2
	2000	3.3	±0.6
	3000	4.5	±0.45

between the values for AlN films and those for Al_2O_3 films, not withstanding in the case of bulk materials. For high-purity AlN and Al_2O_3, the bulk values are of the order of 180 to 250 and 30 $W \cdot m^{-1} \cdot K^{-1}$, respectively. These results indicate that the thin films are in an amorphous rather than a crystalline state. It was also shown that there is a thickness effect, indicating that the reduction of the value could possibly be related to the crystal size affecting contact resistance between particles and/or due to differences in the mean free path such that the scattering behavior is affected. It seems that the thermal conductivities for both AlN films and Al_2O_3 films are getting closer to a certain minimum value, which was proposed by Albert Einstein, as the film thickness approaches to zero [17, 18]. It should be noted here that the thermal contact resistance, which may exist between the thin film and the substrate, cannot be observed by the present method because it measures the in-plane thermal diffusivity.

A chromatic thin films with a thickness of 1000 or 2400 Å deposited on a half-surface of a PET (polyethylene terephthalate) substrate was also measured. This substance is a kind of cyanine dye and was made to be in an amorphous, but might be partially in a crystalline, state. The chromatic film is prepared by the spin-coating technique. This substance is used as a recording layer of CD-R media of computer systems. The conditions of the composite specimen are listed in Table V. Every measurement was also

Table V. Conditions of the Composite Specimen of Chromatic Thin Films

Sample	Size (mm)	Thickness (μm)	Specific heat capacity ($J \cdot g^{-1} \cdot K^{-1}$)	Density ($g \cdot cm^{-3}$)
PET	30 × 5	112 ± 2	1.18	1.6
No. 1	15 × 5	0.110	Unknown	Unknown
No. 2	15 × 5	0.240	Unknown	Unknown

Table VI. Derived Thermal Conductivity of Chromatic Thin Films Deposited on PET Substrate

Sample	Thermal conductivity $(\mathrm{W \cdot m^{-1} \cdot K^{-1}})$	Uncertainty $(\mathrm{W \cdot m^{-1} \cdot K^{-1}})$
No. 1	8.9	± 2.8
No. 2	6.9	± 0.4

carried out at room temperature in a vacuum of less than 0.01 Pa. Derived thermal conductivities are listed in Table VI. In this case no significant thickness dependence is observed. This result indicates that the films are primarily in an amorphous state.

On the other hand, thermal conductivities of the PET substrate are 0.54 to 0.59 $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$, which is much higher than the value for bulk (solid) materials. The thermal conductivity of the polyimide film (50-μm-thick Kapton-H, supplied by Dupont–Toray Co.) was also measured on six pieces or regions of the specimen. The polyimide film is also found to have a higher thermal conductivity $(0.74 \pm 0.02\ \mathrm{W \cdot m^{-1} \cdot K^{-1}})$ than literature values obtained on bulk (solid) materials. In these experiments, the effect of the bismuth layer was examined by making measurements at different thicknesses of the bismuth layer, and it was confirmed that it could be neglected. Assuming that the thermal conductivity of the bismuth is the same as that of the bulk material $(8.5\ \mathrm{W \cdot m^{-1} \cdot K^{-1}})$, it is possibly much lower; the effect is calculated to be less than 0.017 $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$. The effect of the air layers was also examined by making measurements at different pressures and was confirmed to be less than 1%. In general, plastic films seem to show anisotropy in their thermal conductivity due to the film-marking process.

Results of the derived thermal conductivity of a dielectric substance, $\mathrm{ZnSSiO_2}$ with a thickness of 2.0 μm, deposited on a half-surface of borosilicate glass (200-μm thick) is quite low (less than 0.3 $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$). This substance is used as protective layers which sandwich the recording layer in CD-RW media of computer systems.

6. SUMMARY

A modified ac-calorimetry technique has been used successfully to measure the thermal properties of submicron films deposited on a homogenous substrate by means of the differential method. The method is capable of measuring the thermal diffusivity of a free-standing specimen to a precision of $\pm 0.3\%$. Results for AlN and $\mathrm{Al_2O_3}$ films having thicknesses from 1000 to 3000 Å deposited on glass indicate that the thermal conductivity is significantly lower than that of the bulk material. However, there is very little difference between the values for the AlN films and those for Al_2O_3 films, notwithstanding the difference in values of the bulk materials. The low values indicate that thin films are in primarily an amorphous rather than a crystalline state. It was also shown that there is a thickness effect, indicating that the reduced value could possibly be related to the crystal size affecting the contact resistance between particles and/or due to a difference in the mean free path such that the scattering behavior is affected. It appears that the thermal conductivities for both AlN films and Al_2O_3 films are getting closer to a certain minimum value as the film thickness approaches zero. Results for a chromatic substance having thicknesses of 1000 and 2400 Å deposited on a PET substrate showed a low thermal conductivity but no thickness dependence.

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