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Differential 3ω method for measuring thermal conductivity of AlN and Si_3N_4 thin films

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A B S T R A C T

The thermal conductivity λ of plasma enhanced chemical vapor deposited Si_3N_4 and sputtered AlN thin films deposited on silicon substrates were obtained utilizing the differential 3ω method. A thin electrically conductive strip was deposited onto the investigated thin film of interest, and used as both a heater and a temperature sensor. To study the thickness dependent thermal conductivity of AlN and Si_3N_4 films their thickness was varied from 300 to 1000 nm. Measurements were performed at room temperature at a chamber pressure of 3.1 Pa. The measured thermal conductivity values of AlN and Si_3N_4 thin films were between 5.4 and 17.6 $Wm^{-1}\,K^{-1}$ and 0.8 up to 1.7 $Wm^{-1}\,K^{-1}$, respectively. The data were significantly smaller than that of the bulk materials found in literature (i.e., $\lambda_{AlN} = 250$–$285\ Wm^{-1}\,K^{-1}$, $\lambda_{Si_3N_4} = 30\ Wm^{-1}\,K^{-1}$), due to the scaling effects, and also strongly dependent on film thickness, but were comparable with literature for the corresponding thin films.

1. Introduction

Thin film technology has been developing rapidly in the past decades due to the advances in thin film deposition and characterization techniques, leading to increased and simultaneously decreased structure dimensions of microelectronic devices. As a result, alternate approaches for thermal management of thin films are frequently required in order to achieve a high efficiency and to ensure the reliability of fabricated devices. Thermal conductivity of thin films is an important fundamental material property since the ability to dissipate heat is often the limiting factor to determine the device performance. Therefore, materials with a high thermal conductivity are needed. Aluminum nitride (AlN) is one of the most promising candidates for effective heat conductors in microelectronic devices due to its high bulk thermal conductivity and thermal expansion coefficient which are close to silicon [1]. The bulk thermal conductivity of AlN is 200 times higher than those of silicon dioxide (SiO_2) and silicon nitride (Si_3N_4) [2,3]. However, thermal conductivity of thin films can be substantially different from that of bulk materials due to the scaling effects [1,4–6]. Therefore, precise determination of thermal conductivity for thin films is crucial for designing or analyzing microelectronic devices. Furthermore, the limited thermal conductivity data of AlN available in literature vary significantly from each other, which is mainly attributed to the differences in film preparation processes [1]. Currently, thermal conductivity of AlN films has been determined using a variety of measurement methods, such as laser ablation, AC calorimetric method and photothermal reflectance method [1,3,7–9]. Results using these methods indicate that the thermal conductivity of polycrystalline AlN films is strongly dependent on film thickness, defect density and oxygen content. Clearly, an appropriate method to determine the thermal conductivity of AlN and other dielectric thin films is required. This work reports an approach to measure the thermal conductivity λ of insulating thin film material AlN in the sub-micrometer range, in order to characterize the thickness dependency of the film thermal conductivity. The thermal conductivity of AlN thin films was measured using an improved differential 3ω method. In contrast to commonly used thermal conductivity measurement methods, such as laser flash [5], AC calorimetric method or photothermal reflectance, the differential 3ω method is insensitive to errors from black-body radiation because the effective thickness of the sample is extremely small [10]. Therefore, higher accuracy and better reproducibility of the film thermal conductivity data can be obtained.

2. Experimental details

In this work the thermal conductivity of AlN and Si_3N_4 thin films was determined by applying the differential 3ω method, which was originally developed by Cahill [10]. The 3ω measurement technique was evolved from conventional hot-wire techniques and is currently used to measure the thermal conductivity of dielectric thin films [1,5,11]. A thin metal strip, with a width of $2b$ and a resistance R_h, as

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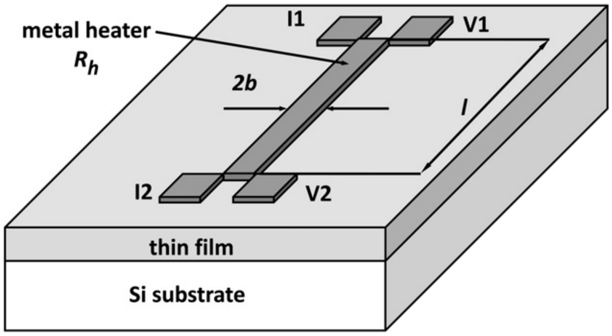

Fig. 1. Schematic layout of the four-pad test structure used to determine the thermal conductivity of a thin film by the differential 3ω method. A metal strip serves as both the heater and the thermometer. The four pads are the connections for current leads (I1, I2) and voltage leads (V1, V2). *l* is the distance between the current leads and *2b* is the strip width.

shown in Fig. 1, is deposited on top of the thin film sample for simultaneous operation as a heater and thermometer. An alternating current with an angular modulation frequency ω is driven through the metal strip causing Joule heating and induces a temperature oscillation $\Delta T(\omega)$ at a frequency of 2ω. This results in a voltage oscillation $\Delta V(\omega)$ along the heating resistor with a third harmonic which depends on the temperature oscillation of the heater. The temperature and voltage oscillations are the key parameters of the differential 3ω method. Thermal conductivity of a thin film can be obtained by comparing the temperature oscillation in a film-on-substrate structure with the corresponding value of the substrate. The temperature variation of the film-on-substrate structure is experimentally measured by detecting the voltage oscillation across the metal heater, which is proportional to the oscillating resistance value. The substrate temperature oscillation can be determined by [12]:

$$\Delta T = \frac{P}{\pi l \lambda_s}\left(\frac{1}{2}\ln\frac{4D}{b^2} + In2 - 0.5772 - \frac{1}{2}\ln(2\omega) - \frac{i\pi}{4}\right) \quad (1)$$

where λ_s is t D the thermal diffusivity of the substrate, P the power supplied to the metal strip, l the length of the metal strip and i the imaginary unit, respectively [12].

If the thin film thermal conductivity λ_f is much smaller than that of the substrate material and also the width $2b$ of the metal strip is much larger than the thickness d_f of the investigated film, the temperature shift induced by the thin film ΔT_f is given by [11]:

$$\Delta T_f = \frac{p\, d_f}{l \lambda_f 2b}. \quad (2)$$

The AlN and Si_3N_4 thin films were prepared on a p-doped Si-substrate of about 640 μm in thickness. The AlN thin films were deposited using a magnetron sputtering process, whereas Si_3N_4 films were deposited using plasma enhanced chemical vapor deposition. The AlN thin film was deposited on a single crystal Si (001) wafer by a radio frequency (rf) reactive magnetron sputtering process with an rf power of 5 kW and a dc power of 100 W. The ambient pressure and temperature in the deposition chamber were adjusted to be 133.3 Pa and 20 °C, respectively. An aluminum target

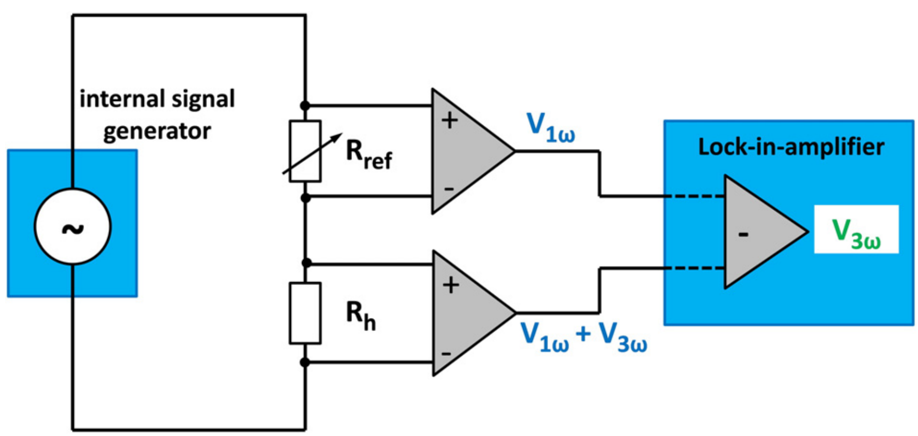

Fig. 2. Schematic circuit diagram used to extract the 3ω voltage component from the voltage signal across the metal strip deposited on the sample.

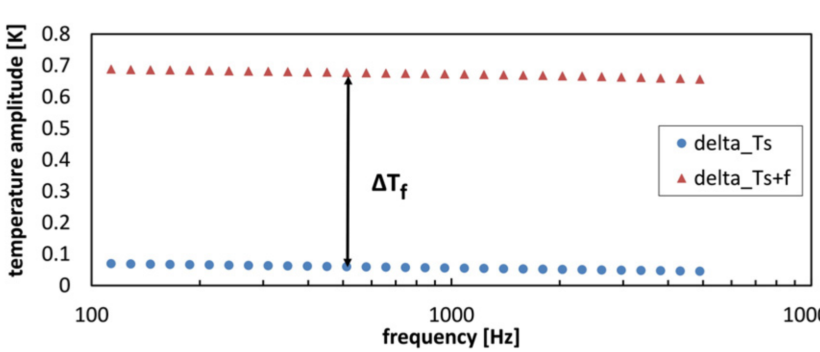

Fig. 3. The temperature amplitudes for the substrate and for the film-on-substrate structure for the 1001 nm Si_3N_4 thin film. The triangles represent the temperature amplitude of the film-on-substrate structure, which is experimentally measured by detecting the 3ω voltage across the 5.5 μm wide Au-heater. The thermal conductivity of the Si_3N_4 is determined by comparing the temperature amplitude of the film-on-substrate structure with the corresponding value in the Si substrate. The circles represent the corresponding temperature amplitude of the Si-substrate, obtained by Eq. (1).

Table 1
Experimental results of the thermal conductivity measurements of AlN and Si_3N_4 thin films using the differential 3ω method.

Material	Film thickness d_f [nm]	Film thermal conductivity value λ_f [$Wm^{-1} K^{-1}$]
AlN	299	5.4
	502	10.5
	600	12.2
	699	14.2
	998	17.6
Si_3N_4	298	0.8
	500	1.2
	601	1.3
	698	1.5
	1001	1.7

of 5 in. was used for depositing the thin film with a gas mixture of nitrogen and argon. Film thickness was varied between 300 and 1000 nm.

The metal heater was deposited onto the thin film sample using a lift-off process. Gold (Au) was used because of its high temperature stability. Between the thin film and the Au strip, a thin titanium layer acts as an adhesion agent. The dimensions of the metal lines used in this study were 5.5 μm width, 9 mm length, and about 500 nm in thickness. The dimension of the metal strip was chosen to be wider compared to the thickness of the thin films, thus the heat flow through the films can be considered as one dimensional [13].

The main challenge of the experimental setup is the reliable extraction of the 3ω voltage signal from the voltage oscillation of the thin film sample, since the amplitude of the 1ω voltage is typically 1000 times higher than the amplitude of the 3ω voltage [14]. Therefore, an appropriate electrical circuit, consisting of a differential lock-in amplifier and a bridge circuit, is needed.

Fig. 2 shows a schematic diagram of the experimental setup used to extract the 3ω component of the voltage along the metal heater. An internal signal generator of the digital lock-in amplifier (Anfatec Instruments eLockIn204/2) produces the alternating heating current. The generated heating current contains a low harmonic distortion, because any third harmonic content in the signal generator induces interfering signals during the thermal conductivity measurement. Due to the finite dynamic reserve of the lock-in amplifier, the suppression of 1ω voltage from 3ω signal is accomplished by a bridge circuit balanced by adjusting the series resistance R_{ref}. To properly detect the 1ω and 3ω voltage signals a differential lock-in amplifier with a bandwidth of 0.1 Hz up to 2 MHz was used. In order to reduce radiation and convection losses, measurements were performed inside a vacuum chamber with a pressure of less than 3.1 Pa.

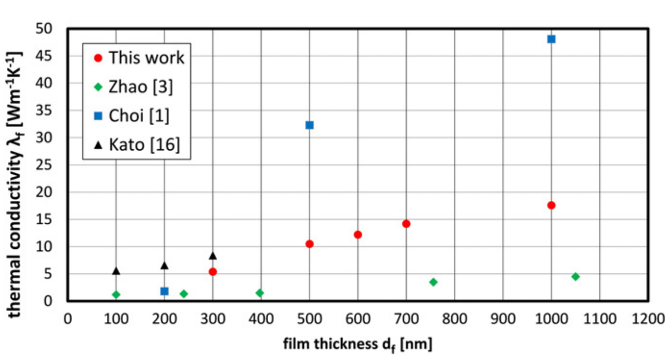

Fig. 4. Thermal conductivity of AlN thin films as a function of film thickness (see Refs. [1,3,16]). Closed circles represent the experimental data obtained in this study by the differential 3ω method. The values are compared with other experimental results for AlN thin films obtained by photothermal reflectance (Ref. [3]), ac calorimetry (Ref. [16]) and standard hot wire method (Ref. [1]).

3. Results and discussion

In this study, the differential 3ω technique was used to analyze the thickness dependency of the thermal conductivity of AlN and Si_3N_4 thin films. Fig. 3 exhibits the raw data obtained from thermal conductivity measurements of the Si_3N_4 thin films deposited on a p-type Si wafer. As the thermal conductivity of Si_3N_4 is well reported [11], it can be used as a reference or a verification of the method. The temperature shift ΔT_f between the triangular and the circular lines in Fig. 3 is caused by the thermal resistance of the thin film. The measured thermal conductivity of the Si_3N_4 film was 1.73 $Wm^{-1} K^{-1}$, which is close to the published value of 1.70 $Wm^{-1} K^{-1}$ for a Si_3N_4 film fabricated by a similar process [11]. The thermal conductivity of the Si substrate determined by Eq. (1) is 140 $Wm^{-1} K^{-1}$, which is approximately 6% smaller than the literature value of the thermal conductivity of pure Si 148 $Wm^{-1} K^{-1}$ [10, 11]. It is evident that this discrepancy is mainly due to the impurity scattering by the doped boron in the silicon substrate [1,10]. The random error of the measurement for a given AlN or Si_3N_4 sample, based on at least ten measurements, was found to be better than 4% in all cases.

Table 1 lists the experimental results obtained for the thermal conductivity of AlN and Si_3N_4 thin films as a function of the film thickness. The experimental values are considerably lower than those of the corresponding bulk materials which are 250–285 $Wm^{-1} K^{-1}$ [1] and 30 $Wm^{-1} K^{-1}$ [11]. Furthermore, the film thermal conductivity values of both AlN and Si_3N_4 increases with film thickness, but the increase rate decreases gradually with increasing thickness. This effect is much stronger for Si_3N_4 than for AlN thin films.

The thermal conductivity values are between 5.4 and 17.6 $Wm^{-1} K^{-1}$ for the AlN film samples with thicknesses varying from 300 to 1000 nm. The reduction of the thermal conductivity values in comparison to the bulk values can be attributed to the shorter phonon mean free path. Slack et al. [15] report that the grain size significantly affected the thermal conductivity due to the grain boundary scattering, because smaller grain size could decrease the thermal conductivity because of the shorter phonon mean free path. The experimental results for our AlN thin films supports this observation. Furthermore, Choi et al. [1] and Pan et al. [9] measured X-ray diffraction (XRD) patterns of the AlN thin films in order to correlate the measured thermal conductivity with the microstructure of the films. According to the result of their XRD result, the increased thermal conductivity of the AlN thin films at a larger film thickness was primarily due to the increase in the grain size [1,9,16].

In Fig. 4, the thermal conductivity of the AlN film as a function of the film thickness is compared with experimental data from other studies obtained by different measurement methods. These methods include hot wire technique [1], AC calorimetric method [16] and photothermal reflectance technique [3]. The results obtained from our differential 3ω method are slightly higher than those reported by Zhao et al. [3], which vary from 1.4 to 4.5 $Wm^{-1} K^{-1}$ for AlN films with thicknesses from 100 to 1000 nm. Kato et al. [16] reported in-plane thermal conductivity values of 5.6 to 8.4 $Wm^{-1} K^{-1}$ in the range of 100 to 300 nm. However, the thermal conductivity values for AlN thin films in the thickness range between 200 and 2000 nm determined by Choi et al. [1] are considerably higher (1.83 to 76.5 $Wm^{-1} K^{-1}$). The discrepancy can be attributed to the difference in the fabrication process and the fact that the differential 3ω method is insensitive to errors from black-body radiation.

The deviation of the AlN thermal conductivity data can also be attributed to the surface roughness of the substrate material. Prior studies have shown that surface roughness affects the interface and the thermal properties of thin films [9,17,18]. Su et al. [17] showed that a reduced thermal conductivity of AlN thin films can result from high surface roughness of the substrate material. It has been shown that for a substrate with 1.2 nm surface roughness defects at the interface impact the thermal boundary resistance (TBR) significantly [17]. However, for substrate surface roughness less than 0.2 nm no planar defects were observed and the TBR was one order of magnitude smaller. In our experiment, the silicon substrate had a surface roughness of less than 0.1 nm. Therefore the TBR was neglected for the determination of the AlN thin film thermal conductivity.

4. Conclusion

This work presents a modified measurement procedure to determine the thermal conductivity of AlN and Si_3N_4 thin films. AlN and Si_3N_4 are widely used in microelectronics and MEMS applications, especially serving as heat spreader films owing to the high thermal conductivity. It is shown that the thermal conductivity of the thin films is substantially lower than that of the corresponding bulk materials. The thermal conductivity of AlN thin films decreases rapidly with decreasing film thickness. Furthermore, the thermal conductivity of the investigated AlN thin films varying 300 to 1000 nm is considerably higher than that of Si_3N_4.

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