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Thermal Conductivity of AlN and SiC Thin Films

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The thermal conductivity of AlN and SiC thin films sputtered on silicon substrates is measured employing the 3ω method. The thickness of the AlN sample is varied in the range from 200 to 2000 nm to analyze the size effect. The SiC thin films are prepared at two different temperatures, 20 and 500°C, and the effect of deposition temperature on thermal conductivity is examined. The results reveal that the thermal conductivity of the thin films is significantly smaller than that of the same material in bulk form. The thermal conductivity of the AlN thin film is strongly dependent on the film thickness. For the case of SiC thin films, however, increased deposition temperature results in negligible change in the thermal conductivity as the temperature is below the critical temperature for crystallization. To explain the thermal conduction in the thin films, the thermal conductivity and microstructure are compared using x-ray diffraction patterns.

KEY WORDS: 3ω method; aluminum nitride; silicon carbide; thermal conductivity; thickness; thin film.

1. INTRODUCTION

The thermal conductivity of a thin film is a fundamental material property that is particularly important in high-power/high-frequency microelectronic devices and micro/nanoscale thermal systems since the ability to dissipate heat is often the limiting factor that determines the

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device performance. Also, the thermal conductivity of a thin film may be substantially different from that of the bulk material because of the microstructure. Therefore, measurement of the thermal conductivity is a critical step for designing or analyzing microelectronic devices and thermal systems.

This work is concerned with the thermal conductivity of aluminum nitride (AlN) and silicon carbide (SiC) thin films. These thin films do not have only high thermal conductivities but also thermal expansion coefficients which are close to those of GaAs and Si [1,2]. Accordingly, AlN and SiC are considered as candidate materials for effective heat spreader films and substantial attention is being paid to the thermal transport properties of AlN and SiC thin films. However, despite the importance of the property, only a limited number of thermal-conductivity data has been reported so far and even the reported values exhibit significant scatter, primarily depending on the film preparation process. Jacquot et al. [3] measured the thermal conductivity of an AlN thin film grown on a silicon substrate by laser ablation. Kuo et al. [4] showed that the thermal conductivity of AlN films is strongly dependent on the substrate. Kato et al. [5] measured the in-plane thermal conductivity of AlN films using the AC calorimetric method. Zhao et al. [6] recently measured the thermal conductivity of AlN thin films in the thickness range from 100 to 1000 nm employing the photothermal reflectance method and explained the results based on the microstructure analysis. Regarding the thermal conductivity of SiC, no reported data are available except for an optical SiC thin film having an amorphous structure [7]. Considering that the thermal conductivity of these thin films depends strongly on the preparation process, i.e., structure, film thickness, impurities, etc., it is evident that further investigations are required for measurement of the thermal conductivity of AlN and SiC thin films.

This work examines the thermal conductivity of AlN and SiC thin films, for potential applications in MEMS (microelectromechanical systems). The films are sputtered on silicon substrates by varying the process parameters, and the thermal conductivities are measured employing the 3ω method [8]. The experimental setup to measure the thermal conductivity is verified by using a silicon dioxide (SiO_2) thin film as a reference case. In the case of AlN, the film is sputtered at room temperature and the thickness of the film is varied from 200 to 2000 nm to observe the size effect. Meanwhile, the thermal conductivity of SiC thin films is measured for two different deposition temperatures, 20 and 500°C, and the effect of elevated temperature on the thermal conductivity is examined. The results of the thermal conductivity measurements are compared with x-ray diffraction

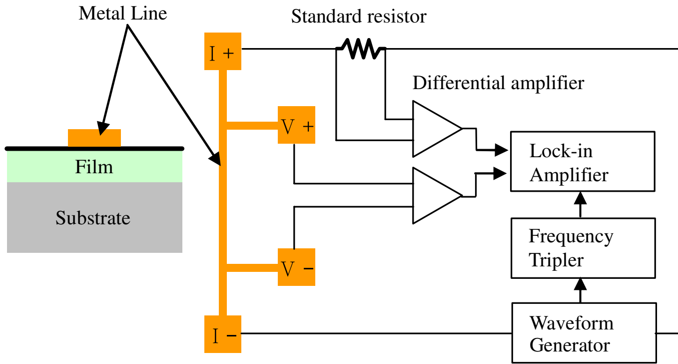

Fig. 1. Schematic diagram of the 3ω method.

(XRD) patterns from the samples for correlating the results with internal material structures.

2. THERMAL-CONDUCTIVITY MEASUREMENT

The 3ω technique is one of most widely used standard methods to measure the thermal conductivity of dielectric thin films [8]. In the method, the surface temperature variation induced by periodic heating of a patterned metal line is measured by monitoring the resistance variation. The sensitivity of the measurement depends on the film thickness and thermal properties. In the typical experimental setup as displayed in Fig. 1, the current input at a frequency of ω induces temperature oscillation at a doubled frequency 2ω, resulting in voltage oscillation across the heating element at a tripled frequency of 3ω.

In the 3ω method, the thermal conductivity of a thin film is determined by comparing the temperature oscillation in a film/substrate structure with the corresponding value in the substrate only. The temperature variation in a film/substrate structure is experimentally measured by detecting the voltage oscillation that is proportional to the resistance variation. The substrate temperature is obtained by [8]

$$\Delta T = \frac{q}{\pi l k_{y_1}} \left(\frac{1}{2} \ln \frac{4\alpha}{b^2} + \ln 2 - 0.5772 - \frac{1}{2} \ln(2\omega) - \frac{i\pi}{4} \right), \tag{1}$$

where $k, \alpha, q, l, b,$ and i are the thermal conductivity of the substrate, thermal diffusivity of the substrate, power supplied to the metal line, length of the metal line, width of the metal line, and imaginary unit, respectively. If the width of the metal line is much larger than the thickness of the film

and also the thin-film thermal conductivity is much smaller than that of the substrate, the temperature shift induced by the thin film is expressed by the following simple heat conduction equation [8]:

$$\Delta T_{\mathrm{f}} = \frac{q}{lk_{\mathrm{f}}} \frac{t}{2b}, \tag{2}$$

where k_{f} and t are the thermal conductivity and the thickness of the film, respectively.

When the thin-film thermal conductivity is comparable to or greater than the substrate thermal conductivity, Eq. (1) cannot be used to precisely describe the temperature oscillation. In this case, the exact solution for the temperature oscillation in the film/substrate structure should be used to determine the thermal conductivity. The general solution for a multi-layered structure was obtained analytically by Borca-Tasciuc et al. [9]:

$$\Delta T = -\frac{q}{\pi l k_{y_1}} \int_0^{\infty} \frac{1}{A_1 B_1} \frac{\sin^2(b\lambda)}{b^2 \lambda^2} d\lambda, \tag{3}$$

where

$$A_{i-1} = \frac{A_i \frac{k_{yi} B_i}{k_{y_{i-1}} B_{i-1}} - \tanh(\varphi_{i-1})}{1 - A_i \frac{k_{yi} B_i}{k_{y_{i-1}} B_{i-1}} \tanh(\varphi_{i-1})}, \quad i = 2, 3, \ldots, n \tag{4}$$

$$B_i = \left(k_{xyi} \lambda^2 + i \frac{2\omega}{\alpha_{yi}} \right)^{1/2}, \tag{5}$$

and

$$\varphi_i = B_i d_i, \quad k_{xy} = k_x / k_y. \tag{6}$$

In the above equations, n represents the number of the layer and the subscripts x and y represent the horizontal and vertical directions, respectively.

The electrical current to heat the metal line is supplied by a function generator (Agilent, Model 333220A). As the 3ω component of the signal is approximately 1000 times smaller than the ω component, the ω component is eliminated by adjusting the gain of a differential amplifier (Analog Devices, Model AD620) with an input impedance of 10 GΩ. The 3ω signal without the ω component is measured by a lock-in amplifier (Stanford Research Systems, Model SR552). The amplitude and the phase of the temperature signal are measured repeatedly by varying the modulation

frequency. The frequency range is chosen to be from 200 to 3000 Hz for the thermal penetration depth to be much greater than the thickness of the thin films.

3. THIN-FILM PREPARATION

The AlN thin film was deposited on a single crystal Si (001) wafer by an rf (radio frequency) reactive magnetron sputtering process with an rf power of 7 kW and a dc power of 100 W. The ambient pressure and temperature in the deposition chamber were adjusted to be 10^{-7} Torr and 20°C, respectively. An aluminum target of 5 inch (12.7 cm) in size was utilized for deposition of the thin film with a gas mixture of N_2 (40 sccm) and Ar (6 sccm). Different samples were prepared by varying the film thickness up to 2000 nm.

The SiC thin film was deposited on a single crystal Si (001) substrate by an ion beam-assisted RF sputtering process. The applied rf and dc powers are 650 and 240 W, respectively. The pressure of the deposition chamber was set to be 5×10^{-4} Torr. Samples were fabricated at two different temperatures, 20 and 500°C, to examine the temperature dependence of the thermal conductivity. An Ar gas flow was employed in the process at a flow rate of 5 sccm.

A metal (Au) pattern of 2500 Å in thickness was deposited on the thin-film sample by the lift-off process to serve as a line heater. The wafer was first coated with a photoresist, exposed to ultraviolet irradiation, and finally developed in the photolithograpy process. Between the thin film and Au layers, a 100 Å-thick Cr layer was inserted to improve the adhesion strength. The Au line was deposited on the Cr surface by the e-beam evaporation process at room temperature (20°C). The photoresist was removed by sonication in acetone for 10 min. Before deposition of the metal patterns, the film/substrates samples were cleaned with acetone and isopropyl alcohol followed by 10 minutes of the main cleaning process using a H_2SO_4 solution (H_2SO_4:$H_2O_2 = 4:1$). After the chemical cleaning process, the samples were rinsed using deionized water and then dried.

4. RESULTS AND DISCUSSION

The resistance of the Au film heater was first calibrated as a function of temperature and the temperature coefficient of the resistance was found to be 0.002567 $\Omega \cdot K^{-1}$ with a linearity better than 0.99998. Figure 2 exhibits the raw data obtained for measuring the thermal conductivity of 500 nm SiO_2 thin film deposited on a p-type Si wafer. As the thermal conductivity of SiO_2 is relatively well known, the thermal conductivity

of the SiO_2 film is measured and compared as a reference case with those from previous investigations. In Fig. 2, the linearity of the temperature signals is better than 0.999. The temperature shift between the solid and dashed lines in Fig. 2 is caused by the thermal resistance of the thin film. The measured thermal conductivity of the SiO_2 film is 1.35 $W{\cdot}m^{-1}{\cdot}K^{-1}$, which is close to the thermal conductivity 1.34 $W{\cdot}m^{-1}{\cdot}K^{-1}$ of a SiO_2 film fabricated by a similar process within 1% [8]. The thermal conductivity of the Si substrate determined by Eq. (1) is 128 $W{\cdot}m^{-1}{\cdot}K^{-1}$, which is approximately 10% smaller than the literature value of the thermal conductivity of pure Si 148 $W \cdot m^{-1}{\cdot}K^{-1}$ [10]. It is evident that this discrepancy is due to the impurity scattering by the doped boron. The random error of the measurement for a fixed sample, based on 10 or more measurements, was found to be better than 1% in all cases. However, the maximum uncertainty considering the sample fabrication process, i.e., sample-to-sample variation based on three different samples, was 8.6 and 4.1% for AlN and SiO_2, respectively. Since the quality of the SiC thin film is estimated to be similar to that of the AlN film, examination of the sample dependence has not been conducted for SiC.

Table I shows the results of the thermal conductivity measurement. The thermal conductivity of the film is substantially lower than that of the bulk material. The thermal conductivity of the AlN thin film shows a

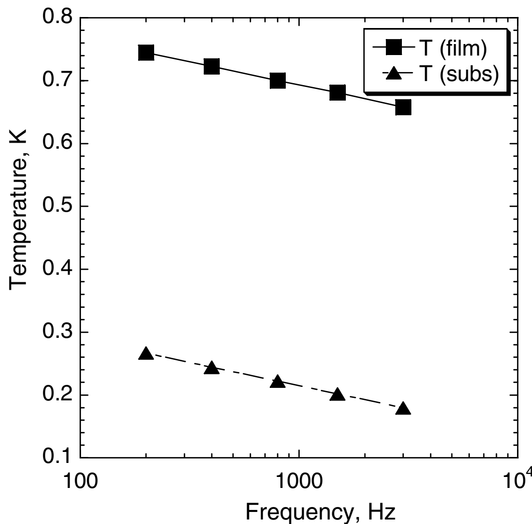

Fig. 2. Typical experimental raw data of the 3ω method (SiO_2 500 nm).

Table I. Experimental Results for Dielectric Thin Films

Material	Thickness (nm)	Deposition temperature	Film value ($W{\cdot}m^{-1}{\cdot}K^{-1}$)	Bulk value [10] ($W{\cdot}m^{-1}{\cdot}K^{-1}$)	Normalized thermal conductivity (film/bulk)
	200	20°C	1.83	285	0.0064
AlN	500	20°C	32.3	285	0.11
	1000	20°C	48.1	285	0.17
	2000	20°C	76.5	285	0.27
SiC	498	20°C	1.44	360 (3C-SiC)	0.0040
	520	500°C	1.49	360 (3C-SiC)	0.0041

pronounced size effect. The thermal conductivity normalized by the bulk value is 0.27 for the sample of 2000 nm in thickness and as small as 0.0064 for 200 nm. The measured values are considerably higher than the previously reported thermal conductivity of AlN by Zao et al., 1.4 to 4.5 $W{\cdot}m^{-1}{\cdot}K^{-1}$ in the thickness range 100 to 1000 nm [6]. Kato et al. [5] reported in-plane thermal conductivity values of 5.6 to 8.4 $W{\cdot}m^{-1}{\cdot}K^{-1}$ in the range of 100 to 300 nm, which is also higher than those by Zao et al. Considering the accuracy of the measurement, the discrepancy is estimated to be mainly due to the difference in the fabrication process, i.e., microstructures and impurities. It is noted that Zao et al. [6] indicated that the thermal conductivity of their AlN thin film is considerably affected by the oxygen impurity based on the results of the XPS (X-ray photoelectron spectroscopy) analysis [6]. Variation of the thermal conductivity with film thickness is plotted in Fig. 3. The line in Fig. 3 is the result of curve fitting

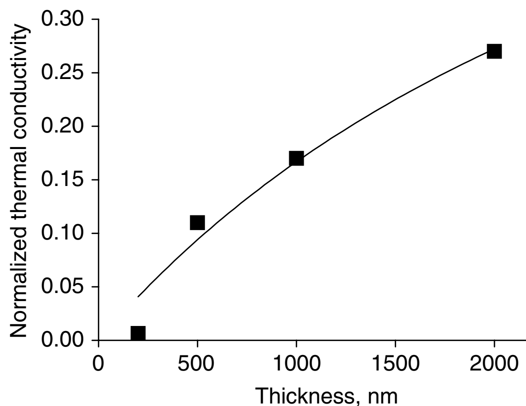

Fig. 3. Normalized thermal conductivity of AlN film as a function of film thickness.

in the form of $C_1t/(t+C_2)$, where C_1 and C_2 are the fitting parameters ($C_1 = 0.7586$, $C_2 = 3448\,nm$). Figure 3 shows that the thermal conductivity increases with the film thickness and the dependence weakens gradually with increasing thickness. In order to correlate the measured thermal conductivity with the microstructure of the film, the XRD pattern has been measured and the results are summarized in Fig. 4. The XRD analysis reveals that the AlN thin films are of crystalline structure regardless of the thickness. On the other hand, the SiC thin films are amorphous regardless of the deposition temperature. According to these results of the XRD analysis, it can be claimed that the increased thermal conductivity of the AlN thin film at a large film thickness is primarily due to the increase in the grain size. In general, the size of a grain is proportional to the thickness of a thin film if other process conditions are unchanged [11]. Therefore, the boundary scattering is enhanced as the thickness of the AlN thin film becomes thin, leading to a decrease in the thermal conductivity. In Fig. 3, the thermal conductivity drop at 200 nm is shown to be particularly large, which is believed to be due to a lattice mismatch at the AlN/Si interface. For thin films, the interfacial region near the substrate generally has a poor microstructure because of the lattice mismatch [12]. Consequently, considering the difference in the lattice constant of AlN (3.11 Å) and Si (5.43 Å), it is expected that the AlN thin film is divided into two layers having difference structures. In this regard, the thermal conductivity

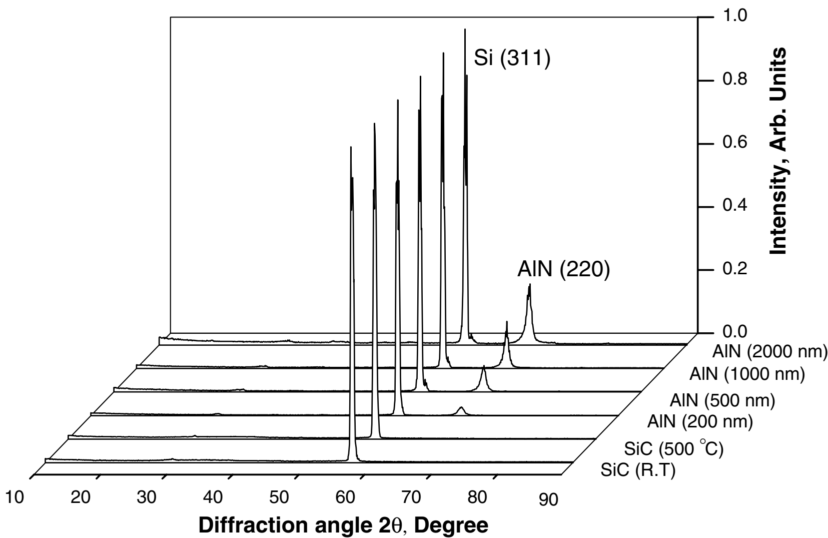

Fig. 4. X-ray diffraction patterns for SiC and AlN films.

at 200 nm in Fig. 3 represents an effective value for the composite structure. Assuming that the film is composed of two distinct layers having thermal conductivities k_1 and k_2 for thicknesses l_1 and l_2, respectively, the effective thermal conductivity k_{eff} is expressed by

$$\frac{(l_1+l_2)}{k_{eff}}=\frac{l_1}{k_1}+\frac{l_2}{k_2}. \quad (7)$$

According to Eq. (7), it is likely that the low thermal conductivity in the interfacial region is responsible for the large drop in the effective thermal conductivity at 200 nm.

Table I indicates that the thermal conductivity of the SiC thin film as well as the AlN film is significantly smaller than that of the bulk material. This reduction of thermal conductivity is because of the amorphous structure of the SiC thin film as indicated in Fig. 4. The amorphous structure in the thin film shortens the phonon mean free path and results in considerably decreased thermal conductivities as compared to the bulk value. The measured thermal conductivity 1.44 $W \cdot m^{-1} \cdot K^{-1}$ is close to the thermal conductivity 1.34 $W \cdot m^{-1} \cdot K^{-1}$ of the SiO_2 thin film whose structure is also amorphous. A relatively small value of the thermal conductivity 0.12 $W \cdot m^{-1} \cdot K^{-1}$ has been reported for an optical SiC film [7]. However, the origin of this difference is not clear as the details of the sample preparation process are unknown for the previously tested film. Unlike the size dependence of the AlN thermal conductivity, the dependence of thermal conductivity on the deposition temperature is not pronounced in the case of SiC. The normalized thermal conductivity is 0.0040 and 0.0041 for the samples deposited at room temperature and at an elevated deposition temperature of 500°C, respectively. As confirmed by the results of the XRD analysis, this small temperature dependence is because even the increased temperature 500°C is not large enough to transform the amorphous microstructure of the SiC thin film into a crystalline structure. Consequently, these results confirm that the effect of process temperature on thermal conductivity is negligible without a significant structural change. The slight increase in the thermal conductivity may be due to partial crystallization of the thin film but the change is not meaningful considering the fact that the film thickness is also slightly different in the two cases.

5. CONCLUSIONS

This work reports the thermal conductivity of AlN and SiC thin films which are widely used in microelectronics and MEMS applications, especially often as heat spreader films owing to the high thermal conductivity. It has been shown that the thermal conductivity is substantially lower than

that of the bulk materials. The thermal conductivity of the AlN thin film decreases rapidly as the film thickness, and thus the grain size, is reduced. On the other hand, negligible change in thermal conductivity has been observed by increasing the SiC film deposition temperature up to 500°C, which is below the critical temperature at which a crystalline structure is formed.

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