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Influence of sample processing parameters on thermal boundary conductance value in an Al/AlN system

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The influence of sample processing parameters on the thermal boundary conductance (TBC) between aluminum and aluminum nitride has been investigated by transient thermoreflectance. An evaporated Al layer on the polished substrate yielded a TBC at ambient of roughly 47 MW m^{-2} K^{-1}. The largest improvement (by a factor of 5) was obtained by plasma-etching of the substrate and subsequent evaporation of the metal layer. Electron microscopy suggests that the differences in TBC were mainly due to the (partial) elimination of the native oxide layer on the substrate. The importance of an adequate model for data extraction on measured TBC is highlighted. © *2011 American Institute of Physics*. [doi:10.1063/1.3560469]

A finite thermal boundary conductance (TBC) is a critical issue for all heat transfer phenomena especially at the microscale and nanoscale. While for theoretical calculations of the TBC the phonon density of states in the two phases forming the interface are considered, recent experimental evidence has highlighted that the chemical nature of the bonds at the interface may be very important for the TBC as well.[1–4] The present communication uses the transient thermoreflectance (TTR) technique[5–12] to determine the thermal conductance at Al/AlN interfaces while varying the condition of the AlN surface prior to deposition of the Al thin layer and the layer deposition method itself.

Samples were made consisting of Al layers deposited by various means on an AlN substrate. The polycrystalline AlN substrate had a measured thermal conductivity of 155 W m^{-1} K^{-1}, lower than its monocrystalline counterparts[13,14] due to the presence of yttria.[15] The samples were washed with alcohol and blown dry with nitrogen before being inserted in the deposition chamber. Sample 1 simply received a 29 ± 2 nm evaporated Al layer. Three samples (number 2, 3, and 4) received an argon-sputtered Al-layer of 32 ± 3 nm, two of which (3 and 4) having undergone a presputter rf etching prior to deposition to remove the first few nanometers of the substrate including a possible native oxide layer. Sample 5 received a 70 ± 1 nm Al layer by evaporation with a prior rf-etch. The thicknesses of the deposited films were measured by focused ion beam (FIB) cross-section and checked by picosecond ultrasonics.[16] Cooling curves were recorded using a conventional TTR setup[11] using a Ti:sapphire laser at 80 MHz repetition rate. Pump fluences of approximately 0.1 J cm^{-2} were used, the pump beam was modulated at 100 kHz and focused on a spot of 40 ± 10 μm in radius. The probe spot had a radius of 10 μm to minimize radial thermal transport effects. Exponential time constants were measured using a fit between 200 and 800 ps. After checking that the measured time constant τ was well below one-fourth of the time between laser pulses in our system (i.e., close to single pulse conditions[17]), an inverse method based on an implicit scheme was used. It was based on the one-dimensional finite differences numerical thermal model described by Norris *et al.*[3,11,18,19] and permitted to extract a value of TBC taking into account the sample's dimensions and thermal properties. The TBC between Al and AlN was calculated for all samples, using both the exponential time constant and the inverse method. To link the results obtained for TBC to the interface structure, lamellae were extracted from three representative samples using a FIB and loaded into a transmission electron microscope for energy filtered transmission electron microscopy (EFTEM) experiments.

Table I shows the results obtained after recording and extracting the TBCs for four measurements on each sample,

TABLE I. Summary of the extracted TBCs with respect to the sample and the data extraction technique. The exponential time constant $\tau^{exp}_{200-800\ ps}$ is taken between 200 and 800 ps. The h_{bd} stands for TBC as measured with an exponential (*exp* superscript) or inverse (*Inv* superscript) method.

Sample number	Etch (min)	Layer	$\tau^{exp}_{200-800\ ps}$ (ps)	h^{exp}_{bd} (MW m^{-2} K^{-1})	h^{Inv}_{bd} (MW m^{-2} K^{-1})	$(h^{exp}_{bd}-h^{Inv}_{bd}/h^{Inv}_{bd})$ (%)
1	⋯	Evap.	1710 ± 360	42 ± 8	43 ± 7	-2 ± 1
2	⋯	Sputt.	570 ± 80	138 ± 20	171 ± 28	-19 ± 6
3	10	Sputt.	580 ± 90	134 ± 16	174 ± 28	-22 ± 6
4	30	Sputt.	510 ± 80	152 ± 19	198 ± 29	-22 ± 6
5	10	Evap.	930 ± 50	183 ± 17	241 ± 46	-25 ± 7

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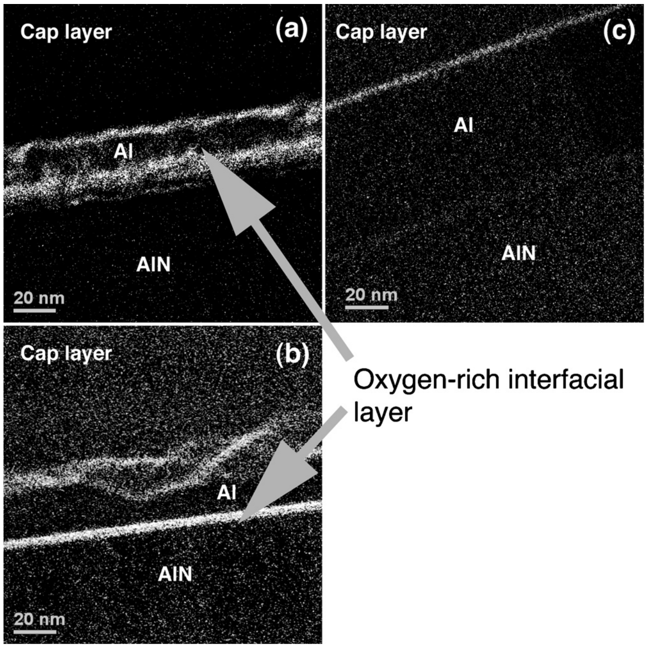

FIG. 1. Energy filtered TEM centered on the oxygen edge at 525 eV in 3 conditions: (a) Al evaporated on AlN without surface treatment (sample 1), (b) Al sputtered on AlN with 30 min rf etching of the surface (sample 4), (c) Al evaporated on AlN with 10 min rf etching of the surface prior to deposition (sample 5). The oxygen-rich layer between the metal and the substrate is 8 ± 1, 3 ± 0.5, and 1 ± 0.5 in thickness for case (a), (b), and (c), respectively.

each of them being an average of ten measurements on the same spot. Depending on the sample, the lock-in signal obtained ranged from 30 to 100 μV with a dc level of approximately 1 V.

Figure 1 shows the EFTEM images obtained with an energy filtered around the oxygen edge at 525 eV for samples with Al (a) evaporated on AlN, (b) sputtered on AlN with 30 min prior rf etch and (c) evaporated on AlN with 10 min prior rf etch. The images show an oxygen-rich layer between the Al and the substrate. The measured TBC increases with decreasing thickness of this layer, cf. Table I. In the case (c) of the evaporated layer on etched substrate, the oxygen signal is very weak and comparison of the electron energy loss spectroscopy (EELS) spectrum around the oxygen edge suggests that the oxygen content of the layer is much lower than in the other two cases, suggesting that (1) the sputtering process may have added oxygen in the early stages of the deposition, possibly due to an inadequate cleaning of the sputtering target and (2) the value of 241 $MW\,m^{-2}\,K^{-1}$ obtained for the evaporated Al on etched substrate is the most adequate value to the actual TBC between Al and AlN with minimal oxide contamination. This value agrees well with the value of 230 $MW\,m^{-2}\,K^{-1}$ given by Stevens *et al.*[11] Moreover, even though rf-etching seems to yield a surface of much better quality, the sputtered Al surface has a rms roughness of 3 nm which represents 10% of its total thickness. This represents the largest source of uncertainty in the results obtained in the thermal model. Also shown in Table I is the comparison between the TBC extracted via the exponential approach and the inverse method. For a TBC of 43 $MW\,m^{-2}\,K^{-1}$ the discrepancy is less than 3% between the values extracted by those two methods. A discrepancy of more than 20% is observed in the case when

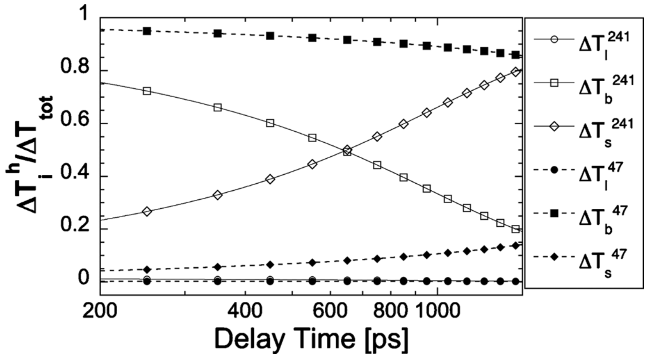

FIG. 2. Contributions of local gradients to the total gradient ΔT_{tot} present in the sample for 2 TBC h of 43 and 240 $MW\,m^{-2}\,K^{-1}$; l, b, and s stand for layer, boundary, and substrate, respectively.

TBC is of 130 $MW\,m^{-2}\,K^{-1}$ or more, and it increases along with the TBC itself. This is rationalized by the fact that the higher the TBC, the more the gradients within the metal layer and the substrate have to develop to bring heat to and remove heat from the interface, respectively.

To illustrate this point the evolution of the ratios between the temperature drop across the interface, ΔT_b, across the metal layer, ΔT_l, and within the substrate ΔT_s, to the one between the metal layer surface and the substrate far from the interface ΔT_0 are shown in Fig. 2 against pump-probe delay time. For a sample consisting of 30 nm Al on an AlN substrate with a TBC of 240 $MW\,m^{-2}\,K^{-1}$ the contribution of the drop within the substrate increases steadily and reaches 50% after 670 ps. On the other hand, a much lower TBC (40 $MW\,m^{-2}\,K^{-1}$) allows the substrate to evacuate heat sufficiently rapidly for the gradient at the interface to account for 90% of the overall gradient up to 1 ns. The temperature drop within the 30 nm Al layer remains smaller than 2% for any delay time. This highlights that not only the layer properties and thickness[13] but also the TBC itself and the substrate thermal diffusivity are important for the choice of the data extraction technique used to measure TBC.

In summary, we present a study of the influence of an AlN substrate surface treatment prior to an Al film deposition on the TBC between these two materials. A rf-etch prior to the Al layer deposition greatly improves the heat transfer between the two phases. Evaporation of Al after a rf-etch seems to be a cleaner method as compared to sputtering. The resulting TBC of 241 ± 46 $[MW\,m^{-2}\,K^{-1}]$ agrees well with results previously published in the literature,[11] and adds to the evidence of the impact of the interface quality on the TBC between two solids.[1–4] Our results further show that for high TBC values in combination with limited substrate thermal diffusivity the inverse procedure is more appropriate than the simple exponential approach.

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