Steady-State Thermal Conductivity Measurements of AlN and SiC Substrate Materials

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Abstract—**Measurements of thermal conductivity on conventional and newly developed electronic ceramics are presented as part of an effort to incorporate the new high thermal conductivity ceramics into microcircuit manufacturing. Motivated by large variations in values claimed by different vendors for ostensibly similar materials, a longitudinal bar apparatus was built to measure thermal conductivities of actual substrate materials on both an individual and a lot sampling basis. The longitudinal bar method is compared to other experimental techniques for measuring thermal conductivity.**

INTRODUCTION

ALUMINUM NITRIDE (AlN) and silicon carbide (SiC) have gained increasing attention as packaging and hybrid substrate materials because of properties that synergistically complement today's high-speed integrated circuits. These properties are high thermal conductivities (comparable to beryllium oxide (BeO)) and thermal expansion coefficients which closely match both silicon and GaAs. Previous work [1]–[3] has shown that AlN can be used successfully to fabricate high-performance thin- and thick-film hybrid microcircuits. Silicon carbide also appears promising, but has not been thoroughly investigated for hybrid applications. Recent reports [4] on boron nitride suggest great potential but to our knowledge no commercial substrates exist at this time.

An important part of data needed for packaging microelectronic circuitry is both the absolute value and the repeatability (sample-to-sample, lot-to-lot, etc.) of certain key design parameters. Although the reported values for the thermal expansion coefficients and strengths of AlN and SiC are relatively constant, the values quoted for thermal conductivity, particularly of AlN, have varied widely (e.g., 70–200 W/m·K). Thus an apparatus to measure thermal conductivity of ceramic materials was designed, fabricated, and tested. The apparatus has been used to verify manufacturers' specification data as well as to determine variations within manufacturing lots.

This study will present a brief overview of thermal conductivity and its measurement, followed by a description of the longitudinal bar apparatus. Experimental results on several materials at both room temperature and 80°C will be reported. Correlation of the longitudinal bar method with the flash diffusivity technique will be described. The effect of hybrid circuit processing on the stability of substrate thermal conductivity will be discussed.

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THERMAL CONDUCTIVITY MEASUREMENT METHODS

Thermal conductivity is a measure of how well a material conducts heat; that is, how small a temperature difference across that material results from a given heat flow through it. Thermal conductivity (K) is determined using the steady-state heat flow equation

$$\frac{Q}{A} = K\frac{\Delta T}{\Delta x} \tag{1}$$

where Q is the heat (in watts) flowing through a cross-sectional area A, perpendicular to the heat flow, and $\Delta T/\Delta x$ is the resultant temperature gradient (see Fig. 1). Steady-state methods typically pass a given amount of heat down a sample and measure the temperatures at various points to determine thermal conductivity; the separation between thermometers (or thermocouples), and the cross-sectional area must be known, and heat leaks from the sample must be minimized.

Several practical methods exist for determining thermal conductivities of ceramic substrates. While each method has its own advantages and disadvantages, the most prevalent techniques are 1) the guarded hot plate; 2) longitudinal bar; and 3) flash thermal diffusivity method. The first two are steady-state methods, where the third is transient. A summary of each of these methods is presented below.

Guarded Hot-Plate Method

This method [5]–[8] is used with thin disk or plate shaped specimens, where the heat flow is in the thickness direction, as it would be in microcircuit or package use. The specimen is placed between a heat source and a "cold plate" or heat sink, both of which contain temperature sensors very close to their surfaces. The sensors measure the temperature drop across the sample after a steady-state heat flow has been established. The heat source is surrounded by a guard heater, maintained at the heat source temperature, that minimizes heat losses out of the edges. In this method, the thermal resistance at the sample interfaces must be minimized and well controlled for consistent results. This makes the method more useful for the lower ranges of thermal conductivities and less accurate for

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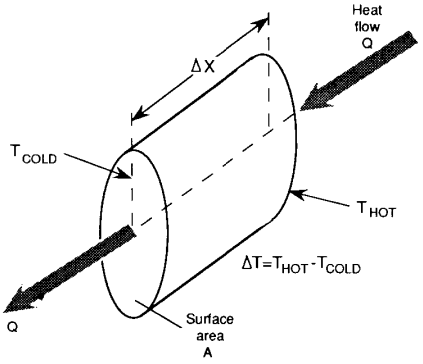

Fig. 1. Schematic representation of sample heat flow for the determination of thermal conductivity.

the higher values obtained with the newer ceramics such as AlN and SiC. Since the sample sizes required in this method are generally larger than the AlN and SiC hybrid substrate materials available for testing, and since flatness variations and camber common in ceramics can cause major differences in the interface resistances, this method was not chosen.

Longitudinal Bar Method

In this method [9], [10], a heat flow is sent down a long piece of material, inducing a temperature gradient that is measured by two (or more) thermometers (thermocouples) contacting the sample. The ratio of the heat flow per unit area to the temperature gradient gives the thermal conductivity (1). Sample shapes are generally bars or rods whose length is at least five times the effective diameter (either the actual diameter for circular specimens or the cross-sectional length plus width divided by two for rectangular (or square) specimens). Since the temperature gradient is measured directly, thermal interface resistances at the heat source and sink are less important. To reduce errors, the apparatus must be designed to minimize heat losses from the sample; this is done by isolating the sample from the environment using insulation and thermal guards. A drawback in using flat ceramics is that the measurement is made with the heat flow parallel to the surface, whereas in typical electronic package applications they are used with the heat flow perpendicular to the surface; any sample anisotropy would cause an inaccuracy in the assumed perpendicular thermal conductivity. The apparatus is relatively simple to design, and can be made with common machine shop tools.

Flash Diffusivity Method

Thermal diffusivity is a measure of a material's thermal response to a transient application of heat. The diffusivity is actually the product of the density, heat capacity, and thermal conductivity; density and heat capacity must be known to extract thermal conductivity from the diffusivity data. In this method [11], a flat sample has a heat pulse applied to one surface, and the temperature-versus-time curve on the opposite surface is measured. The heat pulse is almost always applied with a moderately powered laser. The temperature sensors are usually noncontact optical sensors to avoid inaccuracies due to thermal mass of the thermometer or thermocouples [12]. Its utility is very dependent on the optical absorption and emission properties of the sample material, so it is not as generally useful for wide classes of material as is the longitudinal bar method. Although the flash diffusivity technique is somewhat expensive to set up, it can be automated for high volume use.

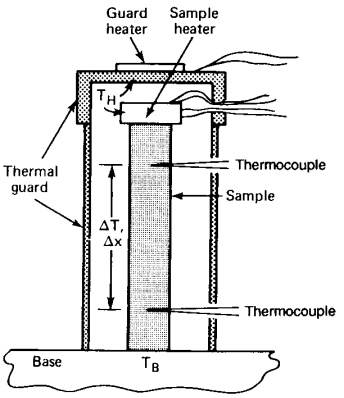

Fig. 2. Diagram of the thermal conductivity apparatus. A heat flow from the sample heater to the base induces a temperature gradient (ΔT) in the sample, measured by the two thermocouples spaced Δx apart. Heat loss out of the sample is minimized by the temperature-compensated guard.

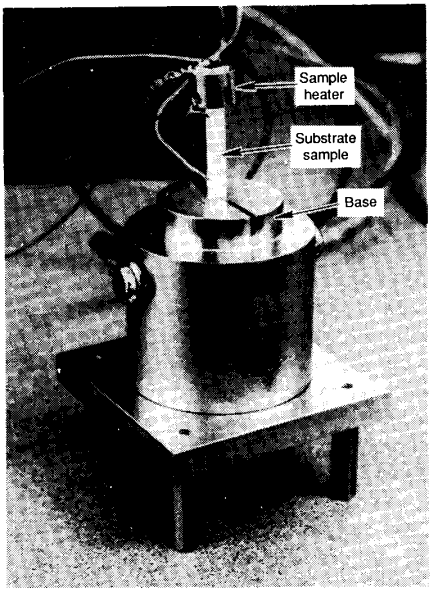

Fig. 3. Photograph of the thermal conductivity apparatus showing a typical test sample in position with the guard and glass wool insulation removed.

Longitudinal Bar Apparatus

The longitudinal bar method was chosen for its versatility, relative simplicity, and economy. Such an apparatus is shown schematically in Fig. 2. The apparatus uses a guarded steady-state measurement technique where the temperature gradient resulting from a given heat flow is measured with two thermocouples in a differential mode. A photograph of a mounted sample is shown in Fig. 3. The question of anisotropy of the thermal conductivity was addressed in AlN by examining the grain orientation with a metallograph; no visible evi-

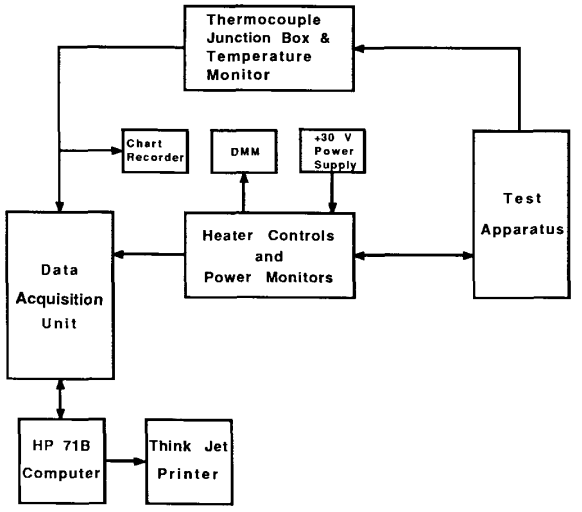

Fig. 4. Block diagram of the thermal conductivity apparatus and supporting hardware.

dence of a preferential orientation was seen, making significant anisotropy in thermal transport properties unlikely.

The test apparatus is connected to several pieces of support hardware (Fig. 4). This hardware controls the sample and guard heaters, measures temperatures at various points in the apparatus, collects the necessary data for computing thermal conductivity, and prints the test results. The system is monitored by an HP-71B hand-held computer. Information about the test sample (i.e., thickness, width, and thermocouple spacing) is entered by the operator at the beginning of a test and is used by the computer along with the measured data (i.e., sample heater power and temperature across the sample) to compute the thermal conductivity of the sample. The system is run open-loop (i.e., no automatic feedback control) and therefore needs an operator to manually set heater levels and maintain sample and guard heater null and to monitor and maintain a stable ambient temperature. For room temperature testing, the system achieves steady-state conditions in as little as 30 min, but requires up to 60 min to reach equilibrium at 80°C.

Experimental Results

Room-Temperature Thermal Conductivity Results

The room-temperature thermal conductivity measurements were made on Al_2O_3, AlN, SiC, and BeO substrates. In most cases, the values measured on this apparatus were close to the manufacturers' specifications. Table I presents the data collected from these tests. The Hitachi SiC has a high thermal conductivity because it contains BeO [13], [14]. In cases where there were large discrepancies, additional measurements were taken. In all cases, the discrepancy remained.

Onn and his collaborators [15] at the University of Delaware, using an evacuated and thermally guarded longitudinal bar system, have reported similar results. Room-temperature thermal conductivity results measured at APL were compared to the Delaware data [16] for AlN and Al_2O_3 substrate materials. The materials had been studied independently by each laboratory. However, the materials were obtained from the same manufacturers with the same quoted thermal conductivity values, but from different lots. Coors 96 percent Al_2O_3, with a quoted value of 26 W/m·K, had a measured value of 28 obtained with the University of Delaware's apparatus and 27 with this study's apparatus. Toshiba's (also known as Norton and now Ceratronics) AlN 170 W/m·K material had a measured value of 173 from Delaware and 171 from this study.

High-Temperature Thermal Conductivity Results

The APL apparatus has been used as high as 80°C without significant thermal losses becoming apparent. Substrates in each of the various thermal conductivity ranges were measured at room temperature, 55°C, and 80°C. Fig. 5 shows these results. BeO and SiC with BeO have a relatively sharp decrease in thermal conductivity with increasing temperature, whereas, the AlN, Al_2O_3, and SiC without BeO appear to have a much smaller decrease with increasing temperature. Kuramoto *et al.* [17] and Kurokawa *et al.* [18] each have published thermal conductivity versus temperature graphs for BeO, AlN, and Al_2O_3 (from room temperature to 200°C) with results similar to our study.

Effects of Thick-Film Processing

A necessary requirement in developing new materials for packaging is their stability to the various processing environments. A natural question in the use of AlN (and SiC) is the stability of its thermal conductivity to thick-film processing; the nitride could, in principle, be converted to oxide in an air-firing furnace. Thermal conductivity would be greatly affected if some appreciable fraction of the bulk nitride was converted to the oxide. As a test of the extent of the possible nitride-to-oxide conversion in the bulk and at the surface, thermal conductivity measurements and surface oxide thickness measurements were performed before and after thick-film firing.

Surface Oxide Measurements: An experiment was conducted to compare the surface compositions of unpolished Tokuyama Soda AlN substrates both before and after furnace firing using the secondary ion mass spectrometer (SIMS). The substrate cleaning and firing consisted of the following successive processing steps: dicing, solvent cleaning, one air firing at 850°C (10-min peak profile), ultraviolet/ozone cleaning, and four additional air firings at 850°C. SIMS profiles for AlN at various points in the processing are presented in Fig. 6. After all processing, the surface oxide signal intensity increased to double that seen after the original dicing treatment. The surface nitrogen decreased by a similar factor. However, the surface is far from being completely oxidized; the estimated atomic fraction of oxygen is less than 5 percent. The crossover of oxygen and nitrogen signals occurred only 2 nm below the surface after dicing and at 8 nm from the surface after all the processing. In the processed substrates, the oxygen level reduced to background levels in a distance approximately 40 nm from the surface. Thus no significant surface conversion was indicated.

TABLE I
ROOM-TEMPERATURE THERMAL CONDUCTIVITY RESULTS FOR VARIOUS CERAMIC SUBSTRATE MATERIALS USING LONGITUDINAL BAR METHOD

Substrate Material	Substrate Manufacturer	Purity/ Color	Thermal Conductivity (W/m·K) Manufacturer's	Measured
Al_2O_3	Adolf Meller	100%/clear	40	42
	MCR	99.6%/white	37	31
	Coors	96%/white	26	27
AlN	Toshiba	grey	70	72
		translucent	170	171
	Tokuyama Soda	translucent	140	154
		translucent	180	179
BeO	Brush Wellman	white	250	282
SiC	Hitachi	grey	270	276

* Measured values shown are adjusted to 25°C from a three-temperature curve fit to conform to the referenced room-temperature manufacturers' values.

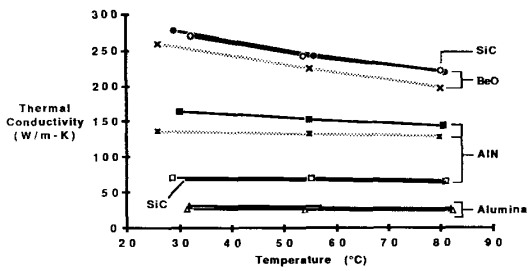

Fig. 5. Thermal conductivity versus temperature characteristics for substrate materials ranging from about 20 to 280 W/m·K. The measured values (solid lines) are compared to the published [17] data (dashed lines).

TABLE II
ROOM-TEMPERATURE THERMAL CONDUCTIVITY RESULTS ON TOKUYAMA SODA'S 160 W/m·K AlN AFTER DICING AND AFTER FIVE AIR FIRINGS AT 850°C

Sample	As Diced	Sample	Fired 5 Times at 850°C
1	163	7	172
2	170	8	166
3	164	9	167
4	169	10	166
5	166	11	171
6	167	12	169
Mean	166.5	Mean	168.4
Std. dev.	2.2	Std. dev.	2.2

Thermal Conductivity Measurements: Four Tokuyama Soda AlN substrates were diced into twelve 0.635 cm × 2.54 cm samples. Thermal conductivity was measured on six samples after dicing without subjecting them to the firing furnace. The other six were fired five times using a standard 850°C 10-min peak thick-film temperature profile. The results are shown in Table II. The oxygen depth data measured by SIMS correlated well with the thermal conductivity results, as no measurable effect was seen in the thermal conductivity.

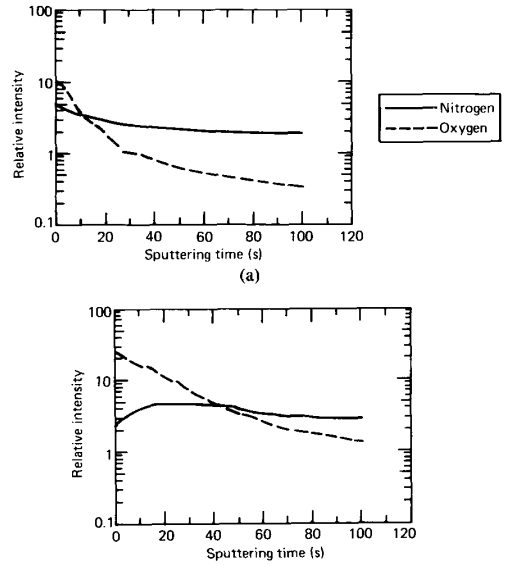

Fig. 6. SIMS profiles of Tokuyama Soda AlN samples. (a) After dicing. (b) After dicing, solvent, and UV/ozone cleaning, and five firings at 850°C.

Method Correlation Study

As an independent check on the thermal conductivity results, a round robin experiment to measure thermal properties on the same samples was conducted using the APL longitudinal bar apparatus and the thermal diffusivity apparatus of Enck and his collaborators at BP Research [19]. The diffusivity apparatus was designed to eliminate the need for surface treatment on the samples. The Al_2O_3, AlN, SiC without BeO, and BeO samples were first measured for thermal diffusivity on 2.54-cm^2 wafers nominally 0.0635 cm thick. They were then diced into strips of 0.635 cm wide for APL thermal conductivity measurements. The results are compared in Fig. 7.

DISCUSSION

The values measured in this work are in general agreement with the manufacturers' quoted values, with a few marked

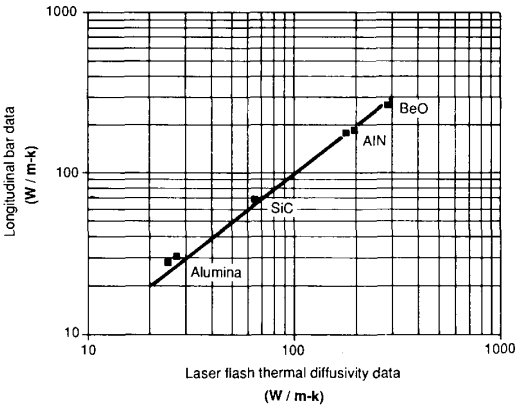

Fig. 7. Results of a round robin thermal conductivity experiment on Al_2O_3, SiC without BeO, AlN, and BeO using the APL longitudinal bar apparatus and BP Research's laser flash diffusivity technique.

differences for certain AlN samples. It is uncertain whether such discrepancies are from the statistical distribution of values within a given lot, from lot-to-lot variations, mislabeling, or actual errors in thermal conductivity measurements. The values measured using the APL longitudinal bar apparatus from room temperature to 80°C on the different materials agree with both previously published data on similar materials and current measurements by other researchers. The APL longitudinal bar data for high thermal conductivity samples are completely consistent with the flash diffusivity results. For low thermal conductivity samples there appears to be an unresolved difference (under 15 percent) between the longitudinal bar and the flash diffusivity method.

Data on the AlN samples subjected to thick-film firing show no measurable change in thermal conductivity, indicating the material to be stable after repeated firings in 850°C oxidizing atmospheres.

The longitudinal bar apparatus appears to be operating as designed; measurements indicate that thermal resistances at the base and heater mounts are low enough not to affect the thermal conductivity measurements. The apparatus can be used, with reasonable care, for lot sample inspection of materials received for circuit manufacturing when high thermal conductivity is important.

Conclusion

SiC materials are at a rather early stage of development to determine their applicability to electronics packaging. AlN materials, however, show great promise. Despite minor discrepancies among thermal conductivity measurement methods; commercially available AlN generally shows high thermal conductivity; it has also been successfully subjected to normal hybrid processing with no adverse effects. If AlN materials properties remain consistent as the manufacturers' formulations mature, as have aluminum oxide ceramics, AlN is likely to find substantial use in circuit manufacturing.

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