MICROELECTRONIC MATERIALS CHARACTERIZATION: AN UPDATE

Photosensitive polyimides and aluminum nitride materials have been used to fabricate an array of advanced high-performance microcircuit devices and structures. The characterization of these materials by fundamental and advanced materials analysis methods has paved the way for the development of these new circuits.

INTRODUCTION

In a previous article,¹ various microelectronic materials analysis and characterization tools and techniques were presented with a focus on ensuring the quality of APL's microelectronic products. Those tools and techniques can also be applied to the development of new materials and products for current and future microelectronics applications. This article discusses the development, characterization, and application of materials for two evolving microelectronics application areas: highdensity substrate interconnections produced by complex, high-speed integrated circuits; and the thermal management of high-power-dissipating integrated circuits. The first area involves the creation of high-speed, controlledimpedance, high-density-substrate interconnections using multilayer thin-film hybrid structures with an organic interlayer dielectric. For the second, the thermal dissipation requirements of today's high-performance integrated circuits will be handled by building both thick- and thinfilm structures on aluminum nitride (AlN) substrates.

THIN-FILM HYBRIDS

The advancement of microelectronics toward more complex and faster devices has resulted in an electronic packaging revolution. Today's packaging techniques must accommodate the high input/output requirements and the increases in speed of modern very large scale integrated (VLSI) devices. A multilevel thin-film hybrid with line widths of 25 μ m (on 99.6% alumina) or less (on silicon) is the preferred technology² to meet the challenge of VLSI technology and to realize fully its potential. A thin-film multilayer hybrid permits the fabrication of resistors and integral ground planes within the hybrid structure. Polyimide has recently been evaluated as an interlayer dielectric for these hybrid structures. Its advantages include a low dielectric constant ($\epsilon_r = 3.5$), a smooth surface for the fabrication of narrow line widths, resistance to high temperatures, resistance to solvents, compatibility with existing thin-film processes, and excellent planarization or leveling for the fabrication of multilevel structures. To use polyimides in multilayer structures, via-holes must be fabricated in the dielectric layers to connect the different conductor layers. Fabricating via-holes in (traditional) nonphotoimagable polyimides³ involves either wet or dry etching methods.⁴ The disadvantages of the traditional methods include poor resolution (wet chemical etching), process complexity, and equipment costs (dry processing). In photosensitive polyimide precursors, via-holes can be patterned directly by applying the precursor, exposing the substrate to ultraviolet light, and developing to remove the unexposed portion of the precursor film, followed by a high-temperature cure.

Until recently,^{2,3} there has been little information in the literature concerning the processing of thick layers of polyimide precursors suitable for use in multilayer hybrid structures. This study addresses the characterization and use of photosensitive polyimide precursors for thin-film multilayer hybrid applications where thick dielectric interlayers (nominally 15 to 25 μ m) are required. The particular polyimide of interest is Probimide 348, manufactured by Ciba-Geigy Corp.⁵ The substrate material used in this study was 99.6% aluminum oxide (alumina), which typically is used for thin-film hybrid applications. Some particular topics to be considered include polyimide polymer chemistry, spin-coating parameters, drying and curing processes, reliable via fabrication, and circuit parameter and design guidelines for thin-film multilayer hybrids. In addition to previous materials analysis methods,¹ results from thermal analysis methods such as thermogravimetric analysis and differential scanning calorimetry will also be discussed.

Polyimide Chemistry

Developing a process for fabricating thin-film circuits using photosensitive polyimides requires an understanding of the basic chemistry involved. In nonphotoimagable polyimides, polyamic acid (the polyimide precursor) is formed by a polycondensation reaction of an acid dianhydride with a diamine base (Fig. 1). In Probimide 348,⁵ the dianhydride of pyromellitic acid (PMDA) and the diamine (oxydianiline, ODA) are used to form the polyamic acid, which is readily soluble in a polar organic solvent such as *n*-methyl pyrrolidone (NMP). The polyimide is converted from the polyamic acid at temperatures high enough to remove most of the NMP and initiate the ring closure (Fig. 1). The ring closure, or im-



Figure 1. The chemistry of PMDA-ODA polyimide.

idization reaction, results in the release of two molecules of water for every polymer repeat unit.

The photosensitized polyamic acid in Probimide 348 contains photoreactive groups that can form cross-links with adjacent polymer chains upon exposure to ultraviolet light. This cross-linkage leads to a differential solubility with respect to the exposed and unexposed portions of the film; thus, it can be developed in much the same way as negative photoresist.⁶ The photochemical reactions of the photosensitive polyimides are shown in Figure 2.

Thermogravimetric Analysis

The Perkin-Elmer Model TGS-2 Thermogravimetric System was used to analyze uncured samples of the polyimide precursors. With this technique, a sample is heated at a constant, preferably linear, rate while the sample weight is monitored continuously. The weight change with temperature is plotted, and the thermal stability of the original sample, the composition and thermal stability of intermediate products, and the composition of the residue may be determined. The sample weight was approximately 10 mg, and the heating rate was nominally 5°C/min. Before the thermogravimetric analysis, the samples were placed on a hot plate (nominally at 80°C) to dry and were exposed to ultraviolet light to achieve photo cross-linking of the polyimide precursor. The percentage weight loss versus temperature for the Probimide 348 is shown in Figure 3. It lost 36% of its weight during the curing process, consistent with the data reported by the manufacturer,⁵ but it exhibited little weight loss (solvent loss) until the boiling point of the NMP was reached (205°C). Most of the weight loss above



Figure 2. The chemical reactions of photosensitive polyimide precursor.



Figure 3. Thermogravimetric analysis of Probimide 348 photosensitive polyimide precursor. The scan rate is 5° C/min. The sample weight is about 10 mg.

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205 °C is due to the loss of water resulting from the imidization of the polyamic acid (Fig. 1).

Differential Scanning Calorimetry

The photosensitive polyimide was analyzed using a Perkin-Elmer DSC-2 Differential Scanning Calorimeter (DSC), which measures the change in the amount of heat (liberated or absorbed) with the change in temperature. Heat or enthalpic changes can be either endothermic or exothermic and are a result of phase transitions such as boiling, vaporization, etc.; bond-breaking reactions; changes in crystalline structure; and chemical reactions. The DSC method is ideal for use in polymer chemistry to characterize glass transition temperatures, polymerization, thermal decomposition, and oxidation-type reactions.

Before analysis, the specimens were treated as described for the thermogravimetric analysis. The result of the analysis is shown in Figure 4. The DSC spectrum for the Probimide 348 showed two characteristic endothermic peaks at 185 and 220°C. The rapid change in slope above 250°C is due to the evaporation of water formed during the imidization reaction.

Mechanical Properties

Stresses in cured polyimide films consist of "intrinsic" stresses resulting from film shrinkage and thermal stresses resulting from differences in the coefficients of thermal expansion of the film and the substrate. Intrinsic stresses from solvent loss alone may or may not be significant, depending on the cure cycle. If the film is only partially cured or is cured too rapidly, so that a significant portion of solvent remains in the specimen after the cure cycle, large stresses can accumulate in the film when it is returned to room temperature. These stresses are much higher than those present when the film is cured slowly at temperatures up to 400°C and then brought back to room temperature, probably because of a higher coefficient of thermal expansion⁷ owing to the presence of unevaporated NMP in the film.

The coefficient of thermal expansion for PMDA-ODA has been reported to be 20 ppm/°C.⁷ The glass transition temperature for this polyimide has been reported to be approximately 385°C.⁸ The coefficient of thermal expansion for a common substrate material such as alumina is about 6.7 ppm/°C. Thus, cooling down from the glass transition temperature can result in large accumulated stresses. These stresses should, however, be well below the fracture strength of the film,⁹ provided there is sufficient adhesion to the substrate and there are no flaws in the film surface.

Film stresses should not increase significantly with film thickness, but adhesion may be degraded if the film thickness inhibits photo-cross-linking of the polyimide near the substrate. Increased film thickness can also increase substrate warpage by introducing bending moments that are proportional to the cube of the largest substrate dimension.¹⁰

Electrical Properties and Circuit Characteristics

The electrical properties of cured PMDA-ODA have been fully characterized in the literature.^{4,11-13} An evaluation of polyimide as a suitable interlayer dielectric for use in



Figure 4. Differential scanning calorimetry of Probimide 348 photosensitive polyimide precursor. The scan rate is 20°C/min.

very high speed integrated circuits (VHSIC) and VLSI devices depends on a complete characterization of its electrical properties (dielectric constant and dissipation factor), a knowledge of the specific characteristics of the logic family that is to be interconnected, and the determination of the electrical parameters for a given multilevel configuration. The electrical parameters will depend on the use and placement of distributed power and ground planes within the multilevel stack, the thickness of the dielectric layers, and the geometry of the conductor lines. The requirements of the device and the specific application will determine the range of acceptable electrical parameters.

Electrical parameters essential to good circuit performance include signal-line capacitance and resistance for low-speed applications and characteristic impedance, attenuation, and cross-talk immunity for higher-speed applications. Polyimide, with its inherently low dielectric constant, should provide an excellent medium for moderate- to high-speed data transmission, and its dissipation factor is low enough (typically below 0.006 at 25°C for fully imidized specimens) for most high-speed silicon devices.

A device's output impedance is important in determining the optimum signal-line impedance for the unterminated line.¹⁴ For example, ringing—stemming from reflections in improperly terminated lines—can be eliminated by matching the driver-source impedance to the line impedance. The output impedances for high-to-low and low-to-high transitions typically are different; an exact match is not possible, but a good compromise can usually be found. Generally, the higher the output impedance, the less capacitance that a source can drive for a given propagation delay.

The critical length of a signal line buried in polyimide (with a dielectric constant of 3.5) for a high-speed pulse with a rise or fall time of 1 ns would be about 16 cm, compared with about 10 cm for a typical thick-film dielectric. Speed can be increased by fabricating signal lines with narrower traces, which result in lower crossover capacitance and, thus, a higher propagation velocity. The characteristic impedances for covered microstrip lines as a function of dielectric thickness for signal lines 25, 37.5, and 50 μ m wide are shown in Figure 5. Since the relative dielectric constant is low, the impedance for the covered microstrip is only slightly different from that of the microstrip configuration. The microstrip line, which has been well characterized in the literature, should be adequate for design purposes below microwave frequencies. Today, the unloaded output impedances for most devices are in the 50- to 65- Ω range; thus, dielectric layers of 13 to 25 μ m will have to be fabricated. While the manufacturer should strive to achieve desirable values, thickness variations across the substrate resulting from processing and material variables will limit the ability to manufacture signal lines with tightly controlled impedances.

Polyimide Application

Thick polyimide coatings can be applied several ways, the foremost being spin coating. The cured thickness of Probimide 348 as a function of spin speed for various spin times is shown in Figure 6. The thickness decreases monotonically as a function of both spin speed and time. The effect of spin speed and time on the degree of uniformity (standard deviation, σ , of the film thickness divided by the average film thickness, \bar{x}) is illustrated in Figure 7. The data indicate that, for a given spin time, there is a range of spin speeds that minimize the degree of uniformity, expressed as σ/\bar{x} . An optimization analysis of response parameters^{15,16} yields the most uniform films for a given thickness. The most uniform Probimide 348 material was obtained at a thickness of 14 μ m.

When exposing polyimide with small via structures (using proximity techniques for sloped via sidewalls), only a small amount of nonuniformity can be tolerated. The total thickness variation across a substrate (greater than several square centimeters in area) can easily exceed 4 to 6 standard deviations owing to the edge bead from the spin coating operation. The edge bead limits the minimum mask-to-substrate distance in the proximity exposure mode. With this limitation in via-hole exposure distance, the process window decreases as the film thickness increases, resulting in reduced via size. For a thickness variation of 6σ , a 1-mm-thick film of uncured polyimide exposed in the proximity mode can tolerate, at most, a degree of uniformity of 0.025. Films this uni-



Figure 5. The characteristic impedance of covered microstrip lines in polyimide.

form are difficult to achieve. Larger via-holes require less uniform films. For predictable results, the via-hole diameter should be 2.5 times the thickness of the cured film to ensure sloped via sidewalls with angles $\geq 45^{\circ}$ and sufficient area at the via-hole bottom to maintain low contact resistance.

A via test pattern was generated with 50-, 75-, and $100-\mu m$ round and square via-holes. The pattern included



Figure 6. The thickness of a fully cured Probimide 348 polyimide film layer as a function of spin speed and spin time.



Figure 7. The uniformity of film thickness for a fully cured Probimide 348 polyimide film layer as a function of spin speed and spin time.

an underlying ground plane and a second layer of metallization that could be probed to measure the via resistance. A Dektak Stylus Profilometer (8-µm stylus) was used to profile the via-hole sidewalls before and after curing. Scanning electron microscopy and electrical resistance tests were used to evaluate the via quality. Via fabrication with the Probimide 348 has been quite good. The precursor is highly photoreactive at the 436-nm peak in a mercury arc emission spectrum.⁴ Relatively short exposures using an I-line suppression filter were sufficient to fabricate via-holes in 50-µm-thick films. The uniformity of development was quite good for both round and square via-holes. Proximity settings were varied between 5 and 15 μ m. At the 15- μ m setting, the $50-\mu m$ vias were almost completely closed, but the degree of closure was fairly uniform across the substrate. Figures 8 and 9 illustrate a 50- μ m round via and a 75- μ m square via in a 20- μ m-thick Probimide 348 polyimide film using a 10- μ m proximity setting, 630 mJ/cm², filtered (365-nm I-line suppression filter) exposure. The 10-µm proximity setting yields nicely sloped sidewalls and an opening at the bottom of the via-hole large enough for electrical conduction.¹⁷

Circuit Fabrication

Multilayer thin-film test circuits (Fig. 10) and hybrid substrates have been fabricated using photoimagable polyimide. The bottom layer metallization is made up of sputtered chromium (0.2 μ m thick) to serve as an adhesion layer to the bare ceramic, followed by a sputtered copper conductor layer (1.4 μ m thick), and another layer of sputtered chromium serving as an adhesion layer for the first dielectric layer. Most of the ceramic is metallized since that layer acts as an interdigitated power and ground plane. Vias through the first dielectric layer connect the power and ground to subsequent layers. The large areas of chromium offer an excellent intermediate surface for adhering the polyimide film to the ceramic substrate. The interdielectric metallization also consists of a sputtered chromium adhesion layer (0.2 μ m thick), a sputtered copper layer (1.4 μ m thick), and a second chromium adhesion layer. The top-layer metallization is a sputtered chromium adhesion layer followed by a sputtered copper or gold layer, depending on the particular application. When conductor thicknesses greater than 3 µm are desirable, metal layers are typically plated to the desired thickness. Plated interlayer conductors can then be sputtered with a chromium adhesion layer before patterning. Metallizing the top layer with chromium/copper followed by plated nickel and gold yields a metallization scheme that is compatible with most soldering, bumping, and wire-bonding processes.

ALUMINUM NITRIDE SUBSTRATES/ CIRCUITS

Aluminum nitride (AlN) is rapidly becoming a useful substrate material for hybrid fabrication because of its many desirable properties, including (1) a high thermal conductivity comparable to that of beryllium oxide; (2) a coefficient of thermal expansion that closely matches those of silicon and gallium arsenide; and (3) an appar-



Figure 8. A round 50- μ m-diameter via developed from 750- μ m-thick (uncured) Probimide 348 photosensitive precursor.



Figure 9. A square 75- μ m-wide via developed from 750- μ m-thick (uncured) Probimide 348 photosensitive precursor.



Figure 10. A three-conductor-level thin-film test pattern fabricated using Probimide 348 on a 5 cm by 5 cm alumina (99.6% Al_2O_3) substrate.

ent compatibility with materials, metallizations, and processing associated with hybrid fabrication. Fundamental

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materials characterization and substrate properties have been described.¹⁸ Two previously developed hybrids originally designed to be fabricated with alumina substrates have been fabricated using AlN substrates. One was a chip and wire power hybrid using thin-film metallization; the other was a low-power chip and wire hybrid with multilayer thick-film metallization. The fabrication and testing of these hybrids to MIL-STD-883C is described. In addition to hybrid processing with the different metallization schemes, determinations of fundamental electrical, mechanical, and material properties were carried out using a custom-designed test pattern (Fig. 11). Film adhesion was monitored by tensile testing and compared with previous adhesion results obtained by shear testing.^{18,19} Resistivity and other related electrical behaviors were derived from measurements on four-point-probe and large, square (500) resistor test patterns. Material properties and hybrid process effects were evaluated with surface analysis techniques.¹¹ Because of the wide variations reported for thermal conductivity, an apparatus to measure thermal conductivity of the substrates has been designed, fabricated, and tested.²⁰

Two types of hybrids were fabricated to evaluate the possibility of replacing alumina substrates with AlN in practical applications. A low-power chip and wire hybrid, used previously in a medical application, was fabricated to test the viability of multilayer thick-film metallization. A high-power chip and wire hybrid, used previously in the AMPTE²¹ and Geosat²² satellite power control systems, was fabricated using thin-film metallization and high-heat-transfer assembly procedures. Photographs of the finished AlN hybrids are shown in Figures 12 and 13, respectively.

Fabrication of Thick-Film Hybrids

AlN substrates with dimensions of 1.78 cm by 2.90 cm and a thickness of 0.0635 cm were printed with three conductor layers, two dielectric layers (each printed twice), and two via backfills using a DuPont inking system.¹⁹ In previous studies^{18,19} a multilayer test pattern with a 0.51 cm by 0.76 cm print area was successfully printed and fired on AlN using the DuPont system. This time, the DuPont 5704 dielectric crazed when it was printed and fired on the larger dimensioned substrates because of the mismatch between the coefficients of thermal expansion of the dielectric and the AlN. An AlN study²³ reported similar crazing results when using DuPont 5704 dielectric, but the blistering they reported was not observed on our substrates. Additional substrates were then printed and fired with a specially formulated Ferro AlN ink system.²⁴ Printing and firing, continuity, and visual inspection results were excellent for both the alumina and the AlN substrates using the Ferro system. Following firing and inspection, the substrates were epoxied to the packages with Ablefilm 517. All capacitors, diodes, chip resistors, and crystals were epoxied with Ablebond 84-1LMINB (low mobile ion, no bleed) conductive epoxy, and the integrated circuits were attached using Ablefilm 517 nonconductive epoxy. The circuit was interconnected with thermosonic gold wire bonds, 25.4 μ m (1 mil) in diameter.



Figure 11. A test pattern on an AIN substrate.



Figure 12. Low-power hybrid fabricated using a multilayer thick-film conductor-dielectric system on an AIN substrate.

Fabrication of Thin-Film Hybrids

Alumina from the MRC Co. and AlN substrates from Tokuyama Soda (0.762 cm by 3.68 cm) were coated on both sides by physical vapor deposition with a chromium interface layer (0.05 μ m thick), followed by a copper deposit of 2 μ m. The thin-film metallized substrates were patterned using photolithography techniques but were not etched. Next, the substrates were electroplated on both sides, first with 11 μ m of copper and then with 2 μ m of gold. Following the pattern plating, the photoresist was removed and the thin, underlying, evaporated, chromium-copper plating conduction layers were etched. The metallized substrates were solder reflowed



Figure 13. High-power hybrid fabricated using a thin-film conductor system on an AIN substrate.

onto the packages using a gold-tin eutectic (80% by weight gold, 20% by weight tin). Eight HEXFET dies were attached using a tin-lead eutectic solder (63% by weight tin, 37% by weight lead) and ultrasonically wire bonded with 0.5% magnesium-doped aluminum wire, 127 μ m (5 mils) in diameter.

Hybrid Testing

The thick-film hybrid was tested to MIL-STD-883C environmental levels, except that the large package warranted a lower g force during centrifuge testing. Supply voltage, current measurements, and basic functional parameters for baseline testing before lidding showed no differences between the thick-film hybrids fabricated with AlN or with alumina. The prelidding baseline testing for the thin-film power hybrid included checking gate and drain leakage currents, threshold voltage, saturation voltage at three currents, and the voltage drop of the maximum-length conductor path at a high current level of 2.6 A. The results show no differences between the thin film hybrids fabricated with either AlN or with alumina. Electrical testing before and after burn-in also showed no differences between the two hybrid designs constructed with the two substrate materials.

Destructive Physical Analysis of Hybrids

Following environmental testing and burn-in (over 1000 hours at 125°C), several qualified hybrids were delidded and subjected to destructive physical analysis. In general, there were no observable changes in appearance or in physical parameters (strength, adhesion, etc.). Destructive wire pull testing was performed on the multilayer thick-film hybrids. The average pull strengths were similar both before and after burn-in, with essentially the same relative distribution of failures.²⁵

Resistivity/Resistance

Resistance measurements at room temperature were made using a constant 100-mA current source (for the 0.4- μ m-thick thin films, a 10-mA current source was used) and a precision voltmeter. For the thin films, resistance measurements of the 500-square meander line



Figure 14. Resistivity-versus-thickness characteristics for thinfilm gold on polished sapphire, 99.6% alumina, and polished AIN substrates.

and the two four-point probe patterns were averaged to calculate the resistivity-versus-thickness behavior of the gold films (Fig. 14). The purpose was to characterize the thin-film metallization on AlN, particularly effects on the electrical resistivity of the AlN's surface finish, and to look for possible substrate-film interactions.

From Figure 14, it is clear that the AlN's rougher surface finish (compared with those of the alumina and the single-crystal sapphire) had a marked influence on film resistivity. The metal film resistivity of all the substrates exhibited similar characteristic inverse thickness relationships, but the AlN resistivities were consistently higher than those of the other, smoother aluminum oxide materials. Resistivities for gold on the aluminum oxide materials were essentially the same once the film thickness reached 1.1 μ m. The extrapolated bulk resistivity for the deposited gold films is approximately $3.0 \times 10^{-6} \Omega$ cm. Even for a film thickness of 2.9 μ m, the AlN displayed a resistivity approximately 35% higher than the apparent bulk value for the other films. The extrapolated film resistivity agrees favorably with the currently accepted bulk gold resistivity²⁶ of 2.4 \times 10⁻⁶ Ω cm.

The measured results for the thick-film resistor and the four-point probe patterns are shown in Table 1. Measured values for the Ferro materials were lower than the calculated resistance and resistivity values based on Ferro's published sheet resistance. Measured values for the DuPont materials were slightly higher than their published sheet resistances. Similar results for DuPont materials were reported in a previous study¹⁸ using a different pattern. Ferro's nominal thick-film gold resistivity is $5.6 \times 10^{-6} \Omega$ cm and DuPont's is $4.7 \times 10^{-6} \Omega$ cm.

Mechanical Results

Epoxy-coated studs (0.71 cm in diameter) were clamped to the thin- and thick-film gold circles on the test substrates. The assemblies were subsequently cured at 150° C for one hour and were tensile tested using a Quad Group Sebastian III tester. None of the gold cir-

| Table | 1. | Electrical | resistance | and resistivity | measurements | for the tw | o thick-film | gold systems | on AIN | and 96% |
|-----------|----|------------|------------|-----------------|--------------|------------|--------------|--------------|--------|---------|
| AI_2O_3 | su | bstrates. | | | | | | | | |

| | Calcu | lated ¹ | Measured | | | | | | |
|-------------------------|--|--------------------|--------------------------------------|---------------|---------------|---------------|--|--|--|
| | | | Coors Al ₂ O ₃ | | Toshiba AlN | | | | |
| System | DuPont | Ferro | DuPont | Ferro | DuPont | Ferro | | | |
| | Resistance (Ω) | | | | | | | | |
| 500-square resistor | $1.0 \\ (\leq 2)^2$ | 2.7 (≤5.4) | 1.17 (2.3) | 2.29 (4.5) | 1.18 (2.3) | 2.29 (4.4) | | | |
| 250-square resistor | 0.5 (≤2) | 1.35 (≤5.4) | 0.59 (2.3) | 1.14 (4.3) | 0.59 (2.3) | 1.11 (4.3) | | | |
| | Bulk Resistivity ($\times 10^{-6} \Omega$ cm) | | | | | | | | |
| 4-point probe (Fig. 11) | 3.8 (≤2) | 6.9 (≤5.4) | 4.8 (2.5) | 5.4 (4.6) | 4.7 (2.5) | 5.6 (4.5) | | | |
| 4-point probe (Fig. 11) | 3.8 (≤2) | 6.9 (≤5.4) | 4.6 (2.4) | 5.7 (4.9) | 4.6 (2.4) | 5.8 (4.9) | | | |
| | | | | | | | | | |

¹Calculated using the manufacturers' sheet resistance and the measured thickness.

²Values in parentheses are sheet resistance in m Ω /square.

cles separated from any of the substrate materials. In almost every case, the substrate broke. The only two exceptions occurred with sapphire and 99.6% alumina, where there was a cohesive failure or separation in the bulk of the epoxy at $\geq 1760 \text{ kgf/m}^2$ (2.5 psi). There were, however, significant differences in the breaking strengths of the various substrate materials. Both the single-crystal sapphire and the 99.6% alumina polycrystalline material (with or without thin-film metallization) typically fractured at 1900 kgf/m². The polished Tokuyama Soda AlN (metallized or not) fractured even lower, at 378 kgf/m². Substrates tested without metallization were cleaned only in ultraviolet light/ozone, with no further processing, to ensure that the metal processing had no effect on the strength of the substrates.

Surface Analysis

An effective cleaning and stress-annealing method for thick-film substrates¹⁸ has been to pass uncoated substrates through the firing furnace. Concern was expressed that the operation might be detrimental to AlN substrates, since some of the surface might be converted to oxide (air firing), and thus the effective thermal conductivity might be significantly altered (i.e., AlN might be converted to $Al_x O_y$ to a great depth). An experiment was designed to compare the surface compositions of unpolished AlN substrates before and after furnace firing, using secondary ion mass spectrometry. The substrate cleaning and firing consisted of the following successive processing steps: dicing, solvent cleaning, one firing at 850°C (10-min-peak profile), ultraviolet/ozone exposure, and four additional firings at 850°C.

Figure 15 compares the profiles for the as-diced AlN with AlN subjected to the cleaning and firing cycle. Af-

ter processing, the surface oxide composition was twice that of the original material, and the surface nitrogen was halved, but the surface was far from being completely oxidized (an estimated atomic fraction of oxygen of less than 5%). The overlap of oxygen and nitrogen in the bulk of the AlN substrate occurred 2 nm from the surface after dicing and 8 nm from the surface after all the processing. In the treated substrates, the oxygen level was reduced to the original background level noted for the as-diced sample at about 40 nm from the surface.

SUMMARY

A process has been presented for the fabrication of multilevel thin-film structures. A three-metal-level thin-film test structure resulting from this method is illustrated in Figure 10. Probimide 348 is a most promising dielectric for thick-layer applications. The uniformity is best for film thicknesses around 14 μ m, which should be suitable for use with the 25- μ m traces and the 50- μ m viaholes. The photo speed is excellent; it requires about 35 mJ/cm² μ m⁻¹ to provide excellent resolution.

The soft-baked film is transparent even for relatively thick films (> 30 μ m), making it ideal for alignment to underlying patterned features and registration marks. The shelf life of the precursor is about 4 months when it is refrigerated at temperatures just above 0°C.

Additional process development is needed to determine process parameter windows for the optimal resolution of via-holes and fine-line geometries. Via-hole shapes must also be optimized to yield sloping sidewalls (preferably 45°) to permit more uniform sputtered sidewall metallization. The process parameters include softbake time, exposure parameters such as proximity dis-



Figure 15. Secondary ion mass spectrometry profiles of Tokuyama Soda AIN samples. **A**. After dicing. **B**. After dicing, solvent and ultraviolet/ozone cleaning, and five firings at 850°C. Red indicates oxygen; black indicates nitrogen.

tance and exposure energy, development method (spray or immersion), and development time. Improving the reliability and yield of multilevel dielectrics will require improvements in the patterning of metal pads surrounding via-holes to circumvent photoresist breakdown, particularly when the film thickness exceeds 10 μ m and the via-hole diameter exceeds 100 μ m.

A low-power, thick-film hybrid and a high-power, thin-film hybrid were successfully fabricated and screened to MIL-STD-883C using the AlN substrate materials. The electrical verification for both AlN hybrids was equivalent to results obtained for companion hybrids built with alumina.

In-depth properties and processing characteristics for the AlN substrate were obtained through adhesion and electrical measurements, surface analytical techniques, and thermal conductivity tests. Thin-film conductors on AlN are more resistive than their counterparts on 99.6% alumina or on optically polished sapphire because of AlN's poorer surface finish. Polished AlN appears somewhat weak, but that caused no problems in hybrid processing. Even five furnace firings in air only slightly affected the surface oxygen content. Initial thermal conductivity measurements of AlN and alumina substrates are in basic agreement with their manufacturers' specifications.

In summary, AlN has performed satisfactorily in the fabrication of both thin- and thick-film hybrids. No major adverse effects were encountered with its use. It appears ready to meet the hybrid producers need for substrate material that is nontoxic, has high thermal conductivity, and has a controlled coefficient of thermal expansion.

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