

# Fabrication of Air-Channel Structures for Microfluidic, Microelectromechanical, and Microelectronic Applications

Dhananjay Bhusari, Hollie A. Reed, Michael Wedlake, Agnes M. Padovani, Sue Ann Bidstrup Allen, and Paul A. Kohl, *Member, IEEE*

**Abstract**—A method is presented for fabricating micro-air-channel structures encapsulated by a dielectric material using a sacrificial polymer based on polynorborene (PNB) chemistry. A spin-coated film of PNB was patterned to define the exact geometry of the air-channels using conventional lithographic and etching techniques. The sacrificial polymer was encapsulated with a permanent dielectric material. The composite was then raised to elevated temperatures to produce gaseous products which permeate through the encapsulating material ( $\text{SiO}_2$ ,  $\text{SiN}_x$  or other polymer) leaving behind minimal solid residue. Air-channels integrated with metal interconnections can be formed via a Damascene, or in-lay process. After patterning the sacrificial polymer, copper was electroplated, followed by encapsulation with the dielectric. Various issues pertaining to the processing steps have been investigated and are discussed, such as type of encapsulants, feasible air-channel sizes, and processing conditions. Such air-channel structures are believed to have potential applications in microelectronics, displays, printers, multilevel wiring boards, microscale chemical reactors on a chip, and microelectromechanical devices. [586]

## I. INTRODUCTION

A vast array of microfluidic and microelectromechanical devices have been disclosed in the recent years that offer the potential to improve our way of life significantly by impacting the medical, pharmaceutical, environmental, electrical, and chemical technologies. It has been proposed that microfluidic devices could be used to perform a variety of tasks including combinatorial chemical synthesis, genetic diagnostics, and microchemical analysis and sensing [1]–[5]. One particular application that is already showing commercial promise is the use of microfluidic systems for the combinatorial synthesis of drug compounds [2]. The advantages of such microfluidic systems include lower labor requirements, low reagent usage and costs, and accelerated compound synthesis. One can imagine synthesizing thousands of compounds simultaneously using a single, highly integrated microfluidic device. The applications of air-channels in microelectronics include their use as low dielectric

constant medium between the metal interconnections in the integrated circuits to lower the LC delays in interconnects, enabling faster signal transmission [6]; creation of bridges in multilevel wiring boards; fabrication of microdisplays with enhanced resolution; fabrication of ink-jet printing heads with finer printing resolution, etc. In microelectromechanical devices, compressible micro-air-cavities can be used to mitigate problems with excess thermal expansion of materials; microtunnels can be used for cooling of miniature devices as well as many other applications.

However, to realize these applications, the device needs to be constructed with a complex three-dimensional (3-D) network of interconnected air-channels that can be used to transport different fluids independently. Moreover, such network of air-channels should be capable of integrating with various active as well as passive electrical and electromechanical components. While significant advancements have been made in recent years in the design of microfluidic devices and their applications, more effort is required to improve the technique currently used to fabricate these devices. The most prevalent method for fabrication of these devices involves bonding of layers of ultra-flat glasses to one another, each glass layer containing the patterned fluid channels already etched into it, to form the required complex 3-D network [7]. There are several severe limitations with this material system and layering process. First, the bonding of glass plates together leads to an obvious source of defects and low device yields [7]. The requirement that the layers be extremely flat (for proper bonding) eliminates the ability to build onto structures that have large amount of surface topography. This limits the system design and most importantly, affects the possibility of integration into integrated circuit (IC) manufacturing environments and onto more conventional IC substrates and devices. A more conformal layering technique would greatly simplify the construction process and ultimately make the design of the devices more flexible. An additional limitation to glass bonding technique is that the construction of metal lines and other structures in the glass layer is very difficult. In more complex systems that integrate electrical and nonelectrical processes, this difficulty in metallizing the upper fluidic layers is a severe limitation. Examples of applications in which the integration of metal patterns into the fluidic layers is important include electrohydrodynamic pumping system and the use of sensors in the fluid channel network.

Manuscript received June 4, 2000; revised February 27, 2001. This work was supported by the National Science Foundation. Subject Editor A. J. Ricco.

D. Bhusari is with Cypress Semiconductor Corporation, San Jose, CA 95134 USA.

H. A. Reed, A. M. Padovani, S. A. Bidstrup Allen, and P. A. Kohl are with the School of Chemical Engineering, Georgia Institute of Technology, Atlanta, GA 30332 USA (e-mail: paul.kohl@che.gatech.edu).

M. Wedlake is with Advanced Micro Devices (AMD), Inc., Sunnyvale, CA 94088 USA.

Publisher Item Identifier S 1057-7157(01)05032-6.

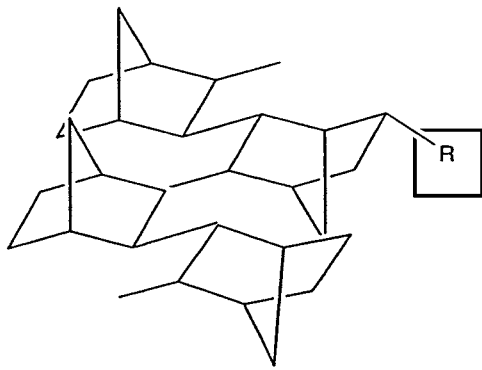


Fig. 1. Generic structure of polynorbornene.

Another important issue with regard to pumping and fluid control is the use of regions within the channels that are selectively made either hydrophobic or hydrophilic to control fluid movements. Currently, a step that is separate from the fabrication process for the channels must be performed to selectively modify the surface of the channels. In general, if the fabrication and surface modification of channels could be performed in one single step, it would greatly simplify the fabrication process and reduce the cost of manufacturing these devices. Finally, laser ablation is one of the more common methods used to pattern these rigid glass layers before they are bonded together [8]. However, laser ablation is extremely slow for relatively complex devices and limits the overall flexibility of the structures that can be manufactured [8]. A process that uses more conventional lithographic techniques would be more desirable. Some work has been done using other fabrication techniques, but there is still a general lack of materials and processes that would allow for the low cost integration and manufacture of microfluidic structures with more complex electrical structures such as IC devices.

We propose here a novel, general-purpose, buried microchannel technology that is amenable to mass production of micrometer size systems, since this technology utilizes silicon fabrication methods and equipment (CMOS compatible) leading to high volume, foundry implementable, mass formation of microsystems. The core of this technology is its ability to create a void of any arbitrary shape and size within a suitable dielectric medium, by using a sacrificial material as a 'place holder' that can be patterned into desired shape using the conventional lithographic techniques. After encapsulating the patterned layer of sacrificial material with suitable dielectric, the whole structure is raised to elevated temperatures which causes thermal decomposition of the sacrificial material, with the gaseous decomposition products permeating through the encapsulating dielectric; thus leaving a void behind in the space it had previously occupied within the dielectric medium. The use of a sacrificial material (whose decomposition products can permeate the solid overcoat) can potentially simplify the processing of microfluidic structures, allow finer features to be fabricated, and enable multilayer build-ups.

The sacrificial polymer used in this study is based on polynorbornene chemistry. The generic structure of the norbornene monomer is shown in Fig. 1. The "R" group attached to these monomers can be tailored to impart the desired properties to the polymer and achieve some specific purposes, such as adhesion

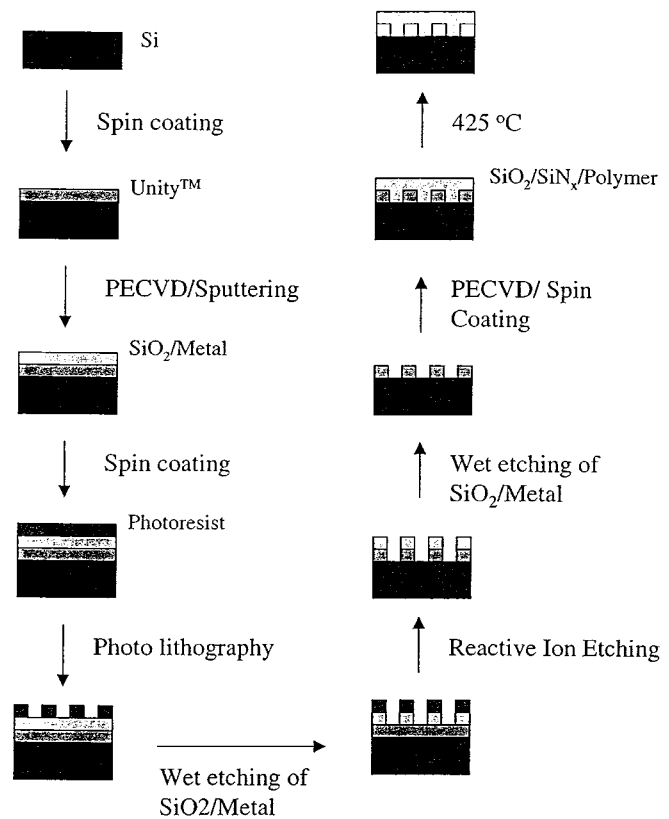


Fig. 2. Process for fabrication of air-channels encapsulated with  $\text{SiO}_2$ ,  $\text{SiN}_x$ , or polymer.

to the desired substrate and mechanical or thermal properties [9], [10]; and also to render the inner surface of the resultant cavity either hydrophilic or hydrophobic. The molecular weight of the polymer can be controlled to high accuracy by regulating the amount of chain transfer agent used in the polymerization. In order to meet specific application requirements, the molecular weight may range from a few tens of thousands to a few million amu. The specific polymer used in this study is poly butylnorbornene (hereafter generally denoted as PNB) with 10 wt.% of the polymer functionalized with triethoxysilyl (TES) side groups, to provide adequate adhesion of the polymer with the Si or  $\text{SiO}_2$  substrate. The TES groups provide excellent adhesion to metals (Cu, Au, Ag, Al, Ta, W, Ti) and oxides [9]. This is a highly desirable property for the application of PNB as a sacrificial material for forming air-channels because many organic adhesion promoters may leave solid decomposition products and residues in the air-channels upon high temperature decomposition of the polymer.

## II. EXPERIMENTAL

The process flow for fabrication of air-channel structures is depicted in Fig. 2. The thickness of the polymer film (i.e., ultimately the height of the air-channel) was controlled by both the weight fraction of the polymer in solution as well as the spin speed. A 10 to 23 wt.% solution of PNB in mesitylene was spin coated onto 3000 Å thermal  $\text{SiO}_2$  at spin speeds between 2500–4500 rpm, resulting in layers 1 to 12  $\mu\text{m}$  thick. In the experiments where the air-channels were to be fabricated between

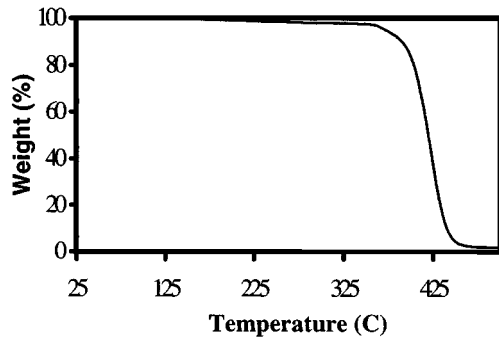


Fig. 3. Thermogravimetric spectrum of 90/10 BuPNB/TESNB recorded at the ramp rate of 3 °C/min.

Cu lines, a layer of Ti/Cu/Ti with a thickness of 20/100/20 nm was deposited by dc sputtering on the thermally grown SiO<sub>2</sub> layer before spin coating the polymer. After spin coating, the film was softbaked by heating on a hotplate at 120 °C for 3 min to evaporate the solvent. Samples that were to be electroplated or sputtered underwent an additional solvent removal step by heating in a N<sub>2</sub> purged tube furnace ramped at 5 °C/min to 200 °C and held for 2 h. Next, either SiO<sub>2</sub>, Cr, or Al was deposited to serve as a hard mask for the subsequent patterning of PNB. The SiO<sub>2</sub> mask was deposited by plasma enhanced chemical vapor deposition (PECVD) at 200 °C using the gas mixture of N<sub>2</sub>O and SiH<sub>4</sub> diluted in N<sub>2</sub>. Oxide thickness ranged from 0.5 to 1.0 μm, depending on the thickness of the PNB. Typical PECVD conditions were: chamber pressure = 550 mTorr; gas flow rates = 1400 sccm N<sub>2</sub>O and 400 sccm of 2% SiH<sub>4</sub> in N<sub>2</sub>; RF frequency = 380 KHz and RF power = 50 W. Aluminum and chromium masks were deposited using dc sputtering at room temperature. The aluminum deposition rate was 4.0 Å/s, and that of chromium was 1 Å/s. The hard mask was patterned by a standard photolithography method using a positive photoresist (Shipley Microposit 1813 or 1827) and exposing it to UV radiation for a total dose of 100 mJ. The resist was developed in Shipley Microposit 319 or 354 developer for 50 to 120 s. The exposed areas in the photoresist were then removed. SiO<sub>2</sub> was removed at a rate of 250 nm/min in 6 : 1 buffered oxide etch (BOE) solution diluted to 10 : 1 in water. Aluminum was etched using PAN (phosphoric acid/ acetic acid/ nitric acid) etch Type A heated to 50 °C at a rate of 100Å/s. Cr was etched using Mantek Co. CR-7S Chromium etchant. The SiO<sub>2</sub> and the PNB was removed by reactive ion etching (RIE). The RIE conditions were: gas flow rates = 40 sccm Ar, 15 sccm O<sub>2</sub>, and 5 sccm CHF<sub>3</sub>; chamber pressure = 300 mtorr; dc power = 300 W at room temperature. The typical etch rate of PNB under these conditions is approximately 250 nm/min. After etching the exposed PNB, the hard mask was removed, leaving only a patterned PNB film on the substrate. This was followed by either blanket coating the desired encapsulation material (such as SiO<sub>2</sub>, SiN<sub>x</sub> or other low-*k* polymer) or electroplating Cu, if the air-channels were to be created between the Cu lines. SiO<sub>2</sub> and SiN<sub>x</sub> were deposited using PECVD and the polymeric encapsulant was spin coated or doctor bladed under appropriate conditions. After encapsulating the patterned PNB, it was decomposed at 425 °C for 2 h in a Lindberg tube furnace constantly purged with N<sub>2</sub> in order to minimize residual oxygen in the furnace.

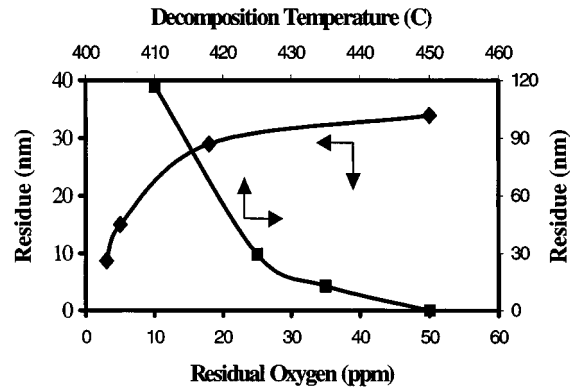


Fig. 4. Thickness of the residue after thermal decomposition of 90/10 BuPNB/TESNB at various decomposition temperatures and different levels of residual oxygen. For the decomposition temperature series, the level of oxygen was 3 ppm and for the oxygen level series, the decomposition temperature was 425 °C.

### III. RESULTS AND DISCUSSION

#### A. Decomposition Characteristics of PNB

The thermal decomposition characteristics of PNB were studied by carrying out thermogravimetric analyzes (TGA). The dynamic TGA spectrum of the PNB, recorded at the ramp rate of 3°C/min under constant N<sub>2</sub> purging, is shown in Fig. 3. The spectrum demonstrates that this polymer is highly stable and exhibits negligible weight loss at temperatures less than 350 °C. Decomposition occurs between 370 °C and 425 °C. Stability at temperatures below 370 °C is desirable so that processing steps following application of PNB can be performed without polymer degradation. This includes PECVD deposition of the mask or encapsulating SiO<sub>2</sub>. The sharp transition to gaseous products at higher temperatures minimizes the temperature window required for decomposition.

The factors greatly influencing the amount of residue left after decomposition of PNB include weight percent and type of substituents on the polynorbornene backbone, decomposition temperature, and residual oxygen concentration during decomposition. Fig. 4 presents the thickness of the residue as a function of decomposition temperature and residual oxygen level, as measured by XPS profiling after decomposition of a 7.5-μm-thick film of PNB. The rate of sputtering of the residue by Ar-ions during XPS profiling was calibrated by measuring the thickness of the residue of a patterned PNB film by atomic force microscopy (AFM). The PNB films were coated on a gold layer for the XPS studies in order to enable accurate measurement of the residue thickness. As can be seen from Fig. 4, 425 °C appears to be the optimum temperature for PNB decomposition. Incomplete decomposition occurs at temperatures lower than 425 °C while higher temperature yields no appreciable benefit. Longer times at temperatures less than 425 °C could mitigate this problem but at the expense of processing time. Therefore, 425 °C was used as the decomposition temperature in all subsequent experiments. The amount of solid decomposition products produced was found to be directly proportional to the partial pressure of oxygen in the furnace. This is because higher levels of residual oxygen result in oxidation of the polymer producing some amount of nonvolatile products. In Fig. 4, the sam-

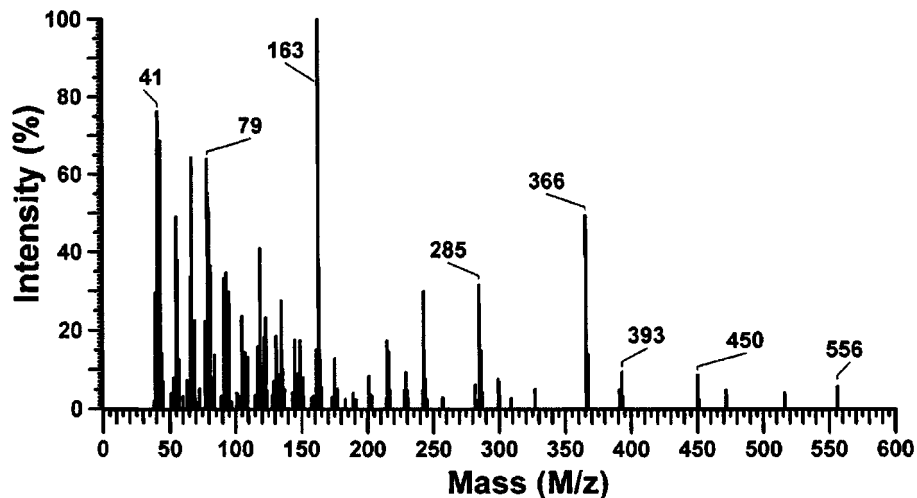


Fig. 5. Mass spectrum of 90/10 BuPNB/TESNB recorded at 425 °C.

ples decomposed at 425 °C had an oxygen concentration of 3 ppm. The residue in Fig. 4 was uniform across each sample and reproducible from sample-to-sample. The silicon surface after decomposition (with the residue shown in Fig. 4) had a hydrophobic nature, as determined by a water drop (contact angle of a water drop) experiment. Heating the surface in air to above 300 °C changed the surface to hydrophilic. This behavior is being investigated further and will be the subject of a future publication.

A mass spectrum of the decomposition products of the PNB was studied. A typical mass spectrum of a free standing film of PNB recorded at 425 °C is shown in Fig. 5. The major peaks at 41 to 79 correspond to  $C_3$ ,  $C_4$ ,  $C_5$  and  $C_6$  fragments. The peak at 163 is for triethoxysilane norbornene. The peaks at 285 and 366 correspond to dimers and trimers respectively. A more detailed study of the thermal decomposition products of PNB will be the subject of a future publication.

### B. Air-Channels With $SiO_2$ or $SiN_x$ Encapsulation

Air-channel structures encapsulated in  $SiO_2$  or  $SiN_x$  were fabricated by PECVD blanket coating the overcoat material onto the patterned PNB film, followed by decomposition of the PNB at 425 °C. Fig. 6(a) and (b) present the scanning electron micrographs of the cross section of air-channel structures encapsulated in  $SiO_2$  and  $SiN_x$ , respectively. The air-channels shown in these pictures are the same shapes as the patterned PNB without deformation or distortion of the encapsulating layer. Both  $SiO_2$  and  $SiN_x$  have adequate permeability to the gaseous products formed at elevated temperatures and can withstand the pressure needed for permeation. Pertinent processing issues with  $SiO_2$  and  $SiN_x$  air-channels are the temperature for deposition of these layers and the possible dimensions of the air-channel structures. The  $SiO_2$  and  $SiN_x$  deposition was limited to 200 °C and 230 °C, respectively. Cracks occurred in the glass at higher temperatures. The most important factor limiting the deposition temperature of both  $SiO_2$  and  $SiN_x$  films is the mismatch between the coefficient of thermal expansion of PNB (CTE = 80 to 85 ppm/°C) and  $SiO_2$  (CTE = 0.6 to 0.9 ppm/°C), or  $SiN_x$  (CTE = 1.0 to 2.2 ppm/°C). Due to this mismatch, the

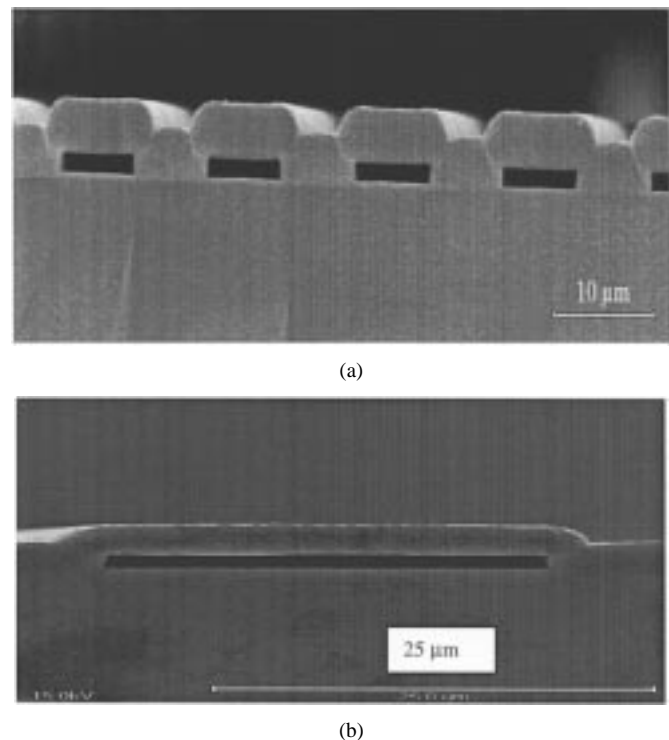
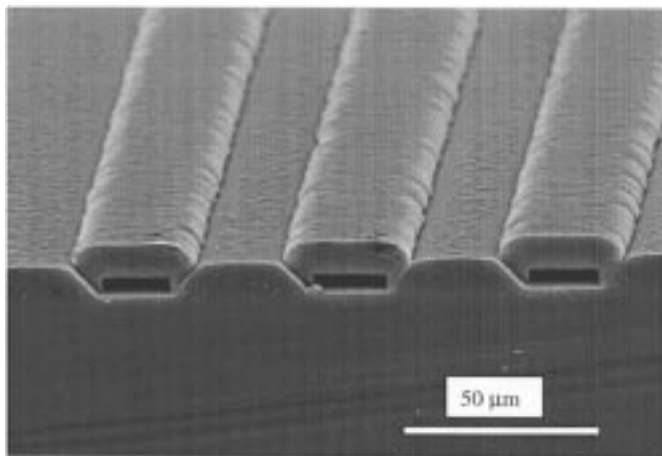
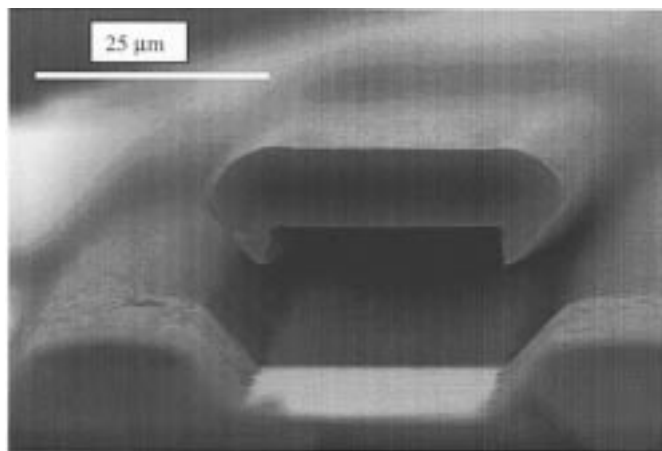


Fig. 6. Typical air-channel structures encapsulated in (a)  $SiO_2$  and (b)  $SiN_x$ .

overcoat films tend to crack when excessive stress is developed between the substrate and the film during cooling from the deposition temperature. The limits of temperature are found to be 200 °C for deposition of  $SiO_2$  and 230 °C for  $SiN_x$  on PNB. Good mechanical strength of the air-channel was found over a range of dimensions of the structures. Step coverage at the corners of PNB by the encapsulating material was critical. Fig. 7(a) shows an example of poor step coverage, which was more critical for  $SiO_2$  than for  $SiN_x$ .  $SiN_x$  yields satisfactory step coverage over a rather broad range of growth parameters whereas  $SiO_2$  coating suffers from inferior step coverage over a large range of parameters. This problem can, however, be mitigated by varying the PECVD source gas or deposition conditions. As can be seen from Fig. 7(a), the boundary between the horizontal



(a)

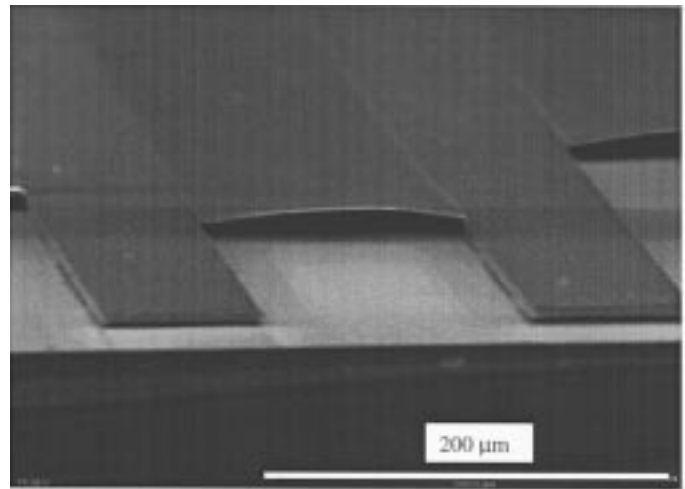


(b)

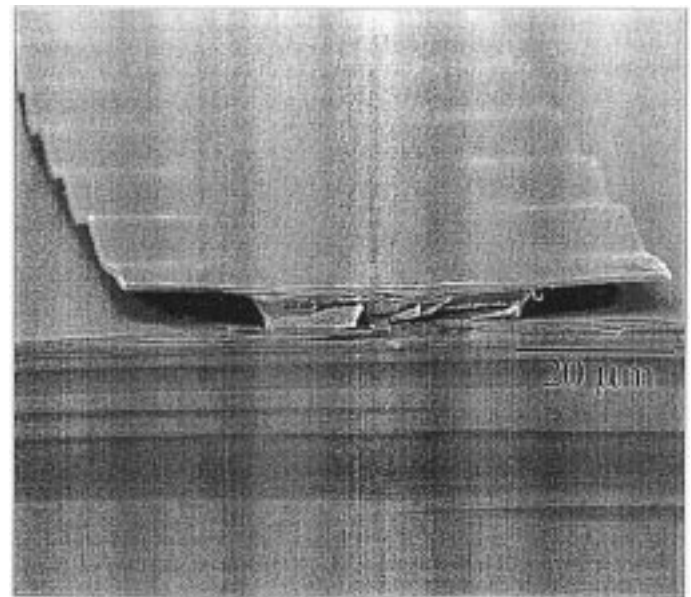
Fig. 7. (a) Example of poor step coverage with PECVD  $\text{SiO}_2$  and (b) the resulting damage to the overcoat.

and vertical faces of the  $\text{SiO}_2$  coating is the weakest point in the air-channel structure and cleavage occurs along this plane during decomposition or post-decomposition handling, as can be seen in Fig. 7(b).

The limiting factor for air-channel dimensions of  $\text{SiO}_2$  and  $\text{SiN}_x$  structures has been found to be the length of the unsupported span of the overcoat layer (i.e., width of the air-channel). Fig. 8(a) shows an example of bowed  $\text{SiO}_2$  after decomposition. This deformation occurs because the  $\text{SiO}_2$  layer is deposited at  $200^\circ\text{C}$ , and its coefficient of thermal expansion is less than that of the substrate. The layer is then subjected to annealing at  $425^\circ\text{C}$  with high pressure developing inside the cavity, possibly resulting in further deformation. In practice, air-channels as wide as  $50\ \mu\text{m}$  and up to  $10\ \mu\text{m}$  in height are readily fabricated. There appears to be no minimum width or maximum length limits;  $0.28\ \mu\text{m}$  wide air-channels have recently been fabricated [11]. Overcoat thicknesses of 1 to  $15\ \mu\text{m}$  ( $\text{SiO}_2$  and  $\text{SiN}_x$ ) have been found to be possible without deformations. Thicker overcoats had greater mechanical strength but were less permeable. In addition, the intrinsic stress in the overcoat film as well as stresses generated at the interface due to mismatch in CTE between the overcoat and PNB may also play an important role in the structural deformation of the overcoat. Fig. 8(b) shows an example of corrugation of the overcoat layer ( $\text{SiO}_2$ )



(a)



(b)

Fig. 8. Examples of structural deformation of the overcoat. (a) "Curving up" of the overcoat for an excessively wide air-channel and (b) "corrugation" of the overcoat resulting from release of intrinsic and thermal stresses.

when a portion of the underlying PNB was removed. With the PNB present, the strain in the glass was contained.

The feasibility of fabricating multilayered air-channel structures has also been investigated. Fig. 9(a) and (b) show scanning electron micrographs of the cross section of such structures with two levels of air-channels. The top level is nonplanar in Fig. 9(a) because of the resulting topography in the surface of the first level, since no CMP or other planarization methods were used. The multilevel structures were processed by two approaches; i) the first level was completely processed including decomposition of the PNB and then the second level was formed and decomposed separately; ii) the first level PNB was patterned and blanket coated with  $\text{SiO}_2$ , then the second level was fabricated and both levels were decomposed simultaneously. Both these approaches have been found to work equally well. In the first approach, the mechanical strength of the first level has to be adequate to withstand the processing of the second level on top. In

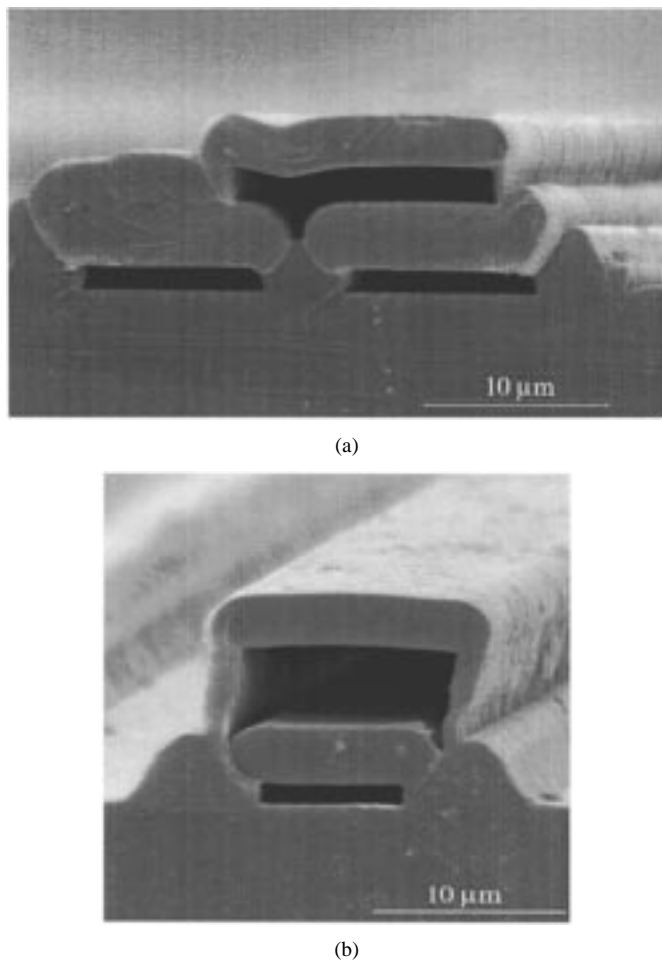


Fig. 9. Multilevel air-channels encapsulated in  $\text{SiO}_2$ .

the second approach, diffusion of the decomposition products through the overcoat material has to be adequate to allow such diffusion through multiple layers of overcoat. In actual practice, the choice of approach would be dictated by the number of layers to be processed and other processing conditions.

The feasibility of making holes in the encapsulating layer has also been investigated. These holes are intended to serve either as inlet/outlet for the fluids or to connect the successive levels of air-channels. Holes as large as  $100\ \mu\text{m}$  in diameter have been successfully wet etched into the encapsulating  $\text{SiO}_2$  prior to thermal decomposition of the PNB, using a negatively photosensitive polyimide as a mask. Although it is possible to etch the holes into the encapsulant after formation of the air-channels, doing it prior to polymer decomposition helps in enhancing the dimensional stability as well as mechanical strength of the air-channel structure by providing an outlet for the PNB decomposition products, thereby preventing the pressure build-up in the cavities. Secondly, it also prevents the etching solution to enter the channels, wherefrom it will be otherwise difficult to remove and may also damage the inner surface of the channels.

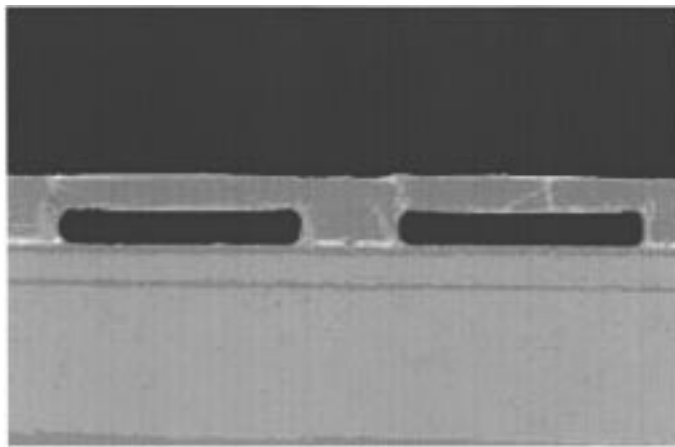
### C. Air-Channels Encapsulated in Polymer

The fabrication of air-channels encapsulated in polymer may be used to reduce the effective dielectric constant as well as the modulus of the encapsulating polymer. However, the polymer

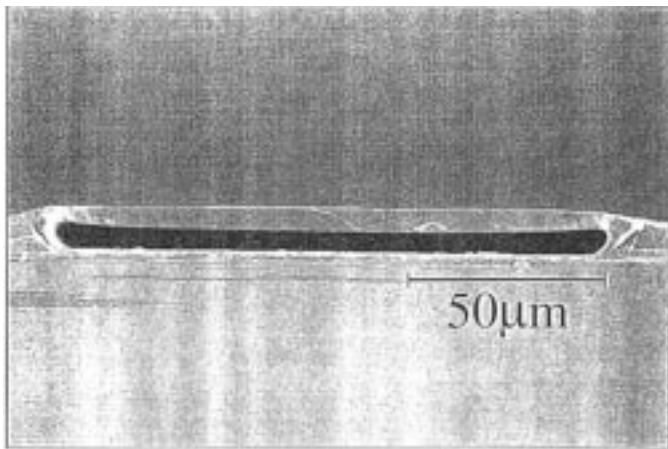
must withstand the decomposition temperature of the sacrificial material. If the overcoat material is to soften or flow, the air-channel would be severely distorted. In addition, as with  $\text{SiO}_2$  and  $\text{SiN}_x$  overcoats, the polymer must have adequate mechanical strength to span the air-channel without sagging, collapsing or rupturing during the polymer decomposition. Polyimides are a good choice for an overcoat because they display high glass transition temperature, high thermal stability, low dielectric constant, low modulus, low moisture adsorption, and relatively low stress. Using the fabrication process in Fig. 2, polymer overcoated air gaps were created with and without copper channels. Air gaps were fabricated in several shapes and sizes with three polyimide overcoats: Amoco Ultradel 7501, Dupont PI-2611, and Dupont PI-2734.

Ultradel 7501 is a preimidized photosensitive polyimide with a dielectric constant of 2.8 and a glass transition temperature greater than  $400\ ^\circ\text{C}$ . This polymer is cured between  $200\text{--}350\ ^\circ\text{C}$  and is characterized by low shrinkage. A main focus of the study using 7501 was to determine limitations of the air-channel size, shape, and overcoat material. Variables investigated for the formation of air gaps without metal lines included PNB thickness (height of the air-channel), hard mask material, width of the air-channel, and thickness of overcoat material. The height of the air-channel was varied by changing the thickness of the spin-coated PNB. Two compositions of PNB in mesitylene were applied at a range of spin speeds to achieve PNB thickness from  $4$  to  $12\ \mu\text{m}$ . Air-channels were fabricated in different three sizes:  $140\ \mu\text{m}$  channels with  $60\ \mu\text{m}$  spacing,  $70\ \mu\text{m}$  channels with  $30\ \mu\text{m}$  spacing and  $35\ \mu\text{m}$  channels with  $15\ \mu\text{m}$  spacing. In addition, wider air-cavities were also fabricated in the shapes of circles and squares ranging from  $2\ \text{mm}$  to  $1\ \text{cm}$ . The overcoat polymer was deposited by either spin-coating or doctor-blading. Overcoat thickness ranged from  $0.1$  to  $28\ \mu\text{m}$ . Fig. 10(a) and (b) show air-channels with 7501 overcoat. Fig. 10(a) shows  $35\ \mu\text{m}$  wide air-channels with  $15\ \mu\text{m}$  spacing. With these smaller cavities, the polymer is able to planarize the surface. Fig. 10(b) shows an air-channel  $140\ \mu\text{m}$  wide with  $60\ \mu\text{m}$  spacing. As the spacing between the air-channels is increased, the planarity decreases. Various shapes were seen with the wider air-channels. Sometimes the overcoat collapsed in the middle as in Fig. 10(b) and sometimes the overcoat arches upwards as in Fig. 11. Collapse occurred due to flow into the channel while expansion was driven by the pressure build-up. The overcoat material is sensitive to the decomposition ramp rate and conditions. Before PNB decomposition, rectangular channels are seen with straight side-walls and a flat top. During decomposition, the overcoat can undergo some flow, as can be seen by the rounded edges of the air-cavity. In addition, as the PNB decomposes, it produces gaseous decomposition products. The larger the initial amount of PNB present, the more gaseous products produced. As the height of the air-channel increases (i.e., more PNB present), more arching up of the overcoat is seen. Problems also occurred with very thin overcoats ( $< 1\ \mu\text{m}$ ). During decomposition either the thin overcoat film collapsed filling in the cavity, or the overcoat ruptured and cracked. These problems were not observed with thicker overcoats.

Air-channels were fabricated with Dupont PI-2611 and PI-2734. PI-2611 is a low thermal coefficient of expansion



(a)



(b)

Fig. 10. (a) and (b) Air-channels encapsulated in Amoco Ultradel 7501 with variations in the overcoat shape.

polyimide based on polyamic acid chemistry while PI-2734 is a photosensitive polyamic ester. Both polymers are cured between 350–400 °C, have a dielectric constant of 2.9 and have a glass transition temperature greater than 350 °C. Both polymers work equally well for making air-channels. Fig. 11 shows an air-channel fabricated with PI-2734. The curvature in the top of the air-channel is more pronounced with PI-2611 and PI-2734 than with Ultradel 7501, likely due to reduced glass transition temperature. Since the glass transition temperature in these two materials occurs before the decomposition temperature, some deformation occurs during decomposition, dependent on the ramp rate and decomposition conditions.

Hard mask materials included SiO<sub>2</sub>, aluminum and chromium. SiO<sub>2</sub> was used as the hard-mask for air-channels less than 1 μm high. However, the RIE etch gas CHF<sub>3</sub>, which attacks the triethoxysilane groups in PNB, also attacks the SiO<sub>2</sub>, causing mask erosion. Etch rate of the mask under the conditions used in polymer etching was determined to be 0.05 μm/min. The PECVD SiO<sub>2</sub> exhibits stress and cracking when deposited beyond 1 μm thick on PNB and was therefore not a feasible mask for PNB etching beyond 4 μm. In addition, the BOE used to etch SiO<sub>2</sub> also attacks the TES groups in the underlying PNB layer. Both chromium and aluminum hard masks were used as alternatives and were deposited using dc

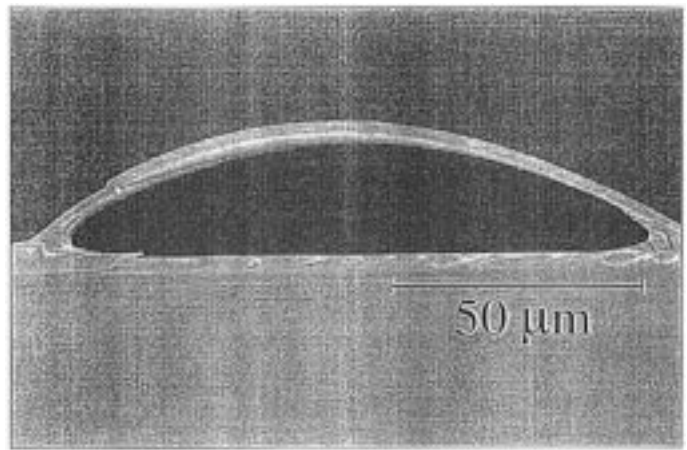


Fig. 11. Air-channel overcoated with Dupont PI-2734 displaying curving up of the overcoat.

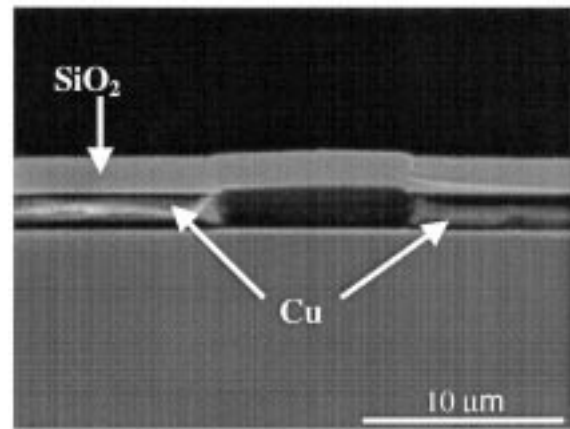


Fig. 12. Air-channels fabricated between copper line and encapsulated with SiO<sub>2</sub>.

sputtering to a thickness of 2000 Å. Problems occurred with the chrome mask from stress, likely due to CTE mismatch with the polymer. The chromium film appeared cracked immediately upon removal from the sputterer. Although the film held together during lithography and RIE, it transferred the cracked pattern into the surface of the underlying PNB layer, causing distortion in the final air-channel shape. Aluminum did not display the same problems as chromium. In addition, the etch profile improved compared to the SiO<sub>2</sub> mask, since a much thinner layer could be used, reducing shadowing effects while etching in the RIE. However, for air-channels with copper lines, a SiO<sub>2</sub> mask must be used because the Cr and Al etchants attack the underlying Ti-Cu-Ti seed layer.

#### D. Air-Channels Between Metal Lines

In order to demonstrate the feasibility of fabrication of air-channels between metal lines for their application as low-*k* intralevel dielectric in ICs as well as in microfluidic devices integrated with electrical components, air-channel structures were fabricated between copper lines and encapsulated with SiO<sub>2</sub> and polyimide, two preferred interlevel dielectric materials in the IC industry. Fig. 12 presents a scanning electron micrograph of such a sample. For these experiments, as mentioned earlier,

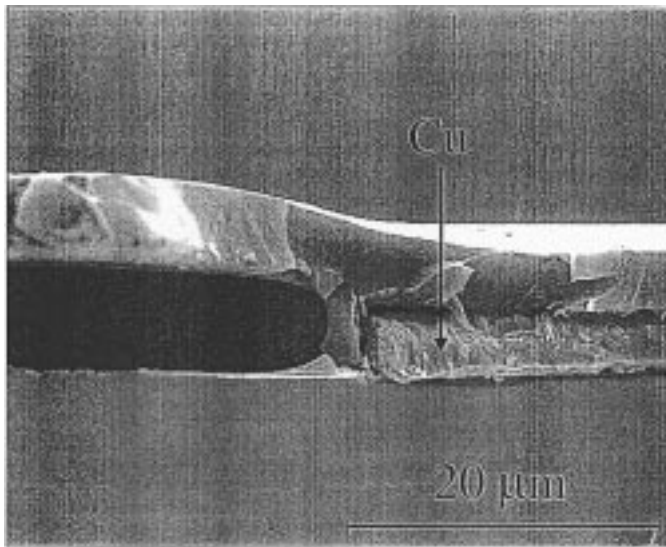


Fig. 13. Air-channels fabricated between copper lines that are insulated from the air-channel. The overcoat is Amoco Ultradel 7501.

PNB was laid and patterned on top of a Ti/Cu/Ti layer so as to allow subsequent electroplating of Cu between the patterned PNB. The time duration of electrodeposition and current were controlled to achieve same thickness of Cu as that of the PNB, and then the overcoat was applied.

Although the metal lines are electrically interconnected through the bottom metallic layer in this particular case, these structures are intended to serve as test vehicles for demonstrating the feasibility of fabrication of such metal/air-channel structures. In fact, other alternate routes for fabricating the metal/PNB structures are also possible. For example, metal can be blanket coated on patterned PNB followed by chemical mechanical polishing (CMP) or alternately the metal can be first inlaid and patterned by additive, semi-additive or subtractive means and then the PNB can be blanket coated followed by either CMP or etching by using RIE. Air-channels as narrow as 280 nm in width between 600 nm wide copper lines have been achieved by using IC compatible damascene type metallization and CMP processes [11].

Insulated Cu channels were fabricated with 7501 overcoat as shown in Fig. 13. After electroplating the copper slightly beyond the thickness of patterned PNB, the copper is etched back using nitric acid. This isotropic etch removes copper from the top and side surfaces of the Cu channels, leaving a small space between the Cu and PNB features. The polymeric overcoat has excellent gap-fill capabilities and fills-in this small space. Upon decomposition, the air-channel forms leaving Cu channels overcoated with polymer and a thin insulating layer of polymer on the sides.

#### IV. CONCLUSION

A process for fabrication of air-channel structures within a dielectric or dielectric/metal medium by using a sacrificial

polymer as 'place holder' is presented. The polymer is first spin coated and patterned into a desired geometry by photolithography and reactive ion etching techniques, and then blanket coated with suitable encapsulating layer. The polymer is then decomposed into gaseous products at elevated temperatures, which diffuse out through the encapsulating layer; leaving behind a void in the volume it had previously occupied. This method thus offers excellent control over the geometry of the air-channels. Conventional dielectric materials such as SiO<sub>2</sub>, SiN and polyimides have been found to be suitable encapsulants. Various processing as well as materials issues are discussed at length.

#### ACKNOWLEDGMENT

The intellectual and materials contributions of Dr. R. Shick and Dr. L. Rhodes (BFGoodrich Corporation) are gratefully acknowledged. The authors would also like to thank Dr. G. Prentice and Dr. R. Khosla, Program Managers at National Science Foundation, for all their support and help.

#### REFERENCES

- [1] P. Gravesen, J. Branebjerg, and O. S. Jensen, *J. Micromech. Microeng.*, vol. 3, p. 168, 1993.
- [2] R. F. Service, *Science*, vol. 282, p. 396, 1998.
- [3] C. H. Mastrangelo, M. A. Burns, and D. T. Burke, *Proc. IEEE*, vol. 86, p. 1769, 1998.
- [4] Z. Moukheiber, *Forbes*, vol. 161, p. 76, 1998.
- [5] R. Srinivasan, I.-M. Hsing, J. Ryley, M. P. Harold, K. F. Jensen, and M. A. Schmidt, *Proc. Solid State Sensor and Actuator Workshop*, 1996, p. 15.
- [6] P. A. Kohl, Q. Zhao, K. Patel, D. Schmidt, S. A. Bidstrup-Allen, R. Shick, and S. Jayaraman, *Electrochem. Solid State Lett.*, vol. 1, p. 49, 1998.
- [7] N. F. Raley, J. C. Davidson, and J. W. Balch, *Proc. SPIE*, vol. 3224, p. 185, 1997.
- [8] P. Pethig, J. P. H. Burt, A. Parton, N. Rizvi, M. S. Talary, and J. A. Tame, *J. Micromech. Microeng.*, vol. 8, p. 356, 1998.
- [9] N. R. Grove, P. A. Kohl, S. A. Bidstrup-Allen, R. A. Shick, B. L. Goodall, and S. Jayaraman, *Proc. Mater. Res. Soc. Symp.*, vol. 476, 1997, p. 3.
- [10] R. A. Shick, B. L. Goodall, L. H. McIntosh, S. Jayaraman, P. A. Kohl, S. A. Bidstrup-Allen, and N. R. Grove, *Proc. IEEE Multichip Module Conf.*, 1996, p. 182.
- [11] D. M. Bhusari, M. D. Wedlake, P. A. Kohl, C. Case, F. P. Klemens, J. Miner, B. C. Lee, R. J. Gutmann, J. J. Lee, R. Shick, and L. Rhodes, *Mater. Res. Soc. Symp. Proc.*, vol. 612, 2000.



**Dhananjay Bhusari** received the B.S. and M.S. Degrees in physics from Nagpur University, Nagpur, India in 1986 and 1988, respectively; and the Ph.D. degree in solid state physics from University of Poona, Pune, India, in 1994.

From 1988 to 1993, he was a Council of Scientific and Industrial Research Fellow in National Chemical Laboratory, Pune, where studied the structural short range order in hydrogenated amorphous silicon based alloys. From 1994 to 1998, he was a Visiting Researcher at the Institute of Atomic and Molecular Research, Academia Sinica, Taipei, Taiwan, where he worked on nanocrystalline diamond and other superhard materials. Currently, he is working at Cypress Semiconductor Corporation, San Jose, CA.





**Hollie A. Reed** received the B.E. degree in chemical engineering from Youngstown State University, Youngstown, OH, in 1998. She is currently pursuing the Ph.D. degree from the school of Chemical Engineering at the Georgia Institute of Technology, Atlanta.

Her research interests are mainly in the areas of microfabrication and chemical processing, with applications in microfluidics, wafer-level packaging and testing, and lab-on-a-silicon chip.

**Michael Wedlake** received the B.S. degree in chemical engineering from The University of Texas and the M.S. degree from Georgia Institute of Technology, Atlanta. He is currently employed by Advanced Micro Devices (AMD), Inc., Sunnyvale, CA.



**Agnes M. Padovani** received the B.S. degree in chemical engineering from the University of Puerto Rico, Mayaguez, Puerto Rico, in 1997 and is currently pursuing her Ph.D. degree in chemical engineering from the Georgia Institute of Technology, Atlanta. Her research areas focus on the development and characterization of low dielectric constant materials for advanced interconnects.



**Sue Ann Bidstrup Allen** received the S.B. degree in chemical engineering from the Massachusetts Institute of Technology (MIT), Cambridge, in 1981 and the Ph.D. degree from the University of Minnesota, Minneapolis, in 1986.

She spent two years as a Postdoctoral Associate in the electrical engineering department at MIT. She currently holds the position of Professor in the School of Chemical Engineering at the Georgia Institute of Technology, Atlanta. Her area of research is focused on the application of polymeric materials in micro-

electronics.

**Paul A. Kohl** (M'92) received the Ph.D. degree from The University of Texas at Austin in 1978.

From 1978 to 1989, he worked at AT&T Bell Laboratories. He is currently Regents' Professor of chemical engineering at the Georgia Institute of Technology, Atlanta.

