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Improved fabrication of micro air-channels by incorporation of a structural barrier

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Abstract

The fabrication of air-channels for microelectromechanical systems and microfluidic devices using polynorbornene and polycarbonates as thermally or photolitically decomposable materials to form air-gaps in dielectric materials has been reported. In this study, the incompatibility of some overcoat polymers with the sacrificial materials was addressed. SiO_2 was used as a barrier layer for the fabrication of single- and multi-layer air-channels via different sacrificial and overcoat materials. The structural rigidity of SiO_2 mitigates problems associated with overcoat polymers that can easily deform at the processing temperature (overcoat cure or sacrificial decomposition temperature). The chemical inertness and low permeability of SiO_2 allows the use of solvent-cast polymers, for which the solvents would have otherwise dissolved the sacrificial material.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Air-gaps or air-channels are of interest in a range of microelectronic applications. For example, air-gaps can provide ultra low-k electrical interconnects for electrical devices [1, 2]. A wide spectrum of microelectronic and microelectromechanical systems (MEMS) applications need thermally decomposable sacrificial materials for the fabrication of structures and moving parts. Microfluidic devices have tremendous applications in a variety of fields including chemical synthesis, drug discovery, biomedical testing and analysis [3–5]. In such devices, liquids and gases are allowed to pass through the micro air-channels with a wide variety of cross-sectional and feature dimensions. Chemical processing in micro air-channel devices provides several advantages over large-scale techniques including low reagent and analyte consumption, highly compact and portable systems for easy transportation, fast processing times and the potential for disposable systems.

Sacrificial polymers have been used to make enclosed air-channels by providing temporary placeholders that are patterned to the desired shape and geometry of the eventual air-gap [6–10]. After patterning, the sacrificial material is encapsulated by an overcoat material, such as an inorganic glass or an organic polymer. Polymer overcoats can provide advantages such as larger achievable dimensions, lower overcoat film stress, lower elastic modulus and selective permeability. The overcoat polymer is deposited then processed or cured, and subsequently the structure is heated to the decomposition temperature of the sacrificial material. The air-gap is formed when the sacrificial material decomposes, and its gaseous products permeate through the overcoat polymer. The air-gap takes on the exact shape of the sacrificial material, if the overcoat polymer is rigid at the process temperatures, and the solvent for the overcoat polymer does not dissolve the sacrificial material [9, 10]. Several acceptable combinations of sacrificial materials and overcoat polymers have been found, but the approach is not universal. In some cases, there is a solubility problem between the overcoat polymer and the sacrificial polymer. For example, a norbornene-based overcoat, Avatrel 2090P, can be used with a polypropylene carbonate (Unity 2207P) sacrificial material

but not with a polycyclohexene carbonate (Unity 2507P) sacrificial material. The Avatrel solvent, mesitylene, dissolves Unity 2507P but not Unity 2207P. Further, when building an air-gap on a polymer base, Unity 2207P sacrificial material cannot be used on an Avatrel base because the solvent for Unity 2207P, anisole, dissolves Avatrel. In other cases, the overcoat material softens or loses mechanical strength at the decomposition temperature and sags or flows into the cavity, or permanently deforms (e.g. expands) from pressure build-up inside the cavity. Thus, complex materials processing and design rules have evolved. In order to accommodate a wider variety of overcoat polymers, the patterned sacrificial material should be resistant to *any* solvent used to cast the overcoat polymer (where the solvent may lead to the dissolution of the sacrificial material). Also, the polymer overcoat must provide excellent mechanical strength to span the eventual air-gap during and after fabrication without sagging or expanding. This is especially critical at the highest temperature used in the process (e.g. the decomposition temperature of the sacrificial material) because the overcoat polymer may be near or above its glass transition temperature, and the sacrificial polymer may exist in liquid form.

In this paper, we report a general approach enabling the use of any solvent or overcoat polymer to fabricate air-gaps. Plasma deposited SiO_2 has been introduced as a barrier between the sacrificial material and overcoat polymer as a general solution to the solvent-incompatibility and soft-overcoat problems. We have demonstrated the fabrication of air-channels that would otherwise be impossible to fabricate without the SiO_2 layer. The deposition of a thin layer of SiO_2 above the patterned sacrificial material before overcoating the dielectric polymer successfully addresses the overcoat sagging and solvent incompatibility problems. The use of SiO_2 on top of an 'incompatible' polymer base is also shown. This strategy was used to fabricate single- and multi-layer air-channels. SiO_2 on top of the overcoat polymer also provides structural support to overcoat materials that soften and sag at or near the decomposition temperature.

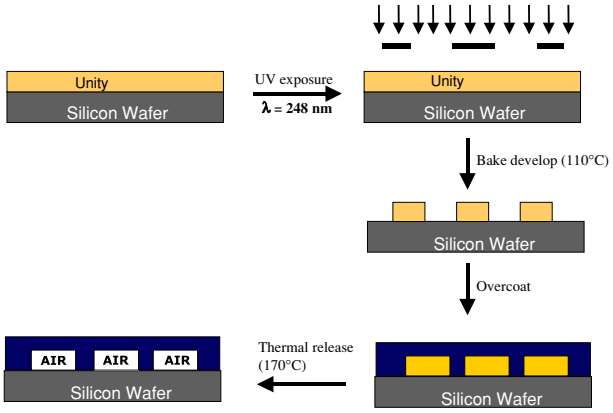

Figure 1. Fabrication sequence for the formation of embedded air-channels in polymer dielectric material using photosensitive polycarbonate sacrificial materials.

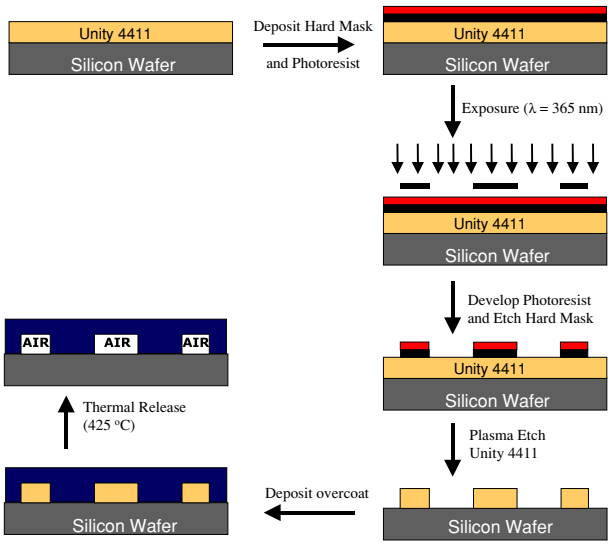

Figure 2. Fabrication sequence for the formation of embedded air-channels in polymer dielectric material using Unity 4411.

2. Experimental details

Numerous overcoat polymers were used in this study: Pyralin PI-2540, PI-2555, PI-2611, PI-2734 and PI-2771, and HD4000 (HD Microsystems, Parlin, NJ); Avatrel EPM and Avatrel 2090P (Promerus LLC, Brecksville, OH); SU-8 2025 (Microchem Corp., Newton, MA); Cyclotene 3022-63 (Dow Chemical, Midland, MI), Sumiresin Excel CRC-8650 (Sumitomo Bakelite, Japan); and Photoneece DL-1000 and PWDC-1000 (Dow Corning, Midland, MI). These were used as the overcoat polymers in the fabrication of both single- and double-layer micro air-channels. The sacrificial materials: Unity 2207P (containing polypropylene carbonate), Unity 2507P (containing polycyclohexene carbonate), Unity 3807P (containing polynorbornene carbonate) and Unity 4411 (containing polynorbornene) were externally obtained (Promerus LLC, Brecksville, OH).

Air-channels were fabricated with the photosensitive polycarbonate-based Unity sacrificial polymers via the process flow shown in figure 1. First, Unity was spun onto the silicon wafer (typical thickness 14 μm). The film was then soft-baked at 110 °C for 10 min to remove the solvent. The photosensitive Unity was photopatterned using a clear-field mask having 100 μm wide lines and 240 μm wide spaces using 248 nm UV exposure at a dose of 1 J cm^{-2}. This was followed by bake development at 110 °C for 10 min to decompose the UV irradiated area. Plasma enhanced chemically vapor deposited (PECVD) silicon dioxide (deposited at 100 °C to 150 °C) was used as a thin-film structural material. The encapsulating polymer was then deposited and soft-baked at 80 °C for 5 min. Finally, the Unity was decomposed at 180 °C for 1 h.

The air-gap fabrication process with Unity 4411 begins with deposition of the sacrificial material onto the substrate. A 30.8 wt% polymer solution in mesitylene was used to spin-coat 5 μm thick films. In the case of non-photosensitive Unity 4411, a hard mask and reactive ion etching were used for patterning as shown in figure 2 [9]. Air-channels were fabricated using a clear-field mask pattern containing four different size line/space patterns: 140 μm wide channels with 60 μm spacing, 70 μm wide channels with 30 μm spacing, 35 μm wide channels with 15 μm spacing, and 15 μm wide channels with 5 μm spacing. The overcoat polymers were deposited and cured according to recommended procedures from the manufacturers. PECVD silicon dioxide

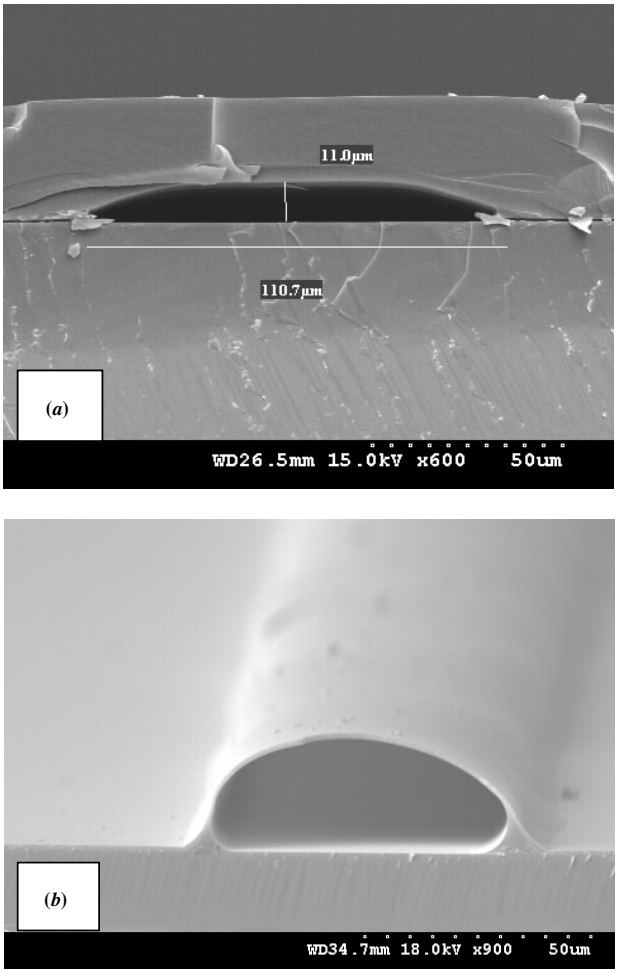

Figure 3. Air-channel fabricated using Unity™ 2207P as a sacrificial polymer and overcoat: (*a*) Avatrel 2090P and (*b*) SiO_2.

was deposited at 200 °C in a commercial system operated at the specific temperature stated. After depositing the overcoat, the decomposition of the remaining Unity 4411 sacrificial material was performed at a maximum temperature of 425 °C using a nitrogen-purged Lindberg horizontal tube furnace. The resulting air-channels were examined. In general, the air-channels were clean from residue and defects.

3. Results and discussion

Figures 3(*a*) and (*b*) show cross-sectional SEM images of embedded air-channels that were fabricated using Unity 2207P as a sacrificial material and Avatrel 2090P and PECVD SiO_2 as overcoat materials, respectively. The combination of sacrificial material (Unity 2207P) and overcoats (Avatrel or SiO_2) are compatible with each other (no solvent interference or mechanical deformation), and demonstrate favorable results from air-channel fabrication.

The first 13 rows of table 1 describe the different sacrificial materials and overcoat polymer combinations. Those combinations that were 'compatible' (no solvent or thermal/mechanical property problems) are identified by 'G', while those that are not compatible are identified by 'B' (destruction of the sacrificial material or deformation of the overcoat was visually observed). Table 1 shows that the

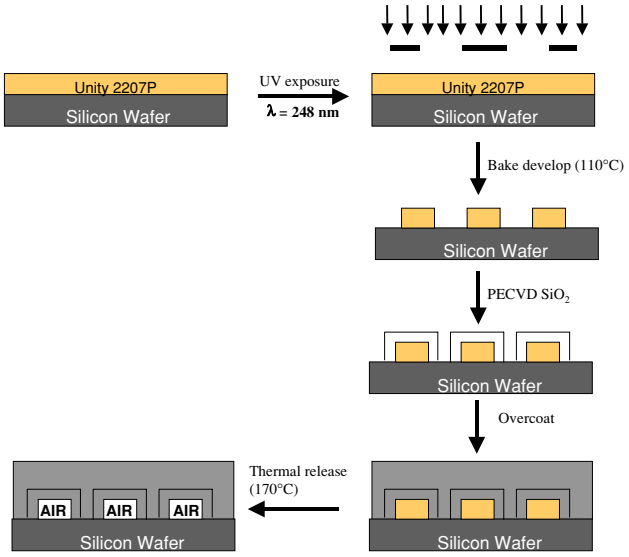

Figure 4. Fabrication sequence for the formation of embedded air-channels incorporating SiO_2 layer.

selection of the sacrificial materials and the overcoat polymers used to fabricate the microchannels is limited due to either solvent incompatibility between the sacrificial material and the overcoat polymer, or poor temperature resistance of the overcoat material to the decomposition temperature of the sacrificial material. Those combinations that did not work were reprocessed with a thin layer of SiO_2 between the sacrificial material and the overcoat polymer.

First, the solvent incompatibility issue will be addressed. If the solvent used to cast the overcoat polymer dissolves the sacrificial material, the pattern defined by the sacrificial material is distorted or destroyed. This processing issue is addressed by adding an additional step to the process—depositing a thin layer of PECVD SiO_2 as a barrier to prevent the sacrificial feature from contacting the overcoat solvent. Figure 4 shows a typical process: a 4.5 μm thick pattern of Unity 2207P was formed, followed by the deposition of a thin layer of SiO_2 (∼100 to 1000 Å), as described in the experimental section. The overcoat polymer was then spin-coated and soft-baked at 85 °C for 10 min. The Unity 2207P was decomposed at 180 °C in a nitrogen atmosphere to form the air-channels. Unity 2507P and Unity 3807P patterns dissolve in the solvent of the Avatrel dielectric polymers, resulting in the removal or gross distortion of the Unity pattern. Figure 5(*a*) shows a cross-sectional scanning electron micrograph (SEM) image of an air-channel formed using Unity 2507P, a thin SiO_2 layer and Avatrel EPM as the overcoat polymer. The thin layer of PECVD SiO_2 (∼4000 Å) as a solvent barrier layer can be clearly seen in figure 5(*b*). If the SiO_2 had not been used, the Unity would have dissolved in the Avatrel solvent (mesitylene) and no air-channel would have been produced. Rows 14 to 23 of table 1 summarize the combinations of sacrificial materials and polymer overcoats which are compatible when SiO_2 is used.

Experiments were performed to optimize the thickness of the SiO_2 when used as a solvent barrier and support layer. Samples were fabricated with four variations in SiO_2 thickness (1023 Å, 790 Å, 530 Å and 283 Å) on the Unity 2507P patterns. The build-up sequence consists of: Unity 2507P–SiO_2–Avatrel

Table 1. The sacrificial polymers (Unity 2207P, Unity 2507P, Unity 2807P and Unity 4411) and dielectric overcoat materials compatibility chart.

Entry	Dielectric overcoat material	Overcoat solvent	Unity 2207P	Unity 2507P	Unity 3807P	Unity 4411
1	Avatrel EPM™	Mesitylene	G	B	B	B
2	Avatrel 2090P™	Mesitylene	G	B	B	B
3	Sumiresin Excel CRC-8650	GBL	B	B	B	B
4	Pyralin PI-2540	NMP	B	B	B	G
5	Pyralin PI-2555	NMP	B	B	B	B
6	Pyralin PI-2611	NMP	B	B	B	G
7	Pyralin PI-2734	NMP	B	B	B	G
8	Pyralin PI-2771	NMP	B	B	B	B
9	SU-8 2025	GBL	G	B	B	–
10	Hitachi HD4000 polyimide					B
11	Cyclotene 3022-63	Mesitylene	G	B	B	B
12	Photoneece DL-1000	GBL	B	B	B	B
13	Photoneece PWDC-1000	GBL	B	B	B	B
14	SiO_2 + Avatrel EPM™	Mesitylene	G	G	G	B
15	SiO_2 + Avatrel 2090P™	Mesitylene	G	G	G	B
16	SiO_2 + Sumiresin CRC-8650	GBL	G	G	G	G
17	SiO_2 + PI-2611	NMP	G	G	G	G
18	SiO_2 + PI-2734	NMP	G	G	G	G
19	SiO_2 + Dow Corning PI	NMP	G	G	G	G
20	SiO_2 + SU-8 25	GBL	G	G	G	G
21	SiO_2 + Cyclotene 3022-63	Mesitylene	G	G	G	G
22	SiO_2 + Photoneece DL-1000	GBL	G	G	G	G
23	SiO_2 + Photoneece PWDC-1000	GBL	G	G	G	G

G = compatible and air-channels can be formed; B = incompatible; NMP = *N*-methyl pyrolidinone; GBL = gamma-burtyrolactone.

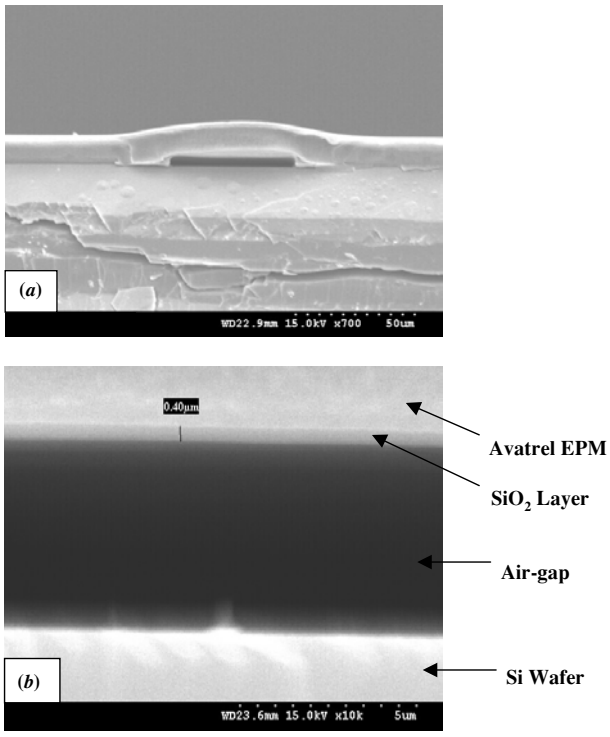

Figure 5. SEM image of an air-channel encapsulated by Avatrel (*a*); a closer view of an air-channel where a thin layer of SiO_2 is vividly shown below the overcoat polymer (*b*).

2090P and the resulting air-gaps are shown in figure 6. Only the thinnest SiO_2 layer (283 Å) was unable to prevent distortion of the Avatrel after the Unity was decomposed. Thus, the minimum thickness of SiO_2 to prevent solvent dissolution was found to be 530 Å (figure 6(*c*)). Figure 6(*d*) shows an SEM micrograph of the collapsed Avatrel layer due to solvent interaction during Avatrel coating.

Epoxy-based overcoats are of interest in a number of MEMS applications. Figure 7 shows an SEM image of the cross-sectional image of a micro air-channel fabricated using SU-8 2025 epoxy as the overcoat for 13 μm thick Unity patterns. The SU-8 2025 encapsulant was spin-coated on to the Unity 2207P patterns at 3000 rpm for 30 s. Even though SU-8 contains an unacceptable solvent (Unity dissolves in gamma butyrolactone), the SiO_2 barrier layer was not required because the high viscosity of SU-8 2025 slowed the dissolution of the Unity. The Unity patterns stayed intact while the SU-8 2025 was dispensed and processed at 95 °C for 8 min on a hot plate. The SU-8 2025 film was later UV exposed (300 mJ cm^{-2} at 365 nm). After exposure, the SU-8 was baked at 65 °C for 1 min and 95 °C for 6 min. The final heating step (12 min at 130 °C then ramped to 190 °C and held for 60 min) resulted in the full decomposition of the Unity.

The ability to fabricate multi-layered structures requires that the Unity sacrificial polymer be processed on top of a base other than silicon or glass. Ideally, the base material should be insoluble in the solvent system used by the sacrificial material. In the cases where the solvent for the sacrificial material dissolved the base layer, a film of SiO_2 was added prior to deposition of the sacrificial material, thus preventing the sacrificial material solvent from contacting the base polymer. Figure 8 shows a stack of two air-channels fabricated on an Avatrel base with SiO_2 as a protection layer, Unity 2507P and SU-8. The SiO_2 was effective in preventing swelling and distortion of the base layer.

The Unity 4411 sacrificial material does not have the same difficulty with solvent incompatibility as the polycarbonate-based Unity sacrificial materials, due to its low solubility in many organic solvents. However, a key concern is the

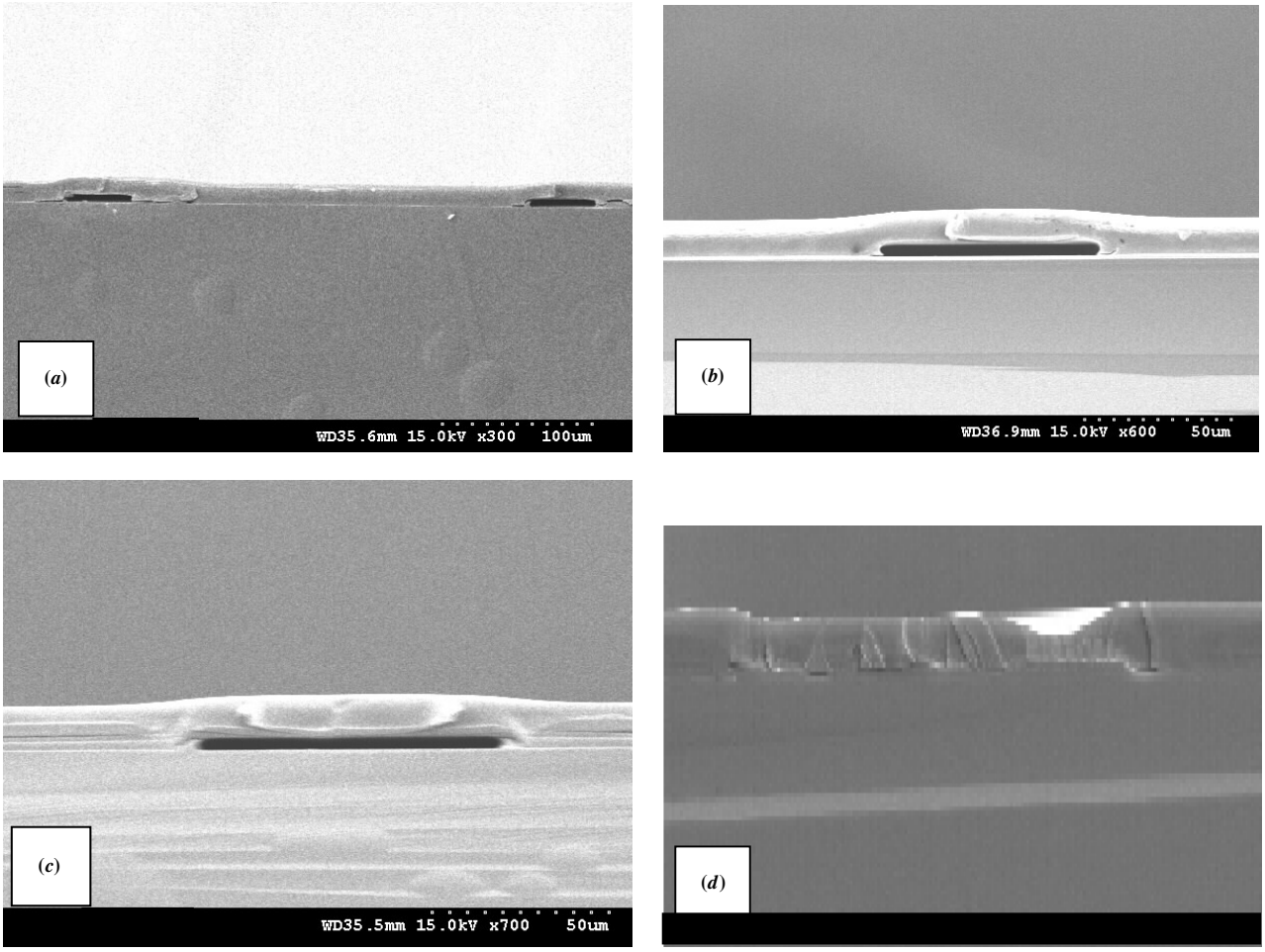

Figure 6. SEM micrographs of the cross-sections of air-channels with different thicknesses of SiO_2 layer deposition. The material sequence of deposition is: Unity 2507P + SiO_2 + Avatrel 2090P. Thicknesses of SiO_2 on the four different samples: 1023 Å (*a*), 790 Å (*b*), 530 Å (*c*) and 283 Å (*d*). The thicknesses was measured using a Plasmos Ellipsometer.

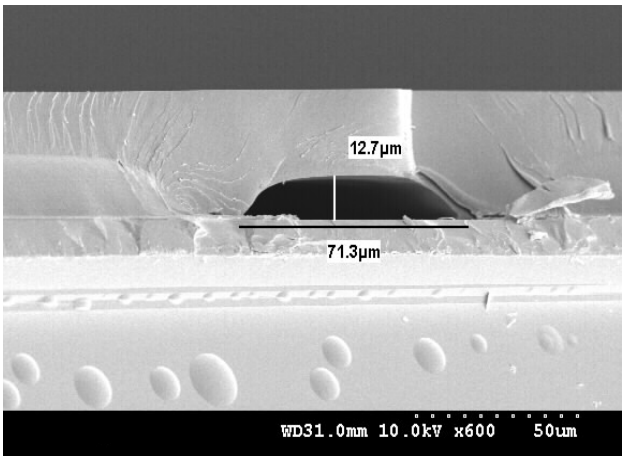

Figure 7. Air-channel fabricated using Unity™ 2207P as a sacrificial polymer and SU-8 2025 overcoat.

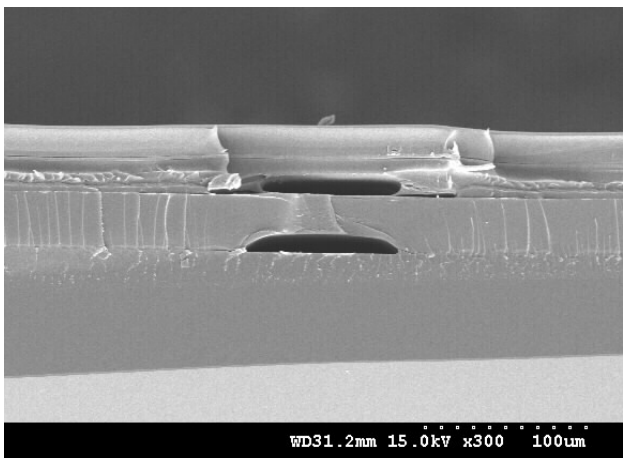

Figure 8. SEM micrographs of the double-layer embedded air-channels on an Avatrel 2090P base using Unity 2507P as the sacrificial material and SU-8 2025 as the overcoat material.

high temperature (>400 °C) required for decomposition of the sacrificial material. If the overcoat material softens or flows during the decomposition, the air-channel becomes severely distorted. The overcoat materials for use with Unity 4411 were chosen based on: (i) applications or interest in the microelectronics industry; (ii) curing temperature (below the temperature at which Unity 4411 starts to decompose); and (iii) thermal stability measured by the glass transition temperature (∼400 °C). Air-gaps have been successfully fabricated using Unity 4411 and three polyimide overcoat materials: Pyralin PI-2611, PI-2734 and PI-2540. The common

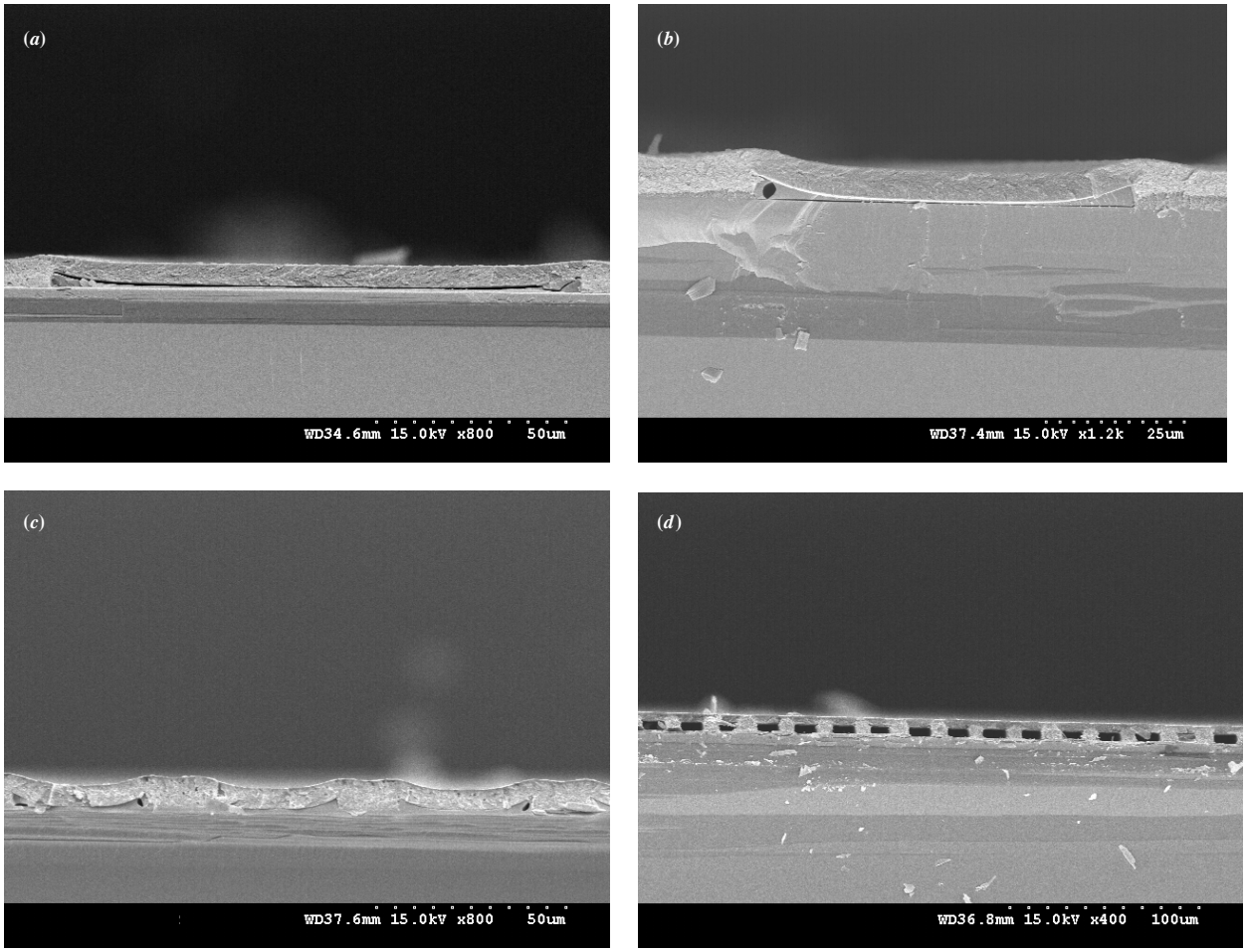

Figure 9. SEM images of features following the decomposition of 5 μm Unity 4411 overcoated with PI-2771 overcoat polyimide and channel widths of (*a*) 140 μm, (*b*) 70 μm, (*c*) 35 μm and (*d*) 15 μm.

factor between these polyimides is a high T_g (>350 °C). Several other polyimides, including PI-2555, PI-2771 and HD4000, sag into the channel during the decomposition, except in the case of small channels with a width of less than 15 μm. The T_g of PI-2555 is >320 °C, the T_g of PI-2771 is 294 °C, and the T_g of HD4000 is 350 °C. Figures 9(*a*)–(*d*) show SEM cross-sections of various sized features from a 5 μm thick Unity 4411 film overcoated with PI-2771 and decomposed. Only the smallest width features produced air-channels, as shown in figure 9(*d*). Some remaining sacrificial material is seen in the corners of the channels in figures 9(*a*)–(*c*) from incomplete decomposition. Identical results to PI-2771 are seen using either HD4000 or PI-2555 as the overcoat material. The other overcoat materials listed in the first 13 rows of table 1 do not work for any dimensions due to poor thermal stability.

The reason polymer overcoats fail with Unity 4411 has been attributed to a loss in mechanical properties or flow of the polymer when the overcoat polymer passes through the T_g. As the Unity 4411 decomposes, it passes through a liquid-like stage. If the overcoat material is above its T_g, surface tension can pull it into the channel as the sacrificial material decomposes. One approach to mitigating this problem is to add a thin layer of SiO_2 as a rigid support layer either between the sacrificial and overcoat polymers or on top of the overcoat polymer layer.

Sample sets were fabricated with SiO_2 between the sacrificial material and the overcoat, and SiO_2 above the overcoat material: (i) overcoat polymer only; (ii) 5000 Å of SiO_2 + overcoat polymer; (iii) overcoat polymer + 5000 Å SiO_2; and (iv) overcoat polymer + 2 μm of SiO_2. The overcoat polymer in sample sets (iii) and (iv), with SiO_2 on top of the polymer, was cured before deposition of the glass. A summary of the results for the various sized air-gap structures and overcoat combinations with Unity 4411 is given in table 2. A 5000 Å layer of SiO_2 deposited between the sacrificial material and the overcoat polymer corrects the problem with the overcoat collapsing or filling the channel. Figures 10(*a*)–(*d*) show cross-sectional SEM images of various sized structures overcoated with SiO_2 and PI-2771. Some of the resulting air-cavities are dome-shaped. The amount of the overcoat deformation and the height of the resulting channel increases with increasing channel width. The results for PI-2555 are similar to those for PI-2771, except the structures with SiO_2 and PI-2555 deformed less. Many of the air-channels were rectangular. However, even in the cases where the resulting air-channel had a dome-shaped cross section, the glass layer is visibly intact. Figure 11 shows SEM images of air-channels fabricated with Unity 4411 overcoated with PI-2771, then a SiO_2 layer on the top surface. Glass thicknesses of 5000 Å and 2 μm are demonstrated. When the glass is above the overcoat polymer, the additional support

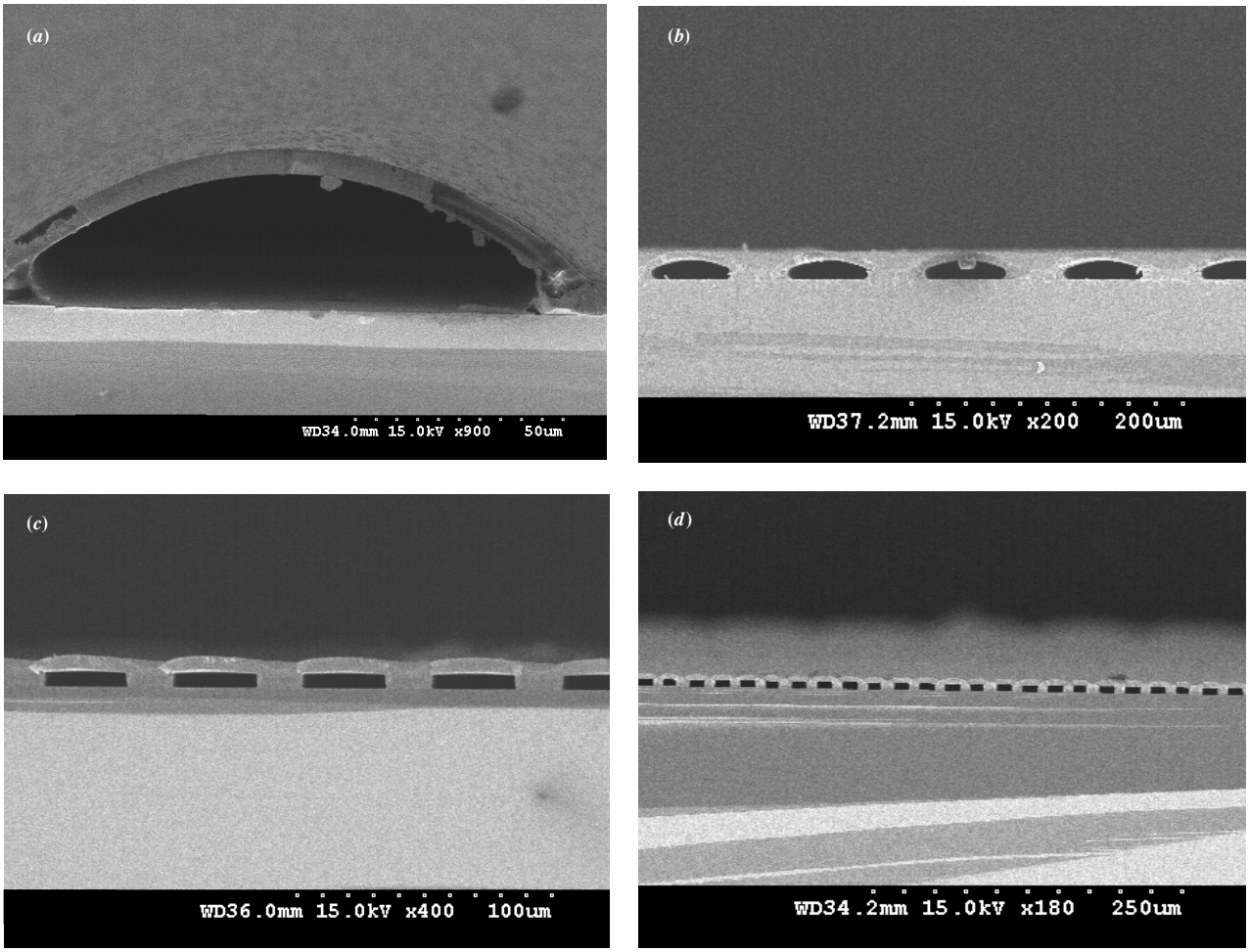

Figure 10. SEM images of air-gaps overcoated with 5000 Å SiO_2 and 9 μm PI-2771. The widths of the air-gaps shown are: (*a*) 140 μm, (*b*) 70 μm, (*c*) 35 μm and (*d*) 15 μm.

Table 2. Overcoat results with Unity 4411.

Overcoat layer(s)	Unity 4411 (μm)	Glass (Å)	Overcoat polymer (μm)	15 μm	35 μm	70 μm	140 μm
PI-2771 only	5.46	–	9	G	B	B	B
SiO_2+ PI-2771	5.46	5 360	9	G	G	G	G
PI-2771 + thin SiO_2	5.49	5 360	9	G	G	G	G
PI-2771 + thick SiO_2	5.49	17 000	9	G	G	G	G
PI-2555 only	5.46	–	5	G	B	B	B
SiO_2+ PI-2555	5.46	5 360	5	G	G	G	G
PI-2555 + thin SiO_2	5.46	5 360	5	G	G	G	G
PI-2555 + thick SiO_2	5.46	17 000	5	G	G	G	G
HD4000 only	5.23	–	15	G	B	B	B
HD 4000 + thin SiO_2	5.23	5 360	15	G	G	G	G
HD 4000 + thick SiO_2	5.23	17 000	15	G	G	G	G
CRC-8650 only	5.46	–	10	B	B	B	B
SiO_2 + CRC-8650	5.49	5 360	10	G	G	G	G
PBO CRC-8650 + thick SiO_2	5.03	17 000	10	G	G	G	G

G = compatible and air-channels can be formed; B = incompatible.

helps the softened overcoat polymer withstand the pull-down force from surface tension between the overcoat polymer and the decomposing Unity. However, a minimal amount of sag by the overcoat into the channel continues to be present for wider channels, as in figures 11(*a*)–(*c*). When the glass is deposited over the polymer, the surface tension will continue to act on the softened polymer since it is in contact with the Unity 4411 when decomposition begins. A thicker glass layer improves the final shape of the structures. In addition, the SiO_2 on each of the polymers is cracked following the decomposition. This is attributed to stress developed from the CTE mismatch between the polymers ($\sim$30–50 ppm $^\circ C^{-1}$) and the SiO_2 ($\sim$0.5 ppm $^\circ C^{-1}$) when cooling to room temperature. However, the glass can subsequently be removed by a plasma or wet-chemical etch. When the glass is removed, the overcoat material is not affected and the air-gap retains its original

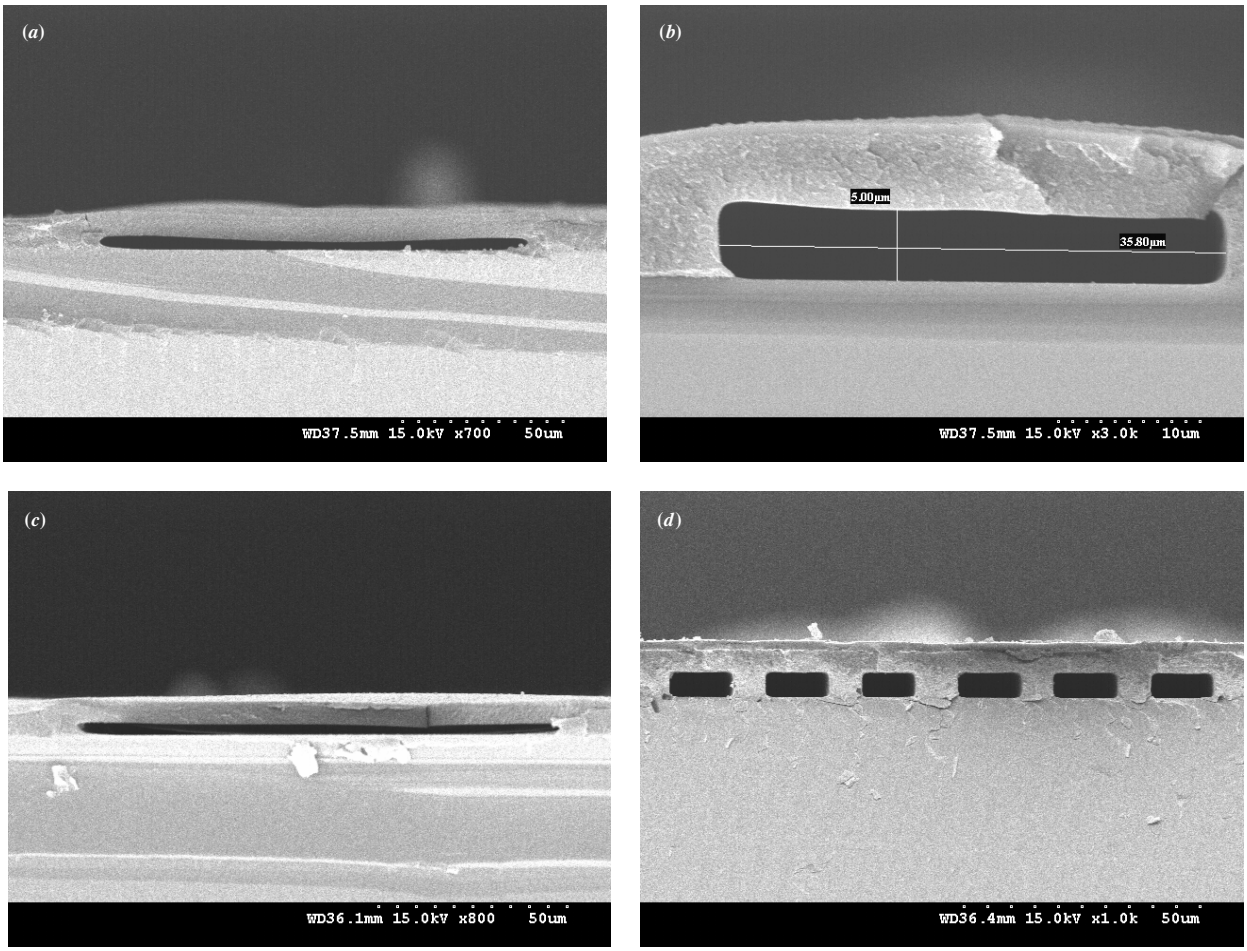

Figure 11. SEM cross-sectional images of air-gaps overcoated with PI-2771 and SiO_2. Dimensions are: (*a*) width: 140 μm, overcoat: 9 μm PI-2771 and 5000 Å SiO_2; (*b*) width: 35 μm, overcoat: 9 μm; PI-2771 and 5000 Å SiO_2; (*c*) width: 140 μm, overcoat: 9 μm PI-2771 and 2 μm SiO_2; and (*d*) width: 15 μm, overcoat: 9 μm PI-2771 and 2 μm SiO_2.

shape. In addition to SiO_2, other glasses such as silicon nitride have been used in air-channel studies and their applicability here is likely. This investigation is underway.

4. Conclusion

A new process for the fabrication of micro air-channels incorporating SiO_2 as a mechanical support layer and solvent barrier was demonstrated. The SiO_2 structural support mitigates solvent incompatibility and overcoat softening issues and enables the use of a wide variety of sacrificial materials and polymer overcoat combinations. The fabrication of two-layer micro air-channels was demonstrated, by the addition of a SiO_2 deposition step wherever needed to prevent material dissolution. In addition, the SiO_2 offers mechanical support to the polymer overcoat whether it is above or below the polymer layer.

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