INSTITUTE OF PHYSICS PUBLISHING JOURNAL OF MICROMECHANICS AND MICROENGINEERING
J. Micromech. Microeng. 14 (2004) 625–631 PII: S0960-1317(04)71690-3

Wafer bonding using microwave heating of parylene intermediate layers

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Received 10 November 2003
Published 6 February 2004
Online at stacks.iop.org/JMM/14/625 (DOI: 10.1088/0960-1317/14/4/025)

Abstract
This paper describes a novel wafer bonding technique using microwave heating of parylene intermediate layers. The bonding is achieved by parylene deposition and thermal lamination using microwave heating. Variable frequency microwave heating provides uniform, selective and rapid heating for parylene intermediate layers. The advantages of this bonding technique include short bonding time, low bonding temperature, relatively high bonding strength, less void generation and low thermal stress. In addition, the intermediate layer material, parylene, is chemically stable and biocompatible. This bonding technique can be used for structured wafers also because parylene provides a conformal coating. Therefore, this is a very attractive bonding tool for many MEMS devices. The bonding strength and uniformity were evaluated using diverse tools. Fracture mechanisms and the effects of bonding parameters and an adhesion promoter were also investigated. The bonding with a structured wafer was also successfully demonstrated.

1. Introduction

Wafer bonding is an important process in the fabrication and packaging of many microelectromechanical system (MEMS) devices as well as integrated circuits (IC). Wafer bonding techniques can be classified into two categories, direct wafer bonding and intermediate layer bonding. The common direct wafer bonding techniques are silicon–silicon fusion bonding [1] and silicon–glass anodic bonding [2]. While these bonding techniques provide high bonding strength and hermetic sealing, they require high temperature processing and even high voltage for anodic bonding. The indirect, intermediate layer bonding includes adhesive bonding and eutectic bonding. The eutectic bonding does not require high temperature processing but it requires specific material composition such as metal alloy to achieve a low eutectic point [3]. Consequently, the direct wafer bonding technique and the eutectic bonding technique cannot be used in many MEMS devices that contain diverse types of substrates, polymers and electrically sensitive materials. In contrast, adhesive bonding has more widespread applicability. It is a low temperature process, no electrical power is required and there is no limitation for substrate materials.

Many spin-coated amorphous polymers such as polyimides, benzocyclobutene (BCB), Nafion and photoresists have been investigated as intermediate layers for wafer bonding [4–9]. Although the intermediate layer bonding can be obtained at a relatively low temperature and there is no limitation for the substrate material, they also have some disadvantages. First, the solvent release and the polymer curing process often generate voids resulting in non-uniform bonding. Second, the intermediate layer is not usually chemically inert and biocompatible. As a result, these materials may not be a good choice for micro total analytic systems (μTAS) and BioMEMS devices. Third, the spin-coating method is inappropriate for structured wafers such as microfluidic devices because the spin-coated polymer fills up the concave structures and causes contamination problems.

Parylene, polyparaxylylene, can be a good alternative to the conventional adhesive materials. Parylene is deposited

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Table 1. The material properties of parylene, BCB and polyimide.

Material properties	Parylene C	BCB (Dow chemical)	Polyimide (PI2555, DuPont)
Polymer type	Crystalline thermoplastic CVD	Amorphous thermoset spin-coated	Amorphous thermoset spin-coated
Thermal properties			
Glass transition temperature (°C)	<90	>200 (80% cure)	325
Melting temperature (°C)	290	–	–
Thermal expansion coefficient at 25 °C, $\times 10^5$ (C^{-1})	3.5	4.3	5.0
Barrier properties			
Water absorption (%)	<0.1	~0.1	N/A
Solvent resistance	Inert	N/A	N/A
Electrical properties			
Dielectric constant	2.95	2.65	3.4
Dissipation factor at 1 MHz	0.013	0.0008	0.0018 at 1 kHz
Other properties			
Young's modulus (Gpa)	3.2	2.9	2.5
Optical transparency	Transparent	Transparent	N/A
Biocompatibility	Biocompatible	Biocompatible	N/A

by the chemical vapor deposition (CVD) process providing conformal coating regardless of surface morphology. Parylene is also chemically stable, biocompatible and optically transparent. The material properties of parylene are listed in table 1 and compared with BCB and polyimide. In spite of its superior properties, parylene has not been considered as an intermediate layer for wafer bonding because it is a crystalline and thermoplastic polymer. However, recently a parylene/parylene thermal bonding technique has been developed to fabricate microchannel structures [10, 11]. The bonding was achieved by the lamination of two thin parylene layers under high temperature and pressure. Recently, this parylene/parylene bonding technique was applied to wafer-to-wafer bonding [12, 13]. Particularly, Kim and Najafi investigated the effect of bonding temperature on the bonding strength using convection heating and pull tests, and they achieved a bonding strength of about 3.6 MPa with the bonding temperature above 230 °C. However, the bonding mechanism and the other bonding parameters such as pressure, time, parylene thickness and adhesion promoter were not investigated in their study.

In this paper, variable frequency microwave (VFM) is introduced as a new heating technique of parylene intermediate layers for wafer bonding application. VFM can provide uniform, fast and selective heating because it is based on energy dissipation of dielectric materials at a high frequency. In this bonding technique, the substrates are not heated significantly and thus stress due to differences in thermal expansion could be drastically decreased. Therefore, this technique will be specifically useful for bonding two substrates consisting of dissimilar materials and having different coefficients of thermal expansion. In addition, this new heating technique along with a new intermediate layer material, parylene, provides a rapid, low-temperature, uniform, chemically stable and biocompatible wafer bonding. This technique can also be used for bonding structured wafers.

The bonding and fracture mechanisms, and the effect of major bonding parameters (temperature, time, pressure and thickness) on the bonding strength have been thoroughly investigated. The effect of an adhesion promoter treatment has also been investigated. The bonding strength and uniformity were evaluated by diverse methods such as pull tests, die shear tests, c-mode scanning acoustic microscopy (c-SAM) and scanning electron microscopy (SEM).

2. Variable frequency microwave heating of parylene

Microwave heating of materials occurs due to dielectric loss mechanisms. The principal mechanism of coupling microwave radiation to polymer dielectrics is through dipole orientation by the electric field. The amount of microwave energy absorbed by a material is a function of the applied electric field and dielectric properties of the material as shown in equation (1) [14].

$$P_{\mathrm{absorbed}} = 2\pi f E_{\mathrm{rms}}^2 \varepsilon_0 \varepsilon'', \tag{1}$$

where P_{absorbed} is the dissipated or absorbed power per unit volume, E_{rms} is the root mean square electric field strength, ε_0 is the permittivity of free space, ε'' is the relative loss factor and f is the frequency. It is also known that microwaves can accelerate chemical reaction rates. For example, the curing time of adhesives can be reduced as much as 10–20 times compared with convection heating [15].

The variable frequency microwave is a modified microwave technology that allows uniform and selective heating. By rapidly sweeping through a bandwidth of frequencies, VFM not only provides a uniform energy distribution inside the chamber but also eliminates the problem of arcing that is usually observed in fixed frequency microwaves. This permits processing of samples with metal, electrical circuits and high value semiconductor assemblies without any damage.

'Parylene C' (poly-monochloroparaxylylene) has a dielectric constant of 2.95 and a dielectric loss factor of 0.013 at the frequency of 1 MHz. It must be noted that the dielectric loss factor of parylene is much higher than other spin-coated polymers while dielectric constants are similar. This suggests that parylene can be effectively heated by VFM. This thought was verified by preliminary heating tests.

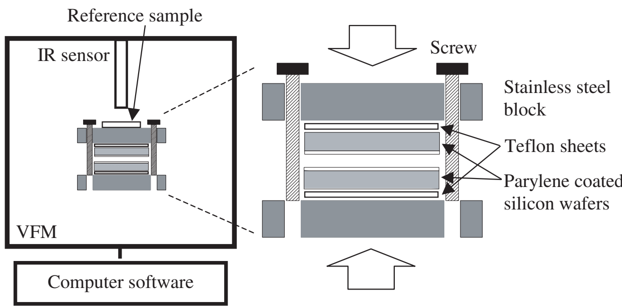

Figure 1. Wafer bonding apparatus.

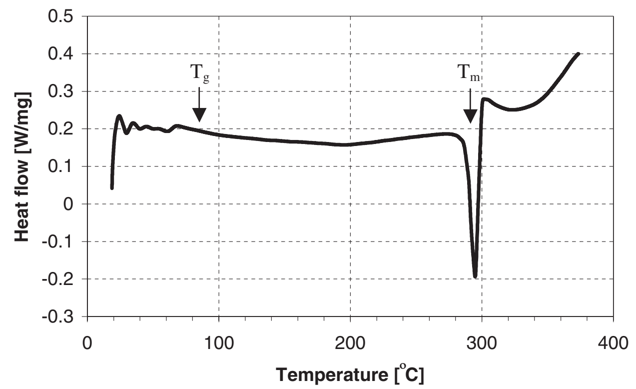

Figure 2. DSC data of parylene C.

10 μm thick parylene was deposited on silicon chips ($\sim$2 $\times$ 2 cm^2) and heated by VFM. The temperature of parylene was measured by an infrared camera installed in the VFM chamber. Parylene was heated linearly up to about 170 °C, but above that temperature, parylene was oxidatively degraded, causing a non-linear heating curve. However, it must be noted that this heating characteristic is only for the parylene that is exposed to air during the heating. In the wafer bonding application, parylene intermediate layers are not exposed to air. As a result, the degradation is not observed in the intermediate parylene layers.

3. Experimental details

3.1. Wafer bonding

Silicon wafers with a diameter of 7.5 cm were used for the bonding experiments. All wafers were cleaned with organic solvents and dried completely followed by parylene C deposition using a parylene deposition system, Labcoater II (Specialty Coating System, Indianapolis, IN). Figure 1 shows the bonding apparatus used in this study. Two parylene-coated wafers were put together between stainless steel blocks and compressed with screws. Pressure was controlled by a torque wrench, and Teflon sheets (0.7 mm thick) were inserted between the wafers and steel blocks to provide uniform compression. The compressed sample is then placed in a VFM chamber. A commercially available VFM, MicroCure 2100 (Lambda Technologies, Morrisville, NC) was used in this study. Air was constantly purged into the VFM chamber during the heating. The bonding temperature and time were programmed using the VFM software. In order to monitor the temperature of parylene, a reference parylene film coated on a silicon chip was always placed on top of the bonding sample so that the infrared camera might read the temperature.

The four major bonding parameters investigated in this study were temperature, pressure, time and thickness. In order to choose the proper bonding temperatures, the glass transition temperature (T_g) and melting temperature (T_m) of parylene C were measured using differential scanning calorimetry (DSC Q1000, TA Instrument, New Castle, DE) as shown in figure 2. T_m was very clearly at 290 °C, but the DSC data did not show a distinct T_g. However, the T_g of parylene C is known to be around 90 °C. Hence, three different bonding temperatures (120, 160 and 200 °C) were chosen between T_g and T_m of parylene. Pressure was controlled by a torque wrench and the three different torque values used in this study were 1.2, 2.4 and 4.0 N m. These torque values were converted to pressure values using the torque versus force relation as shown in equation (2).

$$F = \frac{T}{r\mu + \frac{d}{2}\tan(\alpha + \rho)}, \qquad (2)$$

where F is the force, T is the torque, r is the average radius of the screw head, μ is the friction constant (generally 0.15–0.25), d is the diameter of the screw, α is the pitch angle of the screw and ρ is $\tan^{-1}\mu$. According to this equation, the initial pressure values for the torques used in this study were 0.45, 0.9 and 1.5 MPa. However, it must be noted that the applied pressure was not constant due to the viscoelastic behavior of Teflon sheets. Bonding time was varied from 10 to 120 min and parylene thickness was varied from 1 to 10 μm.

Another important parameter that needs to be considered is the pre-treatment of the silicon surface because fracture may occur at the parylene/silicon interface instead of parylene/parylene interface during bond strength test. An adhesion promoter recommended by the parylene manufacturer has been used to see its effect. The adhesion promoter was a mixture (50:50:1) of isopropyl alcohol, DI water and A-174 (gamma-methacryloxytropyl trimethoxy silane, Specialty Coating Systems). The treatment procedure was as follows. Silicon wafers were immersed in isopropyl alcohol for 5 min. Then, wafers were immersed in the mixed adhesion promoter solution for 30 min. Then, the wafers were air dried. The wafers were then immersed in isopropyl alcohol again for 5 min. Finally, the wafers were dried with nitrogen gas and baked on a hot plate at 100 °C.

3.2. Evaluation of the bonding

The bonding strength was first evaluated qualitatively by a razor blade insertion test. For the samples that could not be separated by razor blade insertion, two quantitative tests, a die shear test and a tensile pull test, were performed. For the die shear test, the bonded wafers were diced into 4 $\times$ 4 mm^2 pieces and mounted on a glass slide using super glue. The die shear tester used in this experiment was STM 100K (Micro Systems Inc., Anyang, Korea). At least ten samples were tested for each bonding condition, and the bonding strength values were averaged. For the pull test, the bonded wafers were diced into 9 $\times$ 9 mm^2 pieces and each piece was mounted to a cylindrical plastic holder fabricated by

Table 2. Bonding strength at different bonding conditions.

Bonding parameters				Bonding strength		
Temperature (°C)	Pressure (MPa)	Time (min)	Thickness (μm)	Adhesion promoter	Die shear test (MPa)	Pull test (MPa)
120	0.9	60	2	No	N/A	N/A
120	1.5	120	2	No	N/A	N/A
160	0.9	30	2	No	N/A	N/A
160	0.9	120	2	No	7.94	–
160	0.9	90	1	No	7.07	–
160	0.9	90	5	No	5.78	–
160	0.45	60	2	No	7.09	–
160	1.5	10	2	No	7.75	3.45
160	1.5	30	2	No	5.97	5.38
160	1.5	30	1	No	6.76	–
160	1.5	30	5	No	6.22	–
160	1.5	30	2	Yes	12.68	9.16
200	0.9	90	1	No	6.64	–
200	1.5	30	2	No	5.33	–
200	1.5	10	2	No	4.92	–

N/A: wafers were separated with razor blade (parylene layers were not bonded).
Reference value: the fracture strength of bulk silicon is 6.6 ± 0.9 GPa [16].

rapid prototyping for convenient grip. A strong epoxy resin (Epoxy907, Miller-Stephenson, Sylmar, CA) was used for the mounting. Pull tests were performed using MicroTester 5548 (Instron Corporation, Canton, MA). At least five samples were tested for each bonding condition, and the bonding strength values were averaged.

Bonding uniformity across the wafer was investigated using a c-mode scanning acoustic microscope, the so-called c-SAM (series D-9000, Sonoscan. Inc., Elk Grove Village, IL). This enabled the non-destructive visualization of the interface between bonded wafers. The microscopic bonded interface was also investigated using a scanning electron microscope (Hitachi S-800, Hitachi High Technologies, Pleasanton, CA). A bonded sample was broken in liquid nitrogen and coated with a thin gold coating for SEM imaging.

3.3. Bonding with structured wafers

Rectangular microchannels (both width and depth were 100 μm) were fabricated on silicon wafers using deep reactive ion etching (RIE). The standard BOSCH process was used for the fabrication. The photoresist mask used for deep RIE was removed completely by both wet treatment and oxygen plasma etching. Then 2 μm thick parylene was deposited on the structured silicon wafer. After parylene deposition, this structured silicon wafer was bonded to a parylene-coated pyrex glass substrate. Bonding quality was inspected visually through the transparent glass plate.

4. Results and discussion

4.1. Bonding strength

Table 2 is the summary of the bonding strength test results. All values were averaged. For every sample, a razor blade insertion test was performed first just after the bonding experiment. The samples bonded at 120 °C were separated by razor blade insertion regardless of other bonding parameters. The separated wafers still had parylene layers on the surface indicating that parylene layers had not been bonded with those bonding parameters. When the bonding temperature was 160 °C, parylene/parylene bonding was achieved depending on pressure and time, and the bonded wafers were not separated by razor blade. However, the parylene thickness did not make any difference in the bonding strength. The shortest process condition for parylene/parylene bonding in our experiments was 10 min at 160 °C and 1.5 MPa.

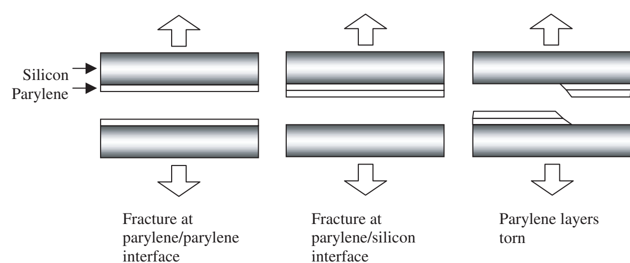

Figure 3. Different fracture mechanisms in bonding strength test.

The bonding strength measurement through the die shear test revealed that the bonding strength did not change much with the changes of bonding parameters. The bonding strength ranged from 5 to 7 MPa. The tensile pull test result also showed similar characteristics. The bonding strength ranged from 3.5 to 5.5 MPa regardless of the bonding parameters. However, the adhesion promoter-treated sample showed almost two times the strength both in shear (12.68 MPa) and tensile strength (9.16 MPa). All the tested samples were inspected visually to see where the fracture had occurred. This inspection provided a good interpretation of the above bonding strength result. The fracture mechanism in this experiment can be classified into three cases as shown in figure 3. First, when the parylene intermediate layers were not bonded, the fracture occurred at the parylene/parylene interface. In this case, the samples were usually separated during the razor blade test. Second, when the parylene intermediate layers were bonded well, the fracture occurred at the parylene/silicon interface during the bonding strength test. In this case, the bonded parylene intermediate layers were observed to remain only on one wafer after the bonding test. This

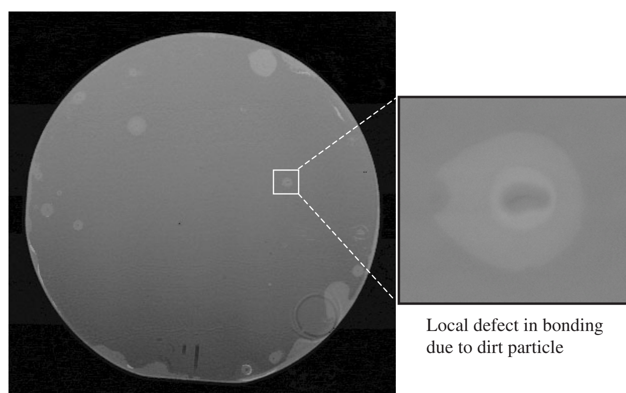

Figure 4. C-SAM image of a bonded wafer (7.5 cm in diameter).

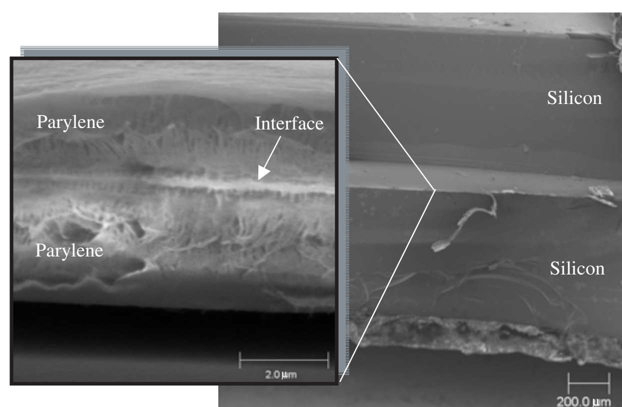

Figure 5. SEM image of the bonded interface.

was because the parylene/parylene bond was stronger than the parylene/silicon bond. The adhesion strength of the parylene/silicon bond was not influenced much by the bonding parameters. Consequently, the bonding strength did not change much as the bonding parameters varied in this case. The third case is that the bonded parylene layers were torn during the bonding strength test. The adhesion promoter-treated sample belonged to this case. Both parylene/parylene bonding and parylene/silicon adhesion were so strong that the fracture generated damage in the parylene layers. But still the fracture mainly occurred at the parylene/silicon interface.

Another characteristic was that the bonding strength value often showed a large deviation (∼50%) from the average value. This is considered to be mainly due to the existence of particles. This issue will be discussed more in the bonding uniformity section. However, the adhesion promoter-treated sample was quite different. It showed uniform bonding strength in both shear and tensile tests. The deviation from the average value was less than 20%.

4.2. Bonding uniformity and interface

Figure 4 shows the c-SAM image of a bonded sample. The bonding condition for this sample was 160 °C, 1.5 MPa for 30 min and the intermediate parylene thickness was 2 μm. The bright parts indicate non-bonded areas in this image. The bonding uniformity was good in the center of the wafer but the edge was not as good. Although the overall bonding uniformity was good, a few local defects were observed. Most local defects were found to have tiny dirt particles in them as shown in the magnified image of figure 4. Dirt particles can sit on parylene layers either during the venting process of the parylene coating procedure or during the preparation of the bonding. It was difficult to remove dirt particles completely because the parylene deposition and bonding were done in a normal lab environment—not in a cleanroom. This existence of particles can also explain why some of the samples showed very weak bonding strength during the die shear test. Better bonding quality is expected if the bonding experiment is done in a cleanroom environment.

Figure 5 shows the SEM image of the bonded interface. The bonding condition for this sample was 160 °C, 1.5 MPa for 30 min and the intermediate parylene thickness was 2 μm. The magnified view in figure 5 clearly shows the two parylene layers (approximately 2 μm thick) and the interface. A uniform interface layer that is less than 200 nm was observed between two parylene layers. This thin interface layer is considered to be the inter-diffusion layer of the two parylene layers indicating a good bond. Many different spots were inspected in the SEM, and uniform bonding was observed between the two parylene layers.

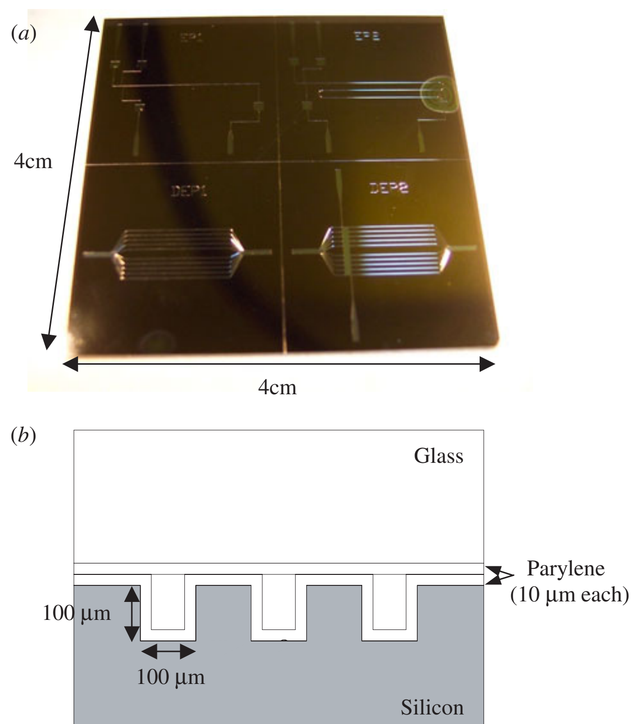

Figure 6. The bonding of a structured wafer (*a*) a bird view, (*b*) a schematic diagram of the cross-section.

4.3. Bonding with structured wafers

A structured wafer that has microchannels was successfully bonded to a glass substrate using parylene intermediate layers. Again the bonding condition for this sample was 160 °C, 1.5 MPa for 30 min and the intermediate parylene thickness was 2 μm. The bonding quality was quite good as shown in figure 6. But still a few local defects due to the existence of particles were observed visually. Figure 6(*b*) shows a diagram

Table 3. Comparison of different adhesive bonding techniques.

	BCB	Polyimide	SU-8	Parylene
Bonding temperature (°C)	250	275	90	160
Bonding time	~2 h	~2 h	5 min	<30 min
Tensile bonding strength (MPa)	N/A	8.2	20.6	9.2
Void sources	Release of solvents	Byproducts of imidization and release of solvents	N/A	Dirt particles
References	[5]	[4, 5]	[9]	

Reference value: the bonding strength of anodic bonding is approximately 30–40 MPa [17].

for the cross-section of the bonded microchannel. It must be noted that the inner surface of the channel has conformal parylene coating.

5. Conclusions

A new low temperature wafer bonding technique of using microwave heating of the parylene intermediate layer was developed. The bonding is achieved by parylene deposition and lamination using microwave heating. Variable frequency microwave heating provides uniform, selective and rapid heating for parylene intermediate layers. Table 3 compares this technique with other adhesive bonding techniques. The tensile bonding strength of the new technique is slightly higher than the bonding strength of the Polyimide technique reported in the literature. Also, this bonding technique requires a lower bonding temperature and less bonding time than BCB and Polyimide techniques. The possible sources of void generation in the new technique are dirt particles and trapped air because there is no solvent release or byproduct in this process. Non-destructive scanning acoustic microscopy inspection revealed that the main void source was dirt particles.

There are many other advantages in this bonding technique over other adhesive bonding techniques. This technique will be specifically useful for bonding two substrates consisting of dissimilar materials and having different coefficients of thermal expansion because the substrates are not heated significantly during the bonding process. In addition, the intermediate layer, parylene, has biocompatibility and good barrier properties such as chemical inertness and a low permeation coefficient. Also, the CVD deposition of parylene enables this technique to be used for structured wafers. Therefore, it is believed that this new bonding technique can be a very attractive bonding tool, especially for MEMS devices.

The bonding strength test and the inspection of the fractured sample showed that fractures occurred at different interfaces depending on the bonding parameters. When the temperature was too low, the time was too short or the pressure was too low, parylene/parylene was not bonded well and as a result, fractures occurred between two parylene layers even by razor blade insertion. But when the bonding parameters are high enough to provide good parylene/parylene bonding, the fracture always occurred at the parylene/silicon interface without breaking the parylene/parylene bond. The shortest process condition for parylene/parylene bonding in our experiments was 10 min at 160 °C and 1.5 MPa. The cross-sectional SEM image of the well-bonded sample revealed the existence of a uniform inter-diffusion layer (<200 nm) between two parylene layers. In this case, the changes in the parameters did not make much change in parylene/silicon bond strength. The bonding strength ranged from 5 to 7 MPa in the die shear test and 3.5–5.5 MPa in the tensile pull test regardless of the changes in bonding parameters. However, when the wafers were treated with an adhesion promoter before parylene deposition, the bonding strength was increased by 100% (shear strength ~12.68 MPa, tensile strength ~9.16 MPa). The overall bonding uniformity was good but a few local voids due to dirt particles were observed. The bonding with a structured wafer was successfully demonstrated using a deep RIE etched silicon wafer and a transparent glass substrate.

Acknowledgments

The authors would like to thank Zhuqing Zhang for DSC and s-SAM measurement. Also the help of Inho Yoon and Soongi Lee with SEM imaging is greatly appreciated.

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