A Modified Quasi-Creep Model for Assessment of Deformation of Topas COC Substrates in the Thermal Bonding of Microfluidic Devices: Experiments and Modeling

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ABSTRACT: The effects of thermomechanical properties of dissimilar polymer plates on thermal bonding were investigated and the resultant deformation of cover Topas COC plate was modeled using a simplified quasi-creep model. The appropriate conditions for thermal bonding for minimal deformation of both the Topas cover and substrate plates could be established through simulation using the quasi-creep model. Both the cover plate and the substrate containing microchannels were fabricated by injection molding. The elastic modulus of the COC plate at different temperatures was measured using three-point bending test. The thermal bonding was conducted at different temperatures, pressures, and holding times. The deformation of the cover plate (consisting of Topas with a lower glass transition temperature, T_g) into the microchannel of the substrate plate (consisting of Topas with a higher T_g) was found to be significant even at lower bonding pressures when the bonding temperature was higher than a critical temperature. Such deformation was dependent on the viscoelastic creep behavior of the material and the thermal bonding temperature and load. This deformation behavior was predicted by the numerical model, and the predicted results agree well with the experimental data. The bonding strength of the sealed microchannels was evaluated using the burst test. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 867–873, 2011

Key words: creep; thermal bonding; modeling; viscoelastic properties; COC

INTRODUCTION

Cyclic olefin copolymer (COC) has been used widely as the substrate material for the fabrication of microfluidic devices due to its high mechanical stability, good chemical, and excellent optical properties.[1] Many manufacturing methods of the microchips have been explored and successfully used, such as hot embossing or imprinting,[2,3] micro-injection molding,[1,2,4] soft lithography,[5,6] laser photo-ablation,[7,8] X-ray lithography,[9] and plasma etching.[2,10] Micro-injection molding involves the use of a precisely made master from which many identical polymer microstructures can be made. The injection molding technique is highly versatile (microchannel sizes ranging from 10 μm to few hundred micrometer can be molded) and has been widely used to fabricate microchannels in a wide variety of thermoplastic materials.[11]

One of the key challenges in the manufacturing of microfluidic devices is to seal a substrate containing microchannels with a cover plate of the same material. There are many methods for bonding the polymeric plates, such as thermal bonding, thermal lamination, adhesive tape, glue layer, and laser bonding. Direct thermal bonding is desirable in many applications as it allows the formation of enclosed microchannels with a uniform surface composed entirely of the same or similar polymeric material. However, high bonding pressure may induce global and localized geometric deformation of the cover plate and the substrate,[12] which may impair the performance of the microfluidic device.

If the substrate and the cover plates are made from the same material, then the bonding temperature should be selected carefully to prevent the deformation of the microchannel. The advantage of using two different grades of the same polymer material, e.g., a COC with higher glass transition temperature (T_g) for the substrate plate and another COC with lower T_g for the cover plate, is that the dimensions of the microchannel in the substrate could have little change during thermal bonding.

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This can be achieved by selecting a bonding temperature to be close to the softening temperature of the cover plate which has a lower T_g to obtain good bond strength. The deformation of the microchannel plate in the substrate with a high T_g can then be minimized. However, some polymer material may protrude from the cover plate into the microchannel in the substrate plate at high bonding pressure, or may even fully block the microchannel if inappropriate bonding conditions are utilized. Therefore, a study of the deformation of the cover plate in the vicinity of the microchannel of the substrate plate with temperature and pressure is important as this determines the final cross section of the microchannel. Modeling of such deformation coupled with burst pressure assessment of the sealed microchannel will facilitate determination of the appropriate conditions for thermal bonding of such microfluidic devices. This is the focus of the present study.

The amorphous polymer COC exhibits temperature-dependent viscoelastic behavior, which determines the elastic and plastic deformation of the polymer with stress. The elastic component is recoverable after unloading while the plastic part leads to permanent deformation. The range of the linear stress–strain relationship of most thermoplastics is rather limited, whereas the permanent strain is quite large when the stress exceeds the linearity limit,[13] which is the case during thermal bonding. The viscoelastic creep behavior of the COC at different temperature and pressure has to be taken into account in the assessment of deformation of the cover plate in the vicinity of the microchannel during thermal bonding.

Although there are some previous studies on polymer substrate deformation during thermal bonding,[12,14] they have not characterized the relationship between deformation of the polymer substrates with the thermal bonding conditions. This article describes the fabrication of microfluidic device by thermal bonding using two grades of cyclic olefin copolymer (COC). It is demonstrated that the optimal thermal bonding conditions can be established through the assessment of the simulated deformation of the cover plate in the vicinity of the microchannel of the substrate plate. The numerical simulation incorporated a quasi-creep model that describes the viscoelastic response of the COC material.

EXPERIMENTAL

Material

Topas with grades 5013L-10 and 6015S-04 were purchased from Ticona Company (USA). It is a COC copolymerized from norbornene and ethylene using a metallocene catalyst. The properties of the two grades of Topas were listed in Table I.

TABLE I
Properties of Topas 6015S-04 and 5013L-10

Grades	Glass transition temperature (°C)	Molecular weight M_n (g/mol)	Molecular weight M_w (g/mol)	M_w/M_n
6015S-04	158	31,200	76,400	2.45
5013L-10	134	41,300	99,200	2.40

Injection molding

Both the cover and substrate plates were fabricated from COC pellets using an injection molding machine (Battenfeld injection molding technology Pte Ltd). Both the plates were 75 mm long and 40 mm wide. The base plate contained two 50 μm deep straight microchannels that were molded in the 2 mm thick Topas 6015S-04 substrate. The 1 mm thick cover plate was molded using Topas 5013L-10. These two grades of Topas were selected because of their different glass transition temperatures.

Three-point bending test

The elastic moduli of the cover plate at different temperatures were measured by three-point bending tests using the universal testing machine (Instron, series 5569). A video extensometer with high precision strain measurement was used in testing. Tests at elevated temperatures were carried out in a chamber, in which accurate control of the testing temperature was maintained. The chamber was heated up to the designated temperature and then held at that temperature for at least 20 min before testing began to ensure a uniform temperature distribution within the specimen. The elastic moduli were derived from the linear portion of the stress–strain curves from the flexure test. The bending span was 40 mm, the load head was 3 mm in radius and the cross head speed was 1 mm/min. The dimension of the specimen was 75 mm × 40 mm ×1 mm.

Thermal bonding and confocal microscopy measurement

The thermal bonding was conducted on a hydraulic press (Carver, Inc.) fitted with heated plates and an analog load gauge, where temperature between the plates can be kept uniform with an accuracy of 0.1°C. All the features including ports and channels are integrated in the microchannel substrate plate, while the cover plate consisted of a flat featureless plate. Thus, exact alignment of the substrate and cover plates was not necessary for thermal bonding.

The bonding temperature was selected to be near the glass transition temperature of the cover plate (134°C). This is much less than the T_g of the substrate plate and thus little or no deformation to the molded

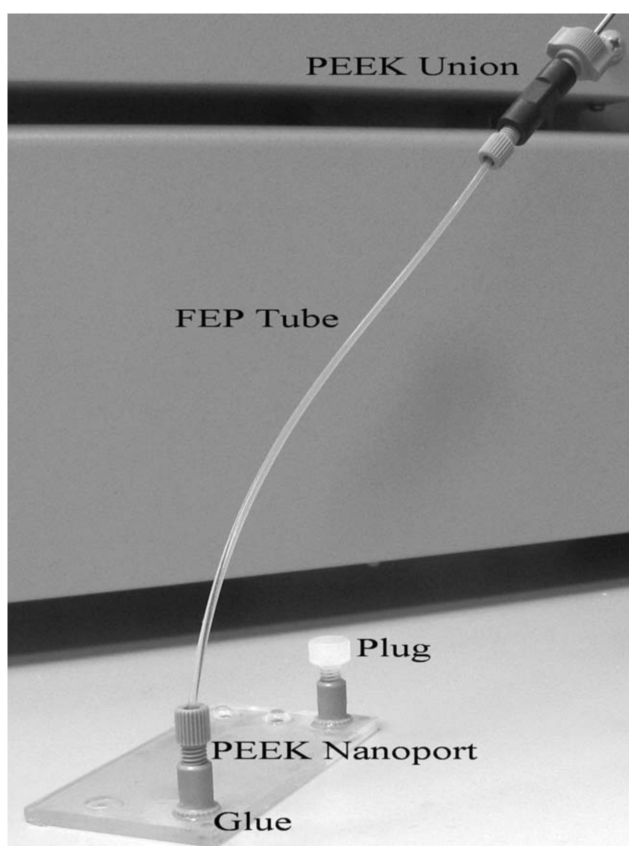

Figure 1 Burst test setup with a syringe pump connected with bonded polymer plates by FEP tube with a PEEK ZDV union.

microchannel was expected in the process. The bonding pressure was applied within 10 min after the platens were heated up to the desired temperature. After a specific bonding time, the samples were taken out to cool under room temperature conditions. After thermal bonding, the plates were debonded using the burst pressure test and examined using a confocal microscope (Nikon eclipse L150). The deformation of both the cover and substrate plates in the vicinity of the microchannels was examined.

Burst pressure test

The burst pressure of the thermally sealed microchannels was measured using the experimental setup shown in Figure 1. The outlets of the sealed microchannel in the microfluidic device were connected using plastic tubing to a syringe pump (ISCO Teledyne Technologies Company, Model 100DM), which is fitted with pressure transducer with an error accuracy of 0.5% in the range of 0.069–68.9 MPa. Before the test, a PEEK NanoPort assembly (Science Team Services, Singapore) was glued onto the thermally bonded plates using Araldite epoxy adhesive. After the epoxy adhesive was applied, the substrates and the port were clamped for 24 h at room temperature. After that, it was then connected to the syringe pump via a natural FEP (fluoroethylene propylene) tube with a PEEK (polyether ether ketone) zero-dead-volume (ZDV) union, see Figure 1. The ZDV union is designed for connecting 1/16″ OD tubing and is pressure rated to 41.4 MPa.

After pumping water into the device for around 1 min, the outlet port was plugged when there was water flowing out from the outlet. The pressure increased slowly when it was below 1 MPa, but subsequently at a faster rate until it reached a maximum before it dropped suddenly. The peak value corresponded to the burst pressure.

RESULTS AND DISCUSSION

All the experimental results obtained are averaged values from three identical tests, standard deviations are also calculated for the tests.

Elastic modulus

The three-point bending tests were conducted at 25, 122, and 125°C. The bending stress σ and bending strain ε were determined using the relationships:

$$\sigma = \frac{3Wl}{2bh^2} \quad (1)$$

$$\varepsilon = \frac{6fh}{l^2} \quad (2)$$

where W is load (N), l is the span between the support (mm), b and h are the width and thickness of the specimen (mm), respectively. f is the deflection of the specimen with load (mm).

The elastic modulus was derived from the linear portion of the stress–strain curves of the flexure tests, and the results were given in Table II. Only the bending modulus of the cover plate material (Topas 5013) and not the substrate plate was determined since the deformation during thermal bonding is confined mainly to this plate.

Thermal bonding and burst pressure testing

The original microchannel was designed with a V-shape with a depth of 50 μm and a width of 100

TABLE II
Elastic Moduli of Topas 5013 Cover Plate at Different Temperatures

Temperature (°C)	Elastic modulus (GPa) (± standard deviation)
25	4.41 ± 0.03
122	0.88 ± 0.06
125	0.85 ± 0.04

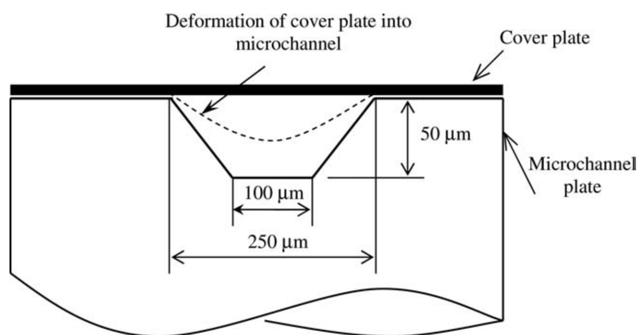

Figure 2 Sketch of cover plate with microchannel plate in thermal bonding.

μm at the bottom and 250 μm at the top, see Figure 2. It was found that the polymer material from the softened cover plate would sometimes flow into the microchannel during thermal bonding and the depth of this deformation was dependent on the bonding pressure, temperature, and the holding time. A typical deformation on the debonded cover plate as measured using the confocal microscope was shown in Figure 3. It should be noted that debonded cover plate in Figure 3 has been inverted to facilitate measurement of the profile of the deformation. In the device, the deformation extends from the cover plate into the cavity of the molded microchannel in the substrate plate.

The initial thermal bonding experiments were conducted at 130, 134, and 140°C, respectively, but at the same pressure and time, as shown in Table III. These represented temperatures that were much lower than the glass transition temperature of the substrate plate which contained the molded micro-

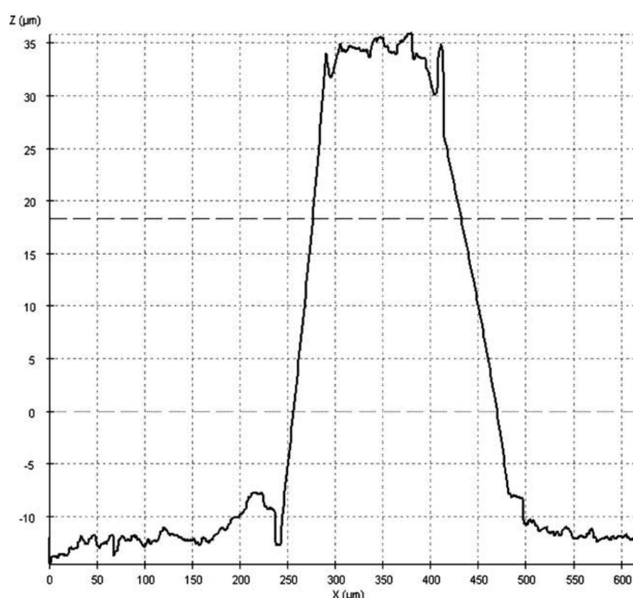

Figure 3 Debonded 5013 cover plate with protrusion into 6015 microchannel (bonding conditions: T = 134°C, P = 0.89 MPa, time = 10 min.).

TABLE III
Bonding Conditions and the Corresponding Deformation of Cover Plate

Temperature (°C)	Time (min)	Pressure (MPa)	Height of deformation of cover plate (μm)	Bonding results
130	10	0.89	38	No bonding
134	10	0.89	42	Poor bonding
140	10	0.89	50	Poor bonding

channel, but near the T_g (134°C) of the cover plate. It was found that the bonding was poor at a low bonding pressure of 0.89 MPa, where the plates were bonded nonuniformly and sometimes air bubbles were visible between the plates. Furthermore, it is apparent from Table III that the deformation of the 5013 cover plate was quite significant, with the softened material protruding between 80 and 100% into the microchannel (which had a depth of 50 μm). However, the geometry of the microchannel remained the same as that before thermal bonding. Therefore, the substrate with microchannel can be regarded rigid in the numerical simulation.

The above results clearly showed that a combination of low pressure and high temperature (relative to the T_g of the cover plate) leads to poor bonding. To improve bonding and reduce the deformation of the cover plate into the microchannel plate, the bonding temperature was reduced while the bonding pressure was increased (see Table IV). It should be noted that larger deformation leads to a corresponding decrease in microchannel cross-sectional area for fluid flow in the device. Table IV indicates that the deformation of the cover plate was appreciable with protrusion heights of more than 20 μm for thermal bonding at 128°C although the burst pressure of these thermally sealed microchannel was more than 5 MPa. The true burst pressure of such specimens could not be determined due to failure of the epoxy glue (it can only withstand a maximum pressure between 5 MPa and 6 MPa) at the outlet joint during testing.

Table IV also indicates that thermal bonding at 122°C or below will lead to small deformation and good thermal bond strength for normal applications. For example, only a very small deformation of 5 μm and a burst pressure of 1.7 MPa was obtained for the sealed microchannel for bonding at 120°C and a bonding pressure and time of 1.85 MPa and 10 min, respectively.

MODELING AND SIMULATION

Modified creep model

During thermal bonding at a given temperature and pressure, the viscoelastic COC material experiences

TABLE IV
Thermal Bonding at Lower Temperatures, and Corresponding Deformation and Burst Pressure

Temperature (°C)	Time (min)	Bonding pressure (MPa)	Height of deformation of cover plate (μm)	Burst pressure (MPa)
120	10	1.85	5	1.67 ± 0.15
122	10	1.85	11	2.11 ± 0.22
125	10	1.85	22.5	3.67 ± 0.20
128	5	2.59	20	>5
128	10	1.85	37.5	>5
128	10	2.59	42	>5
128	10	3.71	50	–
130	10	1.85	47.5	–

a time-dependent increase in strain, this is known as viscoelastic creep. This creep is quite severe since thermal bonding is conducted at a high temperature near the glass transition temperature. Since the cover plate and substrate plate are held for a fixed duration of 10 min at specified temperature and pressure, the deformation of the plates during the bonding process can be assumed to be akin to that in a quasi creep test.

A modified five-constant creep model which combines the effects of bonding pressure and temperature is proposed to account for the viscoelastic behavior of the Topas COC during thermal bonding. This modified model is similar to some creep models showing the relationship of strain with stress and temperature.[15,16] However, the proposed model is different from other creep models in that an additional constant, T_0, which is regarded as a critical deformation temperature, is introduced. In the proposed modified model, at temperatures below T_0, the short term creep deformation is assumed to be sufficiently small that it can be neglected. However, when $T > T_0$, the short termdeformation increases significantly with increasing temperature. This concept of a critical deformation temperature T_0 is similar to the notion of the heat deflection temperature (HDT) in polymers where the short term deformation under a fixed load suddenly increases significantly at the HDT.

The proposed modified creep model can be written as:

$$\dot{\varepsilon} = c_1[\sinh(c_2P)]^n \exp\left(-\frac{c_3}{T - T_0}\right) \tag{3}$$

where $\dot{\varepsilon}$ is creep strain rate, P and T are applied pressure and temperature, respectively. c_1 is a material constant, c_2 is a pressure-dependent parameter, n is a power-law index, c_3 is a parameter related to temperature, and T_0 is a critical deformation temperature.

The creep strain rate was obtained based on the assumption that the deformation was linear with time, as the holding time was set within 10 min. The constants in eq. (3) were obtained by curve fitting based on experimental results and were listed in Table V. The fitted value of T_0 was found to be 114°C. The HDT of a polymer is known to be pressure dependent, and decreases with increasing pressure. The supplier, Ticona Company (USA),[17] indicates that the HDT of Topas 5013 decreased from 127 to 116°C when the applied pressure was increased from 0.45 MPa to 1.8 MPa. Therefore, an HDT value of 116°C was used in the range of bonding pressure between 1.85 MPa and 2.5 MPa (see Table IV). This is in good agreement with the calculated value of 114°C for T_0.

The deformation of the cover plates with applied pressure and temperature for a fixed bonding time of 10 min can be determined using eq. (3) as shown in Figure 4. The comparisons of the fitted with measured deformations at different temperatures and pressures were shown in Figure 5(a,b). It can be seen that there is good agreement between the predicted and experimental results with deviations of

TABLE V
Constants in Modified Creep Model

c_1 (1/s)	c_2 (MPa^{-1})	n	c_3 (K)	T_0 (K)
1.36×10^{-2}	3.15×10^{-6}	0.3	23.12	387.3

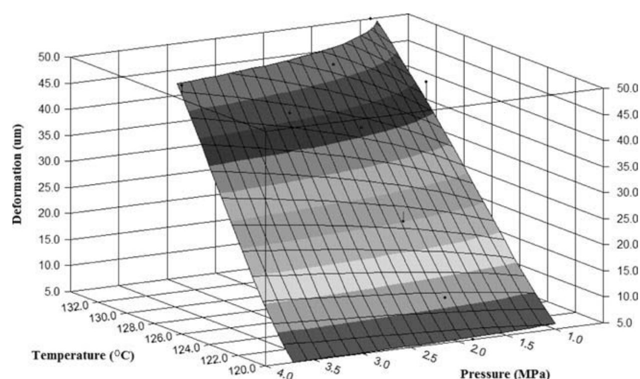

Figure 4 Relationship of deformation with temperature and pressure.

less than 10%. Furthermore, it can be observed that temperature had a more significant effect than pressure on the deformation of the cover plate.

Simulation of protrusion in cover plate

The modified creep model was incorporated into ANSYS^{TM} to determine the height of the protrusions in the cover plate at the same combination of temperature and pressure that were utilized experimentally.

A 2D plain strain finite element model was established to simulate the creep deformation of the cover plate. The element plane 182 with 4-node in ANSYS^{TM} was implemented. Coarser mesh was created in the area in contact with the substrate but away from the microchannel, while finer mesh was utilized in the area in the vicinity of the microchannel to obtain accurate simulation results. A half 2D model was used due to symmetry, with the appropriate symmetric boundary condition applied. The vertical degree of freedom was constrained at the bottom of the cover plate except for the nodes close to the microchannel. The pressure load was applied on the top of the cover plate.

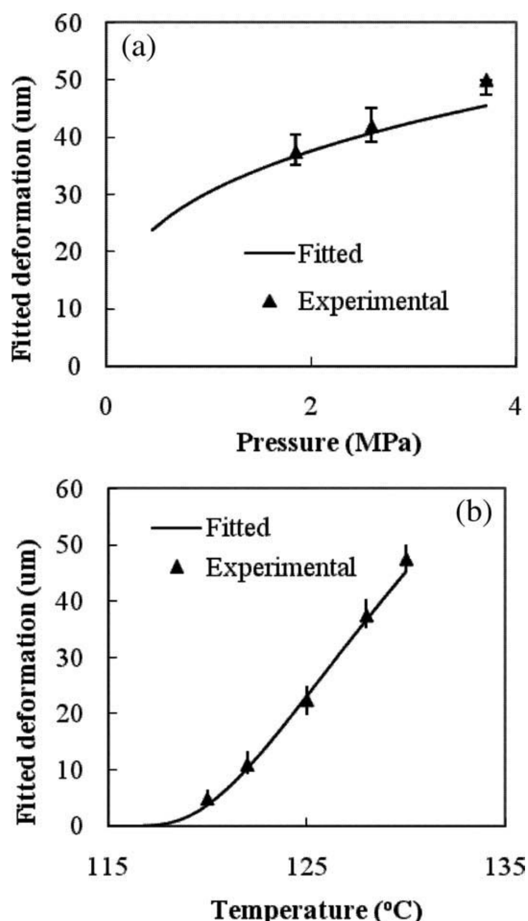

Figure 5 Fitted creep deformations at different temperatures and pressures. (a) Pressure = 1.85 MPa, Time = 10 min. (b) Temperature = 128°C, Time = 10 min.

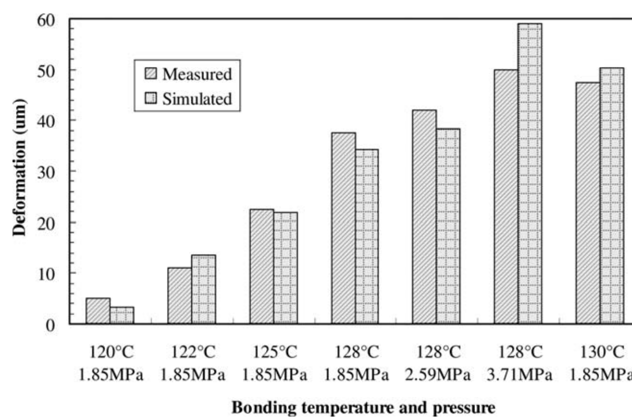

Figure 6 Comparison of measured and simulated deformation at different conditions.

The elastic moduli, together with the parameters in eq. (3), were input into the model. A static analysis was conducted to calculate the deformation at different temperatures, see Figure 6. There is a good agreement between the experimentally measured and simulated deformation of the cover plate at different conditions. The deviation between them was less than 10%, which is acceptable. There is an exception (for 128°C, 3.71 MPa) with an error of 18%. However, this can be attributed to the fact the deformation of the cover plate for this particular case was constrained and restricted because the deformation had completely filled the microchannel in the substrate plate. Hence, it is evident that the material flow from the cover plate into the substrate plate in the vicinity of the microchannel cavity can be modeled utilizing ANSYS with the modified creep model.

CONCLUSIONS

Disposable microfluidic plates with different glass transition temperatures (with/without microchannels) were manufactured by injection molding, and the sealed devices were fabricated by thermal bonding at a temperature near the T_g of the cover plate, but much lower than the T_g of the substrate with the microchannel. As such, the deformation of the substrate with microchannel can be negligible in thermal bonding. The deformation of the cover plate was investigated at different bonding temperature and pressure. The corresponding bonding strength was determined by the burst pressure test. It was found that bonding temperature dominates the deformation of the polymer plate. Therefore, it is advisable to perform thermal bonding at a lower temperature for a smaller deformation of the cover plate. The process of thermal bonding can be modeled as a quasi creep test and the deformation of the cover plate can be predicted accurately using a quasi creep model. The model contains a critical deformation temperature, T_0, which represents the

heat deflection temperature at high pressure. The deformation predicted by numerical simulation with the creep model has good agreement with the obtained experimental results. Therefore, the model can be utilized to predict the appropriate conditions for thermal bonding of the microfluidic device.

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