Investigation on CO₂ laser irradiation inducing glass strip peeling for microchannel formation

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The study investigates the use of CO₂ laser to induce glass strip peeling off to form microchannels on soda lime glass substrate. The strip peeling exhibits a strong dependence on the energy deposition rate on the glass surface. In spite of the vast difference in the combination of laser power and scanning speed, when the ratio of the two makes the energy deposition rate in the range 3.0-6.0 J/(cm² s), the temperature rising inside glass will be above the strain point and reach the softening region of the glass. As a result, glass strip peeling is able to occur and form microchannels with dimensions of 20-40 μm in depth and 200-280 μm in width on the glass surface. Beyond this range, higher energy deposition rate would lead to surface melting associated with solidification cracks and lower energy deposition rate causes the generation of fragment cracks. © 2012 American Institute of Physics. [doi:10.1063/1.3670362]

I. INTRODUCTION

Microchannels forms the backbone of microfluidic devices.¹ Glass microchannels are normally formed by wet chemical etching, which involves multiple steps such as photomask manufacturing, photolithography, and hydrofluoric acid etching.² It is noted that microfluidic channels are generally designed according to the particular requirements of each application, it is frequently necessary to modify the channel design in order to optimize the performance of a microfluidic device. This demand drives to develop rapid prototyping techniques of versatile microfluidics. Laser techniques may provide a high level of process flexibility in microfluidic manufacturing, allowing the iterations of the design and fabrication of microfluidics with improvements in short time cycle and cost effectiveness.³⁻⁵ Laser technique is thus considered a faster alternative to conventional techniques such as wet chemical etching with photomask, lithography, electron beam writing, or photo-patterning in terms of rapid prototyping.^{2,6}

In fact, great progress has been made in fabrication of glass microchannels using lasers in the past decades. One common method is the use of laser ablation to remove material for microchannel fabrication. Laser ablation refers to photon sputtering process, the explosive removal of material from a sample under the irradiation of an intense laser pulse.^{7,8} In the laser ablation of a solid material, a pulsed laser is used to heat a solid surface and a small quantity of material is vaporized. Through further photon absorption, the ejected material is heated up until it ionizes and expands from the sample surface as a plasma cloud. The material removal rate ranges from a fraction of a monolayer to a few monolayers per pulse, which is directly proportional to laser fluence. There is a threshold in laser fluence or intensity below which only laser-induced desorption, without destruction of the sample surface, occurs. Some of the earlier studies on microchannel fabrication using laser ablation are, for instance, nanosecond (ns) ultraviolet (UV), visible, and infrared (IR) lasers ablation of fused quartz and Pyrex glass,^{9,10} femtosecond (fs) near infrared (NIR) laser ablation of quartz,¹¹ water-assisted fs-NIR laser ablation

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of the rear surface of silica glass,¹² and ns-IR Nd:YAG laser-induced thermal energy or plasma to machine microchannels in three-dimensional (3D) quartz.^{13,14}

Another approach is the use of laser irradiation followed by wet chemical etching to remove material for microchannel fabrication. The glass property in region of laser irradiation is modified and the laser irradiated part is preferentially etched away during etching process. Some earlier studies on microchannel fabrication with this method are, for instance, UV light irradiation of photostructurable glass followed by post wet chemical etching,^{15–17} fs-NIR direct writing of photostructurable glass followed by post wet chemical etching^{18–21} fs-NIR laser irradiation of silica followed by post wet chemical etching,²² and instant etching during laser irradiation, i.e., laser-induced backside wet etching (LIBWE).^{23–25} Instead of using wet chemical etching, an effective method has been developed to fabricate 3D homogeneous microchannels by water-assisted fs direct writing inside porous glass followed by postannealing for consolidation recently.^{26,27} Microchannels with arbitrary lengths and configurations could be achieved inside glass.

Fundamentally, when laser is employed to machine a material, the optical absorption of the material to the laser beam is a significant concern, which determines the laser machining competency. As glasses absorb laser energy in a way which is dependent highly on the incident wavelength,^{28,29} photon absorption mechanism plays a key role in laser machining of glass materials. When the wavelength of a laser beam is above $5\text{ }\mu\text{m}$, laser energy is highly absorbed by glasses, such as borosilicate, fused silica, and soda lime.²⁹ CO_2 laser radiation at wavelength of $10.6\text{ }\mu\text{m}$ is strongly absorbed by a Si-O vibration mode.^{30–33} However, CO_2 laser is rarely used to directly ablate glass materials to fabricate microchannels due to the cracks induced by thermal stress.^{32,34,35}

So far, Zhao *et al.*³⁶ reported that surface channels a few μm deep can be obtained through the mechanisms of thermo-capillary force, vaporization-induced material removal, and recoil pressure in CO_2 laser irradiation of silica glass. Özcan *et al.*³⁷ reported that channels around $20\text{ }\mu\text{m}$ deep with free of bumps, redeposition, and cracks on both sides of channel can be obtained by CO_2 laser ablation of polymer layer protective silica glass substrates. Very recently, Chung and Lin³⁸ reported that clean and crackless channels were obtained by CO_2 laser ablation of a Pyrex 7740 glass slide which was immersed in water. Yen *et al.*³² demonstrated that the maximum channel depth of $225\text{ }\mu\text{m}$ can be fabricated when a CO_2 laser was used to ablate a borofloat glass substrate that was heated by means of an underlying hot plate ($150\text{--}300^\circ\text{C}$).³²

Instead of laser ablation, a few years ago, we found that a continuous strip of glass layer could be peeled off under CO_2 laser irradiation of borosilicate glass and channels were left behind along the laser beam path.³⁹ However, no continuous glass strips were observed when the CO_2 laser irradiated the soda lime glass in the similar experiments. Two years later, Wang and Lin⁴⁰ observed that continuous chips were obtained when a CO_2 laser with a line-shape beam was used to cleave a soda-lime glass substrate at beam-rotation angle of 0° to the cutting direction. In contrast, surface cracks were obtained instead when the beam rotation angle was at 45° or 90° .

The difference in the thermal shock resistance of the two glass types is believed to be one of the main reasons for the obtained results.³⁹ Microcracks are not readily formed for glass of high thermal shock resistance like borosilicate. The absence of microcracks implies that the forces associated with the thermal shock are allowed to accumulate in the material. There would be a point where these forces are high enough to separate the bonds holding the material together and form a continuous glass strip. As a result, strip formation is only possible for the borosilicate glass but not for the soda-lime glass. Soda lime glass is the most economic and prevailing type of glass, used in mirrors, windowpanes, microscopic slides, printed circuit substrates, and electronic applications. Therefore, this study focuses on CO_2 laser microprocessing of soda lime glass.

Be it ablation or strip peeling, both phenomena are through CO_2 laser depositing its photon energy on the glass surface. The amount of energy deposited into glass and the energy deposition rate are the crucial factors in determining the thermal gradient, resulting in equilibrium or non-equilibrium physical and chemical reactions at glass surface, i.e., ablation, melting, thermal stress, and the crack formation during interaction between laser energy and glass. Thus, it will be interesting to study the effects of CO_2 laser energy deposition on the surface phenomena induced by laser irradiation. The related process parameters to be investigated are thus laser

beam energy and laser beam scanning speed, as they determine the laser energy deposition on the glass surface. Special effort was made to investigate how the laser energy deposition could enable the softening of glass for strip peeling off, resulting in formation of microchannels on soda lime glass surface for microfluidics applications.

II. EXPERIMENT

In the experiments, the soda lime glass used was commercially available soda lime glass slides, microscope slide Cat No. 7101, with a thickness of 1.1 mm and area of 26 mm by 76 mm. The CO₂ laser used was a Synrad J48-2 W, with a wavelength of 10.6 μm . The laser beam has a Gaussian profile energy intensity distribution and M^2 of 1.2. Maximum output power under continuous wave (CW) mode operation is 25 W. This laser allows modulating of its CW mode into pulse mode through an external pulse generator with a frequency of 5 KHz. The output laser power could be tuned through pulse width modulation, i.e., by adjusting the pulse duty cycle (DC). The laser power from a single pulse was varied by changing the DC so as to study the various energy depositions.

The original beam diameter of the laser output is 3.5 mm. The laser beam was doubly expanded to a diameter of 7 mm and was focused by an F-Theta-Ronar lens of 150 mm in focal length. The spot size was calculated to be about 347.05 μm at the focal point. During laser irradiation of the glass surfaces, a positive defocus, i.e., the focal point was 1.5 mm above the glass sample surface, was employed in order to reduce the possibility of crack generation through the reduced laser energy density deposited at the glass surface. The laser beam was scanned on the glass surface through a galvanometer scanner at a scan speed of up to 2000 mm/s.

The surface morphology of the laser irradiated samples was observed using optical microscope and scanning electron microscope (SEM, Zeiss EVO50). The geometric dimensions of the microchannels formed by laser-induced glass strip peeling were characterized in three-dimensions (3D) as well as two-dimensions (2D) using a Stylus Profilometer (Taylor-Hobson Form Talysurf Series 2). The vertical resolution of the stylus profilometer is 10 nm and lateral resolution 0.125 μm .

III. RESULTS AND DISCUSSION

A. Glass strip peeling off induced by laser irradiation

To investigate the glass strip peeling, various laser conditions were employed to irradiate the glass sample surfaces. Fig. 1 is a plot of laser scan speed against the laser power (duty cycle). It was observed that a continuous strip of glass layer was peeled off with a single beam scan over the moderate range (i.e., in region 3) of laser power and scan speed. When the laser irradiation parameters were outside the moderate range, cracks were easily produced instead of strip peeling. For example, at a given laser power, lower scanning speed resulted in glass surface melting and led to crack generation along the beam path in the rapid melting-solidification process (in region 4). With an increase in scan speed outside the moderate range, crash-like cracks and fragments were produced along the laser irradiated belt (in region 2). Further increase in scan speed resulted in isolated cracks or no noticeable changes in the beam path (in region 1).

Different morphologies with respect to the appearance regimes of peeling, melting, and cracking induced in the four parameter regions are shown in Fig. 2. The details of the observations to the glass strips peeled by laser irradiation and the grooves formed after strip peeling off are shown in Fig. 3. The strips show a bent shape and they were bent up from the starting point of laser scanning. It could indicate that the glass surface layer experienced a thermal softening and cooling shrinking process under laser irradiation. Due to the peeling feature, the morphology of the groove bottom (Fig. 3(e)) shows a corresponding appearance to the strip bottom (Fig. 3(c)). And the strip thickness represents the groove depth. The grooves have clean edges and are free of cracks.

Figs. 1 and 2 thus clearly show that there is an optimal range in laser power and scanning speed for strip peeling off such that microgrooves are able to be formed on soda lime glass

FIG. 1. Moderate range (in region 3) of laser parameters for glass strip peeling off.

surface. However, in the view of engineering, in order to find the suitable laser conditions for strip peeling off and, therefore, microgrooves formation, plenty of experiments are required to be carried out physically by trial-and-error method. If without the assist of computational modeling or other effective instructions, there is difficulty in selection of the suitable laser process parameters for peeling off glass strips.

B. Optimal energy deposition in inducing strip peeling off

To avoid trial-and-error method for glass strip peeling, it would be necessary to understand how the laser thermal energy deposition can induce glass surface layer softening and shrinking such that strip peeling enables microgroove formation to occur. Laser as a thermal source

FIG. 2. Morphologies induced by laser irradiation under various scan speed ranges. (a) Groove formed by laser peeling at a moderate scan speed, (b) melting with cracks at a slow speed, (c) continuous cracking belt at a high speed, and (d) discontinuous cracking belt at a high speed.

FIG. 3. CO₂ laser-induced peeling of glass strips from a soda lime slide under a laser duty cycle of 0.15 and scan speed of 240 mm/s. (a) A plane view of glass strips, (b) an enlarged plane view of a continuous glass strip, (c) top view of a micro-groove formed by glass strip peeling, (d) top view of a glass strip, and (e) backside view of the glass strip. (a) and (c) are optical images and (b), (d), and (e) are SEM images.

deposits its energy into the glass substrate during the laser irradiation of the glass surface. The deposited thermal energy heats the glass to different temperatures and may cause the glass to vaporize, melt, peel off, and crack. Therefore, despite the vastly different process parameter combinations of laser power and scanning speed (Fig. 1), the appearance of which of the four regimes would clearly depend on the laser energy delivered to glass surface. The total energy deposited on the glass surface per unit area is related to the pulse energy and the pulse number at the area. Pulse energy is related to the initial output of laser power and pulse number is a parameter which is determined by the laser beam scanning speed. Indeed, the combination of the two parameters, the pulse energy and the scanning speed, dictates the energy deposition rate of a single scan of the laser beam.

If the energy deposition rate of a single scan is defined as the amount of energy deposited on the glass surface per unit area per unit time, it typifies the moving laser energy along the scanning path, i.e., the energy deposited while the laser beam traversed per unit area in 1 s. For the single scan of laser beam, the value of energy deposition rate is equal to that of the total energy deposited at per unit area in 1 s. The value during a single scan may be approximately written as Eq. (1) which is obtained by dividing the product between pulse energy and pulse number with the area scanned in 1 s. The total energy deposited per unit area on the glass surface during a single scan can be calculated as Eq. (2) below.

$$R_E = \frac{(W \cdot \tau) \cdot K}{2\omega \cdot L_0}, \quad (1)$$

where,

R_E -Energy deposition rate of a single scan along the beam path, $J/(\text{cm}^2 \cdot \text{s})$.

W -Average power of a single pulse.

τ -Pulse width, i.e., time duration of pulse duty cycle, s.

$(W \cdot \tau)$ -Total photon energy (E_p) of a single pulse, i.e., pulse energy, J.

K -Pulse number in 1 s, i.e., pulse frequency of laser beam, Hz (s^{-1}).

$(W \cdot \tau) \cdot K$ -Total energy deposited in an area scanned in 1 s, J.

ω -Laser beam radius on the glass surface, cm.

L_0 -Scanning distance of laser beam, cm, for 1 s.
 $(2\omega \cdot L_0)$ -Area scanned by laser beam, cm^2 , in 1 s.

$$E_T = \left(\frac{(w \cdot \tau) \cdot K}{2\omega \cdot L_0} \right) \cdot \left(\frac{L_0}{v} \right). \quad (2)$$

Or after simplification as

$$E_T = \frac{W \cdot \tau \cdot K}{2\omega \cdot v}, \quad (3)$$

where,

E_T -Total energy deposited per unit area on the glass surface, J/cm^2 .

v -Scanning speed of laser beam, cm/s .

$\frac{L_0}{v}$ -Scanning time of a single scan used to scan a length of L_0 , s.

Under the varied laser energy deposition, the high absorption coefficient of the CO_2 laser irradiation causes substantial heating of a thin surface layer and its deposition amount determines the degree of the temperature rising at the laser irradiation area.³⁰ Heat flow within the irradiation zone is difficult to model accurately. Most successful models utilize finite element or finite difference analyses with parameters varying both spatially and temporally. However, these models are cumbersome and require extensive computing facilities. For explaining the laser-caused thermal heating, a steady-state model may be used. In the thermal analysis of laser irradiation of the soda lime glass substrate, we refer to the previously reported studies^{40,41} and the following assumptions are made

- (1) The CO_2 -laser beam is regarded as a surface heating source.
- (2) On the surface of the glass without laser heating, the superficial heat irradiation is negligible.
- (3) The thermal properties of the glass are isotropic and temperature-independent.
- (4) Heat conduction in the specimen is negligible, as the deposited energy is on the thin surface layer and it is far greater compared to heat loss.
- (5) The convection and thermal radiation in the surrounding environment are considered into the laser energy absorption efficiency of soda lime glass (absorption efficiency of 80%, page 13, line 14-15).
- (6) The heating phenomena due to phase changes are neglected.
- (7) Inertia effects are negligible during stress development.

Based on the above assumptions, the mathematical heat transfer model can be established and it was expressed by Eq. (4), thus, the difference in temperature induced by laser irradiation can be easily obtained.

$$Q = (A \cdot L_0 \cdot \rho) C_p \Delta T, \quad (4)$$

where,

Q -Heat added to glass from laser irradiation for 1 s (J). Its value is the same as the energy amount deposited into glass by laser irradiation for 1 s.

A -Vertical plane area; the plane is perpendicular to the laser scanning direction and through beam propagation axis in the absorption layer inside glass, cm^2 .

ρ -Mass density of glass, g/cm^3 .

$(A \cdot L_0 \cdot \rho)$ -Mass of the glass heated by laser irradiation.

C_p -Specific heat of glass, $\text{J}/(\text{g}^\circ\text{C})$

ΔT -Temperature rise caused by laser irradiation, $^\circ\text{C}$.

It is noted that the area A is related to the thermal transferring thickness inside glass, which is difficult to measure experimentally or give a theoretical value. In this study, we simply employed the cross-sectional area of the microgroove as an approximately estimation of the area A . Absorption efficiency of soda lime glass to CO_2 laser beam energy was hypothesized to

be 80%.^{40,42} Thus, a plot to express roughly the temperature rising against the energy deposition rate was made and shown in Fig. 4. It shows that the energy deposition rate at the range 3.0–6.0 J/(cm²·s) during a single scan of laser beam for 1 s could heat the glass surface above its strain point of 514 °C and in the softening region more or less than 720 °C (<1000 °C).^{41,43} Glass strip peeling off was induced easily in this temperature region. At higher energy deposition rate above 7.0 J/(cm²·s), the temperature calculated was above 2000 °C (without considering the fusion of heat). Serious melting associated with solidification cracks appeared on the glass surface (Fig. 2(b)). With decrease in energy deposition rate below 3.0 J/(cm²·s), the temperature calculated was below softening point of 720 °C and resulted in a continuous cracking belt along the laser scanning path. When the energy deposition rate was less than 1.5 J/(cm²·s), temperature calculated went down below strain point 514 °C, and isolated crack was produced under the pulse shock of laser irradiation (Fig. 2(d)).

It is also noted that the glass strip peeling is in a form of soft bending of a solid layer of glass (Figs. 2(a) and 2(b)). It implies that the glass layer temperature reached the glass softening point during the laser heating process. The softening of the glass layer makes it possible to deform or bend, and thus to be peeled off as a strip by contraction along the laser beam scan path without cracking. In contrast, when the glass is in the molten state, strip peeling could not occur. Similarly, when the temperature is below the softening point but exceeding the glass strain point, cracking belt is produced and, therefore, no strip peeling can be achieved.

FIG. 4. Optimal energy deposition rate in inducing glass strip peeling off (Temperature calculation without considering the fusion of heat of the glass, and the bottom graph is a magnified view of the region at lower energy deposition rates.).

FIG. 5. Schematic of thermal gradient induced by laser irradiation.

An illustration has been made to show the thermal gradient induced by laser irradiation in Fig. 5. The strain point represents the point of elastic limit and softening point represents the point of plastic deformation. Because of the strip bending up features and the Gaussian temperature distribution in the laser beam, the average temperature in the strip layer should be at least above the strain point. As glass is cooled, the inside does not cool as fast as the outside of the glass. The inside stays softer or molten and expands while the outside is starting to get stiff and begins to contract. If the glass is cooled too fast, the expansion and contraction are frozen into place and this creates stress in the glass. The glass will eventually break to relieve this built up stress. For the strip peeling off, the cracking would occur at a region with a temperature between the strain point and the softening point.

Under the average temperature in the irradiation zone (strip layer) above the strain point, the laser peeling process may be explained by the expansion and contraction cycles generated during the laser beam and glass interactions. When the laser beam irradiates the glass, the affected region gets heated up by the high beam power intensity, whereas the surrounding regions remain at a much lower temperature. This would create a steep temperature gradient between the affected and nonaffected regions. Rapid cooling takes place by convection and radiation as the laser beam leaves the affected region and causes the glass to contract. The expansion and contraction of the glass substrate causes the boundary of the affected region to experience stress forces. The induced forces are expected to be proportional to the thermal expansion coefficient of the material.

As the laser beam energy distribution is similar to the Gaussian distribution, the center region of the groove gets heated up more than the other regions. The highest power density is at the center region, it has the highest temperature change and thus the greatest expansion than the other regions of the glass substrate. Upon cooling, the center region undergoes the greatest amount of contraction. The expansion and contraction action creates the greatest force in the center. The bending momentum is greater along the length of the glass strip than that across the width. Laser irradiation thus leads to the removal of the glass material in the form of a continuous strip (laser peeling) and microgroove could be formed on glass substrate surface.

Therefore, from Figs. 4 and 5, the energy deposition rate may be used as a guide to facilitate the selection of the laser-induced peeling parameters for groove formation on the soda lime glass surface. Laser irradiation at whatever pair of laser power (i.e., duty cycle) and scanning speed, as long as the ratio of them from the Eq. (3) is located in the range $3.0\text{--}6.0\text{ J}/(\text{cm}^2\cdot\text{s})$, the temperature rising inside glass would be above the strain point and in the softening region of the glass. As a result, glass strip peeling should be able to occur and form microgrooves on the glass surface.

C. Microchannel fabrication

Under the optimal laser energy deposition rates of $3.0\text{--}6.0\text{ J}/(\text{cm}^2\cdot\text{s})$ in Figs. 1 and 2, various microchannels were fabricated on the soda lime glass substrates. The cross-section of the microchannel was measured to be a U shape, as shown in Fig. 6. A smooth sidewall was

FIG. 6. Profiles (3D top and 2D bottom) of the microchannel formed at energy deposition rate of $4.5 \text{ J}/(\text{cm}^2 \cdot \text{s})$ with laser duty cycle of 0.15 and scan speed of 240 mm/s.

FIG. 7. Optical image of a microchannel cross-section showing no clear ridge appeared around the channel edge during laser thermal peeling process.

FIG. 8. Changes in microchannel dimension against the laser energy deposition rate.

achieved in the microchannel. The part appeared to be rough and shallow at the right bottom of the microchannel, however, which could be caused by asymmetric distribution of laser energy or a tilted laser beam. The dimension of the microchannel measured from stylus profiler was $36\text{ }\mu\text{m}$ in depth and $220\text{ }\mu\text{m}$ in width. Because even a small bump generated along the edge of the microchannels during the laser thermal peeling process will dramatically decrease the following bonding yield, cross sectional view of microchannel were also carried out under optical microscope. The microchannel was mechanically sectioned along its axis and its section was sand-polished for carrying out the channel sectional observations. The image in Fig. 7 shows that no obvious ridge appeared around the channel edge, which is in agreement with the measurement from stylus profilometer (Fig. 6). Under the optimal deposition rate from $3.0\text{ J}/(\text{cm}^2\cdot\text{s})$

FIG. 9. 3D profiles of microchannels fabricated at energy deposition rate of $3.72\text{ J}/(\text{cm}^2\cdot\text{s})$ with duty cycle of 0.2 and scan speed of 380 mm/s . (a) A curved shape with a radius of 2 mm and (b) multi-scans of laser beam horizontally at a overlapping pitch of $150\text{ }\mu\text{m}$.

to 6.0 J/(cm²·s), the microchannel size was varied from 20 μm to 38 μm in depth and from 200 μm to 280 μm in width (Fig. 8).

As the microchannels used in microfluidics are not only in a linear straight structure but also often in a non-linear structure, fabrication of the microchannel in a curved shape at various radii was attempted. Furthermore, the horizontal overlapping of the laser beam with a certain pitch between the two scans was also carried out such that wider channel widths could be achieved. For example, a 540 μm width channel was obtained by scanning the laser beam with three overlapped passes at a horizontally shifted pitch of 150 μm. The profiles of the above mentioned microchannels are shown in Fig. 9. It needs to be mentioned that when the radius of microchannel goes to smaller, for instance, below 1 mm, the channel was clogged without strip peeling off. Nonetheless, the results could show that laser peeling process is flexible and efficient to fabricate various microchannels on glass substrate.

IV. CONCLUSION

A continuous strip of glass layer can be peeled off along the moving path of the laser beam under a single scan of a CO₂ laser beam, and thus form microgrooves on the soda lime glass substrate. The energy deposition rate is a crucial factor in determining the rise of temperature of the glass and its value determines the regimes: melting, cracking, and peeling induced by laser irradiation on glass surface.

The study reveals that in spite of the different combinations of laser power and scanning speed, as long as the ratio of the two results in the energy deposition rate in the range of 3.0-6.0 J/(cm²·s), the temperature rising inside glass will be above the strain point and reach the softening region of the glass, leading to glass strip peeling on the glass surface. Outside this range, higher energy deposition rate results in surface melting associated with solidification cracks. Inversely, lower energy deposition rate causes the thermal cracks on glass surface.

Various microchannels with dimensions of 20-40 μm in depth and 200-280 μm in width have been successfully fabricated on the soda lime glass substrates by the laser-induced peeling process. The process is flexible and efficient, which could offer potentials for microfluidic channel fabrication on the glass substrates without post-process and wet chemicals.

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