## A New Approach to Seal Polymer Microfluidic Devices Using Ultrashort Laser Pulses

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We report on the joining of two different transparent thermoplastic polymers using infrared femtosecond laser pulses. Solely being based on nonlinear absorption, the developed micro-welding process for cyclo-olefin copolymers and polycarbonate does not require any intermediate absorbing layers or any surface pre-processing of the welding partners. In addition, it circumvents any heat damages at the outer surfaces of the welding partners. Femtosecond laser welding of cyclo-olefin copolymers is improved by increasing the numerical aperture of the focusing setup resulting in a wider and more homogenous welding cross-section. Welding results are classified by shear strength and fracture modes are analyzed using scanning electron microscopy. Furthermore, we demonstrate, to the best of our knowledge, first results of ultrashort pulse laser welding of transparent polycarbonate, one of the most used transparent polymers. In view of an optimized and stable micro-welding process, we study the influence of laser power and focal position on welding results. The application of ultrashort pulse laser welding for Lab-on-chip fabrication technologies is demonstrated by sealing an ultrashort pulse laser ablated microfluidic device based on a transparent polymer substrate.

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#### 1. Introduction

Lab-on-chip (LOC) systems gain an increasing attention to be part of a point of care diagnostics medical device. State of the art systems are based on transparent polymer substrates due to low material costs as compared to glass [1], biocompatibility [2], and easy processing [3]. Joining and sealing of transparent polymers for LOC applications are one of the most challenging parts in the microfluidic chip production. Beside conventional joining technologies such as thermal or solvent bonding [4], laser welding exhibits no risk of channel collapse. However, conventional welding technologies employing laser diodes or fiber lasers emitting in the near infrared wavelength regime require an absorbent joining partner to perform common laser transmission welding [5]. Clear to clear laser transmission welding is based on a coating of one of the transparent chips with a thin absorbent layer such as graphite [6] or metal [7] serving as an absorber for the laser power, yet imposing the danger of objectionable contamination of the microfluidic system. Another possibility of laser welding of transparent materials is the processing at a material specific semitransparent wavelength. Most polymers such as polymethylmethacrylate (PMMA), polycarbonate (PC), or cyclo-olefin copolymers (COC) show a semitransparent absorption in the wavelength region around 2 µm. Laser welding using a thulium fiber laser emitting at a wavelength between 1800 nm and 2  $\mu$ m is reported by Ruotsalainen et al. [8]. Mingareev et al. [9] performed transmission and butt welding experiments at a welding speed of 5 mm/s in a contour welding process using different

transparent polymers. Thulium laser welding of polycarbonate using a quasi-simultaneous process design at an effective welding speed of 37 mm/s is demonstrated by Mamuschkin et al. [10]. As being based on linear absorption process, welding results are, however, strongly influenced by the thickness of the upper joining partner and the heat impact inside the upper chip may lead to a surface damage. A new approach to join transparent polymers has been introduced by the authors using ultrashort laser pulses [11]. Due to nonlinear absorption of the laser power, an internal controllable heat source is generated. By adjusting the location of the internal heat source to be at the interface between two transparent chips, a melting and welding is initiated. A potential channel collapse or heat influence to the microfluidic system is prevented as well as it circumvents the application of an absorbing layer at the interface of the joining partners. Thus, an extra process step and a potential source of chip contamination are avoided. The generation of the heat source is based on heat accumulation, which requires a high repetition rate laser as shown and calculated by Volpe et al. [12]. This welding technology is also common for welding of glass as shown by Miyamoto et al. [13-16] using ultrashort laser pulses. Volpe et al. [12; 17] and Mizuguchi et al. [18] report on the femtosecond laser welding of PMMA at a welding speed of below 1 mm/s. Welding seams are classified by optical inspection, leakage tests and a maximum shear strength of 21.4 MPa. Roth et al. [11; 19] demonstrate the ultrashort pulse laser welding of transparent COC, an emerging polymer for LOC applications [20; 21; 2]. Using a welding speed of up to 75 mm/s, a shear strength of 37 MPa is achieved. Furthermore, the cross-sectional behavior of the welding region is studied in both contributions.

In this report, we expand the possibilities of ultrashort pulse (USP) laser welding of transparent polymers by studying and demonstrating the weldability of polycarbonate, one of the most common transparent polymers. Furthermore, we indicate the focusing setup as one of the major influence factors on welding seam quality. In addition to that, ultrashort pulse laser welding is used to seal a microfluidic device based on a COC polymer substrate as to demonstrate the usability of this technology in LOC fabrication.

### 2. Experimental

#### 2.1 Laser system

We used an ultrashort pulse laser (Light Conversion, Pharos-10-600) with a fundamental wavelength of 1028 nm having an adjustable pulse duration from 220 fs to 15 ps and variable pulse repetition rate up to 610 kHz. The laser beam is focused by a fixed lens with a focal length of 50 mm. The numerical aperture of the focused beam is controlled by a variable beam expanding telescope. Samples are positioned using linear translation stages (Aerotech, Pro 165 LM). The focal position is controlled by a linear z-stage (Aerotech, Pro 165).

#### 2.2 Materials and characterization

We processed COC plates (size of 11×22 mm) from Ticona GmbH (brand name TOPAS 6017). The material is characterized by a glass transition temperature of 170 °C and a tensile strength of 58 MPa. The plates had a thickness of 1.5 mm and a surface roughness Ra of 25 nm. Polycarbonate samples are obtained from Covestro AG (brand name Makrolon 2458). Plates with a thickness of 3.2 mm are cut to samples (size of 11x22 mm). Makrolon 2458 is characterized by a glass transition temperature of 146 °C and a tensile strength of 65 MPa. The layout of the platelets for the tensile shear tests consists of 2 parallel welding seams with a length of 10 mm and a lateral distance of 500 µm. Welding seam dimensions were selected to get a robust chip design and to minimize the impact of single welding defects. The specimen and testing setup are shown in Fig. 1. The spatial extension of the welding seam is characterized by transmission light microscopy (Nikon, Eclipse LVDIA-N). In addition, an overview picture is taken with a digital macroscope (Leica, DVM6). The mechanical quality of the seam is evaluated by measuring the separating force with a tensile tester (Shimadzu, AG-Xplus). In order to calculate the joining strength, the size of the welded area is determined by a laser scanning confocal microscope (Keyence, VK X-200). A scanning electron microscope (Phenom, X Pro) is used to observe fracture modes of separated chips.

#### 2.3 Process setup

Ultrashort pulse laser welding of transparent materials requires a minimized air gap between both joining partners. Based on previous results and in accordance to Volpe et al. [12], we indicate a sufficiently small gap by the appearance of Newton's interference patterns upon white light illumination. The air gap is minimized by using a pneumatic short stroke cylinder (Festo, AEVC-63-10-I-P) and a maximum force of 1870 N. Taking the chip size of 242 mm<sup>2</sup>



Fig. 1 Lap shear test setup (left) and scheme of the cross-section of the laser processing (right). Please note the focal offset  $\Delta z$ .

into account, a maximum contact pressure of 2.58 MPa is applied on the joining partners. The process is stabilized by a fused silica plate with a thickness of 10 mm above the joining partners and a PMMA board below for clamping. A transparent material below the joining partners is necessary to prevent any ablation at the bottom of the chips. The complete process setup is shown in Fig. 1. To achieve a successful welding, the focal position has to be shifted with respect to the interface between both joining partners in propagation direction of the impinging laser beam. This shift is denoted as  $\Delta z$  in this report.

#### 3. Results and discussion

In order to achieve highest nonlinear absorption, the shortest pulse duration of our laser system, 220 fs, is used. The pulse repetition rate is set to the maximum value, 610 kHz, to ensure sufficient heat accumulation to enable adequate joining. Welding seams are generated in a single line single pass process design also known as contour welding.

# 3.1 Influence of the focusing condition on the welding of cyclo-olefin copolymers

Welding of transparent materials using ultrashort laser pulses is based on a heat accumulation at the interface between both joining partners. Independent of the applied laser parameters for welding, the heat source is generated above the focal spot. Thus, its focal offset  $\Delta z$  has to be adjusted for each process parameter variation. The influence of both laser power and focusing condition is depicted in Fig. 2 using a welding speed of 20 mm/s. The focal offset  $\Delta z$  is adjusted in



Fig. 2 Averaged welding seam width for 3 focusing setups using different laser powers at a scanning speed of 20 mm/s and suitable focal position  $\Delta z$ .



Fig. 3 COC welding seam generated at 20 mm/s and a NA of 0.072 in joined state (left) and top face of the bottom laminate in separated state using a scanning electron microscope (right).

a range between - 0.2 mm and - 0.6 mm for the lowest and the highest power, respectively. Inside the process window for the focal offset  $\Delta z$  average welding seam widths can be continuously increased by using higher laser powers. The applicable laser power and achievable welding seam widths are determined by the applied focusing setup. A higher numerical aperture supports the creation of larger welding seams due to the focal offset  $\Delta z$  by enabling the usage of more laser power at constant process quality. A laser power above the specific process regime creates a welding seam which is dominated by welding defects and irregular melt ejection into the minimized air gap around the former welding seam. Melt spread structures are comparable to formations observed by Mizuguchi et al. [18] reporting on the ultrashort pulse laser welding of PMMA.

The maximum applicable numerical aperture is limited by the nonlinear refractive index of the polymer. Using a NA above 0.09 in combination with a corresponding laser power of 1000 mW results in self-focusing and a filamentation along the beam propagation path inside the polymer. This effect prevents the generation of a single heat source at the interface of both joining partners and therefore circumvents a successful welding. Beside the possibility to create wider welding seams the main advantage of using a higher NA is the possibility to create welding seams without heat degenerated spots at the interface layer inside of the welding seam is surrounded by interference patterns indicating a minimized air gap between the joining partners.



Fig. 4 Required focal position  $\Delta z$  for a successful laser welding at different laser powers.



Fig. 5 Averaged welding seam width using different laser powers at a scanning speed of 40 mm/s and required focal position  $\Delta z$ .

The fracture mode of the separated welding seam produced with a NA of 0.072 is depicted in Fig. 3 (right) and shows a rough but homogenous fracture along the welding seam without cracking along heat degenerated areas as observed in COC welding using a smaller NA [19].

#### 3.2 Welding of polycarbonate

Ultrashort pulse laser welding of PC requires a precise alignment of the focal position in relation to the interface. The process window using a welding speed of 40 mm/s and a NA of 0.072 is depicted in Fig. 4. The process window for a successful welding, i.e. the heat accumulation zone is located at the interface between both joining partners, is enlarged and shifted downwards for higher laser powers. Besides generating a welding defect, a misalignment of the focal position leads to an internal heat damage and a polymer degradation inside the chip. Compared to welding of COC polymers, a slightly larger focal offset  $\Delta z$  is necessary. The average welding seam width between PC chips is influenced by the applied laser power as shown in Fig. 5 using a welding speed of 40 mm/s, a NA of 0.072 and a related focal offset  $\Delta z$  suitable for each laser power (c.f. Fig. 4). Welding seams between PC chips appear to be wider and in contrast to the welding seams generated between COC samples have a significantly textured border, visible in Fig. 6. We attribute this effect to the higher Melt Flow Index (MFI) of the welded PC brand type. The MFI describes the viscosity in the heated state and is an essential parameter for thermal processing of polymers. At a higher MFI melted polymer has a lower



Fig. 6 PC welding seams generated at 40 mm/s and a NA of 0.072 in joined state (left) and top face of the bottom laminate in separated state using a scanning electron microscope (right).

viscosity which fosters a melt ejection around the welding seam. The delinquent border of the welding seam including the surrounding melted material is clearly visible in the connected and in the separated state in Fig. 6. Besides the melt ejection around the welding seam irregular heat degenerated spots are visible in the center of the welding seam at the interface between both joining partners.

The shear strength of the welding seams between PC polymer samples is measured following the testing setup by Roth et al. [19]. Measurements are performed for three laser powers and five samples per parameter each with a speed of 40 mm/s resulting in an average shear strength of 11 MPa. In comparison to an average shear strength of 37 MPa obtained by Roth et al. [19] for the welding of COC and 21 MPa achieved by Mizuguchi et al. [18] for the welding shear strength of PMMA, resulting shear strengths for ultrashort pulse laser welded PC are lower. De Pelsmaeker et al. [22] observed similar results for Thulium laser welding of PC and assign low welding strengths to a favored degradation of the polymer instead of welding and a prevention of the formation of entitlements by an intrinsic high chain stiffness.

#### 3.3 Sealing of microfluidic devices on COC substrates

An application of ultrashort pulse laser welding of transparent polymers is demonstrated by sealing of a microfluidic network depicted in Fig. 7 (left). The microfluidic structure was previously fabricated by ultrashort pulse laser ablation on the surface of a COC substrate as reported by Suriano et al. [23]. The microchannel has a total length of 25 mm, a maximum width and depth of 100 µm and connects two circular reservoirs. Furthermore, we added two vertical microchannel arms which are only connected to the fluidic system on one side. With a top diameter of 1.5 mm and being ablated completely through the 1.5 mm thick COC chip reservoirs, also serve as microfluidic connection ports as already used by Hessler et al. [24] to connect a photolithographic produced micro-fluidic device. The structured sample was clamped and welded to an unmachined COC chip. To ensure defect free welding seams, as required for a successful sealing, both laser power and welding speed are reduced. We assign irregular defects inside of welding seams to material defects and impurities at the surface of one of the welding partners. A reduction of laser power lowers the probability to generate those welding defects, however, requires a corresponding reduction of welding speed. The rectangular welding seam around the microfluidic network shown in Fig. 7 is produced at a NA of 0.072 with a laser power of 260 mW at a welding speed of 1 mm/s. The focal offset  $\Delta z$ , required to generate the heat zone at the interface is set to 325  $\mu$ m. Overall, welding seam length is produced without any welding defects, mold ejection or disruptions.

The welding path completely surrounds the device and seals both the reservoirs and the microfluidic channel. Microfluidic channels are filled with a Rhodamine B solution. A top view of the channel and parallel welding seam is depicted in Fig. 7 (right). Interference patterns outside the sealed area are clearly visible and are used to identify a small air gap between the welded chips. The area inside the sealing is mostly filled by the testing liquid due to capillary forces. The structure of these patterns highlights the successful welding and sealing.

The functionality of the seal is analyzed by a static leakage test, for which one reservoir was connected to a common commercial microfluidic pump while the other was hermetically closed. Once again, Rhodamine B is used as a testing fluid and inserted with a pressure of up to 0.6 MPa for 30 min. without showing any leakage outside the welded area. Please note that the maximum pressure was limited by the microfluidic testing setup and not by the strength of the welding seams.

#### 4. Conclusion

In this report, ultrashort pulse laser welding of two transparent polymers is studied. Using a NA of 0.072, welding seams without heat degenerated spots inside the welding seam at the interface between two transparent COC chips can be fabricated. The NA of the focusing setups enables to determine applicable laser power and thus realizable welding seam widths. Besides the welding of COC, we report on the ultrashort pulse laser welding of PC. Using a laser power of up to 1700 mW, a mean welding seam width of up to 160 µm can be achieved at a welding speed of 40 mm/s. To highlight the functionality and to propose a potential application of this welding technology, a microfluidic network on a COC polymer substrate is successfully sealed. The sealing is tested using a fluidic pressure of up to 0.6 MPa without showing any leakage outside the welding seam. These results will foster the application of ultrashort pulse laser welding in the fabrication of LOCs based on transparent polymer substrates.



Fig. 7 Scheme of ultrashort pulse laser welding of transparent polymers for sealing of microfluidic devices (left), top view on a sealed microfluidic device filled with a Rhodamine B solution (right).

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