Welding of PMMA by a femtosecond fiber laser

Annalisa Volpe,[1,2,*] Francesca Di Niso,[1,2] Caterina Gaudiuso,[1,2] Andrea De Rosa,[3] Rebeca Martínez Vázquez,[3] Antonio Ancona,[1] Pietro Mario Lugarà,[1,2] and Roberto Osellame[3]

[1] *Istituto di Fotonica e Nanotecnologie (IFN)-CNR U.O.S. Bari, via Amendola 173, 70126 Bari, Italy*
[2] *Università degli Studi di Bari, Dipartimento Interateneo di Fisica,via Amendola 173, 70126 Bari, Italy*
[3] *Istituto di Fotonica e Nanotecnologie (IFN)-CNR, Piazza Leonardo da Vinci 32, 20133 Milan, Italy*
** annalisa.volpe@ifn.cnr.it*

Abstract: Developing versatile joining techniques to weld transparent materials on a micrometer scale is of great importance in a growing number of applications, especially for the fabrication and assembly of biomedical devices. In this paper, we report on fs-laser microwelding of two transparent layers of polymethyl methacrylate (PMMA) based on nonlinear absorption and localized heat accumulation at high repetition rates. A fiber CPA laser system was used delivering 650-fs pulses at 1030 nm with repetition rates in the MHz regime. The laser-induced modifications produced by the focused beam into the bulk PMMA were firstly investigated, trying to find a suitable set of process parameters generating continuous and localized melting. Results have been evaluated based on existing heat accumulation models. Then, we have successfully laser welded two 1-mm-thick PMMA layers in a lap-joint configuration. Sealing of the sample was demonstrated through static and dynamic leakage tests. This fs-laser micro-welding process does not need any pre-processing of the samples or any intermediate absorbing layer. Furthermore, it offers several advantages compared to other joining techniques, because it prevents contamination and thermal distortion of the samples, thus being extremely interesting for application in direct laser fabrication of microfluidic devices.

OCIS codes: (350.3390) Laser materials processing; (320.7090) Ultrafast lasers; (230.3120) Integrated optics devices; (140.3510) Lasers, fiber; (160.5470) Polymers.

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1. Introduction

Femtosecond laser technology is an effective tool for processing a variety of transparent materials, such as glasses, polymers, and crystals [1]. Fs-laser structuring of transparent materials is finding application in a growing number of fields for the production of photonic and bio-medical devices through the direct fabrication of waveguides [2–4], gratings [5–7], microfluidic channels [8–12], and complete optofluidic devices [13]. Furthermore, based on this technology, novel laser joining techniques of transparent materials have been developed, paving the way towards an easy and versatile integration of devices. Several studies have demonstrated the ability of focused femtosecond laser pulses to weld fused silica [14, 15], borosilicate and/or dissimilar glasses [16–18]. A first demonstration of fs-laser welding of silica glass was achieved by Tamaki et al. [19]. They focused intense femtosecond pulses at relatively low repetition rates (< 200 kHz) at the interface of two glass specimen without the insertion of any intermediate light-absorbing layer. Owing to the very intense laser field, filamentation along the optical axis occurred in the focal region generated by a delicate balance between diffraction caused by plasma formation and self-focusing due to the Kerr effect [20]. The intensity in the filamentary region was high enough to initiate non-linear absorption (multi-photon and tunnel absorption, avalanche ionization), leading to the creation of an electron-ion plasma. Subsequently, localized melting occurred in the focal volume followed by resolidification which resulted in the samples being joined [20].

A different approach to weld transparent materials exploits fs-laser pulses with relatively low pulse energies at much higher repetition rates, towards the MHz regime. Here, the mechanism generating localized melting in the focal volume is heat accumulation. Fs-laser welding of glass substrate in this regime has been reported by several groups [15, 21, 22]. Welding at high frequencies offers several advantages compared to the low repetition rate regime as a much higher processing speed and the possibility of tuning the size of the structural modification by adjusting the writing speed [22].

This novel fs-laser based welding technique could be potentially exploited to join also transparent polymeric materials. In particular, PMMA is a very attractive material for the fabrication of microfluidic devices thanks to its high optical transparency, processability and low cost [23]. Boglea et al. [24] and Acherjee et al. [25] reported on laser transmission welding of PMMA by interposing an opaque layer between the two transparent substrates. The heat generated at the interface by the absorption of the laser in the opaque material, propagates towards the transparent layer, thus melting and joining the two PMMA slides. So far laser lap welding of two PMMA samples without any additional layer was only achieved using a thulium doped fiber laser, emitting continuous wave radiation at a wavelength of 2 μm [26]. At this wavelength PMMA is not transparent and therefore the welding is achieved by laser melting the whole thickness of the two slides. This implies the deposition of a large amount of energy that strongly deforms the material surface and volume around the welded region.

In this paper, we report on a novel approach for welding two PMMA transparent layers based on non-linear absorption and heat accumulation using a 1030 nm femtosecond fiber laser at high repetition rates. The nonlinear absorption process, at the basis of the material modification, allows a localized energy deposition that affects only a very small region around the welding line, leaving the rest of the sample intact. Firstly, the laser-induced modifications are investigated with single and multiple line scanning in the volume of the bulk material. This allowed the identification of the appropriate processing window to achieve homogeneous and continuous melting. Experimental data are compared with simulations of heat accumulation using a simple model. Then, results of laser lap welding of two 1-mm-thick PMMA slides are presented. Finally, this technique is applied to seal a fs-laser-fabricated microfluidic channel. Sealing of the weld is demonstrated by leakage tests under controlled pressure.

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2. Experimental setup

We used an ultrafast fiber laser amplifier from Active Fiber Systems GmbH based on the chirped pulse amplification technique (CPA). The laser source delivers an almost diffraction limited beam (M^2~1.25) at a wavelength of 1030 nm with pulse duration in the range from 650 fs to 20 ps, repetition rate varying from 50 kHz to 20 MHz, maximum pulse energy of 100 μJ or maximum average power of 50 W.

In these experiments we operated the laser system at the shortest pulse duration of 650 fs, at a fixed repetition rate of 5 MHz. The laser micro-machining setup also included a Galilean beam expander and a quarter-wave-plate to convert the linear polarization of the laser beam into circular polarization. The laser beam was then focused into the sample through a 0.3 numerical aperture lens. The focusing lens was mounted on a computer controlled motorized axis (Aerotech Pro115) enabling to precisely position the beam focus inside the bulk of the PMMA work-pieces which were fixed on a XY motorized translation table (Aerotech Pro165LM) with micrometer resolution.

High-purity optical-quality PMMA samples (Vistacryl CQ, Vista Optics Ltd) with standard surface quality (measured roughness R_a<5nm) were used for our experiments without any pretreatment. The laser modification tests in bulk material were performed on 3-mm-thick specimens, while the welding experiments were carried out on two overlapped 1-mm-thick PMMA plates. In the latter case the samples were clamped together by applying a moderate pressure to ensure a strict and uniform adherence between the two plates over the irradiated area.

After laser irradiation, the laser-induced modifications and the bonding area were analyzed through optical microscopy. The quality of the bonding was probed with a static leakage tests under increasing pressure [27]. A through hole was laser-drilled in one of the two PMMA slides before welding. This formed a dead-end reservoir that was connected to a microfluidic pump (MFCS-EZ, Fluigent) by a plastic access port glued to the welded sample. The test was performed by injecting a blue-colored fluid at increasing pressures, from 100 mbar to 1 bar with 100 mbar steps. Each pressure value was maintained for 3 minutes while the sealing was analyzed under the microscope to monitor if a fluid leakage was present (see Fig. 1).

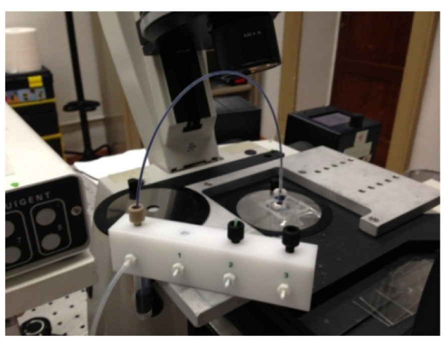

Fig. 1. Picture of a chip connected to the microfluidic pumps for the static leakage tests.

Besides testing the sealing on a single reservoir, the same test was also performed on a complete microchannel connecting two reservoirs, which is the fundamental building block of any microfluidic device. Again, one reservoir was connected to the micropump while the other reservoir was closed. Fluid at increasing pressure was injected in the microchannel following the above protocol and possible liquid leakage around the microchannel was monitored.

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3. Results and discussion

3.1 Laser bulk modification

The type of modifications induced into the bulk PMMA by focused femtosecond laser pulses have been studied varying the pulse energy and the translation speed determining the spatial overlap between consecutive pulses. The laser beam was focused at a depth of 1 mm beneath the surface of 3-mm-thick samples. We firstly produced single line modifications. Then, several parallel lines were scribed aiming to machine larger areas and evaluate the effect of the side distance between the lines on the overall modification.

Figure 2 shows the top view of single line modifications produced at a translation speed of 0.1 mm/s, and different pulse energies ranging from 0.2 μJ to 0.5 μJ. All along the lines, discontinuities of the laser-induced modifications are noticed, accompanied by the appearance of dark spots. The lower the pulse energy the larger the gap between subsequent modifications. When increasing the pulse energy the line modifications become more homogeneous. At the same time the occurrence of dark spots is reduced.

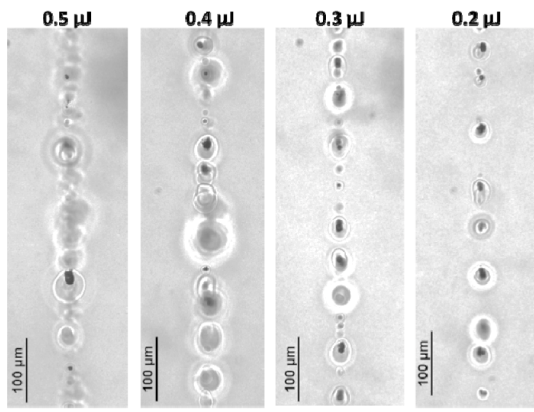

Fig. 2. Optical microscope images (top view) of single line modifications with different pulse energies (rep. rate: 5MHz; translation speed: 0.1mm/s).

From the optical microscope inspection of the cross-sections of the laser irradiated zones (Fig. 3), it can be noticed that the higher the incident pulse energy, the more extended is the laser-induced modification. Above 0.4 μJ of pulse energy, the line modifications exhibit only small discontinuities (see Fig. 2) with transversal dimension longer than 300 μm. By further increasing the laser pulse energy several cracks occurred, propagating towards the sample surface, probably originated by an accumulation of laser-induced defects and/or stresses.

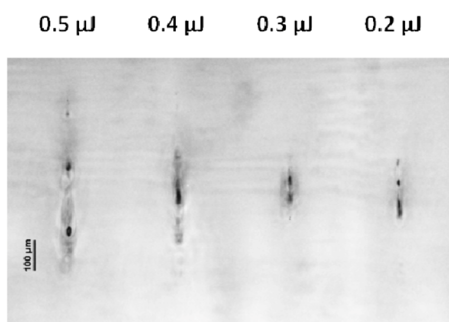

Fig. 3. Optical microscope image of modified tracks observed along the propagation axis of laser pulses at different pulse energy (rep. rate: 5MHz; translation speed: 0.1mm/s).

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The mechanism underlying the generation of the dark spots observed in Figs. 2 and 3 is beyond the scope of this paper. However, these laser-induced damages are typically attributed to ultrahigh pressure waves and micro-explosions that occur within the focal volume leading to the formation of empty voids [28, 29]. This kind of ultrafast light-matter interaction phenomena have been experimentally investigated by Gamaly et al. in different transparent materials (sapphire, glass, polymer) and have been described as follows [30]. Fast deposition of a high energy density in a small and confined focal volume originates melting of the material and the formation of a plasma which further increases absorption of laser light. The subsequent plasma expansion generates a strong shock wave, which propagates into the surrounding volume causing compression of the material at the wave front and decompression on the trailing edge. As a result, during solidification of the melted zone the central region might not be completely filled, leading to the formation of voids buried in the bulk material. We attribute the dark spots observed in our samples to such a mechanism of voids formation, while the lighter modifications consist of resolidified molten material.

In addition to the pulse energy, we have also investigated the influence of translation speed on the type of laser-induced material modification. A series of tests have been performed at a constant pulse energy of 0.4 μJ and variable speed, ranging in the set 0.01 mm/s, 0.025 mm/s, 0.05 mm/s, 0.1 mm/s, 1 mm/s and 10 mm/s. For translation speeds of 1 mm/s and 10 mm/s, no modification of the material was noticed under our experimental conditions. Figure 4 shows the results obtained at relatively low translation speeds, from 0.01 mm/s to 0.1 mm/s. At these speeds, the increased amount of energy deposited per unit volume, together with a larger spatial overlap between subsequent laser pulses, originate significant melting of the material. The lower the speed, the larger the melted area, as revealed by the images shown in Fig. 4.

For the following experiments aiming at modifying larger areas by multiple line scanning we have selected the highest speed (0.1 mm/s) still producing an almost continuous melt of the material. Figure 5 illustrates the machined areas obtained for three different spacing between parallel lines: 50 μm, 25 μm and 10 μm, respectively. The number of voids is clearly reduced with the spacing between lines. This can be explained by assuming that the voids, produced by each laser passage, are filled with the molten material generated by the next laser line. The compressing shock wave accompanying the laser-induced modification helps this filling mechanism. Furthermore, subsequent laser passages may facilitate the release of any mechanical stress generated by the previous ones. We found that the voids completely disappear when the lateral overlap between parallel lines was smaller than 15 μm, as shown in Fig. 5.

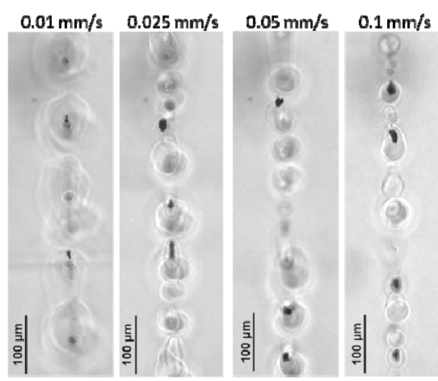

Fig. 4. Top view on the laser induced modifications in PMMA for different translation speed (rep. rate: 5MHz; pulse energy: 0.4 μJ).

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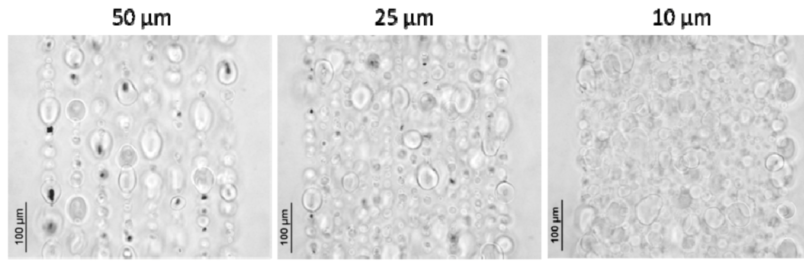

Fig. 5. Microscope images (top view) of the laser induced modification at different spacing between consecutive lines (rep. rate: 5 MHz; pulse energy: 0.4 µJ). The scanning order of subsequent lines goes from right to left.

3.2 Heat accumulation simulation

It is assumed that melting of PMMA in the laser focal volume was originated by a heat cumulative process, since we have worked at relatively low pulse energies and high repetition rates. This hypothesis is also justified by the fact that modifications of the material have been observed only at translation speeds below 1 mm/s, where the spatial overlap between consecutive laser pulses is extremely high. Therefore, we interpreted our experimental observations with a simple thermal model based on the heat diffusion equations that describe the pulsed-laser irradiation of a semi-infinite substrate, with finite absorption A, temperature-independent parameters, and with a Gaussian beam. For times t much longer than the pulse duration τ, as in our case, the cooling cycle after laser irradiation can be approximated by the following analytical expression [31, 32]:

$$\Delta T = \frac{I_a w_0^2 \tau}{2*4\sqrt{\pi} kt \left(Dt\right)^{1/2}}. \qquad (1)$$

where $I_a = A \cdot I$ is the averaged absorbed laser light intensity, w_0 is the radius of the laser focal spot, k is the thermal conductivity and D is the heat diffusivity of the material. Differently from [31], describing a point surface irradiation, Eq. (1) has been divided by a factor of two taking into account that in our case the heat source is located inside the material allowing heat flow into a full solid angle [33].

Figure 6 reports the temperature cooling cycle calculated starting from Eq. (1) of a static laser irradiated point during the first 15 pulses. We assumed a thermal conductivity $k = 0.19$ W m^{-1} K^{-1} and a thermal diffusivity $D = 1.09 \cdot 10^{-7}$ m^2 s^{-1} for PMMA [34]. The absorption coefficient $A = 8.9 \cdot 10^{-4}$ of the laser light has been directly estimated through transmission measurements. The dashed line indicates the temperature increase of the focal volume caused by heat accumulation. Here, the initial sample temperature has been considered to be 25°C. Each laser pulse raises the substrate temperature of a quantity $\Delta T_1 = 4.6 \times 10^{-3}$ °C, calculated by Eq. (1), every time interval $\Delta t = 1/\nu$, where ν is the laser repetition rate of 5 MHz.

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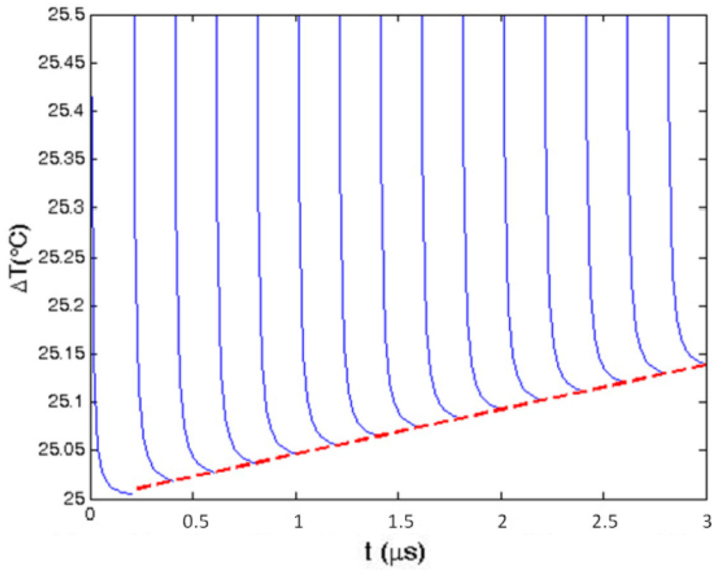

Fig. 6. Simulation of the temperature evolution of the laser irradiated volume in a PMMA sample at 5 MHz of repetition rate and 0.4 μJ of laser pulse energy. The red dashed line describes heating due to heat accumulation.

However, Eq. (1) and the simulation results shown in Fig. 6 refer to the ideal case of a static laser irradiation. In order to take into account the displacement of the sample at a translation speed V, we have estimated the number of pulses N_{lap} overlapping in the same focal spot area as follows:

$$N_{lap} = \frac{2w_0\nu}{V}. \quad (2)$$

The temperature increase ΔT_{HA} of the irradiated focal volume due to heat accumulation can then be calculated by multiplying the quantity ΔT_1 for N_{lap} and is represented in Fig. 7 as a function of the translation speed. For $V < 0.56$ mm/s melting of the focal volume is expected, in fair agreement with our experimental data that showed a modification threshold between 0.1 mm/s and 1 mm/s.

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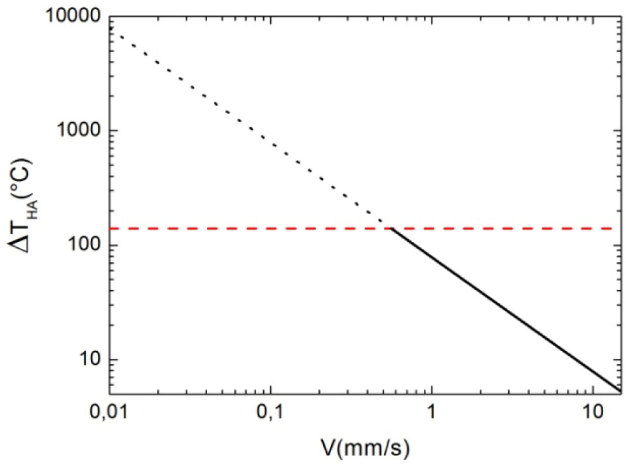

Fig. 7. Calculated temperature increase due to the heat accumulation effect versus translation speed, as predicted by the model described by Eqs. (1) and (2). The PMMA melting threshold (T_{melt} = 160 °C [35]) is highlighted by the red dashed line. The predicted temperature value is reliable for $T<T_{melt}$ (black line), while for $T>T_{melt}$ (black dotted line) the model does not provide a realistic temperature estimation because, owing to the phase transformation, the chosen material parameters do not hold anymore.

3.3 Laser lap welding

Based on the results obtained in the bulk material, we selected a suitable set of working parameters producing homogeneous melting in the focal volume to achieve femtosecond laser lap welding of two 1-mm-thick PMMA plates. The two samples were previously clamped in order to achieve an air gap of a few micrometers range in the welding area, namely where interference effects can be observed. The laser source was operated at 5 MHz with a pulse energy of 0.4 μJ and the beam was focused at the interface between the two plates. The translation speed was set at 0.1 mm/s. The welding seam consisted of a square contour of 1 mm side and 60 μm width, which was obtained by moving the laser beam along a square spiral path with a lateral distance between the closely spaced lines of 5 μm. Figure 8 illustrates the microscope image of the fs-laser welded specimen. The laser-machined area does not show any interference fringes all along its path, thus indicating that melting of the material with subsequent resolidification and joining of the two parts was achieved. Interference fringes are only visible in the immediate proximity of the welding seam, which furthermore appears quite homogeneous and almost completely free from cracks and dark spots.

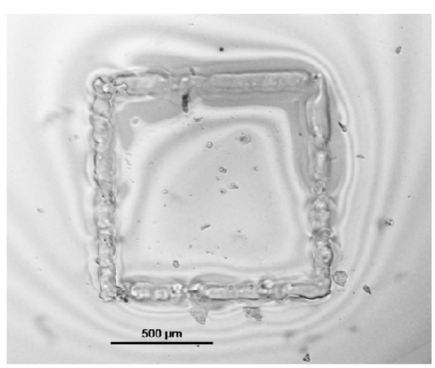

Fig. 8. Microscope image (top view) of a fs-laser lap-welded sample (two overlapped 1-mm-thick PMMA plates) along a square path.

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The sealing properties of the fs-laser lap welding were verified through a static leakage test as described in Section 2. A through hole, with 0.6 mm diameter, was drilled by fs-laser ablation in one PMMA slides. The holey slide was laser-welded to a second standard PMMA slide by a square seam surrounding the hole. This created a dead-end hole, which was connected to a microfluidic pump by gluing a suitable connector on the holey slide (see Fig. 1). By injecting fluid into the dead-end hole at variable pressures, we could test the resistance of the seam. Even at the highest achievable pressure of 1 bar, no leakage of the colored liquid was observed outside the hole and into the gap between the two slides.

3.4 Microfluidic channel sealing and testing

A relevant field of application of PMMA welding is the sealing of microfluidic devices, regardless of the fabrication method employed to produce the microchannel network. In fact, most microfabrication techniques produce open microchannels on the surface of a plastic substrate [36], and thus require subsequent sealing to obtain the final microfluidic network. At present the most used techniques are gluing two layers (either with glue or pressure sensitive thin films) and thermal bonding. Both approaches have disadvantages in terms of channels clogging due to uncontrolled glue diffusion, or deterioration of channel walls and shape due to the applied temperatures and pressures [37].

As a feasibility demonstration of femtosecond laser bonding of PMMA microfluidic networks, we sealed a microfluidic channel previously fabricated by fs laser ablation on the surface of a PMMA slide. The microchannel has a square cross-section with 80µm side and a length of 1 cm, and connects two reservoirs, with 1-mm diameter, that were fs-laser drilled through the whole 1-mm thickness of the PMMA slide [38]. This micro-machined sample was clamped and fs-laser welded to a second standard PMMA slide (Fig. 9(a)). The welding path completely surrounded the device in order to seal both the reservoirs and the micro-fluidic channel. In Fig. 9(b) a detail of the welding seam surrounding the channel and the reservoir is shown. To perform the static leakage test, one reservoir was connected with the microfluidic pump while the other was hermetically closed. A blue liquid was pumped into the microchannel at pressures up to 1 bar observing no leakage from the device. The leakage test was repeated several times. Figure 10 shows a microscope image of the microfluidic channel partially filled with the blue fluid after 5 cycles at the maximum pressure for 3 minutes. It is clearly visible that no liquid leaked outside the microchannel.

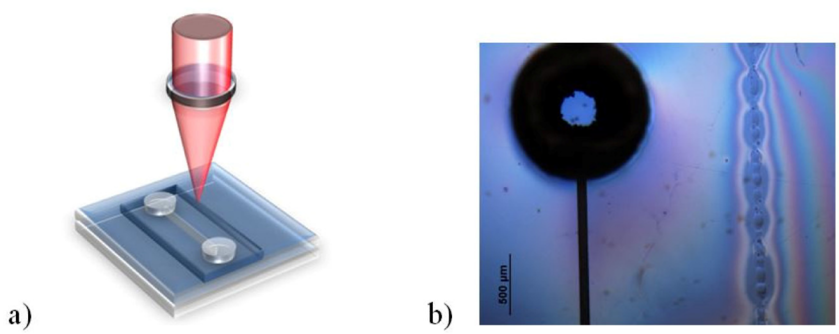

Fig. 9. a) Schematic view of the fs-laser welding process adopted to seal the microfluidic device; b) microscope image (top view) of the welding seam surrounding the microfluidic channel and a reservoir.

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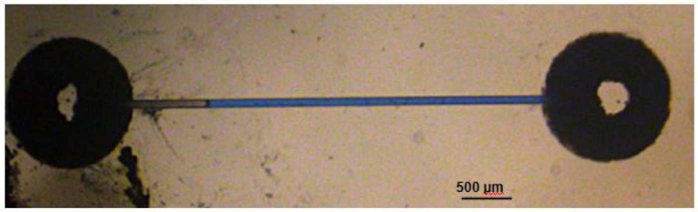

Fig. 10. Microscope image of the microfluidic channel after 5 cycles at 1 bar pressure, showing no leakage of the blue fluid outside of the microchannel.

4. Conclusions

We have demonstrated the feasibility of joining two overlapped transparent PMMA plates by focusing femtosecond laser pulses at the interface. Non-linear absorption of the laser radiation together with heat cumulative processes at high repetition rates and slow translation speeds cause localized melting of the focal volume followed by rapid resolidification and welding.

By using an ultrafast fiber CPA system operating at a wavelength of 1030 nm and a repetition rate of 5 MHz we first investigated the influence of pulse energy and translation speed on the modifications induced in the bulk of a PMMA sample. Single line irradiations produced inhomogeneous modifications characterized by the presence of melted areas interspersed by dark spots probably due to voids formation. We observed that such defects disappear when overlapping several closely spaced parallel irradiation lines, producing a more homogeneous melted area under suitable irradiation conditions. A simple heat accumulation model have been proposed to interpret the main features of our experimental findings.

The identified processing window has been used to weld two 1-mm-thick PMMA layers without any preprocessing of the samples nor any intermediate absorbing layer. A simple microfluidic polymeric device has been assembled taking advantage of this novel joining technique and the effectiveness of the sealing has been proved by a static leakage test with pressures up to 1 bar.

We believe that the present results could pose an important step towards the direct ultrafast laser fabrication and assembly of transparent polymeric microfluidic devices. One of the main advantages of this technique resides in its flexibility to seal channels with complex geometries without introducing potential sources of contamination of the biological samples since it does not employ any chemical additive. In this work we have welded PMMA samples using femtosecond laser pulses nonetheless, being localized melting of the material mainly ascribed to a thermal accumulation process, this novel joining technique could in principle also be effective by employing picosecond laser sources, which would be relevant for a wider industrial impact.

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