

Rapid prototyping of PET microfluidic chips by laser ablation and water-soaking bonding method

Zhifu Yin ✉

School of Mechanical Science and Engineering, Jilin University, Changchun 130012, People's Republic of China

✉ E-mail: yinzf@jlu.edu.cn

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The solution to the commercialisation of polymer microfluidic chips lies in the chosen of a low-cost and concise method. CO₂ laser is an excellent tool for ablating arbitrary-shaped microchannels in most of thermoplastics. However, upheaval area is usually generated along the microchannel due to high temperature during laser ablation process which leads to the difficulty in fully bonding polymer microchannels. In this work, a new bonding method for fully bonding uneven substrates with homogenous surface property was developed based on water-soaking treatment. To prevent edge separating, an extra laser welding on the lateral of the chip was conducted. The influence of laser current on the width and depth of poly(ethylene terephthalate) (PET) microchannel was investigated. The mechanism of water-soaking bonding was explained and the parameters of this method were optimised. The experiments showed that PET chip can be fully bonded without any block or leakage and the bonding strength of PET chip can be as high as 3.4 MPa.

1. Introduction: The last decade witnessed the important development of biological and chemical fields with the employ microfluidic chips [1, 2]. Microfluidic chips are usually fabricated on silicon or SiO₂ substrates, which are costly since they are usually disposable. The demands for economic microfluidic chips stimulated the rapid development of microfluidic chip fabrication by inexpensive material. Nowadays, a shift from silicon and SiO₂ to polymers has carried out [3, 4]. Recently, thermoplastics such as poly(methyl methacrylate) (PMMA), polycarbonate (PC), cyclic olefin copolymer (COC), and poly(ethylene terephthalate) (PET), are attracting attentions for microfluidic chip fabrication due to their low-cost, high transparency, and chemical stability. To fabricate a microfluidic chip by those materials, several well developed microfabrication methods can be employed such as hot embossing [5], injection moulding [6], and micromilling [7]. The above methods, however, are still either complex or low efficiency.

It has been proved that a CO₂ laser is an excellent tool for ablating arbitrary-shaped microchannels in most of thermoplastics. Its low-cost and flexibility makes laser ablation method affordable for volume production of microfluidic chip [8]. Many institutes, laboratories, and companies have already used CO₂ lasers as a basic tool to fabricate microstructures instead of conventional microfabrication methods. For fabrication of high quality microchannels by laser ablation, many works have been carried out. Shaegh *et al.* [9] reported that focused laser beam can fabricate semi-circular microchannels with a diameter of ~300 µm. Zhang and Shin [10] investigated the influence of laser offset on the profile of the microchannels and trapezoidal microchannels can be fabricated with 250 µm width and 250 µm depth. Yan *et al.* [11] proved the speed of laser head can alter the depth of the microchannels without changing its width and deeper microchannels correlate to lower the speed of laser head. Prakash and Kumar [12] analysed beam spot diameters at different work-piece positions. By adjusting the work-piece position, different wide microchannels can be fabricated at a constant laser power. Nowadays, laser ablation has become a well-developed method for fabrication of open microchannels in thermoplastic materials.

To fabricate a complete microfluidic chip, the open microchannels should be sealed. However, upheaval area was usually generated along the microchannel, since high temperature of laser beam makes the material near the edge of the microchannel deformed upwardly [13]. In this case, to fully bond the microchannels,

traditional thermal bonding encounters high microchannel deformation and serious blocking issues due to the adoption of high temperature and pressure [14]. Interlayer bonding method can fully bond the microchannels without the damage of microchannels [15]. However, it gives rise to the inhomogeneity in surface properties of the microchannels, because the foreign material is introduced between the cover layer and the substrate. So upheaval area leads to the difficulty in fully bonding of microchannels with the homogenous surface property. In addition, after microchannels fabrication the flat substrate curves due to the non-uniform temperature distribution during laser ablation. The bonded cover plate and substrate will separate gradually near the edge of the chip because of the high inner stress (we call this edge separating). Up to now, for this issue, there is no suitable solution.

In this work, a bonding method for fully bonding the chips with homogenous material was developed based on water-soaking bonding method. Moreover, edge separating problem was successfully solved by the adoption of laser welding on the lateral of the chip.

2. Experiments: PET, mainly used in the packaging field, has low thermal expansion coefficient, low glass transition temperature (T_g) and good optical transmissivity [16]. In this work, PET sheet with 1 mm thickness, purchased from Gayle Inc Gayle (Burghausen, Germany), was chosen as the substrate material for the microfluidic chip. Fig. 1 schematically shows the process flow for the fabrication of the PET microfluidic chip by laser ablation and water-soaking bonding method.

PET sheet was cut into 2 cm × 3 cm square pieces and the samples were then clean by ultrasound in a cleaner (DZ-1, Jinan Xihua Technologies Co., Ltd, Shandong, China). The clean and baked PET samples were used as a substrate for microchannel fabrication.

As shown in Fig. 1a, the microchannels were ablated into the PET substrate by a conventional laser machine (Liantuo Co., Ltd, Beijing, China). A widely used serpent form microchannel was designed and fabricated in the PET substrate. The width and depth of the microchannel can be changed by adjusting the ablation current. The liquid pools were fabricated in PET cover plate by driller in a separate step as shown in Fig. 1b. PET cover plate with two circular holes was immersed in 40°C water for 24 h to soften its surface layer material. Then the PET substrate with microchannel and PET cover plate were

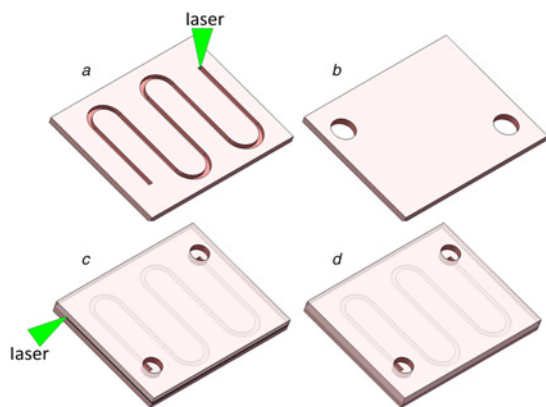


Fig. 1 Illustration of proposed PET microfluidic chip fabrication process
a Laser ablation
b Liquid pools fabrication
c Extra laser welding
d The fabricated micro fluidic chip

bonded together under temperature of 57°C and pressure of 0.2 MPa. Since edge separating always occur after bonded, an extra laser welding on the lateral of the chip was carried out (Fig. 1c). The fabricated microfluidic chip is shown in Fig. 1d.

3. Results and discussion

3.1. Compressive test for T_g estimation: PET is an amorphous thermoplastic material. Its T_g can range from 70 to 150°C [17, 18]. The T_g of PET was not given when we bought the PET sheet. In this work, PET chip was bonded by thermal bonding method after water treatment. It is necessary to estimate the T_g of PET for choosing a suitable bonding temperature during the bonding process. It is known that the strain of PET changes significantly when temperature closes to the T_g of PET under a constant compressive stress. According to this phenomenon, the T_g of PET used in this work can be measured. Fig. 2 shows the strain–temperature curve for PET under a constant stress of 2.5 MPa. The heating speed was 0.03°C/s. One can clearly see that at 72°C the strain increase significantly. It indicates the T_g of PET is about 72°C.

3.2. Influence of laser ablation parameter on the size of the microchannels: For CO₂ laser machine in our lab, only laser current can be changed. So the size of PET microchannel is affected by laser current. The sizes of PET microchannel were measured under different laser current. Fig. 3 shows the profiles of PET microchannels ablated under laser current ranging from 1 to 4 mA. Fig. 4 shows the effect of laser current on the width and depth of PET microchannel. Neither the width nor the depth

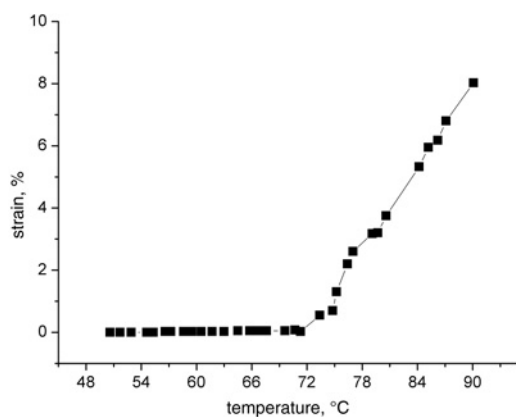


Fig. 2 Compressive test for T_g estimation at different temperatures

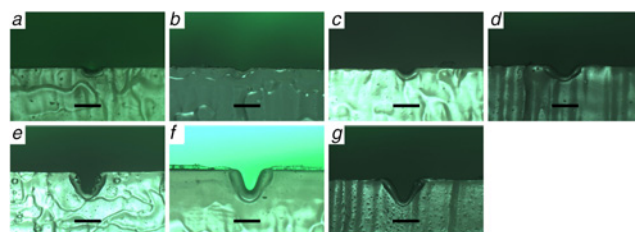


Fig. 3 Profiles of PET microchannels ablated under different laser current
a PET microchannels ablated under laser current of 1 mA
b PET microchannels ablated under laser current of 1.5 mA
c PET microchannels ablated under laser current of 2 mA
d PET microchannels ablated under laser current of 2.5 mA
e PET microchannels ablated under laser current of 3 mA
f PET microchannels ablated under laser current of 3.5 mA
g PET microchannels ablated under laser current of 4 mA

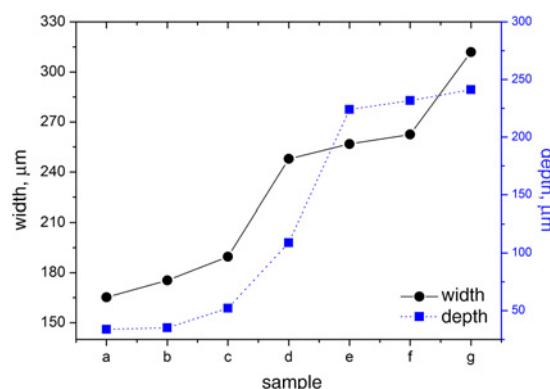


Fig. 4 Effect of laser current on the width and depth of PET microchannel

of PET microchannel follows linear regression based on laser current. For the width, it increases significantly as laser current larger than 2 mA. For the depth, it improves drastically as laser current larger than 2.5 mA. The smallest width and depth for PET microchannel fabricated by laser ablation is about 165 and 34 μm. From the profiles in Fig. 3, we can see that upheaval area generates near the edge of PET microchannel. This can result in the difficulty of fully bonding PET microchannel by traditional thermal bonding method. So in this work, a new bonding method based on water-soaking bonding method was proposed.

3.3. Mechanism of water-soaking bonding method: After microchannel fabrication in the PET substrate, PET cover plate with two liquid pools was immersed in water for 24 h. Then the PET substrate and PET was covered were thermal bonded together. It should be noted that our developed bonding method is different from the widely used solvent assist bonding method. Solvent assist bonding method is based on the dissolve of the polymer in a suitable solvent such as ethanol, isopropanol, or methanol. The solubility parameter of PET is 23 (MJ/m³)^{0.5}. For water it is 47 (MJ/m³)^{0.5}. The solubility parameter of water is much larger than that of PET. According to the solubility principle [19], water cannot dissolve PET even at high temperature. Our bonding method is not solvent assist bonding method at all.

The mechanism of water-soaking bonding method can be explained as follows. PET consists of randomly twined macromolecules with long molecular chains. Microscopically, space exists between PET molecules. While hydrone is a kind of micromolecule with two oxygen atoms and one hydrogen atom. So hydrone can easily go into the space between PET molecules. The molecular mobility of PET is thus increased making Young's modulus of

PET in surface layer decreased [20]. Young's modulus of water soaked PET can be expressed as [21]

$$E_p = \alpha_p \times H_r + \beta_p \times \log(\varepsilon_p) + C_p \quad (1)$$

where E_p is Young's modulus of PET. H_r is the relative humidity. When PET soaks water, H_r can be changed. ε_p is the strain rate of PET. α_p , β_p , and C_p are the material constants for PET.

If a strain rate is a constant value, Young's modulus of PET follows a simple linear regression based on humidity. Lower Young's modulus correlates to higher humidity.

Young modulus of PET before and after water treatment was measured by a Nano-Indentation System (America, 100BA-1C). Fig. 5 shows the measurement results. For water-soaking method, PET sample was immersed in water for 24 h. The result indicates that Young modulus of PET really decreases when it soaks water. However, only Young modulus of the material in PET surface layer can decrease. The thickness of this layer is about 450 nm. It indicates that only the surface layer of PET sample can soak water at the immersing duration of 24 h. PET layer with low Young modulus can serve as a 'glue' the during bonding process. The uneven PET substrate can also be fully bonded by the reflow of the 'glue'. However, the microchannels will not block, since the thickness of the 'glue' is significantly smaller than the size of the microchannel. PET microchannels in surface properties can also be uniform, because water-soaking is only a physical process without chemical reaction.

3.4. Bonding of PET microfluidic chip: After water-soaking, the PET substrate with the microchannel and PET cover plate with two liquid pools were thermal bonded together. The bonding quality is usually evaluated by the bonding rate (the ratio of the bonding area over the whole area of the chip) [22, 23]. For thermal bonding, there are three factors which can influence bonding rate: bonding temperature, bonding pressure, and bonding duration. To fully bond the chips, those three parameters should be optimised.

Near or above T_g , the polymer can easily deform under low pressure, since polymer goes into a rubbery state [24]. To avoid damage of patterned microchannels, bonding process must be conducted below the T_g of PET (always $T_g - 15^\circ\text{C}$). In this work, the bonding temperature of 57°C was chosen. So the bonding rate is only influenced by bonding pressure and duration.

Fig. 6a shows the bonding rate as a function of bonding pressure with bonding temperature of 57°C and duration of 5 min. One can see bonding rate increases with an increase of bonding pressure. From bonding pressure of 0.2–0.4 MPa, bonding rate increase

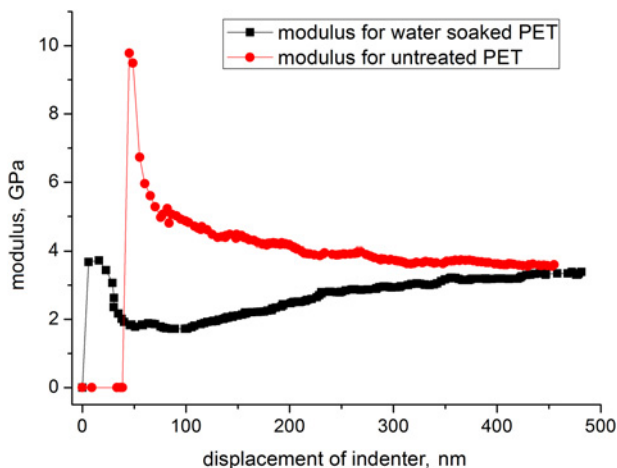


Fig. 5 Young modulus of PET before and after water treatment

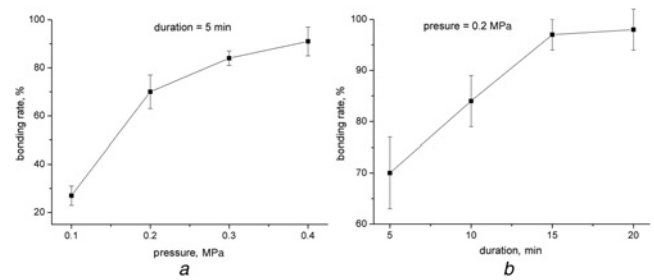


Fig. 6 Influence of bonding parameters on the bonding rate
a Influence of bonding pressure on the bonding rate
b Influence of bonding duration on the bonding rate

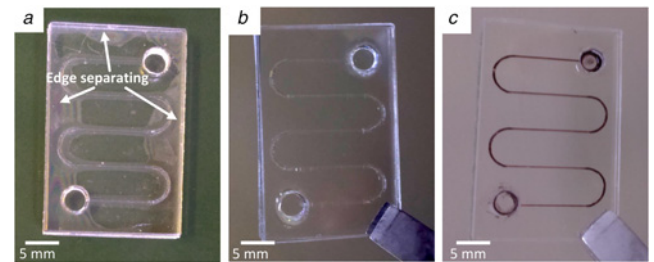


Fig. 7 Photographs of PET microfluidic chips
a Chip with edge separating after bonding
b Chip treated with extra laser welding on its lateral
c Sample filling for the leakage test

significantly (increase by 43%). However, as bonding pressure continuously increases, bonding rate can only increase by ~10%. It is verified higher deformation of the channels correlates to higher bonding pressure [25]. To avoid the damage of PET microchannels, we chose 0.2 MPa as the suitable bonding pressure. Fig. 6b shows the bonding rate as a function of bonding duration with bonding temperature of 57°C and pressure of 0.2 MPa. It is obvious when bonding duration is larger than 15 min, PET chip can nearly fully bonded. Therefore, we chose 15 min as optimised bonding duration.

According to the above discussion, the suitable bonding parameters are bonding temperature of 72°C , bonding pressure of 0.2 MPa, and bonding duration of 15 min. Under those parameters, the chips can be almost fully boned. However, we found that several days later (5–10 days), the edge area in bonded PET chip ruptured from the interface between PET cover plate and substrate (Fig. 7a). We call this edge separating. To solve this problem, we conducted an extra laser welding. After thermal bonding PET chip, a laser was

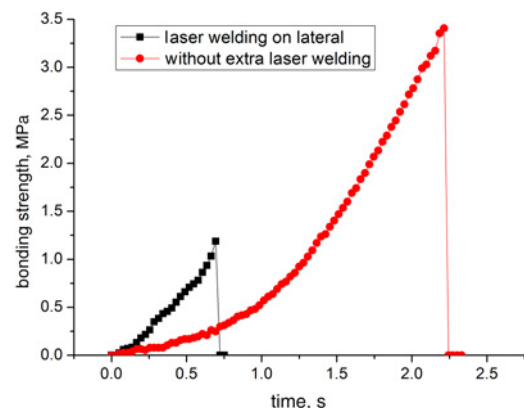


Fig. 8 Bonding strength of PET chips

focused on the lateral of the chip. Then the laser moved along the interface (PET cover plate–PET substrate) making the material near the interface melted. The laser current we chose is the highest current value of 5 mA. At such high laser current, a mass of material near the interface can melt. After cooled, the interface can completely ‘glue’ together. Edge separating cannot occur after 20 days (Fig. 7b). To compare the bonding strength before and after extra laser welding, tensile tests were conducted by an electric stretcher (500TV-SL, Jinlihao electronic tools Co., Ltd, China) (Fig. 8). It indicates that with an extra laser welding the bonding strength can significantly increase by 280%. Tested by sample filling, there was no leakage and block in the entire microchannel (Fig. 7c).

4. Conclusion: In this paper, a bonding method based on water-soaking treatment was developed. The effect of laser current on the width and depth of PET microchannel was studied by experiments. To fully bond PET chip, bonding parameters were optimised. After bonding of PET chip, an extra laser welding process on its lateral was carried out to avoid edge separating in the interface between PET cover plate and substrate. The sample filling and tensile stretching tests demonstrated the absence of leakage over entire PET microchannels and the high bonding strength of 3.4 MPa.

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6 References

- [1] Abgrall P., Gue A.M.: ‘Lab-on-chip technologies: making a microfluidic network and coupling it into a complete microsystem – a review’, *J. Micromech. Microeng.*, 2007, **17**, (5), pp. R15–R49
- [2] Watanabe T., Sassa F., Yoshizumi Y., *ET AL.*: ‘Review of microfluidic devices for on-chip chemical sensing’, *Electron. Commun. Jpn.*, 2017, **100**, (4), pp. 25–32
- [3] Vella S.J., Beattie P., Cademartiri R., *ET AL.*: ‘Measuring markers of liver function using a micropatterned paper device designed for blood from a fingerstick’, *Anal. Chem.*, 2012, **84**, (6), pp. 2883–2891
- [4] Yin Z., Zou H.: ‘Experimental and numerical study on PDMS collapse for fabrication of micro/nanochannels’, *J. Electr. Eng.-Elektrotech. Cas.*, 2016, **67**, (6), pp. 414–420
- [5] Kuo C.-C., Chiang T.-S.: ‘Development of a precision hot embossing tool with microstructures for microfabrication’, *Int. J. Adv. Manuf. Technol.*, 2017, **91**, (1–4), pp. 1321–1326
- [6] Fu G., Tor S.B., Loh N.H., *ET AL.*: ‘The demolding of powder injection molded micro-structures: analysis, simulation and experiment’, *J. Micromech. Microeng.*, 2008, **18**, (7), p. 075024
- [7] Chen P.-C., Pan C.-W., Lee W.-C., *ET AL.*: ‘An experimental study of micromilling parameters to manufacture microchannels on a PMMA substrate’, *Int. J. Adv. Manuf. Technol.*, 2014, **71**, (9–12), pp. 1623–1630
- [8] Chen X., Shen J., Zhou M.: ‘CO₂ laser micromachining for rapid fabrication of a four layer poly(methyl methacrylate) (PMMA)-based microfluidic diluter’, *Lasers Eng.*, 2017, **38**, (1–2), pp. 57–65
- [9] Shaegh S.A.M., Pourmand A., Nabavina M., *ET AL.*: ‘Rapid prototyping of whole-thermoplastic microfluidics with built-in microvalves using laser ablation and thermal fusion bonding’, *Sens. Actuators B, Chem.*, 2018, **255**, pp. 100–109
- [10] Zhang S., Shin Y.C.: ‘Effective methods for fabricating trapezoidal shape microchannel of arbitrary dimensions on polymethyl methacrylate (PMMA) substrate by a CO₂ laser’, *Int. J. Adv. Manuf. Technol.*, 2017, **93**, (1–4), pp. 1079–1094
- [11] Yan Z., Huang X., Yang C.: ‘Rapid prototyping of single-layer microfluidic PDMS devices with abrupt depth variations under non-clean-room conditions by using laser ablation and UV-curable polymer’, *Microfluid. Nanofluid.*, 2017, **21**, (6), Article No. 108
- [12] Prakash S., Kumar S.: ‘Experimental and theoretical analysis of defocused CO₂ laser microchanneling on PMMA for enhanced surface finish’, *J. Micromech. Microeng.*, 2017, **27**, (2), Article No. 025003
- [13] Liu K., Xiang J., Ai Z., *ET AL.*: ‘PMMA microfluidic chip fabrication using laser ablation and low temperature bonding with OCA film and LOCA’, *Microsyst. Technol.-Micro Nanosyst.-Inf. Storage Process. Syst.*, 2017, **23**, (6), pp. 1937–1942
- [14] Matteucci M., Heiskanen A., Zor K., *ET AL.*: ‘Comparison of ultrasonic welding and thermal bonding for the integration of thin film metal electrodes in injection molded polymeric lab-on-chip systems for electrochemistry’, *Sensors*, 2016, **16**, (11), Article No. 1795
- [15] Yin Z., Cheng E., Zou H.: ‘Fast microfluidic chip fabrication technique by laser erosion and sticky tape assist bonding technique’, *J. Nanosci. Nanotechnol.*, 2018, **18**, (6), pp. 4082–4086
- [16] Chantiwas R., Park S., Soper S.A., *ET AL.*: ‘Flexible fabrication and applications of polymer nanochannels and nanoslits’, *Chem. Soc. Rev.*, 2011, **40**, (7), pp. 3677–3702
- [17] Chih-Yu W.: ‘Replication of polyethylene terephthalate (PET) nano-micro structures using ultrasonic nanoimprint’, 2011
- [18] Liao W.-C.: ‘Acidic acrylic polymers for nanoimprint lithography on flexible substrates’, 2005
- [19] Gu X.Y., Lu Y.: ‘Polymer science foundation’ (Chemical Industry Press, Peking, 2003)
- [20] De Anda A.R., Fillot L.A., Rossi S., *ET AL.*: ‘Influence of the sorption of polar and non-polar solvents on the glass transition temperature of polyamide 6,6 amorphous phase’, *Polym. Eng. Sci.*, 2011, **51**, (11), pp. 2129–2135
- [21] Ishiyama C., Higo Y.: ‘Effects of humidity on Young’s modulus in poly(methyl methacrylate)’, *J. Polym. Sci. B, Polym. Phys.*, 2002, **40**, (5), pp. 460–465
- [22] Yin Z., Sun L., Zou H., *ET AL.*: ‘Two dimensional PMMA nanofluidic device fabricated by hot embossing and oxygen plasma assisted thermal bonding methods’, *Nanotechnology*, 2015, **26**, (21), p. 215302
- [23] Du L., Chang H., Song M., *ET AL.*: ‘A method of water pretreatment to improve the thermal bonding rate of PMMA microfluidic chip’, *Microsyst. Technol.-Micro Nanosyst.-Inf. Storage Process. Syst.*, 2012, **18**, (4), pp. 423–428
- [24] Kim N.W., Kim K.W., Sin H.C.: ‘Finite element analysis of low temperature thermal nanoimprint lithography using a viscoelastic model’, *Microelectron. Eng.*, 2008, **85**, (9), pp. 1858–1865
- [25] Jena R.K., Chester S.A., Srivastava V., *ET AL.*: ‘Large-strain thermo-mechanical behavior of cyclic olefin copolymers: application to hot embossing and thermal bonding for the fabrication of microfluidic devices’, *Sens. Actuators B, Chem.*, 2011, **155**, (1), pp. 93–105