Microelectronic Engineering 113 (2014) 93–97

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Three-dimensional metallic microcomponents achieved in fused silica by a femtosecond-laser-based microsolidifying process

Keyin Liu, Qing Yang *, Yulong Zhao, Feng Chen *, Chao Shan, Shengguan He, Xiaole Fan, Lei li, Xiangwei Meng, Guangqing Du, Hao Bian

State Key Laboratory for Manufacturing System Engineering, Xi'an Jiaotong University, Xi'an 710049, PR China
Key Laboratory of Photonics Technology for Information of Shaanxi Province, Xi'an Jiaotong University, Xi'an 710049, PR China

ARTICLE INFO

Article history:
Received 27 April 2013
Received in revised form 8 July 2013
Accepted 23 July 2013
Available online 2 August 2013

Keywords:
3D metallic microstructures
Femtosecond laser
Microsolidifying
Liquid metal

ABSTRACT

Three-dimensional (3D) metallic microdevices have attracted a wide attention in the field of functional microsystems, but the fabrication of 3D metallic structures remains a technical challenge. In this study, a femtosecond-laser-based microsolidifying method was employed to fabricate 3D metallic structures by injecting liquid metal into complex 3D microchannels/cavities in fused silica and solidifying the liquid metal. 3D microchannels/cavities, which were served as micromoulds of the metallic microcomponents, were fabricated in fused silica by taking full advantage of the improved femtosecond laser irradiation followed by chemical etching (FLICE) technology. A PDMS-glass injection device was employed to finish the injection of liquid metal and solidification process. This technology will enable the maskless and facile fabrication of complex true-3D metallic conductive microcomponents for a wide array of micro-applications.

1. Introduction

Metallic microcomponents are significant for most microsystems, including integrated circuit (IC), microfluidic systems and microelectromechanical systems (MEMS) [1–5]. As three-dimensional (3D) metallic devices enable more efficient and flexible electric/electromagnetic conduction and manipulation than planar ones, metallic microcomponents with complex 3D structures have been used in a wide range of functional microsystems, such as high-sensitivity microsensors, on-chip nuclear magnetic resonance (NMR) [6–10].

Although mass efforts have been taken towards the creation of 3D metallic microstructures, it is still a technical challenge to achieve intricate 3D metallic conductive microstructures by the reported methods. The conventional fabricating method of metallic conductive microdevices usually involves a tedious and expensive lithographic process which has been mostly used to provide planar copper (Cu) microwires [2,3,6] or multilayer metallic microstructures [5]. The LIGA (Lithographie, Galvanoformung and Abformung) technology [8] can achieve more complicated metallic microdevices by utilizing deep X-ray lithography, but owns limited processing capability of 3D metallic microstructures. 3D replication methods have been also reported to fabricate 3D microcoils [9,10], but are accompanied with complex fabrication procedures and only simple straight microcoils have been reported. A femtosecond-laser-induced electroless plating technology has been reported to fabricate 3D metal nanowires on nonplanar substrates [11]; however, intricate 3D metal structures are still hard to be fabricated for the configuration of the metal nanowires is decided according to the upper surface of the substrates. A microsolidics technology has been reported to fabricate metallic microstructures in polymer materials (polydimethylsiloxane, PDMS) [12], and the metal forming process involved in the technology enhances the flexible fabrication of metallic microstructures by employing liquid metal. The injection moulding method provides a useful approach to the fabrication of metallic microstructures [12,13], but is restricted by the lithographic process to fabricate intricate micromoulds in 3D.

Recently, the femtosecond laser has been used to fabricate 3D microstructures in glassy materials [14–17], and the technology of femtosecond laser irradiation followed by chemical etching (FLICE) has been the primary method to fabricate 3D microcavities/microchannels in fused silica. In this paper, a facile method of combining the FLICE technology and the microsolidifying technique utilizing low-melting-point metal was proposed to fabricate intricate 3D metallic conductive microcomponents in fused silica. 3D microcavities/microchannels in fused silica, which were served as the micromoulds, were achieved by taking full advantage of the improved FLICE technology. A PDMS-glass injection device was

* Corresponding authors. Tel.: +86 29 83395171 (Q. Yang), tel.: +86 29 82668420 (F. Chen).
E-mail addresses: yangqing@mail.xjtu.edu.cn (Q. Yang), chenfeng@mail.xjtu.edu.cn (F. Chen).

0167-9317/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.mee.2013.07.017

employed to finish the microsolidifying process. The liquid gallium metal (Ga, mp ≈ 30 °C) was introduced into the fabricated 3D microchannels by applying a vacuum; and solid metallic micro-components including microwires, microelectrodes and microcoils were achieved after a cooling process. As the improved FLICE technology is theoretically free to fabricate arbitrary 3D microcavities/microchannels in fused silica, flexible and rapid fabrication of 3D metallic microdevices which is particularly compatible with microfluidic can be achieved by the femtosecond-laser-based microsolidifying method.

2. Methods development

2.1. Fabrication of 3D micromoulds in fused silica

3D microcavities in fused silica, which were served as the micromoulds, were fabricated by the selective etching of fused silica after femtosecond laser irradiations. Methods of fabricating complex 3D microstructures with extra accesses and laser power compensation were employed to improve the processing capability, as shown in Fig. 1(a). The femtosecond laser micromachining system consisted of a femtosecond laser source (wavelength: 800 nm, pulse duration: 50 fs, repetition rate: 1 kHz), a microscope objective, a programmable three-axis stage, a CCD camera and a laser beam control system. During the laser irradiation process, the bulk fused silica ($10 \times 10 \times 0.8\ mm^3$) was fixed on the three-axis stage; and the designed patterns were written in fused silica with femtosecond laser pulses by translating the bulk fused silica along the programmed pattern path. The chemical etching process was carried out in hydrofluoric (HF) acid solution assisted by the ultrasonic bath. The processing parameters were kept constant as: scanning speed of 10 $\mu m\ s^{-1}$, circular polarization laser and HF solution concentration of 10%.

2.2. Injection of liquid metal and formation the solid metallic microstructures

A microfluidic-compatible injection device was employed to inject liquid metal into the 3D micromoulds. The injection device consisted of a fabricated fused silica chip, a cover of PDMS substrate, and steel tubes which can be used to introduce both liquid objects and electric power into the microsystem, as shown in Fig. 1(b) and (d). Typically, the liquid objects include biochemical

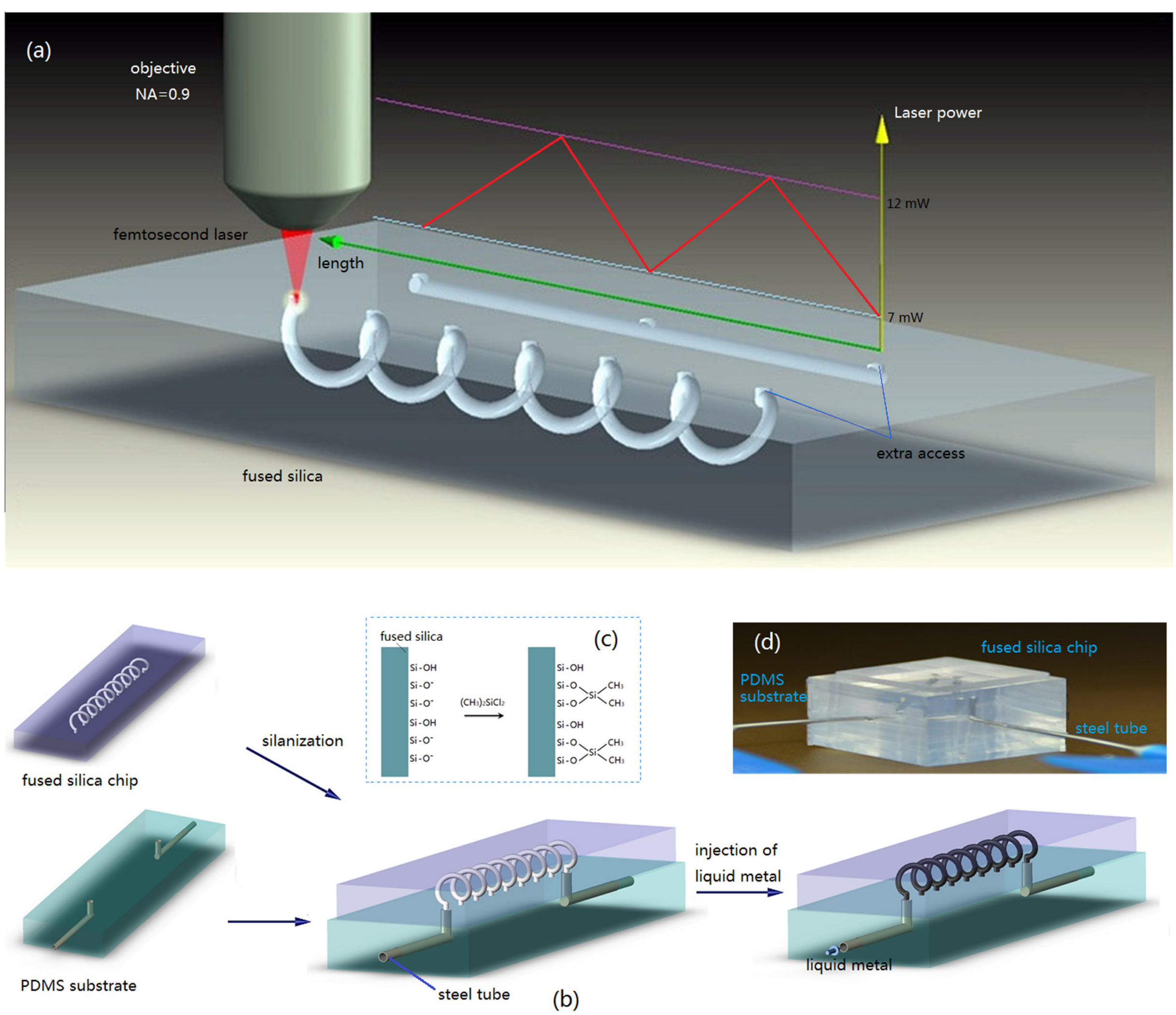

Fig. 1. Schematic illustration of the femtosecond-laser-based microsolidifying process: (a) the improved FLICE technology of writing complex 3D microstructures with extra accesses and laser power compensation, (b) illustration of the microsolidifying process and the microfluidic-compatible injection device, (c) the silanization process of the inside surface of the microchannels, and (d) the picture of the injection device.

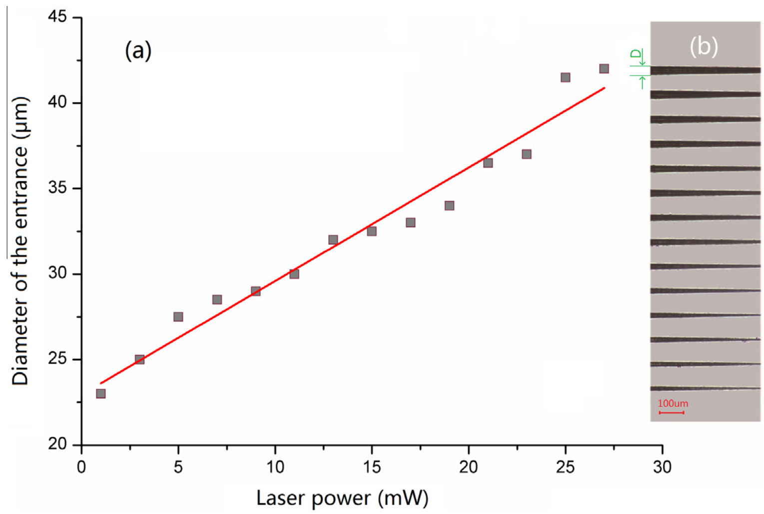

Fig. 2. The dependency relationship of the femtosecond laser power and the microchannel diameter, after HF acid etching for 60 min: (a) diameters of the microchannel entrances fabricated by laser power of different levels and (b) the image of the fabricated microchannels.

solutions for the assay process in microfluidic applications and liquid metal for fabricating the metallic microstructures.

In the experiment, the metal gallium was used to form the 3D metallic conductive microstructures in the micromoulds. An infrared heat lamp was used to melt the metal gallium and maintain the temperature at 45 °C during the injection process. A source of negative pressure was applied to the outlets of the microfluidic channels by connecting them to a syringe; and the liquid gallium was pulled through the microchannels rapidly (<1 s). After filling the channels with gallium, the device was removed from the infrared heat lamp and cooled in the icebox (−1 °C). The liquid gallium inside the microcavities/microchannels was then solidified into solid metal structures.

To facilitate the injection of liquid gallium into the micromoulds, a silanization process to decrease the free energy of the metal–silica interface of the channels was employed before the injection of gallium, as illustrated in Fig. 1(c). Dimethylchlorosilane solution in methanol (6%) was introduced into the 3D micromoulds with the injection device, and the device was stored at 25 °C until the solution had emptied in the microchannels.

3. Results and discussion

3.1. Enhancement of the FLICE processing capability

The microchannels of millimetres fabricated by the traditional FLICE technology are usually strongly tapered that a conical geometry in the microchannel is unavoidable, and the FLICE technology is generally used to provide microchannels in fused silica of less than a few millimetres [16,17]. To address this problem, methods of writing complex 3D microstructures with extra accesses and laser power compensation were employed to improve the processing capability, as illustrated in Fig. 1(a).

The extra accesses acted as extra entrances for the etching solution during the etching process. The amount and distribution of the extra access was designed according to the geometry of the

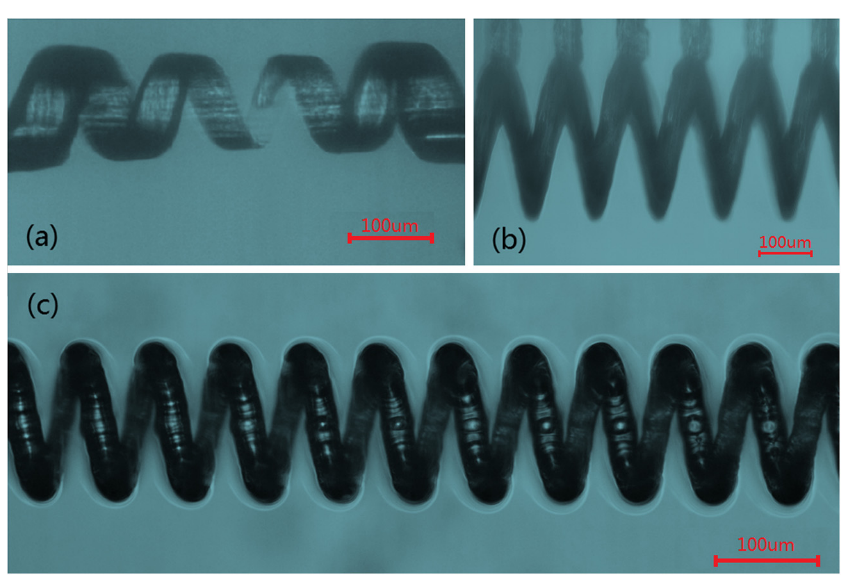

Fig. 3. Fabricating results of helical microchannels in fused silica: (a) by the traditional FLICE technology, (b) the extra access at the helical microchannel, and (c) by the improved FLICE technology of writing complex 3D microstructures with extra accesses and laser power compensation.

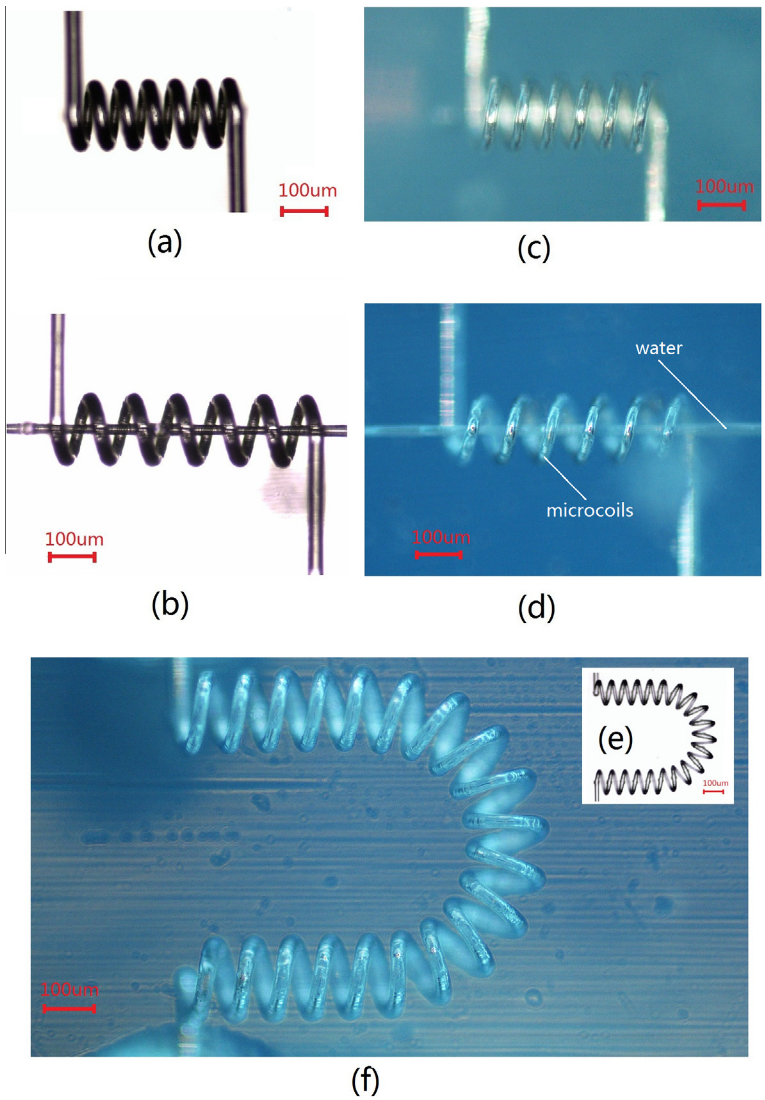

Fig. 4. Solid metallic conductive microstructures fabricated by the femtosecond-laser-based microsolidifying method: (a, c) simple helical microchannels and microcoils, (b, d) microchannels of intricate crossbridge structure and straight microcoils with a central microchannel filled with water, and (e, f) U shape helical microchannels and microcoils.

micromoulds. This method actually divided the fabrication of bulk microfluidic chips by FLICE technology into several smaller parts, and each part could be etched by HF solution simultaneously.

A fabricating strategy of tuning laser power during the laser irradiation process was used to improve the microchannel geometry quality. According to our previous study [18], the diameter of microchannels is increased with laser power. Dependency relationship of the femtosecond laser power and the microchannel diameter was investigated. Laser power ranging from 1 to 27 mW was utilized to fabricate straight microchannels in fused silica, and the diameters of the microchannel entrances were measured after HF acid etching for 60 min. A linear increase of the microchannel diameter with the increasing laser power was testified, as shown in Fig. 2. In the experiment, the laser power was modified linearly from 7 mW at the entrances/extra access to 12 mW in the middle of two adjacent entrances by a computer-controlled attenuator to compensate the conical geometry in the microchannel, as shown in Fig. 1(a).

Complex 3D microchannels of uniform diameter were achieved by the improved FLICE technology. Fig. 3 shows the fabricating results of helical microchannels in fused silica by the traditional and improved FLICE technologies. An extra access was designed at every coil of the helical microchannel by the improved FLICE technology, as shown in Fig. 3(b). Without the method of employing extra accesses and laser power compensation, helical microchannels of more than three coils can hardly be realized, as shown in Fig. 3(a). Meanwhile, the conical geometry of the microchannels was acute. By combing writing complex 3D microstructures with extra accesses and laser power compensation, helical microchannels of arbitrary length and uniform sections were achieved, as shown in Fig. 3(c). Thus, the improved FLICE technology can be used to provide more complex microchannels whose configurations are decided by programming the pattern in 3D.

3.2. Fabrication of 3D metallic conductive microstructures

For microchannels with a diameter >35 μm, the liquid gallium can easily fill the microfluidic channels completely; nonuniform wetting of the microfluidic channels was rarely observed. However, liquid gallium in narrower microchannels (diameter <30 μm) was often accompanied with nonuniform wetting points, and sometimes the liquid gallium in the microchannels was separated as disconnected parts. As the diameter of the microchannels decreased to 20 μm, it became even tough to pull the liquid gallium through the microfluidic channels. This phenomenon was caused by the poor micro-scale wetting characteristics of liquid gallium with fused silica.

To improve the wetting characteristics and fabricate more miniaturized metallic devices, silanization of microcavities before the injection of gallium was carried out using dimethylchlorosilane, as described in Fig. 1(c). After this process, the inside surface of the channels was coated with a layer of silane; and the silane decreased the free energy of the metal–silica interface of the channels and made it possible to wet the inside walls with liquid gallium. This process enabled efficient fabrication of metallic structures in microchannels with a narrower diameter (20–30 μm).

Fig. 4 shows the 3D microchannels and the fabricated solid metallic conductive microstructures, including simple microcoils (Fig. 4(c)), straight microcoils with a central microchannel which is filled with water (Fig. 4(d)), and U shape microcoils (Fig. 4(f)). The 3D microcoil is a kind of typical metallic microcomponents that can be used as microinductors for LC or microsensing applications, electromagnets or heating elements; and the 3D microcoils of different geometries and constructions can extend their applications with different functions. The intricate crossbridge structure of the microcoils indicates that the femtosecond laser based micromoulding method can be used to fabricate more complex 3D metallic microdevices.

The 3D metallic microcomponents fabricated by the femtosecond-laser-based microsolidifying technology can be easily integrated into microfluidic systems. Fig. 4(d) shows a microfluidic system consisting of a straight microcoil and a central microchannel along the axis of the microcoil; and deionized water, which can be replaced by biochemical solutions, was easily introduced into the microfluidic system. Position and configuration of the metallic microcomponents is decided during the femtosecond laser irradiation process and inherently aligned with the microfluidic channels. Besides, the microfluidic-compatible micromoulding process enables convenient application of the metallic conductive microcomponents without extra alignment and packaging steps. Biochemical solutions and electric power can be directly applied into the microfluidic system through the steel tubes of the micromoulding device.

4. Conclusion

In conclusion, this paper presents a femtosecond-laser-based microsolidifying technique to fabricate intricate three-dimensional (3D) metallic microcomponents. 3D metallic objects were achieved by solidifying liquid metal in 3D micromoulds embedded in fused silica; and the micromoulds with complex 3D configurations are fabricated by a femtosecond laser micromachining technology. The femtosecond-laser-based microsolidifying method owns several advantages over other techniques to fabricate 3D metallic microstructures. The fabricating procedure is facile and maskless, and makes it possible to co-fabricate microfluidic channels and metallic microcomponents, each with arbitrary configuration. The fused silica applied in the fabricating process owns excellent electrical insulation, biochemical inertia and light transparent properties, and is highly suitable for most micro-applications.

The femtosecond-laser-based microsolidifying method also has some limitations. The improved femtosecond laser irradiation followed by chemical etching (FLICE) technology involved in the fabricating process is accompanied with extra access structure which may have side effects on the metallic microdevices' function or microfluidic performance. However, the side effects of the extra access structure could be further studied, and treatment processes could be taken to eliminate side effects of the extra access structure if needed, such as decreasing the length of extra access or filling the extra access with polymeric materials. Meanwhile, the method cannot be used to inject liquid objects into "dead-end" channels. In addition, the low melting point of Gallium may not meet a wide range of operation temperature of microfluidic applications. However, as a vast kind of low-melting-point metals can be used to form the 3D metallic microstructures [12], this approach will be beneficial for fabricating functional microsystems intergraded with 3D metallic wires, electrodes, electromagnets or heating elements.

Acknowledgements

This work is supported by the National Science Foundation of China under the Grant No. 61176113, the Program for Changjiang Scholars and Innovative Research Team in University (IRT1033) and the Fundamental Research Funds for the Central Universities of China.

References

[1] D.S. Lee, P.J. Chen, G.B. Lee, Biosens. Bioelectron. 25 (2010) 1820–1824.
[2] A.T. Le, W.S. Cho, Y.S. Kim, J.B. Lee, C.O. Kim, H. Lee, Sens. Actuators A 135 (2007) 547–551.
[3] M.G. Allen, IEEE Trans. Magn. 39 (2003) 3073–3078.
[4] M.A.M. Gijs, Microfluid Nanofluid 1 (2004) 22–40.
[5] H.Y. Li, L. Xie, L.G. Ong, A. Baram, I. Herer, A. Hirshberg, et al., IEEE Electron Device Lett. 33 (2012) 432–434.
[6] R. Rong, J.W. Choi, C.H. Ahn, J. Micromech. Microeng. 16 (2006) 2783–2790.
[7] Y.Y. Cao, N. Takeyasu, T. Tanaka, X.M. Duan, S. Kawata, Small 5 (2009) 1144–1148.
[8] C.K. Malek, V. Saile, Microelectron. J. 35 (2004) 131–134.
[9] L. Dongkeon, H. Hiroshi, Z. Yi, I. Toshihiro, M. Ryutaro, Microelectron. Eng. 88 (2011) 2625–2628.
[10] L. Dongkeon, M. Harutaka, H. Hiroshi, M. Sohei, I. Toshihiro, T. Masaharu, M. Ryutaro, Microelectron. Eng. 86 (2009) 920–924.
[11] X. Binbin, X. Hong, N. Ligang, Z. Yonglai, S. Kai, C. Qidai, X. Ying, L. Zhiqiu, L. Zhihong, M. Hiroaki, S. Hongbo, Small 6 (2010) 1762–1766.
[12] A.C. Siegel, D.A. Bruzewicz, D.B. Weibel, G.M. Whitesides, Adv. Mater. 19 (2007) 727–733.
[13] K.M. Bae, J.S. Ko, T.W. Lim, D.Y. Yang, B.S. Shin, H.S. Lee, S.H. Park, Microelectron. Eng. 88 (2011) 3300–3305.
[14] Y. Liao, J. Song, E. Li, Y. Luo, Y. Shen, D. Chen, Lab Chip 12 (2012) 746–749.
[15] C.G.K. Malek, Anal. Bioanal. Chem. 385 (2006) 1362–1396.
[16] V. Maselli, R. Osellame, G. Cerullo, R. Ramponi, P. Laporta, Appl. Phys. Lett. 88 (2006) 191107.
[17] C. Hnatovsky, R.S. Taylor, E. Simova, V.R. Bhardwaj, D.M. Rayner, P.B. Corkum, Opt. Lett. 30 (2005) 1867–1869.
[18] S.G. He, F. Chen, K.Y. Liu, Q. Yang, H.W. Liu, H. Bian, et al., Opt. Lett. 37 (2012) 3825–3827.