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Facile fabrication of true three-dimensional microcoils inside fused silica by a femtosecond laser

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Abstract
This paper presents a facile method for the fabrication of on-chip three-dimensional (3D) microcoils inside fused silica. The main fabrication process involves two steps: (1) creating 3D helical microchannels inside fused silica using an improved femtosecond laser irradiation assisted by chemical etching (FLICE) technology and (2) conductive treatment by injecting metal gallium into the helical microchannels. The high aspect ratio 3D helical microcoil was prepared inside fused silica with a diameter of 100 μm, a length of 880 μm and a pitch of coil of 44 μm. The method is flexible to fabricate microcoils inside fused silica with arbitrary geometry configurations and different coil properties. This type of microcoils can be easily integrated into 'lab on a chip' (LOC) platform inside fused silica substrate.

(Some figures may appear in colour only in the online journal)

1. Introduction

Microcoils have a wide variety of applications. Typical examples include sensors for measuring magnetic fields and detecting flaws in materials, microinductors for electronic circuit, micropositioners and microactuators for microsystems [1–4]. In particular, the microcoils are diffusely used in micro-total analysis systems (μTAS), or 'lab on a chip' (LOC) as electromagnetic generators for the magnetic bead-based chips, microactuators for sample sorting and separating, excitation and detection coils for micro nuclear magnetic resonance (NMR) spectroscopy and so on [5–9]. In recent years, much effort has been made to fabricate microcoils. Generally, there are two types of strategies for fabricating microcoils. One type of strategy employs lithography and electroplating conducting material on top of an insulating substrate to fabricate planar spiral-shaped microcoils [10, 11]. However, these flat coils result in inhomogeneous magnetic field, low inductance value and small quality factor. The second is focused on three-dimensional (3D) microcoils, which own the stronger and more uniform magnetic field than the planar microcoils. The processes include multilayer photolithography and electroplating [12–14], winding metal wire around yoke and creating helical conducting paths on cylinder [15–18]. But these methods are overly complex and difficult to fabricate on-chip 3D microcoils. There is an unmet need for a facile method to fabricate the integratable on-chip 3D microcoils inside the material, especially inside fused silica for μTAS applications. Fused silica is a kind of ideal material for LOC applications due to its very high optical transparency, low background fluorescence, chemical inertness and hydrophilicity [19], and an increasing number of LOC platforms have been created inside fused silica. The fabrication of on-chip 3D microcoils inside fused silica is urgently needed by such LOC platforms for the miniaturization and integration of systems.

Recently, femtosecond (fs) laser micromachining becomes a new tool to fabricate 3D structures inside transparent materials [19]. Different types of 3D microstructures have been demonstrated inside glass and fused silica [19–23]. And femtosecond laser irradiation assisted by chemical etching (FLICE) is a main strategy for fabricating 3D microstructures inside fused silica [24, 25]. In this paper, we present a facile method to fabricate on-chip 3D microcoils inside fused silica. The main fabrication process

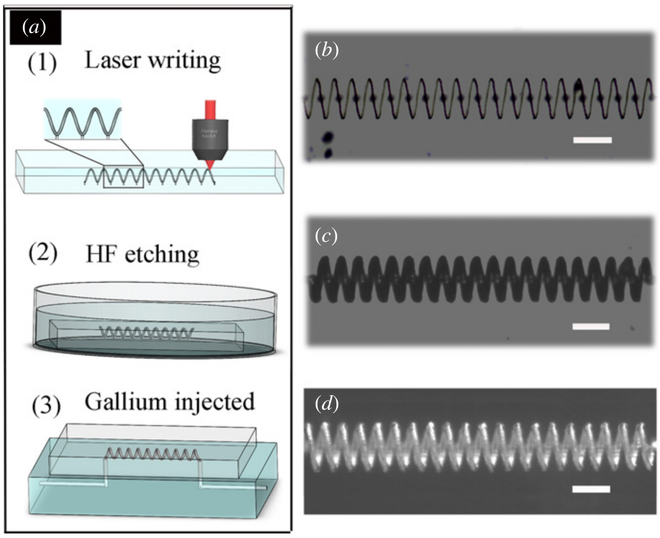

Figure 1. (*a*) Schematic diagram of the fabrication process. (*b*) The optical micrograph of the helical line written in the laser writing process. (*c*) The optical micrograph of helical microchannel obtained after etched in a 10% HF acid solution for 45 min. (*d*) The optical micrograph of microcoil realized by injection of gallium into the helical microchannel. The scale bar equals 100 μm.

involves two steps: (1) creating 3D helical microchannels inside fused silica using an improved femtosecond laser irradiation assisted by FLICE technology and (2) conductive treatment by injecting the metal gallium into the helical microchannels.

2. Methods and experiments

The fabrication procedures of the microcoils are schematically depicted in figure 1, which involves two steps of producing 3D helical microchannels inside fused silica and injecting metal gallium into the channels. To fabricate 3D helical microchannels, a fs laser beam, which is created by a Ti:sapphire pulsed laser oscillator–amplifier system (wavelength = 800 nm; pulse duration = 30 fs; repetition rate = 1 kHz), is focused inside the fused silica substrate ($1.2 \times 1.2 \times 1.2$ mm^3) by a microscope objective lens (NA = 0.9, objective magnification is 100, working distance is 2 mm, depth of field is 0.5 μm, Nikon). The helical line is written by the laser via translating the 3D-stage (the sample was fasten on the stage, the *xyz* resolution of the stage is 40 nm, the model of the stage is H101A ProScan^{TM} II upright stage made by prior scientific) along the pattern path with a speed of 10 μm s^{-1}, as shown in figure 1(*b*). In the laser writing process, the circularly polarized laser is employed for the higher etch rate for fabricating complex 3D microchannels compared to linearly polarized laser [26]. The laser power is temporally modulated by a computer-controlled attenuator to implement the power compensation. The geometry configurations of height, pitch and circle diameter of the helical line can be controlled by computer. Then, the sample with helical line is immersed in a 10% hydrofluoric (HF) acid solution (diluted by deionized water) at room temperature. Because the etching ratio of the laser-modified area is much higher than the areas without irradiation, the helical microchannels could be obtained after etching for tens of minutes, as shown in figure 1(*c*). Meanwhile, to facilitate the HF penetrating into the helical microchannels, an ultrasonic bath is used during the chemical etching process.

Then, the metal gallium is injected into the fabricated helical microchannels to achieve the conductive microcoils. Here, the gallium with a purity of 99.99% is used as the metallic conductors. To create the connector for the injection process, a polydimethylsiloxane (PDMS) film is prepared on the fused silica sample and two syringe needles are inserted inside the PDMS film to connect the entrances of the helical channel. Before the injection, the metal gallium is heated by an alcohol burner. The gallium is rapidly liquefied once the temperature reaches the gallium melting point of 29.8 °C. The liquid gallium is then injected into the helical microchannels by a syringe pump from a syringe needle with a low speed of 0.1 mL h^{-1} and the gallium is simultaneously sucked by a suction pump at another entrance. In the injection process, the temperature is maintained at 45 °C with an infrared heat lamp. Then, the sample with channels filled by metal gallium is cooled at room temperature. After the gallium is solidified completely, the PDMS film is peeled by an ultrasonic bath oscillation assisting process. The fabrication result is shown in figure 1(*d*).

3. Results and discussion

3.1. Formation mechanism of the helical microchannels

The mechanisms of the femtosecond laser irradiation assisted by FLICE technology is based on selectively increasing chemical etching rate by femtosecond laser irradiation [19]. The increase of etching rate is due to the self-ordered nanocracks induced by femtosecond laser irradiation. The self-ordered nanocracks benefit the permeation of the acid solution into the material along the path of femtosecond laser irradiation. After chemical etching, the channels will be formed along the path of laser writing because the etching rate of the laser-modified area is much higher than the areas without irradiation. However, the increase of chemical etching rate is practically limited. The lengths of the channels fabricated by the previous FLICE process are generally limited to a few millimeters, and the channels are often conical with wide entrances and narrow central parts because the channel entrances often suffer a longer etching time than the central parts.

3.2. Improvement of the FLICE process

The long helical microchannels with high uniformity are required for obtaining the high inductance of the microcoils. To overcome the limitation of length and uniformity of the channels fabricated by the previous FLICE process, we propose an improved FLICE process by introducing extra access ports and power compensation methods. By creating a series of laser-scanned lines which connect a sample surface and the internal structures, extra access ports are prepared by the chemical process, as shown in figure 2(*a*).

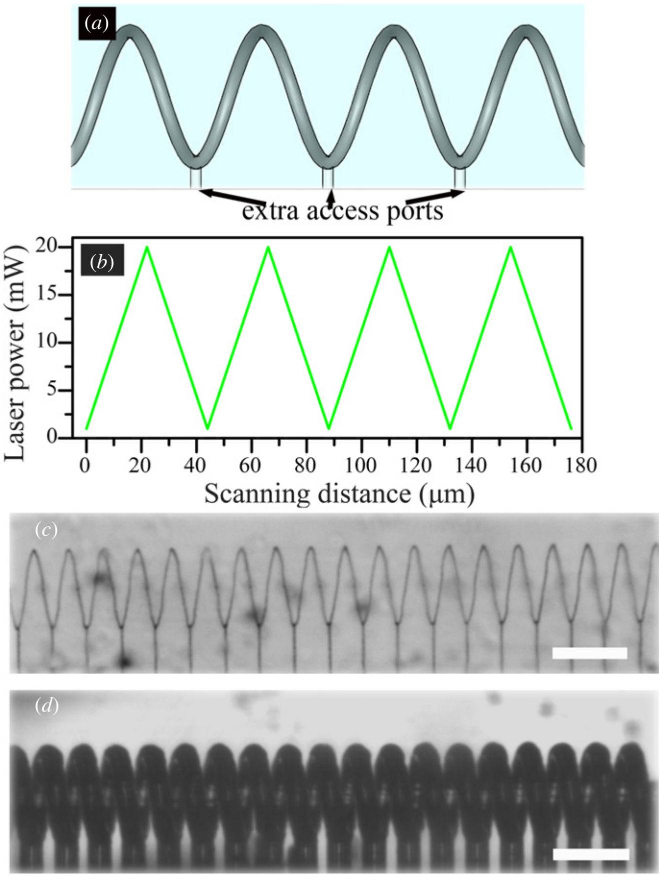

Figure 2. (*a*) Schematic diagram of extra accesses introduced into helical channel. (*b*) The laser power curve in the laser writing helical line process. (*c*) The side view of the helical line written by the laser. The length of the extra access ports is 70 μm, and the diameter of the line is about 2 μm. (*d*) The side view of helical microchannel obtained after etched in a 10% HF acid solution for 50 min. The length of the extra access channels is 45 μm. The diameter of the channel is 25 μm. The scale bar equals 100 μm.

In that case, HF acid solutions can directly penetrate into the central parts of the channels rapidly. Then, each part of the microchannels can be etched simultaneously. If adequate access ports are introduced along the expected way, 3D helical channels with arbitrary lengths could be realized inside fused silica and glasses. Besides, to solve the conical-shape problem of the fabricated mcirochannels, we investigate the power dependence of the diameters of the chemically etched microchannels. The results indicate that the diameter of the microchannels linearly increase with increasing the laser power. According to the phenomenon of diameter variability, a laser power compensation method is introduced into the fabrication process by linearly and periodically changing the laser power, as shown in figure 2(*b*). After the introduction of extra access ports and power compensation, the long helical microchannels with uniform diameters are fabricated inside fused silica, as shown in figure 1(*c*). In the experiment, the extra access ports are introduced to each turn of the helical microchannels with the lengths of 70 μm below the microchannels, as shown in figure 2(*c*). Figure 2(*d*) shows the side view of the helical microchannels obtained after etched in a 10% HF acid solution. The laser power is changed linearly and periodically from 3 mW to 20 mW each turn by tuning a computer-controlled attenuator. To decrease the effect of the extra access ports on microcoils, in the laser writing process, the helical line can be written closer to the undersurface of the sample, and then the length of the extra access ports will be decreased. Besides, after HF acid solution etching, the extra access ports can be polished away or blocked by insulating light curing adhesive.

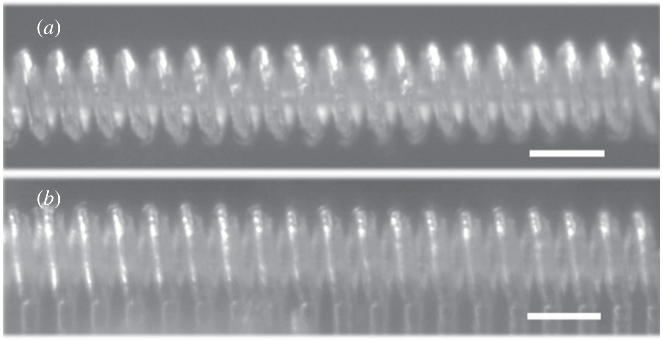

Figure 3. (*a*) The top view of 3D helical microcoil fabricated inside fused silica. (*b*) The side view of the 3D helical microcoil. The scale bar equals 100 μm.

3.3. Conductive treatment of the microcoils

The microsolidic process [27], an approach for achieving 3D conductive structures inside material by injecting metal into the microchannels, is implemented for the conductive treatment. In this paper, the metal gallium is used as the metallic conductors. The choice of gallium is motivated by its low melting point (29.8 °C) and low resistivity compared to other liquid metals. To inject the low-fluid-mobility liquid gallium into the narrow and complex helical microchannels, the injection pressure is needed to be properly controlled. So a syringe pump is employed to inject the liquid gallium from an entrance of the helical channel. Besides, in order to facilitate the injection of liquid gallium, a suction pump is used to supply suction at another entrance. In this process, the sample temperature should be maintained above the melting point of gallium to avoid the gallium solidification.

The 3D helical conductive microcoil is well prepared inside fused silica after the metal gallium being injected into the helical microchannel, as shown in figure 3. The length, diameter and pitch of the coil are 880 μm, 100 μm and 44 μm, respectively. The diameter of the gallium wire is 25 $\pm$ 2 μm. The inductances, L, of the helical micro-coils can be estimated by Wheeler's formula:

$$L = \frac{10\pi \mu_0 N^2 r^2}{9r + 10h_{\text{coil}}} \quad (1)$$

where $\mu_0 = 4\pi \times 10^{-7}$ H m^{-1} is the permeability in free space, N is the number of turns, r is the coil radius and h_{coil} is the length of the coil. The inductance of the liquid–metal coil is calculated to be 4.26 nH. The direct-current resistance of the microcoils is 12 Ω, as measured with a digital LRC bridge (TH2817B).

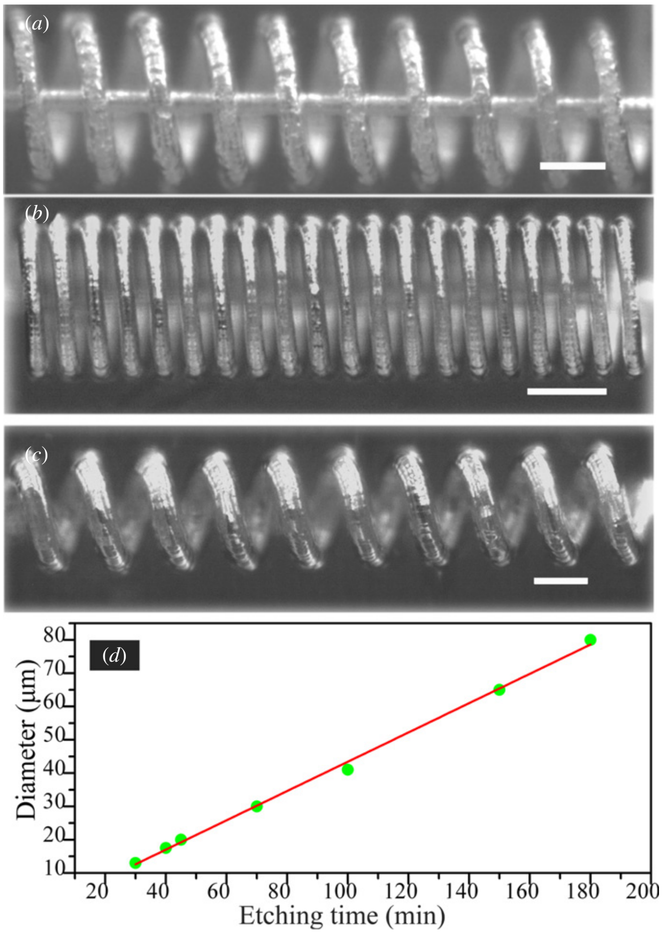

Figure 4. (*a*–*c*) The optical micrograph of microcoils with different configurations including the turns, pitch, diameter of coil and the diameter of the channel (it is also the wire diameter). (*a*) The microcoil with coil core injected gallium. For (*a*)–(*c*), the etching time are 105 min, 70 min and 150 min, respectively. The scale bar equals 100 μm. (d) Etching time dependence of the diameter of the channel.

3.4. Control of geometry configurations of the microcoils

The flexibility of coil fabricating technology is required for a wide range of applications: for example, sensors and actuators need high inductance and a high winding density, the coils for microscale magnetic resonance analysis require high quality factors and a homogenous magnetic field [16].

For the proposed method, the turns, length, diameter and pitch of coils can be arbitrarily changed in the laser writing process. The wire diameter (the channel diameter) can be flexibly controlled by controlling the etching time of helical channels. The diameters of the channels with different etching times are measured and the results are plotted in figure 4(*e*). It indicates that the diameters of the microchannels linearly increase with increasing the etching time. These geometry configurations, including the turns, length, diameter, pitch of coils and wire diameter, will decide the coil properties such as inductance and resistance. The 3D helical microcoils with different geometry configurations and coil properties are fabricated by the improved FLICE assisted by the microsolidics method, as shown in figures 4(*a*–*c*) (figure 4(*a*): the number of turns, length, diameter, pitch of coils and wire diameter are 10 950 μm, 260 μm, 95 μm and 42 μm, respectively; figure 4(*b*): the number of turns, length, diameter, pitch of coils and wire diameter are 20, 880 μm, 200 μm, 44 μm and 35 μm, respectively; figure 4(*c*): the number of turns, length, diameter, pitch of coils and wire diameter are 10, 1250 μm, 230 μm, 125 μm and 65 μm, respectively.). Besides, the method is flexible to fabricate microcoils with or without coil core. By writing central line to the helical line in the laser writing process, the 3D helical microchannels with central microchannels can be easily obtained after HF acid etching processing. The microcoils with coil core can be achieved after injecting liquid gallium into the helical microchannels and central microchannel. Figure 4(*a*) shows a microcoil with gallium coil core.

4. Conclusions

In summary, we present a facile method for fabricating 3D helical microcoils inside fused silica. The high aspect ratio 3D helical microchannel was demonstrated inside fused silica with a length of 880 μm, a diameter of 100 μm, a pitch of coils of 44 μm and the wire diameter of 26 μm. By introducing extra access ports and the power compensation method into the FLICE process, the uniform 3D microcoils with arbitrary geometry configurations and different properties can be achieved. This paper provides a new way to realize integration of microcoils with microfluidics inside fused silica using femtosecond laser micromachining assisted by the microsolidics method. It would be beneficial for developing the novel and high-quality microfluidic systems, such as magnetic-bead-based microfluidic systems, biochemical fluorescence detection and biophotonic systems [26].

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