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# Scalable shape-controlled fabrication of curved microstructures using a femtosecond laser wet-etching process

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ABSTRACT

Materials with curvilinear surface microstructures are highly desirable for micro-optical and biomedical devices. However, realization of such devices efficiently remains technically challenging. This paper demonstrates a facile and flexible method to fabricate curvilinear microstructures with controllable shapes and dimensions. The method composes of femtosecond laser exposures and chemical etching process with the hydrofluoric acid solutions. By fixed-point and step-in laser irradiations followed by the chemical treatments, concave microstructures with different profiles such as spherical, conical, bell-like and parabola were fabricated on silica glasses. The convex structures were replicated on polymers by the casting replication process. In this work, we used this technique to fabricate high-quality microlens arrays and high-aspect-ratio microwells which can be used in 3D cell culture. This approach offers several advantages such as high-efficient, scalable shape-controllable and easy manipulations.

## 1. Introduction

The ability to generate microstructures with curvilinear surfaces on transparent materials allows for fabricating various micro-optical and biomedical devices [1–3]. For example, curved microstructures on glasses and polymers can be used as microlenses which are widely used in optical communications, display and lighting systems, imaging devices and microfabrications [4–6]. In addition, microwells with curved surfaces have potential applications in three-dimensional (3D) cell culture, single cell trapping and analysis, microbioreactor arrays and lab-on-chip systems [7–9].

Several strategies have been adopted for fabricating curved microstructures, such as lithographic thermal reflow technique, gray-tone photolithography, ink-jet process, diamond milling, PDMS processes and beam direct writing [10–14]. However, these methods still have drawbacks in fabricating high-quality curved microstructures. Lithography-based techniques require expensive equipments and complicated procedures, and are difficult to fabricate high-aspect-ratio microstructures; ink-jet and PDMS process can generate spherical-shaped microstructures, but the shapes are not controllable; diamond milling and beam direct writing methods can be used for producing microstructures with tunable shapes, but suffer from long processing times. For most current microfabrication techniques, precise fabrication of scalable and shape-tunable curved microstructures with high-aspect-ratios is still challenging.

To overcome these limitations, we have developed a simple, high-efficient maskless technique for large-area gapless MLAs using a femtosecond laser wet etch (FLWE) process in the previous works [15–17]. In the FLWE process, the ultrafast laser delivers intensity- and time-controlled, programmable arranged, individual pulses to a glass chip. The sample is then subjected to wet-etch processing. The laser pulses change the physical and chemical properties of the glass in the focal spots, and the wet-etch processing that follows carves out a unique microlens array pattern. It offers several advantages: 1) ability to create high-aspect-ratio concave curved microstructures rapidly; 2) flexibility in tuning the dimensions and profiles of the structures; 3) ease of manipulation and a few fabrication procedures. This work presents a improved femtosecond laser wet-etching process (FLWE) for fabricating high-quality microlens arrays with controllable shapes especially aspherical microstructures. And these special microlens arrays are highly desired in biomedical field for example microlens arrays with high-aspect-ratio concave microstructures can be used as microwells for cell culture and analysis.

## 2. Experimental

The fabrication process is schematically depicted in Fig. 1. The materials used in the experiments were polished silica glass chips with dimensions of $15 \times 15 \times 2$ mm$^3$. The laser source was 800 nm, 30 fs and 1 kHz Ti:sapphire oscillator-amplifier system (FEMTOPOWER Compact Pro, FEMTOLASERS). The beam was focused normally onto the surface of the silica glass via an objective lens (NA $= 0.5$) and created the laser-induced modifications of the materials. After the laser treatments, the samples were immersed in the water-diluted hydrofluoric (HF) acid solution with a concentration of 5% for several minutes. Because of the laser-induced modifications of the materials, the laser-treated regions were chemically etched out rapidly with little damage of the

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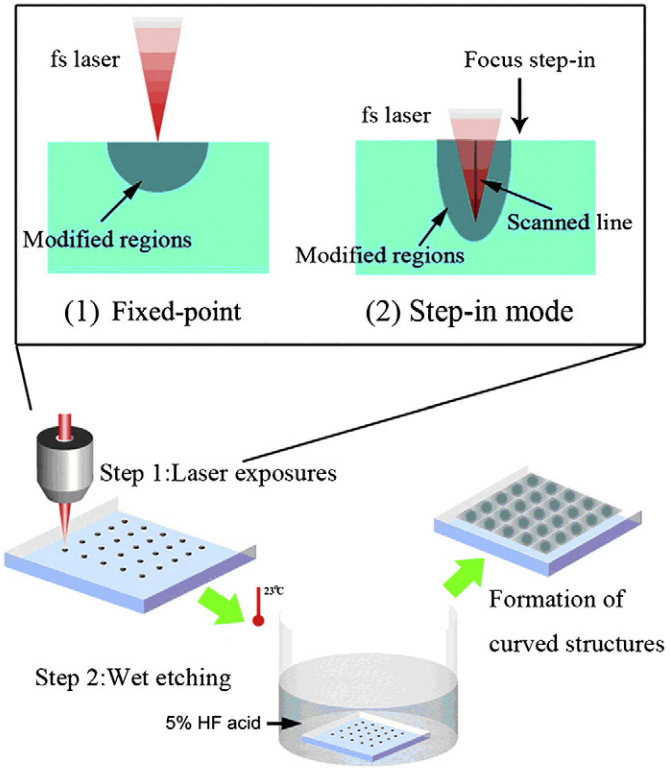

**Fig. 1.** Schematic diagrams of the fabrication process and the laser exposure methods.

original materials, and the concave microstructures formed. The whole etching process was monitored by an optical microscope system (OM, NIKON CV-100) equipped with a CCD camera. When the structures were completely fabricated, the samples were rinsed in the deionized water and dried by hot air.

To fabricate microlenses and high-aspect-ratio microwells, two types of laser exposure methods were used: the fixed-point irradiation and the step-in irradiation, as shown in Fig. 1. For fabricating microlenses with relatively small sag heights, fixed-point laser irradiations were adopted. The laser exposing time was controlled by the mechanical shutter. The laser power, which was tuned by a variable attenuator, ranges from 0.3 mW to 7 mW. We will demonstrate the power dependency of the lens diameter and sag height in the next section. The exposure duration was about 500 ms. In the experiments, long exposure time of each point will increase the structural uniformity of the microlenses, but decreased the fabrication efficiency. The time of 500 ms is the experimentally optimized parameter, which allows for generating 5000–6000 exposure spots per hour. So to speak, over 10,000 microlenses can be fabricated within 3 h by the above parameters.

For high-aspect-ratio microwells, step-in exposure method was used to increase the depth of the laser-induced modification. By translating the sample in the direction parallel to the optical axis ($z$-axis), the focused laser beam was drilled inside the transparent sample. Benefited from the nonlinear absorption regime of the photons, the femtosecond laser can penetrate the wide-band materials when the energy density is below the damage threshold of the materials and induce breakdown when it exceeds that value. This process, in most cases, was used to write or produce modifications and damages directly embedded inside transparent materials. Here, a line-patterned modification region was created in the sample (Fig. 1) by the focal spot of the laser pulses.

This laser-induced chemical process can produce concave microstructures on silica glasses. To fabricate convex microlenses, we used PDMS (Sylgard 184, Dow Corning) casting method: the degassed mixture of PDMS and curing agent with a ratio of 10:1 was poured on the glass molds and cured for 100 min in temperature of 90 °C. For the microwells, their PDMS replicas could be used for a second replication to create concave structures on hydrogels, which are suitable for cell culture. The results were investigated by a field-emission scanning electronic microscope (FE-SEM, JEOL JSM-7000F). The samples for SEM observations are pretreated by coating a thin film of Pt atoms with

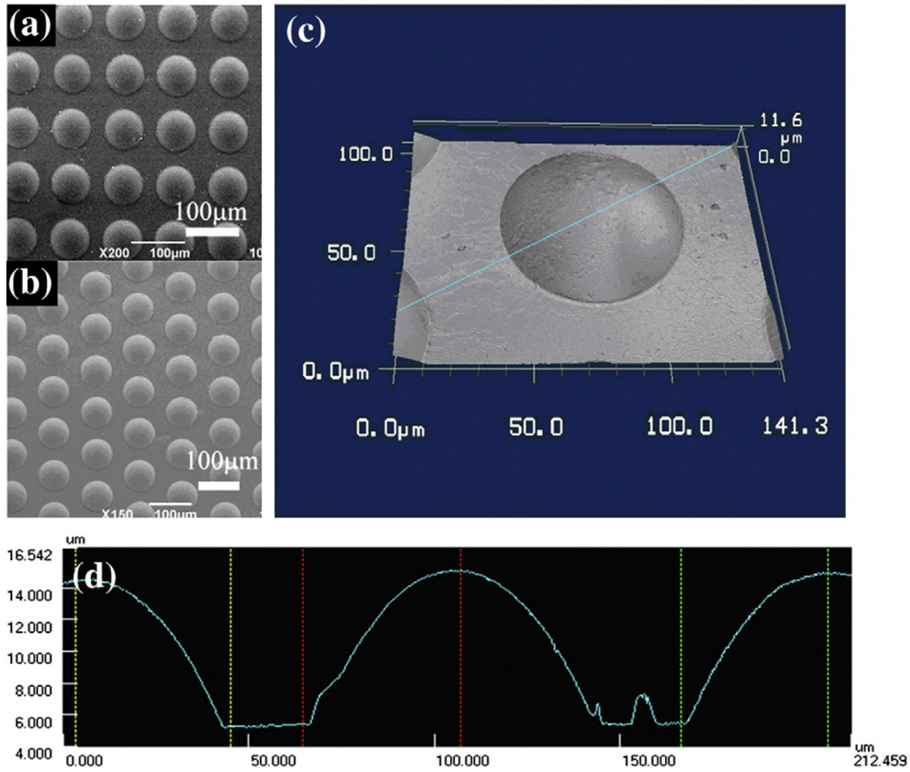

**Fig. 2.** Images of the fabricated microlens arrays. (a) Rectangular-packed microlens array. (b) Hexagonal-packed microlens array. (c) 3D profile of a microlens. (d) Cross-sectional profiles of the microlenses in the rectangular-packed array.