Silicon-Based Suspended Structure Fabricated by Femtosecond Laser Direct Writing and Wet Etching

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Abstract—Suspended structures have attracted a great deal of attention in the field of micro-technology and nanotechnology for their important functions. However, the fabrication of the suspended structures is typically cumbersome and time-consuming and requires complicated processes, such as multiple deposition, photolithography, and etching. Here, we report a new method of fabricating flexible suspended structures by femtosecond laser direct writing followed by wet chemical alkaline (KOH) etching. Our proposed method will enable convenient and flexible fabrication of suspended structures for a wide variety of applications in the field of micro-technology and nanotechnology.

Index Terms—Femtosecond laser, suspended structure, silicon, wet etching.

I. INTRODUCTION

X ITH the rapid development of micro- and nanotechnology, suspended structures have attracted a great deal of attention for their unique characteristics [1]–[3]. In microelectromechanical systems (MEMS), suspended structures are widely used to produce various semiconductor sensors and actuators combined with functional films and high-performance circuits [4]-[7]. Moreover, suspended structures have brought attention to applications afforded by the extremely long photon lifetime of whispering-gallery modes (WGMs) [8] and open microfluidic systems [9]-[12]. In recent years, many methods have been developed to fabricate suspended structures, such as surface micromachining [2], bulk micromachining [3], [4], LIGA [13], and threedimensional (3D) mask technology [14]. Each of these techniques has its own unique advantages; however, these methods rely on multiple deposition, photolithography and etching, which entail time-consuming processing steps.

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Furthermore whole-goal design will limit the flexibility of suspended structure fabrication seriously. Given these challenges, it is clear that a convenient and simple method to fabricate suspended structures is needed.

Femtosecond lasers have been widely used as a powerful tool for three-dimensional devices fabrication [15]-[20]. However, direct generation of suspended structures on silicon by femtosecond laser is challenging owing to the broad absorption of silicon in the UV-VIS-NIR region. Here, we propose a new method to fabricate suspended structures with the widely used Ti-Sapphire femtosecond laser. First, flexible two-dimensional (2D) patterns are printed on undoped (100)-oriented silicon by femtosecond laser irradiation; the silicon oxide patterns act as masks during the subsequent wet chemical alkaline (KOH) etching process. A variety of complex suspended structures can be obtained by carefully controlling the laser power and the etching parameters. Scanning Electron Microscope (SEM) observations of the structures and the optical performance of the fabricated devices themselves indicate that this is a simple, practical method to fabricate 3D suspended microstructures.

II. EXPERIMENTS

Suspended structures were produced on silicon using a two-step process (schematically depicted in Fig. 1). First, $\langle 100 \rangle$ -oriented silicon was irradiated in air by high-frequency pulses of femtosecond laser oscillator (800 nm, 80 MHz, 120 fs) with a long focal length objective (NA=0.8). A two-galvano-mirror set and a piezo stage were used toreduce the programmable horizontal and vertical scanning movements of the focused laser spot; a detailed description of this procedure is available in reference [21]. Because of the high precision of femtosecond laser direct writing and accurate positioning of translation stages, a variety of complex 2D patterns could be flexibly fabricated at any local area, as shown in Fig. 1(a).

Then the irradiated substrate was immersed in KOH solution and rinsed with deionized water; thereafter, the predesigned suspended structures were realized.

III. RESULTS AND DISCUSSION

A smooth helical line produced by 110-mW (pulse power intensity about 1 TW/cm²) [22] femtosecond laser writing is shown in Fig. 2. No obvious thermal melting zone was observed, and very few sputtering particles surrounded the ablation area. After the substrate was etched in KOH for 2 min,

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Fig. 1. Schematic of suspended structure fabrication by laser fabrication assisted with wet chemical alkaline etching.

the sputtering particles and the unmodified area outside the pattern were removed, and the modified area maintained in its original position, as shown in Fig. 2(c). Specially, there is a visible triangular micro-holder below the helical line, which is attributed to the anisotropic etching process of silicon in KOH. Typically, silicon (100) is etched in a layer-by-layer manner by KOH, and triangular defects are produced when the orientation of silicon is (111). Longitudinal etching is effected by the naked (111) crystal phase in the boundary beneath the line, which was confirmed by an apex angle of 70-degree in the cross-section of the holder and further confirmed by crystal phase analysis. When the sample was etched for 10 minutes, the etching depth was greater, but the line width remained the same. A 5% hydrogen fluoride (HF) etchant was used for comparison; the difference in results is apparent in Fig. 2(d), where the laser-irradiated pattern is shown to be completely etched. This phenomenon can be attributed to the different etch ratios of silicon/silicon dioxide in KOH and in HF.

As further proof, the surface composition of the lines array and helical line after femtosecond laser irradiation was analyzed by Energy Dispersive X-ray (EDX). As shown in Fig. 2(e-f), approximately 48% oxygen and 52% silicon were detected on the surface of the modified structures regardless of line scanning or mapping. Therefore, the laser induced pattern is mainly a kind of partially oxide silicon, or silicondioxide, owing to the laser-induced thermal oxidation of silicon in air. This flexible pattern can serve as a mask and is assisted by KOH etching.

The dependency of the line width on the power of the laser was studied systematically. Although the diameter of a laser focus is determined by the formula $D=1.22*\lambda/NA=1.22\mu m$, it changes significantly with exposure power and time owing to the Gaussian light intensity profile. As shown in Fig. 3(a), the line width increased with the



Fig. 2. (a-b) are the designed structures written on silicon without etching while (b) is the zoomed up image; (c-d) are the structures etching with KOH and HF for 2 min, respectively; (e-f) are the EDX results of line arrays and helix patterns.

laser power. The laser power was measured before the objective lens and had a diameter of 2 mm. The dot interval for line scanning was 100 nm and the beam scanning speed is 20 μ m/s. At a power of 65 mW (0.61 TW/cm²), close to the threshold, the line width was approximately 660 nm at an exposure time of 10 ms, whereas at 165 mW (1.52 TW/cm²), the line width was approximately 1 μ m. Specially, it showed a linear change from 835 nm to 1003 nm when the power increased from 85 mW (0.8 TW/cm²) to 165 mW (1.52 TW/cm²). In principle, the lines should have been wider as the laser power approached the threshold; however, in reality, lines with a width of less than 600 nm were obtained. This phenomenon was attributed to the high reflectivity of silicon before ablation and the rapid decline after ablation at a wavelength of 800 nm. In this case, therefore, the second pulse would ablate a wider line. In addition, thermal effects were inevitable with pulse accumulation. When the exposure time changed from 1 ms to 10 ms, the tendency of the width to increase was similar but smaller (by less than 100 nm). The longitudinal depth is shown in Fig. 3(b); the laser intensity distribution is shown in the inset. As the power increased (with an exposure time of 10 ms), the thickness of the area modified by the laser increased from 1.1 μ m to 1.6 μ m. The effective depth was less than the effective length calculated by the formula $h = 2n\lambda/NA^2 = 2.5\mu m$ owing to optical absorption and scattering.

Fig. 4 shows various structures obtained by laser direct writing followed by wet etching in 20% KOH at 70 $^{\circ}$ C. The samples shown in Fig. 4(b-c) were etched for 2 min, those shown in Fig. 4(e-g) were etched for 3 min. A distinct



Fig. 3. The dependency of structure precision with laser parameters. (a) Line width with pulse power intensity and exposure time; (b) line thickness with power intensity.



Fig. 4. SEM images of various structures. (a) Chinese dragon; (b-d) top view line arrays, circular rings, and rings arrays; (e-g) triangular, square, and pentagonal barriers.

two-layer, 3D structure appeared for the triangular, square, and pentagonal barriers. It is noteworthy that some ripple structures formed in the vertical direction (Fig. 4(e-f)), but these structures were absent in the lateral direction. This phenomenon can be attributed to the anisotropic etching which occurred in response to the silicon crystal orientation. Fig. 4(a) shows a winding Chinese dragon with a 3D profile produced by



Fig. 5. SEM images of various designed suspended structures. (a-d) line, disk, helix and hexagon.

laser direct writing and 5 min wet etching. Compared with the samples shown in Fig. 4(b-d), the base of the dragon has a significant shrinkage due to the anisotropic etching effect. More complex suspended structures can be fabricated by taking advantage of this effect. The abovementioned results indicated that the structure height increased with etching time and that the lateral etching effect takes advantage of the sample being in good contact with the etchant. Therefore, silicon under linear patterns would be etched more quickly than silicon under planar patterns. Thus, some complex 2D patterns that combined linear and planar patterns were designed and generated on silicon by femtosecond laser direct writing; those patterns acted as flexible masks in the subsequent wet chemical alkaline etching. By controlling the etching time to produce a state in which the silicon under the linear mask was completely etched, the linear part of designed pattern was suspended, and the planar part was connected with the silicon substrate. Thus, fabrication of various 2D patterns on silicon can permit corresponding suspended structures to be obtained.

As shown in Fig. 5, suspended structures including straight line, disk, helix, and hexagon were successfully fabricated using this method. Fig. 5(a) shows a suspended straight line that was fabricated using a dumbbell pattern, then etched for 7 min in 20% KOH at 70 °C. Fig. 5(b) shows a suspended disk that was etched for 10 min under the same etching conditions as the sample shown in Fig. 5(a); and only a simple disk pattern was just employed to fabricate it. To test the robustness of our method, more complex suspended structures such as suspended spiral coil and hexagon were successfully obtained in a similar manner, as shown in Fig. 5(c-d). Fig. 5(c)shows a suspended helix supported by two square bases. The micro peaks on the base are due to a second masking effect caused by silicon oxide particles falling onto the substrate during the etching process. A suspended hexagon supported by two supporting pyramids is shown in Fig. 5(d). It should be noted that the two pyramids on the base were not intentionally designed, but they contributed to the anisotropic etching. The ratio of etching rate for silicon in (110), (100), and (111)

orientations is 200:100:1. Therefore, these two pyramids were naturally generated, and the suspended hexagon was obtained.

IV. CONCLUSIONS

In summary, we have successfully demonstrated a simple and flexible method to fabricate suspended structures by femtosecond laser direct writing followed by wet chemical alkaline etching. Unlike conventional methods for fabricating suspended structures, the mask we used was created by targeted femtosecond laser irradiation rather than by complicated and time consuming processes. Moreover, local design is easy to realize because femtosecond laser direct writing is capable of extremely high precision and the accurate positioning by the use of translation stages. In addition, multiple deposition, photolithography, and etching steps have been replaced by a single wet chemical alkaline etching step. Therefore, this novel method for fabricating suspended structures is a good candidate for both current and future applications in microand nanotechnology.

REFERENCES

- I.-Y. Huang, C.-H. Sun, and K.-Y. Hsu, "Improving bandwidth and return loss of Si-based MEMS antenna using suspending and electromagnetic band-gap structures," *Sens. Actuators A, Phys.*, vol. 174, pp. 33–42, Feb. 2012.
- [2] S. K. Pavuluri, C. H. Wang, and A. J. Sangster, "A high-performance aperture-coupled patch antenna supported by a micromachined polymer ring," *IEEE Antennas Wireless Propag. Lett.*, vol. 7, pp. 283–286, 2008.
- [3] I. Papapolymerou, R. F. Drayton, and L. P. B. Katehi, "Micromachined patch antennas," *IEEE Trans. Antennas Propag.*, vol. 46, no. 2, pp. 275–283, Feb. 1998.
- [4] E. Öjefors, K. Grenier, L. Mazenq, F. Bouchriha, A. Rydberg, and R. Plana, "Micromachined inverted F antenna for integration on low resistivity silicon substrates," *IEEE Microw. Wireless Compon. Lett.*, vol. 15, no. 10, pp. 627–629, Oct. 2005.
- [5] A. B. Kaul, E. W. Wong, L. Epp, and B. D. Hunt, "Electromechanical carbon nanotube switches for high-frequency applications," *Nano Lett.*, vol. 6, no. 5, pp. 942–947, Apr. 2006.
- [6] C.-Y. Lee and G.-B. Lee, "Micromachine-based humidity sensors with integrated temperature sensors for signal drift compensation," *J. Micromech. Microeng.*, vol. 13, no. 5, p. 620, May 2003.
- [7] S. P. Natarajan, T. M. Weller, and A. M. Hoff, "3-D micro coaxial transmission lines with integrated MEM capacitors," *IEEE Microw. Wireless Compon. Lett.*, vol. 17, no. 12, pp. 858–860, Dec. 2007.

- [8] M. Borselli, K. Srinivasan, P. E. Barclay, and O. Painter, "Rayleigh scattering, mode coupling, and optical loss in silicon microdisks," *Appl. Phys. Lett.*, vol. 85, no. 17, p. 3693, Oct. 2004.
- [9] C. Wang and M. Madou, "From MEMS to NEMS with carbon," *Biosens. Bioelectron.*, vol. 20, no. 10, pp. 2181–2187, Apr. 2005.
- [10] D. Lee, Z. Ye, S. A. Campbell, and T. Cui, "Suspended and highly aligned carbon nanotube thin-film structures using open microfluidic channel template," *Sens. Actuators A, Phys.*, vol. 188, pp. 434–441, Dec. 2012.
- [11] B. P. Casavant *et al.*, "Suspended microfluidics," *Proc. Nat. Acad. Sci. USA*, vol. 110, no. 25, pp. 10111–10116, Apr. 2013.
- [12] Y. Hanada, K. Sugioka, H. Kawano, I. S. Ishikawa, A. Miyawaki, and K. Midorikawa, "Nano-aquarium for dynamic observation of living cells fabricated by femtosecond laser direct writing of photostructurable glass," *Biomed. Microdevices*, vol. 10, no. 3, pp. 403–410, Dec. 2008.
- [13] F. Cui, W. Liu, W. Chen, W. Zhang, and X. Wu, "Design, fabrication and levitation experiments of a micromachined electrostatically suspended six-axis accelerometer," *Sensors*, vol. 11, no. 12, pp. 11206–11234, Nov. 2011.
- [14] H. Mazraati, M. Gharooni, S. Darbari, S. Mohajerzadeh, and F. Salehi, "Realization of suspended silicon-based structures using a smart threedimensional etching method," *J. Vac. Sci. Technol. B*, vol. 32, no. 6, p. 062002, Oct. 2014.
- [15] F. Chen *et al.*, "Maskless fabrication of concave microlens arrays on silica glasses by a femtosecond-laser-enhanced local wet etching method," *Opt. Exp.*, vol. 18, no. 19, pp. 20334–20343, Sep. 2010.
- [16] A. Kiani, K. Venkatakrishnan, B. Tan, and V. Venkataramanan, "Maskless lithography using silicon oxide etch-stop layer induced by megahertz repetition femtosecond laser pulses," *Opt. Exp.*, vol. 19, no. 11, pp. 10834–10842, May 2011.
- [17] M. Haque and P. R. Herman, "Chemical-assisted femtosecond laser writing of optical resonator arrays," *Laser Photon. Rev.*, vol. 9, no. 6, pp. 656–665, Nov. 2015.
- [18] L. Yuan, M. L. Ng, and P. R. Herman, "Femtosecond laser writing of phase-tuned volume gratings for symmetry control in 3D photonic crystal holographic lithography," *Opt. Mater. Exp.*, vol. 5, no. 3, pp. 515–529, Mar. 2015.
- [19] S. Juodkazis, Y. Nishi, and H. Misawa, "Femtosecond laser-assisted formation of channels in sapphire using KOH solution," *Phys. Status Solidi-Rapid Res. Lett.*, vol. 2, no. 6, pp. 275–277, Dec. 2008.
- [20] S. Juodkazis, H. Misawa, T. Hashimoto, E. G. Gamaly, and B. Luther-Davies, "Laser-induced microexplosion confined in a bulk of silica: Formation of nanovoids," *Appl. Phys. Lett.*, vol. 88, no. 20, pp. 201909.1–201909.3, May 2006.
- [21] D. Wu, J. Xu, L.-G. Niu, S.-Z. Wu, K. Midorikawa, and K. Sugioka, "In-channel integration of designable microoptical devices using flat scaffold-supported femtosecond-laser microfabrication for couplingfree optofluidic cell counting," *Light, Sci. Appl.*, vol. 4, p. e228, Jan. 2015.
- [22] R. Buividas, S. Rekštytė, M. Malinauskas, and S. Juodkazis, "Nanogroove and 3D fabrication by controlled avalanche using femtosecond laser pulses," *Opt. Mater. Exp.*, vol. 3, no. 10, pp. 1674–1686, 2013.