Sub-micron resolution three dimensional structure writing using two photon absorption process

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ABSTRACT

Three-dimensional (3D) microstructure writing using the two photon absorption (TPA) process has potential applications in the fabrication of photonic crystals and micromechanical devices. Ormocore and SU-8, two commercially available photoresists, were used to produce 3D structures and compare their writing performances. A 40X objective (NA 0.65) was used to produce high aspect ratio structures with high resolution. The resultant widths and heights of the lines written in the resists were measured for various exposure conditions. Walls with ∼320 nm width and aspect ratios of ∼40 were produced in Ormocore. Other standing 3D structures were also written to demonstrate the capability of the resists.

Keywords: Two photon absorption, three dimensional, 3D, structure writing, Ormocore, SU-8

1. INTRODUCTION

Three dimensional (3D) structures can be created by scanning the sample in three dimensions and changing the physical properties in a small interacting volume. Photopolymerization or isomerization is a simple way to modify the properties of a suitable material. For photopolymerization using a two photon absorption (TPA) process the absorption of energy is proportional to the square of the intensity of light. This property confines the TPA processes in three dimensions at the focal point where the intensity is maximum enabling 3D writing. On the other hand, single photon absorption (SPA) processes are based on the linear response of the material to the light intensity and do not have this optical sectioning capability.

3D microstructure with nanometer (nm) scale resolution have potential application in producing compact micro or nanostructures, photonic crystals, and microtools for cell manipulation in microfluidic systems. In particular, it is possible to write custom designed photonic crystal structures for optical communication devices.

Most of the research groups have used Ti:Sapphire laser oscillators for 3D writing to date. The average parameters for such laser oscillator systems are 80 MHz repetition rate, 100 femtosecond (fs) pulse width, and 800 nm wavelength. The average beam power to produce such microstructures is 10-50 mW. Oil immersion, 100x objectives with high numerical aperture (NA) of 1.4 were used to produce high resolution 3D structures.[1–3] On the other hand, structures with high aspect ratio but relatively lower resolution were reported for 10x and 60x objectives with NA values of 0.30[4] and 0.85,[5] respectively. The best resolution reported for TPA writing was 120 nm using SCR 500 resin.[1] For commercially available resists Ormocore[2] and SU-8[3] the minimum dimensions written by TPA method were 150 and 475 nm, respectively.

Ormocore has mostly been used for high precision writing of complex structures. The average aspect ratio for volume pixels (voxels) produced in Ormocore using a 100x, 1.4 NA objective is around 5:1 as reported by Serbin et al.[2] On the other hand, SU-8, a widely used resist for X-ray lithography, has been explored for high aspect ratio structures. Teh et al[4] reported aspect ratios of 23:1 for vertical photoplastic planes and 50:1 for photoplastic pillars.

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The structures reported in this paper are also written by a 800 nm Ti:Sapphire laser with the typical laser parameters for TPA writing. Different structures were produced in both SU-8 and Ormocore to demonstrate the 3D writing capability using low NA objective. Moreover, simple lines at different control parameters were produced to characterize the 3D writing process. Very high aspect ratio walls written in Ormocore using TPA process are also presented in this article.

2. EXPERIMENTAL DETAILS

An 80 fs, 800 nm Ti:Sapphire laser oscillator (MaiTai, Spectra Physics) and a 40X objective with 0.65 NA was used was for writing of submicron resolution 3D structures. Exposure was controlled by a Newport electronic shutter having a minimum shutter opening time of 10 ms. A 3-axis computer controlled dc motor stage from Physik Instrument (PI) with a minimum resolution of 7 nm was used to control the sample position. The unidirectional repeatability and backlash of the stage were 0.1 μm and 2 μm, respectively. Codes written in Labview 5.1 were used to write structures at different positions and at different powers with different scanning speeds. The transmittance of the objective was measured to be 0.70 and power given in this article is the power after the objective considering this transmittance.

A 50 μm layer of SU-8, from MicroChem Corp., was coated on 0211 glass substrate by spinning the photoresist at 3000 rpm. The substrate was then baked at 65°C for 3 minutes and at 95°C for 6 minutes according to the manufacturers recommendations. Post exposure baking was 1 minute at 65°C and 5 minutes at 95°C. After the 3D laser writing the sample was then developed for 5 minutes in SU-8 developer, rinsed with IPA and dried using N_2 gas flow. Adhesion of SU-8 on glass was enhanced by putting a 1 μm thick adhesive layer of XP Omnicoat between the SU-8 and glass. For Ormocore a 20 μm thick layer was coated at 3000 rpm. After baking 2 minutes at 80°C it was exposed to write the microstructures. It was then developed in OrmoDev for 1 minute, soaked in IPA for 15 sec and left in DI water for 1 to 2 minutes. Finally it was then dried by N_2 gas flow.

3. RESULTS

For a moderate NA objective, as used in the experiment, elliptically shaped voxels will be produced with high aspect ratios. It thus limits the true 3D writing when a voxel is required to be more like a dot or sphere. Considering the limitation of using a 40X objective simple elongated 3D structures were written in the resists to characterize the process.

3.1. 3D structures

Partially open square spirals and vertical plane on posts structures were designed to be implemented in the TPA absorption writing. The base and pitch of the square spiral were 10 μm. The final arm was scanned only for a length of 7.5 μm. A 10 μm wide vertical plane on posts structure was also designed to demonstrate the feasibility of a floating structure. It is also a test to find the strength of the resist and show the stability of writing in a solid resist. All the structures were written at different focal positions and at different scanning speeds.

Figures 1 shows the SEM images of different 3D structures produced in the resists. The 3D structures were made at different powers and different scanning speeds. A beam power of 20 mW at 75 μm s^{-1} scanning speed has produced the best 3D structures. Lower power and/or higher scanning speed made the structures too weak. On the other hand, higher power and/or lower scanning speed produced long and wide voxels. There are significant distortions due to the physical limitations of the scanning capability of the positioning stage assembly which is clearly visible for the plane on posts structures. Figure 2 shows an attempt to write a complex structure. A 30° tilted 'U of A' was written in Ormocore at a beam power of 20 mW and the scanning speed was 75 μm s^{-1}. Writing of 'o', an octagon with 2 μm side arms, of the 'U of A' was also limited by the hysteresis of the mechanical stage (2 μm).

3.2. Characterization of 3D writing

Simple lines were also drawn at different scanning speeds for a particular power. The higher the scanning speed, the less time the resist was exposed to the beam. For that reason, higher scanning speed produced lines with smaller width and height. The characteristic height of the line was taken as twice the distance from the center of the voxel where the radius was maximum to the upper tip of the voxel. This characteristic height should be related to the Rayleigh range of the focused beam. Lines were drawn at (3, 7, 10, 30 and 70) μm s^{-1} for (15, 20 and 25) mW beam powers. All the lines were drawn for a total of 5 seconds to avoid the initial lower velocity region due to finite acceleration. There were 10 μm to 20 μm separation distances between lines at different velocities. A focal height scan with 1 μm steps was also performed for each speed. Heights and widths were measured from SEM images. Figure 3(a) and 3(b) show lines drawn in Ormocore and SU-8 at different scanning speeds with each row at different focal height.

The measured characteristic heights and widths of different lines are given in Figure 3(c) and 3(d) for Ormocore and SU-8, respectively. There were no standing lines of Ormocore on the substrate for scanning speed greater than 10 μm s^{-1} at 15 mW beam power. Moreover, measuring the width was a difficult process for the standing lines due to the vague edges of the structures. Therefore, results of only 20 mW and 25 mW are presented in the graph.

The minimum width and characteristic height obtained for Ormocore were 320 nm and 2.5 μm, respectively for 20 mW beam power and 70 μm s^{-1} scanning speed. Lines with minimum width of 1.3 μm and 11.4 μm characteristic height were produced in SU-8 for 15 mW beam power and 70 μm s^{-1} scanning speed.

3.3. Ultra high aspect ratio walls

The real total height of a line or wall was higher than the characteristic height of the line. The height of the floating vertical walls, as shown in Figure 1(c) and 1(d), are comparable to the measured characteristic heights as given in Figure 3(c) and 3(d). But for the same beam power and scanning speed, when the floating plane is closer to the substrate there is a secondary structure just beneath the vertical plane as shown in Figure 4(a) and there is a gap between these two planes. Surface adhesion chemistry, the catalytic effect of surface, and reflection enhancement of the field intensity near the surface may be possible reasons for such structures. Bogdanov et al[6] reported wider base/foot of pillars due to emission of photoelectrons from the substrate surfaces. In that article the 8 μm pillars were produced using x-ray lithography and the pillars were 9 μm wide at the bottom. For smaller distances between the substrate and the focal point of the beam there will be no gap between these two sections and they join to form a tall wall with very small width. Figure 4(b), 4(c) and 4(d) show ultra high aspect ratio walls fabricated in Ormocore with 20 mW beam power and 70 μm s^{-1} scanning speed. The height of the wall is 12.6 μm while the width is 310-330 nm. Thus the aspect ratio for the wall is around 40. This is the largest aspect ratio reported for lines/walls written using TPA process. Previously the highest reported aspect ratio was 23:1 as produced by Teh et al.[4]

4. CONCLUSION

Different 3D structures were produced using two photoresists, Ormocore and SU-8, for an 800 nm, 80 fs, Ti:Sapphire laser pulses together with a 40X objective. Simple 3D structures were chosen to characterize the writing process and demonstrate the 3D writing capability. A systematic series of lines were written in order to characterize the 3D writing using TPA process. The minimum width measured for lines drawn at 70 μm s^{-1} scanning speed at 20 mW laser power in Ormocore was 310-330 nm. The height of the line was 12.6 μm giving an aspect ratio of $\sim$40. An enhancement of the resist writing at the surface was found which helped to create these high aspect ratio structures. Complex structure writing was limited by the hysteresis of the stage. This effect may be reduced by writing structures with slower scanning speed, lower beam power or using optical scanners instead of mechanical scanning stages.

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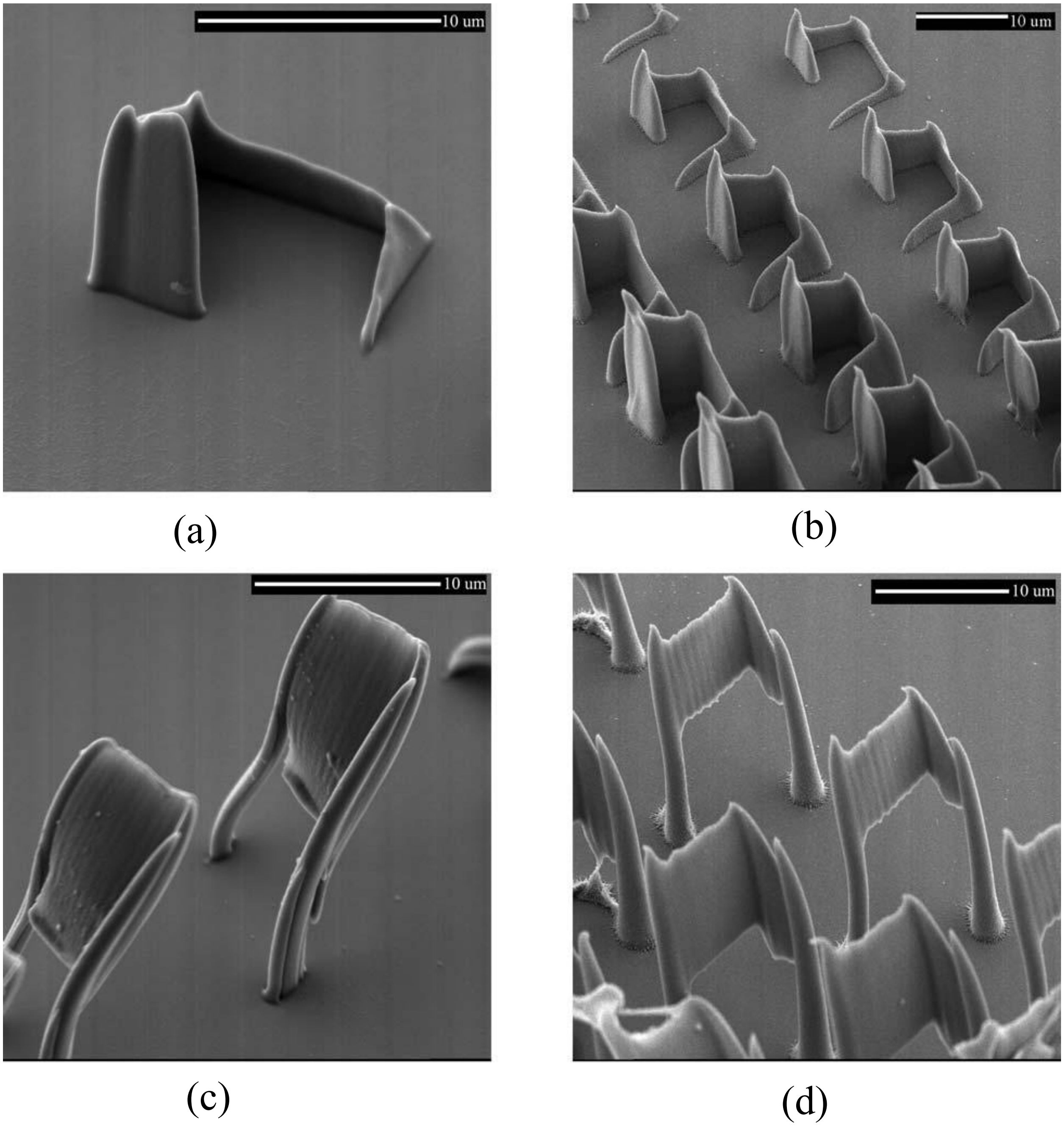

Figure 1. (a) Projection of a square spiral written in Ormocore, (b) similar structures written in SU-8 resist, (c) Vertical planes on posts produced in Ormocore, (d) similar planes written in SU-8, (e) An attempt to write a more complex structure in Ormocore. The beam power was 20 mW while writing all the structures in the figure. The scanning speed for structures written in Ormocore was 75 μm s^{-1}. The speeds was different for different columns of SU-8 structures. The right most column was written at 75 μm s^{-1}, the next left at 50 μm s^{-1} and the next left (if visible) was written at 25 μm s^{-1}.

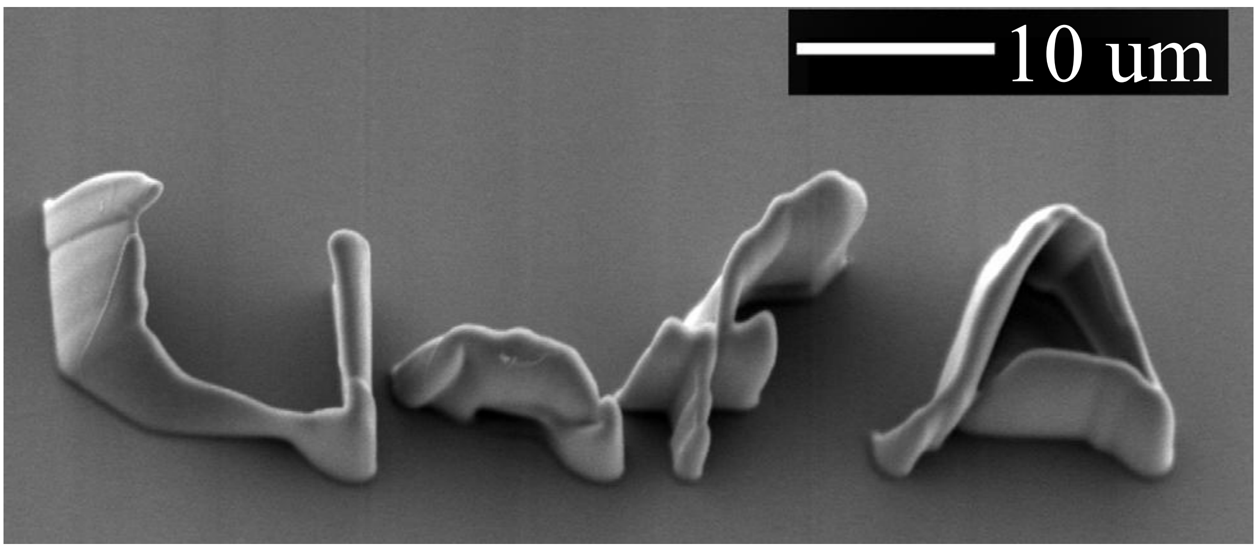

Figure 2. An attempt to write a more complex structure in Ormocore. The beam power was 20 mW and scanning speed was 75 μm s^{-1}.

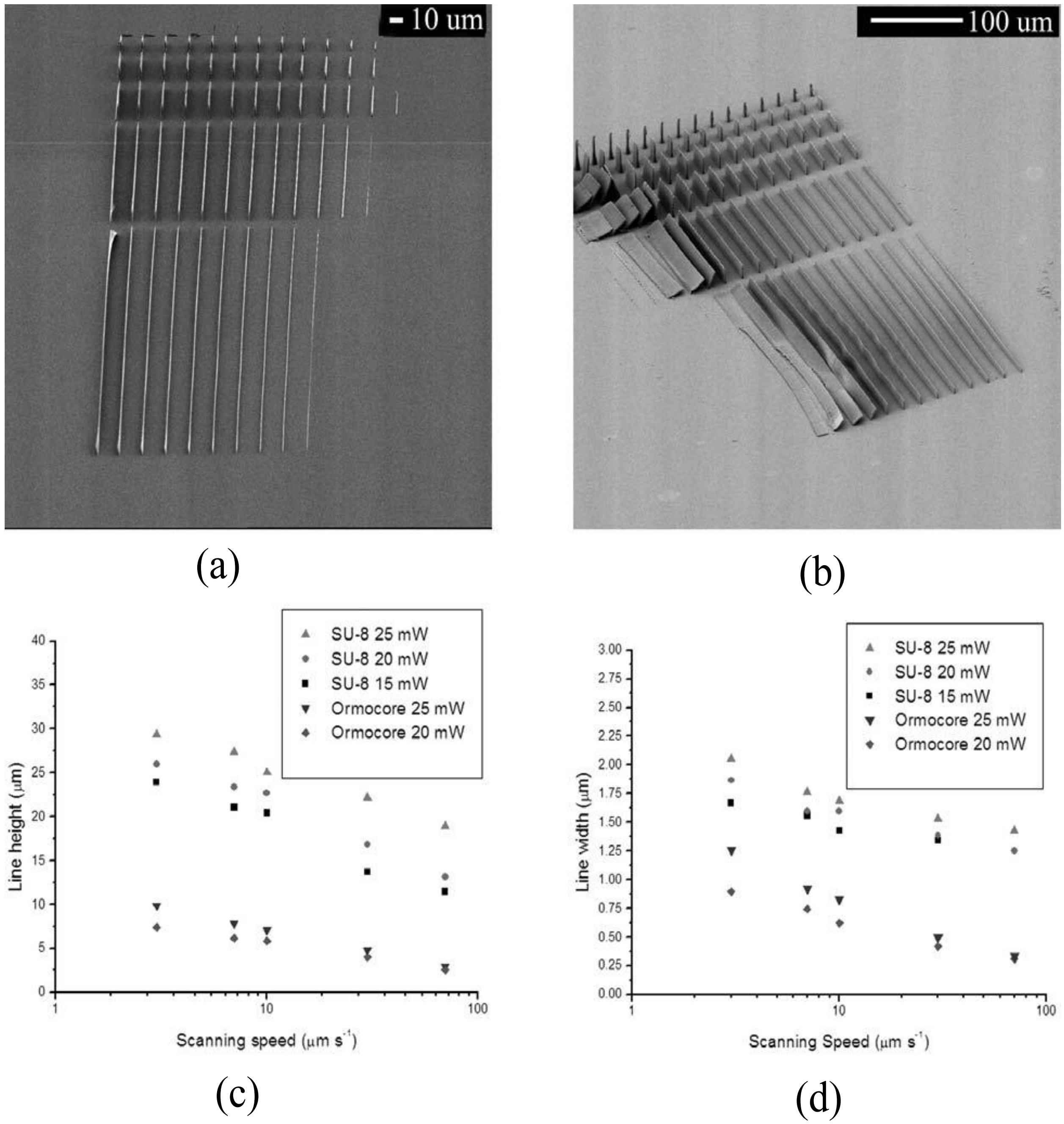

Figure 3. (a) and (b) Lines drawn with different velocities in Ormocore and SU-8 respectively. Longest lines were drawn at 70 μm/s. The shorter line segments were drawn at velocities of 30, 10, 7, 3 and 1 μm s^{-1}. All lines were drawn for a period of 5 seconds. The beam powers while writing lines in Ormocore and SU-8 samples shown in the figure were 20 and 15 mW, respectively. (c) characteristics heights and (d) width of lines for both Ormocore and SU-8 at different scanning speeds and beam powers.

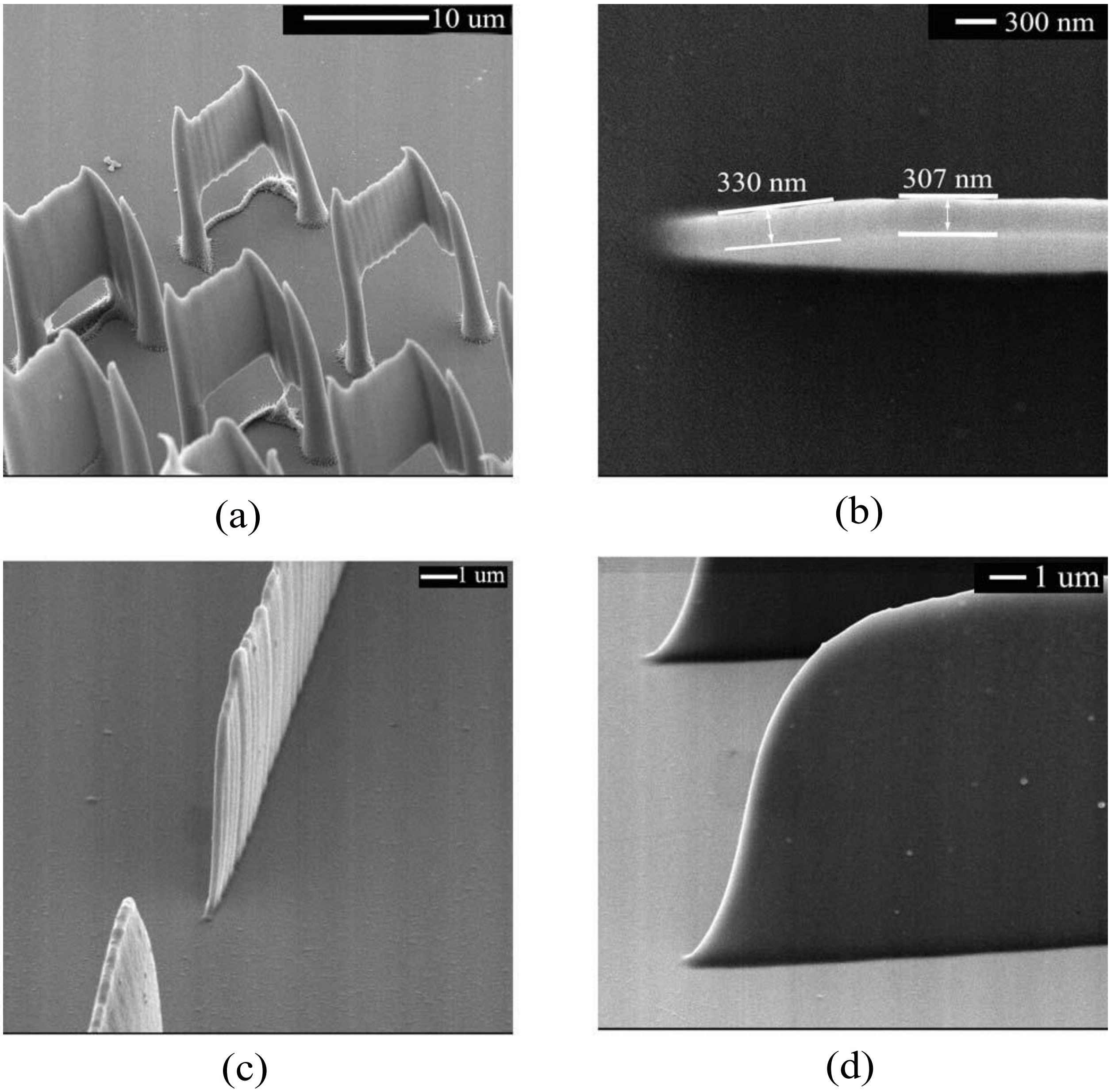

Figure 4. (a) Formation of structures along the beam path but at positions away from the focal point. Standing plate structures written in SU-8 resist for beam power of 20 mW and scanning speeds of 75 μm s^{-1} and 50 μm s^{-1}. (b) top, (c) side and (d) front view of the wall with ultra high aspect ratio. The structures in (b), (c) and (d) were fabricated in Ormocore. The beam power and scanning speed were 20 mW and 70 μm s^{-1}, respectively.