Consolidation of Flame Hydrolysis Deposited silica with a 9.3 μm wavelength CO₂ laser

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Consolidation of flame hydrolysis deposited silica soot with a 9.3 μ m CO₂ laser has been demonstrated. A range of laser parameters were investigated and the surface roughness of the resulting silica layers were characterised with a stylus profiler and white light interferometer. The surface roughness parameters were Ra = 68.9 nm, Rq = 83.8 nm perpendicular to the trajectory of the translated laser beam for a speed of 300 mm s⁻¹ and an average laser power of 42.5 W, and Ra = 29.5 nm, Rq = 36.18 nm along the trajectory of the translated laser beam for a speed of 250 mm s⁻¹ and an average laser power of 35.8 W.

Introduction: Additive manufacture is a disruptive technology currently being applied and developed in a range of technology sectors including aerospace, medicine and photonics [1-3]. Through additive manufacture a route to achieving previously unrealisable designs and material combinations can be made. This work discusses preliminary consolidation results that will enable additive manufacture of optical quality flame hydrolysis deposition (FHD) glass using CO₂ laser consolidation at a wavelength of 9.3 µm.

FHD is a commercial technique used to deposit layers of doped silica onto the surface of a substrate for planar lightwave circuits and device integration [4,5]. The process is capable of depositing layers of silica soot several microns thick with levels of dopants tailored to suit a range of applications. There is a secondary stage to consolidate the deposited soot, typically within a furnace at temperatures exceeding 1200 °C. This thermal processing is not localised as the temperature of the entire wafer is raised. This limits design combination with thermally sensitive, metallic or active photonic structures, such as those based on semiconductor technologies. Use of a furnace also limits the materials which can be used in combination, for example the typical long ramp-up ramp-down thermal cycles can result in notable boundary diffusion.

CO₂ lasers are widely used for machining glass [6,7] and have previously been used for smoothing and polishing of silica [8,9]. These techniques were performed with CO₂ lasers operating with a wavelength of 10.6 μm . As silica has a large absorption peak between 9 to 9.5 μm there is a corresponding absorption in the range of 50 times greater than for 10.6 μm [9-10]. This paper demonstrates the use of a 9.3 μm source for highly localised heating and consolidation of FHD silica soot on a silicon wafer, offering the potential to reduce processing time and relax the constraints on dopants and substrate used in FHD. To the knowledge of the authors this is the first example of FHD consolidation with a 9.3 μm laser. Furthermore, this enables the practical realisation to scale this approach and enable additive manufacture of FHD.

Experimental method and results: Silica soot was deposited through FHD onto the surface of a 6" silicon wafer with a 15 μ m thermally grown oxide layer. The wafer was placed on an Aluminium plate to aid heat dissipation and a Synrad Firestar 100 W CO₂ laser operating at a repetition rate of 20 kHz and a wavelength of 9.3 μ m was focused onto the soot, achieving a full spot size of ~290 μ m. A pair of galvanometer actuated mirrors were used to raster scan the beam over a 5 mm² area (figure 1). Initial tests of a wide range of spot translation speeds and laser duty cycles were performed to determine the optimal parameters for consolidation. The spot translation speed was then set at values from 50 to 400 mm s¹. The power of the laser was controlled through setting the duty cycle at values from 19 to 31%, giving 29 to 47.7 Watts average power. The consolidated array of squares shown in figure 2(a) were then produced. The laser was run from cold, with a power stability of \pm 3.8 %. With high laser duty cycles and low spot translation speeds the soot initially consolidates, however excessive heat generation to the consolidated

areas cause significant cracking as laser fluence increases. The FHD soot surrounding these squares also becomes partially sintered due to the amount of energy localised to the area. At low laser duty cycles the FHD soot does not consolidate completely but power is absorbed primarily by the surface of the soot, leaving a bottom layer of unconsolidated soot between the substrate and consolidated surface. For duty cycles in the range 21 to 29 % and spot translation speeds from 100 to 300 mm s⁻¹ the soot is consolidated down to the substrate with no cracking evident. A comparison between poorly and well consolidated regions is shown in figure 2(b).

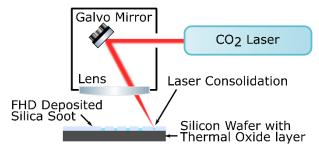


Fig. 1 CO₂ laser light is directed by galvanometer-actuated mirrors through a focusing lens onto a silicon wafer coated in FHD deposited silica soot. The soot is consolidated by the heat generated through absorption of the laser light, forming a silica layer.

The remaining soot from the laser processed samples was removed and the surface of the consolidated regions were measured with a KLA-Tencor P-16+ stylus profiler over a 1 mm length of the squares perpendicular to the lasers raster scan direction. Figure 2(c) shows the measurement for the square consolidated with 400 mm s⁻¹ spot translation speed and 31% duty cycle (47.7 W). The high power allowed consolidation of the silica soot, however the high spot translation speed meant that the consolidation remained highly localised and the heat did not propagate into the surrounding material, an outcome that could be exploited for patterning photonic structures. This leads to the peaks and troughs seen in the plot, which are a consequence of the 80 μ m resolution of the raster scanning.

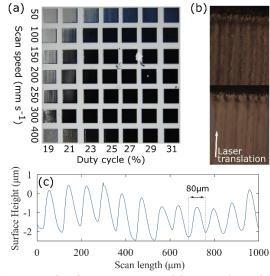


Fig. 2 Images and surface measurements of the array of consolidated silica soot

(a) shows the array of consolidated squares for a range of spot translation speeds and laser duty cycles. Low powers and speeds show partial consolidation, whereas low speeds and high powers cause cracking of the consolidated glass. (b) shows microscope images of a poorly consolidated section for a speed and duty cycle of 150 mm s⁻¹ and 21% respectively (top), and the smoothest consolidation section for a speed and duty cycle of 300 mm s⁻¹ and 27% respectively (bottom). (C) shows a stylus profiler measurement for 31% duty cycle and 400 mm s⁻¹ speed where the 80 µm resolution of the raster scan is indicated in the figure and relates directly to the achieved surface profile.

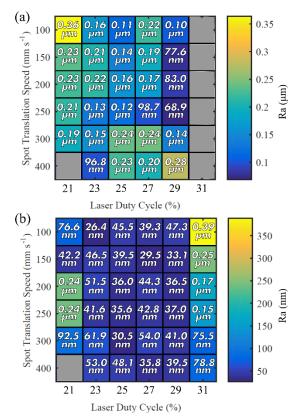


Fig. 3 Plots of surface roughness measurements of the array of consolidated silica soot.

(a) shows a plot of the surface roughness of consolidated areas measured with a stylus profiler perpendicular to the lasers raster scan direction. (b) shows the same measurement performed with a white light interferometer parallel to the lasers raster scan direction. Log(Ra) is plotted to highlight the different surface roughness's achieved. Ra values and their units are shown on the plot and the grey areas in each figure represent Ra values greater than 1 μ m.

Figure 3(a) shows a surface plot of the roughness parameters for each consolidate square measure with the stylus profiler. Grey areas indicate regions where Ra was greater than 1 μ m. Measurements for duty cycles of 19% and spot translation speeds of 50 mm s⁻¹ are not plotted due to partial consolidation or severe cracking preventing accurate measurements. The lowest surface roughness parameters achieved were for a spot translation speed of 300 mm s⁻¹ and a laser duty cycle of 27% (42.5 W), giving Ra = 68.9 nm and Rq = 83.8 nm.

A white light interferometer (Zemetrics Zescope) was then used to measure the surface quality of the consolidated ridges parallel to the lasers raster scanning direction, with the results shown in figure 3(b). The lowest surface roughness parameters achieved were for a spot translation speed of 100 mm s^{-1} and a laser duty cycle of 23% (35.8 W), giving Ra = 26.4 nm and Rq = 32.0 nm. The difference in spot translation speed and duty cycle for the two measurements suggest different parameter spaces for surface roughness quality and overall consolidation of the soot for the 9.3 µm process. The dominant parameter is most likely duty cycle, which controls the pulse duration of the laser and the amount of energy transferred to the soot at a given time. It would be possible to improve roughness through a secondary surface processing of the consolidated silica with the CO2 laser [8]. The surface roughness achieved is a promising first result, which will allow for the development of laser consolidation processes without the need for furnace processing. It will be possible to build upon this process to test consolidation of 3D photonic structures and consolidation on substrates that would not survive furnace processing. Due to the short absorption depth of 9.3 um light in silica and the demonstrated retention of subsurface unconsolidated soot at low powers, consolidation of silica overlaying existing structures would be without causing damage to the layers beneath.

 ${\it Conclusion:}\ \ Demonstration\ of\ consolidation\ of\ FHD\ silica\ soot}$ with a 9.3 $\mu m\ CO_2$ laser has been made for different laser parameters and the

surface roughness of the resulting silica layers were characterised. The highly localised heating of the laser can allow for substrates and photonic structures to be realised which would otherwise be vulnerable to homogeneous thermal processing within a furnace.

Future work will include investigation of further parameter ranges, characterisation of the processed glasses and consolidation of layers with different dopant levels. Consolidation on to exotic substrates which would not survive furnace processing will also be explored, as well as a direct comparison with a $10.6 \, \mu m \, \text{CO}_2$ laser.

(All data supporting this study are openly available from the University of Southampton repository at http://doi.org/10.5258/SOTON/D0346)

Acknowledgments: The authors would like to acknowledge EPSRC grant EP/M013243/1 for funding this research.

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