Additive Manufacturing of Glass

Selective laser melting (SLM) is a technique for the additive manufacturing (AM) of metals, plastics, and even ceramics. This paper explores using SLM for depositing glass structures. A CO₂ laser is used to locally melt portions of a powder bed to study the effects of process parameters on stationary particle formation as well as continuous line quality. Numerical modeling is also applied to gain insight into the physical process. The experimental and numerical results indicate that the absorptivity of the glass powder is nearly constant with respect to the processing parameters. These results are used to deposit layered single-track wide walls to demonstrate the potential of using the SLM process for building transparent parts. Finally, the powder bed process is compared to a wire-fed approach. AM of glass is relevant for gradient index optics, systems with embedded optics, and the formation of hermetic seals. [DOI: 10.1115/1.4028531]

Introduction

AM has drawn significant attention as it moves from rapid prototyping to the fabrication of production parts. Powder bed processes such as selective laser sintering (SLS) or SLM are among the most popular techniques for making complex three-dimensional (3D) parts directly from Computer Aided Design (CAD) models. In these processes, a laser beam is scanned relative to the powder bed. The laser locally heats the powder, melting it (or partially melting it in the case of SLS) which causes the powder to coalesce. The unprocessed powder is removed to reveal the solid part. Besides partial and full melting, there are three other consolidating mechanisms for SLS and SLM technologies; solid state sintering, chemical induced binding, and liquid-state sintering [1]. The SLS/SLM process has been widely studied for manufacturing metal parts, such as iron based alloys [2,3], steels [4–11], aluminum alloys [12,13], magnesium [14], titanium alloys [15–17], and copper alloys [8,18]. It can also be used in sintering/melting ceramic parts [19–22], glasses [23–26], and polymers [27–29].

A critical challenge for metal and ceramics powder SLS/SLM processing is the densification of the parts [8,19]. With the correct processing parameters, densities as high as 99% in SLM process have been reported [13,16], and SLM produced parts can have mechanical properties comparable or even exceeding those of conventionally cast parts [13]. This success has seen the use of SLM for AM of production parts in the aerospace and medical industries albeit at low volumes. SLS/SLM has also been applied to fabricate parts with low density and high porosity. This is particularly useful for replacement of bone because the pores provide space for bone tissues to grow [17].

The properties of additively manufactured parts depend strongly on each layer and in turn on each laser-melted track. For example, Yadroitsev et al. [9] studied the single track melting in metal powder SLM process. They explored effects of the processing parameters to show a considerable negative correlation between the thermal conductivity of bulk material and the range of optimal scanning speed for the continuous single track melting. In determining the optimal parameters for stainless steel powders, Childs et al. [10] showed that the absorptivity changing with the scanning speeds. Sammons et al. [11] set up a model dynamically related the process inputs (laser power, material mass flow rate, and scan speed) to the melt pool dimensions and temperature. Similar studies have been applied to ceramic powders. Yves-Christian et al. [20] studied net shape forming of Al₂O₃–ZrO₂ parts using SLM and demonstrated the effectiveness of preheating the powder bed prior to laser exposure.

Glass has widespread applications such as windows, optics (imaging and nonimaging), and hermetic seals. Traditionally, glass powder is melted in a furnace, and cast in molds to form specific shapes. There is comparatively little literature available about AM or laser melting of glass. SLM was proposed for fabricating structures on mars from indigenous materials [30] and researchers from Hewlett-Packard (HP) experimented with extruding glass fritted polymers [31]. Several references have reported on laser sintering ceramics and glasses using low melting temperature binders to form composites. This has included ammonium phosphate [21], UCAR 430 Acrylic Polymer Latex [22], and monolithic HBrO₂ [23]. Marchelli et al. [24] studied 3D printing of glass using maltodextrin as a binder which could be burned out in a kiln. Kloccke et al. [25] studied laser sintering borosilicate glass volumes including post deposition densification in a furnace. While these studies demonstrate the ability of the SLS process to form solid glass/ceramic parts, the parts were not transparent. On the other hand, Niino and Yamada [29] demonstrated forming a transparent polymer parts by infiltrating high porosity SLS deposited resin blocks with index matched plastic. The physical properties of glass are significantly different from metals or plastics. Glasses typically have much lower thermal conductivity and thousands of times of lower viscosity in the liquid phase than metals [32]. In addition, glass is a brittle material which leads to cracking due to thermal fracture [33]. Finally, soda-lime glass is very transparent in the visible and near-IR [34] which creates processing issues with lasers commonly used in SLM/SLS.
In this paper, we apply the SLM process to soda-lime glass. We start by using a stationary laser beam to form isolated particles. This shows that the melted volume scales with the energy input. Next we experiment with single track scanning and identify the parameter sets required for the deposition of continuous lines. Both stationary and scanned melting are modeled numerically which helps to understand the results. Finally, solid walls are built using a powder bed process. These results are compared to those achieved using a wire fed process. This demonstrates that transparent parts can be built using powder bed SLS but it is more straightforward to start with a fully dense glass filament.

Experimental Setup

Figure 1 shows a schematic of the experimental setup. The powder bed is 5 x 5 cm$^2$ and 0.25 cm deep. It is supported on a refractory block which is positioned by three-axis numerically controlled stages (direct-drive brushless servo motors in x and y with a lab-jack pantograph type mechanism in z) under a fixed focus laser beam. While soda lime glass is nearly transparent at visible and near-IR wavelengths, it is opaque with low reflection to longwave infrared radiation [34]. For this reason, we use a continuous wave Coherent GEM100 CO$_2$ laser ($\lambda = 10.6 \text{ } \mu\text{m}$) to locally heat the glass. The laser is focused onto the surface of the powder bed with an $f/3$ lens. The Full Width at Half Maximum (FWHM) diameter of the beam is measured to be $70 \mu\text{m}$ at its focus. Spherical soda lime glass particles (diameters ranging from 1 to 37 $\mu\text{m}$) from the Mo-Sci Corporation are used throughout this study.

Particle melting: The glass powder is irradiated by the laser beam with the stage in a fixed location. The temperature rises to the point that the powder softens and fuses, coalescing to form a small particle. Conduction away from the melted region also heats the surrounding material and surface tension draws surrounding powder into the particle. Experimentally, the laser power was adjusted between 10 and 40 W while the exposure duration from 0.1 to 20 s. The beam was focused to a size of $70 \mu\text{m}$ for the experiment. The diameter of the melted region was measured with an optical microscope.

Single track melting: The powder bed is scanned in one direction under the laser beam. The height of the stage was adjusted relative to the fixed laser focus to produce three different beam sizes, 70 $\mu\text{m}$, 200 $\mu\text{m}$, and 350 $\mu\text{m}$. At each beam size, lines were drawn at different scan speeds ranging from 0.5 to 100 mm/s. The laser power was adjusted from 10 to 50 W. The track width was measured using an optical microscope prior to removing the fused laser power was adjusted from 10 to 50 W. The track width was drawn at different scan speeds ranging from 0.5 to 100 mm/s. The laser power was adjusted from 10 to 40 W while the exposure duration from 0.1 to 20 s. The beam was focused to a size of $70 \mu\text{m}$ for the experiment. The diameter of the melted region was measured with an optical microscope.

Glass walls: Glass walls were fabricated by melting single tracks in a powder bed layer-by-layer. After each track was written, the stage was lowered and powdered glass was leveled to form a new layer. We also added an electrical strip heater to the substrate which minimizes temperature gradients in the workpiece. Finally, we introduce an alternative wire-fused process. After deposition, the pieces are polished to observe their transmission characteristics.

Numerical Modeling

There are two principal numerical methods to model the melting process; finite element method (FEM) and finite volume method (FVM). FEM models generally only model the conduction heat-transfer. Melting and coalescence are accounted for by locally changing the density and specific heat when the temperature exceeds the melting point. This approach was used by Osakada and Shiomi [35] to model the balling process of metal powder laser melting and by Childs et al. to simulate the single track melting process with metal and polymer powders [10,36]. Paul et al. [37] studied thermal deformation on part errors in metal powder based AM by a 3D thermomechanical FEM model. It is important to note that the fluid dynamics of the melting process is neglected in FEM.

FVM also simulates the heat transfer but includes the melting and resolidification process to capture the liquid motion within the melt pool. This is handled numerically by incorporating the enthalpy of fusion into the specific heat leading to a mushy zone. The volume of fluid (VOF) model can also be incorporated into a FVM model and used to track the free surface of the melt pool. VOF is widely used to simulate welding, for example. Tsai et al. used FVM with VOF to model the laser keyhole welding, arc welding, gas tungsten arc welding, and Metal Inert Gas (MIG) welding processes [38–41]. Zhang et al. presented a model of the SLS process of two-component metal powders by FVM [42,43]. An FEM and FVM coupled model, which can predict laser welding geometry formation and joint strength, was created by Marimuthu et al. [44]. Jamshidinia et al. [45] and Mahamood et al. [46] also modeled melting process of metal powder by FVM. In this paper, we use ANSYS FLUENT 14, a commercial FVM solver to model the melting process. The solidification/melting model calculates the liquid fraction throughout the whole domain based on the temperature (not using the VOF method). As in the experiment, the size melted region of the powder bed was measured at different laser powers and exposure durations or scanning speeds. The following assumptions are made to simplify the analysis of glass circulation and heat transfer:

1. The molten glass acts as an incompressible, homogeneous, and Newtonian fluid.
2. Variation of glass composition in the melting process is neglected, and chemical reactions are neglected. This ignores the effects of any evaporation of volatile species from the glass surface, or the presence of gas bubbles on the thermal physical properties [32].
3. The density does not change with temperature after coalescence.
4. Glass is opaque to the laser beam with a constant absorptivity which allows the laser beam to be modeled as a heat flux.
5. Radiation heat transfer within the glass is neglected.

Given these assumptions and material properties, the governing equations for modeling molten glass can be expressed as

Continuity Equation: \[ \frac{\partial p}{\partial t} + \nabla \cdot (\rho \mathbf{v}) = 0 \] (1)
Momentum Equation: \[ \rho \frac{D\mathbf{v}}{Dt} = \nabla \cdot (\mu \nabla \mathbf{v}) - \nabla p + \rho g \] (2)
Energy Equation: \[ \rho C_p \left( \frac{DT}{Dt} + \mathbf{v} \cdot \nabla T \right) = \nabla \cdot (k \nabla T) \] (3)

where $\rho$, $\mathbf{v}$, $p$, and $T$ denote the density, velocity vector, pressure, and temperature, respectively. All of the thermal properties except for the density, $\rho$, are functions of the local temperature. Outside the molten region, the velocity vector is equal to zero, and the equations reduce to the heat diffusion equation. The thermal properties of soda lime glass powder used for the numerical study are shown in Table 1. This neglects the porosity of the glass powder bed by initially modeling it as solid glass. However, this is not as significant as in metal powder bed processes because there is less of a mismatch between the properties of glass and air, specifically the thermal conductivity.
Table 1  Thermal properties of soda lime glass [32]

<table>
<thead>
<tr>
<th>Property</th>
<th>Powder state</th>
<th>Molten</th>
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<tbody>
<tr>
<td>$\rho$ (kg/m$^3$)</td>
<td>1400</td>
<td></td>
</tr>
<tr>
<td>$\mu$ (Pa·s)</td>
<td>2500</td>
<td>T $&lt; 1073$ K</td>
</tr>
<tr>
<td>$c_p$ (J/kg·K)</td>
<td>10 exp($-2 + 3600/(T - 573)$)</td>
<td>$T &gt; 1073$ K</td>
</tr>
<tr>
<td>$k$ (W/m·K)</td>
<td>1.5</td>
<td>Powder state</td>
</tr>
<tr>
<td>$h_q$ (J/kg)</td>
<td>3.5–4.5</td>
<td>Molten</td>
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<td></td>
<td>523,000</td>
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Boundary conditions: The laser beam is treated as a heat flux on the top surface of the powder bed. It follows a Gaussian distribution [47] with heat flux, $q''$, given by:

$$q'' = \frac{2P}{\pi r_0^2} \exp\left[-2(r/r_0)^2\right]$$

where $P$ is the laser power, $\alpha$ is the absorptivity, $r_0$ is the laser beam waist, and $r$ is the distance from the beam center. The boundary condition of the exposed face of the glass is

$$-k \frac{\partial T}{\partial n} = h(T_i - T) + \varepsilon \sigma(T_4^4 - T^4) + q''$$

which includes convection, radiation, and the laser energy input. On the side surfaces and far away from the center of the laser beam, the $q''$ term is absent. The substrate rests on a piece of thermal insulation material so the lower boundary is treated as adiabatic.

Results and Discussion

Particle melting: Figure 2 shows how the melted particle’s diameter, $d$, varies with the exposure duration and laser power. Four particles were made under each set of experimental conditions, and the error bars denote the minimum and maximum diameters. Surface tension causes the particles to coalesce to a nearly spherical shape with volume roughly proportional to $d^3$. The thermal energy imparted to the system varies with the laser power and the exposure duration, $E = P \times \tau$. A good first order approximation is that the melted volume is proportionate to the thermal energy. For longer exposure lengths, there is more time for the heat to diffuse without melting the glass. In addition, some powder will agglomerate to the outside of the sphere without fully melting. The linear relationship of power to volume implies that the absorptivity is insensitive to the temperature of the melt pool (which would be higher for larger powers) as well as other parameters in the study.

Figure 3 shows the particle diameter determined from simulation, where the size of the particle was determined by the molten region at a given time. The numerical model shows similar trends to the experimental results, however, on average the simulation underestimates the diameter by a factor of 0.76. The numerical model neglects heat diffusion and melting after the end of the heat pulse. Another contributing factor is that in the experiment, unmelted powder coming into contact with the melt pool becomes attached and fused to the particle increasing its diameter.

Single track melting: Depending on the laser power, scanning speed, and beam size, we identify four regimes. Tracks are either (A) continuous lines (B) discontinuous lines; (C) discrete particles; and (D) only partially melted. When the scanning speed is high and the power is sufficiently low, there is no observable change to the powder bed. To vary the beam size, the height of the powder bed is adjusted relative to the fixed focus of the laser beam to give measured beam diameters of 70 µm, 200 µm, and 350 µm.

Figure 4 shows the experimental parameters leading to each of the four regimes. At each height, there is an optimal region that produces continuous lines. Generally, this regime is broader for the smaller, tighter focused beam. At a given scan speed, as the power increases the melt pool becomes unstable and the track breaks up to form discrete particles. The shape of particles is irregular and they form at irregular intervals. When the power is lower than the continuous range, the track is also discontinuous but forms regular round shaped particles at regular intervals. Figure 4 shows that the range of parameters producing continuous lines is broadest for the focused beam with the smallest diameter, shrinking as the beam size is expanded.

The width of the continuous lines decreases with increasing scan speed or decreasing power. This is shown in Fig. 5. The volume of the continuous track is proportionate to the square of the width and scales with the ratio of the power to the scan speed. This relationship is similar to what was observed for the particle melting experiments. In both cases, the volume is linearly dependent on the energy intensity which supports the assumption that the absorptivity of glass to the CO2 laser is insensitive to temperature.

For all three beam sizes, all continuous tracks are in the width range of 0.5–1.5 mm which defines the line-to-line pitch for building continuous parts. For larger beam diameters, there is no set of scan speeds that produce continuous tracks at low power (10 W, 20 W) because the peak energy intensity of the laser beam is insufficient to deposit continuous tracks.

Figure 6 shows simulation results of the scanned tracks for a beam diameter of 70 µm. As would be expected, the maximum

![Fig. 2 Experimental results showing particle diameter as a function of exposure duration and laser power. The image in the inset shows the result for $P = 10$ W and $\tau = 1$ s.](image1)

![Fig. 3 Particle diameter as a function of exposure duration and laser power from simulation](image2)
temperature in the melt pool decreases with high scan speed and lower power. The peak temperature of the melt pool exceeds 2000 K for most points, indicating that vaporization would occur within the melt pool. In the experiment, we observe the presence of visible fumes rising from the melt pool under these conditions. The model does not capture the instability leading to the breakup of the melt pool and all lines in the simulation are continuous. For example, when the parameters that produced discrete particles in the experiment (regime C) are simulated, the resulting track width is smaller than the continuous lines. The model does adequately predict the experimental width of the lines in the continuous regime (A) to a difference of less than 100 μm.

Glass walls: The next step is to deposit a simple one-track wide wall layer-by-layer. After each track is written, the workspace is lowered and a fresh layer of powder spread over the powder bed. This introduces the additional parameter of the layer thickness, s. Figure 7(a) shows a photograph of a wall built with a layer-to-layer thickness of s = 1 mm using laser power of 50 W and a scanning speed of 20 mm/s with a beam spot size of 70 μm. These parameters created consistent tracks with a width of 0.8 mm (shown in Figs. 4 and 5). The part in the figure is 10 layers thick and is not transparent. In addition, it proved to be brittle and broke apart into smaller pieces during polishing. The inset of Fig. 7(a) shows one piece after polishing both sides to remove the sides. Porosity and small cracks in the part lead to significant scattering limiting transparency.

Much of the cracking is due to thermal stresses created in the part during the deposition process. The wall in Fig. 7(a) was deposited without heating the underlying substrate which leads to large thermal gradients in the glass between the top and bottom of the part. This demonstrates the need for annealing to relieve stresses in the part.

Figures 7(b) and 7(c) show a part built on a substrate held at 530 °C. It was deposited using the same laser/speed parameters as Fig. 7(a) but with a thinner layer-to-layer thickness of h = 0.5 mm. In addition, following deposition the piece was surrounded with an insulating fiber blanket to allow it to cool to room temperature gradually over about an hour. Despite this partial annealing step, the part still broke during polishing. However, the polished piece (shown in the inset of Fig. 7(b)) demonstrates significant transparency. Figure 7(c) shows that the transparent core is sandwiched between two thick layers of partially fused glass. It is only in this transparent core where the temperature is sufficient to melt and fully fuse the glass. Air bubbles become trapped in the partially melted glass and cannot escape due to the high viscosity of the lower temperature glass. This is a larger issue for thicker layer heights and led to the lack of transparency in Fig. 7(a).

We also experimented with feeding a glass filament into the melt pool. The filament is fully dense and can be melted and fused to previous layers. Figure 8 illustrates this process. In this work, the filament is a 1 mm diameter glass stringer (Bullseye Glass Co.) and is fed by hand at ~1 mm/s. The laser power is set to

![Fig. 4 Shape distribution of tracks: (a) beam size 70 μm; (b) beam size 200 μm; (c) beam size 350 μm; and (d) photographs of different track regimes](image1)

![Fig. 5 Continuous line width distribution. White, gray, and black correspond to a beam sizes of 70 μm, 200 μm, and 350 μm, respectively.](image2)

![Fig. 6 Simulated results of single track scanning showing with inset showing temperature distribution in the powder bed](image3)
25 W and the stage is scanned at 1 mm/s. The substrate for this experiment was a glass microscope slide and was maintained at a temperature of 530 °C throughout the experiment. The sample was also allowed to cool gradually. These steps permit deposition of large pieces without cracking. Figure 8(b) shows the piece as deposited, and Fig. 8(c) shows the same piece after polishing. These results show a continuous transparent piece that is much more robust than the powder bed results. It is worth noting that in addition to having a smooth free surface, the wire-fed process is more conducive to locally varying the composition of the melt pool which will be advantageous for gradient index optics.

Figure 9 shows brightfield reflected light microscope images of the polished samples in Figs. 7(a) and 8(c). The sample produced with the powder bed process has a significant portion of the image covered by bubbles which scatter light. The wire fed process produces a smoother surface without the inclusions. It is possible to heat the melt pool to the point that convection within the molten region becomes violent due to boiling. However, moderate heating successfully softens the glass to the point that it bonds with the underlying layers without trapping air. These results suggest starting with fully dense feedstocks will be a more direct way of manufacturing optical quality components using AM.

Conclusion

This paper explored melting soda lime glass using a CO2 laser for AM. Generally, the deposited volume is proportionate to the laser energy incident on the glass for both stationary and scanned experiments. Optimal parameters were determined for scanning single tracks in a powder bed. Both experiments were simulated...
regimes which estimate the temperature of the melt pool. These results were the basis for building walls using a powder bed process. This showed the importance of layer height and controlling the temperature profile within the heated part. Finally, these results were compared with a wire fed process using a fully dense starting material. Both approaches are shown to have the potential for depositing optically transparent parts using AM. However, the wire-fed process appears to be more robust and promising.

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References


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