



Lasers in Manufacturing Conference 2021

Manufacturing of fused silica parts by means of Laser Glass Deposition

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Abstract

Additive manufacturing (AM) of polymers and metals is already established in the industry. Materials such as glass create significant challenges based on their material properties. Especially mechanical and thermal properties as well as the viscosity behavior are difficult to handle. So far, only few specialized glass AM processes exist and are established in research and development.

The Laser Glass Deposition (LGD) process offers the possibility to deposit glass fibers without using binder materials. For the application area of optical components, manufactured parts must fulfill high requirements for transparency, surface quality, material purity and homogeneity of the material. Investigations on the printing of individual single-layer quartz glass structures have already been carried out with the LGD process. Within this article the influence of laser power, axis speed and fiber feeding speed on the deposition characteristics is investigated shortly. Subsequently, a multilayer deposition is investigated to manufacture solids with an optical transparency.

Keywords: Laser Glass Deposition; Fused silica; Glass fibers; Multilayer deposition

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1. Introduction

Glass materials are widely utilized in many areas that are also strongly represented in everyday life, such as displays, domestic objects or windows. Further areas such as medical technology or optics are strongly characterized by special designs of components [Hench et al.]. The conventional manufacturing of glass components cannot comprehensively satisfy these requirements for special designs, thus the conventional manufacturing spectrum is severely limited and new manufacturing processes have to be applied. The additive manufacturing enables almost limitless design freedom for the manufactured components. With the help of additive manufacturing processes, it is possible to handle a wide range of different materials. The application of metals and polymers in such manufacturing processes has already been extensively researched and the processes already fulfill industrial standards [Gebhardt]. Qualified processes for the utilization of amorphous materials, such as glass, are almost non-existent and have also been considered exclusively in research and development to this point. The main challenges for a successful application of such a technology are the mechanical and thermal properties as well as the viscosity behavior of the glass material at increased temperatures. In addition to the material-specific properties, high requirements are placed on the manufactured components in terms of their transparency and surface quality. [Rettschlag et al.]

Possible fields of application for the additive manufacturing of glass are, for example, optics, predominantly in the field of high-power optics, and biomedical applications, for example, individual special designs. In the further future, applications in the field of architecture are also conceivable. For the experiments presented in this work, the Laser Glass Deposition (LGD) process is used for processing fused silica fibers. For reproducible additive manufacturing of high-quality components, the influence of the process parameters like laser power and material flux on the quality of layered fused silica structures is investigated. The manufactured samples are measured topographically using a 3D profilometer and the induced material stresses are detected using a polarimeter.

2. Experimental setup and experiments

Additive manufacturing of glass can be performed with different types of the starting material and manufacturing processes adapted to them. A CO₂ laser-based fiber extrusion process, LGD, is being developed at the Laser Zentrum Hannover e.V. (LZH) for a manufacturing of optically transparent components without binders in order to obtain pure glass components as a final product. The setup of the experimental system used has already been described in detail in previous publications [Rettschlag et al., Kranert et al.]. For the experiments carried out in this article, HSQ300 quartz glass fibers from Heraeus with a diameter of 400 µm and a 50 µm polymer coating were chosen. This polymer coating is used only to ensure that the fiber is transported into the process zone without breakage and is burned in the process zone without any residue [Rettschlag et al. 2019]. GE 214 fused silica substrates with the dimensions 100 x 50 x 3 mm³ were used as substrates.

With the help of statistical experimental design and on the basis of preliminary experiments, a suitable number of 25 tests for preliminary experiments was determined for a specified parameter range. These preliminary tests serve to narrow down the parameter range for printing wall structures and to define suitable quality zones for the printed components. For these pre-tests, walls were manufactured from twelve fiber layers and with a length of 35 mm. The length of a single wall is limited by the substrate dimensions. The fabricated wall structures are evaluated with regard to the degree of fusion of the individual layers to each other. The following Fig. 1 shows the result of the sample evaluation and the classification into the quality ranges defined here.

	135 W	141 W	146 W	152 W	158 W
90 mm/min	2	3	4	4	5
108 mm/min	1	2	3	4	4
120 mm/min	1	2	3	4	4
144 mm/min	1	2	3	3	4
180 mm/min	1	2	2	3	3

Number	Definition
1	Low fusion of the individual layers Detaching of the glass fiber
2	Only slight fusing of the individual layers Partially optically transparent Regular surface
3	Optically transparent Fusion of the individual layers Regular surface
4	Strong fusion of the individual layers Optically transparent Unregular surface
5	Sublimation of the glass material

Fig. 1. Tabular summary of the results of the pre-tests to explain the quality levels.

Based on the pre-tests, further experiments on the additive manufacturing of wall structures will be carried out. Particular focus is placed on the height of the individual layers, since these define the surface roughness of the printed components, but also represent a dimension for the process velocity. In comparison to the filament-based polymer printing process, the layer height in the LGD process is not determined exclusively by a movement of the print head in the z-direction, but also by the degree of fusion of the fiber, i.e. how viscous the fiber is during printing. In order to control the degree of melting of the glass fiber, the process parameters must be precisely coordinated.

Both the laser power input and the speed ratio between axis speed and fiber transport speed influence the energy input into the fiber. Before the wall structures are produced, the individual layer height is determined for each parameter set from the deposition of a single fiber layer. From this, the component height of the wall structure is calculated and then compared with the test results. Within a test run, the set layer height is kept constant. The wall structures are all manufactured in a dragging manner, i.e. the direction of deposition of the fiber corresponds to the direction of movement of the axis. After each deposited layer, the fiber is automatically separated in the process by the incoming laser radiation and a new layer is then printed on top. For these tests, the parameter sets listed in the following Tab. 1 are examined.

Table 1. Parameter sets for the manufacturing of different wall structures

Parameter set	Laser power [W]	Axial speed [mm/min]	Fiber speed [mm/min]	Number of layers)
A	135	90	90	
B	141	90	90	1, 2, 4, 8
C	146	90	90	
D	158	90	180	
E	163	90	180	1, 4, 8, 16
F	169	90	180	

Following the performed experiments, the manufactured samples are analyzed with focus on two selected quality properties. The topology of the wall structures is measured with a Keyence VR-3200 profilometer. This stripe projection technique allows a non-contact and damage-free evaluation. To counteract possible

measurement errors due to the transparency of the glass material, the samples are treated with a chalk spray. The average particle size of the chalk spray is $3.8 \mu\text{m}$ and therefore has no significant influence on the measurement results, as the measurement accuracy of the measuring device is $\pm 3 \mu\text{m}$. As a second characteristic, the residual stress of the components is measured with a Strainmatic M4/140 polarimeter from Iliis and the local stresses are evaluated. In the following section, the obtained test results as well as the topography data and residual stresses of the manufactured specimens are shown and analyzed.

3. Results and discussion

For a first examination of the influence of the laser power on the quality on a wall structure of different heights, the material flow is chosen as constant. This means that the axis speed and the fiber feed rate are set equal. The tests are performed with the parameters A to C from Table 1. For each parameter set, a measurement of the topography is performed to assess the properties of the wall structure. Similarly, the measured wall height is compared with the previously calculated expected value. The expected value is obtained by multiplying the determined height of a single layer by the number of printed fiber layers. Following the investigations with constant material flow, samples with a disproportionate material flow are manufactured and evaluated. Here, the fiber feed rate is selected to be higher than the axis speed in order to deposit more material.

3.1. Constant material flow

Parameter set A results in only low melting of the fiber, which leads to inadequate bonding of the individual layers. The average temperature recorded in the process zone during the process is only $1780 \text{ }^\circ\text{C}$, which corresponds to a value just above the softening temperature. The wall of eight layers is not completely preserved after the process; the upper four layers are separated.

Figure 2 shows the measurement results for parameter set B as an example. From the deposition of a single layer, a constant layer height of 0.29 mm was selected within this parameter set. The topographic measurement shown in Fig. 2 demonstrates that the surface of the manufactured wall is nearly homogeneous. The additional deposited areas above the wall result from the fiber separation process. Here, an adjustment is still necessary so that the fiber can be separated without any residues.

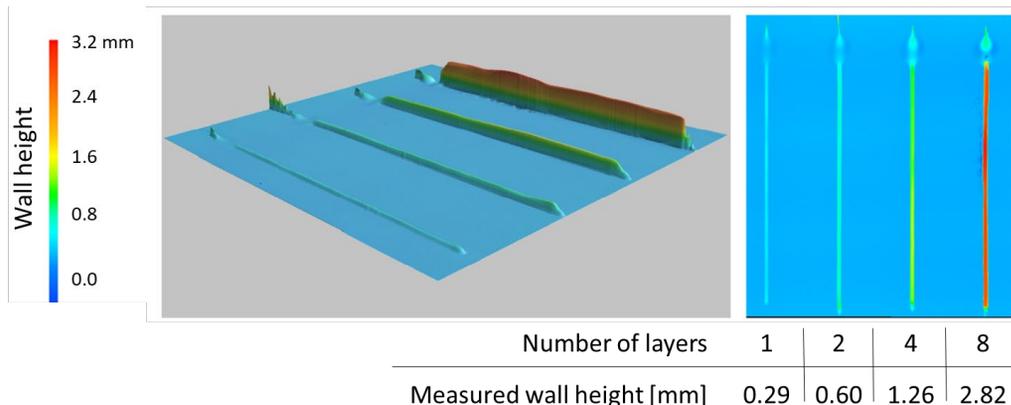


Fig. 2. Height profile of additively manufactured wall structure at a laser power of 141 W and constant material flow. Both the number of layers and the measured wall height are indicated.

The measured values for the wall heights do not show a linear progression but an exponential tendency. This can be explained by the thermal influence. With increasing distance between process zone and substrate, the thermal input into the fiber decreases due to the missing substrate-side heat input. Despite this observation, the wall structure exhibits good strength, but the transparency is limited by the clearly visible layer structure (Fig. 3).

Compared to parameter set B, the results of parameter set C show uneven surfaces and a strong fusion of the individual fiber layers. With long process times, the deposited glass is subjected to a disproportionately large heat input, which greatly reduces the viscosity of the glass and causes the material to flow. Fig. 3 shows the walls manufactured with eight fiber layers of the three parameter sets for comparison. In the top figure, it is clearly shown that the bonding of the fibers was not strong enough, so the structure is broken. In the middle figure, the individual layers are still clearly visible, but the bonding could be significantly improved. The bottom figure shows a strong fusion of the individual layers, but with an unevenness in the structure. The cause of this is still unknown, but it is suspected that slight fluctuations within the supplied process gas might have created it.

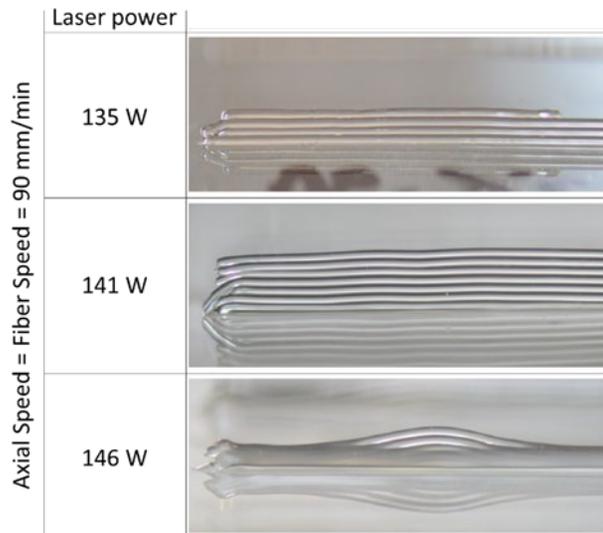


Fig. 3. Comparison of the walls out of eight layers. Above parameter set A; mid: parameter set B; bottom: parameter set C.

Despite the high process temperatures, fluctuations in laser power in the single-digit watt range are still enough to produce significant differences in the quality of the manufactured components. Therefore, a good matching of the process parameters to the required application and property of the printed components is necessary.

Finally, the internal stresses of the material after the process are investigated using the polarimeter. In general, it can be seen that the stresses in the material increase with process time. For single layers, these are about 9 MPa. For the walls with eight layers, stresses up to 17 MPa could be measured. However, no stress cracks occurred in the substrate material during the tests carried out here.

3.2. Disproportionate material flow

In order to achieve a disproportionate, i.e. increased material flow, the axis speed and fiber feeding rate are not kept equal. In the tests carried out here, the axis speed is again set to 90 mm/min (compare section 3.1), but the fiber feeding rate is 180 mm/min, i.e. twice the speed. To guarantee melting of the applied material, the laser powers are set higher than in section 3.1. In this section, parameter sets D to F are considered (Tab. 1).

From the results of parameter set D, it can be seen that increasing the material flow at higher powers from 158 W can counteract the sublimation of the material. However, the surface of the manufactured wall structure is strongly wavy and uneven. Similarly, irregularities are visible on the side regions of the wall. These irregularities are a result of the material being applied to the process zone not being fully softened, so that no oriented placement of the fiber is possible, causing the fiber to wobble. Increasing the laser power should eliminate this effect. It is also observed that the wall height is significantly lower than expected (3.15 mm instead of 6.88 mm) when up to 16 layers are deposited. The expected wall height was again calculated using the single layer height. Since the process is programmed with a fixed expected height, the feeder nozzle is too far away from the printed structure when the number of layers exceeds 10, so that selective deposition is no longer possible. This problem must be counteracted in further experiments.

A further increase of the laser power by 5 W to 158 W (parameter set E) results in a reduced single layer height to 0.27 mm. The manufactured components have a mostly homogeneous surface. The individual layer heights of the structures are lower than in parameter set D (D: 0.20 mm, E: 0.12 mm), but the material has softened more uniformly. Due to the increased heat input into the fiber and the substrate, the viscosity of the glass is significantly reduced compared to section 3.1, which allows for increased bonding to the substrate, but also increases the structure width. The original fiber structure is not preserved, so that the individual layers are no longer recognizable and a good transparency of the wall structures can be observed (Fig. 4). Due to the low viscosity of the glass material, the shaping is largely determined by the surface tension, so that filigree structures cannot be produced with this process strategy. However, it is possible to manufacture transparent components.

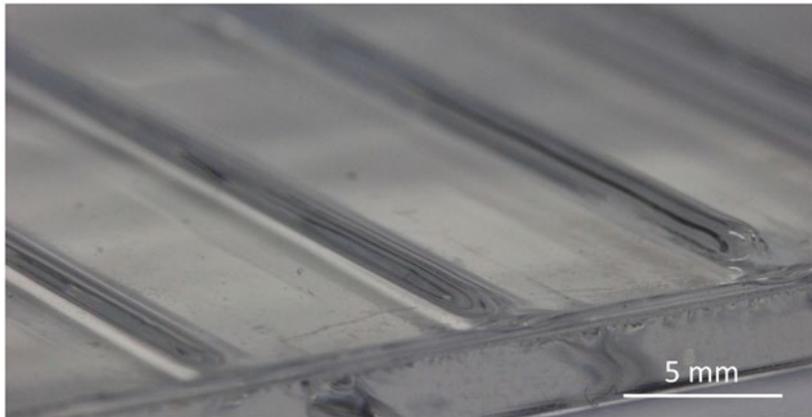


Fig. 4. Photographic image of the wall structures with 1, 4 and 8 layers of the parameter set E.

For the last parameter set, a layer height of 0.22 mm was determined and adjusted. The results show very uniform surfaces (Fig. 5). For the wall structure consisting of 16 layers, a slight peak can be seen in the initial

area (Fig. 5), which is probably caused by the process strategy and the separation of the fiber. This effect must be reduced or completely avoided in subsequent experiments. When depositing multiple layers, the layer height remains constant at about 0.12 mm, as in parameter set E. The power increase again favors fiber detachment. The power increase again favors the flow of the material in the process zone, which makes it possible to produce very homogeneous components. However, evaporation of the material during the deposition process can also be observed, which is why parameter set E is selected for further tests.

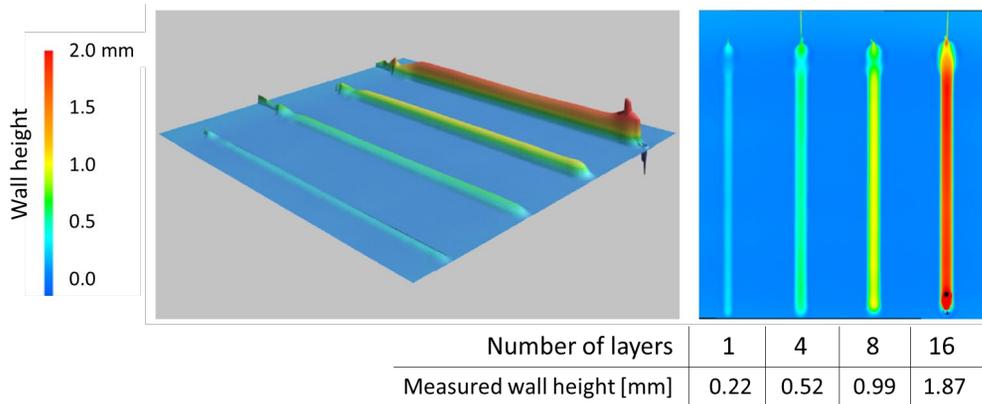


Fig. 5. Height profile of additively manufactured wall structure at a laser power of 169 W and a disproportionate material flow. Both the number of layers and the measured wall height are indicated.

3.3. Long term process of a wall structure of 80 layers

As a final test, a wall of 80 layers is manufactured in a fully automated printing process. The process parameters for this test are: Laser power of 163 W, axis speed of 90 mm/min, fiber feeding speed of 180 mm/min and set layer height of 0.15 mm. The layer height is obtained from the results determined in section 3.2. After the successful manufacture of the wall structure, a change in the quality properties with increasing wall height can be observed (Fig. 6 left).

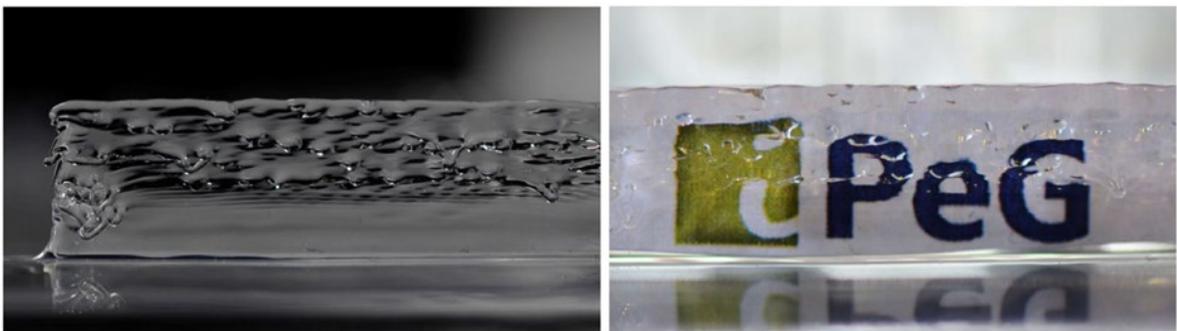


Fig. 6. Printed wall structure made of 80 layers. Left: Image of the manufactured structure after the process; Right: Demonstration of optical transparency through the manufactured wall structure.

The first 30 to 40 layers are well fused, so that no separating layers are visible to the human eye. After these layers, a transition area is visible, where the single-layer structure can already be seen. After this transition area, an uneven deposition of material can be observed, resulting in reduced transparency (Fig. 6 right).

These results demonstrate once again that the process temperature is a significant factor influencing the uniform deposition of the glass material. As the component height increases, this temperature decreases while the process parameters remain constant, which increases the viscosity of the glass material to be applied and leads to uneven material application. Possibilities to avoid this could be, on the one hand, that the laser power is stepwise increased after a defined number of layers. On the other hand, the laser power could be actively controlled by measuring the average process temperature with a pyrometer.

4. Conclusion

In summary, it can be concluded that a successful investigation of additively manufactured basic wall structures could be fabricated using the laser glass deposition process. Within this experimental setup, a height dependence of the print quality could be determined for different laser powers and material flows.

Essentially, this work presents an overview of the influencing factors. It is noticeable that even small parameter variations have a considerable influence on the result of volume deposition. The individual layers also exhibit different properties with increasing component height. In particular, the fusion of the individual layers is decisive here, whereby the laser power could be determined as the main influencing factor for the results carried out here.

For components consisting of several layers, a constant material deposition cannot be guaranteed with the current setup, as a change in temperature conditions is caused. With the aid of dynamic parameter adjustment, it should be possible to achieve constant print quality for different component heights. This can be realized, for example, with the aid of pre-programmed process adjustments based on the results obtained here or by means of active process control using a temperature control system. The integration of a substrate preheating is also necessary to reduce internal stresses. Furthermore, for an unrestricted deposition of the glass fibers for the production of structural components, a modification to a direction-independent fiber feeding should be carried out.

Acknowledgements

The experiments were conducted within the framework of the project “GROTESK – Generative Fertigung optischer, thermaler und struktureller Komponenten” funded by EFRE – NBank (ZW6-85018307).



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