Glass Powder Injection Molding – Ceramic Processing Applied to Glass Components

Conventional shaping of glass components involves a glass melt. The glassmaker is familiar with technologies like drawing, blowing, pressing, floating, casting, and rolling of molten glass. Such shaping techniques are limited with respect to the geometric complexity of the desired components. For processing of sharp edges the glass components need additional finishing steps. Also shaping of holes and undercuts are uncommon for the mentioned glass forming techniques. Powder based shaping methods are an alternative. The article shows how ceramic injection molding can be applied to glass powders.

Introduction

For attaining microstructures in flat glass substrates for microelectronic applications or for micro-reactors the so-called Foturan® glass can be used. This special glass type is sensitive to light of a certain wave length. Due to a chemical reaction it crystalizes during subsequent thermal treatment. The crystalized areas can be removed by etching with fluoric acid which attacks the crystalized material up to twenty times faster than glassy material. Applying a mask during light exposure allows selective material removal during the etching process. In this way microstructures with aspect ratios up to 10 : 1 can be realized. Nevertheless, grinding is necessary for attaining planar surfaces for covering the microstructures with a lid. Furthermore, handling of fluoric acid is inconvenient and harmful.

When shaping of glass is not performed from the molten state but using glass powders (dry pressing, powder injection molding) a subsequent consolidation of the glass powder compacts is achieved by sintering. The advantage of glass sintering is the possibility of manufacturing porous glass components, especially for filtration applications. The temperature necessary for glass sintering is remarkably lower than the temperatures for shaping of glass melts and, moreover, porous sintered glass components are machinable. In case of powder pressing the geometry of sintered glasses is limited to relatively simple geometries. Another disadvantage of sintered glasses is that the residual pores interfere with optical properties.

Keywords
powder injection molding,
glass powder

Glass powder injection molding

Ceramic injection molding is the technology of choice for a large-scale, fully automized production of components with complex geometry including holes, undercuts or threads. Micro-components or microstructured parts can be made by this technique as well as multifunctional products. Due to the highly abrasive behavior of glass particles and the limited optical properties of sintered glass components, powder injection molding did not play any role for glass shaping so far. Therefore, the goals of the project GlasPIM which is introduced here were:

- Application of ceramic injection molding technique to glass powders for attaining complex shaped sintered glass components with sharp edges, and
- Development of sintered electrical conducting glass-carbon-composites by means of a graphite containing glass powder matrix for applications as metal free heating conductors, damping resistors, braking resistors, terminating resistors, powder resistors or micro-reactors.

As initial glass powders, three commercially available glass powders (trade names®: 8250, 8330, 8470) from SCHOTT AG were evaluated. For the development of carbon-free glass components a binder system basing on Licomont®, eMBe GmbH, was chosen. The injection molding feedstocks were prepared by a shear roller (BSW 135-1000, Bellaform) having a solid content of 59 mass-%. This

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device allows an almost complete deagglomeration of the powders and a very homogeneous coating of the powder particle surface with a thermoplastic binder film. For preventing impurities in the feedstock from wear by the abrasive glass powder particles, all steel compon-ents of the shear roller getting in close contact with the feedstock had been replaced by ceramic components previously. For attaining an electrical conductivity in the sintered glass components, 5 mass-% of graphite powder (Timrex T15, KS25, SFG15 from *Timcal*/CH) was added to the initial glass powders and homogeneously distributed. Thus, inert graphite particles are arranged between sinter active glass particles in the powder mixture. For the carbon content, a compromise has to be found considering the formation of the percolation network on the one side and the density of the glass matrix on the other side. While low graphite contents allow many glass-glass particle contacts and good sintering conditions, the percolation network can become interrupted leading to a high resistivity. A high graphite content leads to a high conductivity, but reduces glass-glass particle contacts and constrains sintering what in turn results in low sinter densities. Following these boundary conditions, the electrical properties of the sintered glass-carbon-composites can be adjusted by variations in the density of the percolation network of the graphite particles.

Injection molding was carried out by means of an *Arburg* Allrounder 320S with different tools from *Fraunhofer IKTS* and *Kläger Spritzgusstechnik*. A special tool was designed and constructed for developing a glass micro reactor specified by *mikroglas GmbH*. The desired component can be described as a micro mixer with a Y-shaped channel structure with two inlet and two outlet channels covered by a planar lid.

Debinding and sintering of the injection molded glass components is challenging and much more difficult compared to injection molded ceramic parts when the advantage of the initially shaped details have to be maintained with high precision. This is caused by the different sintering behavior of glass and ceramic powders. In contrast to ceramic powders the glass powder particles soften during heating and densification takes place by viscous flow of a glassy melt. Regarding the fact that the viscosity of glass covers several orders of magnitude within the temperature range which is relevant for the sintering process, it becomes clear that even slightly exaggerated sintering temperatures or holding times can lead to deformations and roundings. Hence the boundary conditions for the sintering process must be evaluated precisely in order to determine the tight ranges of process parameters. Due to the fact that depending on the glass composition softening of the glass particles can start at relatively low temperatures, debinding of the organic components is critical too. It is notable that the binder removal has to be finished before the glass powder starts softening and closing of the pores in the powder compact. Otherwise, residual carbon would be entrapped and may form gas bubbles during sintering in the glass components or can turn the sintered components to grey. This is especially of interest, when pure glass components are manufactured because mechanical and optical properties depend

Tab. 1
Properties of sintered glass-carbon-composites

Feedstock	Resistivity [Ω cm]	Relative Density [%]
Glass 8250 with 5 mass-% Timrex T15	4,2	95,7
Glass 8470 with 5 mass-% Timrex T15	9,5	94,5
Glass 8250 with 5 mass-% Timrex SFG15	4,8	92,2
Glass 8470 with 5 mass-% Timrex KS25	10,2	95,5

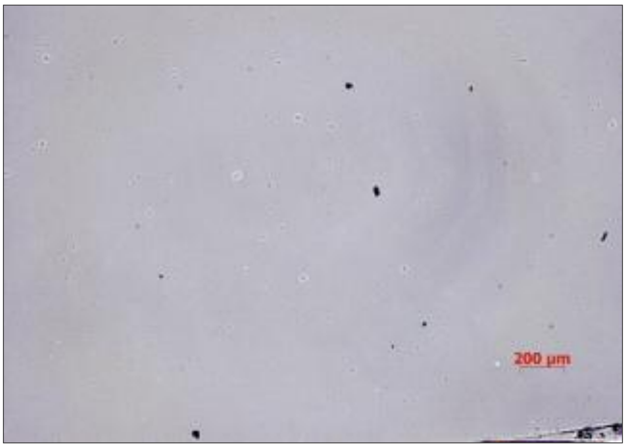

Fig. 1
Light microscopic image of a cross-section of injection molded sintered glass powder 8330

Fig. 2
Sintered disc (thickness 0,8 mm) of injection molded glass powder 8330

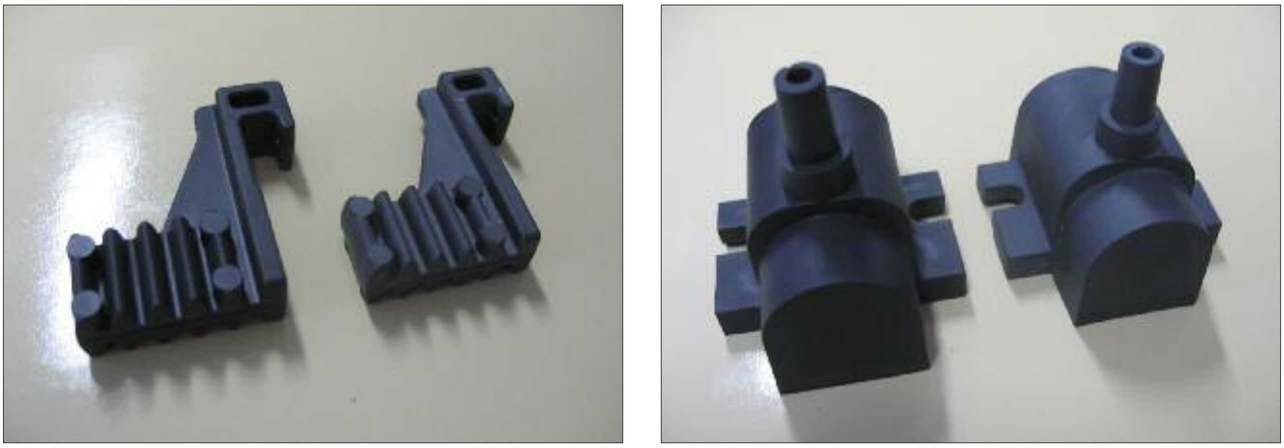

Fig. 3
Injection molded demonstrator components of carbon-containing sintered glass in the green and in the sintered state

strongly on a pore and carbon free microstructure. In glass-carbon-composites these issues are less noteworthy, because they are usually black and the optical appearance does not play a crucial role. Also the debinding process is not that critical because the percolating carbon-network contains a residual porosity, which provides paths for the evaporation of gaseous carbon species.
After sintering the electrical resistance of the carbon containing glass components was measured. Additionally, electrical heating experiments had been carried out with meandering components made of this material. For all samples density and shrinkage were measured. With respect to the averaged densities of the glass powders and the used carbon phases, sintering densities between 92 – 96 % of the theoretical density could be reached for the described glass-carbon-composites.

Sintered glass components and properties

The residual carbon content of the carbon-free glass components after debinding in air at 600 °C was analyzed with 0,007 mass-%. The relative green densities of the debindered injection molded parts ranged from 65 to 70 % and were therefore about 6 – 8 % higher than those of green pressed glass compacts. Sintering of the injection molded components made of powder 8330 had been carried out at 810 °C under vacuum. With >99 % of the theoretic density after sintering, the densities of the compacts were also slightly higher than those of pressed sintered glass components. Fig. 1 shows a cross-section of a sintered glass component. According to the state of the art residual small, roundish pores limit the transparency of the sintered glasses so far. For discs with a thickness of 0,8 mm, the total transmission of light in the visible wavelength range attained 55 – 68 % (Fig. 2).
Sintered discs made of the glass powder 8330 were tested after a grinding process by the double annulus test (DIN ISO 6474), showing a mechanical strength of 55 ± 5 MPa.
The carbon-containing feedstocks were prepared by means of the glass powders

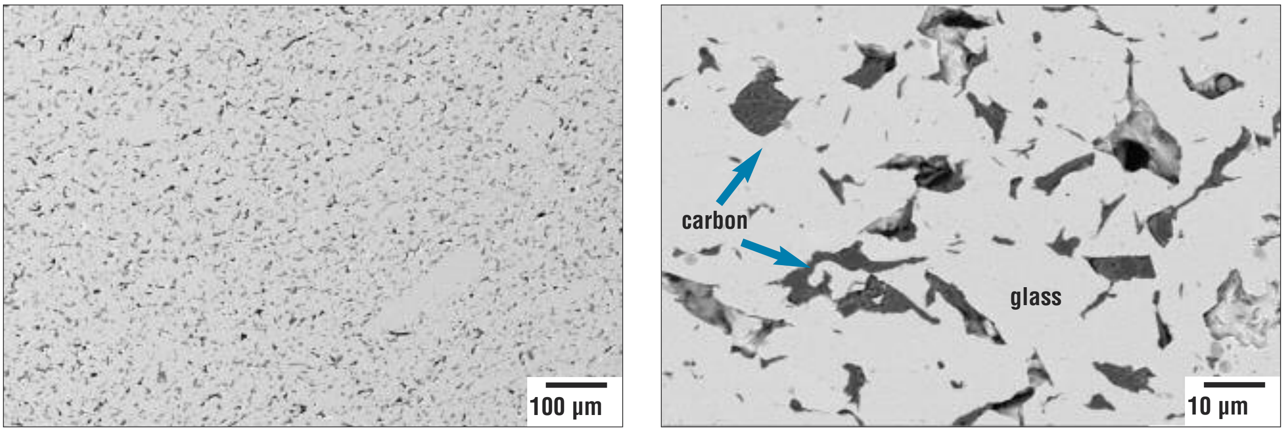

Fig. 4
SEM images of a sintered glass-carbon-composite (glass 8470 with 5 mass-% Timrex T15) showing the microstructure with
an electrical conducting and percolating network of graphite platelets

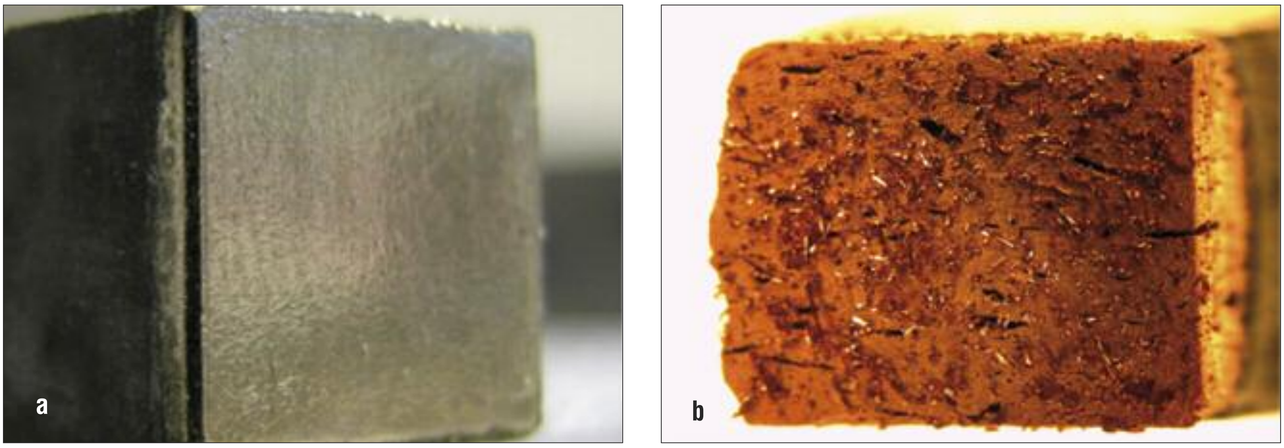

Fig. 5
Sintered glass-carbon-composites with metallic electrical contacts based on a galvanic nickel layer (a), and a cold flame sprayed copper layer (b)

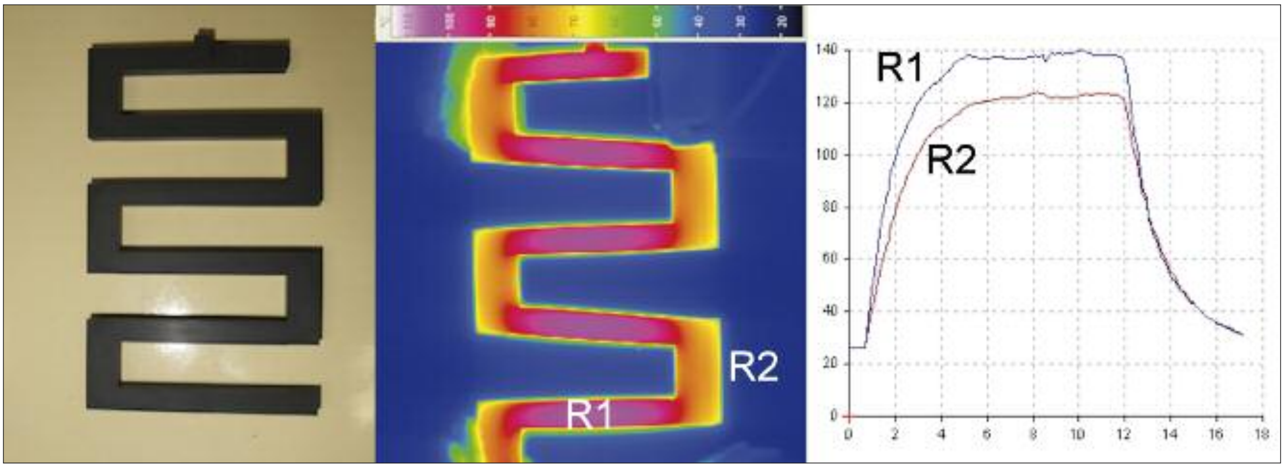

Fig. 6
IR camera image of the heated sintered glass component and heating curves for two locations

8470 and 8250, respectively, and a carbon content of 5 mass-%. Relative sintered densities of more than 90 % could be achieved. Two different injection molded demonstrator components in the green and in the sintered state can be seen in Fig. 3. They illustrate the geometrical complexity realized by PIM including sharp edges without mechanical reworking.

Fig. 4 shows cross-sections of a carbon-containing sintered glass component. The graphite particles are embedded in the glass matrix. Despite of the presence of carbon which usually suppresses sintering, the glass particles had joined with neighboring glass particles and formed the stable matrix with only little remaining porosity. The graphite phase established a three-dimensional framework above the percolation limit which gives the composite electrical conductivity.

When an electrical current is driven through the sintered glass-carbon-composites, the components are heating up due to ohmic losses inside the microstructure. This effect is the basis for the application of this new class of materials as glass-based heating elements. Testing components manufactured by injection molding (Fig. 6) were contacted and exposed to an electrical current. Low ohmic electrical contacts can be realized by sputtering of metallic layers, by galvanic coatings (i.e. nickel as shown in Fig. 5a) or cold flame spraying of copper (Fig. 5b). It is pointed out that the electrical contacts represent the only metallic components in these heating elements.

For testing the heating characteristics of glass-carbon-components, a constant amount of electrical power input was applied and the time-temperature-profiles were recorded by an IR imaging camera. As an example, it was possible to reach a maximum value of 140 °C at 14 W for a glass-carbon-composite having a resistivity of 0,7 Ω cm at 25 °C. An IR camera image of the heated glass component in comparison to the real component and a corresponding heating profile are shown in Fig. 6. The produced heat depends on the local cross-section of the component varying between 4,5 mm × 4,5 mm for the long (R1), and 6,3 mm × 4,5 mm for the short (R2) branches.

For the demonstration of the potential of glass injection molding for manufacturing

Fig. 7
Injection molded micro-reactor component made of carbon-free and electrically conductive sintered glass, and Y-channel structure with inlets

of components containing micro-shaped features a special micro-reactor (design provided by *Mikroglas*®) has been chosen. This reactor is regularly manufactured by shaping of a Foturan® substrate as described in the introduction. For injection molding of sintered glass micro-reactors both, electrical conductive and carbon-free glass powder feedstocks were used. Fig. 7 shows the micro-reactor part with a double Y-channel structure and two in- and outlets, respectively. The flatness of the component was detected by 3D coordinate measurement. Geometrical details of the micro-channels were investigated by white light interferometry (Fig. 8).

Channel depth and width was 480 and 340 µm, respectively, in electrical conductive sintered glass components and 470 and 310 µm, respectively, in carbon-free sintered glass micro-reactors. Computed tomography was applied to estimate the edge sharpness. It was found that an edge radius of about 110–140 µm can be realized which is a remarkable demonstration of the detail grade that can be maintained in injection molded glass based components after sintering.

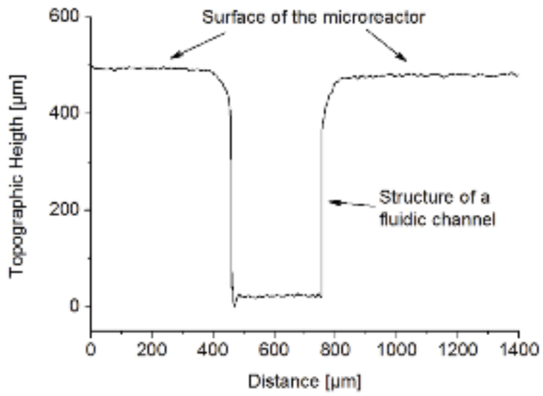

Fig. 8
Profile of channel in the micro-reactor determined by white light interferometry

Conclusion

In the project GlasPIM, powder injection molding has been successfully applied for shaping complex, sharp edged components made of different glass powders. The sintering process has been identified as a critical process in order to minimize an unwanted viscous flow of the glass while preserving microstructured details of the shaped components. Electrically conductive, heatable sintered glass components were prepared by adding certain amounts of graphite powder to the glass powder feedstock. The applicability of sintered glass-carbon-composites as metal-free heating elements was demonstrated successfully by thermoelectrical heating experiments of selected glass-carbon-components. Furthermore, carbon-free sintered glass components could be prepared by injection molding showing a theoretical density of >99 % and a relatively high total transparency in the visible light wavelength range.

Thus, powder injection molding offers a promising opportunity for high efficient large-scale production of different kinds of sintered glass components with complex shape, sharp edges and microstructures for manifold applications. Electrical conductive sintered glass-carbon-composites have been developed as an alternative new material, which is cheap, flexible moldable and nearly metal free for the application as heating elements and possibly also for power resistors. Further effort has to be spent on the optimization of optical quality of sintered glass components and the integration of glass-carbon-components in insulating functional housings.

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