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Silica gel glasses after laser irradiation

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The paper describes a 'combined' sol-gel process allowing us to fabricate large-sized silica glasses. A comparative analysis of the laser thermosplitting of fused and gel silica glasses is also presented. Basic characteristics and main specific features of the silica-gel glasses obtained are given. The distribution of thermoelastic fields in fused and gel-silica glasses after exposing them to laser thermosplitting is analyzed. Regularities in splitting silica glasses by the laser thermosplitting method have been found. To achieve reliable thermosplitting effects and stable dividing microcracks, special conditions are needed, favouring the formation of initiating microcracks.

Key words: sol-gel method, silica glasses, thermosplitting, microcracks

1. Introduction

Theoretical and experimental studies of the problem of splitting brittle non-metallic materials by laser methods are described in a great number of papers. However, new materials recently developed require an adaptation of known technologies to make them applicable for treatment of these materials. Quality of cutting silica glasses, especially gel glasses, is one of the most important problems [1]. This method yields high-purity activated silica glasses for fibre optics, optoelectronics and laser optics at low temperatures eliminating the fusion stage. The sol-gel transition occurs due to polycondensation, hydrolysis and gel formation followed by heat treatment yielding dense gels. Unlike fused glasses, the sol-gel ones contain fewer impurities resulting from the quality of the initial materials. They are also synthesized at lower temperatures. The sol-gel technology is believed to be an energy- and resource-saving process [2]. Its another advantage is simplicity of the necessary equipment.

At present, three directions of the sol-gel technology are employed to produce silica glasses suitable for optical applications. The first variant is the alkoxide method.

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Glass is formed by hydrolysis, polycondensation of products of hydrolysis and heat densification of gels to the state of pore-free glass. The drawbacks of this technique are time-consuming process of synthesis, high probability of cracking when bulks are dried and sintered and small bulk size.

The other technique involves polymerization of colloids of ultradispersed silicate powders in liquid environment. Large-sized gels obtained by this technique can be dried without cracking. Yet, the process requires elevated temperatures of sintering (over 1450 °C) and does not allow one to obtain complex compositions and homogeneously activated glasses.

The third technique is a combination of the two. It comprises the advantages of the former two variants [2]. The initial components are usually ethoxy- and methoxysilanes with aerosils or other ultradispersed powders serving as fillers. The introduction of aerosols into sols increases the concentration of the solid phase in the liquid and the gel strength, facilitates and accelerates the drying of the porous bulks. Vitrification temperatures of such combined systems are usually within the range of 1200–1350 °C, depending, among others, on the filler content. Such systems are most prospective from the viewpoint of industrial application – yet, their properties are poorly understood.

A number of processes occurring during the production of gel glasses and inside the samples formed are not fully understood. A comparative analysis of the regularities of the laser thermosplitting of fused and silica glasses is also an important task.

2. Experiment and materials

The glasses studied were obtained by the 'combined' sol-gel process. The schematic diagram of the process is presented in Fig. 1. Certain characteristics of these glasses are similar to those of fused silica glasses (Table 1) [3]. This is why common methods used to study silica glasses were applied in our case. Bulks of gel glass may have large sizes making them suitable for practical applications. However, their structure on the submicron scale is different than that of fused glasses. The glasses have more chaotic structure and contain more defects.

Table 1. Comparison of characteristic features of gel lasses and fused glasses

Characteristic	Gel glasses	Fused glasses
Density, g/cm^3	2.201	2.206
Specific heat, J/(kg×°C)	1325	880
Heat conductivity, W/(m×°C)	0.7	1.34
Linear thermal expansion coefficient, (1/K)×10^{-7}	5.7	5
Yung's modulus, GPa	7.3	7.8
Poisson's ratio	0.158	0.165
Microhardness, MPa	7000–8500	6860–8850
Refractory index	1.458	1.458

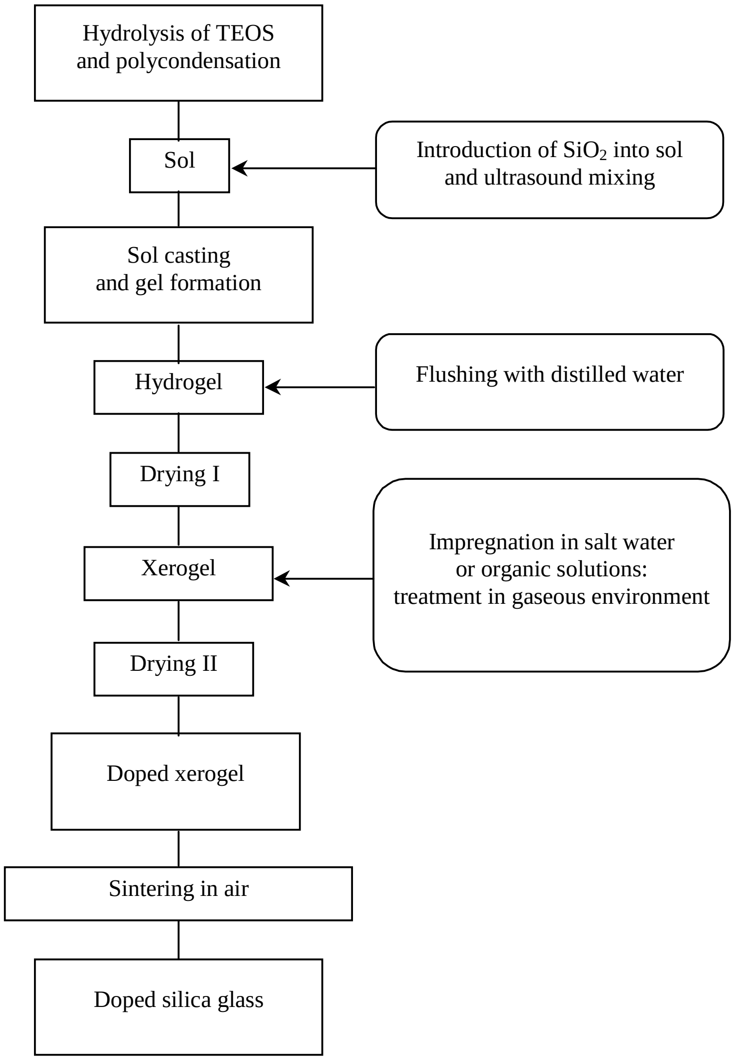

Fig. 1. Schematic diagram of the sol-gel process to obtain silica glasses

Experimental studies of the laser thermosplitting of fused and gel-silica glasses were performed using a set-up comprising the following units: a CO_2 laser with the power of output radiation of 60 W operating in the continuous mode at $\lambda = 10.6$ μm, an *xyz* positioner with the software control, an optical-mechanical device of the radiation energy channel.

Experimental studies have yielded the technology of laser cutting of fused and silica gel glasses. Optimal technological parameters providing the best results are listed in Table 2.

Table 2. Optimal technological parameters for the technology of laser cutting of glasses

Material	Laser beam	Sizes of glasses, mm		Cutting speed, mm/s	Power density of a laser radiation, 10^6 W/m^2
		Thickness	Cross-section		
Fused quartz glass	ellipse A = 4.5 mm B = 1.1 mm	2	30×30	10	5
Silica gel glass	ellipse A = 2 mm B = 0.5 mm	2	30×30	17	3.2

Our studies have shown that the processes of cutting gel and fused silica glasses by laser thermosplitting are similar. This occurs provided that special measures are applied to attain conditions favourable for formation of the initial microcracks. In such a case, preliminary softening of glass due to the generation of a defect zone along the cutting line is inefficient.

The main features of the technology developed are: the supply of the air–water mixture into the cutting zone which acts as a refrigerant and the initiation of the thermosplitting process by glass softening within the region where the refrigerant is supplied.

3. Numerical analysis of thermoelastic fields

The analysis of thermoelastic fields is of practical importance since it allows one to study the features of the mechanism of dividing microcrack formation. Due to difficulties arising when using analytical methods in calculating thermoelastic fields in the non-linear problem formulation, the finite-element method [4] was used.

Thermal characteristics of silica glasses depend to a great extent on temperature while with the laser thermosplitting the temperature of silica glass varies within a broad range. To eliminate large errors in calculations, one should take into account these dependencies. According to literature data [5], we suggest that upon increasing temperature from the ambient value to the vitrification temperature, the glass thermal conductivity increases two times and the specific heat increases by the factor of 1.5. To achieve high-quality glass by laser thermosplitting, the temperature of the specimens to be cut should not exceed the vitrification temperature. Thus, in calculations we have taken into account that the temperature of glass within the region of laser beam action can vary from 20 to 1100 °C.

We used the supply of the cooling air–water mixture into the cutting zone previously heated by a laser beam. The mixture rate was 0.8 m/s [6], which gives the heat transfer coefficient of 6800 W/(m^2×K) used in our calculations.

As the initial size of the region heated depends on the depth of radiation penetration into the material, the model of a surface heat source was applied to simulate the effect of laser radiation. When a CO_2 laser is used to cut silica glasses, this depth is comparable with the radiation wavelength.

To calculate temperature stresses, we employed the formulation of the quasi-static problem implying that the stress state stabilises sufficiently faster than the thermal equilibrium. The error of the values of the temperature stress caused by the neglect of the inertial effects is very small [7].

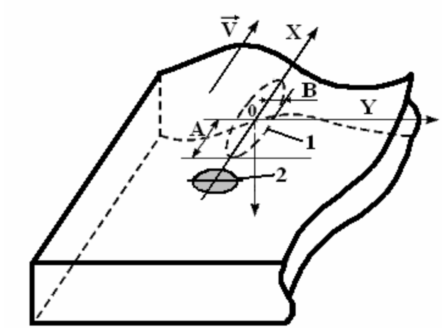

Fig. 2. Scheme of arrangement of the laser beam and cooling agent in a plane of processing: 1 – elliptical laser beam, 2 – cooling agent

Figure 3 shows the temperature fields on the surface of the specimen during cutting. The solid lines are isotherms corresponding to the temperature values shown in the plots. The maximum surface temperature of gel silica glass reaches 1152 °C. Thus, the brittle fracture mode occurs in the specimens.

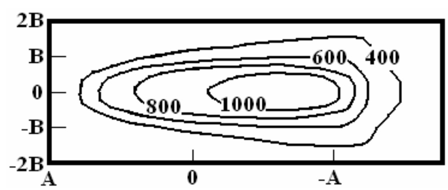

Fig. 3. Distribution of temperature fields (in °C) on the surface of a quartz glass synthesized by the sol-gel method

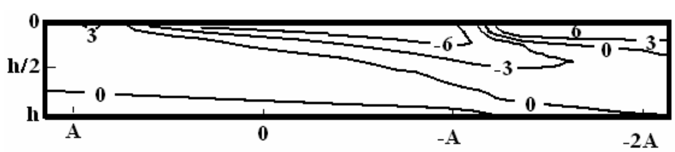

Fig. 4. Distribution of fields of σ_{22} stresses (in MPa) in a plane of splitting of a silica glass synthesized by the sol-gel method

In Figures 4 and 5, the distribution of fields of stresses (σ_{22}) which give the largest contribution to the development of the dividing microcracks in the splitting plane of the silica glass obtained by the sol-gel method is shown. One can conclude that the maximum values of σ_{22} occur on the sample surface in the zone of action of the cooling agent. These values do not exceed 10 MPa. That is not enough for splitting the sample without formation of the initial microcrack. Thus, as has been mentioned above, the necessary requirement of the reliable thermosplitting process is initializing of the dividing microcrack in the zone of action of the cooling agent.

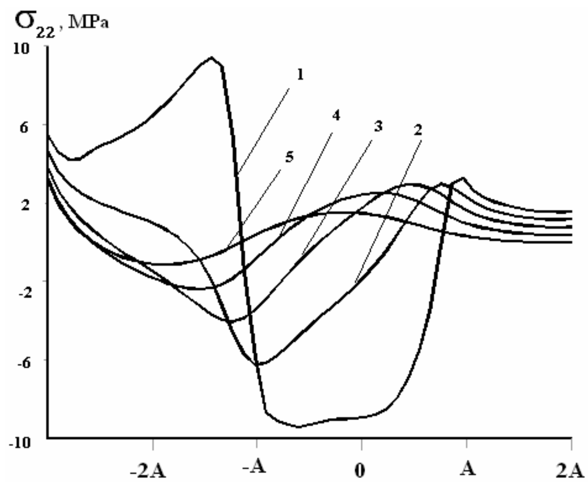

Fig. 5. Dependence of stresses σ_{22} (in MPa) to spacing interval up to the centre of an elliptical laser beam in silica glass synthesized by the sol-gel method (Y = 0 mm), 1 – Z = 0, 2 – Z = 0.3 mm, 3 – Z = 0.6 mm, 4 – Z = 0.9 mm, 5 – Z = 1.2 mm

A further development of the crack is governed by the distribution of compressive stresses caused by the laser beam. The zones of location of these stresses stretch around the zone of tensile stresses resulting from the cooling agent action. This enables division of the silica glass in the controlled thermosplitting mode involving the first stage of the sample damage yielding high quality of surfaces of articles being processed.

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