Sintering, crystallization and foaming of La$_2$O$_3$·SrO·5B$_2$O$_3$ glass powders - effect of the holding temperature and the heating rate

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**ABSTRACT**

The peculiarities of sinter-crystallization of La$_2$O$_3$·SrO·5B$_2$O$_3$ fine glass powder (below 1 μm) are investigated and discussed. The sintering was studied by contactless optical dilatometry and porosity measurements. The crystallization process was evaluated by differential scanning calorimetry (DSC) and the crystalline phase formation was analyzed by X-ray diffraction (XRD). Finally the microstructures of the samples sintered at different temperatures were observed by field emission scanning electron microscope (FESEM).

The specific aim of this study was to highlight the influence of sintering temperature and heating rate on the densification process and the final structure of glass-ceramic samples. It was shown that zero water adsorption and minimum closed porosity of ~3% were obtained at heating rate of 2 °C/min and at about 1 h holding at 690 °C. The temperature increase accelerated intense foaming process that resulted in formation of total porosity of 35–45%. It was also demonstrated that the foaming depended on the heating rate: the higher the heating rate – the more intensive was the bloating. The observed foaming process was related to intensive crystallization and re-crystallization of La$_2$O$_3$·SrO·5B$_2$O$_3$ crystal phase. The phase formation processes entirely completed after 30–60 min at 730–740 °C with the formation of intriguing and unusual crystal structures.

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- Structure

**1. Introduction**

One of key advantages of the glass-ceramics is the possibility to create innovative materials with various combinations of properties and different microstructures, depending on the initial glass composition and the applied heat-treatment [1–3]. The majority of glass-ceramics is obtained by controlled bulk crystallization of glasses with original chemical compositions. However, since most of the glass forming systems crystallize on the surface, it is necessary to add the appropriate nucleating agents, which can initiate the bulk crystallization. This ad-

An alternative method for glass-ceramic production is the simultaneous sintering and crystallization of the glass powders. This technique is suitable especially for compositions with problematic bulk nucleation. In this case, the crystallization process begins on the surface of the sintering glass particles while the surface defects act as nucleation centers. The main advantages of this technique are that the addition of nucleating agent in the batch is not necessary and that the products with complicated shapes could be manufactured. In addition, the melting time might be significantly reduced since a very high degree of refining of the parent melt is not necessary.

This method, known as sinter-crystallization, implies that the densification and the crystallization occur in similar temperature intervals, so that each of these processes might have influence on the other. When sintering completes before the actual beginning of the crystallization, it might be assumed that the densification is controlled by viscous flow [4–6]. However, usually both processes take place simultaneously and the densification of the glass powder might be hindered by the crystalline phase formation [7,8]. At the same time, the phase formation trend might be influenced by the densification because during sintering the number of surface defects might decrease.

By employing sinter-crystallization it is possible to obtain glass-ceramics for a wide range of applications: for building and construction [1,2,8], materials for electronic industries, solders, special coatings and cements [1,2], as well as new products for dental or other medical
purposes [1,2,9–11]. The samples might also have different microstructures (dense or foamed materials) with nano, micron or even millimeter glass powder sizes and/or crystals dimensions.

Lanthanide alkaline borate glasses showed improved durability and relatively high glass-transition temperatures, so therefore similar glass-ceramics could be used as specialized hermetic sealers [12]. These glasses also could have application in various electronic industries as display panels, low-temperature co-fired ceramics and packaging where low-temperature sintering of glassy or glass-ceramic powders is required [13,14].

In the presented study the sinter-crystallization of glass powder with molar composition \( \text{La}_2\text{O}_3\cdot\text{SrO}\cdot5\text{B}_2\text{O}_3 \) was investigated. The formed crystal phase (i.e. \( \text{La}_2\text{O}_3\cdot\text{SrO}\cdot5\text{B}_2\text{O}_3 \)) showed good potential as the laser hosts and nonlinear optical materials and usually it is synthesized by conventional solid state reaction techniques [15].

This stoichiometric composition was studied together with other \( \text{La}_2\text{O}_3\cdot\text{SrO}\cdot\text{B}_2\text{O}_3 \) glasses in previous works [16,17]. It was featured that it forms only \( \text{La}_2\text{O}_3\cdot\text{SrO}\cdot5\text{B}_2\text{O}_3 \) as a crystal phase (i.e. total crystallization is possible) and that the crystallization is surface induced. It was also shown that, when glass powders below 63 \( \mu \)m were tested for sinter-crystallization, the densification process is partially inhibited by the intensive phase formation.

In this work new research on sinter-crystallization of a very fine fraction (below 1 \( \mu \)m) of \( \text{La}_2\text{O}_3\cdot\text{SrO}\cdot5\text{B}_2\text{O}_3 \) glass is presented. The sintering process and the phase formation are studied in detail by various techniques, while the microstructures of samples, heat-treated at different temperatures, are observed by field emission scanning electron microscope (FESEM). The primary aim of the presented work is to highlight the influence of the sintering temperature and the heating rate on the densification process and glass-ceramics structure.

In the next part of this research, which will be presented in the near future, unusual results for the variation of the porosity and the glass-ceramic structure during the crystallization process will be presented and discussed.

2. Experimental

The mixture of the reagent-grade of \( \text{H}_2\text{BO}_3 \), \( \text{SrCO}_3 \) and \( \text{La}_2(\text{CO}_3)_3 \) was used to prepare batch for glass making composition as follows: 14.3 mol\% of \( \text{La}_2\text{O}_3 \), 14.3 mol\% of \( \text{SrO} \) and 71.4 mol\% of \( \text{B}_2\text{O}_3 \). The batch was placed in a platinum crucible and the glass was melted at 1200 °C for 30 min in an electric furnace. In order to minimize the boron evaporation during the melting, the platinum crucible was covered with platinum plate and the melting was carried out for a short time at relatively low melting temperatures. The melt was cast and cooled on a stainless steel plate in air at room temperature. Thus obtained glass was transparent and without visible bubbles.

The initial glass powder was obtained by crushing and grinding in an agate mortar and preliminary sieving (below 2 mm). Thus obtained glass grains were milled for 90 min at 400 rpm in SFM-1 DESK-TOP PLANETARY BALL MILLE. The final particle size distribution was acquired by a Zetasizer Nano ZS device (Malvern Instruments, U.K.) in the dispersion of obtained fine glass powder which was made by mixing with deionized water in an ultrasonic bath for 2 min.

The sintering process was studied in details using horizontal optical dilatometer (Misura – HSML ODLT 1400, Expert System Solutions, Italy). The initial green samples (50 mm × 5 mm × 3–4 mm) were obtained by mixing the glass powder with 7% PVA (polyvinyl alcohol) solution and subsequent uniaxial pressing at 40 MPa using hydraulic press (Nannetti · Italy). The addition of PVA increases the green strength and decreases the initial porosity, but a preliminary burn-out step at 280 °C was necessary before the sintering experiments. Thus obtained samples were isothermally sintered for 1 h at different temperatures using various heating rates. The sintering conditions of the samples are summarized in Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heating rate, °C/min</th>
<th>Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-680</td>
<td>2</td>
<td>680</td>
</tr>
<tr>
<td>2-690</td>
<td>2</td>
<td>690</td>
</tr>
<tr>
<td>2-700</td>
<td>2</td>
<td>700</td>
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<tr>
<td>5-700</td>
<td>5</td>
<td>700</td>
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<td>10-700</td>
<td>10</td>
<td>700</td>
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<tr>
<td>30-700</td>
<td>30</td>
<td>700</td>
</tr>
<tr>
<td>10-720</td>
<td>10</td>
<td>720</td>
</tr>
<tr>
<td>10-740</td>
<td>10</td>
<td>740</td>
</tr>
</tbody>
</table>

Table 1. Samples labels and sintering conditions (1 h holding time).

The apparent sample densities, \( \rho_{ap} \), were calculated by precise measurement of their volume and weight (using electron gauge and balance, respectively). Their skeleton densities, \( \rho_s \), were measured by gas (Ar) pycnometer (AccuPy 133, Micromeritics, USA). In addition, the water soaking, \( W \), was measured after standard test in boiling distilled water for 3 h. The results were used to estimate the open, \( P_O \), and the closed, \( P_C \), porosities using following relations:

\[
P_O = \frac{W \times \rho_s}{\rho_a}
\]

(1)

\[
P_C = \frac{100 \times (\rho_a - \rho_s) / \rho_a}{100}
\]

(2)

In Eq. (2) \( \rho_{ap} \) is the absolute density of the parent glass powder, measured by gas (Ar) pycnometer.

The crystallization behavior was studied by DSC analysis (SDT Q600 TGA/DS, TA Instruments, USA) with Al₂O₃ as the referent material. Glass powder samples of about 10 mg were heated up to 900 °C at 20 °C/min after 30 min holding at the following temperatures: 690, 700, 720 and 740 °C in a dry air atmosphere (flow rate 100 cm³ min⁻¹). These results were compared to the sample heat-treated non-isothermally without preliminary step. The heating rate of 20 °C/min was selected in order to increase the accuracy of the crystallization enthalpy evaluation.

XRD technique was used to determine the crystalline phase compositions of crushed and milled samples. The XRD patterns were obtained using a Philips PW-1710 (PANalytical, Netherlands) automated diffractometer with a Cu tube operated at 40 kV and 30 mA. The instrument was equipped with a diffracted beam curved graphite monochromator and a Xe-filled proportional counter. The diffraction data were collected in the 2θ Bragg angles from 5 to 70° at every 0.02° step, counting for 1 s. The divergence and scattering slits were fixed 1 and 0.1, respectively. All of the XRD measurements were performed at room temperature.

The microstructures of the obtained glass-ceramic were observed by FESEM (Mira Tescan, Czech Republic). The samples were crushed and gold sputtered, so the surface and the fractured surface were analyzed by FESEM (operated at 20 keV, using BSE and SE detectors).

3. Results and discussion

3.1. Sintering and porosity

The size distribution of the particle after the milling is shown in Fig. 1. According to these results the average particle diameter is approx. 700 nm. This particle size practically is inferior with two orders of magnitude to the preliminary results at low temperatures (680, 690 and 700 °C) when heating rate of 2 °C/min was used, there is none or negligible crystallization. In the crystallization interval (700, 720 and 740 °C), where substantial and even very intensive crystallization occurs during the holding step, heating rate of 10 °C/min was applied. Using stated procedure two samples denoted 2-700 and 10-700 were obtained after 1 h sintering at 700 °C (see Table 1).
The dilatometric results at low temperature sintering, shown in Fig. 2, demonstrated that after 1 h at 680 °C the densification was not completed. After 55–60 min at 690 °C the sintering rate diminished to zero and the linear shrinkage reached ~13.7%. The same shrinkage value was obtained after 15–20 min at 700 °C. Subsequently, slow expansion of sample 2-700 was observed, leading to a decrease in the linear shrinkage to ~13.3% after 1 h step.

The sintering results at 10 °C/min (see Fig. 3) demonstrated that the densification at 700 °C completed after 10–15 min holding (i.e. similarly to sample 2-700), while the maximum shrinkage at 720 °C was reached after only 2–3 min. In the sample 10-740 the densification completed during the heating stage at about 730 °C. After densification had finished, “foaming” process started in those three samples with increasing rate following the rise of the holding temperature. The “foaming” effect however stopped after 30–40 min holding time at 720 °C and after 5–7 min at 740 °C, at shrinkages of about 5 and 3%, respectively. This behavior highlights an interesting connection between the phase formation and the bloating.

This relationship will be further studied in details in the second part of our work. Hot stage microscopy (HSM) and simultaneous thermal analysis (DTA-TG) results in the temperature interval between T_g and T_m will be presented. New XRD, pycnometric, dilatometric and FESEM results for the variation of the structure at 700 °C up to 15 h holding times will be shown. The probable reasons for the foaming phenomenon and its subsequent inhibition during the crystallization will be debated.

Thus obtained samples were used also to measure the skeleton density, the apparent density and the water soaking. The results are summarized in Table 2, while the related variations of open and closed porosities vs. the holding temperatures are presented in Fig. 4.

The porosity results entirely confirm the data presented in Figs. 2 and 3. After 1 h heat-treatment at 680 °C the sintering was uncompleted...
and the porosity was mainly open. At 690 °C no open porosity was observed and the closed porosity was ~3%, which is the minimum value for all the samples. After 1 h at 700 °C the formation of additional closed porosity (~2–3%) was measured, while at 720 and 740 °C, due to intensive bloating, the total porosity increased drastically. Temperature increase resulted in transformation of open to closed porosity: at 720 °C both open and closed pores were present, while at 740 °C the porosity was mainly open.

It is interesting to note that sample 10-700 had slightly higher porosity than the sample 2-700, which indicates that the final degree of densification depends also by the heating rate. In order to highlight the effect of the heating rate additional dilatometric experiments at 700 °C were performed using heating rates of 5 °C/min and 30 °C/min. The dilatometric and the porosity results for the samples 2-700 and 10-700 and newly obtained specimens 5-700 and 30-700 are summarized in Table 2.

### Table 2

<table>
<thead>
<tr>
<th>Sample</th>
<th>Skeleton density (g/cm³)</th>
<th>Apparent density (g/cm³)</th>
<th>Water soaking (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial glass powder</td>
<td>3.576 ± 0.002</td>
<td>2.8 ± 0.1</td>
<td>4.1 ± 0.1</td>
</tr>
<tr>
<td>2-680</td>
<td>3.539 ± 0.002</td>
<td>3.5 ± 0.1</td>
<td>0</td>
</tr>
<tr>
<td>3-680</td>
<td>3.487 ± 0.002</td>
<td>3.6 ± 0.1</td>
<td>0</td>
</tr>
<tr>
<td>2-700</td>
<td>3.388 ± 0.002</td>
<td>3.4 ± 0.1</td>
<td>0</td>
</tr>
<tr>
<td>5-700</td>
<td>3.381 ± 0.002</td>
<td>3.4 ± 0.1</td>
<td>0</td>
</tr>
<tr>
<td>10-700</td>
<td>3.307 ± 0.002</td>
<td>3.3 ± 0.1</td>
<td>0</td>
</tr>
<tr>
<td>30-700</td>
<td>3.187 ± 0.002</td>
<td>3.2 ± 0.1</td>
<td>0.2 ± 0.02</td>
</tr>
<tr>
<td>30-720</td>
<td>2.594 ± 0.003</td>
<td>2.5 ± 0.1</td>
<td>7.9 ± 0.2</td>
</tr>
<tr>
<td>10-740</td>
<td>3.331 ± 0.003</td>
<td>1.6 ± 0.2</td>
<td>20.2 ± 0.4</td>
</tr>
</tbody>
</table>

The plots in Fig. 5 elucidate the obvious result that the degree of densification, attained during the heating stage, decreased with the heating rate increase: the sintering isotherm at 2 °C/min starts at shrinkage of about 8%, while the one at 30 °C/min begins at 2% shrinkage.

The maximum reached shrinkage also decreased with the heating rate, which was not expected. In fact, for the sintered glass-ceramics usually the densification is favored at higher heating rates [7,18]. In our case, in the sample 2-700 the maximum shrinkage of ~13.7% was reached after 15–20 min holding time, while in the 30-700 the maximum shrinkage was only ~11.4% and this value was reached after shorter holding times of 3–5 min. This strong effect probably is also a result of the “overshooting” of 6–7 °C, which occurs at the highest heating rate of 30 °C/min. In the others samples this effect is not so obvious.

Other interesting detail was that the “foaming” effect also depended on the heating rate: at 2 °C/min the shrinkage decrease after 1 h holding at 700 °C was ~0.4%, while at 30 °C/min it was ~1.6%.

The results for open and closed porosity of these four samples again confirmed the dilatometric tests and highlighted that the increase in closed porosity occurred with rising heating rate from ~3% in the sample 2-700 to ~11% for the sample 30-700. For the last sample even started the formation of some open porosity (~0.6%).

These results could be explained assuming that the densification of used very fine powders practically completed before the beginning of crystallization. In this case, the usage of higher heating rates and/or higher holding temperature leads to densification process at lower viscosity. As a result, an earlier “sealing” of the surface carries out which leads to an entrapment of larger amount of glasses and higher porosity. During the crystallization probably an additional amount of gas is formed which leads to the “foaming” effect. The higher the holding temperature, the lower is the viscosity and more intensive is the bloating. Similar results for sinter-crystallization of nano glass particles are presented and discussed also by other authors [19].

### 3.2. Phase formation and structure

In the first step, the phase formation was estimated by DSC analysis (plot is shown in Fig. 7). The glass transition range was 650–660 °C, indicating that the sintering process at 680–690 °C, carried out at very high viscosities. This is typical for nano-sized glassy particles [19,20], because at viscous flow sintering the densification rate increases linearly with the decrease of particle size [4]. The onset of the crystallization exo-peak and its maxima were at 755 and 784 °C, respectively, whereas the melting temperature was observed at about 950 °C.

In order to better elucidate relation between the processes of...
sintering and phase formation, DCS was additionally used in order to estimate the degrees of transformation reached after 30 min holding time at different temperatures. For this purpose, additional runs at heating rate of 20 °C/min were made after isothermal heat-treatments at 690, 700, 720 and 740 °C. Thus obtained exothermic plots are presented in the inset of Fig. 7. The crystallized fraction, α, formed during the isothermal step, is estimated based on Eq. (3), as previously described in the literature [21]:

\[ \alpha = 1 - \frac{A}{A_h} \]  

(3)

where \( A_h \) is the normalized area of the exothermic peak of parent glass sample and \( A \) is the area of the exothermal peak after isothermal thermal treatment. The results for crystallization peak temperatures, \( T_p \), enthalpies of crystallization (i.e. the normalized areas) and obtained \( \alpha \) values are shown in Table 3.

### Table 3

<table>
<thead>
<tr>
<th>( T_p ) (°C)</th>
<th>( \Delta H ) (J g(^{-1}))</th>
<th>( \alpha )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial glass powder</td>
<td>782</td>
<td>231</td>
</tr>
<tr>
<td>690 °C</td>
<td>781</td>
<td>226</td>
</tr>
<tr>
<td>700 °C</td>
<td>779</td>
<td>205</td>
</tr>
<tr>
<td>720 °C</td>
<td>764</td>
<td>88</td>
</tr>
<tr>
<td>740 °C</td>
<td>–</td>
<td>–</td>
</tr>
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</table>

These results demonstrate that the degree of crystallization after 30 min at 690 °C is insignificant and the peak temperature is practically the same as the one obtained for the parent glass powder. After temperature holding at 700 °C about 13% decrease in the enthalpy was observed and the peak temperature shifted by 3 °C. At 720 °C the degree of transformation reached approx. 60% and the peak temperature of residual crystallization exothermic plot decreased by 18 °C. Finally, the results of 740 °C run showed that the phase formation completed for a time period shorter than 30 min.

The XRD spectra of the samples 2-680, 2-690, 10-700, 10-720 and 10-740 are summarized in Fig. 8. In general, these results confirm the DSC data. After 1 h holding at 680 and 690 °C, the crystallinity of the XRD spectra is practically identical to the parent glass results. This proves that the crystallization rate for this temperature range is very low and justifies the statement that the sintering process of used fine powders is related to viscous flow mechanism. The spectrum for the sample 10-700 reveals \( \text{La}_2\text{O}_3\cdot\text{SrO}\cdot5\text{B}_2\text{O}_3 \) crystallization behavior supporting the hypothesis that the slow bloating effect at this temperature is related to the beginning of phase formation. These results agree well with the dilatometric data that declared the bloating at 700 °C started after 10–20 min. Also, DSC results confirmed the previously stated by showing a low degree of transformation after 30 min step at this temperature.

It can be also noted that, regardless of the different heating rates all XRD spectra of the samples for 1 h temperature hold at 700 °C (i.e. 2-700, 5-700, 10-700 and 30-700) are very similar. This indicates that the intensity of the bloating during the holding periods depends also on the initial amount of trapped gas in the samples. Higher values of maximum sintering shrinkage (see Fig. 6) result in lower residual porosity values and less intense is the subsequent bloating effect.

Finally, the spectra of the samples 10-720 and 10-740 showed very high crystallinity. The results are in agreement with the DSC data, stating that for 30 min at 720 °C the degree of transformation is approx. 0.6. Based on the data, complete phase formation could be reached for 30–60 min at 730–740 °C.

The structures (both surface and fracture) of the samples 2-680, 2-690, 10-700, 10-720 and 10-740 were observed by FESEM. These investigations clarify the data obtained by the other methods, and partially explain the interesting volume variations accompanying the phase formations.

In Fig. 9 typical images of the amorphous samples 2-680 and 2-690 are shown. Fig. 9-a and -b present the surface of the sample, heat-treated at 680 °C, at low and high magnifications, respectively. The
Fig. 9. SEM images of samples 2-680 (a - surface and b - surface) and 2-690 (c - fracture).

Fig. 10. SEM images of samples 2-700 (a - surfaces) and 2-700 (fracture - b and c).
densification was not completed and several open pores were still present on the sample surface. It is interesting to note that the surface is not homogeneous: along with well separated 1–2 micron particles, connected by typical necks, well sintered areas up to 10–20 μm (which is a significantly bigger size than the one of the parent glass powders) are observed. This non-homogeneity, which also is observed in the fracture of the sample 2-680, might be explained by the “cluster” model of the glass particle sintering [22]. In fact, this approach proposes a faster sintering of clusters, composed by finer particles, and predicts the formation of bigger size pores in the final stage of densification.

Images 9-c demonstrates the fracture of the sample 2-690 and elucidates that the residual porosity in the volume is minimal, which is in excellent agreement with the pycnometric results for ~3% closed porosity. In accordance with the XRD data – no evident phase formation was identified. The surface of this sample is smooth and completely sintered.

According to the DSC and XRD results, in the samples sintered for 1 h at 700 °C, some crystallization already occurred. As a consequence, the dilatometric and pycnometric data indicated the start of bloating, which led to some increase in the porosity. It was also shown that higher is the heating rate used, more intensive is this effect. These features were well established by the SEM observations. Typical image of the surface of sample 10-700 is shown in Fig. 10-a. It demonstrates the beginning of some surface bloating, as well as the formation of new tiny open pores, which are not observed in sample 2-700 (i.e. at lower heat rate). Fig. 10-b and -c show images of the sample 2-700 fracture at different magnifications. In the first photo, the increase in closed porosity is well illustrated, while in the second - the formation of some crystal phase is well distinguished.

The increase in holding temperature and the related intensive crystallization entirely changes the structure and the morphology of glass-ceramic. An image from the surface of sample 10-720, showing the formation of huge amount of open and closed pores with different sizes, is presented in Fig. 11-a, while the fracture of this sample is shown in the Fig. 11-b. The observation at higher magnification demonstrates not only the increased porosity in the bulk, but also highlight that two quite different structures were formed. This particularity is elucidated in Fig. 11-c. The first structure is “sponge-like” and is characterized by the presence of different spherical closed pores of micro- and nano-size (in the left side of the image); the second one is denser and presents 5–10 microns crystal structures, formed by “sheets” of 10–30 nm thickness (in the central part of the image).

The structure of glass-ceramic 10-740 is similar to 10-720. However, it might be noted that the size and the amount of formed open canal pores is bigger, which is in good agreement with the lower apparent density and higher water soaking (see Table 2).
that the volume and the dimensions of the “sheet” crystal structures also increase. A typical image of this morphology is presented in Fig. 11-d. Since the XRD results confirm the formation of a single crystal phase, corresponding to the parent glass composition (i.e. La₂O₃·SrO·5B₂O₃), these transformations might be related to the intensive re-crystallization process. In fact, the formed final crystal structures have dimensions several times larger than the parent glass powder sizes.

The properties of studied sinter-crystallization process eventually might be used for the development of various materials: from low temperature compact amorphous soldering materials to nonlinear optical glass-ceramics with controlled porosity [13–15].

4. Conclusions

The sintering and the crystallization pattern of La₂O₃·SrO·5B₂O₃ glass powders with the average particle diameter of 700 nm are studied by various techniques.

Notwithstanding of the high crystallization trend, the sintering of used fine particles completes before the beginning of phase formation. As a result, well-sintered amorphous samples with negligible closed porosity of ~3% could be obtained at heating rate of 2 °C/min and one hour holding at 690 °C. The subsequent crystallization led to the beginning of the foaming, which intensity drastically increased with the rise of holding temperature. When heat treatment at 740 °C was applied, completely crystallized glass-ceramics with huge open porosity of ~35% were formed. It was also demonstrated that the degree of densification decreased with the heating rate increase (i.e. lower the heating rate, lower was the formed porosity).

The foaming process is related to intensive crystallization and re-crystallization of La₂O₃·SrO·5B₂O₃ crystal phase. During the phase formation two quite different structures are formed: the first is “sponge-like” and is characterized by the presence of different spherical micro and nano sized closed pores; the second is denser and presents 5–10 micron crystall structures, formed by “sheets” of 10–30 nm thickness.

In the next part of this research, additional results elucidating the variation of porosity and glass-ceramic structure during the crystallization process will be presented and further discussed.

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References