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Study of crystallization, microstructure and mechanical properties of lithium disilicate glass-ceramics as a function of the sintering temperature

Estudo da cristalização, microestrutura e propriedades mecânicas de uma vitrocerâmica de dissilicato de lítio em função da temperatura de sinterização

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ABSTRACT

Objective: The purpose of the present study was to synthesize and characterize lithium disilicate glass-ceramics through the Li_2O-SiO_2 system for determining the most satisfactory sintering parameter by evaluating the crystalline composition, microstructure and mechanical properties. **Material and methods:** The glass-ceramics were prepared from a glass precursor by means of the melting/cooling technique with a composition of 33.33 Li_2O and 66.67 SiO_2 (mol.%). The specimens were compressed by the uniaxial pressing technique and three different thermal treatments were used for sintering: 850 °C (Group 1), 900 °C (Group 2), and 950 °C (Group 3), which were determined based on the differential scanning calorimetry (DSC) result. The glass-ceramics were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Archimedes method, microhardness and biaxial flexural strength analyses. **Results:** The results regarding XRD predominantly showed lithium disilicate phase for all the heat treatments performed. Moreover, grains with a needle form were more predominantly observed in the SEM images for Group 3, as well as a higher densification and consequently higher mechanical properties. In contrast, Group 1 presented the lowest mechanical properties and

RESUMO

Objetivo: O presente estudo teve como objetivo sintetizar e caracterizar uma vitrocerâmica de dissilicato de lítio através do sistema Li_2O-SiO_2 para determinar o parâmetro de sinterização mais satisfatório através da avaliação da composição cristalina, microestrutura e propriedades mecânicas. **Material e Métodos:** As vitrocerâmicas foram preparadas a partir de um vidro precursor pelo método fusão/resfriamento utilizando a composição de 33.33 Li2O e 66.67 SiO_2 (mol.%). As amostras foram prensadas utilizando uma técnica de prensagem uniaxial e três tratamentos térmicos diferentes foram utilizadas para sinterização: 850 °C (Grupo 1), 900 °C (Grupo 2), e 950 °C (Grupo 3), que foram determinados através do resultado da análise diferencial de calorimetria. As vitrocerâmicas foram caracterizadas através das análises de difração de raios X (DRX), microscopia eletrônica de varredura (MEV), métodos de Arquimedes, microdureza e ensaio de flexão biaxial. **Resultados:** Os resultados de DRX mostraram predominantemente a fase de dissilicato de lítio para todos os tratamentos realizados. Além disso, grãos com forma agulhada foram mais predominantemente observados por MEV no grupo 3, assim como uma densificação maior e consequentemente valores maiores das propriedades mecânicas. Em contraste, o grupo 1 apresentou os menores valores de propriedades mecânicas e densificação, e também densification, as well as the highest porosity. **Conclusion:** The present study demonstrated how extremely important it is to follow the heat treatment recommended by the manufacturers of ceramics, including time and temperature, which possess direct effects in the crystalline phase formation, as well as in the material's microstructure and mechanical properties.

KEYWORDS

Crystallization; Glass-ceramics; Lithium disilicate.

a maior porosidade. **Conclusão:** O presente estudo demonstrou como é extremamente importante seguir o tratamento térmico recomendado pelos fabricantes de cerâmica, incluindo tempo e temperatura, que possuem efeitos diretos na formação da fase cristalina, assim como na microestrutura do material e propriedades mecânicas.

PALAVRAS-CHAVE

Cristalização; Vitrocerâmicas; Dissilicato de lítio.

INTRODUCTION

There has been an increase in research in recent years related to ceramic materials as a biomaterial often used in oral rehabilitations due to their versatility in replacement function and form to the dental element [1–4]. In the range of ceramics commercially available, lithium disilicate (Li2Si2O5) glass-ceramic has received a great deal of interest predominantly due to its mechanical properties and high potential to be used as dental materials [5,6].

Glass-ceramics are normally obtained by the crystallization of suitable glass through controlled heat treatments, which transforms the vitreous parent glass into a uniform microcrystalline ceramic after internal or external nucleation [7]. Li_2O-SiO_2 is a binary system, which has been widely studied involving various fabrication methods; one such method is through the sintering of glass frits [3,5,8], which can form lithium disilicate phase after a controlled and slow crystallization process. Understanding and controlling glass crystallization mechanisms has great scientific and technological importance, as it allows internal regulation as well as oriented development of the grains without producing micro-cracks or porosity, thereby providing satisfactory mechanical strength and translucency which are paramount features for glass-ceramics [2].

Some studies have reported that the resulting crystal form and phase after the sintering process has a paramount influence on the crystalline structure and mechanical properties achieved by the material, which are dependent on the composition of the parent glass as well as on the sintering temperature and time [6,9,10]. In addition, it has been reported that the final temperature has a major impact on the sintering process and has therefore gained much attention [11]. In order to achieve a lithium disilicate phase, the sintering temperatures in previous studies were less than 900 °C [3,5,12,13], which is much lower than the melting temperature (T_m), while the likely consequences of a higher final temperature for the binary system are still not well known.

In view of the above, this study aimed to synthesize and to determine the most satisfactory sintering treatment to obtain an experimental lithium disilicate glass-ceramic from the binary Li_2O-SiO_2 system (33.33 Li_2O.66.67 SiO_2, mol%). The crystallization temperature varied from 850 °C to 950 °C, which is near Tm, aiming to disclose the effects of these different temperatures on crystallization, microstructure and mechanical properties of the studied glass-ceramics.

MATERIAL AND METHODS

Preparation and characterization of SiO_2-Li_2O glass

First, Li_2CO3 and SiO_2 (Sigma-Aldrich, Brazil) powders were weighed in a stoichiometric composition (33.33 Li_2O.66.67 SiO_2, mol%), mixed in a mill (Marconi, MA 500, Brazil) for 2 h for homogenization. Next, the mixture was introduced into a ZAS crucible (zirconia-alumina-silica) and melted in a laboratory furnace (Fortelab, MEV-1700/V, Brazil), which was performed in two steps: 720 °C for 15 min with heating of 10 °C/min, and then 1400 °C for 2 h with heating increments of 5 °C/min. The first step removed the CO_2 gas released in the decarbonation reaction, while the second step promoted glass melting and its homogenization. After the end of the second step, the molten glass was quickly spilled into water to obtain the frit, which was collected, dried and milled in a planetary mill (Fritsch, Pulverisette 5, Germany) for 30 min with a rotation of 220 rpm. Zirconia balls (10 and 20 mm of diameter) were used in a weight ratio of 1:10 between the glass powder and the zirconia balls, respectively. The SiO_2-Li_2O glass powder was subsequently passed through a sieve, aiming to obtain a particle size of less than 73 μm (200 mesh). Moreover, a binder (5% volume/volume solution of polyvinyl alcohol-PVA) was added to the glass powder and the new mixture was passed through the sieve (200 mesh) again to ensure that the formed granules were less than 73 μm.

X-ray diffraction and Differential scanning calorimetry

The X-ray diffraction (XRD) technique was performed by a diffractometer (Shimadzu XRD7000, CuKα radiation, $2\theta = 10\text{-}70°$, 40 mA, 40 kV) to analyze and attest that the obtained glass was amorphous. The glass transition, crystallization and melting temperatures of the glass powder were determined by differential scanning calorimetry (DSC). The analysis was performed using the simultaneous thermal analyzer (STA 449 F3 Jupiter®-NETZSCH) in a nitrogen atmosphere, with a heating rate of 10 ºC/min to 1200 ºC, and 0.03 g of ground glass powder was used for each analysis.

Preparation and characterization of lithium disilicate glass-ceramic (Li_2Si_2O5)

The powder was compacted into disc form in a hydraulic press (Marcon, MPH10, Brazil) by uniaxial pressing with a pressure of 14.7 MPa applied for 1 minute, using 0.4 g per specimen in a matrix with 15 mm of diameter. The mold was lubricated with stearic acid to reduce the friction between the matrix and the glass powder particles before each uniaxial pressing.

The thermal treatments were performed based on the temperatures corresponding to the structural modifications obtained by the DSC in order to achieve the lithium disilicate phase formation. The heat treatments were performed in a furnace (JUNG, LF00914, Brazil). the specimens were polished with SiC #400, 800 and 1200 according to the ISO standard CD 6872, and attained final dimensions of 12 mm in diameter and 1.2 mm thickness.

The crystalline phases present in the lithium disilicate glass-ceramics were also determined by a diffractometer (the same mentioned previously) and one disc of each group was used. The JCPDS International Center for Diffraction Data assisted in identifying the crystalline phases.

The microstructure was analyzed by scanning electron microscopy (SEM, FEI, Czech Republic). The discs were coated with gold by sputtering (EMITECH SC7620 Sputter Coater, East Sussex, United Kingdom), with one disc from each group being selected and prepared with 10% hydrofluoric acid for 20 seconds (on each surface).

Density and apparent porosity measurements were performed on three specimens of each lithium disilicate glass ceramic group using the Archimedes method in water. The sample mass measurements were carried out on a scale under various conditions: dry, immersed, and wet or saturated in water. The density and apparent porosity were calculated by the following equations (1 and 2):

$$\text{Equation 1} \qquad D = \frac{Md}{Mw - Mi} \times \rho_{water}$$

$$\text{Equation 2} \qquad P = \frac{Mw - Md}{Mw - Mi} \times 100$$

Where D is the density and P is the porosity. Md is the dry mass; Mw is the wet mass and Mi is the immersed mass.

Regarding hardness, five Vickers indentations were performed in the center of 5 discs of each group in order to create a defect. The specimens were indented with 1 kg for 12 seconds, and then the size of the indentation was measured.

Next, the biaxial flexural strength (BFS) test was performed in a universal testing machine (Emic DL, São José dos Pinhais, PR, Brazil). The specimens were positioned over three support spheres (3 mm in diameter) which were equally spaced at a circumference of 10 mm. The load was applied by a circular flat piston (1.5 mm in diameter) at 1 mm/min until the failure. Fifteen specimens from each group were tested, and a piece of adhesive tape (3M ESPE) was fixed to their compression side to minimize defects caused by the metallic tip, as well as to keep the fragments in position. The BFS of the monolithic discs was calculated according to equation 3:

$$\text{Equation 3} \qquad S = -0.2387P(X - Y)/d^2$$

Where S is the maximum tensile stress (MPa), P is the total load applied to cause the fracture (N), and d is the specimen thickness at the origin of the fracture (mm). X and Y are parameters related to the elastic properties of the material (Poisson's ratio and elastic modulus).

Statistical Analysis

The data were analyzed individually in one-way ANOVA tests, and Post hoc multiple comparisons were performed using Tukey's test at α=5%.

RESULTS

Characterization of SiO_2-Li_2O glass

The frit obtained from the melting/cooling method was an amorphous glass which was confirmed by the presence of a halo in Figure 1. Moreover, Figure 2 shows the thermal analysis results of SiO_2-Li_2O glass. The glass transition (T_g) occurred at 470 °C, the crystallization temperature (T_c) was the highest exothermic peak which occurred at 580 °C, and the melting temperature (T_m) occurred at 1040 °C. Thus, the heat treatment was planned (Table I) according to these data, using the 1st temperature step for binder elimination (PVA), the 2nd step for nucleation and the crystallization process, and the 3rd step for crystallization and densification. A heating rate of 2 °C/min was used in the 1st step and then increased to 5 °C/min in the 2nd and 3rd steps.

Table I - Thermal treatments used to obtain the experimental lithium disilicate glass-ceramic

		Group 1	Group 2	Group 3
300 °C/1h	580 °C/1h	850 °C/3h	900 °C/3h	950 °C/3h
1st step	2nd step	3rd step		

Characterization of lithium disilicate glass-ceramic ($Li_2Si_2O_5$)

The crystalline phases of lithium disilicate ($Li_2Si_2O_5$, JCPDS 00-040-0376) and quartz (Figure 3) were identified by XRD analysis. Next, SEM micrographs (Figure 4) depicted the needle grains most clearly for Group 3 (Figure 4-C), as well as showing a better densified structure. On the other hand, the structure was less dense when the material was sintered at lower temperatures such as 850 °C (Figure 4-A) and 900 °C (Figure

4-B), and the grains possessed a subtle needle format.

All the treatments presented statistical differences in relation to density, apparent porosity, hardness and BFS. Group 3 achieved the highest values for density, hardness and BFS results (Table II), while Group 1 reached the lowest values due to its higher porosity.

Table II - Means values and SD of porosity, density, hardness and BFS for SiO_2-Li_2O glass-ceramics after heat treatments

	Group 1 (850 °C)	Group 2 (900 °C)	Group 3 (950 °C)
Porosity (%)	33.89 (0.37) A	27.67 (0.36) B	11.30 (0.40) C
Density (g/cm³)	1.61 (0.01) C	1.74 (0.01) B	2.11 (0.01) A
Hardness (HV)	43.24 (5.87) C	81.50 (5.50) B	249.6 (12.9) A
BFS (MPa)	43.69 (11.25) C	90 (11.92) B	179.99 (7.72) A

Data is presented by means (SD). Different letters show statistical differences.

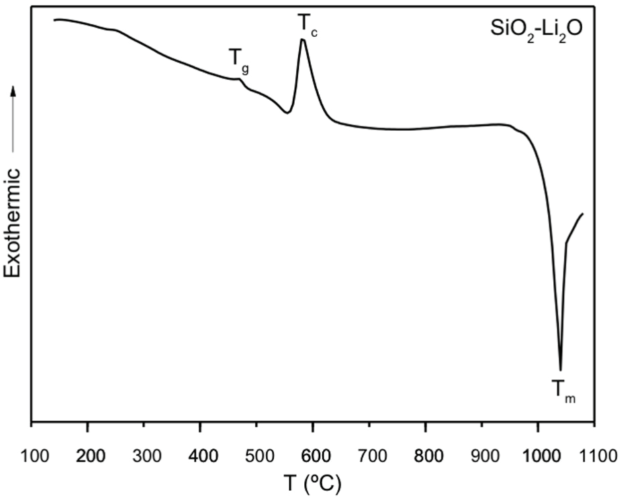

Figure 2 - DSC curve of the SiO_2-Li_2O glass.

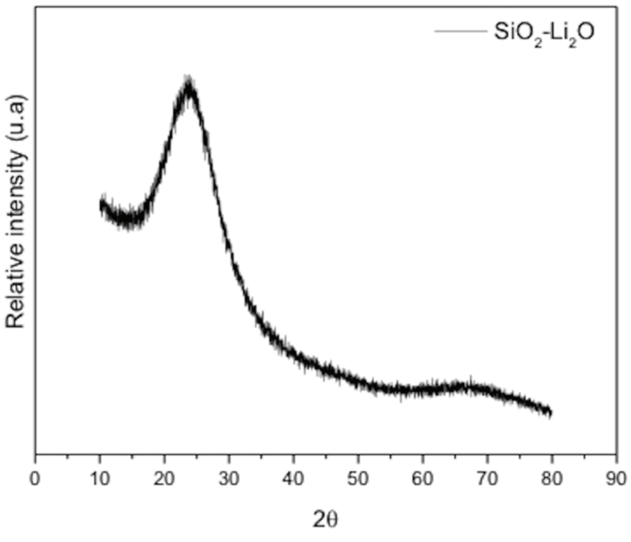

Figure 1 - XRD pattern of the SiO2-Li2O glass obtained from melting/cooling method.

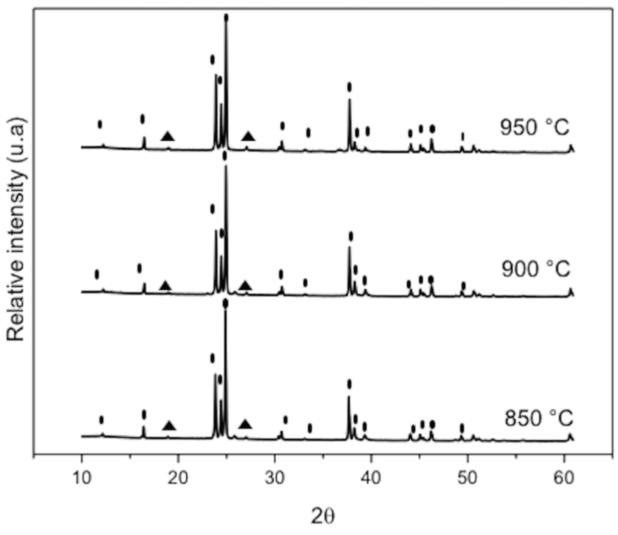

Figure 3 - XRD patterns of the SiO_2-Li_2O glass-ceramics after heat treatment. Lithium • disilicate, ▲Quartz.

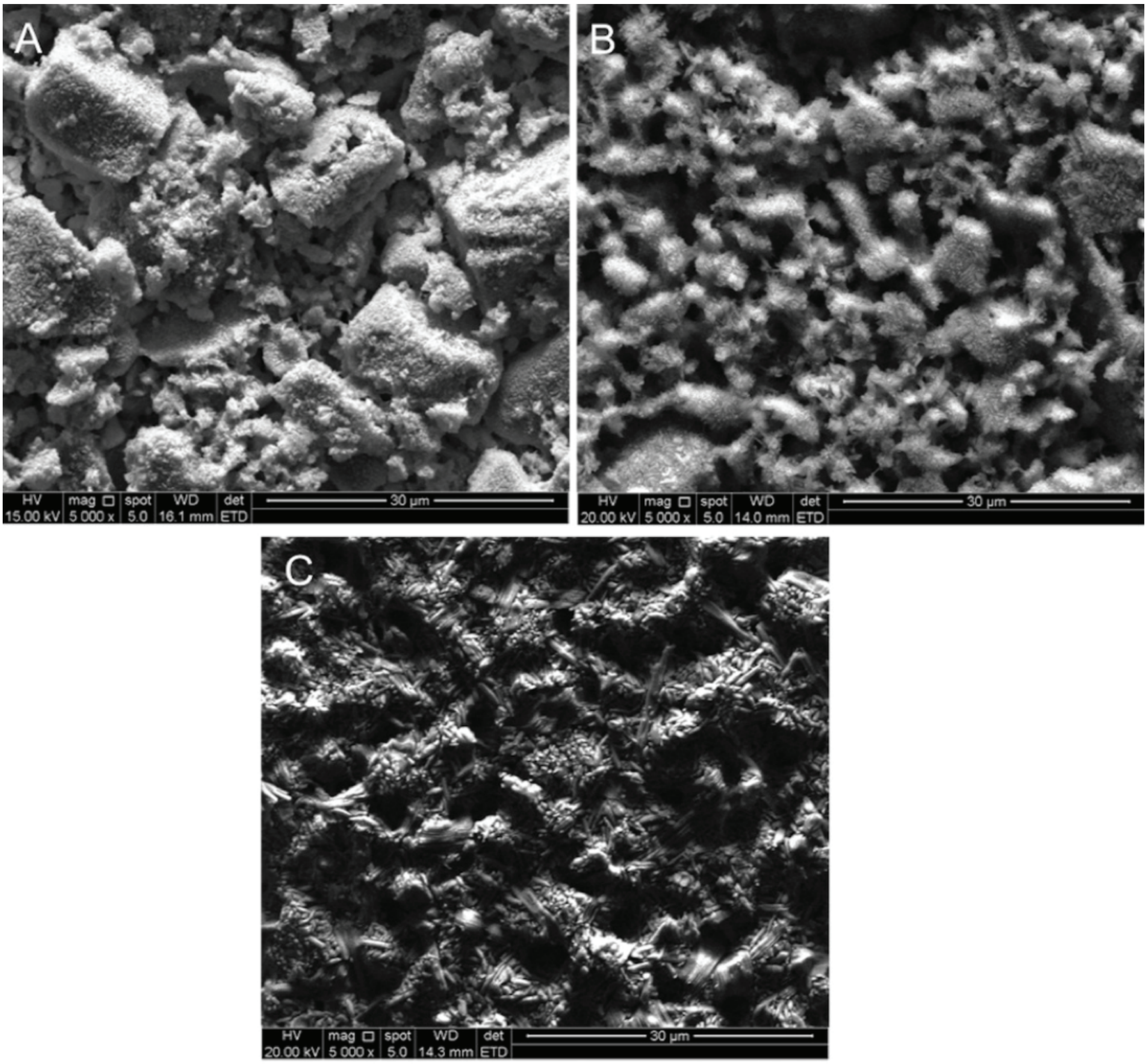

Figure 4 - SEM micrographs of the SiO_2-Li_2O glass-ceramics after heat treatments. A) Group 1 (850 °C), B) Group 2 (900 °C) and C) Group 3 (950 °C).

DISCUSSION

The present study synthesized an experimental glass-ceramic derived from the Li_2O-SiO_2 system aiming to study the sintering process, and to evaluate the microstructure, densification and mechanical properties of the new material. Regardless of the analysis, all studied temperatures presented significant differences. Group 3, which was the highest temperature analyzed, achieved the highest mechanical properties, while Group 1 showed the lowest mechanical properties and highest apparent porosity.

The nucleation and crystal growth phases are of primary importance for the microstructural development of glass-ceramics due to their direct interference on the properties of the new material [14,15]. A sequence of phase changes takes place during the conversion of glass to a final ceramic material with a microstructure of interlocking crystals [16]. The nucleation process is the key to control and predict the glass devitrification, which is done by a heat treatment above the glass transition temperature T_g [17]. The T_g found in this study occurred at 470 °C and determined the second step of the heat treatment, which was maintained for one hour to promote a slow nucleation. This stage was followed by the third stage at a higher temperature at which crystal growth took place [18]. It has been reported that the crystal growth rate for lithium disilicate crystals is a function of the reciprocal temperature value [13], which could be observed by the micrographs, in which the lithium disilicate crystals inevitably increased by increasing the sintering temperature. Thus, the use of a final sintering temperature of 950 °C for the presented system was safe, not causing any material melting, and was also able to improve the crystal growth.

Moreover, it has been reported that temperature and time are directly related to phase transformations in the synthesis of ceramics [16,19]. The lithium metasilicate (Li_2SiO_3) phase grows fast after nucleation and completely decomposes at 820 °C [3,20], and is a precursor for lithium disilicate crystallization [21]. Thus, the lowest temperature used for the crystallization of the glass-ceramics was at 850 °C, since the aim was to achieve the lithium disilicate phase for all groups as it crystalizes easily, and the availability of reliable thermodynamic data makes it a preferred model system for studying glass-ceramics [22,23].

In relation to the micrographs, increasing the temperature resulted in more noticeable needle-like particles of lithium disilicate, suggesting that high temperatures are preferable for strengthening the material achieved by a dense crystal microstructure. The mechanical properties analyzed were consequently improved, including the biaxial flexural strength (BFS), which remarkably increased from 51 MPa to 160 MPa in comparing Groups 1 and 3, respectively. Moreover, the final temperature also had a major impact on the densification process [8,11] and hardness, which significantly increased when the temperature also increased. These findings can be mainly attributed to efficient sintering behavior, the high relative density, and also the interlocking structure with rod-shaped crystals homogenously embedded in the matrix, which plays an important role in hindering crack propagation and elevating flexural strength [5,8].

Additionally, the flexural strength characterizes the maximum stress level supported by the material under flexural stress, thus providing the most clinically relevant mechanical specification for brittle dental ceramics [14]. In a previous study, Yuan et al. 2013 [14] reported that crystals with a larger size may provide stronger mechanical strength by developing a more organized microstructure. Indeed, an increase in the crystal size and flexural strength was observed when the sintering temperature increased, agreeing with a previous study [24], which reported a similar finding and also observed a decrease in the open porosity of the lithium disilicate glass-ceramics. Likewise, the porosity of the presented glass-ceramic was also abruptly reduced by an increase of 100 °C, which can be directly related to the crystal shape that was transformed into rod-shaped crystals at higher sintering temperature.

Therefore, this study analyzed the effects of heat treatments on the crystalline composition, microstructure, porosity, density, hardness and biaxial flexural strength of glass-ceramics of the Li_2O-SiO_2 system. The studied temperatures presented no significant effect on the composition of the crystalline phases of the glass-ceramics. However, there was a significant effect on the crystal size, apparent porosity and density, with higher temperatures resulting in larger crystals, as well as in a denser and less porous material, without melting the material.

Likewise, higher hardness and BFS values were also evaluated, which significantly increased when the temperature also increased. However, this study presented some limitations such as the use of a simple binary system and the pressureless sintering, which could be modified in further studies in order to obtain glass-ceramics with higher mechanical properties and an even denser microstructure.

CONCLUSION

The effect of sintering temperatures on the lithium disilicate glass-ceramics showed that its morphology and densification affected their mechanical properties. Group 3 sintered at 950 ºC resulted in higher hardness, density, and BFS values due to its lower porosity. Therefore, it is of utmost importance to follow the protocols recommended by the manufacturers of ceramics, including the times and temperatures to achieve the better properties for glass-ceramics. A sintering mistake can compromise the restorative material's performance such as reducing its mechanical strength and translucency due to a higher porosity, resulting in a weak and opaque material.

Declarations of interest

None

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