Nucleation Heat Treatment on the Crystalline Fraction and Crystal Morphology of Lamav Cad Lithium Disilicate

Bruna de Freitas Vallerini (✉ bruvallerini@hotmail.com)

Universidade Estadual Paulista Julio de Mesquita Filho Faculdade de Odontologia Campus de Araraquara <https://orcid.org/0000-0002-8432-9625>

Mariana de Oliveira Carlos Villas-Bôas

Universidade Federal de Sao Carlos Centro de Ciencias Exatas e de Tecnologia

Lígia Antunes Pereira Pinelli

Universidade Estadual Paulista Julio de Mesquita Filho Faculdade de Odontologia Campus de Araraquara

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Abstract

Thus, this study aimed to evaluate the influence of different nucleation heat treatments on the crystalline fraction and crystal morphology of the LS2 LaMaV CAD. Discs (12 mm diameter x 1.2 mm thickness) of LS2 LaMaV CAD and IPS e.max CAD (C, control group, n=3) were made. LS2 LaMaV CAD discs were annealed at 380°C/2h followed by different nucleation heat treatments in which time and temperature were varied composing 4 groups (n=3): T1 (1h30/500°C), T2 (3h/500°C), T3 (6h/500°C) and T4 (6h/480°C). Scanning Electron Microscopy images were obtained to characterize the microstructure and to quantify the crystalline fraction using the ImageJ software. X-ray diffraction with Rietveld refinement was performed. ANOVA one-way with Tukey post-hoc test ($\alpha = 0.05$) was used to analyze the ImageJ images and the Rietveld refinements for X-ray diffraction data. X-ray diffraction analysis showed peaks of approximately 86% of lithium disilicate for all groups. The morphology of crystals was in lath-shaped and homogeneous format for all groups; the group that most resembled the C group was T1. The crystalline fraction (%) was: C = 59.33 ± 0.47 , T1 = 61.63 ± 0.61 , T2 = 61.07 ± 1.09 , T3 = 56.39 ± 0.32 , and T4 = 57.48 ± 0.90 , all the groups were statistically different to the C group. It was concluded that the nucleation temperature influenced the size and quantity of the crystals of lithium disilicate; treatment that showed the best result was that that received nucleation treatment for 1 hour and 30 minutes at 500°C.

1 Introduction

Over the years, ceramic materials have received high visibility in the dental market, mainly in oral rehabilitation in cases involving aesthetics, due to their high capacity to mimic dental tissues. Dental ceramics with high crystalline content allowed to replace the metal infrastructures used in metal-ceramic restorations with fully ceramic structures due to the biocompatibility, aesthetic capacity, and adequate mechanical resistance of these materials [1, 2].

Lithium disilicate glass-reinforced ceramics ($\text{Li}_2\text{O} \cdot 2\text{SiO}_2$), are one of the most commercially successful materials in dentistry [3] and frequently the choice of professionals, due to the wide range of indications for making crowns and fixed dental prosthesis, since it has a high flexural strength of 300 to 400 MPa, high fracture toughness of 2.8 and 3.5 $\text{MPa} \cdot \text{m}^{1/2}$ and excellent optical properties [4–6]. Also, these glass-ceramics have the possibility of being milled, simplifying the manufacturing methods and enabling the applicability of these materials to laboratories and chairside systems through computer-aided design/computer-aided manufacturing (CAD/CAM) technology, being one of the main advantages of this system and resulting in excellent clinical approval [7, 8].

It is considered that the greater the aesthetics of ceramic materials, the lower their fracture resistance, since they present a greater glass phase and a lower crystalline phase, as is the case of glass-ceramics that are considered a special group of materials and have their microstructure composed of one or more crystalline phases embedded in glass matrix [3], with crystallinity ranging from 30–70% and crystals with needle morphology with dimensions from 3 μm to 6 μm [3, 9]. This configuration results in a microstructure with lithium disilicate crystals interlocked lath-shaped that promote an increase in the

flexural resistance of these materials and result in the limitation of the propagation of the crack, because the conformation of the crystals make that, during the deflection of the crack, the path that the crack would have to go through increases [3, 9–11] and, consequently, consumes more energy.

However, other factors such as crystal size, phase type and porosity influence the resistance of these materials [3], as well as the translucency which is dictated not only by the amount of crystalline phase but also by the size of the crystals and the refractive index of the two phases, the control of these factors can allow resistance and aesthetics[4]. Thus, with an appropriate design of the microstructure that can be modified by changes in time and temperature of the nucleation heat treatments and crystalline growth, it is possible to develop materials improving their properties from new microstructural designs [9, 12, 13].

Glass-ceramics result from the process of controlled crystallization, its properties being basically dependent on two factors, the composition of the reagents established and the heat treatment adopted [3, 9, 14], which when carefully modified can result in promising microstructural designs with excellent properties [9, 12, 13]. The conventional route of the glass-ceramics production process consists of four stages: melting, homogenization, induction of the internal nucleation by means of the nucleation heat treatment, and growth of the crystals with one or more heat treatments, which significantly affect the evolution of the crystalline fraction [15]. That is, the crystalline fraction is controlled through the nucleation and crystal growth stages, which can vary according to temperature and time for both conditions [9, 12, 13, 16]. The heat treatments to which the glass-ceramic is submitted allow to obtain the desired microstructure and optimize the material properties, increasing the mechanical resistance of fragile materials [9, 12, 13].

Based on the good characteristics of lithium disilicate glass-reinforced ceramics, the Laboratory of Vitreous Materials of the Federal University of São Carlos developed a lithium disilicate glass-ceramic (LS2 LaMaV CAD) in search of better properties compared to the materials already available in the dental market. Even knowing that changing the time and temperature of nucleation modifies the microstructure of these materials, it is timely to assess the effect of different times and temperatures of nucleation on the microstructural characteristics and crystalline fraction of lithium disilicate LS2 LaMaV CAD, in order to obtain a homogeneous material with improvements in microstructural design and larger crystal fraction.

2 Materials And Methods

The phases of the materials assessed by X-ray diffraction, the quantification of the crystalline fraction and the morphology of crystals analyzed by the images obtained in a scanning electron microscopy were used to determine the most suitable nucleation heat treatment in order to obtain homogeneous crystals of lithium disilicate, as well as a crystalline fraction that allows optimizing the mechanical properties of LS2 LaMaV CAD. The heat treatments with their respective times and temperatures are shown in Table 1, as well as the nomenclature adopted by the experimental groups.

Table 1
– Nomenclature adopted to the groups of LS2 LaMaV CAD with their different times and temperatures of nucleation heat treatment

Groups	Time (h)	Temperature (°C)
T1	1.5	500
T2	3	500
T3	6	500
T4	6	480

The reagents of experimental groups were placed in a polyethylene bottle and homogenized for 4 hours. After the homogenization of the whole mixture, the glass-ceramic was melted in a platinum crucible at 1450°C for 3 hours in an oven Deltech Inc. (Denver, Colorado, USA), during which time the glass was poured and ground by hand three times to ensure complete homogenization. The glass was poured into stainless steel molds with cylindrical geometry.

The annealing was done in an oven EDG 7000 (EDG, São Carlos, São Paulo, Brazil) at 380°C for 2 hours. After this step, the cylindrical samples were cut in a SYJ-150 Low Speed Diamond Saw (MTI, Richmond, California, USA) with a diamond disk (Series 15LC Diamond, Buehler, Illinois, USA) at 200 rpm and water cooled to obtain 12 discs with dimensions of approximately 12 mm in diameter x 1.2 mm in thickness, measured by means of a digital caliper (500-144B, Mitutoyo Sul Americana, Suzano, SP, Brazil). The sintering shrinkage for this material is about 3%.

The samples were randomly divided into four groups (n = 3) to perform the different nucleation heat treatments (Table 1), followed by the treatments of lithium metasilicate and lithium disilicate * crystalline growth (Figure 1), isothermic cycles were adopted for all heat treatments. After heat treatments, the samples were again measured with the aid of a digital caliper, and a sintering shrinkage greater than 3% was not observed.

After all heat treatments (nucleation, lithium metasilicate phase growth, and lithium disilicate phase growth), the surfaces of the disks were standardized using silicon carbide # 200, # 320, # 400, # 600, #1200 (401Q, 3M ESPE, Sumaré, SP, Brazil) and polishing with aqueous solution of cerium oxide in a politrax (METMAXIMILLE, Bela Vista, SP, Brazil) at 200 rpm .

IPS e.max CAD was used as the control group, the blocks were machined in a cylindrical shape and cut in a SYJ-150 Low Speed Diamond Saw with diamond disk (Series 15LC) at 200 rpm and water, obtaining 3 disks with approximate dimensions of 12 mm of diameter x 1.2 mm of thickness, measured by means of a digital caliper. A crystallization cycle was carried out in a Programat P310 oven (Ivoclar Vivadent, Schaan, Liechtenstein), according to the manufacturer's recommendations (820°C for 7 minutes). The samples were measured with the aid of a digital caliper before and after crystallization treatment and

there was not sintering shrinkage greater than 3% was observed. The finishing and polishing of the samples of IPS e.max CAD followed the same procedures described for the LS2 LaMaV CAD groups.

All samples were analyzed by an X-ray diffractometer (RINP2000, RIGAKU, São Paulo, SP, Brazil) with Cu-K $\alpha_{1,2}$ radiation, with a scan between 10° and 80°, continuous scanning at 3°/min. The crystalline phases were identified with the PDF + 2 and the refinements of the crystalline structures were performed by the Rietveld method[17, 18].

The characterization of the crystals was performed for all groups (n = 3) using a scanning electron microscope (Jeol, JSM6610LV, Akishima, Tokyo, Japan) to observe the geometry, quantity, and size of the crystals. The samples were not acid etching before it was covered with carbon.

Quantification of the crystalline fraction was performed using the ImageJ software (National Institutes of Health, Bethesda, Maryland, USA), obtaining the estimated percentage of glass phase and a crystalline phase. Six images of each group were selected for standardization with respect to dimensions (1092 × 776 pixels), excluding the edges and information bar of the images; white balance setting by temperature 8 and tint 5 and basic settings (exposure = -4, brightness = 22, contrast = 100, vibration = 37 and saturation = 100), all these adjustments were made in the Fotor Photo Editor program (version 3.5.1). The images were analyzed using the Make Binary function of the ImageJ software, which converts the images to black and white, allowing a more accurate estimate of the percentages [19].

The values of crystalline fraction obtained in the Image J Software were analyzed by ANOVA one-way with post-hoc Tukey ($\alpha = 0.05$), the analyses were performed using IBM SPSS Statistics 25.0 (IMB Corp., Armonk, NY, USA). The DRX data and scanning electron microscopy images were analyzed descriptively.

* Temperatures and times of the heat treatments of growth of the lithium metasilicate and disilicate protected by the patent process.

3 Results

X-ray diffraction showed that the phases of T1, T2, T3, and T4 groups had no differences and there was little variation in quantities among the four groups, with values close to 86% for lithium disilicate, 8% for lithium phosphate and 6% for lithium metasilicate (Table 2). The control group showed the predominance of lithium disilicate (91.37%) with the presence of lithium phosphate (8.63%) and absence of lithium metasilicate.

Table 2
– Quantitative phase analysis of the groups measured by XRD with Rietveld refinement method.

Group	Lithium disilicate (%)	Lithium phosphate (%)	Lithium metasilicate (%)
Control	91.37	8.63	-
T1	86.57	8.11	5.33
T2	86.22	8.02	5.82
T3	85.79	7.28	6.89
T4	85.71	8.67	5.61

Figures 2, 3, 4, and 5 show the microstructure of LS2 LaMaV CAD of groups T1, T2, T3, and T4, respectively. It was possible to observe that the T1 group (Fig. 2) has crystals with lath-shaped morphology and uniform dimensions, being the group that most resembled the microstructural morphology of the control group (Fig. 6).

When analyzing the images related to groups T2 and T3 (Figs. 3 and 4, respectively) it was observed that the crystals morphology was lath-shaped, both with a crystal heterogeneity, but greater for the T3 group (Fig. 4). T3 also has a greater amount of glass matrix in relation to the other groups, which may have a negative impact, since it allows the propagation of cracks between the crystals. In relation to the T4 group (Fig. 5) it was possible to observe homogeneous crystals and with a better distribution among the glass matrix, the microstructure observed for this group is considered promising, since it seems to have had higher nucleation. The morphology of the experimental groups showed the orientation of the crystal like an interlocking pattern similar to the control group.

Regarding crystalline fraction, there was a statistical difference between experimental groups (T1, T2, T3, and T4) and the control group ($p < 0.05$) (Table 3). Analyzing Table 3, increasing the time of the nucleation heat treatments resulted a decrease in the crystalline fraction. However, keeping the same time, and varying the temperature in 20°C (T3 group comparing with the T4 group) resulted in a tendency to increase the crystalline fraction.

Table 3
– Means (%) of the crystalline fractions of T1, T2, T3, T4 and control groups

Group	Mean (%)
Control	59.33 ± 0.47 ^a
T1	61.63 ± 0.61 ^b
T2	61.07 ± 1.09 ^c
T3	56.39 ± 0.32 ^d
T4	57.48 ± 0.90 ^e
Different lowercase superscript letters indicate statistically significant difference between rows (p < 0,005);	

4 Discussion

The choice of lithium disilicate in this study is due to this material being one of the main materials indicated for the preparation of dental ceramic restorations. This is due to the favorable aesthetics of lithium disilicate that can mimic dental tissues, to the fracture strength values (± 350 MPa), and the replacement of metal infrastructures (metal free) [1, 8, 20].

Other companies than Ivoclar Vivadent Company started researches of lithium disilicate, among them the Laboratory of Vitreous Materials from the Department of Materials Engineering of Federal University of São Carlos, which aims to develop a lithium disilicate with better microstructure and crystal morphology, resulting in the improvement of the properties of lithium disilicate.

According to the literature [14, 16], the properties of glass-ceramics depend on their composition and the heat treatment to which they are subjected. The heat treatments have a significant effect on the crystallization process [16], controlling the formation of the phases, the crystallinity, the size, and the shape of the crystals [9, 12, 13, 21], allowing to project the crystals from the control of the crystallization step, making it possible to optimize the phases present and its microstructure by developing materials with the desired properties [9, 12, 13, 22].

The findings of the X-ray diffraction of the LS2 LaMaV CAD groups agree with those reported in the literature [3, 20, 22–25], with values close to 86% for lithium disilicate, 8% for lithium phosphate and 6% for lithium metasilicate, showing the lithium disilicate like predominant phase. The control group showed the predominance of lithium disilicate (91.37%), with the presence of lithium phosphate (8.63%) and absence of lithium metasilicate, according to the findings of Belli et al. [26] in 2019 and Wang et al. [27] in 2019.

Huang et al. [22] in 2013 and Lien et al. [23] in 2015 showed that the glass-ceramic when receiving heat treatment for additional crystallization with temperatures below 780°C presents a higher amount of lithium metasilicate, which promotes a low resistance to this material and is ideal for milling. When this glass-ceramic is treated at temperatures above 780°C there is an increase in the transformation of the lithium metasilicate to lithium disilicate, providing an increase in its resistance[22, 23]. Both the commercial material and the LS2 LaMaV CAD were subjected to the crystallization temperature of the lithium disilicate phase at a temperature above 780°C; in the control group the crystallization was performed at 820°C for 7 minutes of threshold and in the LS2 LaMaV CAD the temperature adopted was above this value *. The fact that LS2 LaMaV CAD samples have approximately 6% of the lithium metasilicate phase may have occurred in function of the temperature adopted in the lithium disilicate crystal growth treatment and is also verified in Huang et al.[22] in 2013 data showing that there is a maximum peak of lithium disilicate when adopting temperature up to 800oC falling in volume above and below this temperature.

Regarding the crystalline fraction, an interesting behavior was observed: the increase of the heat treatment time resulted in a decrease of the crystalline fraction ($T1 = 61.63 \pm 0.61$, $T2 = 61.07\% \pm 1.09$ and $T3 = 56.39 \pm 0.32$). However, analyzing the T3 and T4 groups separately (same times but at different temperatures), the crystalline fraction increased ($T3 = 56.39 \pm 0.32$ and $T4 = 57.48 \pm 0.90$), most probably due to the difference in nucleation temperature (20oC less). By associating the scanning electron microscopy images and the crystalline fraction values, heterogeneous crystals with different lengths were observed in the T3 group; this was probably due to the growth of the crystals during the nucleation stage (with a temperature of 500oC) which was not expected. The decrease in the nucleation temperature in 20oC resulted in more homogeneous crystals (T4 group). In view of the observed, it can be inferred that the decrease in temperature provided more homogeneous nucleation, besides a greater crystalline fraction in relation to T3.

Regarding the geometry of the crystals, it can be observed that comparing all the experimental groups, the one that presented characteristics that most resemble the control group was the T1 group. It can be seen that samples T2 and T3 have crystals with less homogeneous dimensions. Group T1 visually shows larger average crystal sizes with respect to the T4 group, as can be analyzed in Figs. 2 and 5, respectively. The differences obtained between the microstructures of the samples T1 and T4 are important for the future evaluation of the impact of crystal sizes on mechanical properties, since according to Hallmann et al. [2] in 2018 and Höland and Beall [28] in 2007 the differences in mechanical properties for materials with similar chemical compositions are most likely related to divergent microstructures. The orientation, shape, and distribution of crystal sizes, homogeneity, and the proportion of the glassy matrix and crystalline phase control the flexural strength and fracture toughness of the glass-ceramic materials [2, 28]. The surface morphology showed for all groups that the crystals have lath-shaped [6, 29] and an interlocked microstructure that is an important characteristic for the mechanical performance of the material [3, 10, 11, 23, 30] because it allows during the deflection of the crack that the path that it would have to travel increases and consequently consumes more energy [3, 10, 11].

The evaluation of the crystalline fraction and the crystal morphology carried out in this study shows the importance of steps that are often unknown by the dentist, but which have an impact on the choice and use of materials in oral rehabilitation. The fact that the crystalline fraction renders the glass-ceramic more resistant and the morphology of the crystals also exerts a strong influence in this property reinforces, even more, the seriousness with which the research must be carried out when it is intended to launch new materials in the dental market.

Based on the tests performed, the groups that present microstructural and morphological characteristics more similar to the IPS e.max CAD (control) were the groups nucleated for 1 hour and 30 minutes at 500°C (T1) and 6 hours at 480°C (T4) and will be the target for future mechanical tests. The T1 group was the one that most resembled the control mainly when analyzed in terms of the microstructure. The T4 group will be selected because it possesses a crystalline fraction considered very close to the control, with more homogeneous nucleation and visibly smaller crystals, which may allow a better crystals interlocking which according to the literature for lithium disilicate resulted in higher values of resistance mechanics [2, 4, 10, 14]. It should be emphasized that the conclusions of this work were based only on the analysis of the microstructure and in the crystalline fraction, requiring data of mechanical resistance in order to be applied safely in the dental area.

* Temperatures and times of the heat treatments of growth of the lithium metasilicate and disilicate protected by the patent process.

5 Conclusions

Based on the methodology developed, it was concluded that changes in the nucleation heat treatment influenced the size and quantity of the crystals, which allows changing them according to the characteristics desired. The group that presented the best result was the one treated for 1 hour and 30 minutes at 500°C.

Declarations

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Figures

Figure 1

Stages of the heat treatments adopted in the experimental groups (LS2 LaMaV CAD)

Figure 2

SEM image of the LS2 LaMaV CAD sample of T1 group. Used scale represented in the image (5μm)

Figure 3

SEM image of the LS2 LaMaV CAD sample of T2 group. Used scale represented in the image (5μm)

Figure 4

SEM image of the LS2 LaMaV CAD sample of T3 group. Used scale represented in the image (5μm)

Figure 5

SEM image of the LS2 LaMaV CAD sample of T4 group. Used scale represented in the image (5μm)

Figure 6

SEM image of the sample IPS e.max CAD (control group). Used scale represented in the image (5μm)