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Impact of crystallization firing process on the microstructure and flexural strength of zirconia-reinforced lithium silicate glass-ceramics

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ABSTRACT

Objectives. The aim of this study was to characterize the microstructure of two zirconia-reinforced lithium silicate (ZLS) glass-ceramics and evaluate their mechanical properties before and after the crystallization firing process (CFP).

Methods. Field emission-scanning electron microscope (FE-SEM) and energy-dispersive X-ray spectroscopy (EDS) analyses were performed for microstructural characterization. To evaluate the pattern of crystallization and the molecular composition of ZLS glass-ceramics, was used X-ray diffraction (XRD). Vickers hardness, fracture toughness by the indentation method, and biaxial flexural strength were also measured. One hundred and forty ceramic discs were produced (diameter = 12 mm; thickness = 1.2 mm) and allocated among four groups (n = 30): Sfir, Sunf-ZLS Vita Suprinity; and Cfir and Cunf-ZLS Celtra Duo; fired and unfired, respectively. Statistical analysis was performed and Weibull failure probabilities were calculated.

Results. Cfir showed the highest characteristic strength (251.25 MPa) and hardness (693.333 ± 10.85 GPa). Conversely, Sunf presented the lowest characteristic strength (106.95 MPa) and significantly lowest hardness (597.533 ± 33.97 GPa). According to Weibull analysis, Sunf had the highest structural reliability (m = 7.07), while Sfir presented the lowest (m = 5.38). The CFP was necessary to crystallize zirconia in the partially crystallized ZLS glass-ceramics. Sfir had a lower percentage of crystallized zirconia than did Cfir. Fractographic analyses showed that all failures initiated from an inherent critical defect in Sunf and from processing defects in the remaining groups.

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Significance. The CFP had a direct influence on the flexural strength and microstructural characteristics of both ZLS materials.

1. Introduction

The constant search for all-ceramic restorative materials that combine aesthetics and strength has contributed to the development of reinforced glass-ceramics [i.e., zirconia-reinforced lithium silicate (ZLS) glass-ceramics] by the companies Vita and Dentsply, in conjunction with the Fraunhofer Institute for Silicate Research (Germany), and marketed separately as different products, i.e., Vita Suprinity (Vita) and Celtra Duo (Dentsply) [1,2]. Both ZLS glass-ceramics are indicated for inlays, onlays, full-contour anterior and posterior crowns, and also for implant-supported prostheses [3,4].

The glassy matrix of vitreous ceramics is highly susceptible to crack propagation [5] and to degradation under fatigue loads [1]. Glass-ceramics such as those studied here have been developed to overcome those deficiencies and create even higher-performance ceramic materials [1]. The manufacturers claim that the zirconia dioxide content incorporated into ZLS glass-ceramics is ten times higher than that in other glass-ceramics [6,7]. This component is arranged in small grains and could result in higher resistance and better superficial polishing [8]. Previous studies, however, have called attention to the low efficacy of zirconia for stopping crack propagation [9], as this is simply a form of commercial appeal [1]. Moreover, ZLS materials continue to belong to a class of sensitive acid ceramics, meaning that their surface can be etched by hydrofluoric acid [10].

ZLS glass-ceramics are marketed in a pre-processed state, available as pre-fabricated ceramic blocks, which allows for fewer internal flaws and a high-quality and reliable ceramic restoration produced in a mechanized/subtractive way [3]. To make the milling process easier, ZLS glass-ceramics are available in a meta-sintered (intermediate) stage and only after undergoing a CFP in which the lithium silicate crystals will grow and the ceramic gains its final color and strength [1,11]. Recently, the simplification of the clinical steps pushed manufacturers to produce blocks in fully crystallized state that can be milled and cemented, although Dentsply reports lower flexural strengths for “mill and polish only” specimens, which led us to the belief that full crystallization is one step further. Thus, the differences in crystalline phases in vitreous ceramics – mainly the effects on mechanical properties and toughening mechanisms depending on the crystallization state – remain to be determined.

The aim of the present study was to evaluate the microstructure of ZLS glass-ceramics in partially or fully crystallized states, as well as to evaluate the mechanical properties of these ceramics before and after the crystallization process. To evaluate the ceramics, we performed crystallization firing schedules and not only the stain/glaze firing recommended by Celtra Duo manufacturer (Dentsply). The null hypothesis tested was that the ZLS glass-ceramics would have the same mechanical properties and microstructural characteristics before and after the crystallization firing.

Table 1 – Crystallization chart firing of Vita Suprinity and Celtra Duo.

	Vita Suprinity	Celtra Duo
Initial chamber temperature (°C)	400	400
Time at the initial temperature (min)	8	8
Temperature rate increase (°C/min)	55	55
Crystallization temperature (°C)	840	830
Holding time (min)	8	10
Ending temperature (°C)	680	700

2. Materials and methods

Zirconia-lithium silicate glass-ceramic materials (Vita Suprinity - Vita Zahnfabrik, H. Rauter GmbH & Co., Bad Säckingen, Germany, batch number 49270; and Celtra Duo - Degudent GmbH, Hanau, Wolfgang, Germany, batch number 18018171) were used to produce 140 ceramic discs. The specimens were further divided into two test conditions, with or without crystallization firing.

2.1. *Specimen preparation*

The ceramic blocks were machined in a conventional lathe (Nardini, Americana, São Paulo, Brazil) to obtain cylinders (~12 mm in diameter) that were cut into discs in a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA), with the final thickness of 1.2 mm.

The ceramic specimens were randomly separated, and half the discs of both materials were subjected to a crystallization cycle in a specific oven for each ZLS glass-ceramic (Vita Vacumat 6000MP, Vita Zahnfabrik; Multimat, Dentsply/Sirona (Table 1). ‘The crystallization firing of Vita Suprinity followed the manufacturer’s recommendations while that of Celtra Duo was based on the materials’ appearance and personal conversations with a technician and Dentsply consultant. For this process, the specimens were positioned inside the oven on top of a porous refractory material, allowing for homogeneous heat distribution. A metallic device with a central cavity and the desired dimensions (diameter = 12 mm; thickness = 1.2 mm) (Fig. 1) was used to allow the specimens to be better polished in the polishing machine (Ecomet 250 Grinder Polisher, Buehler), with silicon carbide papers of #400-, #600-, #1200-, and #2500-grit (Buehler). The specimens final dimensions were according to ISO 6872:2008 [12], that is, 12 mm in diameter and 1.2 mm thick. A mirror-like polishing was performed (Fig. 2).

All samples were manually polished with felt wool discs (Kota, São Paulo, Brazil) at 5000 rpm, under refrigeration by the same abrasive liquid solution with a diamond powder in