DENTAL MATERIALS 31 (2015) e316–e324

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.intl.elsevierhealth.com/journals/dema

Extended glaze firing improves flexural strength of a glass ceramic

Iana L. Aurélio[a], *Sara Fraga*[a], *Lucio S. Dorneles*[b], *Marco A. Bottino*[c], *Liliana G. May*[d,*]

[a] *Dental Science Post Graduate Program, Federal University of Santa Maria, Santa Maria, Rio Grande do Sul, Brazil*
[b] *Department of Physics, Federal University of Santa Maria, Santa Maria, Rio Grande do Sul, Brazil*
[c] *Department of Dental Materials and Prosthodontics, São Paulo State University, São José dos Campos, São Paulo, Brazil*
[d] *Department of Restorative Dentistry, Federal University of Santa Maria, Santa Maria, Rio Grande do Sul, Brazil*

ARTICLE INFO

Article history:
Received 27 January 2015
Received in revised form
18 April 2015
Accepted 24 October 2015

Keywords:
Glass ceramic
Leucite
CAD/CAM
Machining
Roughness
Thermal cycle
Glaze
Biaxial flexure strength

ABSTRACT

Objective. To investigate the effect of firing protocols on flexural strength, surface roughness, and crystalline structure of a leucite-based glass ceramic.

Methods. Discs produced by automated machining were distributed into five groups ($n = 30$) according to the applied firing protocols, conducted above (790 °C) or below (575 °C) the ceramic transition temperature (T_g) (625 ± 20 °C): C – control, no heat treatment; G790 – glaze firing (790 °C) for 1.5 min (manufacturer-recommended); G790-SC – G790 modified by slow cooling; EGF790-SC – extended G790 for 15 min, with slow cooling; and EF575-SC – extended firing below T_g at 575 °C for 15 min, with slow cooling. Discs were subjected to biaxial flexural tests and results were assessed using Weibull analysis. Surface roughness was measured before and after treatments. One specimen from each group was used for X-ray diffraction (XRD).

Results. Highest values of characteristic strength (σ_0) were obtained for EGF790-SC (211.7 MPa). Regimens EF575-SC, G790-SC, and G790 produced σ_0 values (167.9, 157.7, and 153.7 MPa, respectively) lower than the control (C) (187.7 MPa). The Weibull modulus (m) was statistically similar between groups. All treatments reduced the mean roughness (Ra) of the specimens. Extended cycles (EGF790-SC, EF575-SC) decreased the mean amplitude (Rz). XRD revealed no crystalline phase transformation and slight changes in size of leucite crystallites.

Significance. Increased values of fracture strength and decreased surface roughness for a leucite-reinforced glass ceramic are achieved by extended glaze firing after machining. Crystalline structure is not modified. Glaze cycles, manufacturer-recommended or modified by slow cooling, and firing below T_g, significantly reduce fracture strength.

* *Corresponding author at*: Federal University of Santa Maria, Faculty of Odontology, MDS-PhD Graduate Program in Oral Science, Prosthodontics-Biomaterials Units, R. Floriano Peixoto, 1184, 97015-372 Santa Maria, Brazil. Tel.: +55 55 3220 9276; fax: +55 55 3220 9272.
E-mail addresses: lamadridiana@hotmail.com (I.L. Aurélio), sara.odonto@yahoo.com.br (S. Fraga), lucio.dorneles@ufsm.br (L.S. Dorneles), mmbottino@uol.com.br (M.A. Bottino), liligmay@gmail.com (L.G. May).

http://dx.doi.org/10.1016/j.dental.2015.10.012
0109-5641/

1. Introduction

Clinical studies involving leucite-reinforced glass ceramic restorations report survival rates between 76 and 97%, with bulk fracture being one of the main reasons for failure of these restorations, totaling 3–11% of failures [1–4]. Cohesive chipping of the ceramic is mentioned in the literature, but this has not been considered as a failure, since this type of defect does not prevent functioning of the restorations [1,2,4].

Glass ceramics are widely used in indirect restorations that require aesthetic appeal, because they mimic the dental tissues and provide a mechanical strength that is greater than that of feldspathic ceramics. The improvements in the mechanical properties of these materials are a result of the higher content of crystals, such as leucite [5–7], and the development of prefabricated ceramic blocks for CAD-CAM (Computer Aided Design, Computer Aided Machining) systems, produced using a standardized process that results in a more homogeneous material [8,9]. However, even with these fracture toughness advances, the ceramic components maintain their brittle behavior, accumulating residual stresses as a result of the machining process, and are thus susceptible to different failures [10–12]. On the other hand, it is believed that machining might introduce thin compressive stresses to the surface of the ceramic with a positive effect on its strength [13,14]. Thus, an understanding of these effects is essential for predicting possible failure mechanisms and assessing the reliability of restorations made by CAD-CAM systems.

Different heat treatments have been employed for glass ceramics in an attempt to control residual stresses [15,16], while potentially reducing some of the damage accumulated during the various steps of fabricating the restorations. These treatments are conducted at temperatures near the glass transition temperature (T_g), where the material viscosity is reduced, and structural rearrangements promote relaxation of internal stresses in the ceramic [17,18]. However, cycles that reach temperatures close to the material softening point [19,20], in order to promote healing of possible defects, have also been reported. A relevant aspect in firing treatments for glass ceramics is the way in which the cooling stage is conducted, since it is the rate (fast or slow) at which this process occurs that will determine how much tension will be released or accumulated [16,21]. Below T_g, the glass behaves as a solid and all structural contraction reverts to residual stress locked inside the material [22]. Higher cooling rates usually result in thermal contraction and solidification being non-uniform because of temperature gradients, leading to the development of stresses [23], and possible weakening of the material.

Materials consisting of different phases, such as leucite-reinforced glass ceramics (with two phases, glassy and crystalline), should be treated with extreme caution because these phases behave differently during heat treatment [24–26]. This situation requires thermal schemes that meet the requirements for structural balance between phases. Some studies show that, depending on the regimen adopted, alterations in the crystals, and even in the constituent phases of the materials, may occur [27,28].

There are studies showing that routine laboratory thermal cycles, such as glazing, may be associated with mechanical and structural changes, which could reduce the fracture strength of ceramic materials [11,29–32]. Thus, these treatment schemes must be taken into account when trying to accurately predict the long-term performance of restorations. However, there is inconsistency in the literature regarding different thermal protocols applied to ceramic materials. Existing data on the influence of these treatments on structural and mechanical characteristics of the material are inconclusive.

The present study aimed to investigate the effect of different firing protocols on the fracture strength of a leucite-reinforced glass ceramic. The first hypothesis tested was that cycles that adopt heating baths at temperatures above the glass transition temperature, followed by slow cooling, would produce higher values of fracture strength. In addition, we sought to evaluate the effect of these treatments on surface roughness and crystalline structure of the ceramic (crystallite size and phase changes), testing a second hypothesis that presumed increases in strength values would be associated with a reduction in surface roughness. A third hypothesis was that a possible decrease in strength would be associated with an increase in crystallite size and crystal phase changes.

2. Materials and methods

2.1. Machining of ceramic discs by CAD/CAM

Discs with 13.5 mm diameter and 1.4 mm thickness were obtained by automated machining of leucite glass ceramic fully-sintered blocks (14 mm × 14 mm × 18 mm) (IPS Empress CAD C14L, Ivoclar Vivadent AG, Liechtenstein), using a CEREC inLab MC XL milling unit (Sirona Dental Systems Gmbh, Germany). A specific pair of diamond burs for glass ceramics (cylinder pointed bur 12S and step bur 12S, Sirona Dental Systems Gmbh, Germany) was used for machining under intense irrigation. The replacement of the pairs of burs was performed on machine request or failure of a bur.

At the end of the machining process, slight polishing was manually performed, using 400 and 600 grit silicon carbide paper on the upper surface of the discs, to achieve a thickness of 1.4 mm (±0.05 mm) and flatten the surface. The bottom side of the discs was kept unchanged (as-machined) and had its surface roughness measured. After that, discs were randomly distributed among the five experimental groups ($n = 30$ in each group), using the Random Allocation version 1.0 program (developed by M. Saghaei, Department of Anesthesia, Isfahan University of Medical Sciences, Isfahan, Iran). Statistical analysis was performed in order to validate the randomization. No statically significant difference was found in the initial roughness among the groups ($p > 0.05$ in Kruskal–Wallis test). Four additional discs were used for complementary microstructural analysis.

2.2. Determination of glass transition temperature (T_g)

Differential Thermal Analysis (DTA) [33] was performed to estimate the T_g of the ceramic. The analysis was performed in a thermogravimetric analysis instrument (Mettler TGA/SDTA 851e) with approximately 20 mg of material at a heating rate of 1 °C/min from room temperature to 1000 °C, in an oxygen