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Extended glaze firing on ceramics for hard machining: Crack healing, residual stresses, optical and microstructural aspects

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ABSTRACT

Objective. To evaluate the effect of extended and conventional (manufacturer-recommended) glaze firings on crack healing, residual stresses, optical characteristics and crystalline structure of four ceramics for hard machining.

Methods. Rectangular specimens were obtained by sectioning densely sintered feldspathic (FEL), leucite- (LEU), lithium disilicate- (DIS), and zirconia-reinforced lithium silicate-based (ZLS) prefabricated ceramic blocks and divided into groups according to the applied glaze firing (n = 5): conventional glaze/manufacturer-recommended (G), extended glaze (EG) and control/no heat treatment (C). Defects generated by indentation were analyzed by scanning electron microscopy before and after firing (n = 1) to evaluate crack healing. Residual stresses were determined by the indentation technique. Color differences (ΔE) after firing were measured by CIEDE2000 formula, and translucency variations were quantified by contrast ratio. Stability of crystalline microstructure was analyzed by X-ray diffraction.

Results. Regardless of the material, EG had greater ability than G to heal defects, and produced compressive residual stresses, while G generated tensile stresses. Color differences produced by EG were: imperceptible for FEL and LEU ceramics; perceptible, but still clinically acceptable for DIS; clinically unacceptable for ZLS. G produced no perceptible color change. The DIS and ZLS ceramics became ≈1% more opaque after G, ≈4% and ≈15%, respectively, after EG. The crystalline phase of all the ceramics remained stable after G and EG.

Significance. Extended glaze firing could be an alternative to finish feldspathic, leucite-, and lithium disilicate-based ceramic restorations, since it provides greater crack healing than the conventional glaze firing. It develops tolerable residual stresses, and produces clinically acceptable color alterations, without altering the microstructure of these materials.

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1. Introduction

Feldspathic, leucite-, lithium disilicate- and lately, zirconia-reinforced lithium silicate-based ceramics [1] are materials available for so-called “hard machining” [2], in which densely sintered blocks are milled into the desired restoration format. Recently, Belli et al. [3] presented promising life estimates for posterior single unit all-ceramic hard-machined restorations: the expected time for 10% of the restorations to fail was ≥10.9 years. However, ceramic fracture still appears as one of the main technical failures or complications in clinical studies [4–8], and the brittle nature of these materials remains a challenge for the machining process [9,10].

The fabrication of glass ceramic restorations frequently also includes the application of stain oxides and glazing for finishing, which are important for mimicking tooth structure and appearance [11]. According to Denry [9], the inability of manufacturer-recommended finishing procedures, such as glaze firings, to reduce the hard-machining damage indicates that large flaws produced by grinding during the milling stage may become fracture origins. Furthermore, some results suggest a possible deleterious effect of glaze firing on machined glass ceramic materials [11,12]. Therefore, there is a need for studies evaluating the feasibility of alternative firings that will maximize the performance of densely sintered ceramics for CAD/CAM systems.

With this intent, Aurelio et al. [11] extended the glaze firing dwell time at 790 °C from the manufacturer-recommended 1.5 min [13] to 15 min, and followed this by slow cooling, rather than immediate furnace opening, for a hard-machined leucite glass ceramic. The extended glaze had significantly increased the ceramic flexural strength compared to the conventional glaze firing. The authors credited this result in part to the possibility that the extended firing had induced a decrease in flaw depth and sharpness, increasing the stress needed for crack initiation.

Healing of defects in ceramic materials, either by thermal diffusion [14,15] or by oxidation mechanisms [16,17], has been previously studied. Some studies show the ability of certain heat treatments to reduce the viscosity of the glass matrix, causing a decrease in the depth of fissures, crack–tip blunting [18,19], and even sealing of defects [20,21] via capillarity [14,22]. Healing seems to be favored by materials containing silicon (Si), which when subjected to the proper heat treatment, go through an oxidation reaction and generate viscous SiO_2-based products (crystalline or amorphous) which tend to seal the defects [23,24].

Both the magnitude and the profile of residual stresses developed after thermal treatments seem to be important aspects that impact on the ceramics’ mechanical behavior. Studies demonstrate that firings involving significant thermal gradients should be avoided [25,26], since they produce transient and residual stresses on the ceramic surface that induce the propagation of cracks [27]. On the other hand, formation of a protective compressive stress layer on the material surface has been detected after glazing firings [18,19].

Ideally, firings should not interfere with the desired effect of glaze (e.g., brightness, smoothness) or the optical characteristics of the restoration. Thermal cycles must also meet the requirements for a structural balance of the glassy and crystalline phases, as slight variations in the microstructure resulting from firing may produce new mechanical, chemical or physical properties for a specific material [28]. Some studies suggest that, depending on the firing regime adopted, metal oxides responsible for the color of the material may become unstable [29,30] and that heat treatment may lead to alterations in the material constituent phases [12,31,32]. Therefore, it is necessary that studies compile information regarding the effect of heat treatments on the optical properties and microstructure of ceramics.

Given this context, the present study aimed to evaluate the effect of extended and conventional (manufacturer-proposed) glaze firings on crack healing, residual stresses, optical properties and crystalline microstructure of four ceramics intended for hard machining, having different microstructures. The first hypothesis was that an extended glaze firing would have a better ability to heal defects and that it would result in a compressive residual stress state, as compared to a conventional glaze firing. The second hypothesis was that the thermal cycles would not alter either the optical properties (beyond the clinically unacceptable established threshold) or the crystalline phase of the ceramic materials investigated.

2. Materials and methods

Descriptions of the microstructure, composition, and indications of the four ceramics for hard machining used in this study are given in Table 1.

2.1. *Specimen preparation*

Rectangular specimens were obtained by sectioning densely sintered prefabricated ceramic blocks (Table 1), with a diamond disk at low-speed, under water-cooling, in a sectioning machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA). The upper and lower surfaces of the specimens were flattened with 400 and 600 grit silicon carbide papers. One face then received mirror polishing, using 1200 and 2000 grit silicon carbide papers followed by 0–2 μm diamond polishing paste (Christensen Roder, Porto Alegre, RS, Brazil), resulting in specimens of 1.5 ± 0.5 mm thickness at the center (210 MAP micrometer, Starrett, USA). All specimens were cleaned in an ultrasonic bath (1440 D—Odontrobras, Ind E Com Equip Med Dental LTDA, Ribeirão Preto, Brazil) using isopropyl alcohol for 10 min.

2.2. *Experimental groups: glaze firings applied to the ceramics*

Specimens of each ceramic material were randomly allocated to groups having designated firing protocols (Table 2). The control group (C) did not receive any heat treatment; while the conventional glaze (G) and extended glaze (EG) groups were fired in a VITA VACUMAT 6000 MP furnace (VITA—Germany). The G firings strictly followed the manufacturers’ recommendations. The furnace was opened at the end of the heating stage, providing abrupt cooling. The extended schedules (EG) were conducted with the same initial temperature, pre-heating time, and temperature increase rate as the