### ARTICLE IN PRESS

DENTAL MATERIALS XXX (2018) XXX-XXX



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## Fatigue failure load of an adhesively-cemented lithium disilicate glass-ceramic: Conventional ceramic etching vs etch & prime one-step primer

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#### ARTICLE INFO

Article history: Received 7 February 2018 Received in revised form 13 April 2018 Accepted 30 April 2018 Available online xxx

Keywords: Fatigue test Glass ceramic Mechanical behavior CAD/CAM restorations Monolithic restorations Topographical changes

### ABSTRACT

*Objectives.* To evaluate the effect of different glass-ceramic surface treatments and aging on the fatigue failure load of a lithium disilicate glass-ceramic adhesively cemented to a dentin analogue material.

Methods. One hundred and twenty (120) disc-shaped lithium disilicate specimens ( $\emptyset = 10 \text{ mm}$ , thickness = 1.5 mm) were produced and randomly allocated (n = 20) into 6 groups, considering 2 study factors: "surface treatment" in 3 levels (SIL—silane application only; HF5+SIL—5% hydrofluoric acid etching and silane application; ME&P—etching with an one-step ceramic primer), and "storage" in 2 levels (baseline—storage for 7 days; aging—storage for 90 days + 12,000 thermal cycles). Ceramic discs were adhesively cemented to discs of a dentin analogue material ( $\emptyset = 10 \text{ mm}$ , thickness = 2.0 mm) following the manufacturers' instructions. The fatigue failure load was determined by the staircase approach (250,000 cycles; 20 Hz; initial load = 1050 N [~70% of mean load-to-failure]; step size = 52.5 N [5% of initial load]). Micro-morphologic, fractographic, and atomic force microscope analysis were also performed. Fatigue failure load data were evaluated by one-way ANOVA, Bonferroni and t-tests for independent samples.

Please cite this article in press as: Scherer MM, et al. Fatigue failure load of an adhesively-cemented lithium disilicate glass-ceramic: Conventional ceramic etching vs etch & prime one-step primer. Dent Mater (2018), https://doi.org/10.1016/j.dental.2018.04.012

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DENTAL MATERIALS XXX (2018) XXX-XXX

Results. HF5+SIL presented higher fatigue failure load in both conditions (baseline and aging); ME&P presented intermediary mean values, while the SIL group presented the worst performance. All groups had a statistically significant decrease in the fatigue performance after aging.

*Significance.* Hydrofluoric acid followed by silane application showed the best fatigue performance for an adhesively-cemented lithium disilicate ceramic. Aging negatively influenced the fatigue performance for all tested groups.

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### 1. Introduction

The long-term success of indirect ceramic restorations in Restorative Dentistry substantially depends on achieving an adequate adhesion to the substrates (remaining tooth structure and restorative materials) [1]. An enhancement of the stress distribution (occlusal forces transmissions) is consequently provided through the restorative assembly, increasing its ability to support the challenges involved in the oral environment [2].

In regards to the surface treatment to promote adhesion to the intaglio ceramic surface, the materials' composition and its microstructure must be considered [3]. In this sense, glass-ceramics such as lithium disilicate based glass-ceramics are acid-sensitive, i.e. topographical changes are promoted by glassy matrix removal when the ceramic surface is etched with HF acid [4]. This ability plays a crucial role in promoting micromechanical properties interlocking with resin cement [5]. Thus, the conventional surface treatment protocol for bonding glass-ceramics is composed of two consecutive steps: HF acid etching followed by silane coupling agent application. While the HF acid etching promotes topographical changes, the silane is responsible for promoting chemical adhesion between the inorganic matrix of the glass ceramic and the organic matrix of the resin cement through siloxane links [6,7]. Besides the chemical adhesion, silanes have been shown to reduce the contact angle and increase the wettability of the resin cement on the ceramic surface [8].

However, other factors to be considered are that HF acid etching has the potential to impair the mechanical properties of glass ceramics [3,9–11], and it is also considered as a highly toxic hazardous material [12,13]. The impairment of the mechanical properties is generated by introducing critical defects by glass corrosion/dissolution by HF acid etching, as critical defects are responsible for premature failure under lower loads (in comparison to the nominal strength of the ceramic material) [14]. The high toxicity is due to the potential of HF acid to generate necrosis of soft tissues and bones over prolonged exposure time [13]. In response to HF acid etching, these two deleterious mechanisms have been shown to be time- and concentration-dependents [11,13,15,16].

Recently, a new self-etching ceramic primer (ME&P; Monobond Etch & Prime, Ivoclar Vivadent; Schaan, Liechtenstein) has been introduced as a simplified alternative to the traditional HF acid etching+silane protocol for treating the glass-ceramic surfaces. ME&P is basically a ceramic primer composed of ammonium polyfluoride, a silane system based on trimethoxypropyl metacrylate, solvents (alcohols and water), and a pigment (providing visibility with a green color aspect) in an all-in-one system (one-step etching technique). Therefore, this simplified technique for adhesion requires shorter time, might prevent the weakening effect of glass-ceramics (mild etching-less aggressive), and decreases hazardous potential. Until now, a few studies have been published on the bond strength of ME&P, demonstrating promising results [17–20]. However, data about fatigue performance of adhesively cemented restorations using the ME&P as ceramic surface treatment has not been tested yet.

Therefore, based on the current and aforementioned knowledge regarding a lack of data comparing the fatigue performance of ceramic restorations cemented with this new etching approach (ME&P) in relation to the conventional protocol (HF acid etching + silane application), this study aimed to evaluate and compare the effects of ceramic surface treatments and aging on the fatigue failure load of a lithium disilicate glass-ceramic adhesively cemented to a dentin-like analogue material. The study hypotheses were: (1) the ceramic surface conditionings will promote similar fatigue performance; (2) the fatigue failure loads will not be influenced by aging/thermocycling.

#### 2. Materials and methods

The description of materials used in the present study, commercial name, manufacturers, and batch numbers are presented in Table 1.

#### 2.1. Preparation of the ceramic specimens

Lithium disilicate ceramic blocks (IPS e.Max CAD, Ivoclar Vivadent; Schaan, Liechtenstein) were shaped into cylinders by a drilling machine (SBE 1010 Plus, Metabo; Nürtingen, Germany), under water cooling. Next, a cutting machine (Isomet 1000, Buehler, Lake Bluff, USA) with a diamond disc (Buehler Isomet Wafering blade series 15LC no. 11-4276 with 6 inches' diameter (152 mm) and 0.020 in (0.5 mm) in thickness, being a medium coarse diamond grinding tool) was used to produce 120 disc-shaped ceramic specimens ( $\emptyset$  = 10 mm), simulating the average occlusal table of a first molar [21]. The occlusal side of all the ceramic discs were flattened and polished (EcoMet/AutoMet 250, Buehler) with silicon carbide papers (#120-, #400- and #1200-grit, 3M; Sumare, Brazil) until a final thickness of 1.5 mm (minimum thickness recommended by the manufacturer for a posterior restoration),

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