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Precision of different fatigue methods for predicting glass-ceramic failure

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ABSTRACT

This study aimed to characterize the fatigue behavior using two fatigue methods, boundary and staircase, and to predict the probability of failure (P_f) of zirconia-reinforced lithium silicate glass-ceramic (ZLS). Bar-shaped specimens of ZLS (18 × 4 × 1.2 mm) were fabricated. Thirty specimens were subjected to a three-point flexural strength test using a universal testing machine with 0.5 mm/min crosshead speed, in 37 °C distilled water. Flexural strength data were analyzed with Weibull statistics. Eighty-six bars were subjected to cyclic fatigue using boundary and staircase methods. Fatigue tests were performed in a pneumatic cycling machine (2 Hz, 37 °C distilled water) for 10^3 and 10^4 cycles. Fatigue data were analyzed using an inverse power law relationship and log normal-lifetime distribution. Fracture toughness (K_{Ic}) was determined using V-notched specimens (18 × 4 × 3 mm) and the short beam toughness method (n = 7). Vickers hardness (VH) was evaluated (4.9 N, 20 s). Fractographic and EDS analyses were also performed. ZLS showed a characteristic strength of 197 MPa, Weibull modulus of 4, VH of 6.67 GPa and K_{Ic} of 1.93 MPa m$^{1/2}$. After 10^3 cycles, for both methods, there was a degradation of 78% of the initial strength. There was no significant degradation when the number of cycles increased from 10^3 to 10^4. Both methods resulted in similar P_f and precision at 40 MPa (~50% P_f). Yet, staircase shows good accuracy and precision in predicting the stress amplitude for a P_f near 50%; while boundary is also effective for P_f lower than 50%. The fatigue methods evaluated show similar accuracy and precision for predicting the P_f of a glass-ceramic when simulations were made in the range of stress levels and lifetimes used in the fatigue tests.

1. Introduction

Ceramics successfully achieve the aesthetic and biocompatibility requirements for dental restorations. Improvements on the mechanical behavior and processing quality of ceramics were obtained by the development of materials with high crystalline content and the introduction of the CAD/CAM (computer-aided design/ computer-aided manufacturing) technology in dentistry (Borba et al., 2011; Lohbauer et al., 2008; Sailer et al., 2015).

Glass-ceramics reinforced with crystals, such as leucite and lithium disilicate, can be used to produce monolithic restorations for the anterior region as they show a good balance between optical and mechanical properties (Della Bona et al., 2004). The literature reports an annual failure rate of 0.69% and a 5-year survival rate of 96.6% for these glass-ceramics, catastrophic fracture being the most frequent failure mode (Sailer et al., 2015).

More recently, a zirconia-reinforced lithium silicate glass-ceramic (ZLS) was developed to produce inlays, onlays, partial crowns, veneers, anterior and posterior crowns. ZLS features a dual microstructure with a very fine phase of crystals of lithium metasilicate and lithium disilicate (0.5–0.7 µm) and a glassy matrix containing zirconia (Belli et al., 2017; Ramos Nde et al., 2016). According to the manufacturer, ZLS is composed of ZrO_2 (8–12 wt%), Li_2O (15–21 wt%) and SiO_2 (56–64 wt %). Previous studies reported that ZLS presents fracture toughness between 1.3 and 4.7 MPa m$^{1/2}$, flexural strength from 207 MPa to 611 MPa, elastic modulus between 66 and 103 GPa, and hardness of 6.53 GPa (Belli et al., 2017; Elsaka and Elnaghy, 2016; Traini et al., 2016; Wendler et al., 2017). The bond strength between ZLS and resin cements varies from 20 to 30 MPa (Hu et al., 2016; Sato et al., 2016). However, the literature regarding ZLS lacks clinical trials and only a few laboratory studies simulate in-service conditions (Monteiro et al., 2018; Preis et al., 2017; Wendler et al., 2018).

It is important to characterize the fatigue behavior of dental ceramics because fatigue tests can simulate the oral environment conditions more closely than fast fracture tests (Baran et al., 2001; Wendler et al., 2018). Fatigue failure in ceramics occurs when the material is subjected

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to low stress levels over time, in a humid environment, leading to slow and subcritical growth of the crack, until it reaches a critical size (Wiskott et al., 1995; Gonzaga et al., 2011). In addition, extrinsic and intrinsic fatigue mechanisms may be involved in the degradation of the mechanical properties of the ceramics, depending on their composition and microstructure (Ritchie, 1999; Belli et al., 2014).

Nevertheless, conventional fatigue tests are time-consuming and require a large number of specimens. Therefore, accelerated fatigue methods were developed such as the boundary (Lodi et al., 2017; Maennig, 1975; Vicari et al., 2018; Zhang and Griggs, 2003) and the staircase techniques (Dixon and Mood, 1948; Collins, 1993). For both methods, the frequency of failure after a preset lifetime (number of cycles) is the dependent variable. In the staircase technique, specimens are individually tested and depending on the presence or absence of failure, the stress for the subsequently tested specimen is decreased or increased (Dixon & Mood, 1948; Collins, 1993). For the boundary technique, two groups of specimens are evaluated for each lifetime, one is tested with a stress amplitude corresponding to a high probability of failure and the other with a stress amplitude corresponding to a low probability of failure (Maennig, 1975; Zhang and Griggs, 2003).

Ceramics are flaw-sensitive and different mechanisms are involved in fatigue failure, which results in high data scatter. Therefore, it is important to investigate how accurate and precise these fatigue methods are in predicting the probability of failure of dental ceramics. Accuracy refers to how close the predicted values are to the true value, while precision refers to the variability of the estimates. Thus, this study aimed to evaluate the precision of two fatigue methods, boundary and staircase, for predicting the probability of failure (P_f) of a glass-ceramic, testing the hypothesis that the methods show similar precision. In addition, this study characterized important mechanical properties (flexural strength, microhardness, fracture toughness) and the fatigue behavior of a zirconia-reinforced lithium silicate glass-ceramic.

2. Material and methods

2.1. Specimens preparation

One hundred and sixteen bar-shaped specimens (1.2 mm × 4 mm × 18 mm) of zirconia-reinforced lithium silicate glass-ceramic (ZLS, VITA Suprinity, Vita Zahnfabrik, Germany) were fabricated. Specimens were obtained by cutting CAD/CAM blocks using a diamond disc in a metallographic cutting machine (Miniton, Struers, Copenhagen, Denmark) under constant water irrigation. They were flattened and polished (240–1200 grit papers) and edges were chamfered using a metal device and 800 grit paper, according to ISO 6872 recommendations (ISO-, 6872, 2015). ZLS specimens were subjected to a crystallization cycle in a ceramic furnace (VITA Vacumat 6000 MP, Vita Zahnfabrik, Germany), following the manufacturer recommendations.

2.2. Three-point flexural strength test

Thirty specimens were subjected to a three-point flexural strength test using a universal testing machine (DL 2000, EMIC, São José dos Pinhais, Brazil) with 0.5 mm/min crosshead speed. The test was performed in 37 °C distilled water. The flexural strength values (σ_f) were calculated using Eq. (1) (ISO-, 6872, 2015):

$$\sigma_f = \frac{3Pl}{2wb^2} \tag{1}$$

where P is the fracture load (N), l is the distance between the supports (span = 12 mm), w is the width (mm) and b is the thickness (mm) of the specimen.

The Weibull modulus (m) and characteristic strength (σ_0) were determined by analyzing the flexural strength data, according to the two-parameter Weibull distribution. The 95% confidence intervals were calculated through tabulated values.

2.3. Fatigue test

Specimens for the cyclic fatigue test were tested in the same configuration as the fast fracture test (three-point flexure). Therefore, Eq. (1) was used to define the load level applied to induce the required stress. Specimens were tested in fatigue using a pneumatic mechanical cycling machine (Biopid, Biocycle, Sao Carlos, Brazil) with 2-Hz frequency in 37° C water.

Eighty-six specimens were tested in fatigue. Sixteen specimens were used to determine the first stress level for fatigue testing. For each method, twenty specimens were considered for testing in each lifetime, 10^3 and 10^4 cycles. Nevertheless, a different number of specimens was required for each technique. For staircase, 40 specimens were needed. For the boundary technique, one group of specimens was evaluated in two different lifetimes, resulting in an amount of 30 specimens.

In both methods, the probability of failure (P_f) of the specimens can be estimated only for lifetimes used in the fatigue tests; therefore, more than one lifetime was choosen. Yet, for the boundary technique at least two lifetimes are required to complete the method (Maennig, 1975; Zhang and Griggs, 2003). Lifetimes used in this study were based in ZLS flexural strength data.

2.3.1. Boundary method

To define the stress level for the first testing protocol, specimens were tested individually during a preset lifetime until the first one survives (Maennig, 1975; Zhang and Griggs, 2003). The initial stress for the first specimen ($\sigma_{initial}$) was determined considering 75% of the mean value obtained in the flexural strength test, being 132 MPa (σ_f x 0.75). $\sigma_{initial}$ was reduced using a step value of 5.5 MPa ($\delta = 0.04 \times \sigma_{initial}$) for each specimen, until the first specimen survived 10^3 cycles (n = 16).

Next, a group of 10 specimens were tested at this first stress level (σ_1) - 46 MPa - during 10^3 cycles, and 90% of the specimens failed (n = 10; i = 9). The second stress level (σ_2) was calculated according to Eq. (2) (Zhang and Griggs, 2003):

$$\sigma_2 = \begin{cases} \sigma_1 + S.\left(1 - \dfrac{i}{n}\right).\sigma_1 & \rightarrow \quad i < 0.5n \\ \sigma_1 - S.\dfrac{i}{n}.\sigma_1 & \rightarrow \quad i \geq 0.5n \end{cases} \tag{2}$$

where i is the number of specimens that failed up to the preset number of cycles in σ_1; n is the total number of specimens tested in σ_1; and S is a constant selected to minimize the chance of all or none of the specimens failing at σ_2, being 0.178.

Thus, another 10 specimens were tested with σ_2 (40 MPa, 10^3 cycles), and 40% failed (n = 10; i = 4). The specimens that survived 10^3 cycles at σ_2 were allowed to run out through the next lifetime (σ_2 for 10^3 cycles was used as σ_1 for 10^4 cycles). Three more specimens failed, resulting in 70% failure rate between 0 and 10^4 cycles (n = 10; i = 7). Based on these data, the second stress level (σ_2) for the second lifetime (10^4 cycles) was calculated, resulting in 34 MPa (Eq. (2)). Ten new specimens were cycled and 5 failed between 0 and 10^4 cycles (n = 10; i = 5). Fig. 1 presents the boundary fatigue protocol performed.

To find the probability of fracture at σ_1 and σ_2, Eq. (3) was used:

$$P_f = \frac{(i+0.1)}{(n+0.2)} \tag{3}$$

To predict the stress levels corresponding to 50% ($\sigma_{50\%}$) and 5% ($\sigma_{5\%}$) failure probabilities of ZLS after 10^3 and 10^4 cycles, the fatigue data curve was adjusted using the Weibull parameters (m and σ_0) obtained with the fast fracture flexural test (Table 1). The best fit for each fatigue dataset was obtained after shifting to the right on the ln σ axis by adding 1.53 (a_t) for 10^3 cycles and 1.65 for 10^4 cycles, using the