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Influence of leucite content on slow crack growth of dental porcelains

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ABSTRACT

Objectives. To determine the stress corrosion susceptibility coefficient, *n*, of seven dental porcelains (A: Ceramco I; B: Ceramco-II; C: Ceramco-III; D: d.Sign; E: Cerabien; F: Vitadur-Alpha; and G: Ultropaline) after aging in air or artificial saliva, and correlate results with leucite content (LC).

Methods. Bars were fired according to manufacturers' instructions and polished before induction of cracks by a Vickers indenter (19.6 N, 20 s). Four specimens were stored in air/room temperature, and three in saliva/37 °C. Five indentations were made per specimen and crack lengths measured at the following times: ~0; 1; 3; 10; 30; 100; 300; 1000 and 3000 h. The stress corrosion coefficient *n* was calculated by linear regression analysis after plotting crack length as a function of time, considering that the slope of the curve was $[2/(3n+2)]$. Microstructural analysis was performed to determine LC.

Results. LC of the porcelains were 22% (A and B); 6% (C); 15% (D); 0% (E and F); and 13% (G). Except for porcelains A and D, all materials showed a decrease in their *n* values when stored in artificial saliva. However, the decrease was more pronounced for porcelains B, F, and G. Ranking of materials varied according to storage media (in air, porcelain G showed higher *n* compared to A, while in saliva both showed similar coefficients). No correlation was found between *n* values and LC in air or saliva.

Significance. Storage media influenced the *n* value obtained for most of the materials. LC did not affect resistance to slow crack growth regardless of the test environment.

1. Introduction

Porcelains are highly esthetic materials used as monolithic restorations (inlays, onlays and veneers) or as veneering materials over core ceramics in bilayered crowns and fixed partial dentures (FPDs). However, due to relatively low fracture toughness, unwanted fracture rates have been reported for such restorations and prostheses in clinical trials [1–3]. The fracture of ceramics in service occurs with little or no plastic deformation when a small flaw or crack propagates in an unstable manner under applied tensile stress (i.e. catastrophic failure) [4]. For a body containing a crack of length *c*, subjected to a

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tensile stress σ, the stress intensity factor at the crack tip is given by:

$$K_I = \sigma Y \sqrt{c} \quad (1)$$

where Y is a dimensionless constant which depends on the stress mode, shape and dimensions of the material, geometry and length of the crack. Fracture occurs when the critical level of stress intensity factor (K_{Ic}) is reached.

When subjected to a stress intensity factor below the critical level ($K_I < K_{Ic}$), the defects in ceramic materials may present a slow and stable growth, mainly in a humid environment such as the oral cavity. This phenomenon is referred as slow or subcritical crack growth (SCG) and leads to strength degradation over time [5]. The presence of water at the tip of a crack under stress results in the rupture of the metallic oxides bonds of the material, with the subsequent formation of hydroxyls. As a consequence of SCG, a defect may reach critical size (under a determined applied stress) and result in fast fracture [6]. The subcritical crack growth is notable for its extreme sensitivity to applied load and it tends also to depend on the concentration of environmental species, temperature and other extraneous variables [7].

The oral environment has many elements that favor SCG in ceramic restorations, such as: (a) water from saliva; (b) water from the luting cement and from dentin tubules; (c) masticatory stresses; (d) stresses associated with differences in the coefficient of thermal expansion of the restoration components; (e) temperature variations; and (f) pH variations [8]. Ceramics containing glassy phase, like dental porcelains, are highly susceptible to SCG [9], therefore the slow growth of defects is expected to be a common finding in such restorations, resulting in a decrease of their strength and in-service reliability [10].

The phenomenon of SCG in ceramic materials can be characterized by the stress corrosion susceptibility coefficient, n, which is calculated using the following empirical power law equation [11]:

$$v = \frac{dc}{dt} = v_0 \left(\frac{K_I}{K_{Ic}} \right)^n \quad (2)$$

where v is the crack velocity at an applied stress intensity factor (K_I), c the crack size, t the time, v_0 the critical velocity of the crack at the moment of fracture, and K_{Ic} is the fracture toughness. Since $K_I/K_{Ic} < 1$, a higher n value means higher resistance to SCG and consequently longer service life.

The coefficient n can be measured by direct or indirect methods. In the direct methods, the crack velocity (v) is determined by measuring the crack length in a time interval under different levels of K_I. Examples of these techniques are the "double cantilever beam method" and "double torsion" [12]. The main advantage of such techniques is that they allow for determination of v using a large range of K_I. However, they have shortcomings such as the need of large specimens with large cracks [11]. The indirect methods are used to determine the SCG parameters by means of flexural strength tests. The most used techniques are the dynamic fatigue test, which determines strength at different stress rates, and the static fatigue test, which measures time to fracture of different static loads [9]. The indentation fracture method (IF) is an alternative to the previously mentioned techniques in which the lengths of the cracks generated by a Vickers indenter are measured over time [13]. This method allows for determination of n by means of correlation plots between time and crack size.

The microstructure of a ceramic material is known to strongly affect crack propagation and its mechanical properties [14]. In this regard, it has been demonstrated that dental porcelains with higher amounts of leucite are more resistant to fast crack propagation, and have higher fracture toughness due to the phenomenon of crack deflection around leucite particles [15]. Therefore, it is expected that the presence of leucite in the glassy matrix of porcelains will also hinder slow crack propagation. The objective of this study was to determine the stress corrosion susceptibility coefficient, n, of seven dental porcelains after aging in air or artificial saliva, and correlate results with leucite content (LC). The hypotheses to be tested are: (1) the leucite content of the material influences its n value; and (2) the n value will depend on the storage media.

2. Materials and methods

Seven dental porcelain powders were used: A, B, C, D, E, F and G (Table 1). Porcelains A, B, C, D and G are indicated for porcelain-fused-to-metal and all-ceramic restorations, and porcelains E and F are indicated to be used as veneering material for alumina-based cores. The different types of porcelain were chosen in order to provide a wide variation of leucite contents. Porcelains E and F are silica-rich materials, while the other materials are feldspathic porcelains. Materials E and F had to be chosen to represent leucite-free materials because apparently there are no commercial feldspathic porcelains without leucite. Green specimens (5 mm × 6 mm × 30 mm) were prepared by the vibration–condensation method using a stainless steel mold and sintered in a dental porcelain furnace following the firing schedules recommended by the manufacturers (Table 2). The reproducibility of the specimen preparation was monitored by sintered density measured by Archimedes' method (the coefficient of variation was 0.5% or less for most of the porcelains, except for that of porcelain E, which was 1.6%). After firing, the specimens were machined to the dimensions of 3 mm × 4 mm × ~25 mm, following the guidelines in ASTM C 1161 [16]. Then, the 4-mm side was mirror polished using a polishing machine (Ecomet 3, Buehler, Lake Bluff, ILL, USA) with diamond suspensions (45, 15, 6 and 1 μm).

Radial cracks were generated on polished surfaces with a Vickers microhardness tester MVK-H-3 (Mitutoyo, São Paulo, Brazil) with load of 19.6 N and dwell time of 20 s. This load was chosen in order to generate long radial cracks ($c/a > 2.5$, where c is the crack length and a is half of the impression diagonal) in all porcelains. Higher loads caused chipping of the porcelain surface near the indentation. It has been demonstrated that Vickers hardness of ceramic materials is not independent of load [17]. Moreover, a previous work showed that the Vickers hardness of a feldspathic porcelain varied significantly when lower loads were used (from 2.0 to 4.9 N), however the hardness values did not vary when higher loads were applied (from 9.8 to 49.0 N) [18]. In the present work, the indentation load was the same for all materials because the effect of the inden-