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Dynamic Fatigue Behavior of Dental Porcelain Modified by Surface Deposition of a YSZ Thin Film

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Abstract

Purpose: The objective of this study was to evaluate the basic fatigue parameters of a dental porcelain modified by deposition of a yttria-stabilized zirconia (YSZ) thin film and to compare the data to that of an unmodified control.
Methods: Two hundred bars ($2 \times 2 \times 15$ mm^3) were cut from ProCAD blocks. Specimens were wet-polished with 1200-grit SiC abrasive. One surface of each bar was sandblasted with 50 μm Al_2O_3 abrasive (50 psi). Half the specimens were further modified through deposition of a 3-μm YSZ thin film on the sandblasted surface. Depositions were performed using an radio frequency magnetron sputter system (working pressure of 15 mT, 150°C, 30:1 Ar:O_2 gas ratio). Specimens were tested at different stressing rates: 5.0, 0.1, and 0.01 MPa/s (n = 25/group) in deionized water (37°C), and inert strength was determined in air (25°C, 70 MPa/s). All strength measurements were carried out by three-point bending (span = 10 mm) in a servo-electric test system.
Results: The mean flexural strength values (MPa) and standard deviation for the uncoated sandblasted group were: 98.6 (5.5), 90.7 (5.9), and 84.2 (8.5), and for the sandblasted + YSZ thin film group: 125 (9.4), 119.3 (7.8), and 102.8 (7.0), for the highest to the lowest stressing rates, respectively. The fatigue parameters n and lnB were calculated by linear regression of dynamic fatigue data. For the uncoated group, n = 38 and lnB = 4.7 MPa^2/s, and for the coated group, n = 33 and lnB = 10.8 MPa^2/s. Weibull analysis was also performed showing that the characteristic parameter (σ_o) was 113.3 and 125.7 MPa for the uncoated and coated group, respectively.
Conclusions: There was an increase in strength for specimens modified by application of a YSZ thin film. It is hypothesized that thin-film application modifies flaws or residual surface stress states.

Ceramic materials have been used in dentistry for several years. In the past 15 years a new chairside approach for the fabrication of all-ceramic restorations has gained considerable attention—computer-aided design/computer-aided manufacturing (CAD/CAM) technology.[1,2] Even though these restorations have improved in terms of fabrication and material properties, the main cause of clinical failure stills remains the same: fracture of the restoration.[2]

Dental ceramics are inherently brittle and susceptible to fatigue and subsequent premature failure, especially when they are in moist environments, under high forces, and repetitive stresses during the chewing cycle.[3] The performance of all-ceramic systems remains less stable than that of metal–ceramic systems. Porcelain cracking and bulk fracture can affect 5 to 10% of single-unit all-ceramic prostheses within the first 6 years after placement.[4]

The continued search for strong and tough ceramic materials has led to the integration of zirconia in restorative dentistry. Zirconia offers high flexural strength and toughness, showing promising properties not only in its bulk form but also as sputtered, thin-film coatings.[5-7] Sputtering is one of the most commonly used technologies for the deposition of ceramic thin-film materials. Previous studies have shown that the application of alumina thin films may enhance the fracture strength of borosilicate glass and porcelain.[8,9] It is hypothesized that the application of yttria-stabilized zirconia (YSZ) thin films to all-ceramic restorations will lead to increased longevity and reliability of dental prostheses.

To understand the clinical potential and limitations of a dental ceramic modified by different surface treatments, the strength of the substrate material is the first factor that needs to be assessed, because strength is a relative measure of resistance to crack initiation, and an indication of initial flaw distributions.[6] Flexural strength, measured following well-defined standards, is usually considered a basic method to evaluate the mechanical properties of ceramic materials.[10,11] In addition, the strength reliability and variability of brittle materials should be studied, as their failure stresses are statistically scattered as a function of the flaw size distribution in the material.[10]

Weibull analysis, combined with fracture mechanics principles, forms the probabilistic basis for the design of ceramic materials, and is a common method to study strength reliability and variability. The two-characteristic Weibull parameters are the Weibull modulus (m) and characteristic strength (σ).[12] A high Weibull modulus indicates smaller average defects and greater structural reliability.[12,13]

Dynamic fatigue testing has also been used to characterize the mechanical performance of ceramic materials. Fatigue refers to the slow growth of cracks inherent in the material as a result of fabrication.[14,15] Furthermore, the strength of all-ceramic restorations may also be decreased by stress corrosion, a stress-dependent chemical reaction between water and surface flaws that causes the flaws to grow more rapidly to a critical dimension, allowing spontaneous crack propagation.[15,16] Dynamic stress testing was first described by Evans[15] and later defined as a distinct test modality by Ritter et al.[16] This test procedure makes use of several constant stressing rates to perform strength tests. From such data, the fatigue parameters can be calculated. These fatigue parameters, n and B, are, respectively, the crack growth exponent from the crack velocity expression and a materials constant, which is dependent on the test environment and the inert strength.

The aim of the present study was to evaluate the basic fatigue parameters of a dental porcelain modified by deposition of a YSZ thin film, and to compare the data to that of an unmodified control. The hypothesis to be tested was that the YSZ thin film-modified ceramic has a higher flexural strength and fatigue limit when compared to the unmodified control.

Materials and methods

ProCad blocks (Ivoclar Vivadent, Schaan, Liechtenstein; lot F67160) made of leucite-reinforced feldspathic porcelain were used in this study. The porcelain blocks were cut into plates, and then into bars, using a low speed saw. Two hundred bars, measuring approximately $2 \times 2 \times 15$ mm^3 were polished with 1200-grit SiC abrasive and had their edges rounded to limit edge failures.

One surface of each bar was abraded by sandblasting with 50 μm Al_2O_3 powder at a pressure of 0.34 MPa for 10 seconds. Bars were then cleaned with the use of an ultrasonic device in isopropyl alcohol to remove any surface debris prior to sputter coating. The porcelain bars were randomly divided into a control group (uncoated) and a group to be modified with YSZ thin film (coated).

Depositions were performed using a radio frequency magnetron sputter system (Model SC-400; CVC, Rochester, NY). The target material was 99.99% pure zirconia doped with 3 mol% yttria (SCI Engineering, Columbus, OH). The porcelain bars were placed in a jig directly above a sputtering target (US Gun, San Jose, CA) positioned in the center location of the system (75-mm working distance). Depositions were performed at a working pressure of 15 mT, 150°C temperature, and a 30:1 Ar:O_2 ratio of oxygen input flow rate. To ensure steady-state plasma flux, presputtering of the target material was performed for 10 minutes before the shutter was opened, exposing the bars' surfaces to the plasma. After coating, the specimens were stored for 48 hours at room temperature (25°C) in ambient conditions.

Fracture strength values were then obtained from 25 specimens per group using a three-point bending fixture (span = 10 mm) in a servo-electric mechanical testing system (Evolution; MTS, Minneapolis, MN) in air at 25°C. The materials were tested at a very high stressing rate (70 MPa/s) to avoid subcritical crack propagation and record the inert strength of the material. The variability of the flexural strength values was analyzed using the two-parameter cumulative Weibull distribution function. Strength data of ceramic materials usually show an asymmetrical distribution. The Weibull distribution is often used for this type of analysis. The description of Weibull distribution is given by the formula:

$$p(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)\right]^m \quad (1)$$

where p is the probability of failure, σ is the strength at a given p, σ_0 is the characteristic parameter at the fracture probability of 63.2%, and m is the Weibull modulus.[12] The statistical variability of estimates for the two-characteristic Weibull parameters, σ_0 and m, were determined by a maximum-likelihood approach.

Dynamic fatigue tests were carried out in an environmental chamber filled with distilled deionized water at 37°C, to indirectly measure the environment-dependent slow crack growth parameter. Specimens were tested at three stressing rates (n = 25/group): 0.01, 0.1, and 5 MPa/s for the coated and uncoated groups. The dependence of strength on stressing rate, caused by subcritical crack growth is described by:

$$\sigma_f = \left[B(n+1)\sigma_i^{n-2}\dot{\sigma}\right]^{1/n+1} \quad (2)$$

where σ_i is the inert strength, and B is a parameter associated with A, n, fracture toughness, crack geometry, and loading configuration. The SCG parameter, n, and fatigue parameter, B, can be determined from a plot of $\ln \sigma_f$ as a function of $\ln \dot{\sigma}$ by linear regression of the data:

$$\ln\sigma_f = \frac{1}{n+1}\ln\dot{\sigma} + \ln\beta \quad (3)$$

where

$$\beta = \frac{1}{n+1}\left[\ln B + \ln(n+1) + (n-2)\ln\sigma_i\right]. \quad (4)$$

Table 1 Mean flexural strength of uncoated and coated porcelain

Stressing rates (MPa/s)	Uncoated (MPa ± SD)	Coated (MPa ± SD)
70 (inert)	109.8 (7.5)[d]	119.4 (14.3)[b]
5	98.6 (5.5)[c]	125 (9.4)[b]
0.1	90.7 (5.9)[b]	119.3 (7.8)[b]
0.01	84.2 (8.5)[a]	102.8 (7.0)[a]

Similar superscript letters in columns indicate that groups are not statistically different ($p > 0.05$).

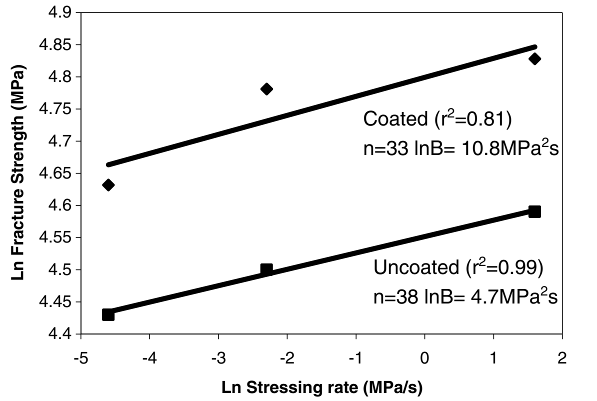

Figure 1 Dynamic fatigue data and fatigue parameters of the uncoated and coated porcelain.

Results

The results (Table 1) showed a significant increase in the strength of the coated group when compared to the uncoated group for all stressing rates tested. Analysis of variance followed multiple comparisons; Tukey tests were also carried out for comparisons among groups. A significant decrease in strength values was noticed from the highest to the lowest stressing rate for the uncoated group; however, no significant differences were observed for the YSZ-coated group, except for the lowest stressing rate.

Weibull statistics (Table 2) demonstrated that the coated groups showed higher values for the characteristic strength than did the uncoated groups, regardless of the stressing rate. The Weibull modulus varied according to each stressing rate evaluated, remaining between 8.9 and 19.2 for the coated group and between 12 and 19.2 for the uncoated one. The dynamic fatigue parameters showed (Fig 1) relatively similar n values for the uncoated (n = 38) and coated groups (n = 33), with lnB being higher for the coated group.

Randomly selected specimens were placed on SEM stubs, gold sputter-coated, and examined under a scanning electron microscope (Model JSM-6300; JEOL Ltd., Tokyo, Japan) at 15 KV. Figure 2 shows SEM micrographs taken from the surface of representative specimens with and without a YSZ coating, and also from the specimen fracture surfaces (after loading) to confirm the thickness of the deposited thin film.

Discussion

The development of new techniques or processes to improve mechanical reliability and increase the strength of ceramics is of great importance for biomedical applications. In particular, in dentistry there are a number of restorative ceramic materials being used that have relatively low strength and fracture toughness, such as machinable dental porcelain.[1,2,17] The use of the RF sputtering technique to deposit YSZ thin films on dental porcelain shows promising results. This technique could be applied to all-ceramic materials in principal, to modify inherent surface flaws, suppress crack propagation, thereby improving fatigue behavior and fracture resistance, and potentially having a broader application than developing new materials and material systems.

In our study, the strength of YSZ-modified porcelain significantly increased compared to the uncoated material. As strength is likely related to microcrack-like defects inherent in the micro-structure of the processed porcelain, it is possible that the initial flaws or defects present in the specimens might be blunted or passivated by the thin film, reducing the incidence of crack initiation. In addition to the flexural strength values, the characteristic strength values of the coated specimens were higher than the corresponding values of uncoated specimens for all stressing rates tested (Table 2). On the other hand, the Weibull modulus was similar in both groups, which leads us to conclude that the homogeneity of flaw distribution might not have changed, and similar critical flaws are still present in a certain volume of material. A relatively high Weibull modulus indicates better material reliability.[10,12]

Regarding the fatigue of modified and unmodified porcelain, other studies have reported this phenomenon when associated with glass-silicate materials, in which the characteristics of materials change over time under constant conditions.[14,18] It is apparent from the data that slow crack growth is occurring for both sets of samples, as strength values are dependent on stressing rate. It was assumed that the modifying films would improve the fatigue strength of the porcelain examined in this study.

Many materials are subjected to stress corrosion, which is aggravated by surface flaws, and because the coating is probably modifying surface flaws, stress corrosion resistance might be improved, preventing crack propagation. However, the films did not exhibit a large improvement in fatigue characteristics. Although other studies have shown that bulk zirconia is less

Table 2 Fracture strength characteristics of uncoated and coated porcelain according to stressing rates

Parameters Stressing rates (MPa/s)	Weibull modulus (m)		Characteristic strength σ63.2%	
	Uncoated	Coated	Uncoated	Coated
70	14.5	8.9	113.3	125.7
5	19.2	14.4	101	129.3
0.1	16.8	19.2	93.4	122.6
0.01	12	14.4	87.7	106

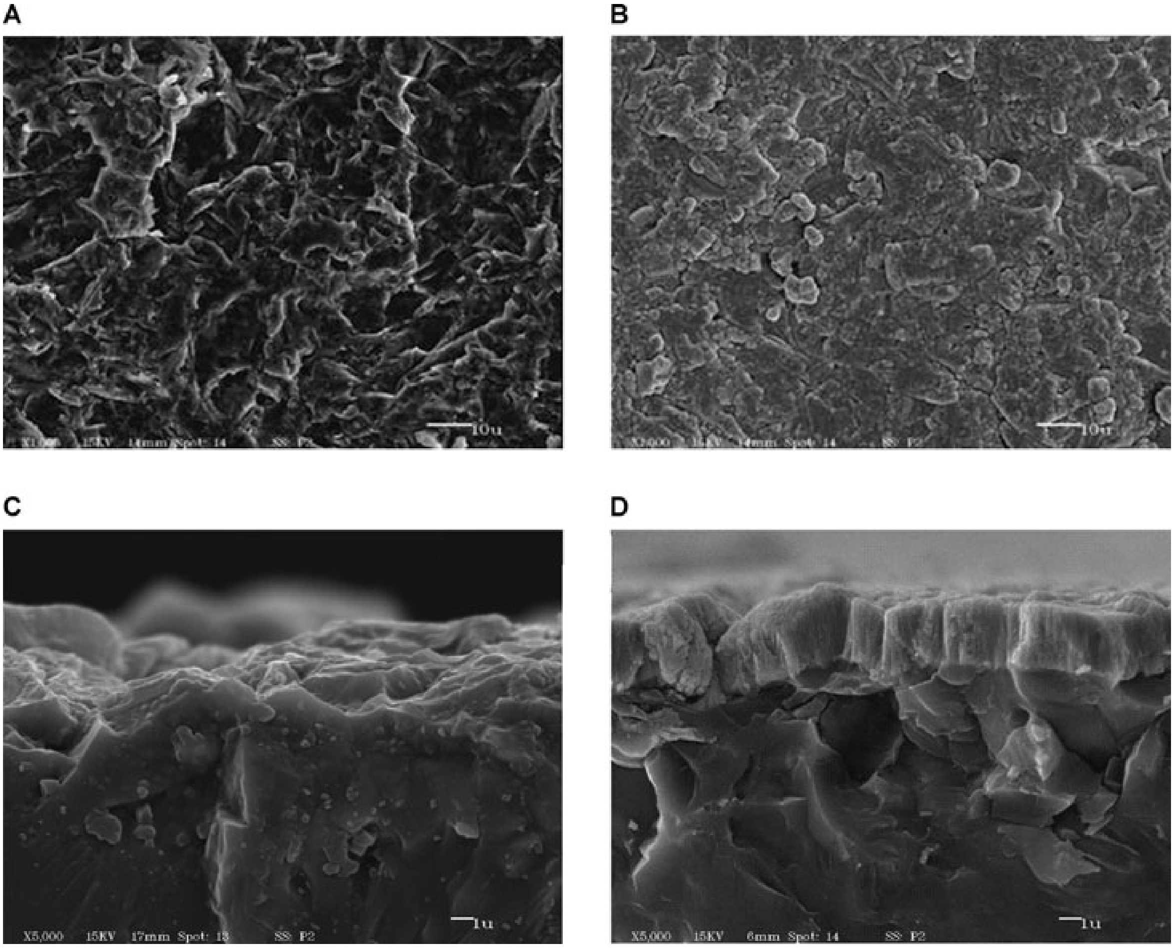

Figure 2 Scanning electron micrographs of the sandblasted surface (A) and the fracture surface of a ceramic bar (B), and surface (C) and fracture surface of a ceramic bar with YSZ coating (D).

susceptible to stress corrosion than porcelain, zirconia is still prone to degradation.[18,19] Previous studies have also shown that the mechanical properties of thin films can change over time.[7] It is therefore possible that a protective layer against water adsorption/absorption may prevent or lessen the severity of environment-assisted degradation of the zirconia films over time.

SEM micrographs show a coherent interface between the YSZ thin film and the porcelain substrate. In addition, the columnar grain structure of the films can be seen. Because most cracks will propagate through grain boundaries, it is undesirable to have parallel-aligned grain boundaries to the applied flexural forces.[20] A preferred microstructure would be one that is more equiaxed, yet still dense and fine-grained. The effect of different types of thin films on the strength of a brittle substrate is under investigation.

Different factors can influence the fracture mode of ceramic crowns, including the geometry of the crown.[19,21] In this study, standard specimens for three-point bending were used. A limitation of this study was that the specimens were prepared according to standardized preparation criteria. Further studies are necessary to evaluate the effect of these thin films on the cyclic fatigue of actual dental ceramic crowns or crown analogues, to better predict the clinical behavior of a YSZ-modified material.

It is assumed that thin film modified dental ceramics will be stronger than unmodified equivalents based on the results; however, this increase might not be enough to justify the use of the technique if the material cannot withstand long-term cyclic loading. Therefore, it is important to try to develop a film that will indeed show a significant increase in the dynamic fatigue characteristics of a given material. Different deposition parameters have been studied to produce a film that can show more stability in a moist environment, such as the oral cavity.[7,20,22] In vitro studies that replicate clinical conditions are fundamental, especially for the development of new techniques, and should be performed before clinical studies are carried out.

Conclusion

The increase in strength of modified porcelain with a 3% YSZ thin film may be due to surface flaw modification. Although an increase was observed in the strength of machinable porcelain when a YSZ thin film is applied to its surface, further studies are necessary to develop a film that will enhance not only the flexural strength of brittle substrates, but also their long-term performance.

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