Effect of Sandblasting on the Long-Term Performance of Dental Ceramics

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Abstract: A study has been made of the effects of sandblasting on the strength of Y-TZP and alumina ceramic layers joined to polymeric substrates and loaded at the top surfaces by a spherical indenter, in simulation of occlusal contact in ceramic crowns on tooth dentin. The sandblast treatment is applied to the ceramic bottom surface before bonding to the substrate, as in common dental practice. Specimens with polished surfaces are used as a control. Tests are conducted with monotonically increasing (dynamic) and sinusoidal (cyclic) loading on the spherical indenter, up to the point of initiation of a radial fracture at the ceramic bottom surface immediately below the contact. For the polished specimens, data from the dynamic and cyclic tests overlap, consistent with a dominant slow crack growth mode of fatigue. Strengths of sandblasted specimens show significant reductions in both dynamic and cyclic tests, indicative of larger starting flaws. However, the shift is considerably greater in the cyclic data, suggesting some mechanically assisted growth of the sandblast flaws. These results have implications in the context of lifetimes of dental crowns. © 2004 Wiley Periodicals, Inc.* J Biomed Mater Res Part B: Appl Biomater 71B: 381–386, 2004

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INTRODUCTION

In dental clinical practice, essential criteria for selection of crown materials are aesthetics and resistance to fracture and deformation under long-term cyclic conditions. Structural ceramics with high modulus, hardness and strength—most notably alumina and zirconia—are attractive candidates for cores in porcelain–veneer anterior crowns. However, ceramic-based crowns are vulnerable to lifetime-threatening damage at the occlusal and cementation surfaces.[1–5] Radial cracking at the cementation surface is particularly dangerous, because of its capacity to propagate to the margins and thus to split the crown. This mode is associated with flexure of the crown on the relatively compliant dentin from top-surface occlusal loading, placing the crown undersurface in tension. Analogous radial cracking may well operate in polyethylene-supported ceramic acetabular liners in total hip replacements.[4,6,7] Critical loads for the initiation of radial and other cracks tend to diminish steadily with time, from intrusion of moisture into starting flaws at the cementation surface[8–10] as well as from secondary mechanical degradation mechanisms. It follows that the state of the interior ceramic surface[11] can be an important factor governing the ultimate lifetime of ceramic-based prosthetic systems.

Sandblasting of the interior surface is a common practice in all-ceramic crown restorations—the roughened surface enables a strong mechanical bond with resin-based dental cements.[12–23] However, sandblasting introduces its own surface flaws and defects that can compromise the strength of the crown. Countering this is the potential introduction of a compression stress into the damage layer, associated with overlap of local fields around adjacent microcracks with residual openings[24,25] as well as from tetragonal to monoclinic phase transformations.[26] A recent study on the strength of three Y-TZP dental ceramics suggests that the relative importance of these countervailing effects depends on the material microstructure as well as on the severity of the sandblast treatment.[27,28] However, the role of sandblasting on the long-term strength of dental ceramics under the kind of sustained and cyclic loading pertinent to dental function has gone largely ignored.

In the current study, the effect of sandblasting on the long-term strength of alumina and Y-TZP layers on polycarbonate substrates is evaluated experimentally, using Hertzian contact with spherical indenters to simulate the basic features

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of occlusal loading. Although our experiments are here confined to monolithic ceramic layers, the critical radial cracking mode is the same in the ceramic cores of porcelain-veneered bilayers,[29] so the results have a certain generality in the context of dental crowns. A conventional sandblast protocol is applied to the ceramic undersurface prior to bonding that surface to the substrate. We examine two forms of loading: constant stressing rate (dynamic fatigue), and periodic stressing at prescribed frequency (cyclic fatigue). Control tests are conducted on specimens with polished ceramic undersurfaces to establish a baseline for comparison. Comparison of time-to-failure data from these tests enables contributions to fatigue from slow crack growth and mechanical sources to be differentiated.

METHODS AND MATERIALS

A dense fine-grain alumina (AD995, CoorsTek, Golden, CO) and a medical grade 3 mol % yttria-stabilized zirconia (Prozyr Y-TZP, Norton, East Granby, CT) were chosen as the ceramic test materials. These materials are representative of those used in dental and biomechanical applications. Micrographs of the microstructures are shown in Figure 1. Note the relatively fine, homogeneous and equiaxed structure of Y-TZP. Pertinent properties of the materials, measured in earlier studies, are given in Table I.

Alumina and Y-TZP plates with surface dimension 20 × 20 mm were ground and polished (1 μm finish) to a thickness $d \approx$ 1 mm or less. These plates were divided into two groups. The first group was set aside in its as-polished state. The second group was subjected to a sandblast treatment with 50 μm Al_2O_3 particles for 5 s at a standoff distance 10 mm and a compressed air pressure 276 KPa. These specimens took on a matt appearance characteristic of ground ceramic surfaces. Relative amounts of tetragonal (t) and monoclinic (m) phases before and after the sandblasting were measured by X-ray diffractometry (XRD) (D500, Siemens Corp, NY). Small-scale nanoindentations (Nanoindenter XP, MTS Systems Corp., Oakridge, TN) were placed in selected as-polished and sandblasted surfaces, and effective Young's moduli E evaluated from instrumented load–displacement data.[30] For these tests, a relatively high nanoindentation load 1 N was chosen, corresponding to 15–20 μm contact diameter; that is, considerably greater than the grain size. A bonded interface technique[31] was used to obtain section views of the subsurface damage. The ceramic plates were bonded damage-face down onto a clear polycarbonate substrate (Hyzod, AlN Plastics, Norfolk, VA) 12.5 mm thick with a thin (10 μm) layer of epoxy adhesive (Harcos Chemicals, Bellesville, NJ). The interlayer thickness is not crucial because the elastic modulus of the epoxy is similar to that of polycarbonate (Table I).[32]

The bilayers were loaded at their top surfaces using a tungsten carbide (WC) sphere indenter of $r = 3.18$ mm mounted into the crosshead of a mechanical testing machine. Constant stressing rate tests were carried out on a screw-driven machine (Model 5500R, Instron, Corp, Canton, MA), in laboratory atmosphere (~50% humidity). Cyclic tests were carried out on a hydraulic testing machine (Model 8500, Instron, Corp, Canton, MA) at a frequency of 10 Hz. In all tests, the ceramic plate undersurfaces were viewed *in situ* from below the contact using a video camcorder (Canon XL1, Canon, Lake Success, NJ) equipped with a microscope zoom system (Optem, Santa, VA), and the time t_R to radial crack initiation duly recorded. Test durations over the range 0.1 to 10^6 s were accomplished by varying the stressing rates in the dynamic tests and the maximum stresses in the cyclic tests. Corresponding strengths S were calculated from the critical loads P_R using the relation[4,11,33]

$$S = (P_R/Bd^2)\log(E_c/E_s) \quad (1)$$

where E_c and E_s are moduli of the ceramic plate and substrate, d is plate thickness, and $B = 1.35$ is a dimensionless constant. Radial cracks did not cause the system to fail completely, but remained contained within the ceramic layer and precipitated a load drop.

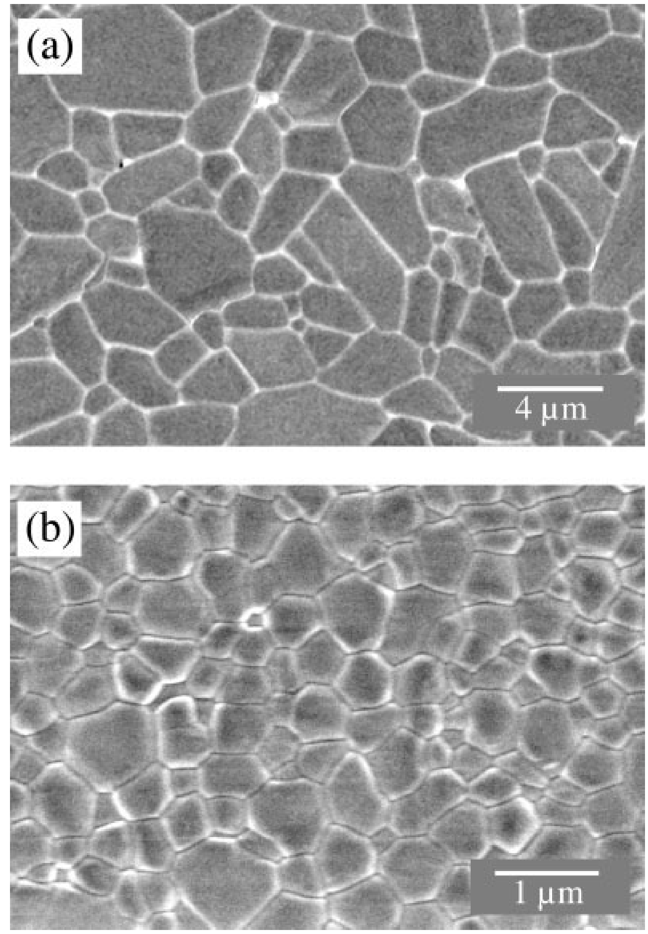

Figure 1. Microstructures of (a) alumina (Al_2O_3) and (b) yttria-stabilized zirconia (Y-TZP). SEM images. Surfaces are thermally etched.

RESULTS

A cross-section SEM view of the sandblast damage in a bonded-interface specimen is shown for Y-TZP in Figure 2.

TABLE I. Properties of Dental Materials[a]

Material	Name	Supplier	Modulus E (GPa)	Hardness H (GPa)	Strength (10 year) S (MPa)	Velocity Exponent N
Core ceramic						
Alumina (dense)	ADS-95-R	CoorsTek	372	19.6	722	26
Zirconia (Y-TZP)	Prozyr	Norton	205	14.0	2325	25
Substrate/Adhesive						
Polycarbonate	Hyzod	AIN plastic	2.3			
Epoxy	RT Cure	Master bond	3.5			
Dentin			16			

[a] Information of product names and suppliers in this article is not to imply endorsement by NIST.

This figure reveals severe sandblasting damage extending ~4 μm below the surface.[27,34] XRD analysis of the Y-TZP before and after sandblasting indicate only a small m-phase content of 4 vol % relative to near-zero on as-polished surfaces (i.e., barely larger than an uncertainty bound of ~3%). Young's modulus determinations from nanoindentation experiments indicate a value $E = 231 \pm 29$ GPa for sandblasted surfaces compared to $E = 270 \pm 3$ GPa for as-polished surfaces (means and standard deviations, 25 indentations). Notwithstanding the relatively large scatter in data from the sandblast tests, indicative of substantial point-to-point variations in surface state, the difference in values suggests a significant increase in microcrack density within the damage layer.[35]

Figure 3 plots maximum stress S versus effective time t_R to radial fracture from dynamic fatigue and cyclic fatigue tests (Eq. 3), for polished Y-TZP and alumina bilayers. This plot, from an earlier study,[9] is reproduced here to establish a comparison base for the sandblast data below. Data points are individual test results, arrows indicate runouts after 10^7 cycles. Solid lines are best fits to the data using the radial fracture relations

$$S^N t_R^c = 2AN^{0.47}, \text{ (cyclic)} \quad (2a)$$

$$S^N t_R^d = A(N+1), \text{ (dynamic)} \quad (2b)$$

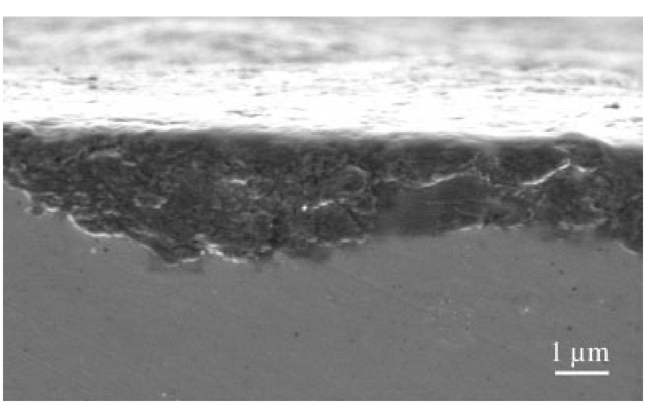

Figure 2. Micrograph showing partial top and cross-section view of sandblast damage (50 μm Al_2O_3 particles) in Y-TZP.

based on a slow crack growth model,[8] where t_R^c and t_R^d are actual fracture times, N is a crack velocity exponent, A is a load-, time-, and thickness-independent quantity. Data from cyclic and dynamic tests can be reduced to a common curve by defining "effective" fracture times

$$t_R = t_R^c, \text{ (cyclic)} \quad (3a)$$

$$t_R = [2N^{0.47}/(N+1)]t_R^d, \text{ (dynamic)} \quad (3b)$$

with Eq. 3b obtained by dividing Eq. 2a into Eq. 2b.[10] The dynamic and cyclic fatigue data overlap within the scatter, enabling determination of common parameters A and N for each material independent of loading condition.

Figure 4 is an analogous plot for sandblasted specimens. The solid lines are carried over from Figure 3 as a zero-damage baseline for comparison. The dynamic fatigue data

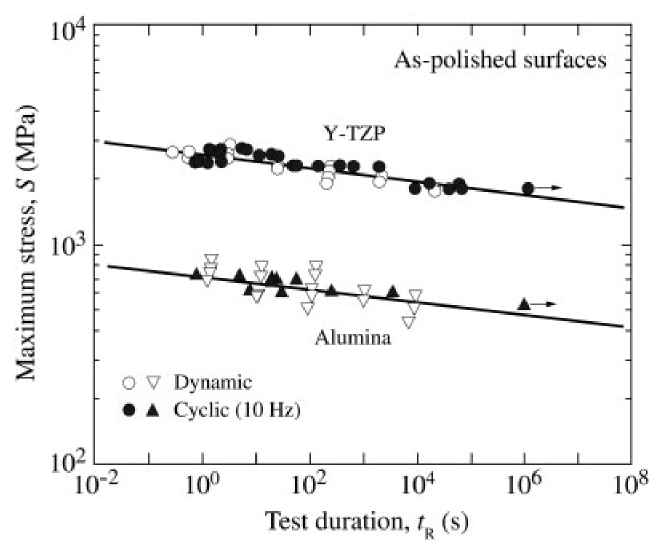

Figure 3. Maximum tensile stress S in ceramic layer versus effective time to radial fracture t_R for as-polished Y-TZP and alumina plates bonded to polycarbonate substrates. Data represent individual tests at constant monotonic stressing rates (unfilled symbols) and in cyclic loading at 10 Hz (filled symbols). Solid lines are data fits in accordance with slow crack growth relations. Arrows indicate runouts. From ref. 10.

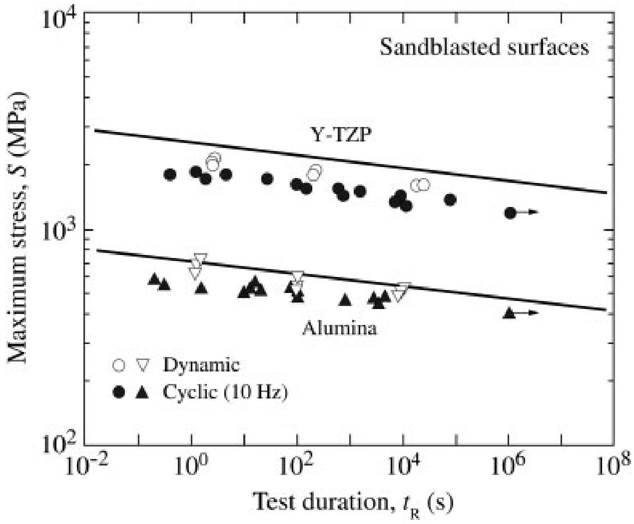

Figure 4. Same as Figure 3, but for sandblasted Y-TZP and alumina plates. Solid lines are fits from Figure 3 for as-polished surfaces.

show minor reductions (<10%) in strength; the cyclic fatigue data show substantially larger reductions (~30% in zirconia, 20% in alumina). The reduced strengths are consistent with the introduction of larger, microcrack flaws in the sandblast treatment (Fig. 2). The fact that the cyclic data fall below the dynamic data suggests some kind of enhanced, mechanically driven flaw extension in repeat loading. Nevertheless, the data sets remain effectively parallel to those for polished surfaces, indicating that the same slow crack mechanism governs the kinetics.

From the standpoint of lifetime of dental crowns in the sandblasted state, it is more practical to consider sustainable load *P* rather than stress *S*, because of a clinical tendency to relate oral conditions to biting *forces*. Such loads can be obtained from Eq. 1 for any prospective ceramic of prescribed thickness on any given substrate material. Due allowance can also be made for the presence of an intervening dental cement of modulus E_i and thickness *h* between crown and dentin, by replacing actual substrate modulus E_s in Eq. 1 with "effective" substrate modulus E_*[36]

$$E_* = E_i(E_s/E_i)^L \quad (4)$$

where $L = L(h/d)$ is an empirical Weibull function

$$L = \exp\{-[\alpha + \beta \log(h/d)]^\gamma\} \quad (5)$$

with $\alpha = 1.18$, $\beta = 0.33$, and $\gamma = 3.13$.[36] As an illustrative example, Figure 5 is plotted by thus converting the cyclic fatigue data for sandblasted alumina and Y-TZP in Figure 4 to equivalent critical load data for ceramic plates of thickness $d = 1.5$ mm on thick dentin substrates with $E_s = 16$ GPa and cement interlayers of thickness $h = 100$ μm and modulus $E_i = 5$ GPa. The plots include 95% confidence bounds to facilitate extrapolations to lifetimes at $t_R = 10$ years. The shaded area in this figure indicates a nominal extreme range of masticatory forces, $P_R = 0–400$ *N*, corresponding to normal oral function.[3] A requisite for guaranteed long lifetime is that the operational loads at 10 years should remain above the shaded region, achieved by both materials but especially by Y-TZP.

DISCUSSION

We have explored the effect of surface condition on the long-term strength of two ceramic crown materials, alumina and Y-TZP, using sphere contact to simulate occlusal loading on crown-like bilayers. Although our tests have been conducted in laboratory atmosphere and not simulated body fluid, the results are clinically relevant because failure occurs from the ceramic undersurface, which does not have direct access to external fluids in crown structures (although internal moisture can be present at the undersurfaces of both epoxy bonded model layer systems and crowns cemented to dentin).[2,8] In polished surfaces, no difference is observed between dynamic (constant load rate) and cyclic (sinusoidal loading) data. These results are consistent with predictions from an earlier study—over a period of some years, slow crack growth degrades the strength by a factor of 2–4.[9,10] Sandblast damage introduced into the ceramic undersurfaces causes further reductions in strength levels, <10% in single-cycle loading but substantially greater, 20–30%, in cyclic loading at 10 Hz. With regard to this last point, 10 Hz is relatively high compared to typical mastication frequencies of ~1 Hz. It has been demonstrated that strength degradation in fatigue tests depends only on total number of cycles,[10] suggesting that the downward shifts in the cyclic data in Figure 4 are likely to overestimate the fatigue effect under clinical conditions. Overall, the present results indicate that the in-

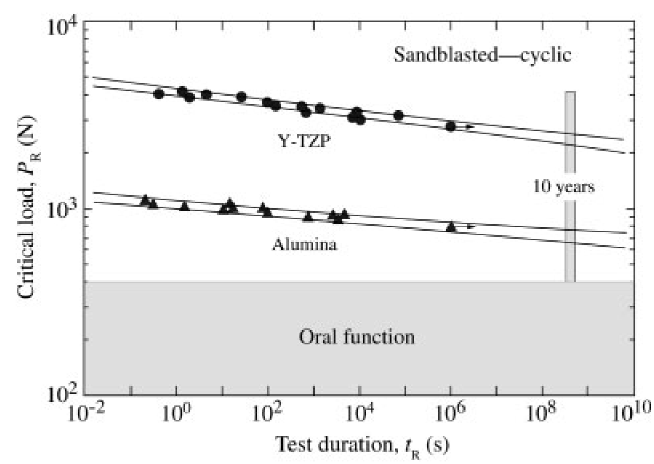

Figure 5. Plots corresponding to cyclic fatigue data in Figure 4 for sandblasted alumina and Y-TZP of thickness 1.5 mm, but in terms of critical loads instead of stress and for dentin-like substrate with intervening dental cement of thickness 100 μm, using Eqs. 1, 4, and 5 to convert the data. Ninety-five percent confidence bounds are used to evaluate uncertainties in sustainable loads at long lifetimes, $t_R = 10$ years. Shaded band indicates nominal oral function range.

troduction of surface flaws outweighs any countervailing strengthening effect from surface compression stresses from sandblasting, either from introduction of microcracks or from phase transformation,[27,28] at least for the materials and under the conditions used in our tests. It can be concluded that surface abrasion treatments can be an important degrading factor in long-term performance of all-ceramic crowns. Any further grinding and abrasion by the dentist during the crown fitting process can only exacerbate the importance of this factor.

This raises questions concerning the underlying nature of the sandblast-induced flaws. Clearly, the flaws must be more severe than those associated with the microstructure. Previous studies indicate that sandblast flaws have the nature of true microcracks.[27,28] Whereas SEM observations (e.g., Fig. 2) may not resolve any such individual microcracks, especially in microstructures with submicrometer grain sizes, modulus reductions inferred from nanoindentation measurements within the damage zones provide supportive evidence for their existence. In sustained loading, slow crack growth extends the microcracks *en route* to radial crack initiation.[8,9] Cyclic loading further exacerbates microcrack extension by some mechanical degradation process, most likely by continual reduction of friction at microcrack walls in repeated shear sliding.[37,38] The degradation nevertheless does not appear to be sufficient to induce the same kind of catastrophic strength losses from crack coalescence that occur from fatigue and fretting in overloaded quasiplastic zones in the immediate contact region.[39–44] The intensity of stress at the lower (cementation) surfaces tends to be somewhat lower than in the contact region at the top surface, "shielding" the subsurface damage to some extent from the immediate occlusal forces.

The data extrapolations in Figure 5 for representative monolithic crowns on dentin suggest that sandblasted alumina, and, especially, Y-TZP crowns, should be able to cope with masticatory forces up to 400 *N* over the long term, despite some strength degradation associated with introduction of larger flaws. In some cases, depending on the specific material microstructure, the sandblast treatment may generate superposed surface compressive stresses, by introducing open microcracks and inducing tetragonal to monoclinic phase transformations.[27,28] Such stresses would only serve to enhance strength still further. For Y-TZP, there is a proviso that the material is not subject to inadvertent hydrothermal degradation, for example, from faulty manufacturing procedure.[45–49] With due care, therefore, Y-TZP would appear to be an ideal candidate for continued development as a dental restoration material.

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