Lifetime of Alumina- and Zirconia Ceramics Used for Crown and Bridge Restorations

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Abstract: The lifetime of a ceramic is dependent on the presence of incidental cracks and their gradual propagation under the conditions of the oral cavity. The objective of this study was to examine the long-term strength of glass-infiltrated alumina- and various zirconia ceramics currently used in CAD/CAM systems to manufacture crown and bridge frameworks. Fracture mechanics were applied to determine characteristic strength (σ_o), Weibull modulus (m), fracture toughness (K_{Ic}), and the subcritical crack growth parameters n and B. Based on these parameters, lifetime diagrams were generated which allowed the evaluation of the long-term behavior. The results showed that in a moist environment, the glass-infiltrated alumina- and some zirconia ceramics have a high susceptibility to subcritical crack growth. Zirconia ceramics with an alumina oxide content of 0.25 wt %, however, exhibited the highest initial and most favorable long-term strength, and should therefore be suitable for crown and bridge restorations. © 2006 Wiley Periodicals, Inc. J Biomed Mater Res Part B: Appl Biomater 80B: 317–321, 2007

Keywords: oxide ceramics; characteristic strength; fracture toughness; subcritical crack growth; lifetime

INTRODUCTION

Because of the brittle properties of ceramic materials, the indication spectrum for dental ceramics was considerably limited in the past. However, a supercritical loading that results in the immediate brittle fracture of a ceramic restoration is rarely observed in vivo, e.g. in cases of trauma or extreme para functions. In contrast, the subcritical stresses are of greater clinical importance. For instance, such stress occurs during cyclic masticatory loading and also when very small manufacturing-related structural flaws are exposed to the corrosive oral environment. This can lead to crack initiation and further propagation.[1] If the external loading continues, the initially subcritical crack growth may reach a critical crack length and causes an unstable spread of cracks ultimately followed by failure of the ceramic restoration.

Various studies have already used fracture mechanics to measure the subcritical crack extension and provide information on the susceptibility of a ceramic material to subcritical crack growth and thus a characterization of its long-term behavior.[2–6] Within the validity of linear elastic fracture mechanics, the lifetime of a ceramic material under a given load can be calculated by various fracture-mechanical parameters, especially with the help of the fracture toughness K_{Ic} and the subcritical crack growth parameters n and B.[7] For a certain ceramic material the crack growth parameters n and B are constant for a given environment.[8]

Zirconia ceramics used in dentistry are characterized by exceptionally high strength properties which are based on the "phase transformation effect," i.e., the tension-induced tetragonal-to-monoclinal phase transformation of metastable zirconia particles.[9] The polycrystalline material zirconia ceramics contains—in contrast to conventional dental ceramics—almost no glass. The glass-infiltrated alumina ceramics of the In-Ceram system (Vita Zahnfabrik, Bad Säckingen, Germany) are an exception. This system has recently been expanded to include a zirconia-reinforced variation. In the industrial production of zirconia ceramics the densely sintered ceramic blanks are compressed in a hot isostatic pressing (HIP) process, in order to improve the ceramic's resistance to microcrack growth and hence the long-term behavior of the material.[10] However, when zirconia ceramics are used in CAD/CAM systems, zirconia blanks are milled both, immediately after cold isostatic pressing as green body sample and after compressed sintering as a hardened (sintered) blank.[11]

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TABLE I. Composition of the Investigated Oxide Ceramics in Weight Percent

	In-Ceram Alumina	In-Ceram Zirconia	Zirconia TZP	Zirconia TZP-A
Crystal phase				
ZrO_2 (%)	–	33	95	94.75
Al_2O_3 (%)	100	51	–	0.25
Y_2O_3 (%)	–	–	5	5
CeO_2 (%)	–	16	–	–
Glass phase	Lanthanide glass	Lanthanide glass	–	–
Nonhipped/hipped	–/–	–/–	–/×	×/×

The objective of this study was to test the long-term strength of two well-known glass-infiltrated alumina ceramics in comparison to various zirconia ceramics currently used in the construction of metal-free crown and bridge frameworks.

MATERIALS AND METHODS

Specimen Preparation

In addition to the glass-infiltrated alumina ceramics In-Ceram alumina and In-Ceram Zirconia (Vita Zahnfabrik, Bad Säckingen, Germany), the zirconia ceramics TZP (Tetragonal Zirconia Polycrystal) and TZP-A with an aluminum oxide component of 0.25 wt % (Metoxit, Thayngen, Switzerland) were tested (Table I). Specimens of Zirconia TZP were examined only in a "hipped" state, and specimens of Zirconia TZP-A in both the "hipped" and "nonhipped" state, meaning with and without having undergone a HIP process. All oxide ceramics belong to the so-called hard-core ceramics, which are used as material for crown and bridge frameworks and must therefore subsequently be veneered with additional veneer ceramics in a dental laboratory.

The In-Ceram specimens were cut from industrially presintered In-Ceram Celay blanks (type AB-28 and ZB-33) into the requisite size using a low-speed saw (Wirtz-Buehler, Düsseldorf, Germany) equipped with a 100-μm-thick diamond blade, and then infiltrated with lanthanide glass. Specimens of Zirconia TZP and TZP-A were delivered by the Metoxit Company in the desired dimensions.

Characteristic Strength and Weibull Modulus

The fracture-mechanical parameters of the ceramic materials were determined according to the German Industrial Standards (DIN) for high-performance ceramics (testing machine: Z-1445; Zwick, Ulm). The 4-point bending test was used to evaluate the fracture strength of the bar specimens according to DIN EN 843–1. Before testing, the tensile stress side of the specimens was successively smoothed with 320-, 600-, 1200-, and 1400-grit SiC wet abrasive papers down to a surface roughness of $R_{\max} < 2$ μm. The polished specimens were 1.5 mm thick, 3.0 mm wide, and 30.0 mm long. Finally, all specimens were annealed and for each type of ceramic material the fracture strength was determined for a total of 30 specimens. The calculation of the characteristic strength σ_0 and the Weibull modulus m was carried out by a self-written computer program. The values for the Weibull modulus were numerically corrected according to the correction table in DIN ENV 843–5.

Fracture Toughness

The critical fracture toughness was determined on notched test specimens with the SEVNB (single edge V-notch beam) method. Before fracture testing, 10 specimens for each type of ceramic material were notched (to simulate an incipient crack) using a 100-μm-thick diamond saw blade. The notches were then reworked using the "razor-blade method" to reduce their width at the bottom.[12] When applying this technique, an average notch root radius of 44.7 μm ($\pm$11.4) was created (Figure 1). The relative notch depth $\alpha = a/h$ for each specimen was between 20 and 30% of the respective specimen height h. The notch depth was exactly measured with a measuring light microscope. All specimens were 3.0 mm thick, 6.0 mm wide, and 30.0 mm long, and tested in their sintered state after firing. The specimens were placed in the 4-point bending test and then the fracture force was mea-

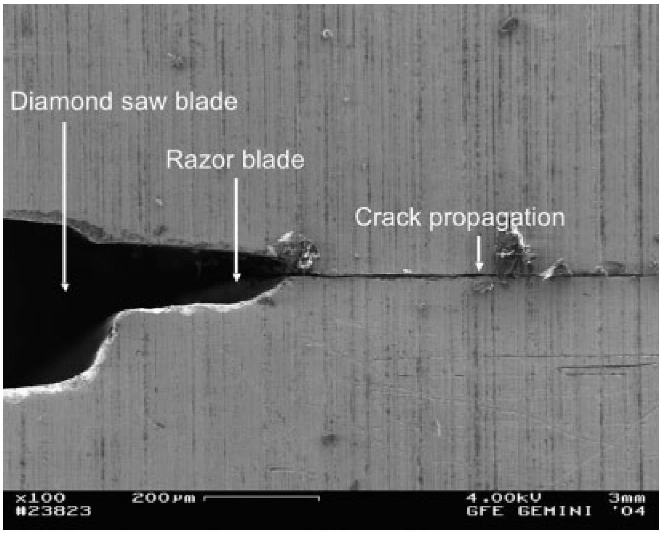

Figure 1. Scanning electron micrographs of a V-notch in a TZP-A specimen with a notch width of 39.9 μm. Notch root radii of the notched specimens used for the fracture toughness test were measured on several high-magnification images.

TABLE II. Summary of the Measured or Calculated Fracture-Mechanical Parameters

	In-Ceram Alumina	In-Ceram Zirconia	Zirconia TZP (hipped)	Zirconia TZP-A (non hipped)	Zirconia TZP-A (hipped)
Characteristic strength σ_0 (MPa)	385 (395–374)	502 (514–491)	937 (952–922)	1185 (1215–1156)	1218 (1252–1187)
Weibull modulus m	11.9 (15.2–9.3)	13.9 (17.7–10.9)	18.4 (23.7–14.9)	12.8 (16.3–10.0)	13.3 (17.1–10.1)
Fracture toughness K_{Ic} (MPa$\sqrt{}$m)	4.0 (± 0.2)	6.2 (± 0.8)	9.4 (± 1.5)	11.1 (± 0.4)	11.5 (± 1.5)
Crack growth parameter n (air/water)	18.7/16.5	23.5/21.9	35.1/16.5	−/20.8	−/38.4
Crack growth parameter B (MPa^{2}s) (air/water)	$4.8 \times 10^4/4.2 \times 10^3$	$5.3 \times 10^4/2.5 \times 10^4$	$2.2 \times 10^5/2.1 \times 10^3$	$-/6.6 \times 10^5$	$-/4.8 \times 10^1$

90% confidence intervals of the characteristic strength values and the weibull modulus values are in parentheses.

sured, which led to a spontaneous crack progression. Fracture toughness was calculated using the relative crack depth α and the specimen dimensions according to DIN design 51109.

Subcritical Crack Growth Parameters

The crack growth parameters n and B were determined using the dynamic bending test according to DIN ENV 843–3, a method that examines the subcritical crack growth of natural cracks.[13] All bending tests were conducted with the 4-point bending test with a total of 90 specimens for each type of ceramic material. The dimensions and the preparation procedure of the specimens were accomplished as described before. For the dynamic bending test, the testing machine was run in "closed-loop" mode, in order to realize constant loading rates, i.e., constant stress-increase velocities. The loading rates selected were 10^{-2}, 10^{-1}, 10^0, 10^1, and 10^2 MPa/s. Prior to the fracture test, a Knoop hardness indentation was placed perpendicular to the specimen axis on the tensile stress side of all specimens in order to obtain defined starting points for the crack growth initiating breakage. All specimens of Zirconia TZP or TZP-A were indented with an applied load of 100 N. This load was selected in order to initiate flaws large enough to be mainly in the tetragonal phase part of the investigated materials.[14] Specimens of In-Ceram Alumina and In-Ceram Zirconia were indented with an applied load of 50 and 70 N, respectively.

The dynamic bending test was conducted under conditions similar to those found in the oral cavity, i.e., in distilled water at 36°C. In addition, the crack propagation parameters n and B were determined in air at room temperature (the humidity content was not controlled) for the materials Zirconia TZP, In-Ceram Alumina and In-Ceram Zirconia.

Lifetime Estimation

On the basis of the calculated fracture-mechanical parameters, lifetime diagrams were generated for each type of ceramics according to the following lifetime equation[7]

$$\ln \ln \frac{1}{1-F} = \frac{m}{n-2} \ln t + \frac{m}{n-2} \ln\left(\frac{\sigma^n}{B}\right) - m \ln \sigma_0 \quad (1)$$

$$B = \frac{2}{AY^2(n-2)} K_{\mathrm{Ic}}^{2-n} \quad (2)$$

with F is the fracture probability; t, the service time; n and B, the subcritical crack growth parameters; A, the mathematical value;[7] Y, the 1.275 (geometry factor); m, the Weibull modulus; K_{Ic}, the fracture toughness; σ, the applied stress; and σ_0, is the characteristic strength.

RESULTS

All measured and calculated fracture-mechanical values are displayed in Table II. The results of the lifetime analysis show that in a moist environment a favorable strength behavior was found for the specimens of Zirconia TZP-A (Figure 2). Especially, Zirconia TZP-A in a "hipped" state revealed the highest initial strength of 1218 MPa and a relatively high n-coefficient of 38. In contrast, the low n-values of the

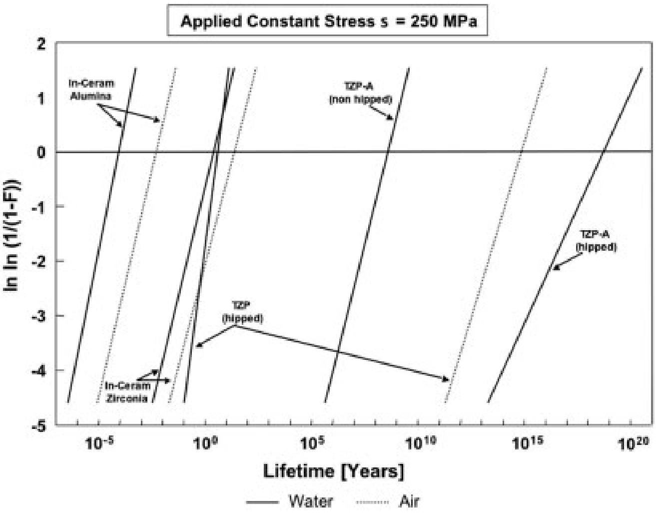

Figure 2. Lifetime diagram of the investigated oxide ceramics shows the long-term fracture probability F in distilled water at 36°C and in air at room temperature after a constant applied stress of $\sigma = 250$ MPa.

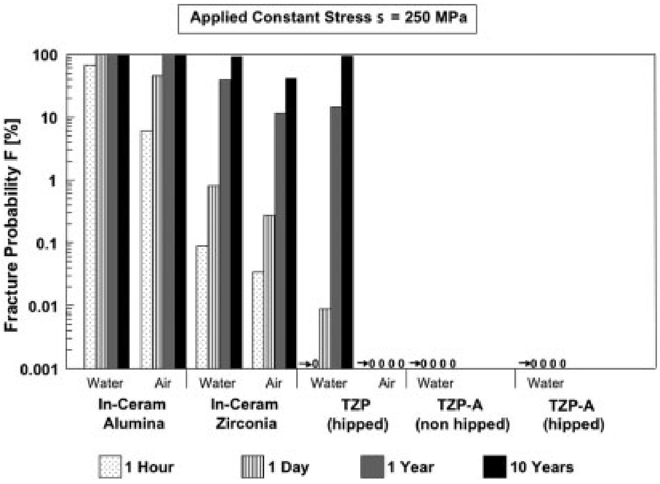

Figure 3. Lifetime diagram of the investigated oxide ceramics shows the long-term fracture probability *F* in distilled water at 36°C and in air at room temperature after a hypothetical constant loading period of 1 h, 1 day, 1 year, and 10 years.

glass-infiltrated alumina ceramics indicated the high susceptibility of In-Ceram Alumina and In-Ceram Zirconia to subcritical crack growth under moist conditions as well as in air.

Comparisons of the fracture probabilities after a period of 1 h, 1 day, 1 year, and 10 years with a constant stress of 250 MPa demonstrate once more the promising mechanical properties of the Zirconia TZP-A specimens (Figure 3). Although a constant loading rate does not reflect very realistically the cyclic mastication process *in vivo*, a comparison of the lifetime predictions is possible. An applied stress of 250 MPa is comparable to the mean value of the maximal occlusal load in relation to one square millimeter chewing surface that can be measured in the posterior tooth region.[15] Similar to the long-term behavior of the glass-infiltrated In-Ceram ceramics, a dramatic loss of strength was observed for Zirconia TZP, but only under moist conditions. Especially, the example of Zirconia TZP showed that a high initial strength does not allow any conclusions about the lifetime of a ceramic, nor does it guarantee a high long-term strength.

DISCUSSION

In all lifetime analyses, the fracture probability of the investigated oxide ceramics was clearly dependent on the duration of loading time (Figure 2). This showed a typical fatigue behavior of ceramic materials. After a hypothetical loading period of 1 year the fracture probability increased to more than 50% for some of the investigated ceramics when a constant stress of 250 MPa was applied (Figure 3). Various studies have already used fracture-mechanical methods to analyze the crack behavior of dental porcelain.[2,16,17] Studies on silicate glasses showed that amorphic vitreous structures, particularly when exposed to the moist conditions of the oral cavity, possess a pronounced tendency to subcritical crack propagation.[18] Thus, for glass- and feldspathic-porcelain only relatively low numerical values for the crack-propagation parameter *n* were found, in some cases much less than 15.[8] These results can help to explain the unfavorable long-term behavior of the vitreous In-Ceram ceramics found in the present study (Figures 2 and 3). Of course, one could assume that the high proportion of aluminum oxide crystals in the ceramic structure of the In-Ceram ceramics and the additional presence of the phase transformation effect in In-Ceram Zirconia impede subcritical crack growth via interaction with the crack front. In terms of long-term strength, these reinforcement mechanisms were not very effective.[19]

The zirconia ceramics TZP and TZP-A exhibited the highest initial strengths as expected.[1] However, it must be pointed out that the average notch radius of 44.7 μm ($\pm$ 11.4) that was created by the razor-blade-method was clearly larger than the particle size of the investigated zirconia ceramics. Regarding a particle size of 0.4 μm ($\pm$0.35) the values of the fracture toughness determined in the present study will probably overestimate the true fracture resistance of the ceramic materials.[12] Furthermore, the influence of HIP was initially not evident in the group of the Zirconia TZP-A test specimens. Clear differences between the ceramics became apparent only upon observation of the long-term behavior. Then, surprisingly, a dramatic loss of strength was registered for "hipped" Zirconia TZP in a moist environment (Figures 2 and 3). Various studies dealing with this phenomenon assume that the tetragonal phase of meta stabilized zirconia is destabilized in a moist environment by hydrolysis reactions between the water and the yttrium oxide, and in time, when the yttrium oxide concentration has dropped below a certain level, it spontaneously transforms into the monoclinal phase.[20,21] In addition, the possibility of direct hydrolysis reactions of water with the zirconium–oxygen bonds has been discussed, which give rise to further weakening of the crystalline structure.[22] As expected, these weakening reactions did not occur in the absence of water, as demonstrated by the examination of the Zirconia TZP specimens in air. Compared to the crack propagation parameters found for the In-Ceram ceramics under air conditions, obviously better long-term behavior was therefore recorded for Zirconia TZP, which, in contrast to the glass-infiltrated alumina ceramics, did not possess a crack-susceptible glass phase (Figures 2 and 3).

However, in comparison to Zirconia TZP, the Zirconia TZP-A specimens exhibited much more stable long-term behavior under moist conditions, although the two ceramics differ only by a very low aluminum-oxide content of 0.25 wt % (Table I). Examinations of zirconia ceramics containing aluminum oxide have shown that added aluminum oxide usually precipitates at the grain boundaries of the zirconia crystals and thus increases the matrix pressure on the metastable crystal phase. This effect is contributed to large suppress transformation reactions into the monoclinal crystal phase.[23] Moreover, the aluminum oxide precipitation on the grain boundaries is thought to prevent water from entering, thus impairing the hydrolysis reactions with yttrium oxide, which commonly occur in moist environments.[20] Both mechanisms ultimately led to a reduction in subcritical crack growth and hence to greatly improved long-term strength of Zirconia TZP-A, especially under moist conditions. A further effect of the added aluminum oxide was an increased initial strength of Zirconia TZP-A. This observation can be explained by crack deflections or crack extensions on the aluminum oxide particles in the zirconia matrix, which lead to an impairment of crack progression and result in a higher strength of the ceramic.[24] Furthermore, the "hipped" Zirconia TZP-A specimens demonstrated somewhat better long-term strength than specimens of "nonhipped" materials (Figure 2). This was apparently a result of further reduction of pore and flaw sizes by exposing the material to final HIP.

In summary, the results of the examinations indicate that Zirconia TZP-A, especially when "hipped", is preferentially suited to the fabrication of all-ceramic crown and bridge frameworks, as it demonstrated by far the highest initial strength and best long-term strength in a moist environment of all the ceramics examined. Further studies must be conducted to determine whether "hipped" Zirconia TZP-A, which must be processed as a densely sintered blank in the fired state, suffers sufficient damage during milling to negate the advantage of greater strength compared to the "nonhipped" material, when the latter is milled as green body sample and thus presumably incurs less damage.

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