J Mater Sci: Mater Med (2007) 18:1219–1224
DOI 10.1007/s10856-007-0131-4

Dynamic fatigue and strength characterization of three ceramic materials

**Erica C. Teixeira · Jeffrey R. Piascik ·
Brian R. Stoner · Jeffrey Y. Thompson**

Received: 24 May 2005 / Accepted: 3 March 2006 / Published online: 3 February 2007

Abstract Fracture strength and fatigue parameters of three ceramic materials submitted to dynamic fatigue were evaluated. A machinable leucite-reinforced dental ceramic, aluminum oxide, and yttria-stabilized zirconia (YSZ) were tested. The inert strength of the materials was determined in air (25 °C) at stressing rates of 70, 250, 400 MPa/s for Porcelain, Alumina and YSZ respectively. The data was analyzed using a two-parameter Weibull distribution. The Weibull modulus (m) and the characteristic of fracture (σ_0) parameters were determined for each material. Specimens were also tested in 3-point bending at different stressing rates in distilled/deionized water at 37 °C (dynamic fatigue) in order to calculate the fatigue parameters n and $\ln B$. The strength for each material was characterized using Strength–Probability–Time (SPT) diagrams for 1 day, 1 year and 10 years. YSZ showed a high-fracture strength σ_0 (1,459 MPa) at a failure probability of 63.2% and high resistance to subcritical crack growth. YSZ and alumina showed better resistance to slow crack growth than porcelain, indicating less susceptibility to strength degradation by stress corrosion. Lifetime predictions after 10 years indicate a reduction of 50%, 36% and 29% in strength for porcelain, alumina and YSZ respectively. YSZ seems to be a very promising material for long-term dental and biomedical applications.

E. C. Teixeira (✉)
Department of Diagnostic Sciences and General Dentistry, University of North Carolina, Chapel Hill, NC 27599, USA
e-mail: teixeire@dentistry.unc.edu

E. C. Teixeira · J. R. Piascik · B. R. Stoner · J. Y. Thompson
Curriculum of Applied and Material Science, University of North Carolina, Chapel Hill, NC 27599, USA

E. C. Teixeira · J. Y. Thompson
School of Dentistry, University of North Carolina, Chapel Hill, NC 27599, USA

J. R. Piascik · B. R. Stoner
RTI International, Center for Materials and Electronic Technologies, Research Triangle Park, Chapel Hill, NC 27709, USA

Introduction

Ceramic materials have been used over the years in dental and biomedical applications, such as hip prostheses and dental crowns. In dentistry, the use of ceramic restorations is desirable due to their wear resistance, biocompatibility, and esthetics [1]. However, some ceramic materials have low to moderate fracture toughness and low endurance limits, limiting their applications [1–3]. New all-ceramic systems, such as leucite-reinforced porcelains and zirconia have been introduced commercially over the last 15 years.

The combination of better manufacturing techniques such as computer-assisted design/computer-assisted manufacture (CAD/CAM) and stronger materials have increased the possibility of using all-ceramic restorations in stress-bearing areas, such as posterior dental applications. However, ceramic materials are brittle in nature and can exhibit delayed fracture [1–4]. It is well known that initial strength can be reduced during a certain application period. Clinical studies of a pressable dental glass-ceramic (Dicor-Caulk Dentsply, Milford, DE), indicated that over longer periods of time failure rates approach 5% per year on molar crowns [5]. The strength of ceramic materials can be influenced by flaws present in the material or induced during fabrication, grinding, and polishing [6–9]. It has been observed in some studies that clinical fracture of all-ceramic dental restorations often initiates along internal surfaces and propagates through the material, leading to a bulk fracture [10, 11].

The effect of a moist environment in conjunction with stress during function encourages crack propagation at lower stress levels than expected after long-term loading, and is referred to as stress corrosion [12, 13]. Environmental effects, such as the influence of water in saliva can modify the energy required to break bonds between atoms in a material, leading to subcritical crack growth (SCG). Subcritical crack growth can initiate at pre-existing flaws, reducing the strength of dental restorations and shortening their lifespan. Fatigue behavior of ceramic materials is often influenced by SCG. This explains why many ceramic materials undergo delayed failure, most likely due to a stress corrosion process involving the stable growth of pre-existing flaws [14]. The SCG behavior can be empirically expressed as:

$$v = \frac{da}{dt} = A\left[\frac{K_I}{K_{IC}}\right]^n \quad (1)$$

where v, a, and t are crack velocity, crack size, and time, respectively. A and n are the material/environment dependent SCG parameters, and K_I and K_{IC} are, respectively, the stress intensity factor and the fracture toughness of the material [15].

Dynamic fatigue testing in which strength is measured as a function of stressing rate has been used to characterize ceramic materials and to indirectly obtain SCG exponents. Knowledge of factors influencing the rate of SCG in a given environment enables the development of relationships between lifetime, applied stress, and failure probability of ceramic materials [4, 14, 15]. In this study, the fatigue and strength parameters of different ceramic materials were obtained through dynamic fatigue and Weibull probabilities. Lifetime predictions diagram were also used to characterize the time-dependent degradation of the tested materials' strength.

Material and methods

Three ceramic materials representative of those used in dental and biomedical applications were chosen for this study. Dense, fine-grained alumina samples in bar form were obtained commercially (Ferro-Ceramic Grinding Inc.). Yttria-stabilized zirconia (Metoxit AG) plates and ProCad blocks (ProCad, Ivoclar, Schaan Liechtenstein) were cut into bars using a low speed saw. All bars, measuring approximately 2 × 2 × 15 mm were polished through 1,200 grit SiC abrasives and had their edges rounded to limit edges failures.

Fracture strength values were obtained from 30 specimens using a 3-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 very high stressing rates in order to avoid subcritical crack propagation and record the inert strength of the material. A constant loading rate of 70 MPa/s for porcelain, 250 MPa/s for alumina and 400 MPa/s for zirconia was applied. The loading rates were chosen based on pilot testing according to the physical and microstructural characteristics of the materials being analyzed in the study. 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:

$$P(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)\right]^m \quad (2)$$

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 [16]. The statistical variability of estimates for the two-characteristic Weibull parameters σ_0 and m were determined by a maximum-likelihood approach. In order to assess material reliability, the two-characteristic Weibull parameters were calculated in addition to their predicted 5% probability of failure, which seems more relevant for biomedical applications.

Dynamic fatigue tests were carried out in distilled deionized water at 37 °C, in order to measure the environment dependent slow crack growth parameter. Porcelain and alumina specimens were tested at four stressing rates of: 0.01, 0.1, 5, 50 MPa/s and 0.06, 0.1, 5, and 45 MPa/s, respectively. YSZ was tested at three different stressing rates of: 0.05, 0.8, and 6 MPa/s. The dependence of strength on stressing rate, caused by SCG is described by:

$$\sigma_f = \left[B(n+1)\sigma_i^{n-2}\dot{\sigma}\right]^{1/n+1} \quad (3)$$

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 with Eq. (3) rewritten as [15]:

$$\ln \sigma_f = \frac{1}{n+1} \ln \dot{\sigma} + \ln \beta \tag{4}$$

where

$$\beta = \frac{1}{n+1} [\ln B + \ln(n+1) + (n-2) \ln \sigma_i] \tag{5}$$

The lifetime t_f is given by:

$$t_f = B\sigma_i^{n-2}\sigma^{-n} \tag{6}$$

The inert strengths were used to obtain the B term in Eq. (5) and to determine lifetime predictions of the different materials. A combination of failure probabilities as a function of strength and time provide the Strength–Probability–Time (SPT) diagrams [13, 17]. Strength values at 1 day, 1 year and 10 years were estimated.

Results

It was observed that most fractures originated from the center region of the specimens in the tensile surface, which was subjected to the highest stress. A scanning electron microscopy (SEM) micrograph of the fractured surface of a porcelain bar is shown in Fig. 1, where a center fracture origin can be observed. The initial flexural strength or inert strength data (measured under dry conditions) of the materials are shown in Fig. 2, where the failure probability is plotted against the strength values by least-squares fit. An inert strength of 113, 265, and 1,080 MPa for porcelain, alumina, and YSZ was respectively determined at a failure probability of 5%.

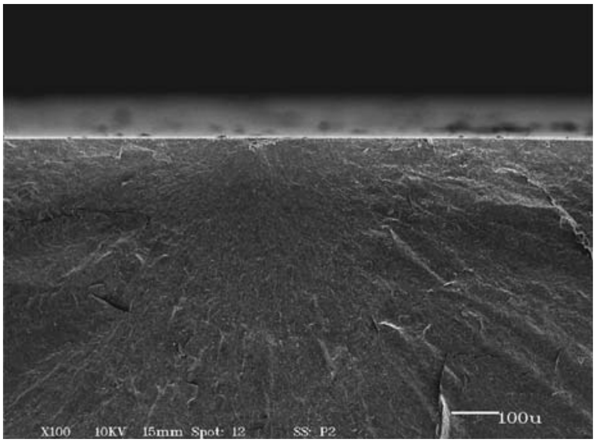

Fig. 1 SEM micrograph of fracture surface of a porcelain bar

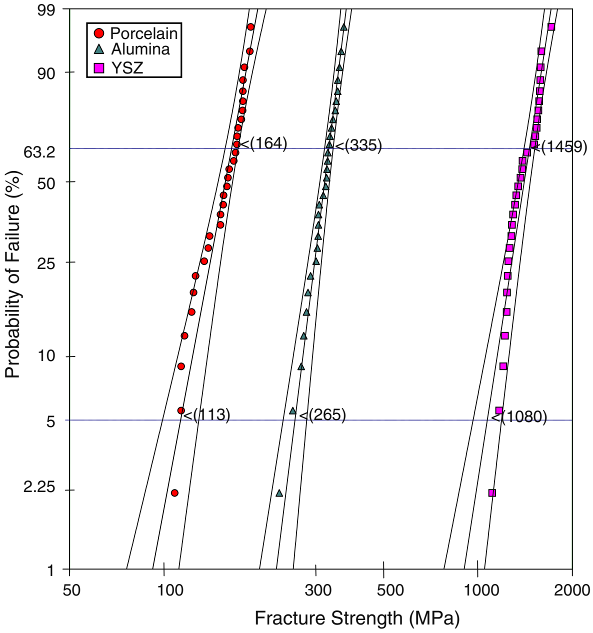

Fig. 2 Weibull probability plot and confidence bounds (95%) for the inert strength of porcelain, alumina and YSZ

The m values observed were in the range expected for ceramic materials 5–15 [3]. Alumina, YSZ, and porcelain showed Weibull modulus of 11.9, 9.6 and 7.9 respectively. The shape parameter (m) describes the relative scattering of the strength data in the asymmetrical distribution.

Dynamic fatigue data (Fig. 3) showed that the SCG parameter estimates were n = 28 and ln B = 3.3 MPa^{2}s for porcelain, n = 43.6 ln B = 8.5 MPa^{2}s for alumina, and n = 56.8 ln B = 12.5 MPa^{2}s for YSZ. n and B are constants, and represent the degree of susceptibility of a material to SCG. Porcelain showed the lowest resistance to stress corrosion (n = 28) and the lowest Weibull modulus (m = 7.9) when compared to alumina

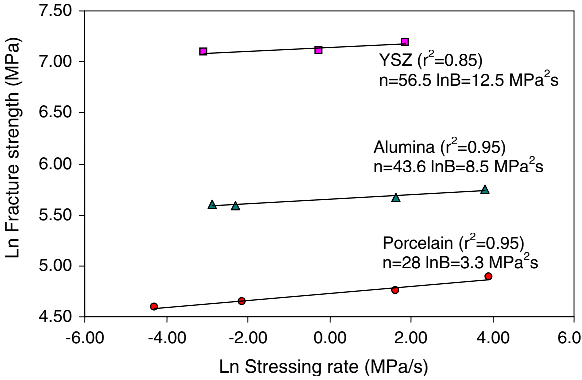

Fig. 3 Dynamic fatigue data and fatigue parameters of the tested materials

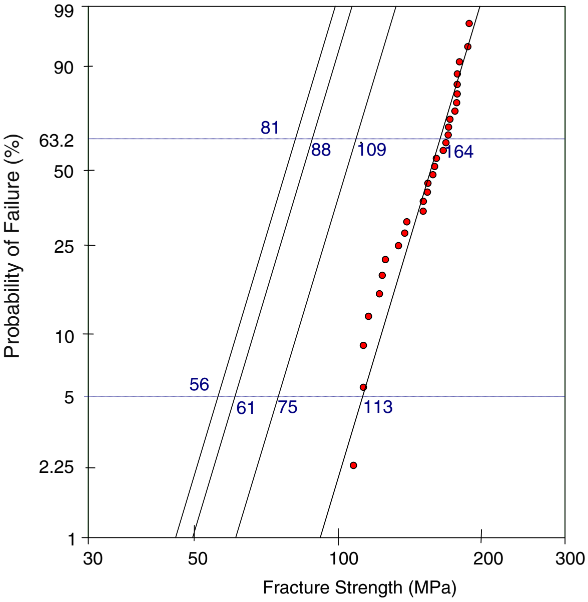

Fig. 4 Lifetime prediction diagram of porcelain based on Weibull probability plot showing strength values at 63.2% and at 5% probability of failure for initial, 1 day, 1 year, and 10 years

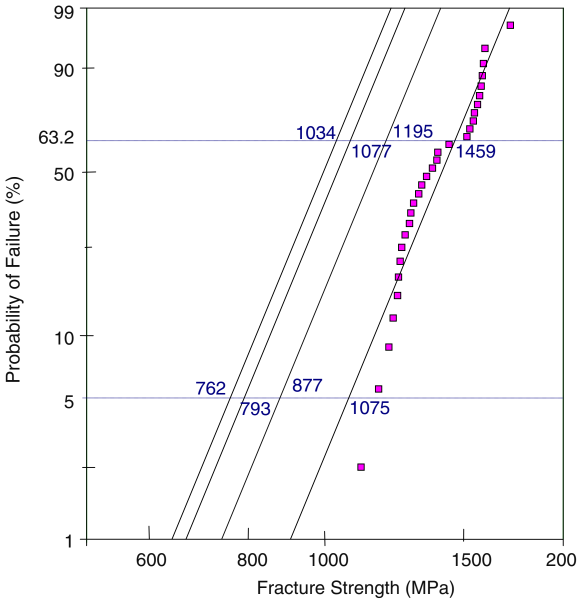

Fig. 6 Lifetime prediction diagram of YSZ based on Weibull probability plot showing strength values at 63.2% and at 5% probability of failure at initial, 1 day, 1 year, and 10 years

and YSZ. One can observe that porcelain displayed relatively strong rate dependence with a low n value, while alumina and zirconia showed much higher values.

SPT diagrams for the three materials are shown in Figs. 4–6, where the characteristic parameters were determined at 63.2% and 5% failure probabilities. Porcelain showed a decrease in strength (P = 63.2%) from 164.1 to 109.3 after 1 day. Lifetime predictions after 10 years indicate a reduction of 50%, 36% and 29% in strength for porcelain, alumina and YSZ respectively. Scanning electron microscopy micrographs of the surface microstructure of porcelain, alumina, and YSZ are shown in Fig. 7. Surface defects such as pores can be observed in the porcelain and alumina specimens.

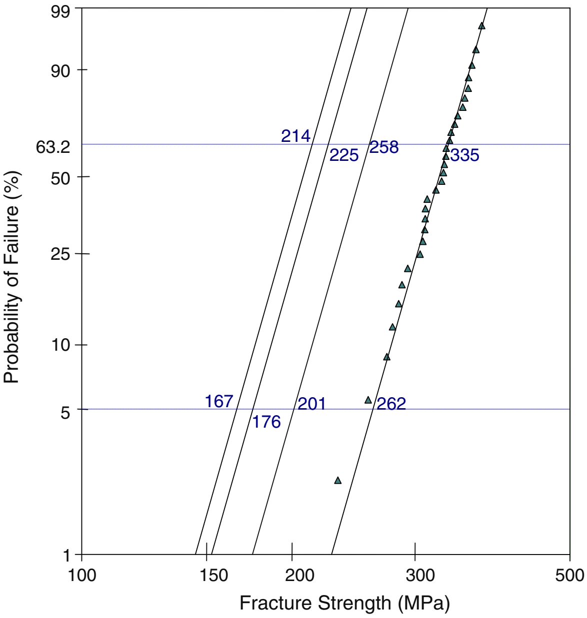

Fig. 5 Lifetime prediction diagram of alumina based on Weibull probability plot showing strength values at 63.2% and at 5% probability of failure at initial, 1 day, 1 year, and 10 years

Discussion

Zirconia-based dental ceramics have been widely studied in the last few years due to their outstanding mechanical properties. It is understood that partially stabilized zirconia undergoes a stress-induced phase transformation from a tetragonal crystal configuration to a monoclinic configuration ($t \rightarrow m$), which seems to be responsible for its high strength. When a crack attempts to propagate through the material the transformation occurs, creating local compressive stress or a compliance region of fine microcracks at the tip of the propagating crack. Either phenomenon results in a reduction in crack tip energy, limiting further crack propagation. This is often referred to as transformation toughening [9].

In vitro reports have observed the effect of different surface modifications, such as sand blasting, grinding, and polishing on the strength of zirconia [8, 9]. However, few studies have reported the SCG parameters when YSZ is subjected to an aqueous environment. Zirconia is known to be sensitive to humidity,

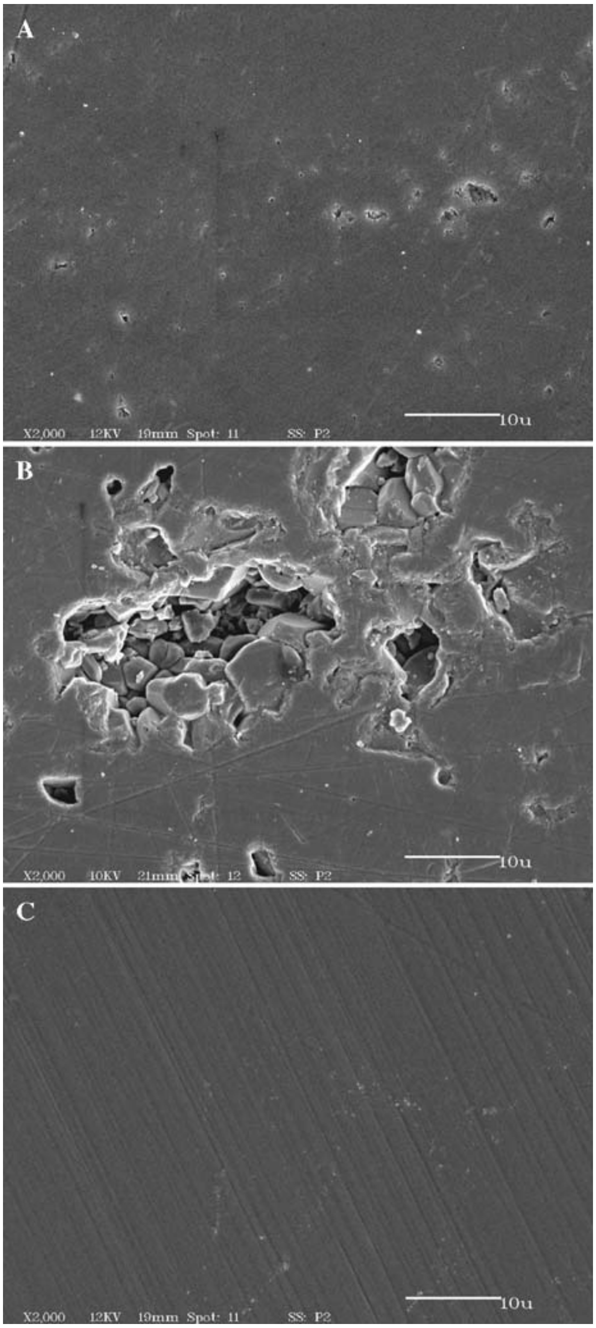

Fig. 7 Surface of characteristic porcelain (**A**), alumina (**B**), and YSZ (**C**) specimen

which is a particularly important issue when prosthetic and orthopedic applications are considered. YSZ may undergo stress corrosion during long term implantation in the human body [18–20]. Qiao et al. [20] observed that ZrO_2 ceramics, with varying Y_2O_3 concentrations presented very different SCG values. The 3% Y_2O_3–ZrO_2 showed the highest *n* exponent (46.5) value after dynamic fatigue testing, indicating low stress corrosion susceptibility.

Thompson and Rekow [21] showed that cyclic loading propagates cracks in a similar manner to dynamic fatigue, and observed that alumina and zirconia were susceptible to SCG, lowering their strength by 20%–50% over a 10-year estimation period [21]. In this study, YSZ showed the smallest decrease in strength when submitted to dynamic fatigue. Its characteristic strength parameter at 10 years prediction was 1,034 MPa, which is still very high when compared to alumina and porcelain. Ceramic dental restorative materials should withstand occlusal loads of 100–200 N, however masticatory forces can reach 900 N [22]. The YSZ tested in this study was fabricated by hot isostatic pressing, in which the material is completely sintered producing a strong and dense material. Although zirconia has high strength and seems to suffer less stress corrosion than most ceramic materials its esthetic properties are more limited than porcelain.

The development of a strong porcelain material would broaden its application. ProCad is a ceramic material based on leucite-reinforced glass-ceramics, with increased strength when compared to traditional feldspathic porcelains [2, 23–25]. Although the results indicate a better resistance to SCG when compared to the data for feldspathic porcelain in the literature, it still is lower than alumina and YSZ. Morena et al. [2] reported a *n* value of 14.6 for feldspathic porcelain. The dependence of the strength on stress rate indicates that SCG is taking place prior to failure [14]. Cyclic fatigue has also been shown to cause strength degradation of porcelain, but it seems to act independently from static or dynamic fatigue effects, making it important to perform both types of tests [25]. In vitro cyclic loading of ProCad crowns showed that the type of cement used can limit the effect of strength degradation [24].

Strength depends on the size of microscopic cracks and pores [8]. The alumina specimens in this study contained a noticeable porosity, probably due to incomplete sintering of the material (Fig. 7), which affected its measured strength values. However, the Weibull modulus for alumina was higher than for YSZ and porcelain. Strength data provide insight into the stresses a material will support for a given flaw distribution and failure mode, but cannot predict structural failure alone [3, 4]. An indirect measure of the flaw distributions in ceramic structures is the Weibull modulus. A smaller m value indicates a broader distribution of flaws and a greater scatter in the distribution of strength values as a function of failure probability [3, 16, 26].

A better controlled manufacturing process for the porcelain blocks used to produce the bars would be expected to produce a high m value. However, the values for all three materials tested were in the expected range for ceramic materials [3]. Further studies characterizing the strength of modified ceramic materials to prevent stress corrosion and enhance long-term performance should be developed and performed.

Acknowledgments Research supported by NIH/NIDCR Grant DE13511-04. Materials contributed by Ivoclar Vivadent.

References

1. K. J. ANUSAVICE, *JADA* **124** (1993) 72
2. R. MORENA, G. M. BEAUDREAU, P. E. LOCKWOOD, A. L. EVANS and C. W. FAIRHURST, *J. Dent. Res.* **65** (1986) 993
3. J. TINSCHERT, D. ZWEZ, R. MARX and K. J. ANUSAVICE, *J. Dent.* **28** (2000) 529
4. J. R. KELLY, *Dent. Mater.* **11** (1995) 103
5. G. SJOGREN and R. LANTTO et al *J. Prosthet. Dent.* **81** (1999) 277
6. Y. ZHANG, B. R. LAWN, E. D. REKOW and V. P. THOMPSON, *J. Biomed. Mater. Res.* **71B** (2004) 381
7. K. PALLIS, J. A. GRIGGS, R. D. WOODY, G. E. GUILLEN and A. W. MILLER *J. Prosthet. Dent.* **91** (2004) 561
8. T. KOSMAC, C. OBLAK, P. JEVNIKAR, N. FUNDUK and L. MARION, *Dent. Mater.* **15** (1999) 426
9. M. GUAZZATO, M. ALBAKRY, L. QUACH and M. V. SWAIN, *Dent. Mater.* **21** (2005) 454
10. J. Y. THOMPSON, K. J. ANUSAVICE, A. NAMAN and H. F. MORRIS, *J. Dent. Res.* **73** (1994) 1824
11. J. R. KELLY, S. D. CAMPBELL and H. K. BOWEN, *J. Prosthet. Dent.* **62** (1989) 536
12. J. A. SALEM and M. G. JENKINS, *ASTM STP* **1409** (2002) 213
13. R. G. CHADWICK, *J. Dent.* **22** (1994) 364
14. D. S. TUCKER, *J. Am. Ceram. Soc.* **73** (1990) 2528
15. J. E. RITTER, in ‘‘Fracture Mechanics of Ceramics’’ (Plenum Publishing Co., NY, 1978), p 661
16. W. WEIBULL, *J. Appl. Mech.* **18** (1951) 293
17. U. LOHBAUER, A. PETSCHELT and P. GREIL, *J. Biomed. Mater. Res.* **63** (2002) 780
18. A. H. DE AZA, J. CHEVALIER, G. FANTOZZI, M. SCHECHL and R. TORRECILLAS, *Biomaterials* **23** (2002) 937
19. Y. ZHANG and B. R. LAWN, *J. Biomed. Mater. Res.* **69B** (2004) 2019
20. G. J. QIAO, W. HONGJIE and J. ZHIHAO, *Int. J. Fatigue* **24** (2002) 499
21. V. P. THOMPSON and E. D. REKOW, *J. Appl. Oral. Sci.* **12** (2004) 26
22. J. A. HANKINSON, E. G. CAPPETTA, *Int. J. Periodontics Restorative Dent.* **14** (1994) 138
23. H. Y. CHEN, R. HICKEL, J. C. SETCOS and K. H. KUNZELMANN *J. Prosthet. Dent.* **82** (1999) 468
24. A. ATTIA and M. KERN, *J. Prosthet. Dent.* **92** (2004) 551
25. S. N. WHITE, Z. C. LI, Z. YU and V. KIPNIS, *Dent. Mater.* **13** (1997) 103
26. J. E. RITTER, T. H. SERVICE and C. GUILLEMET, *Glass Technol.* **26** (1985) 273