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## On the interfacial fracture of porcelain/zirconia and graded zirconia dental structures

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### ABSTRACT

Porcelain fused to zirconia (PFZ) restorations are widely used in prosthetic dentistry. However, their susceptibility to fracture remains a practical problem. The failure of PFZ prostheses often involves crack initiation and growth in the porcelain, which may be followed by fracture along the porcelain/zirconia (P/Z) interface. In this work, we characterized the process of fracture in two PFZ systems, as well as a newly developed graded glass-zirconia structure with emphases placed on resistance to interfacial cracking. Thin porcelain layers were fused onto Y-TZP plates with or without the presence of a glass binder. The specimens were loaded in a four-point-bending fixture with the thin porcelain veneer in tension, simulating the lower portion of the connectors and marginal areas of a fixed dental prosthesis (FDP) during occlusal loading. The evolution of damage was observed by a video camera. The fracture was characterized by unstable growth of cracks perpendicular to the P/Z interface (channel cracks) in the porcelain layer, which was followed by stable cracking along the P/Z interface. The interfacial fracture energy  $G_C$  was determined by a finite-element analysis taking into account stress-shielding effects due to the presence of adjacent channel cracks. The resulting  $G_C$  was considerably less than commonly reported values for similar systems. Fracture in the graded Y-TZP samples occurred via a single channel crack at a much greater stress than for PFZ. No delamination between the residual glass layer and graded zirconia occurred in any of the tests. Combined with its enhanced resistance to edge chipping and good esthetic quality, graded Y-TZP emerges as a viable material concept for dental restorations.

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### 1. Introduction

Posterior dental prostheses are commonly made of porcelain veneer fused to metal (PFM) or zirconia (PFZ) frameworks. Porcelain has good esthetic qualities, but is susceptible to premature fracture. Although the reported incidence of porcelain fracture is higher in PFZ relative to PFM [1–6], the two systems share similar damage forms. Fracture may occur from edge chipping (“cohesive” failure) [1,6–9] or interfacial fracture (“delamination”) [10–12]. The higher porcelain chipping and delamination rates observed in PFZ are due to the development of deleterious tensile residual stresses [13–17] and the low interfacial fracture energy  $G_C$  of PFZ relative to PFM [18,19]. The residual thermal stress occurs during heat treatment due to the mismatch in the coefficient of thermal

expansion (CTE) between veneer and core as well as the low thermal diffusivities characterizing most ceramics. It should be noted that any crown or FPD failure other than edge chipping must involve a crack reaching the interface between veneer and core. Indeed, this has been demonstrated in several *in vitro* studies on fatigue of PFZ restorations [20–23]. Hence, assessing and improving interfacial strength is an important aspect of material design.

There are several approaches to prevent delamination in PFZs. One such approach is to use a glass bonding layer to improve the adhesion of porcelain to zirconia. Unfortunately, this can only marginally, if at all, increase the resistance to delamination. This is because when cracks in the porcelain veneer reach the veneer–core interface, they tend to graze along the weak interface or deflect into the porcelain veneer rather than penetrate the stiffer and tougher zirconia core. A much more effective strategy is to alter the crack growth route so that it penetrates into the zirconia framework via proper engineering of the interfacial microstructure

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[24,25]. This way, the superior strength and toughness of zirconia could effectively hold off the crack, averting porcelain delamination altogether. Using a glass–ceramic infiltration technique, a functionally graded glass–zirconia material has been developed [25]. Previously, we have examined the fracture resistance of this material, including subsurface flexural fracture [26–28] and edge chipping resistance [29,30]. The results showed that this material retains the proven strength benefit of Y-TZP, yet has a superior esthetic quality [24]. Here, we examine the fracture resistance of its interface.

Considerable work has been devoted to assessing the interfacial adhesive strength of porcelain-veneered zirconia restorations. This is generally done by subjecting the material to some form of external force and examining the ensuing fracture pattern [31–34]. While useful for routine screening, this approach yields no direct information on the interfacial fracture energy  $G_c$ . A simple means for evaluating  $G_c$  for dental restorations is the four-point-bending bilayer configuration. Several studies have used this method to determine the interfacial energy of PFM with various metal copings [19,35] or different porcelain-veneered zirconia systems [18]. However, the fracture process in such tests can be complex; even simple configurations, such as thin-film bilayers under tension, are known to fail in an intricate process involving multiple channel cracks in the hard film and delamination between film and substrate (e.g. [36,37]). In addition, the multiple channel cracks may greatly alter the fracture resistance as well as the calculations of interfacial fracture energy [38,39].

In this study, we elucidate the fracture process and fracture resistance of some PFZ systems as well as a newly developed graded Y-TZP coated by a glass layer (hereinafter, we refer to this material as graded Y-TZP) due to tensile loading. Special emphasis is placed on resistance to interfacial fracture. Porcelain/zirconia bilayers and graded Y-TZP samples are loaded in the four-point-bending fixture shown in Fig. 1. Two PFZ systems are considered: porcelain fused directly onto zirconia, and porcelain fused to zirconia in the presence of a glass binder. The latter is motivated by works showing that binders may greatly enhance interfacial fracture resistance [35]. The fracture process is observed from the specimen edge using a video camera equipped with a telephoto

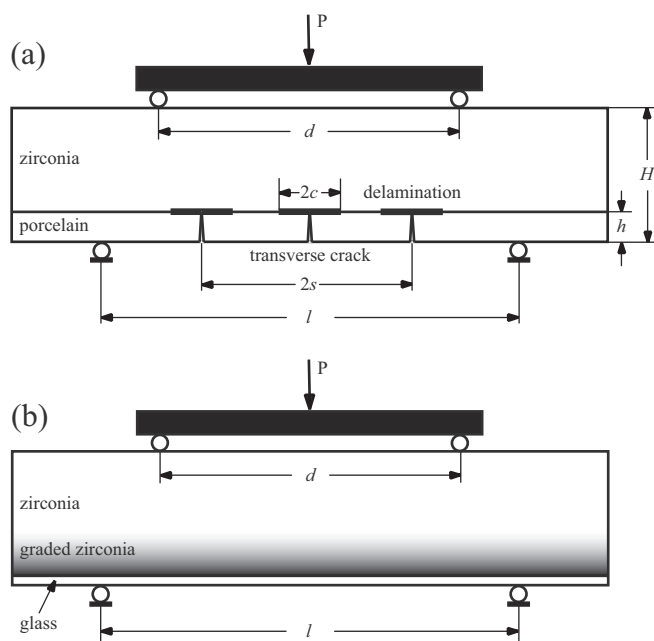
lens. The tests are supplemented by a 2-D finite-element analysis (FEA) for calculating interfacial fracture energy, taking into account the evolving fracture morphology. Section 2 describes the materials and methods used, while Section 3 presents the test results. The latter is discussed in Section 4 in relation to dental restorations.

## 2. Materials and methods

The materials used were PFZ and graded Y-TZP. For PFZ, two configurations were studied: porcelain fused directly onto zirconia (P group) and porcelain fused to zirconia in the presence of a thin glass binder (Y group). Prior to veneering, the zirconia (Tosoh Y-TZP,  $CTE = 10.5 \times 10^{-6} K^{-1}$ ) surface was sandblasted with  $50 \mu m$   $Al_2O_3$  particles for 5 s at a standoff distance of 10 mm and a compressed air pressure of 2 bar. The P group was produced by laying porcelain powder (Heracera Zirconia, leucite-reinforced porcelain,  $CTE = 10.5 \times 10^{-6} K^{-1}$ , Heraeus Kulzer GmbH, Hanau, Germany) onto zirconia and heating to  $870^\circ C$  with a dwell time of 1 min. For the Y group, an adhesive paste  $\sim 0.1$  mm thick (Heracera ZR-Adhesive paste, Heraeus Kulzer GmbH,  $CTE = 10.5 \times 10^{-6} K^{-1}$ ) was first applied to the zirconia veneering surface and fired at  $1050^\circ C$  for 20 min to help wet the zirconia surface. Upon cooling to room temperature, the same porcelain used for the P group was applied and fired. Examination of the interface region in the Y specimens with a high-resolution optical microscope revealed no visible sign of interlaminar glass adhesive, indicating good interdiffusion between glass and porcelain. Hence, both P and Y groups could be characterized by a single veneer layer. The graded Y-TZP was prepared as described earlier [25,40]. Briefly, an in-house prepared glass with composition similar to dental porcelain in the form of powder slurry was first applied on pre-sintered Y-TZP ( $1350^\circ C$  for 1 h). Glass infiltration and densification occurred in a single process at  $1450^\circ C$  for 2 h, resulting in a structure consisting of a  $15\text{--}40 \mu m$  thick residual glass layer followed by a  $120 \mu m$  thick graded layer and finally a bulk Y-TZP. Fig. 2a shows a micrograph of a graded Y-TZP sample broken by bending. A uniform residual glass layer without large voids or flaws can be observed. The glass layer is attached to the Y-TZP core through a graded glass–zirconia layer where the content of intergranular glass is gradually diminishing.

All specimens were fabricated in the desired form: length  $L = 30$  mm, total thickness  $H = 2.7$  mm and specimen width  $b = 2.5$  mm (see Fig. 1). At least seven specimens were prepared for each group. The porcelain thickness  $h$  for both PFZ groups is  $0.4$  mm. The fracture tests were conducted using the four-point-bending test [41]. Fig. 1 shows the PFZ (a) and graded Y-TZP (b) specimens in their loading fixture. The distances between lower and upper supporting pins,  $l$  and  $d$ , were 20 and 10 mm, respectively. Prior to testing, the tensile surfaces of the PFZ samples were polished down to a  $1 \mu m$  diamond suspension finish and then indented at their center points by a Vickers indenter at a load of 5 N to introduce an artificial flaw for crack initiation (crack length  $\sim 45 \mu m$ ). In the case of graded Y-TZP, some specimens ( $n = 10$ ) were tested as fabricated, while in others ( $n = 7$ ) the tensile surface was indented with a Vickers tool at 10 N load (crack length  $\sim 70 \mu m$ ). The specimens were loaded in a standard universal testing machine at a rate of  $0.1 \text{ mm min}^{-1}$ . One specimen side face, polished to mirror surface quality, was observed by a video camera (Canon EOS-7D) equipped with a high-power zoom lens (Optem, Inc.).

Referring to Fig. 1, the load  $P$  produces a bending moment  $M = P(l - d)/4$  in the beam portion bounded by the upper pins. The tensile stress at the lower surface of the PFZ bilayer,  $\sigma_p$ , where fracture initiates, is easily found from beam theory as [38]:



**Fig. 1.** The porcelain/Y-TZP bilayer specimen (a) and graded Y-TZP specimen (b) used, shown in the four-point-bending fixture. Illustration (a) contains the channel and delamination cracks used in the FEM model.

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