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Apparent Interfacial Fracture Toughness of Resin/Ceramic Systems

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Abstract

We suggest that the apparent interfacial fracture toughness (K_A) may be estimated by fracture mechanics and fractography. This study tested the hypothesis that the K_A of the adhesion zone of resin/ceramic systems is affected by the ceramic microstructure. Lithia disilicate-based (Empress2-E2) and leucite-based (Empress-E1) ceramics were surface-treated with hydrofluoric acid (HF) and/or silane (S), followed by an adhesive resin. Microtensile test specimens (n = 30; area of 1 ± 0.01 mm^2) were indented (9.8 N) at the interface and loaded to failure in tension. We used tensile strength (σ) and the critical crack size (c) to calculate K_A ($K_A = Y\sigma c^{1/2}$) (Y = 1.65). ANOVA and Weibull analyses were used for statistical analyses. Mean K_A ($MPa \cdot m^{1/2}$) values were: (E1HF) 0.26 ± 0.06; (E1S) 0.23 ± 0.06; (E1HFS) 0.30 ± 0.06; (E2HF) 0.31 ± 0.06; (E2S) 0.13 ± 0.05; and (E2HFS) 0.41 ± 0.07. All fractures originated from indentation sites. Estimation of interfacial toughness was feasible by fracture mechanics and fractography. The K_A for the systems tested was affected by the ceramic microstructure and surface treatment.

Keywords

structural reliability; fracture; fractography; fracture toughness; Weibull modulus

Introduction

Bond strength tests have been used to predict the clinical performance of repaired fractured ceramic restorations and resin-bonded ceramic restorations, even though most of these tests exhibit a wide variability in fracture patterns and bond strength. The commonly used shear bond test often produces fracture at a distance from the resin-ceramic adhesion zone, and may lead to erroneous conclusions about bond quality. Such failures of the substrate prevent the measurement of interfacial bond strength and limit further improvements in bonding systems (Della Bona and van Noort, 1995; Versluis *et al.*, 1997; Chadwick *et al.*, 1998; Della Bona *et al.*, 2003b).

The microtensile test was developed to eliminate non-uniform stress distribution at the adhesive interface and to minimize the influence of interfacial defects (Sano *et al.*, 1994). This test has been used to measure the bond strength of composite to dental tissues (Sano *et al.*, 1994; Pashley *et al.*, 1995; Schreiner *et al.*, 1998; Shono *et al.*, 1999; Carvalho *et al.*, 2003) and to ceramics (Della Bona *et al.*, 2000; El Zohairy *et al.*, 2003).

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The strength values determined from the microtensile test are considered a reliable indicator of the composite-ceramic bond quality, since fractures occur within the adhesion zone. In addition, the microtensile test produces variable fracture-surface morphology and fracture origins for the same adhesive interfaces within the adhesion zone. Therefore, a careful interpretation of the failure mode is required to prevent inappropriate conclusions about the utility of the microtensile test and the adhesion zone phenomena. Fracture-surface characterization combined with analyses of fracture mechanics parameters is of great importance to our ability to understand and predict bonded interface reliability (Chen and Mecholsky, 1993; Della Bona *et al.*, 2003b).

One method for assessing bond resistance to fracture is an estimation of the apparent interfacial fracture toughness (K_A) of the adhesion zone by the promotion of crack initiation within the bonded interface. The initial critical flaw can be identified by fractography, and K_A can be assessed according to fracture mechanics principles. K_A is expected to be independent of crack size, whereas bond strength is dependent on crack size (Chen and Mecholsky, 1993). Thus, K_A reflects the ability of a material to resist unstable crack propagation at the interface (Mecholsky and Barker, 1984; Tam and Pillar, 1993).

The objective of this study was to use fracture mechanics and fractography to determine the K_A of the adhesion zone of resin/ceramic systems, testing the hypothesis that K_A is affected by ceramic microstructure and ceramic surface treatments.

Materials & Methods

A hot-pressed leucite-based ceramic (E1; IPS Empress, Ivoclar-Vivadent, Schaan, Liechtenstein) and a hot-pressed lithia disilicate-based ceramic (E2; IPS Empress2, Ivoclar-Vivadent, Schaan, Liechtenstein) were selected for the study (Della Bona *et al.*, 2004b). Fifteen ceramic blocks each of the E1 and E2 ceramics were fabricated and polished through 1 μm alumina abrasive (Mark V Laboratory, East Granby, CT, USA). All ceramic blocks were ultrasonically cleaned in distilled water for 10 min after being polished.

The methods for investigating the microstructure, composition, mechanical and physical properties, and the HF-treated ceramic topography of E1 and E2 ceramics have been fully described in previous studies (Della Bona *et al.*, 2000, 2003a,b, 2004a,b).

The ceramic blocks were randomly divided into 3 groups for each ceramic (n = 5) and treated as follows:

E1HF:	9.5% hydrofluoric acid (HF) (Ultradent Products Inc., South Jordan, UT, USA) applied for 1 min on E1 ceramic, rinsed for 30 s, and dried with oil-free air.
E1S:	silane coating (S) (Ultradent Products Inc., South Jordan, UT, USA) applied to E1, allowed to evaporate for 5 min, and air-dried.
E1HFS:	HF applied for 1 min, rinsed for 30 s, air-dried, followed by application of S.
E2HF:	HF applied as for E1HF on E2 ceramic.
E2S:	S coating applied as for E1S on E2 ceramic.
E2HFS:	HF and S applied as for E1HFS on E2 ceramic.

Adhesive (Scotchbond Multi-Purpose Plus, 3M Dental Products, St. Paul, MN, USA) and 2-mm-thick layers of a resin composite (Z100, 3M Dental Products, St. Paul, MN, USA) were applied to the ceramic-treated surfaces and light-cured for 10 s and 40 s, respectively, with the use of a Coltene Coltolux-4 unit (Coltene/Whaledent Inc., Mahwah, NJ, USA; light intensity = 430 mW/cm^2).

The ceramic-adhesive-composite sets were cut by means of a slow-speed diamond wheel saw (model 650, South Bay Technology Inc., San Clemente, CA, USA), which produced bar

specimens with a mean bonding area of 1 ± 0.01 mm^2 (Della Bona *et al.*, 2000). The bar specimens were examined for flaws by optical microscopy (microscope model SCW30L, Fisher Scientific, Bangkok, Thailand). The exclusion criteria included the presence of any obvious flaw or specimen debonding before testing. Thirty specimens *per* group were selected at random and stored in air at 23°C and 50% humidity for 7 days before indentation.

We performed a preliminary study to determine the indentation load (P). A microhardness tester (Model MO Tukon Microhardness Tester, Wilson Instruments Inc., Binghamton, NY, USA) with a Vickers diamond indenter was used to indent the center of the ceramic/resin interface of additional specimens. Indentation loads ranged from 4.9 N to 29.4 N (dwell time, 20 s). An indentation load of 9.8 N was selected based on a constant value of $P/c_o^{3/2}$ plotted against P, where c_o is the dimension of the radial/median crack (Anstis *et al.*, 1981). In addition, some interfaces (E1S and E2S) consistently debonded during the indentation procedure, when loads greater than 9.8 N were used.

After indentation, the crack was allowed to grow and stabilize for 24 h in air at 23°C and 50% humidity before being tested. Each specimen was loaded to failure in tension (Fig. 1) at a crosshead speed of 0.5 mm/min, in an Instron testing machine (Model 1125, Instron Corp., Canton, MA, USA) (Della Bona *et al.*, 2000). The bonded area of each specimen was measured immediately after testing (Digimatic caliper, Mitutoyo Co., Kawasaki, Japan) and used for the calculation of bond strength. We performed a linear regression analysis to determine if the size of cross-sectional area affected the calculated bond strength.

Tensile bond strength (σ) and K_A data were analyzed statistically by one-way ANOVA and Tukey's multiple range test (α = 0.05). We also performed Weibull analysis to evaluate the structural integrity of the adhesion zone (Della Bona *et al.*, 2003b).

We prepared fracture surfaces for SEM examination to determine the mode of failure, which was confirmed by x-ray elemental maps (EDAX). We used quantitative fracture surface analysis (fractography) to determine the critical crack size (c) and to calculate K_A ($K_A = Y\sigma c^{1/2}$), where Y is a geometry factor (Y = 1.65) and $c = (ab)^{½}$, 'a' is the semi-minor axis (Fig. 2B), and 'b' is the semi-major axis of the crack. The mode of failure was determined according to a previous study (Della Bona *et al.*, 2003b).

Results

The elastic modulus (E), hardness (H), and other relevant properties of the materials used in this study were reported previously (Table 1).

The mean bonded area of the specimens was 1.00 ± 0.01 mm^2. Linear regression analysis showed that tensile bond strength was statistically independent of the size of the bonded area. The load and location of indentations were adequate to produce controlled critical defects (Fig. 1).

Ceramics treated with S only showed the lowest *m* value for both E1 and E2 ceramics. The E1HFS group revealed the greatest *m* value of all tested groups. For each surface treatment, the mean σ value was greater for E2 than for E1, except for the groups treated with S only that showed the lowest mean σ value and the greatest coefficient of variation (Table 2). In addition, some specimens treated with S only debonded during the preparation procedure. Therefore, they met the exclusion criteria and were excluded from the random selection process.

The mean K_A value of E2HFS was significantly greater than that for all the other groups ($p \leq 0.0001$). There was no difference in the ranking of the mean σ and K_A values, which varied

with the ceramic microstructure and surface treatment. Both σ and K_A mean values increased when HF and S were used (Table 2).

All fractures originated from the indentation semicircular crack (Fig. 2) that reached the ceramic-adhesive interface (E1-AR in Fig. 1A and E2-AR in Fig. 1B). Most of the bonded E2 ceramic specimens showed 2 microcracks, one at each adhesive interface. The purely adhesive failure (mode 1) and failure from an internal flaw (mode 3) were not found in this study. We observed only failures that were initiated at the ceramic-adhesive interface and propagated through the adhesive (mode 4), and subsequently either reached the adhesive-composite interface (mode 5) or returned to the ceramic-adhesive interface (mode 2). Modes 4 and 5 were the predominant (60%) types of failure. Failure mode 2 was revealed only when S was applied (APPENDIX) (Della Bona *et al.*, 2003b).

Discussion

Evaluation of K_C or K_A requires knowledge of the E/H ratio (Anstis *et al.*, 1981). K_C is determined to an accuracy of 30 to 40% for any well-behaved material. In this context, uncertainties in the value of E/H are relatively unimportant; indeed, this ratio varies between 10 and 50 for most ceramics. Replacement of the 'calibration' constant $\eta(E/H)^{1/8}$ by an averaged quantity $\eta = 0.59\ [(E/H)^{1/8}] = 0.88$ would add no more than 10% to the error in K_C evaluation for a material whose elastic/plastic parameters are unknown (Chantikul *et al.*, 1981). In this study, the calibration constants for all materials were in agreement with the above values: E1 = 0.83; E2 = 0.83; AR = 0.86; and RC = 0.85.

Previous investigators have discussed the generation and limitations of the indentation technique (Hagan and Swain, 1978; Kruzic and Ritchie, 2003), and a brief discussion is included in the APPENDIX.

To test the integrity of bonded interfaces, one can subject a bonded assembly to a variety of loading conditions to control the crack path along the interface or within the interfacial region. Analyses of bond tests have revealed several problems associated with most common test arrangements, suggesting a lack of reliability of such measurements in assessing the adhesive behavior of bonded dental materials (Anusavice *et al.*, 1980; van Noort *et al.*, 1989; Della Bona and van Noort, 1995; DeHoff *et al.*, 1995; Versluis *et al.*, 1997; Sudsangiam and van Noort, 1999).

The non-trimming method we used in this study to obtain specimens for the microtensile test produces less stress in the adhesion zone (Pashley *et al.*, 1999). Since no specimen finishing is necessary, this method also avoids areas of stress concentration produced by polishing materials that differ in hardness and particle size (Della Bona *et al.*, 2000, 2003b).

Effective etching of the ceramic surface is considered an essential step for the clinical success of indirect ceramic-bonded restorations and direct-repaired ceramic prostheses. Structural and surface analyses of etched ceramics have showed that different etching patterns are created according to the ceramic microstructure and composition (Della Bona *et al.*, 2003b), and the concentration, application time, and type of etchant (al Edris *et al.*, 1990; Kupiec *et al.*, 1996; Chen *et al.*, 1998; Della Bona and van Noort, 1998; Jardel *et al.*, 1999; Della Bona *et al.*, 2000).

The results of this study show that there is a synergistic effect of HF and S on the σ and K_A for the systems studied. Yet, HF-treated ceramics produced greater mean σ and K_A values than did S-treated ceramics. Since the silica(Si)-silane(S)-methyl methacrylate (MMA) combination produces the chemical bond between silica-based ceramics and resins, the lowest mean σ and K_A values of E2S may be explained by the reduced amount of silica in E2, compared

with E1 (Della Bona *et al.*, 2004b). Therefore, σ and K_A are affected by the ceramic microstructure and ceramic surface treatments, confirming the study hypothesis.

The evaluation of the structural integrity of the adhesion zone by Weibull analysis revealed the highest Weibull modulus for E1HFS specimens, which is in agreement with the results of previous research (Della Bona *et al.*, 2000). The strength obtained by the microtensile test can be a reliable indicator of the composite-ceramic bond quality, if all fractures occurred within the adhesion zone. In addition, the microtensile test produced variable fracture surface morphology for the same adhesive interfaces within the adhesion zone, which included failure modes 2, 4, and 5.

Compared with optical microscopy observations, a thorough SEM examination of the fracture surfaces and confirmation of composition through the use of x-ray elemental map analysis will ensure a more consistent and complete description of the fracture process and the modes of failure.

An appropriate way to assess the interfacial bond is to analyze the energy *per* unit crack surface area, G_I, that is required for a crack to advance in the bond plane. Toughness is related to the critical strain energy release rate (G_{IC}) and is a measure of the resistance of the bond to fracture, since G_{IC} represents the relative energy required to create new surfaces.

A positive relationship was found between the mean values of σ and K_A, which followed the ranking for both ceramics (E1 and E2), *i.e.*, HF+S-treated > HF-treated > S-treated ceramic surfaces.

The observation of microcracks at both adhesive interfaces for the bonded E2 ceramic specimens may be explained by the greater difference in E and H between the resin components of the adhesion zone and the E2 or E1 ceramic components. The E and H differences between the adhesive and both the composite and the ceramics used in this study result in different residual stresses at the interfaces. In both systems, the strongest interface bond occurred at the adhesive-composite interface. Thus, we expect all failures to occur at the weaker adhesive-ceramic interface, agreeing with previous results from non-indented fractured resin-ceramic systems (Della Bona *et al.*, 2000, 2003b). Therefore, the results suggest that the interfacial crack size, and consequently the K_A value, is associated with the E/H ratio of the substrates.

Since all fractures occurring within the adhesion zone originated from the Vickers indentation, this study suggests that the microtensile test may be preferable to conventional shear or flexural tests as an indicator of composite-ceramic bond quality. A thorough SEM examination of the fracture surfaces following the principles of fractography and confirmation of surface composition through the use of x-ray elemental map analysis produce a more consistent and complete description of the fracture process and the modes of failure. These analyses avoid simplistic interpretations, such as the ‘mixed mode of failure’, that often follows ‘adhesive and/or cohesive’ observations. Thus, the quality of the bond should not be assessed based on bond strength data alone. Yet, estimation of interfacial toughness is possible with fracture mechanics and appropriate fractographic analyses that should reduce the risk for data misinterpretation and provide important information leading to predictions of clinical performance limits, which is the ultimate test of any adhesive system. Future studies should focus on G_{IC} and apply different methods to test the interfacial fracture toughness, validating this procedure.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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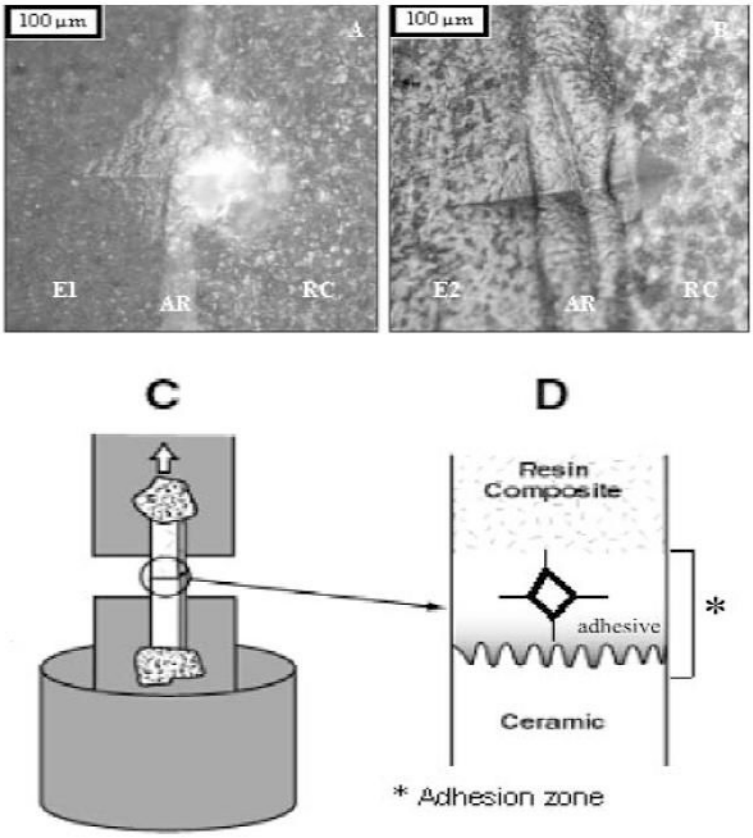

Figure 1.

Optical micrographs of Vickers indentation at the interface of **(A)** HF-treated E1 ceramic bonded to the adhesive resin (AR) and resin composite (RC), and **(B)** HF-treated E2 ceramic bonded to AR-RC. The adhesive resin (AR) layer varied (23-82 µm) in thickness (200x). **(C)** Schematic illustration of a bar specimen fixed to the flat 'grips' of the universal testing machine and loaded to failure in tension; **(D)** close-up view of the indented adhesion zone.

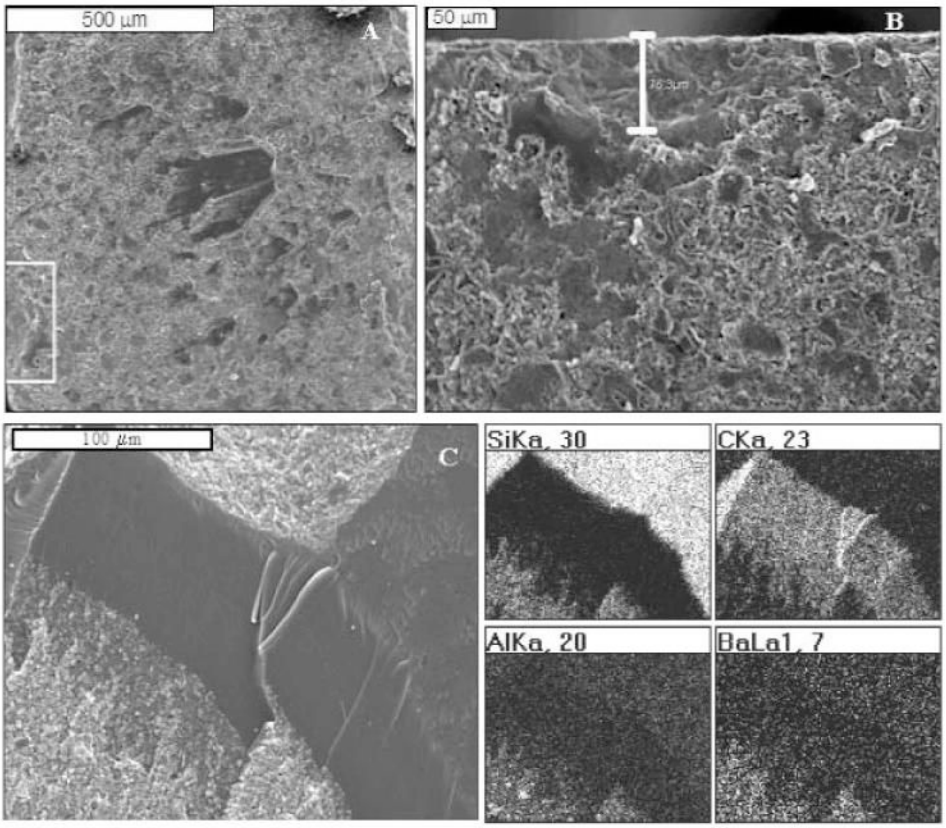

Figure 2.
Representative SEM micrographs of fracture surfaces. **(A)** Semi-circular flaw produced by indentation is the crack origin (white box); the adhesive resin island in the middle of fracture surface has some fracture markings and represents a failure mode 2 (x80). **(B)** Enlargement of white box area of (A) showing the size of the crack semi-minor axis (a = 76.3 µm) (x300). **(C)** SEM image and x-ray elemental maps (lower right) of fracture surface representing fracture mode 5. The label at the top of x-ray maps indicates the elements and their intensity.

Table 1

Mean Values of Density (ρ), Elastic Modulus (E), Poisson's Ratio (υ), Vickers Hardness (H), Four-point Flexural Strength Tested in 37°C Distilled Water, and Fracture Toughness by Fractography and Fracture Mechanics of Empress Ceramics (E1 and E2), the Adhesive and the Resin Composite (Z100)

Material Properties	E1*	E2*	Z100*	Adhesive
Density (ρ)	2.47 g/cm^3	2.51 g/cm^3	2.08 g/cm^3	
Elastic modulus (E)	86 GPa	96 GPa	21 GPa	5.9 GPa
Poisson's ratio (υ)	0.27	0.26	0.25	
Vickers hardness (H)	5.9 GPa	6.3 GPa	1.2 GPa	0.3 GPa
Four-point flexural strength (in water) (σ)	85 MPa	215 MPa	55 MPa[1]	
Fracture toughness (K_{IC})	1.3 MPa•m^{½}	3.4 MPa•m^{½}		

* Data from Della Bona *et al.*, 2000, 2003a,b, 2004a,b.

[1] Three-point flexural strength, tested in a dry environment.

Table 2

Mean Tensile Bond Strength (σ) Values and Standard Deviation (SD), Characteristic Strength (σ_o), Weibull Modulus (*m*), Mean Critical Flaw Size (c) and SD, Modes of Failure, K_A and SD, and Tukey's Test Subsets (α = 0.05) for E1- and E2-treated Ceramics Bonded to Resin

Groups	σ ± SD† (MPa)	σ_o (MPa)	*m*	c ± SD (μm)	Failure mode (%)*	K_A ± SD† (MPa•$m^{½}$)
E1HF	24.0 ± 5.0^{bc}	25.9	4.9	77.0 ± 21.0	4(60); 5(40)	0.26 ± 0.06^{bc}
E1S	21.4 ± 7.6^{c}	24.1	2.6	78.9 ± 20.7	5(60); 4(30); 2(10)	0.23 ± 0.06^{c}
E1HFS	26.4 ± 5.0^{b}	27.3	6.8	82.8 ± 20.5	4(60); 5(30); 2(10)	0.30 ± 0.06^{b}
E2HF	28.1 ± 5.4^{ab}	29.9	5.2	82.5 ± 18.3	4(60); 5(40)	0.31 ± 0.06^{b}
E2S	11.4 ± 5.7^{d}	13.5	1.3	112.7 ± 50	4(60); 2(40)	0.13 ± 0.05^{d}
E2HFS	31.9 ± 8.6^{a}	35.7	5.0	121.5 ± 42	5(60); 4(30); 2(10)	0.41 ± 0.07^{a}

† Within each column, groups with same superscript are not statistically different (P = 0.05).

* Modes of failure are based on Della Bona *et al.*, 2003b.