Journal of Applied Oral Science
J Appl Oral Sci *2005; 13(2): 101-9*
www.fob.usp.br/revista or www.scielo.br/jaos

CHARACTERIZING CERAMICS AND THE INTERFACIAL ADHESION TO RESIN: II- THE RELATIONSHIP OF SURFACE TREATMENT, BOND STRENGTH, INTERFACIAL TOUGHNESS AND FRACTOGRAPHY

CARACTERIZAÇÃO DE CERÂMICAS E ADESÃO À RESINA: II- RELAÇÃO ENTRE TRATAMENTO DE SUPERFÍCIE, RESISTÊNCIA ADESIVA, TENACIDADE DE FRATURA DA INTERFACE E FRACTOGRAFIA

Alvaro DELLA BONA

University of Passo Fundo, Dental School, Passo Fundo, Brazil.

Corresponding address: Prof. Dr. Alvaro Della Bona - Dental School, University of Passo Fundo - Campus I - BR 285 - POBox 611 Passo Fundo, RS 99001-970 - Brazil - Phones: 55-54-581-0657; 55-54-311-5142 - e-mail: dbona@upf.br

Received: February 25, 2005 - Modification: June 20, 2005 - Accepted: June 22, 2005

ABSTRACT

The clinical success of resin bonding procedures for indirect ceramic restorations and ceramic repairs depends on the quality and durability of the bond between the ceramic and the resin. The quality of this bond will depend upon the bonding mechanisms that are controlled in part by the surface treatment that promote micromechanical and/or chemical bonding to the substrate. The objective of this review is to correlate interfacial toughness (K_A) with fracture surface morphological parameters of the dental ceramic-resin systems as a function of ceramic surface treatment. This analysis is designed to identify mechanisms that promote adhesion of these ceramic-resin systems and an appropriate bond test method to yield relevant adhesion performance data.
Uniterms: Ceramics, Resins, Bond strength, Interfacial toughness, Fracture surface.

RESUMO

O sucesso clínico de procedimentos adesivos para restaurações cerâmicas e reparo destas restaurações depende da qualidade e durabilidade da adesão entre a cerâmica e a resina. A qualidade desta adesão dependerá dos mecanismos adesivos que são controlados em parte pelo tratamento de superfície que promove uma união micro-mecânica e/ou química com o substrato. O objetivo desta revisão é correlacionar a tenacidade de fratura aparente da interface adesiva (K_A) com os parâmetros morfológicos da superfície de fratura de sistemas cerâmica-resina em função do tratamento da superfície cerâmica. Esta análise é desenvolvida para identificar os mecanismos que promovem a adesão nestes sistemas cerâmica-resina e uma metodologia apropriada para testar a resistência adesiva produzindo resultados relevantes da performa adesiva.
Unitermos: Cerâmicas; Resinas; Força de adesão; Tenacidade; Superfície de fratura.

INTRODUCTION

The discovery that most dental ceramics could be acid etched to create a micromechanical bond to resin has led to the development of acid-etched and bonded ceramic restorations [39]. This concept was extended to include the repair of fractured dental ceramic restorations in the mouth. Fracture of ceramic restorations leads to increased cost, discomfort, time and labor when a replacement is required [5]. The repair of a fractured ceramic restoration is a challenging clinical situation and there is little documentation on the clinical performance of the repaired restoration. Yet, repair is a more cost effective option, provided the final result is clinically acceptable [30].

Materials and procedures used either to repair fractured ceramic restorations with a resin composite or to bond indirect ceramic restorations using a resin cement are based on the results of bond strength tests that exhibit wide variability in test data and resulting fracture surface characteristics [11,16,21,22,24,26,27,50,55].

In the search for a method that produces a uniform stress distribution across the interface, investigators have evaluated similar adhesive systems under different bond test configurations [22,47,67]. These studies suggest that a tensile bond strength test may be more appropriate to evaluate the bond strength of adhesive interfaces because of more uniform interfacial stresses. However, tensile tests require careful alignment of the specimens to minimize the risk of flexure [24].

The physical contribution to the adhesion process is dependent on the surface topography of the substrate and can be characterized by its surface energy [29,59]. Dynamic contact angle (DCA) analysis has been used to evaluate the surface energy of treated ceramic surfaces and their work of adhesion (W_A) to resin. In principle, the work of adhesion can be related to the apparent interface toughness [29].

Fracture mechanics allows quantification of the relationships between material properties such as toughness, stress level, the presence of crack-producing flaws, and crack propagation mechanisms. Another way to assess the integrity of the bond is to estimate the apparent interfacial fracture toughness of the adhesion zone by promoting crack initiation within this zone. The apparent fracture toughness value (K_A) reflects the ability of a material to resist unstable crack propagation [28,52,71].

The objective of this second part of the review is to correlate apparent interfacial toughness (K_A) with fracture surface morphological parameters of the dental ceramic-resin systems as a function of ceramic surface treatment. This analysis is designed to identify mechanisms that promote adhesion of these ceramic-resin systems and an appropriate bond test method to yield relevant adhesion performance data.

Effect of surface treatment on the contact angle and work of adhesion

The clinical success of either a repaired ceramic restoration or a resin cemented ceramic restoration will depend on the quality and durability of the bond between the ceramic and the resin. The quality of this bond will depend upon the bonding mechanisms that are controlled in part by the specific surface treatment used to promote micromechanical and/or chemical retention with the substrate [24]. Structural and surface analyses of etched ceramics have shown that different etching patterns are created according to the ceramic microstructure and composition, and to the concentration, application time and type of etchant [2,17,18,23-25,40,48]. Surface defects, often located in the glassy matrix, and phase boundaries of heterogeneous ceramic materials are preferably etched by the acids. Alteration of the surface topography by etching will result in changes in the surface area and on the wetting behavior of the porcelain [25,29,59]. This may also change the ceramic surface energy and its adhesive potential to resin [29,41]. Differences in ceramic composition will also produce unique topographic changes after etching procedures [25]. Thus, the ceramic microstructure, composition, and morphologic patterns after surface conditioning should yield potentially useful information on the clinical success of the bonding procedures for indirect ceramic restorations and ceramic repairs [25,30].

It has been reported that the surface changes produced by fluorine content etchants such as hydrofluoric acid (HF), ammonium bifluoride (ABF) and acidulated phosphate fluoride (APF) represent unique patterns [25]. HF etching produces a very aggressive effect on the surface of most acid-sensitive ceramics, where porosities are scattered uniformly throughout the ceramic surface. This pattern is more evident for leucite-based ceramics than for either single-phase or high-content alumina ceramics (Figure 1) [1,2,17,18,23-25,36,74].

ABF etching produces mostly linear defect patterns that were primarily formed because of the etchant attack on existing surface cracks, leucite-induced cracks, and phase boundaries (Figure 2). This etching pattern is also observed after using HF for reduced amount of time and/or in lower concentration, as for SEM preparation for microstructural observations, suggesting that ABF acts as a low power HF etchant [23,25].

The APF etchant seems to build up surface deposits preferentially on the leucite crystal phase (Figure 3) [22-25]. These studies demonstrated that treating the ceramic surface with APF alone produces an insufficient, inconsistent, micromechanical retentive surface and, as a consequence, the lowest bond strengths of resin-based composite to ceramics. Treating the ceramic surface with HF produced a substantial, consistent, surface roughness on acid-sensitive ceramics, mainly because of its action on defects and phase boundaries [23,24]. These results have a positive correlation with the results from contact angle measurements between the ceramic and resin, reported by Della Bona, et al. [29] 2004.

The etched ceramic topography can be explained by the chemical reactivity of the crystals of single-phase materials that depends on crystallographic orientation. In polycrystalline materials, etching characteristics vary among crystal types. Atoms along the crystal boundaries are more chemically active and dissolve at a greater rate than those within the crystals, resulting in the formation of small grooves or linear defects after etching (Figures 1-3) [14,25,30].

Therefore, it has been suggested that (1) differences in ceramic microstructure and ceramic composition are controlling factors in the development of micromechanical retention produced by etching, and (2) the etching mechanism is different for all etchants with HF producing the most prominent etching pattern on most acid-sensitive ceramics [25,30].

Some clinicians also use coarse diamond and/or oral gritblaster (airborne-particle abrasion systems) as the first step for repairing fracture ceramic restorations. It has been shown that other than the scalloped surface created by the rotary instrument, both preparations have a similar topography. In addition, these methods tend to create more stress and sharp cracks onto the ceramic surface, which are

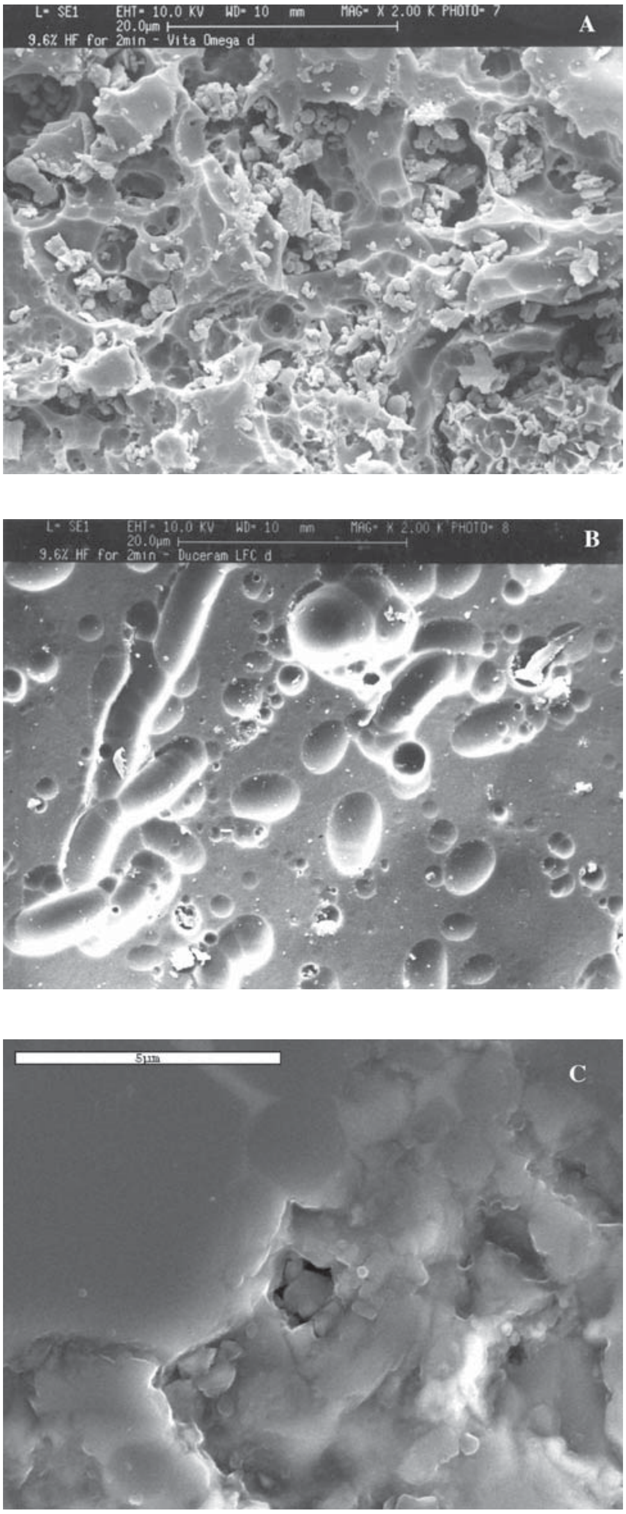

FIGURE 1- Photomicrographs of HF-etched ceramics. (A) Vita Omega dentine ceramic (Vita Zahnfabrik, Bad Sackingen, Germany), a leucite based feldspathic ceramic. (B) Duceram-LFC ceramic (Ducera, Rosbach, Germany), a single-phase low-fusing glass. (C) In-Ceram Zirconia ceramic (Vita Zahnfabrik, Bad Sackingen, Germany), a glass-infiltrated zirconia reinforced alumina-based ceramic. HF etching produces a very aggressive effect on the surface of most acid-sensitive ceramics (A). The acid effect is reduced for either single-phase (B) or high crystalline content ceramics (C). From Della Bona and Anusavice[25], 2002.

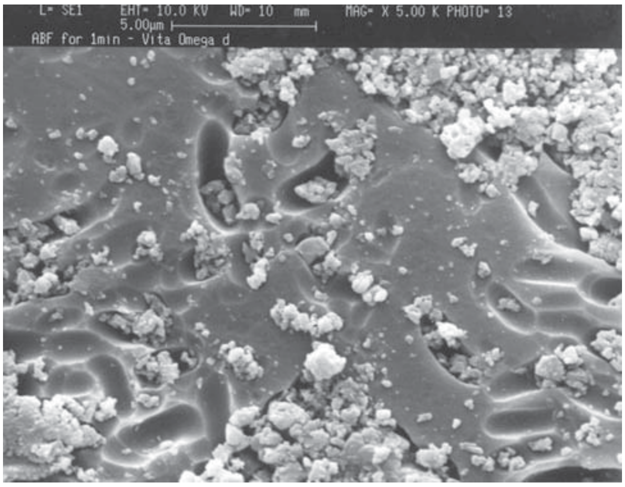

FIGURE 2- Photomicrographs of ABF-etched Vita Omega dentine ceramic (Vita Zahnfabrik, Bad Sackingen, Germany), a leucite based feldspathic ceramic. This acid produces mostly linear defects (grooves) by attacking existing surface cracks, leucite-induced cracks, and phase boundaries. From Della Bona and Anusavice[25], 2002.

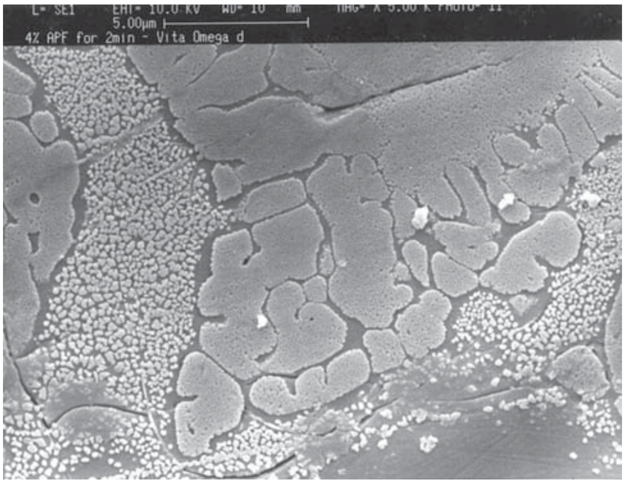

FIGURE 3- Photomicrographs of APF-etched Vita Omega dentine ceramic (Vita Zahnfabrik, Bad Sackingen, Germany), a leucite based feldspathic ceramic. This acid seems to build up surface deposits preferentially on the leucite crystal phase. From Della Bona and Anusavice[25], 2002.

readily attacked by acids and may weaken the substrate [23].

Ceramics with high crystalline content (aluminum and/ or zirconium oxides), also called acid-resistant ceramics, have demonstrated better clinical performance than feldspar-, leucite-, and lithium disilicate-based ceramics, known as acid-sensitive ceramics. However, an increase in mechanical strength, by increasing the crystalline content and decreasing the glass content, results in an acid-resistant ceramic whereby any type of acid treatment produces insufficient surface changes for adequate bonding to resin [24,25,30,31,35,37,51,54,73]. For these acid-resistant ceramics, a silica coating process (silicatization) has been suggested to maximize the bond to resin [30,42,51,54,70,75]. The silica coating systems (Rocatec and Cojet, 3M-ESPE) create a silica layer on the ceramic surface because of the high-speed surface impact of the alumina particles modified by silica. It has been reported that the airborne particles can penetrate up to 15 mm into the ceramic and metal substrates [70]. This tribochemical effect of the silica coating systems may be explained by two bonding mechanisms: (1) the creation of a topographic pattern via airborne-particle abrasion allowing for micromechanical bonding to resin; and (2) the chemical bond of the silica coated ceramic surface, the silane agent, and the resin material. Therefore, a silica-silane chemical bond can occur with acid-resistant ceramics if a silica coating of the ceramic surface is used (Figure 4)[31,45,46,54,75].

Silane has been used to enhance bonding between organic adhesives and ceramics or metals in various industries since the 1940's[62]. The technology of organosilane coating of inorganic filler particles has improved their bonding to matrix resins [13]. This technology also improves the chemical adhesion of ceramic bonded restorations[43,44,66] and resin-bonded ceramic repairs [8,12,18,24,26,27,40,48]. However, the long-time stability of the adhesive bonding using silane coupling agents has been challenged[10,32,33,44,63].

Silane coupling agents bond to Si-OH on ceramic surfaces by condensation reactions and methyl methacrylate double bonds provide bonding to the adhesive. As long as there are adequate Si-OH sites on the ceramic surface, satisfactory bonding should be achieved. Therefore, if the goal is to obtain a thin silane coating on any ceramic surface, the silane protocol should consider the different ceramic microstructures and silane types, and mechanisms to reduce the silane coating thickness, *i.e.*, heat treatment.

Silane is known to be hydrophobic, which is the property believed to reduce hydrolytic degradation of the bond. It also may improve wetting of the ceramic surface by the adhesive, since the silane-coated surface is organophilic to the adhesive. However, the contact angle measurements have proven otherwise[29]. To obtain complete wetting of a surface, the adhesive must initially be of low viscosity and have a surface tension lower than the critical surface tension of the mineral surface[62]. Lee[49] (1975) reported that the surface energy of glass surfaces treated with a silane coupling agent is 36.7 mJ/m^2 and the critical surface energy is 28.0 mJ/m^2 at 20^{o}C. Both values are lower than the surface tension of the resin (39.7 mJ/m^2). This should explain the high contact angle values observed for silane treated ceramic surfaces[29].

Therefore, the adhesion between dental ceramics and resin-based composites is the result of a physico-chemical interaction across the interface between the resin (adhesive) and the ceramic (substrate). The physical contribution to the adhesion process is dependent on the surface topography of the substrate and can be characterized by its surface energy. Alteration of the surface topography, *e.g.*, etching and airborne-particle abrasion, will result in changes on the surface area and on the wettability of the substrate[29,59]. This may also change the surface energy and the adhesive potential [7,29,41,56].

The wetting behavior (wettability) of the resin on the treated ceramic substrate can be characterized using contact angle measurements and surface energy calculations. The wettability of a solid surface by a liquid (*e.g.*, adhesive) can be characterized by Young's equation (Figure 5):

$$\gamma_{SL} = \gamma_{SV} - \gamma_{LV}\ \mathrm{Cos}q \qquad (1)$$

where g_{SV} is the free energy per unit area of the solid surface in equilibrium with vapor, g_{LV} is the surface tension of liquid balanced with its vapor tension, g_{SL} is the interfacial energy, and q is the contact angle. The work of adhesion (W_A) of the liquid drop on a substrate can be expressed by Dupré's equation:

$$W_A = \gamma_{SV} + \gamma_{LV} - \gamma_{SL} \qquad (2)$$

Combining equations (1) and (2) yields the following Young-Dupré equation:

$$W_A = \gamma_{LV}\ (1 + \mathrm{Cos}q) \qquad (3)$$

An increase in the ceramic surface energy can improve the bond strength between ceramic and resin composite. Contact angle values can be used as an indicator of total surface energy and wettability[80]. The dynamic contact angle (DCA) analysis using high-performance liquid chromatography (HPLC) grade water as the probing liquid was used to quantify the influence of surface treatments on contact angle (θ) of a feldspathic ceramic[59]. It was found that chemical and mechanical treatment of ceramic surfaces

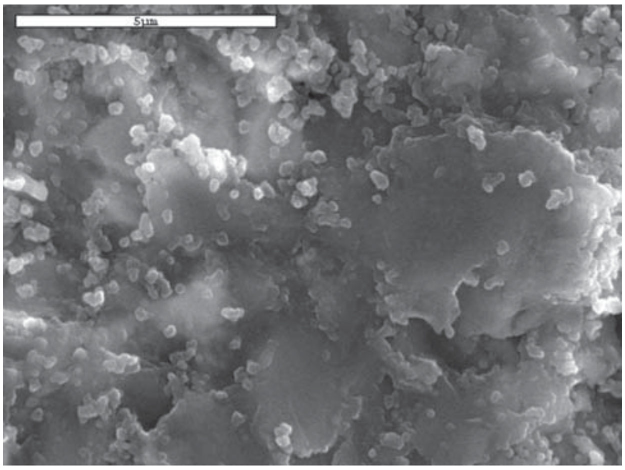

FIGURE 4- SEM Photomicrograph of a silica coated In-Ceram Zirconia ceramic (Vita Zahnfabrik, Bad Sackingen, Germany), a glass-infiltrated zirconia-reinforced alumina-based ceramic. The little grains bonded onto the ceramic surface are alumina (Al_2O_3) particles modified by silica that were sandblasted using the Cojet system (3M-ESPE).

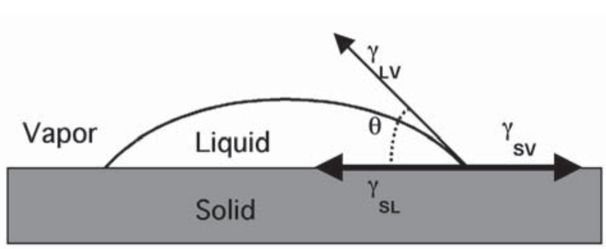

FIGURE 5- Schematic illustration of a liquid drop on a solid surface with energy vectors and contact angle (θ) as described by the equations 1-3. From Della Bona, et al. [29], 2004.

yield increased total surface areas and increased total surface energies. The used of high purity water as a probing medium is valid for analyzing differences between substrates, such as the effect of etching on dental ceramic surfaces. According to equation (3), W_A is dependent on the surface tension of the liquid (*e.g.*, an adhesive) and its contact angle on the substrate. Thus, the values obtained using water as a probing liquid are not useful to calculate the W_A of resin bonded to ceramic. Instead, an adhesive-equivalent fluid resin should be used as the probing medium to characterize the changes in the wetting behavior of dental ceramics caused by any surface treatment[29]. So, to study the W_A for clinical systems, a resin of similar composition to that of the adhesive should be used. This study protocol was used[29] and results showed that untreated ceramics displayed a larger mean contact angle (θ) than the etched ceramics. This improved ceramic wettability by low viscosity resins is resultant of the increase in surface area, which allows a solid to draw more medium onto its surface and exerts greater interfacial force on the specimen. Consequently, roughened surfaces display smaller contact angles and a greater W_A. A good correlation can be observed between the amount of surface disruption and the resulting contact angle. Thus, the greater the surface disruption, the lower the contact angle and the greater the W_A[29].

Bond strength and interfacial fracture toughness of resin/ceramic systems

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 values. The commonly used shear bond test often produces fracture at a distance from the resin-ceramic adhesion zone that may lead to erroneous conclusions on bond quality. Such failures of the substrate prevent measurement of interfacial bond strength and limit further improvements in bonding systems[16,21-23,78].

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 and suggest a lack of reliability of such measurements in assessing the adhesive behavior of bonded dental materials. Several studies have identified the nonuniform stress distributions along bonded interfaces. These variable stress patterns suggest that a standardized research protocol may address only a part of the problem [3,4,9,20,22,69,76,78].

In the search for a method that produces uniform stress distribution across the interface, investigators have evaluated similar adhesive systems under different bond test configurations[22,47,67]. These studies suggest that a tensile bond strength test may be more appropriate to evaluate the bond strength of adhesive interfaces because of more uniform interfacial stresses.

The microtensile test, a tensile bond test with reduced testing area, was developed as an attempt to eliminate the nonuniform stress distribution at the adhesive interface and to minimize the influence of interfacial defects[64]. The reduction in the number and size of defects in the adhesive zone is thought to decrease bulk cohesive failures and increase the tensile bond strengths, regardless of the cross-sectional shape. This test has been used to measure the bond strength of composite to dental tissues[15,57,58,60,64,67,68] and to ceramics [24,27,34].

The non-trimming method to obtain specimens for the microtensile test places less stress on the adhesion zone[58]. As no specimen finishing is necessary, this method also avoids areas of stress concentration produced by polishing materials of different hardness values[24,27].

The evaluation of the structural integrity of the adhesion zone by Weibull analysis is also an important component for an integral analysis of the bonding interface [24,27]. The strength values reported using the microtensile test are considered a reliable indicator of the composite-ceramic bond quality since all 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. However, the ceramic/resin seems to be the weakest bond interface of this system and each ceramic surface treatment have a trend for the mode of failures, which have been studied and classified (Figure 6) [27]. Adhesive failures (mode 1) are not common and normally happened during specimen cutting procedures. HF-treated ceramic specimens usually produce failures that start at the ceramic-adhesive interface and propagate through the adhesive (mode 4), then either reach the adhesive-composite interface (mode 5) or return to the ceramic-adhesive interface (mode 2)[24,27].

Therefore, optical microscopy observation is often not enough to determine the mode of failure of bonding interfaces. A thorough SEM examination of the fracture surfaces following the principles of fractography and confirmation of surface composition through the use of X-

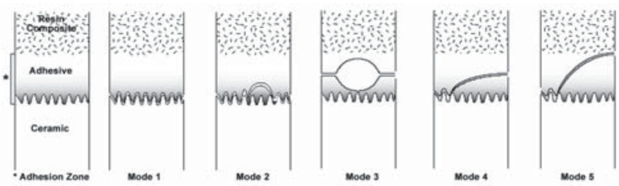

FIGURE 6- Schematic representation (side view) of the modes of failure for the microtensile bond strength test of ceramic bonded to resin composite, based on crack initiation and principles of fractography. Mode 1: adhesive separation at the ceramic-adhesive resin interface. Mode 2: failure starts at the ceramic-adhesive interface, goes into the adhesive resin and returns to the interface. Mode 3: failure from internal flaw (penny-shape internal crack). Mode 4: failure starts at the ceramic-adhesive interface propagates through the adhesive resin. Mode 5: failure starts at the ceramic-adhesive interface, goes through the adhesive resin to reach the resin composite- adhesive interface. From Della Bona, et al. [27], 2003.

ray elemental map analysis (Figure 7) produce a more consistent and complete description of the fracture process and the modes of failure [26,27]. These analyses would avoid simplistic comments such as the "mixed mode of failure" that often follows the "adhesive and/or cohesive" unscientific observations. Thus, when fractography is correctly used to determine the fracture origin, a proper scientific statement on the mode of fracture can be formulated.

Although the mode of failure is an important aspect of bond strength tests, this parameter is not commonly reported. A detailed inspection of the fractured surfaces can indicate the failure mode of a bonded assembly. The fracture behavior of adhesive interfaces will depend on stress level, the flaw distribution, material properties, and environmental effects. Therefore, fracture surface characterization combined with analyses of fracture mechanics parameters are of great importance to understand

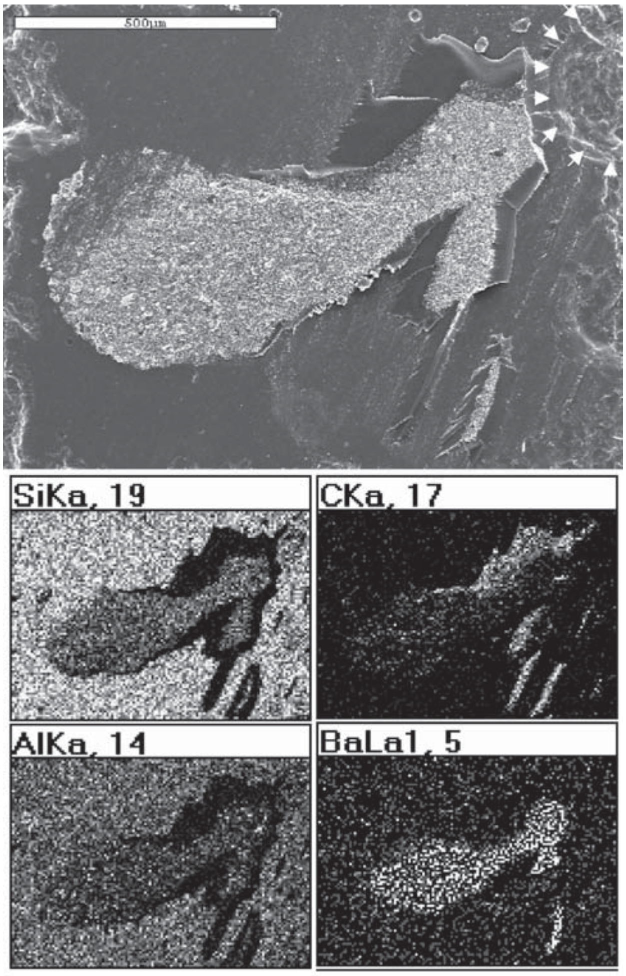

FIGURE 7- SEM image (top) and X-ray elemental maps of fracture surface of IPS Empress ceramic (Ivoclar AG, Schaan, Liechtenstein) bonded to resin composite (Z100, 3M Dental Products, St Paul, USA). The label at the top of X-ray maps indicates the elements and their intensity. The critical flaw is indicated by the white arrows at the top right corner of the SEM micrograph (x100). The fracture starts along the ceramic/adhesive interface, propagates through the adhesive resin to reach the resin composite-adhesive interface (Failure Mode 5). From Della Bona, et al. [27], 2003.

and predict bonded interface reliability[19].

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. Several dentin bond strength studies using microtensile test have reported the modes of failure based on SEM observations[6,53,60,61,65,68,72,79]. These studies based the failure classification on the substrate location where the fracture occurred. Examining the information provided in these studies one concludes that most of the failures occurred within the adhesion zone, as defined by Della Bona, et al.[27] 2003. Yet, an understanding of the fracture mechanics concepts and the analysis of fracture events on the basis of fractography will reduce the risk for data misinterpretation such as the inference that the bond strength must exceed the cohesive strength of the ceramic when the fracture initiates away from the interface. Therefore, the classification of the modes of failure based on principles of fractography (Figure 6) should assist researchers to correctly interpret the fracture phenomena [27].

As demonstrated by finite element stress analyses (FEA), the non-uniformity of the interfacial stress distribution generated during conventional tensile and shear bond strength testing may lead to fracture initiation from flaws at the interface or within the substrate at areas of high localized stress [22,76,77]. To promote crack initiation within the interfacial zone, an interfacial toughness test can be used, as suggested by Della Bona et al. [27], 2003. Those authors used fractography to identify the initial critical flaw and suggested that the interfacial fracture toughness, a more meaningful property than bond strength, could be assessed employing fracture mechanics principles.

The fracture toughness value (K_{IC}) reflects the ability of a material to resist unstable crack propagation. Extensive literature exists on the various techniques used for measuring the fracture toughness of ceramics[28,38]. Fracture mechanics allows quantification of the relationships between material properties, stress level, the presence of crack-producing flaws, and crack propagation mechanisms. Another way to assess the strength of the bond is to estimate the apparent interfacial fracture toughness (K_A) of the adhesion zone by promoting crack initiation within the bonding interface. Strictly speaking, measurement of the toughness at the interface using K_{IC} is undefined. However, tensile tests can be performed in which a crack or defect is the source of failure. Therefore, the apparent fracture toughness of the interface can be calculated from the size of the defect and the strength with the appropriate geometric factor. Thus, the apparent fracture toughness value (K_A) reflects the ability of a material to resist unstable crack propagation at the interface[52,71].

Usually, in order to maintain equal compliances of the specimen halves, *i.e.* for the two halves to have equal strain energy, most of the interfacial fracture toughness tests require $E_1 d_1^3 = E_2 d_2^3$, where E_1 is the elastic modulus for the first specimen half (ceramic), d_1 is the depth of the ceramic rectangular segment, E_2 is the elastic modulus of the composite half, and d_2 is the depth of the composite rectangular segment [27,52,71]. As the microtensile test is a uniaxial tensile test, there is no need for preparing specimens with balanced compliance for the two materials to measure K_A. Yet, the fracture toughness in the form of the stress intensity factor is really a pseudocritical stress-intensity factor, *i.e.* apparent toughness (K_A), considering the fact that it is difficult to define a stress intensity at an interface and one must determine an effective modulus for the two systems[52].

Another appropriate way to assess the interfacial bond is to analyze the energy per unit crack surface area, G_{IC}, that is required for a crack to advance in the bond plane. The toughness relative to the strain energy release rate (G_{IC}) is an accurate measure of the resistance of the bond to fracture since G_{IC} represents the relative energy required to create the new surfaces [52].

This review suggests that (1) microstructure and composition are controlling factors in the development of micromechanical retention produced by etching; (2) roughening the ceramic surface by HF etching and silane coating still yields the highest bond strength values for acid-sensitive ceramics; (3) silica coating acid-resistant ceramics is important to improve bonding to resin; (4) the tensile bond strength and the apparent interfacial fracture toughness of ceramic bonded to resin is affected by the ceramic microstructure and the ceramic surface treatments; (5) the definition of the adhesion zone is critical to classify the modes of failure, which should be an integral component of all failure analyses; (6) the microtensile test may be preferable to conventional shear or flexural tests as an indicator of composite-ceramic bond quality, since fractures occur within the adhesion zone; (7) a careful microscopic analysis of the fracture surfaces and a X-ray elemental map can produce a more consistent and complete description of the fracture process and interpretation of the modes of failure; and (8) there is a positive correlation between the W_A, the tensile bond strength (ó), and the K_A, that is, the higher the mean W_A value, the higher the mean ó and K_A values.

Thus, the quality of the bond should not be assessed based on bond strength data alone. The mode of failure and fractographic analyses should provide important information leading to predictions of clinical performance limits, which is the ultimate test of any adhesive system. Future studies should also focus on optimum surface treatment conditions because of the poor adhesion associated with acid-resistant ceramics.

ACKNOWLEDGEMENTS

This review is based on the knowledge gathered during the graduate course in Restorative Dentistry at the University of Texas Health Science Center at San Antonio, U.S.A., supervised by Dr. James B. Summitt, followed by the MMedSci program in Restorative Dentistry at the University of Sheffield, UK, supervised by Dr Richard van Noort, and, mainly, during the PhD program in Materials Science and Engineering at the University of Florida, USA, supervised by Dr Kenneth J. Anusavice and Dr John J. Mecholsky Jr. I also thank the continuing support of CNPq-Brazil, grant 300659/03-2, for my research work at the University of Passo Fundo.

REFERENCES

1- Aida M, Hayakawa T, Mizukawa K. Adhesion of composite to porcelain with various surface conditions. J Prosthet Dent. 1995;73:464-70.

2- al Edris A, al Jabr A, Cooley RL, Barghi N. SEM evaluation of etch patterns by three etchants on three porcelains. J Prosthet Dent. 1990;64:734-9.

3- Anusavice KJ, Dehoff PH, Fairhurst CW. Comparative evaluation of ceramic-metal bond tests using finite element stress analysis. J Dent Res. 1980;59:608-13.

4- Anusavice KJ. Screening tests for metal-ceramic systems. In: McLean JW, ed. Dental ceramics: Proceedings of the First International Symposium on Ceramics. Chicago, IL: Quintessence Publishing Co.; 1983. p. 371-414.

5- Anusavice KJ, Lee RB. Effect of firing temperature and water exposure on crack propagation in unglazed porcelain. J Dent Res. 1989;68:1075-81.

6- Armstrong SR, Boyer DB, Keller JC. Microtensile bond strength testing and failure analysis of two dentin adhesives. Dent Mater. 1998;14:44-50.

7- Attal JP, Edard V, Degrange M. Factors modifying the accuracy of free energy surface measured by contact angle method. J Biomater Dent. 1990;5:143-55.

8- Bailey JH. Porcelain-to-composite bond strengths using four organosilane materials. J Prosthet Dent. 1989;61:174-7.

9- Ban S, Anusavice KJ. Influence of test method on failure stress of brittle dental materials. J Dent Res. 1990;69:1791-9.

10- Berry T, Barghi N, Chung K. Effect of water storage on the silanization in porcelain repair strength. J Oral Rehabil. 1999;26:459-63.

11- Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent. 2003;89:268-74.

12- Bouschlicher MR, Reinhardt JW, Vargas MA. Surface treatment techniques for resin composite repair. Am J Dent. 1997;10:279-83.

13- Bowen RL. Properties of silica-reinforced polymer for dental restorations. J Am Dent Assoc. 1963;66:57-64.

14- Callister WD. Materials Science and Engineering: an Introduction. 5th ed. New York: John Wiley & Sons, Inc.; 2000.

15- Carvalho RM, Mendonca JS, Santiago SL, Silveira RR, Garcia FC, Tay FR, Pashley DH. Effects of HEMA/solvent combinations on bond strength to dentin. J Dent Res. 2003;82: 597-601.

16- Chadwick RG, Mason AG, Sharp W. Attempted evaluation of three porcelain repair systems - what are we really testing? J Oral Rehabil. 1998;25:610-5.

17- Chen JH, Matsumura H, Atsuta M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. J Dent. 1998;26:53-8.

18- Chen JH, Matsumura H, Atsuta M. Effect of etchant, etching period, and silane priming on bond strength to porcelain of composite resin. Oper Dent. 1998;23:250-7.

19- Chen Z, Mecholsky Jr. JJ. Control of strength and toughness of ceramic/metal laminates using interface design. J Mater Res. 1993;8:2362-9.

20- DeHoff PH, Anusavice KJ, Wang Z. Three-dimensional finite element analysis of the shear bond test. Dent Mater. 1995;11:126-31.

21- Della Bona A, Northeast SE. Shear bond strength of resin bonded ceramic after different try-in procedures. J Dent .1994;22:103-7.

22- Della Bona A, van Noort R. Shear versus tensile bond strength of resin composite bonded to ceramic. J Dent Res. 1995;74:1591-6.

23- Della Bona A, van Noort R. Ceramic surface preparations for resin bonding: a SEM study. Am J Dent. 1998;11:276-80.

24- Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. J Adhes Dent. 2000;2:305-13.

25- Della Bona A, Anusavice KJ. Microstructure, composition, and etching topography of dental ceramics. Int J Prosthodont. 2002;15:159-67.

26- Della Bona A, Anusavice KJ, Hood JAA. Effect of ceramic surface treatment on tensile bond strength to a resin cement. Int J Prosthodont. 2002;15:248-53.

27- Della Bona A, Anusavice KJ, Mecholsky Jr. JJ. Failure analysis of resin composite bonded to ceramic. Dent Mater. 2003;19: 693-9.

28- Della Bona A, Anusavice KJ, Mecholsky Jr. JJ. Fracture behavior of lithia disilicate- and leucite-based ceramics. Dent Mater. 2004;20:956-62.

29- Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. Dent Mater. 2004;20:338-44.

30- Della Bona A. Restaurações em cerâmica pura (metal-free) e mecanismos de adesão. In: Bottino MA. Livro do Ano da Clínica Odontológica Brasileira. 1ª ed. São Paulo: Artes Médicas; 2004. p. 61-86.

31- Dérand P, Dérand T. Bond strength of luting cements to zirconium oxide ceramics. Int J Prosthodont. 2000;13:131-5.

32- Diaz-Arnold AM, Aquilino SA. An evaluation of the bond strengths of four organosilane materials in response to thermal stress. J Prosthet Dent. 1989;62:257-60.

33- Eikenberg S, Shurtleff J. Effect of hydration on bond strength of a silane-bonded composite to porcelain after seven months. Gen Dent. 1996;44:58-61.

34- El Zohairy AA, De Gee AJ, Mohsen MM, Feilzer AJ. Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks. Dent Mater. 2003;19:575-83.

35- Fradeani M, Redemagni M. An 11-year clinical evaluation of leucite-reinforced glass-ceramic crowns: a retrospective study. Quintessence Int. 2002;33(7):503-10.

36- Hayakawa T, Horie K, Aida M, Kanaya H, Kobayashi T, Murata Y. The influence of surface conditions and silane agents on the bond of resin to dental porcelain. Dent Mater. 1992;8:238-40.

37- Hayashi M, Tsuchitani Y, Kawamura Y, Miura M, Takeshige F, Ebisu S. Eight-year clinical evaluation of fired ceramic inlays. Oper Dent. 2000;25:473-81.

38- Hertzberg RW. Deformation and fracture mechanics of engineering materials. 4ª ed. New York: J. Wiley & Sons; 1996.

39- Hussain MA, Bradford EW, Charlton G. Effect of etching on the strength of a luminous porcelain jacket crown. Br Dent J. 1979;147:89-90.

40- Jardel V, Degrange M, Picard B, Derrien G. Correlation of topography to bond strength of etched ceramic. Int J Prosthodont. 1999;12:59-64.

41- Jardel V, Degrange M, Picard B, Derrien G. Surface energy of etched ceramic. Int J Prosthodont. 1999;12:415-8.

42- Jedynakiewicz NM, Martin, N. The effect of surface coating on the bond strength of machinable ceramics. Biomaterials. 2001;22:749-52.

43- Kamada K, Yoshida K, Atsuta M. Effect of ceramic surface treatments on the bond of four resin luting agents to a ceramic material. J Prosthet Dent. 1998;79:508-13.

44- Kato H, Matsumura H, Tanaka T, Atsuta M. Bond strength and durability of porcelain bonding systems. J Prosthet Dent. 1996;75:163-8.

45- Kern M, Thompson VP. Sandblasting and silica coating of a glass-infiltrated alumina ceramic: volume loss, morphology, and changes in the surface composition. J Prosthet Dent. 1994;71:453-61.

46- Kern M, Thompson VP. Bonding to glass infiltrated alumina ceramic: adhesive methods and their durability. J Prosthet Dent. 1995;73:240-9.

47- Kitasako Y, Burrow MF, Nikaido T, Harada N, Inokoshi S, Yamada T, Takatsu T. Shear and tensile bond testing for resin cement evaluation. Dent Mater. 1995;11:298-304.

48- Kupiec KA, Wuertz KM, Barkmeier WW, Wilwerding TM. Evaluation of porcelain surface treatments and agents for composite-to-porcelain repair. J Prosthet Dent. 1996;76:119-24.

49- Lee LH. Adhesion science and technology. American Chemical Society. New York: Plenum Press; 1975.

50- Leibrock A, Degenhart M, Behr M, Rosentritt M, Handel G. In vitro study of the effect of thermo- and load-cycling on the bond strength of porcelain repair systems. J Oral Rehabil. 1999;26:130-7.

51- Madani M, Chu FCS, McDonald AV, Smales RJ. Effects of surface treatments on shear bond strengths between a resin cement and an alumina core. J Prosthet Dent. 2000;83:644-7.

52- Mecholsky JJ Jr., Barker LM. A chevron-notched specimen for fracture toughness measurements of ceramic-metal interfaces. ASTM STP 1984;855:324-36.

53- Nakajima M, Sano H, Urabe I, Tagami J, Pashley DH. Bond strengths of single-bottle dentin adhesives to caries-affected dentin. Oper Dent. 2000;25:2-10.

54- Özcan M, Alkumru H, Gemalmaz D. The effect of surface treatment on the shear bond strength of luting cement to a glass-infiltrated alumina ceramic. Int J Prosthodont. 2001;14:335-9.

55- Pameijer CH, Louw NP, Fischer D. Repairing fractured porcelain: how surface preparation affects shear force resistance. J Am Dent Assoc. 1996;127:203-9.

56- Panighi M, G'Sell C. Physico-chemical study of dental surfaces and mechanisms of composite adhesion. J Biomater Dent. 1993; 8:61-70.

57- Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. Dent Mater. 1995;11:117-25.

58- Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, *et al.*. The microtensile bond test: a review. J Adhes Dent. 1999;1:299-309.

59- Phoenix RD, Shen C. Characterization of treated porcelain surfaces via dynamic contact angle analysis. Int J Prosthodont. 1995;8:187-94.

60- Phrukkanon S, Burrow MF, Tyas MJ. Effect of cross-sectional surface area on bond strengths between resin and dentin. Dent Mater. 1998;14:120-8.

61- Phrukkanon S, Burrow MF, Tyas MJ. The effect of dentine location and tubule orientation on the bond strengths between resin and dentine. J Dent. 1999;27:265-74.

62- Plueddemann EP. Silane coupling agents. New York: Plenum Press; 1991.

63- Pratt RC, Burgess JO, Schwartz RS, Smith JH. Evaluation of bond strength of six porcelain repair systems. J Prosthet Dent. 1989;62:11-13.

64- Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley D. Relationship between surface area for adhesion and tensile bond strength- Evaluation of a micro-tensile bond test. Dent Mater. 1994;10:236-40.

65- Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Tagami J, et al. Long-term durability of dentin bonds made with a self-etching primer, in vivo. J Dent Res. 1999;78:906-11.

66- Sato K, Matsumura H, Atsuta M. Effect of three-liquid bonding agents on bond strength to a machine-milled ceramic material. J Oral Rehabil. 1999;26:570-4.

67- Schreiner RF, Chappell RP, Glaros AG, Eick JD. Microtensile testing of dentin adhesives. Dent Mater. 1998;14:194-201.

68- Shono Y, Ogawa T, Terashita M, Carvalho RM, Pashley EL, Pashley DH. Regional measurement of resin-dentin bonding as an array. J Dent Res. 1999;78:699-705.

69- Sudsangiam S, van Noort R. Do dentin bond strength tests serve a useful purpose? J Adhes Dent. 1999;1:57-67.

70- Sun R, Suansuwan N, Kilpatrick N, Swain M. Characterization of tribochemically assisted bonding of composite resin to porcelain and metal. J Dent. 2000;28:441-5.

71- Tam LE, Pilliar RM. Fracture toughness of dentin/resin-composite adhesive interfaces. J Dent Res. 1993;72:953-9.

72- Tanumiharja M, Burrow MF, Tyas MJ. Microtensile bond strengths of seven dentin adhesive systems. Dent Mater. 2000;16:180-7.

73- Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann H. Fracture resistance of lithium disilicate-, alumina-, and zirconia-based three-unit fixed partial dentures: a laboratory study. Int J Prosthodont. 2001;4:231-8.

74- Tylka DF, Stewart GP. Comparison of acidulated phosphate fluoride gel and hydrofluoric acid etchants for porcelain-composite repair. J Prosthet Dent. 1994;72:121-7.

75- Valandro LF, Della Bona A, Bottino MA, Neisser MP. The effect of ceramic surface treatment on bonding to densely sintered alumina ceramic. J Prosthet Dent. 2005;93:253-9.

76- Van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. J Dent. 1989;17: 61-7.

77- Van Noort R, Cardew GE, Howard IC, Noroozi S. The effect of local interfacial geometry on the measurement of the tensile bond strength to dentin. J Dent Res. 1991;70:889-93.

78- Versluis A, Tantbirojn D, Douglas WH. Why do shear bond tests pull out dentin? J Dent Res. 1997;76:1298-307.

79- Yoshikawa T, Sano H, Burrow MF, Tagami J, Pashley DH. Effects of dentin depth and cavity configuration on bond strength. J Dent Res. 1999;78:898-905.

80- Zisman WA. Relation of the equilibrium contact angle to liquid and solid constitution. Advan Chem Ser. 1964;43:1-51.