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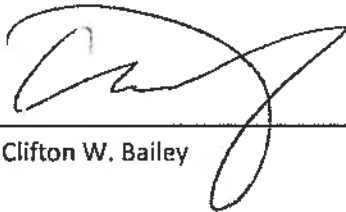
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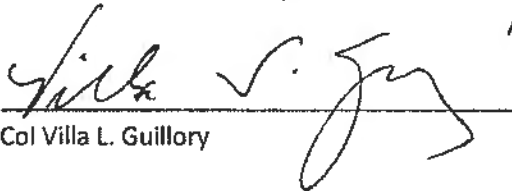External Affairs Approval   Date

Microtensile Bond Strength of New Ceramic/Polymer Materials Repaired with Composite Resin

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30 June 2015

Learning to Care for Those in Harm's Way

Microtensile Bond Strength of New Ceramic/Polymer
Materials Repaired with Composite Resin

Abstract

The new millable ceramic/polymer block materials reportedly are easily repaired with traditional composite-resin restorative materials. The purpose of this study was to evaluate the microtensile bond strength of restorative (Filtek Supreme Ultra, 3M/ESPE) and laboratory (Sinfony, 3M/ESPE) composites to ceramic/polymer materials (Lava Ultimate, 3M/ESPE; Enamic, Vita) utilizing two surface treatments (hydrofluoric acid (HF)/silane or tribochemical coating). All blocks were bonded with Scotchbond Universal Adhesive (3M/ESPE) and veneered with either Filtek Supreme Ultra or Sinfony composite resin. Also, monolithic blocks without composite veneering were included as control groups. The blocks were sectioned into beams, thermo-cycled and loaded to failure on a universal testing machine. A mean microtensile bond strength and standard deviation was determined per group. Data were analyzed with ANOVA/Tukey's tests (alpha=0.05). With the Lava Ultimate resin nano-ceramic material, neither the type of surface treatment nor the type of composite resulted in significantly greater bond strengths. With the Enamic hybrid-ceramic material, the use of hydrofluoric-acid etch/silane or Sinfony laboratory composite resulted in significantly greater bond strengths. All of the composite-resin-repaired ceramic/polymer treatment groups were significantly weaker in bond strength than the cohesive strength of the monolithic block material.

Introduction

Marketers have touted the ability of chairside computer-aided design/computer-aided manufacture (CAD/CAM) restoration systems to allow the clinician to design, mill and deliver an indirect single-tooth restoration in a single appointment obviating the need for additional appointments, repeated anesthesia, and potential problems related to fabrication and delivery of interim restorations (e.g., contamination of the surface to be bonded with eugenol-based material, loss/fracture, after-hours care). Additional potential benefits of single-tooth CAD/CAM restorative treatments include reduction of overhead costs associated with laboratory-fabricated indirect restorations resulting in savings to the clinician and patient.

Heretofore CAD/CAM systems have required costly and messy powders for scanning and restorations in previous generations have had poor marginal fidelity, by laboratory-fabricated standards, due in part to micro-chipping of brittle all-ceramic materials. Because of the propensity to chip, it is unlikely that with current milling configurations (e.g., water-cooled diamond burs) that glass-ceramic materials can be milled more quickly without significant reduction in surface and margin quality. Glass-ceramic materials also have been shown to have higher enamel wear rates than composite-resin CAD/CAM restorations (Mörmann et al, 2013).

As material choices, cost, and workflow improve, the demand for CAD/CAM in dentistry continues to grow. More recent versions of CAD/CAM systems are bypassing traditional impression techniques through utilization of intraoral digital scanners such as the Omnicam, (Sirona, Long Island City, NY), E4D (Planmeca, Roselle, IL), iTero, (Cadent, San Jose, CA) and True Definition Scanner (3M, St. Paul, MN). These systems capture an intraoral scan of the hard and soft tissues. Many of the early CAD/CAM material options (e.g., feldspathic porcelain, leucite-reinforced porcelain) had reduced fracture resistance. Modest gains in durability were gained with the advent of lithium-disilicate-based CAD/CAM material (Fasbinder 2012). Yttrium-partially-stabilized tetragonal zirconia polycrystalline (YTZP) material with its high fracture

toughness and low cost has gained wider acceptance in the dental community for single tooth as well as multi-unit fixed dental prostheses. Improvements in durability of newer generations of all-ceramic systems have disadvantages as well. Lithium-disilicate restorations and YTZP restorations are machined in a partial green or green state and require sintering at high temperatures for approximately 20 minutes to over an hour respectively (Fasbinder 2012). Multi-unit YTZP restorations cannot be separated and soldered if the substructure is found to be warped (Miyazakin and Hotta 2011). Many YTZP units still require application of veneering porcelain which has been shown to delaminate at significantly lower tensile and shear strengths than other substructures veneered with porcelain material (Choi et al, 2009). Lithium-disilicate, leucite-reinforced, and feldspathic porcelains can require correction firings and additional firings for stain and glaze procedures and to repair contacts - adding to the chair time, equipment, and energy consumption required to deliver a restoration.

CAD/CAM composite-resin restorations may alleviate many of the aforementioned challenges to fabrication and repair. Fasbinder et al, (2013) noted that the polymer chemistry of CAD/CAM-fabricated composite-resin restorations, such as Paradigm MZ100 (3M/ESPE), render them more easily adjusted, polished and repaired; although the longevity of these repairs has not been validated by clinical studies. Paradigm MZ100 showed the least amount of opposing enamel wear compared with feldspathic and leucite-reinforced ceramic material but nearly twice the material wear rate (Kunzelmann et al, 2001).

A randomized clinical study of 40 milled Paradigm MZ100 composite inlays and 40 milled Vita Mark II (Yorba Linda, CA) feldspathic porcelain inlays reported the performance of composite inlays equal to porcelain inlays in all categories (shade match, margin discoloration, anatomic form, margin finish, margin adaptation, surface finish, cusp/tooth fracture, caries, restoration fracture and sensitivity). Only one composite inlay fractured over the 10-year study and was identified at the 10-year recall. Direct and indirect composites fabricated by photo-polymerization have been reported to have rougher surface texture due to larger filler particles

and greater wear of the composite matrix material. The aforementioned study reported that Paradigm MZ100 "demonstrated superior surface properties that maintained the surface finish and anatomy over time" (Fasbinder et al, 2013).

A possible challenge of composite-based CAD/CAM restorations is their potential reduced ability to bond to resin cements. Feldspathic, leucite-reinforced, and lithium-disilicate restorations are capable of being etched, silanated and bonded to natural tooth structure with composite-resin cements. The preferred method(s) of bonding methacrylate-based materials to composite-based or the new ceramic/polymer-baed restorations is not clear. Tantbirojn et al (2015) stated that unreacted double bonds of resin-based composites are essential to achieve chemical adhesion of a new layer of composite to an already cured resin-based composite. Methacrylate groups in freshly cured composites are 25-55% unreacted after initial polymerization. For fully cured composites (e.g., millable composite-resin blocks, intraoral composite restorations) there are a reduced number of unreacted double bonds. Due to aging of the composite resin surface in the oral environment, the adhesive strength of composite to composite restorations decreases by 25 to 80% compared to their original strength (Rinastiti et al, 2011).   Various surface treatment techniques have been used in an attempt to improve bond strength by increasing surface roughness and/or chemically treating the substrate surfaces. Examples include acid etching, surface abrasion, silica coating, silanization and bonding agent application. As more than half of the components of composite restorative materials are ceramic-based filler particles, surface treatment and/or application of an adhesive that promotes affinity to both the methacrylate-based resin and the ceramic-based fillers should be advantageous. For instance, a tribochemical system (CoJet, 3M/ESPE) that combines sandblasting, a proprietary silica-coated sand, silanization, and adhesive resin has been reported to increase bond strengths. Moreover, a recently introduced multimode adhesive (Scotchbond Universal, 3M/ESPE) combines methacryloxydecyl phosphate monomer (10-MDP) and silane to enable bonding to various substrates, including metal, non-glass ceramic, and

glass ceramic. The indication for use of this new universal adhesive includes intraoral repair of existing composites (Tantbirojn et al, 2015).

3M/ESPE and Vita corporations have recently introduced new ceramic/polymer materials - Lava Ultimate and Enamic respectively. 3M/ESPE reports that Lava Ultimate has nano-ceramic particles (80% wt.) embedded in a highly cross-linked resin matrix (20% wt.). Lava Ultimate is based on the similar nano-composite chemistry of Filtek Supreme Ultra restorative composite. Vita claims that Enamic is the first hybrid dental ceramic with a dual-network structure. The dominant fine-structure ceramic network (86% by wt.) is reportedly strengthened by a polymer network with a surface-modified polymethyl methacrylate (Vita). Early claims in trade literature regarding these ceramic/polymer materials are that they demonstrate excellent polishability, lasting esthetics, and similar optical properties to glass ceramics (Rosenblatt, 2012). The manufacturers each claim their products have moduli of elasticity similar to dentin, shortened milling times, greater edge quality - resulting in better marginal adaptation and the ability to absorb shock, resist staining and stop crack propagation. Further manufacturer claims are that ceramic/polymer materials are easily adjusted and polished, kinder to opposing dentition and capable of being characterized and repaired utilizing traditional composite resins and light-cure polymerization (3M/ESPE, Vita).

Greater understanding of the bond strengths of some of the more common direct composite resins to 3M/ESPE Lava Ultimate and Vita Enamic ceramic/polymer materials may provide valuable insights into their repairability as well as their customization. Decreased fabrication time, ease of handling and repairability could make the new CAD/CAM ceramic/polymer materials a valuable tool in the dentist's armamentarium. The purpose of this study was to evaluate the microtensile bond strength of restorative and laboratory composites to two ceramic/polymer materials using two surface treatments. The first and second null hypotheses tested were that there would be no difference in microtensile bond strength of composite to these newer ceramic/polymer blocks based on type of composite resin or surface treatment per

block type. Additionally, a third null hypothesis tested was that there would be no difference in microtensile bond strength of the composite to the ceramic/polymer blocks compared to the cohesive strength of the monolithic blocks.

Materials and Methods

Ceramic/polymer materials tested included 3M/ESPE Lava Ultimate and Vita Enamic machinable blocks.   Composite resin veneering materials included Filtek Supreme Ultra Universal Restorative (3M/ESPE) and Sinfony Indirect Lab Composite (3M/ESPE).   Filtek Supreme Ultra is a nano-composite resin which contains only nanomeric particles and nanoclusters as inorganic fillers. The porous nanocluster is infiltrated with silane-coupling agents to allow chemical bonding with the organic matrix (Mitra et al, 2003). Sinfony Indirect Lab Composite is a microhybrid, indirect laboratory composite developed for laboratories and designed to handle like porcelain (3M/ESPE).   One adhesive bonding agent (Scotchbond Universal, 3M/ESPE) was used for all specimens.

Following 3M/ESPE's repair preparation guidelines, two blocks of Lava Ultimate (size 14L) and two blocks of Enamic (size EM14) were treated with a tribochemical system (CoJet Sand). The CoJet roughened specimens were cleaned by soaking in isopropyl alcohol in an ultrasonic cleaner for 2 minutes then air dried for 3-5 seconds with an air-water syringe. Scotchbond Universal Adhesive was applied with a microbrush by rubbing it onto the prepared surface for 20 seconds, air thinning for 5 seconds then light curing for 10 seconds using a Bluephase G2 (Ivoclar Vivadent, Amherst, NY) light-curing unit. Irradiance was monitored with a radiometer (Bluephase Meter, Ivoclar Vivadent) and was considered acceptable if greater than 1000 mW/cm². Scotchbond Universal contains silane, and according to 3M/ESPE, the application of a separate silane-coupling agent is not required after the CoJet tribochemical silica application. Following surface preparation and adhesive placement, half of the specimens received Filtek Supreme Ultra Universal Restorative (shade A4D) and half received Sinfony Indirect Lab

Composite (shade A4). The composites were added in two separate two-millimeter increments and light cured for 20 seconds per increment using the light-curing unit as before.

Following Vita's repair preparation guidelines, two blocks of Lava Ultimate (size 14L) and two blocks of Enamic (size EM14) were etched using 5% hydrofluoric-acid gel (VitaCeramics Etch, Vita) for 60 seconds followed by immersion in distilled water in an ultrasonic cleaner for two minutes. The blocks were air dried for 20 seconds and then silanated (Vitasil, Vita) and allowed to air dry. Scotchbond Universal Adhesive was applied and light cured as before. After surface preparation and adhesive placement, half of the specimens received Filtek Supreme Ultra and half received Sinfony Indirect Lab Composite as before. See Figure 1.
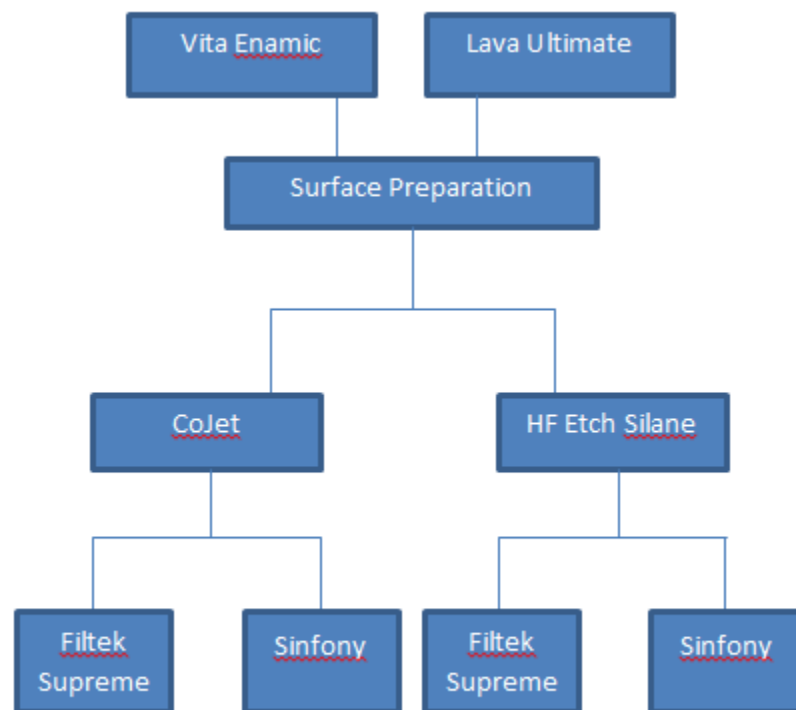

Figure 1.

All blocks were sectioned into approximately 1x1mm beams using a precision saw (Isomet 5000, Buehler, Lake Bluff, IL).   After separating the beams, each individual beam was inspected for defects. Unacceptable specimens were discarded for reasons such as premature fracture during sectioning, chipping, or air inclusions/porosities in the interface. Twenty of the remaining specimens were selected at random from each group (n=20). These specimens were subjected to 500 cycles of thermal cycling (5°C/55°C, 30 seconds dwell time) in a thermal cycling unit (Sabri Dental Enterprises, Downers Grove, IL).

The cross-sectional area of each sample was recorded using a digital caliper, immediately prior to loading. The interfacial surface of each specimen was centered at the interface of a specialized fixture, and luted to the fixture at its lateral surface near the top and bottom of the beams using a cyanoacrylate adhesive (Zap-It, DVA, Corona, CA). Each bar specimen was loaded to failure, at a cross-head speed of 1mm/min on a universal testing machine (Model 5943, Instron, Norwood, MA). The force at time of failure was recorded in newtons and converted to megapascals (MPa = force/cross-sectional area). Following testing, each specimen was examined using 10X stereomicroscope to determine failure mode as either: 1) adhesive fracture at the resin composite/adhesive/ceramic-polymer interface, 2) cohesive fracture in resin composite, 3) mixed (combined adhesive and cohesive) in resin composite and bonded interface or ceramic/polymer and bonded interface or 4) cohesive fracture in ceramic/polymer. The cohesive strength of the ceramic/polymer (control) was determined by determining the microtensile bond strength of solid specimens without bonding composite resin. Blocks were sectioned into beams and tested as before.   A mean and standard deviation was determined per group. Data were analyzed with two-way ANOVAs to evaluate the effect of composite type or surface treatment on the microtensile bond strength of composite to ceramic/polymer ($\alpha = 0.05$). Data were also analyzed with a one-way ANOVA and Tukey's

post-hoc tests to compare the differences between the control and the four treatment groups per ceramic/polymer material (α = 0.05).

RESULTS

No significant difference was found in the bond strength of composite to Lava Ultimate based on type of composite (p=0.857) or surface treatment (p=0.177) with no significant interaction (p=0.110). With the Lava Ultimate resin nano-ceramic material, neither the type of surface treatment nor the type of composite resulted in significantly greater bond strengths. A significant difference was found in the microtensile bond strength of the composite to Enamic based on type of composite (p=0.007) or surface treatment (p=0.042) with no significant interaction (p=0.537). With the Enamic hybrid-ceramic material, the use of hydrofluoric-acid etch/silane or Sinfony laboratory composite resulted in significantly greater bond strengths. The Enamic and Lava Ultimate monolithic control groups were significantly stronger than their respective treatment groups (p<0.001). See Table 1. Filtek Supreme Ultra was associated with more adhesive failures than Sinfony laboratory composite. See Figure 2.

	Mean Microtensile Bond Strength (MPa, st dev)					
	Enamic			**Lava Ultimate**		
	HF etch	CoJet	Total	HF etch	CoJet	
Supreme	24.1 (9.9)	21.5 (8.3)	22.8 (9.1) a	34.3 (17.6)	33.5 (19.6)	33.9 (18.4) a
Sinfony	30.3 (8.8)	25.4 (4.8)	27.9 (7.4) b	29.6 (10.6)	39.3 (7.2)	34.5 (10.3) a
Total	27.2 (9.8) B	23.4 (7.0) A		31.9 (14.6) A	36.4 (14.9) A	
Control	46.8 (18.0)			106.5 (15.3)		
	Within each ceramic/polymer type, groups with the same lower case letter per column or upper case letter per row are not significantly different (P>0.05)					

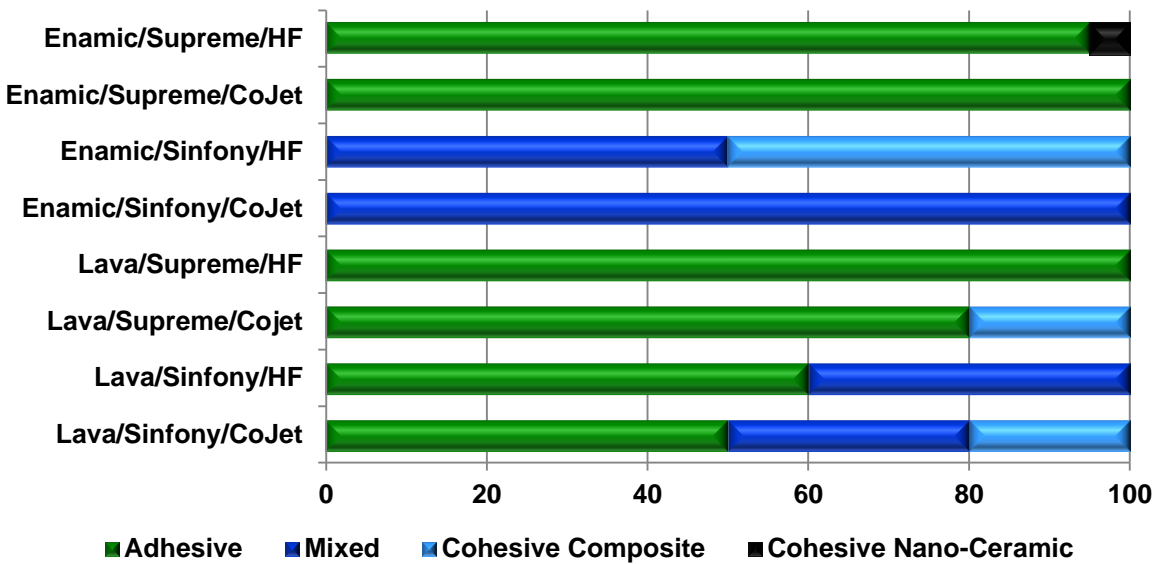Table 1. Mean microtensile bond strength and standard deviations

Figure 2. Failure modes

Discussion

Industrially prepared and polymerized composite-based CAD/CAM restorative materials seem a reasonable alternative to the all-ceramic restorative materials. Fasbinder et al (2013) evaluated survivability/durability of previous generations of composite CAD/CAM restorations (Paradigm MZ100) and found that only one composite inlay fractured over a 10 year period. The new generation of "hybrid ceramics" or "new ceramic/polymer materials" such as Enamic and Lava Ultimate—with their aluminum oxide-enriched, fine-structure feldspar matrix or nanoceramic particles, respectively, seem poised to capitalize on the long-term survival seen with the earlier generation of Paradigm MZ100 composite-resin blocks. The manufacturers claim that these new ceramic/polymer materials are easily adjusted and polished, kinder to opposing dentition and capable of being characterized and repaired utilizing traditional composite resins and light-cure polymerization (3M/ESPE, Vita). Although resin-based bonding to silica-based ceramics is well-documented, only a few studies have been published evaluating

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the surface treatment and bonding to indirect composite materials. Even less research is available evaluating the repairability of the new ceramic/polymer materials.

With Lava Ultimate, the first and second null hypotheses were not rejected. No significant differences were found in microtensile bond strength based on type of composite or type of surface treatment. There was no significant difference in bond strength between the two resin-composite restorative materials. Lava Ultimate consists of nano-ceramic particles embedded in a highly cured resin matrix and has been characterized as having a similar formulation to Filtek Supreme Ultra, a direct nano-composite restorative material (3M/ESPE). Sandblasting the surface of Lava Ultimate with CoJet, a silica-coated aluminum oxide, followed by a silane-containing universal bonding agent, Scotchbond Universal, resulted in greater bond strength when using Sinfony composite; however, in general, there was no statistical significant difference between the two types of surface treatments. The current 3M/ESPE instructions for Lava Ultimate recommend sandblasting with silica-coated aluminum oxide followed by their universal bonding agent.

With Enamic, the first and second null hypotheses were rejected. Significant differences were found in the microtensile bond strength based on type of composite and on type of surface treatment. The use of Sinfony laboratory composite or hydrofluoric-acid etch/silane resulted in significantly greater bond strengths. Sinfony had more mixed and cohesive failures than Filtek Supreme Ultra composite. Mixed and cohesive failures are typically associated with greater bond strength and/or cohesive strength than purely adhesive-type failures (Bottino et al, 2014). Sinfony Indirect Lab Composite is a microhybrid, indirect laboratory composite developed for laboratories and purported to handle like porcelain (3M/ESPE). Enamic has been described as a ceramic substructure infiltrated with a composite material. It consists of an aluminum oxide-enriched, fine-structure feldspar matrix (86 wt%) infused by a polymer material consisting of 14 wt% urethane dimethacrylate and triethylene glycol dimethacrylate (Vita, Spitznagel et al, 2014). Acid-etching with hydrofluoric acid solutions plus the use of a silane has been reported as the

best surface-conditioning protocol for silica-based ceramics (Vargas et al, 2011, Spitznagel et al. 2014). The higher ceramic content of Enamic may have resulted in significantly greater bonds strengths using the manufacturer-recommended combination of hydrofluoric acid and silane compared to the use of the silica-coating system.

With both Enamic and Lava Ultimate, the third null hypothesis was rejected. A significant difference was found in the microtensile bond strength of the composite to the ceramic/polymer materials compared to the cohesive strength of the monolithic material. All of the composite-resin repaired ceramic/polymer treatment groups were significantly weaker in bond strength than the cohesive strength of the monolithic block material. One of the beneficial properties of pre-polymerized composite restorative materials is the high degree of conversion of the polymer matrix. The higher polymerization may contribute to the increased durability and resistance to wear, color stability, and reduced potential for irritation in patients sensitive to unreacted monomers. Conversely, the high rates of conversion may reduce the ability of composite resins or resin cements to bond to the restoration (Bottino et al, 2014). Repair strengths of composite vary widely in the literature and have been found to range between 25 and 80% of the cohesive strength of the intact composite (Teixeira et al, 2005). The repair strength of the hybrid-ceramic material, Enamic, was 54.3% of the cohesive strength of the monolithic material compared to the resin nano-ceramic, Lava Ultimate, which was only 32.1%. However, the resin nano-ceramic had greater cohesive strength (106.5 MPa) and composite repair bond strength (34.2 MPa) compared to the cohesive strength (46.8 MPa) and repair bond strength (25.4 MPa) of the hybrid ceramic.

More studies are necessary to investigate manufacturer claims that the new ceramic/polymer materials are easily characterized and repaired utilizing traditional composite resins and light-cure polymerization. Clinicians should be aware that if attempting to repair a ceramic/polymer material chairside, the repair bond strength of the composite resin material may only be half or less than the original strength of the material. Clinical trials evaluating the

bonding behavior of the new ceramic/polymer materials must be published before these materials can be universally accepted as alternatives to the more traditional all-ceramic systems.

Conclusion

With the Enamic ceramic/polymer material, the use of HF-acid etch/silane or Sinfony laboratory composite resulted in significantly greater bond strengths. With the Lava Ultimate resin nano-ceramic material, neither the method of surface treatment nor the type of composite resulted in significantly greater bond strengths. All of the composite-resin repaired ceramic/polymer groups were significantly weaker in bond strength than the cohesive strength of the monolithic block material.

Disclaimer

The views expressed in this study are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government. The authors do not have any financial interest in the companies whose materials are discussed in this article.

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