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Bond angle effects on microtensile bonds: Laboratory and FEA comparison

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Summary *Objective*. To test the hypothesis that there is a reduction in bond strength when a microtensile load is applied to adhesive junctions prepared at 10, 20 and 30 degrees to the usual perpendicular interface. To evaluate the effect of bond angle and adhesive layer thickness on stress levels within the adhesive joint utilizing FEA.

Methods. Twenty-four non-carious third molars were selected, occlusal enamel removed and polished perpendicular to the long axis of the tooth. The Clearfil SE Bond and Single Bond were applied on the dentin. A 4 mm resin restoration, Z 100, was built up. The teeth were sectioned at 10°, 20° and 30° to the bonding interface ($n=3$). The control ($n=3$) group had all cuts parallel to the tooth longitudinal axis (0° bond angle). The bond values were calculated in MPa and Two-Way ANOVA and Tukey test applied. FEA was performed (1 mm/side square specimens) to obtain the maximum principal stress (MPS) in the microtensile-model for each bond angle and for varying adhesive thickness from 20 μm to 200 μm for each group.

Results. The bond strength results diminish as the angle on the interface increased ($P<0.05$) for Clearfil SE Bond between 0 (control) and 30 degrees, and for Single Bond between 0 (control) and 10, 20, and 30 degrees. The hypothesis can be fully accepted for Single Bond and partially accepted for Clearfil SE Bond. For the FEA, there was a trend toward decreasing MPS as the bond angle increased, while the MPS for each angled group increased with adhesive layer thickness.

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Significance. The MPS results for angled interfaces, exhibited the same trend as the lab values. FEA results indicated an MPS increase with increased adhesive thickness.

Introduction

The science and art of adhesive dentistry has not yet been fully realized. Frequently additional tissue (hard and sometimes soft) must be removed to place and retain restorative biomaterials. Therefore, a durable interfacial adhesion between tooth and biomaterial is essential [1].

The achievement of the bond between adhesive resin and dentin depends on penetration of the primer and adhesive resin into the conditioned dentine surface. This is necessary in order to create micromechanical interlocking between the dentine collagen, and resin, and to form a hybrid layer or resin-dentin interdiffusion zone [13]. Although proper bonding of resins to dentine has proved to be difficult, ongoing advances are improving the reliability and predictability of dentinal adhesion [21,19].

Adhesion analyses of dentin bonding agents have been performed by numerous mechanical testing methods and the enormous number of test variables involved, the variable nature of the heterogeneous dentin, and no real agreement on test standardization, complicate the results.

A microtensile method of bond strength testing was developed by Sano et al. [14] The essence of the microtensile method is the division of resin-bonded teeth into slabs between 0.5 and 1.0 mm thick, which are trimmed in such a manner that tensile force will be concentrated on the bond interface during testing. One advantage of this technique is that the bond interface of a small (ca 1 mm^2) specimen has better stress distribution during loading, so there are fewer cohesive failures than are found with more conventional testing of dentin samples [11,12].

The microtensile test for adhesive bond strength has normally been performed over flat dentin surfaces [11,12,14]. Nevertheless, clinical procedures involving tooth preparation do not produce a flat superficial surface. Therefore, when adhesive agents are applied to tooth preparations, different adhesive bond strength can occur within the same preparation [24]. Ciucchi [5], in 1997, was the first to use a microtensile method on different regions of restored MOD preparations, but the dentin surface to microtensile bond strength testing was flat.

The microtensile bond test would be the most applicable method to test bonds to prepared cavities on sound, as well as caries affected or infected dentin. However, cavity preparation and removal of caries result in a surface that is no longer flat and the variable geometry and surface irregularities, produced by the burs, could be partially responsible for the lower bond values recorded. Studies comparing various bonding agents and bond enhancement procedures to sound, caries affected, and infected dentin will become increasingly important in the future. The role of adhesive bond angle and thickness on microtensile bond strengths should be investigated.

The combination of diverse materials and complex geometry makes stress distribution analysis in teeth very complicated. Simulation in a computerized model would allow for a study of simultaneous interaction of the many variables involved [2].

The ability to achieve a strong and durable bond between the restorative material and tooth structure is of paramount importance for the clinical success of many dental restorations. A fracture toughness test should therefore be an appropriate method for characterization of the intrinsic fracture resistance, and presumably, the in-service reliability of the dentin-composite resin interface [20]. The fracture toughness and the perpetuation of crack growth are, however, dependent on material thickness. As the adhesive layer becomes thicker, it undergoes Poisson contraction from the outer sides of the beam. This contraction may relieve some of the stresses that would otherwise give rise to fracture.

Some difficulties associated with laboratory test methods (fabrication of small specimens, the introduction of the sharp crack, creation of exact and repeatable cement thicknesses, etc.) led the researchers to try a numerical solution. Therefore, the finite element method was applied to fracture mechanisms and was found to give satisfactory results when compared with those obtained experimentally [20].

Finite element analysis (FEA) of stress distribution has also been used to study the sensitivity of bond strengths to specimen design, and changes in testing conditions. Those studies [2,3] show that there is a need for a more critical approach on the design of appropriate tests for evaluating the bond strength of resin composite and ceramic, if the desire for a standardized test procedure is to be achieved. For this objective to be accomplished, a careful examination of bond strength tests is mandatory for correct interpretation of the bond strength data [22].

Therefore, this study was conducted to seek information about the adhesive bond strength on dentin, testing the research hypothesis that there is a reduction in adhesive bond strength when a tensile stress was applied at 10, 20 and 30 degrees to the adhesive junction compared to a non-angled (flat) interface, using two different adhesive systems. A FEA was performed to understand two aspects: first, to identify the stress distribution pattern at different adhesive joint angles; and second to quantify the effect of increasing adhesive thickness at a given joint angle on the stress distribution, represented by the Maximum Principal Stress (MPS). The MPS is associated with the maximum tension stress, and permits comparison of the FEA predictions to the laboratory data.

Materials and methods

Twenty-four recently extracted non-carious third molars were selected and sterilized using 180 Krad of Gamma irradiation [23], under a protocol approved by the NYU College of Medicine Institutional Review Board.

The occlusal enamel of these teeth (Fig. 1A) was removed perpendicular to the long axis of the teeth using a diamond saw (Buehler Isomet low speed saw with Buehler Diamond Wafering Blade-Series 20 HC Diamond, No 11-4215, Buehler, USA) to expose a flat dentin surface (Fig. 1B). The flat surfaces were then polished using a 600-grit silicon paper (Buehler, Phoenix Beta polisher and grinder).

The specimens were randomly divided into two groups (twelve teeth per group) for adhesive application, one total-etching sytem (Single Bond 3M-ESPE) and the other a self-etching system (Clearfil SE Bond Kuraray).

For the Single Bond specimens, before the application of the adhesive agent, 35% phosphoric acid (Scotchbond, 3M-ESPE, Co) was applied for 15 s on the flat dentin surface, and then rinsed for 10 s with water. The excess water was blotted using an absorbent paper, leaving the dentin surface moist.

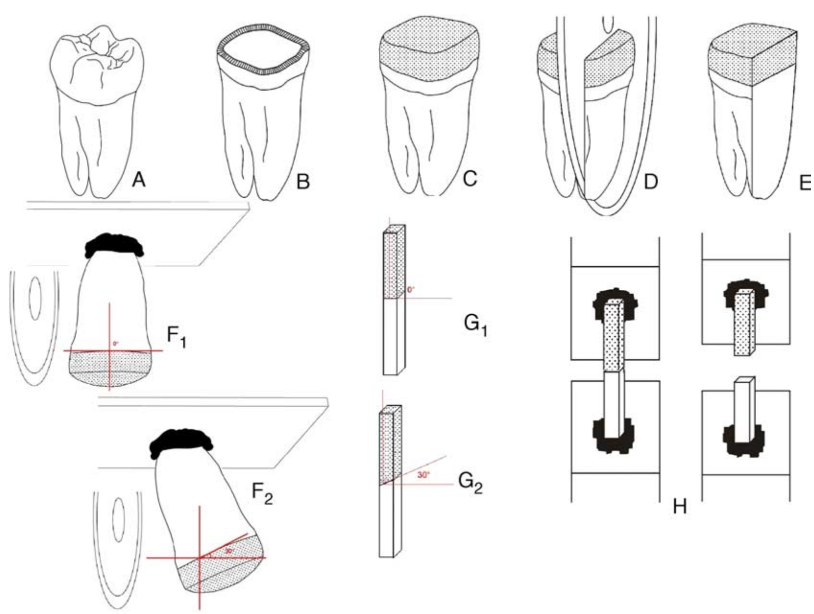

Figure 1 This sequence of pictures shows the MTBS of the cutting procedure. (A) Picture of the tooth; (B) Picture of the flat dentin substrate produced after polishing; (C) Picture of the adhesive and resin applied on the flattened substrate; (D) and (E) Picture of the cutting procedure forming a flat surface at the mesial side of the tooth; (F_1) Picture of the specimen secured to a mold for cutting procedure perpendicular (0 degree) to the interface from buccal to lingual side; (F_2) Picture of the specimen secured to a mold for cutting procedure at 30 degrees to the interface from buccal to lingual side; (G_1) Picture of the specimen cut perpendicular to the interface. Note the interface at 0 degree (flat) represented by the red lines; (G_2) Picture of the specimen cut at 30 degrees to the interface (red lines); (H) Picture of the specimen fixed on the Bencor apparatus for the tensile testing.

Table 1 This table shows the informations on items used in the study.

Material	Composition	Lot/No	Exp.	Company
Single Bond (3M-ESPE, Co)	Bis-GMA; Polyalkenoic acid co-polymer; Dimethacrylates; HEMA; Photoinitiator; Ethanol; Water	3411 2GM	2003-11	3M-St. Paul MN 55144, USA
Scotchbond Etchant (3M-ESPE, Co)	35% Phosphoric Acid; Gel	7523 2XY	2003-11	3M-St. Paul MN 55144, USA
Clearfil SE Bond Primer (Kuraray, Co)	10-Methacryloyloxydecyl dihydrogen phophate (MDP); 2-Hydroxyethyl methacrylate (HEMA); Hydrophilic dimethacrylate; dl-Camphorquinone; N, N-Diethanol-p-toluidine; Water	61243 272	2004-03	Kuraray-200 Park Ave. New York, NY 10166, USA
Clearfil SE Bond (Kuraray, Co)	10-Methacryloyloxydecyl dihydrogen phophate (MDP); Bis-phenol A diglycidylmethacrylate (HEMA); Hydrophobic dimethacrylate; dl-Camphorquinone; N, N-Diethanol-p-toluidine; Silanated colloidal silica	61243 327	2004-03	Kuraray-200 Park Ave. New York, NY 10166, USA
Z100 Composite (3M-ESPE, Co)	BIS-GMA, TEGDMA resins	20020924	2005-03	3M-St. Paul MN 55144, USA
Silicon Paper (Buehler)	600-grit	No: 305118600100	-	Buehler-41 Waukegan Rd. Lake Bluff. IL 60044, USA
Curing Light 2500	-	Serial # 3016930	-	3M-3350 Granada Ave. N Oakdale, MN 55128, USA

Two consecutive coats of Single Bond adhesive (3M-ESPE, Co) were immediately applied, and then genltly dried for 5 s each, avoiding an excess of adhesive agent. Subsequently, the adhered surface was light-cured (Curing Light 2500, 3M-Espe Co) for 10 s.

For the Clearfil SE Bond specimens, the Primer was applied for 20 s on the flat dentin surface, and then dried with light airflow. Subsequently, the bonding agent was applied, and then gently dried with airflow. The bonded surface was then light-cured (Curing Light 2500, 3M-Espe Co) for 10 s.

Following the adhesive procedures, a 4 mm resin restoration was built up on the top of the flat dentin surface using Z100 composite (3M-ESPE Co) in increments of not more than 1.5 mm for each layer, and each layer then light-cured (Curing Light 2500 3M-Espe Co) for 40 s (Fig. 1C). Subsequently, all specimens were stored for a period of at least 7 days in water at 37 °C. Table 1 presents all the materials used in this study.

After aging, the adhered specimens were divided into six groups (three teeth per group) for cutting procedures and the microtensile testing was performed in different directions defined by angles as follows:

Group SB 10. For this group the Single Bond (3M-ESPE, Co) specimens were cut in a 10 degree angle to the long axes of the teeth.
Group SB 20. For this group the Single Bond (3M-ESPE, Co) specimens were cut in a 20 degree angle to the long axes of the teeth.
Group SB 30. For this group the Single Bond (3M-ESPE, Co) specimens were cut in a 30 degree angle to the long axes of the teeth.
Group SE 10. For this group the Clearfil SE Bond (Kuraray, Co) specimens were cut in a 10 degree angle to the long axes of the teeth.
Group SE 20. For this group the Clearfil SE Bond (Kuraray, Co) specimens were cut in a 20 degree angle to the long axes of the teeth.
Group SE 30. For this group the Clearfil SE Bond (Kuraray, Co) specimens were cut in a 30 degree angle to the long axes of the teeth.

In the control groups (one for each adhesive system, three teeth per group) the samples were cut parallel to the long axis of the teeth, and this was considered as a 0 degree angle.

The cutting procedure was performed in '*x*' and '*y*' directions through the resin with a slow-speed diamond saw (Buehler Isomet low speed saw; Buehler Diamond Wafering Blade-Series 20 HC Diamond, No 11-4215, Buehler, USA) under copious water.

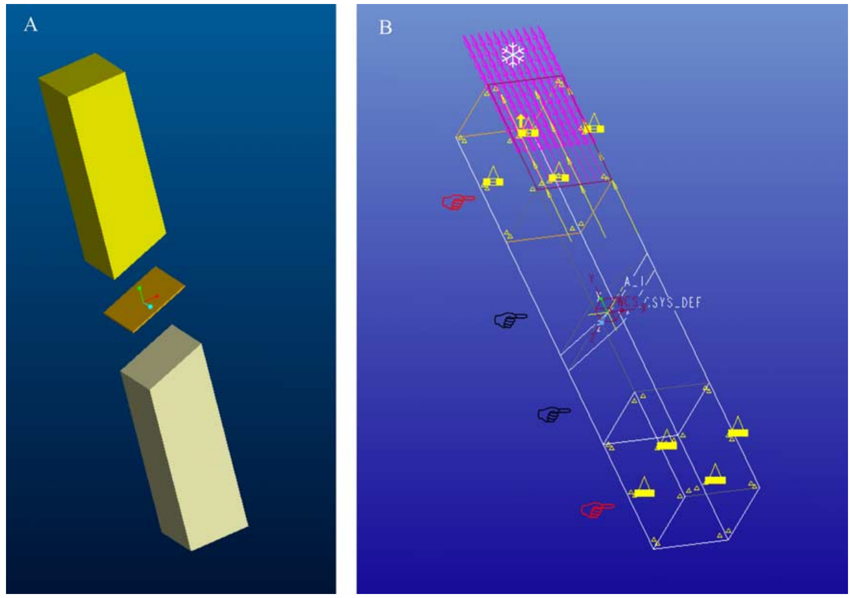

Figure 2 (A) This image shows the specimen modeled for FEA, in which the yellow color represents the resin composite, the brown represents the adhesive layer and the white represents the dentin; (B) This image shows the constrained surfaces (red pointers), the 1.5 mm unconstrained areas (black pointer) and the load direction applied on the resin component that corresponds to the metal plate of the Bencor apparatus (white asterisk).

The cutting procedure in the '*y*' direction was performed perpendicular to the adhesive junction line. The cutting procedure in the '*x*' was performed by first cutting the mesial side to a flat surface (Fig. 1D and E). The tooth was then secured to a wax mold and a protractor was used to draw a line showing the cutting angle at 10, 20 or 30 degrees to the center line. The tooth was positioned at an angle matched to the drawing, parallel to the cutting blade (Fig. 1F_1 and F_2) and then sectioned producing beams in square shape. All beams were cut so the junction line would be at a 10, 20 or 30 degree angle (Fig. 1G_1 and G_2).

Subsequent to the cutting and measuring procedures, each beam was mounted using a cyanoacrylate cement (Krazy Glue Gel, Advanced Formula-ELMER'S Products, Inc.) with the angled-surface of each specimen faced to the microtensile apparatus (Bencor Multi-T; Distributor: Danville Engineering Inc.-USA) and loaded under tension at a given crosshead speed (1.0 mm/min) using a 50 N load cell (Turbo Gauge DFA10 Amtek Chatillon) connected to a universal testing machine (Model TSD 500, Chatillon-Ametek, Agawam, MA, USA) (Fig. 1H).

The beams were then measured along both the *y*-axis and *x*-axis with a digital caliper (Mitutoyo, Japan) to determine the cross-sectional area. The Pythagorean Theorem (information obtained from www.math.com) for sine and cosine was performed for angled specimens to obtain the measurement from the angled sides. The values obtained from the angled face were then multiplied to the value from non-angled face to obtain the final cross-sectional area of each specimen.

The modes of tensile failure were later examined using a Stereomicroscope (Olympus SZX-ILLB100-Olympus Optical Co, Ltd, Japan) and ranked as cohesive in the composite, adhesive, cohesive in dentin and/or mixed.

The adhesive bond strengths were determined dividing force at failure by the cross-sectional area of each specimen and the data converted to MPa and analyzed using Two-Way ANOVA. Multiple comparisons between the groups were made by Tukey test. Statistical significance was defined as $P<0.05$.

As a complementary method to performing in vitro evaluation of microtensile bond strength analysis in different angulations and also in different adhesive thicknesses, an effort was made to establish a geometrical model on

Table 2 This table presents the material properties considered for FEA. Those informations were obtained via www.umch.edu.

Components	μ (Poisson's coeficient)	E (Elasticity-GPa)	Density (g/cm^3)
Z 100 (3M, Co)	0.24	18	2.2
Adhesive	0.42	2	1.2
Dentin	0.31	16	2.14

Table 3 This table shows the microtensile bond strength values (MPa) with change in bond angle, mean, standard deviation, and number of specimens (Mean (SD) (N)). Statistical difference was defined as $P<0.05$.

Group	0 Degree (Control)	10 degrees	20 degrees	30 degrees
Single Bond (3M-ESPE, Co)	44.2 ± 15.8 (61)I	26.7 ± 12.7 (26)II	35.4 ± 14.7 (42)II	32.8 ± 11.3 (26)II
$P<0.05$	yes	yes	yes	yes
Clearfil SE Bond (Kuraray, Co)	55.9 ± 21.1 (39)a	52.6 ± 18.7 (26)ab	47.3 ± 20.7 (37)ab	40.2 ± 16.0 (36)b

computer. The software tool used for FEA in this study was Pro Mechanica (Parametric Technology Co, Waltham, MA, USA) and the geometrical model was generated using Pro/Engineer Wildfire (Parametric Technology Co, Waltham, MA, USA), which is integrated into Pro/Mechanica software (Fig. 2A).

The modeled specimen was assumed with approximately 6 mm in total height and 1 mm^2 in area. The intermediate zone was determined as the adhesive layer and the constraints were defined over four surfaces on each component (dentin and resin) simulating the glue that fixes the sample on the Bencor apparatus (Bencor Multi-T; Distributor: Danville Engineering Inc.-USA) during the microtensile testing (Fig. 2B). 1.5 mm unconstrained area for both dentin and composite was left at their interface area. The fixed portions were outside of this unconstrained area. The constraints on composite component were fixed at four degrees of freedom (free in the Y axis rotation and translation). The model assumed linear-elastic materials behavior and employed isotropic quadrilateral elements-with high element density in the cement and its interfaces. The program automatically generates and meshes elements and tests for convergence. Perfect adhesion between the adhesive and composite resin and adhesive and dentin were assumed. The direction of the applied tensile force was perpendicular to the top surface with magnitude of 10 N. Table 2 shows the material properties used to perform the FEA.

Results

The Single Bond groups, the 0 degree-flat surface (control group) showed the highest bond strength value (44.2 ± 15.8) compared with 10 (26.7 ± 12.7), 20 (35.4 ± 14.7) and 30 degree (32.8 ± 11.3) groups ($P<0.05$). No statistical difference was found between 10, 20 and 30 degrees for Single Bond (Table 3).

For the Clearfil SE Bond, the 0 degree (control group) showed higher bond strength value (55.9 ± 21.1) than 30 degree group (40.2 ± 16.0) at level of significance. No statistical difference was found between 10, 20 and 30 degree groups (52.6 ± 18.7, 47.3 ± 20.7 and 40.2 ± 16.0 respectively) for Clearfil SE Bond system.

In all instances, the Clearfil SE Bond control, 10, 20 and 30 degree groups showed higher bond strength values ($P<0.05$) than their respective Single Bond groups.

For both adhesives groups, the modes of failures of all tested specimens were ranked by percentage. For Single Bond, 77.41% of all tested samples had mixed failure, 6.45% failure in the resin composite, 5.80% cohesive failure in the dentin and 10.32% adhesive failure. For Clearfil SE Bond, 71.03% of all tested samples had mixed failure, 17.39% cohesive failure in the resin composite, 9.42% cohesive failure in the dentin and 2.17% adhesive failure. For this group, 61.50% of the cohesive failures in dentin and 50% of the cohesive failures in resin were observed on the 30 degrees specimens.

The MPS for each angled adhesive layer and adhesive layer thickness is presented in Table 4. The results indicate a trend to decrease in the MPS whenever the angle on the interface increases. However, the MPS for each angled group presents a tendency to increase whenever the thickness of the adhesive layer increases.

Evaluating difference stress maps are a valid way to rapidly depict a specimen behavior at different joint angles and adhesives thickness. The maps on Fig. 3 show that the MPS was always located at the margin of the resin-adhesives interfaces. The tension in the adhesive layer started from the margin (it always started at the same side as the applied load) to the center of the layer. The thickest adhesive layer corresponded to the highest MPS value for all angled adhesive layers.

Table 4 This chart shows the FEA results of Maximum Principal Stress (MPS) analysis for different bond angles and thicknesses of the adhesive.

Thickness (μm)	*Max Principal Stress* (MPa)			
	0°	10°	20°	30°
20	14.7	15.1	15.2	14.4
50	15.5	15.4	15.5	14.6
100	19.1	17.9	17.8	16.5
200	22.4	21.6	20.9	18.8

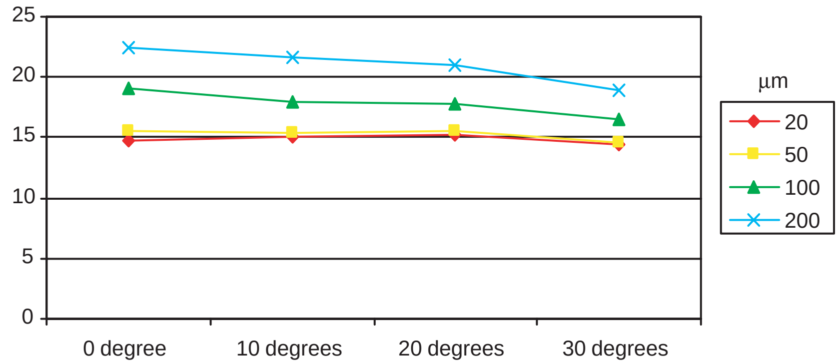

Figure 3 This Graph depicts the MPS for each angled adhesive layer and adhesive layer thickness (µm). There was a trend to decrease the MPS whenever the angle on the interface increases.

Discussion

Since the introduction of the microtensile method by Sano, H. and co-workers [14], many adhesive bond strength studies have been done using this methodology with a flat dentin surface [1,4,10,12,14,15,17,18,25]. The results from flat dentin surfaces sometimes do not mimic what occurs in clinical situations, where little is actually flat. Thus, these prior results require careful analysis when comparing with what happens *in vivo*. This study uses several angled specimens to elucidate what occurs in small portions of actual tooth preparations.

When the clinicians are faced with carious tissue and restorative treatment (Prosthodontics, Operative Dentistry, etc), the cavity preparation

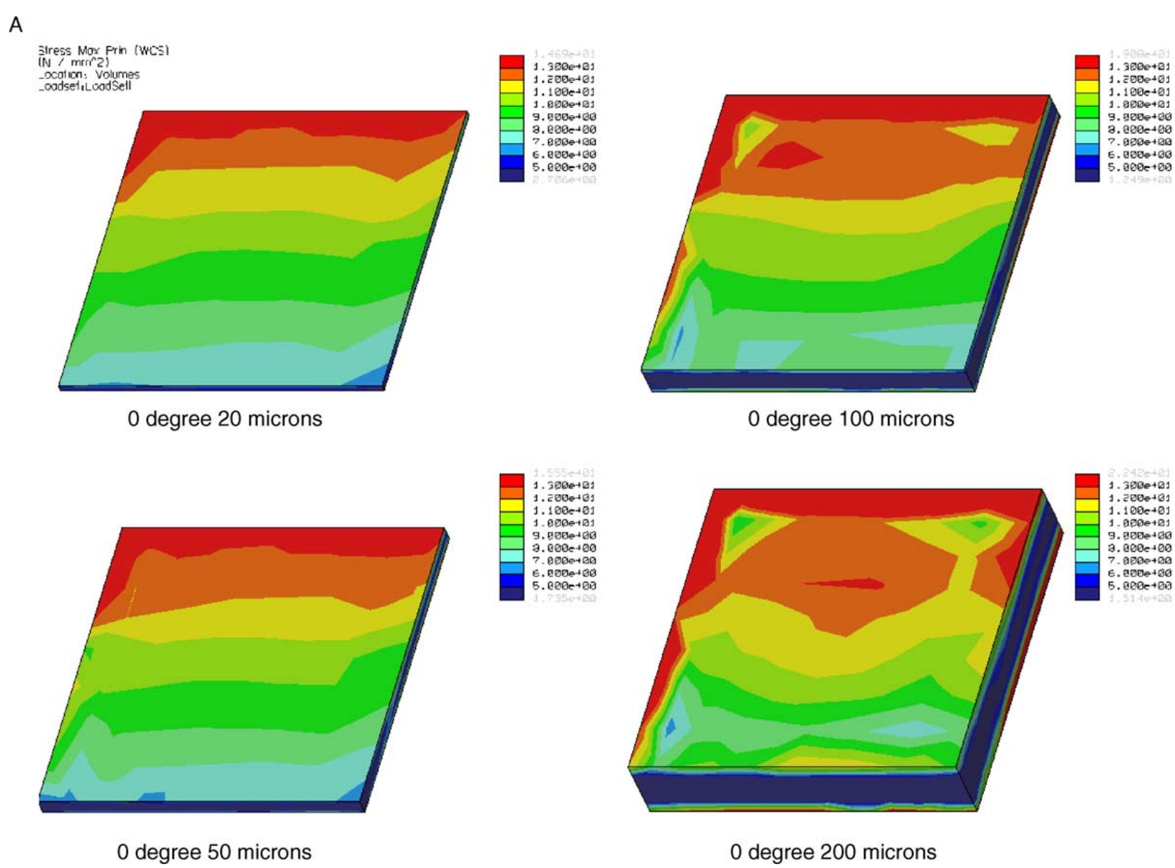

Figure 4. (A-D) are MPS sequences at flat (0 degree), 10, 20 and 30 degree angulations and different thicknesses. The highest stress always started at the same side of the applied load (red color in the scale) and moved to the center of the layer as the thicknesses increased (follow the scale). The near edge is oriented with this specimen faced against the simulated alignment plates of the tensile test apparatus.

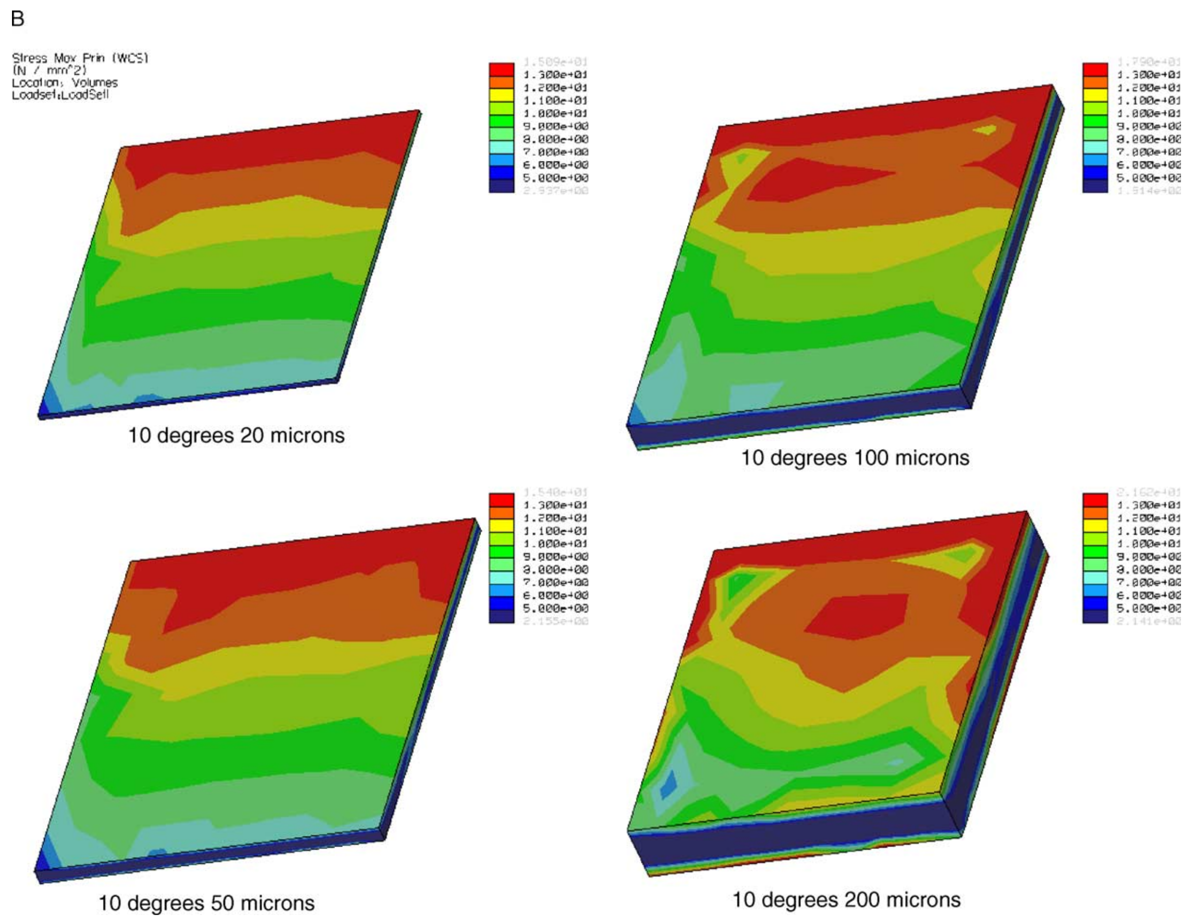

Figure 4 (*continued*)

produces both curved and irregular dentin surfaces. This can probably create both lower bond strengths and more variability on the results.

Studies using the microtensile bond strength method after carious removal by either polymer bur or carbide bur have been performed [16]. In comparison to other dentin bonding studies [1,12, 14,25], the specimens prepared either by the polymer or carbide bur do not have the same perpendicular interfaces as do the usual microtensile bond specimens. In this case the dentin surface becomes irregular with the caries removal process. The lower bond strength data observed from carbide and polymer bur groups might be due to the angulated adhesive junction surface produced after carious removal.

The results from the microtensile testing demonstrated a reduction in the bond strength values with increasing bond line angle. This might be due to two aspects: (1) The higher angulations presented higher cross-sectional areas, thus there is a tendency to incorporate more flaws (bubbles, etc), resulting in lower bond strength values [8]; (2) As the samples have different angles onto the joint interface, the off-axis load applied produces a bending moment, consequently reducing the bond strength values.

Zheng et al. [26] in 2001 discussed that although the Bencor Multi-T apparatus is intended to apply pure tensile force, the alignment of each specimen on the long axis of the testing device may not always be exactly parallel to the long axis of the testing device. This could produce a bending stress distribution within the bonded interface that is perpendicular to bond angle during tensile loading. The authors also observed that the thicker adhesive layer may permit 'self-alignment' of the specimen that corrects for minimal deviations in specimen placement, thereby, improving stress distributions during testing, and yielding higher apparent bond strength.

The FEA model also indicates that bonding (with the low elastic modulus cyanoacrylate adhesive) at only three sides and the end of each portion of the specimen results in a non-uniform stress distribution in the cement layer. This and the potential for off-axis alignment of specimens (as noted above) are limitations of our microtensile test.

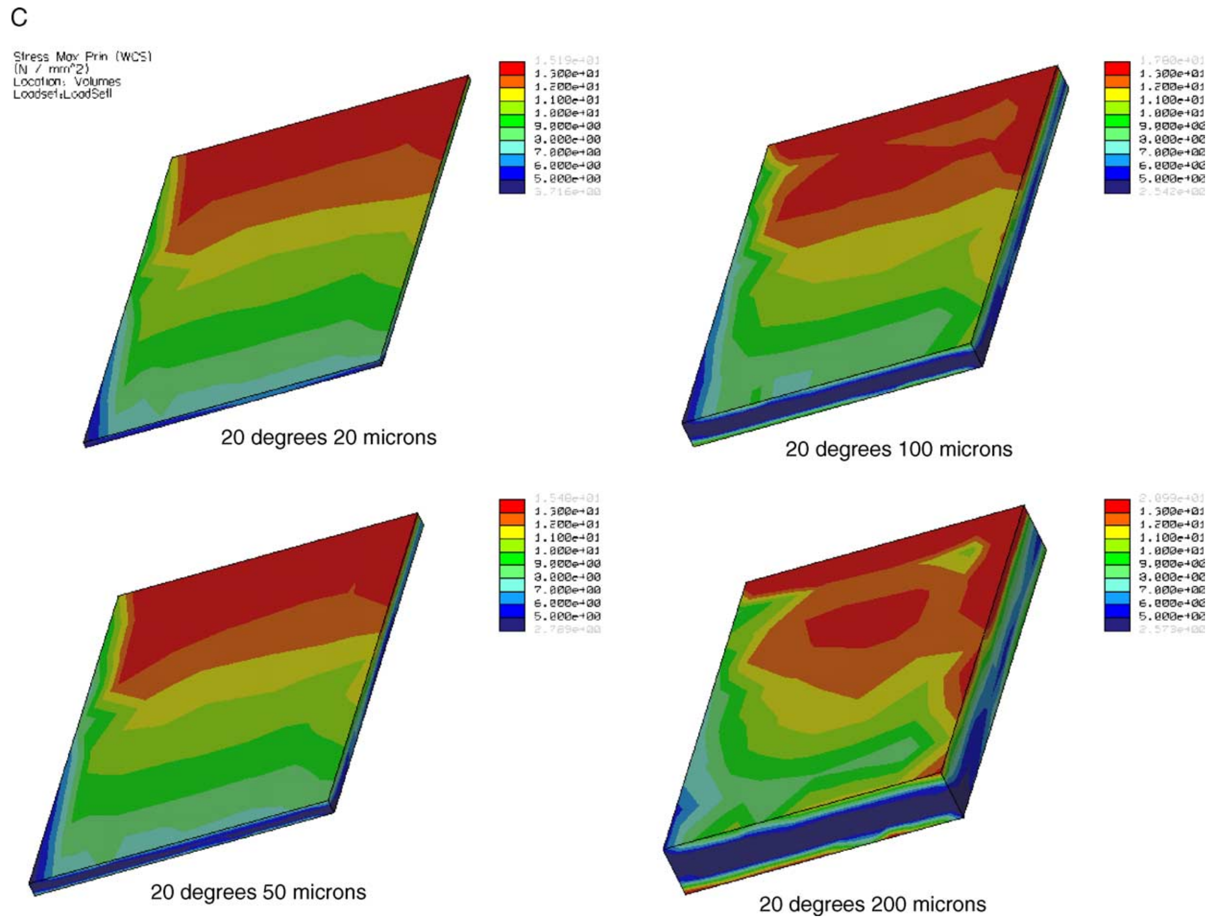

Figure 4 (*continued*)

Proper understanding of the physical phenomena occurring in the adhesively restored tooth during the placement and setting of the materials and throughout tooth functioning is crucial in the determination of the proper placement procedures and material selection [6]. The 3D-FEA is the preferred way to obtain an optimal realistic analysis, and undoubtedly represents a more detailed way to obtain useful mechanical information on the stress distribution at the dentin-composite adhesive interface [3]. Nevertheless, FEA models require experimental validation. This validation must be purposely prepared and definitive [2,9], as we have attempted in our study. Results from FEA that have not been laboratory validated should be viewed with great reserve [9], especially in the case of dentin in which the anisotropy due to the dentinal tubule can hardly be modeled as an isotropic material.

In our 3D FEA results we can observe that the MPS diminished when the angle of the adhesive interface increased from 0 (control) to 30 degrees. The MPS is a consequence of the direction of the applied force. When there are angled interfaces, there are different force components in different directions resulting from the applied load, leading to decreases in the MPS values. The same tendency was observed in the laboratory test results, except for the 10 degree Single Bond group.

In order to obtain information about the adhesive behavior for different adhesive layer thicknesses, MPS was also computed (Fig. 4) with varying adhesive layer thickness on each angled layer. The FEA value indicates that the higher the adhesive thickness, the higher the elastic performance. This is a result of the Poisson contraction from the outer edges of the adhesive, toward the center of the specimen. The greater the thickness of the cement, the more the plastic zone is able to contract. This contraction results in stress relief, and therefore, a higher tolerance of load before a crack results. If the cement layer is thinner, it is closer to the diameter of the specimen. The closeness of the higher stiffness materials prevents the contraction from proceeding as much, and does not allow for as much stress relief. Very recent findings suggest that

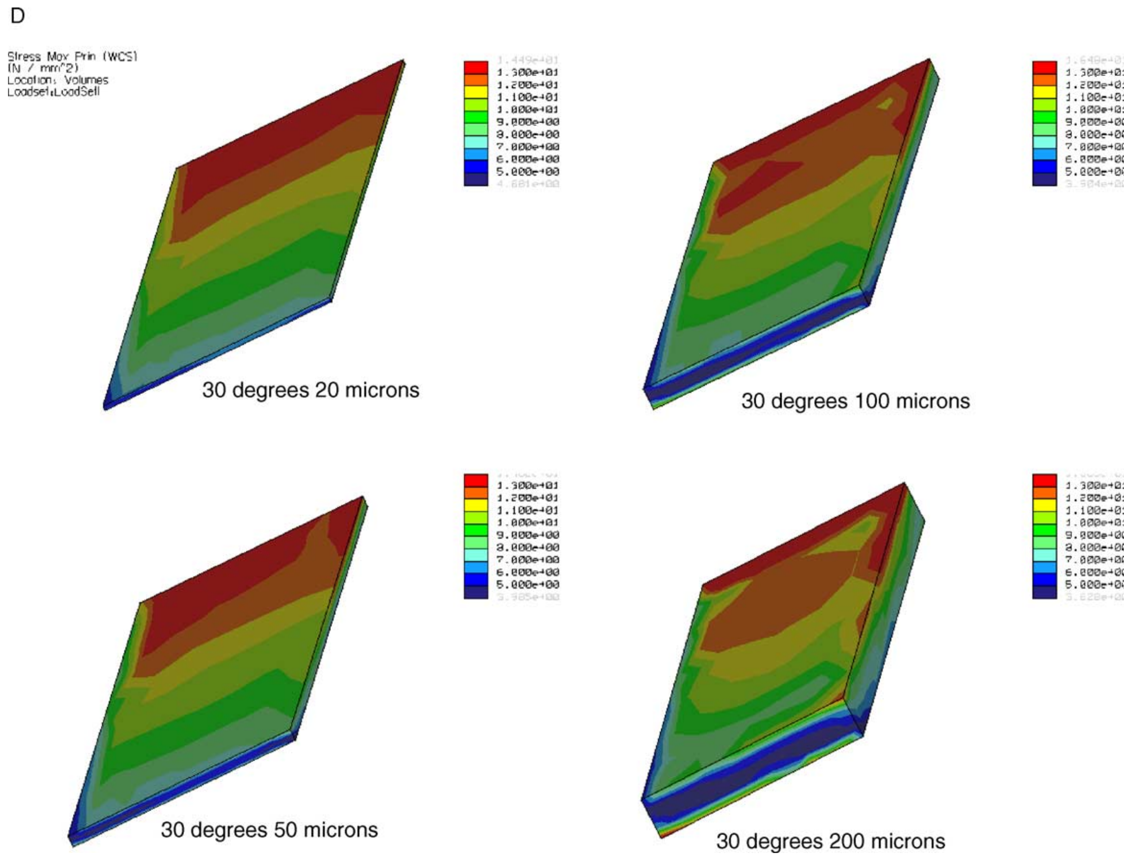

Figure 4 (*continued*)

the functional elastic modulus of resin cements increases with decreasing cement thickness [7]. This effect, if operational in the lower modulus of dental bonding agents utilized here, may be another factor influencing the results.

This study investigated the questions of how angled interfaces present in tooth preparations (but not previously studied), might be simulated in the commonly used Microtensile testing method in order to produce more clinically relevant results. Additionally, we wished to elucidate the value of FEA simulation of laboratory conditions in order to look at the interplay between multiple variables. Microtensile bond strength for different angulations and associated with different adhesive thicknesses are now required to compare to the finite element data. It is also important to complete tests with the angled tests specimens oriented in the test apparatus (Bencor) with the long axis oriented 90 degrees to that employed here, and to simulate the same condition using FEA, in order to see what effect this 'compliance' variable has on the results. Further analyses correlating density and elasticity of the dentin substrate, hybrid layer in model specimen and cavity preparation are also necessary.

Conclusion

The research hypothesis, that increasing bond angle results in reduced microtensile bond strength, can be fully accepted for Single Bond and partially accepted for Clearfil SE Bond. The derived MPS (from FEA) presented the same trends as the lab values. The maximum principal stress exhibited an increase when the adhesive layer thickness increased.

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