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Effect of aging and testing method on bond strength of CAD/CAM fiber-reinforced composite to dentin

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ABSTRACT

Objectives. To evaluate and compare the outcomes of shear (S) and microtensile (μT) bond strength tests of CAD/CAM fiber-reinforced composite (FRC) to dentin. Aging with either fatigue or thermocycling were conducted for comparison with baseline microtensile group.

Methods. CAD/CAM FRC (Trinia, Bicon LLC, Boston, USA) blocks were milled to 3-mm diameter cylinders for shear and to blocks (5 × 5 × 5 mm) for μT. Sixty extracted human molars were flattened to obtain dentin surfaces and randomly divided in four groups (n = 15): (1) SC: samples tested in shear 24 h after bonding; (2) μTC: samples tested in μT 24 h after bonding); (3) μTF: samples submitted to mechanical fatigue prior to μT test, and; (4) μTT: thermocycling prior to μT test. Bonding system was applied onto the FRC material (Cera–Resin Bond, CRB, Shofu Dental, Kyoto, Japan). A conventional three-step adhesive system (All-bond 3, Bisco, Schaumburg, USA) was use with a self-cure resin cement (C&B resin cement, Bisco, Schaumburg, USA). Bond strength tests were conducted at 0.75 mm/min and data analyzed using Weibull distribution ($p < 0.05$).

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Results. Weibull contour plots showed a significantly lower characteristic strength (η) and Weibull modulus (m) for SC (η = 6.9 MPa and m = 1.4) compared to µTC (η = 20.9 MPa and m = 4.5). Fatigued and thermocycled µT groups presented significantly reduced characteristic strength (η = 3.1 MPa and η = 4.1 MPa, respectively) compared to µTC. Weibull modulus was significantly reduced only for SC and µTF groups compared µTC. Failure predominantly occurred at the cement/FRC interface.

Significance. FRC bonded to dentin samples presented lower Weibull modulus and characteristic bond strength when immediately tested in shear compared to microtensile. Aging through thermocycling or mechanical fatigue significantly reduced the characteristic strength in microtensile testing, with the majority of failures emerging between restoration material and cement interface.

1. Introduction

Over the years, clinicians have relied upon laboratory evaluations to choose which adhesive systems to use in their daily practice. Although the relationship between bond strength tests and clinical performance of dental adhesives remains questionable [1,2], recent evidence shows that clinical results can, to some extent, be estimated based upon laboratory results [3–5]. Moreover, mechanical testing of bonded interfaces has provided some valuable information to identify substrate variables [6,7] and to define guidelines for application procedures [8].

The most commonly used tests for the determination of enamel and dentin bond strength are shear and tensile bond strength tests [9–12]. Shear and tensile bond strength tests are performed exclusively in specimens with relatively large bonded areas, usually from 7 to 28 mm^2. However, expressing bond strength in terms of nominal (i.e., average) stress has been questioned due to the heterogeneity of the stress distribution at the bonded interface [10,11,13–15]. Moreover, cohesive failure of both the composite and the dental substrate is a common occurrence that may compromise an accurate assessment of the interfacial bond strength [16]. These limitations led to the use of specimens with smaller bonding areas, below 2 mm^2, with the so-called microtensile and micro-shear tests [11,17,18].

A meta-analytical review of factors involved in dentin adhesion shows that many parameters significantly influence dentin bond strength [9]. Parameters related to the test design and experimental conditions (type of composite and the bonding area, storage condition of the bonded samples, the testing mode and the crosshead speed) could easily be controlled by the use of a standardized protocol, such as that recommended by International Organization for Standardization (ISO) TS 11405, 2003 [19] for adhesion to dental tissues. Unfortunately, and substrate-related variables are more difficult to control, even though their influence is consistent.

The ISO technical specification ISO/TS 11405 [19], reflects this tendency, describing with greater detail the characteristics and preparation of the tooth substrate for the bonding procedure. However, some aspects such as bonding area, testing assemblies or loading conditions are less clear. As a result, a wide variety of experimental protocols exist among researchers, with impact on the outcomes [20,21] eventually hindering sound comparisons.

The stress at failure between the adhesive and the substrate is reported in these tests as the load at failure divided by the cross-sectional area of the bonded surface, which will be referred to as the nominal bond strength [12]. However, studies of lap joints using mathematical stress analysis [22,23], finite element stress analysis or experimentation demonstrate that the stresses at the interface between the adhesive and the substrate are anything but uniform, and are highly dependent on the test geometry and loading configuration adopted [10,11,13], in addition to high frequency of premature failures and large standard deviation values [11,24].

Therefore, the aims of this present study were: (1) to evaluate the bond strength of CAD/CAM fiber-reinforced composite (FRC) to dentin by shear and microtensile tests; (2) to evaluate the isolated effects of mechanical fatigue and thermocycling on microtensile bond strength values. The null hypotheses investigated in this study were: (1) there is no statistical difference between the shear and microtensile tests conducted 24 h after bonding, when using the same materials and methods of manufacture and; (2) Microtensile bond strength values between CAD/CAM FRC and dentin would not be affected by mechanical fatigue or thermocycling compared to microtensile baseline samples.

2. Methods

This study was approved by the Ethic Committee for Human Studies from Bauru School of Dentistry at University of São Paulo, Brazil, (Process # 48748415.8.0000.5417). Factors under study were: 1- bond strength testing method (shear and microtensile) and condition (immediate-control, mechanical fatigue, and thermocycling). The latter two conditions were considered only for microtensile test. The bond strength between CAD/CAM fiber-reinforced-composite (FRC) and human dentin was analyzed according to shear (S) and microtensile (µT) test, described in ISO TS 11405, 2003 [19].

Sixty sound and freshly extracted human 3rd molars, up to 07 days of extraction and age range from 16 to 40 years were used. Upon extraction teeth were cleaned under water and remaining tissue and/or dental calculus were removed. After cleaning the teeth were stored in distilled water according to ISO TS 3696 at 4 °C until preparation of the specimens. The specimens were randomly divided (https://www.randomizer.org) in four groups as: (1) SC (control specimens tested in shear test 24 h after bonded, n = 15); (2) μTC (control specimens tested in microtensile test 24 h after bonded, n = 15); (3) μTF (samples subjected to mechanical fatigue and microtensile test, n = 15) and; (4) μTT (specimens submitted to thermocycling and microtensile test n = 15).

2.1. Shear bond strength test

Fifteen teeth were included with the buccal surface exposed to a PVC matrix and gypsum in order to maintain moisture. The buccal surface was ground with 600-gri silicon carbide abrasive paper under running water to exposure the superficial dentin.

CAD/CAM fiber-reinforced composite blocks (Trinia, Bicon LLC, Boston, MA, USA) were milled to 3 mm (±0,1 mm) diameter cylinders and 4 mm height. The surface of the FRC was polished to 600-grit silicon carbide paper and sandblasted with 45 μm aluminum oxide particles (Al_2O_3) for 10 s at a standoff distance of 10 mm and 2 bar of pressure, to achieve a micro-retentive topography. The samples were cleaned in ultrasonic bath for 10 min. FRC cylinders were cleaned with 37% phosphoric acid for 30 s and running water for 60 s. A bonding system (Cera–Resin Bond, CRB, Shofu Dental, Kyoto, Japan) was applied on FRC surface according to manufacturer's recommendation: one thin coat of CRB1 was applied using a disposable microbrush and left undisturbed for 10 s, followed by application of the one thin coat of CRB2 that was left undisturbed for another 10 s, followed by light curing for 20 s.

A tape with a 3 mm diameter hole was placed on the dentin surface to standardize the cementation area. The dentin surface was slightly dried and conditioned with 37% phosphoric acid for 15 s. Afterwards, the acid was removed with air/water spray (30 s) and excess water removed with absorbent paper.

A dual-cure adhesive system (All-bond 3, Bisco, Schaumburg, USA) and a self-cure resin cement (C&B resin cement, Bisco, Schaumburg, USA) were used according to manufacturer's instructions. A drop of liquid A and B were mixed (10 s) and a thin layer applied by rubbing on the dentin surface for 5 s, gently air dried from 5 cm for 5 s and 10 s of light curing at 1380 mW/cm2 power density by a dental light curing unit (Bluephase 2.0i; Ivoclar Vivadent; Liechstein, Germany). After this step a thin layer of the All Bond 3 Resin was applied and light cured for 10 s. The self-cure resin cement was mixed, placed on the FRC surface and carefully positioned within the tape hole of the prepared dentin. Immediately, a static load of 1 kgF was applied with a cementation device for 8 min. After polymerization the tape was carefully removed and samples stored in distilled water (±37° C) for 24 h until the shear test.

The shear test was performed immediately after removal of the specimen from the water on a universal testing machine (EMIC, São Paulo, SP, Brazil) with a shear device. A stainless steel wire of 0.7 mm diameter was placed on the adhesive interface between tooth and resin exerting force until break at crosshead speed 0.75 (±0,3) mm/min.

After test, each cylinder had the diameter measured and registered with a digital caliper (Mitutoyo, Tokyo, Japan). The area was obtained by the formula:

$$A = \prod r^2$$

where $\prod$ is a constant of 3.14 and r is the radius of the cylinder (mm).

2.2. Microtensile bond strength test

Forty-five teeth were ground on occlusal surface with 600-grit silicon carbide abrasive paper under running water to expose the superficial dentin and provide a regular, smooth and flat surface. CAD/CAM FRC (Trinia, Bicon LLC, Boston, MA, USA) blocks were milled in blocks of 5 × 5 × 5 mm. The surface treatment and bonding procedures were performed as described for shear bond strength test.

After bonding, the resin/cement/dentin blocks were attached with wax to an aluminum base that was fixed to a precision saw cutting machine (Isomet Low Speed Saw; Buehler Ltd., Lake Bluff/IL – USA). Sectioning of samples for bond strength testing was performed under copious water irrigation with a wafering blade (IsoMet Wafering Blades 15LC series, Buehler Ltd., Lake Bluff, IL, USA). Sectioning was always initiated at the tooth towards the resin, with a speed of 100 rpm and weight of 250 g. Serial sections were performed to obtain slices of 1 mm width. The samples holder were removed from the sawing machine, air-dried, and a drop of liquefied sticky wax was injected in between the spaces created during sectioning to stabilize the slices. Afterwards, the sample holder was rotated 90° to conduct the second sequence of cuts at 100 rpm and weight of 150 g.

Approximately 25 sticks with a cross-sectional area of 1 mm^2 were expected to be obtained from each block. The sticks at the periphery, where there was the presence of enamel, were discarded. The sticks were individually analyzed for the presence of defects, such as voids and gaps. The sticks obtained from blocks of the control group were stored in vials with distilled water (37 °C) for 24 h until the microtensile bond strength test (μTC).

Prior to testing, each specimen had the cross-sectional area measured and registered with a digital caliper (Mitutoyo, Tokyo, Japan). Microtensile bond strength test was conducted using a Bencor device fixed in a universal testing machine (EMIC, São Paulo, SP, Brazil). Specimens were fixed parallel to the long axis of the device using cyanoacrylate glue (Super Bonder – Flex Gel, Hentzel Ltda, Itapevi, SP, Brazil). They were loaded in tension to failure at a crosshead speed of 0,75 mm/min.

2.3. Mechanical fatigue test

The specimens of the group μTF (n = 15) were subjected to 500.000 cycles of mechanical fatigue (Model MSFM, Elquip, São Carlos, SP, Brazil) in R-ratio mode at 37 °C and 100% humidity. A load of 115 N was applied with a 3.2 mm stainless-steel ball-shaped stylus at the center of the composite surface, at a frequency of 1 Hz. After fatigue, the blocks were sliced to

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obtain the sticks as previously described and microtensile test was performed.

2.4. Thermocycling test

The specimens of the group μTT (n = 15) were subjected to thermocycling process (Model MSFM, Elquip, São Carlos, SP, Brazil) for 500 cycles between 5 °C and 55 °C in deionized water with a dwell time of 20 s and transfer time of 10 s. After this test, beam specimens were produced and microtensile test was performed.

2.5. Data analysis

Shear and Microtensile bond strength values were recorded for each specimen in MPa using a formula:

$$\mu = P/A$$

where P is the load at the moment of failure (N) and A is the bonding area of the specimen (mm^2).

In some cases specimens could not be tested, due to early failure during positioning of the specimen in the device. For these specimens the zero value bond strength value measured were assumed [25–27].

2.6. Fractographic analysis

The fracture surfaces of the specimens were examined with a stereo-microscope (Leica Zeiss MZE, Mannheim, Germany) and scanning electron microscope (SEM; XL 30 CP; Philips, Eindhoven, Netherlands). Fractographic analysis was performed in all samples to determine the failure origin. Failures were classified as adhesive when occurred only at the FRC/cement or dentin/cement interface; cohesive when occurred only in resin cement, FRC or dentin; and mixed when the failure involved more than two surfaces.

2.7. Statistical analysis

The bond strength values were subjected to Weibull 2-parameters analysis (Synthesis 9, Weibull ++; Reliasoft, Tucson, AZ, USA). The probability of failure as a function of bond strength was plotted to show data scatter and a contour plot with Weibull modulus (*m*) vs. characteristic strength (Eta, reported in MPa where 62.3% of the specimens will be failed, with 95% confidence interval) used to determine differences between groups. Non-overlap of contour plots is accepted as indicating a significant difference between groups [28,29].

3. Results

Some specimens of the microtensile groups (μTC n = 34 (9%), μTT n = 307 (81%), μTF n = 255 (68%) could not tested because they failed prematurely. So, zero bond strength value was assumed for these specimens. In shear bond strength all specimens were tested.

Data from Weibull 2-parameter contour plot (95% confidence intervals) (Fig. 1) shows the characteristic bond strength and Weibull modulus between SC and μTC groups tested immediately. The difference at 95% level is detected if contour overlap between groups does not exist (in such case, samples will be considered to be from different populations) [28–30]. The lowest characteristic strength (η) and Weibull modulus (*m*) was observed for the SC (η = 6.9 MPa and *m* = 1.4), and values were significantly lower than for μTC (η = 20.9 MPa and *m* = 4.5). Fig. 2 depicts a contour plot where the same parameters are compared for all groups tested in microtensile (μTC, μTF and μTT). The fatigued μTF (η = 4.1 MPa and *m* = 1.5) and thermocycled μTT (η = 3.1 MPa and *m* = 2.2) groups were not significantly different between each other for either characteristic strength or Weibull modulus, but both presented characteristic strength significantly lower than μTC (η = 20.9 MPa). Weibull modulus was significantly lower for μTF compared to μTC, but not different between each other (μTF and μTT) (Table 1).

Stereomicroscopy and SEM analysis showed that, for shear specimens, mixed and adhesive failure between FRC and cement were the most common failure mode. On the other hand mixed failures were more predominant than adhesive between FRC and cement in specimens tested in microtensile. Few specimens had adhesive failures between dentin and cement (Table 2). No cohesive failures were observed.

In specimens subjected to shear, SEM micrographs showed the presence of voids on the resin cement interface. Fractographic analysis shows the presence of hackles, wake-hackles and twist-hackles that are very useful for determining the direction of crack propagation and fracture origin [31]. Fractographic marks indicated that bonded interface fractures started at the wired loop contact away from the bonded interface (Figs. 3 and 4).

Stereomicroscope and SEM micrographs of samples subjected to microtensile also showed the presence of voids at the resin cement fractured interface and surface. Most fractures were mixed, followed by adhesive between the FRC and resin cement. The presences of hackles suggest that the tensile strength was in the direction of the long axis of the tooth near to the bonded interface (Figs. 5 and 6).

4. Discussion

The present study evaluated the shear and microtensile bond strength of a fiber-reinforced composite cemented on a dentin substrate with self-cure resin cement. Although both testing methods are recommended by ISO 11405 [19], Weibull analysis showed significance differences in characteristic bond strength values and Weibull modulus. As previously reported in the literature [12,17,32], the microtensile test presented higher bond strength and lower data variability than shear test, which led us to reject the first postulated null hypothesis.

When a resin composite bonded to a flat dentin surface is loaded in tension or shear, the distribution of stresses along the interface is highly non-uniform [10]. The stress distribution is related to features such as the geometry and size of the adherent and their relative elastic modulus. The nominal strength (i.e. the load per unit area of bonded surface) is controlled by the attainment of a critical stress locally at the most sensitive place, usually the edge, of the bonded area [10]. Thus, the flaws present at a bonded interface may act as stress rais-

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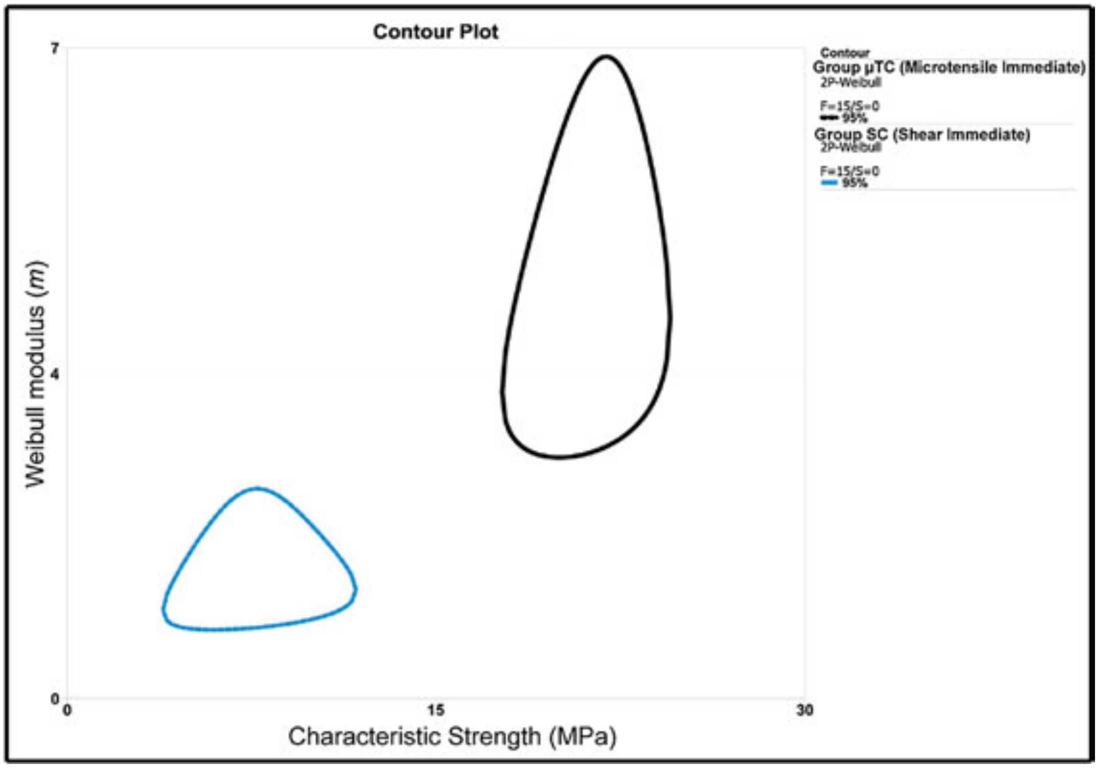

Fig. 1 – Contour plots using 95% confidence bounds for the relationship between Weibull modulus and Characteristic strength for SC and μTC groups tested immediately, in which 62.3% of the specimens will be failed. Non-overlap between contours indicates a significant difference between groups.

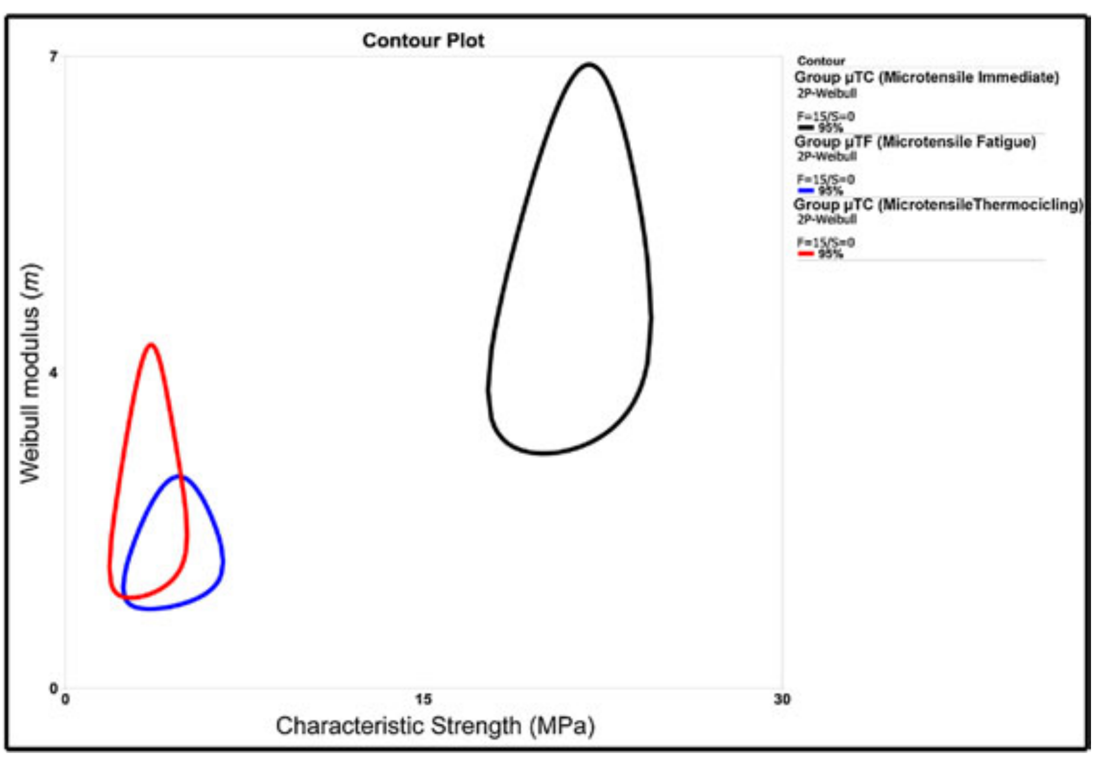

Fig. 2 – Contour plots using 95% confidence bounds for the relationship between Weibull modulus and Characteristic strength for all groups tested in microtensile (μTC, μTF and μTT), in which 62.3% of the specimens will be failed. Non-overlap between contours indicates a significant difference between groups.

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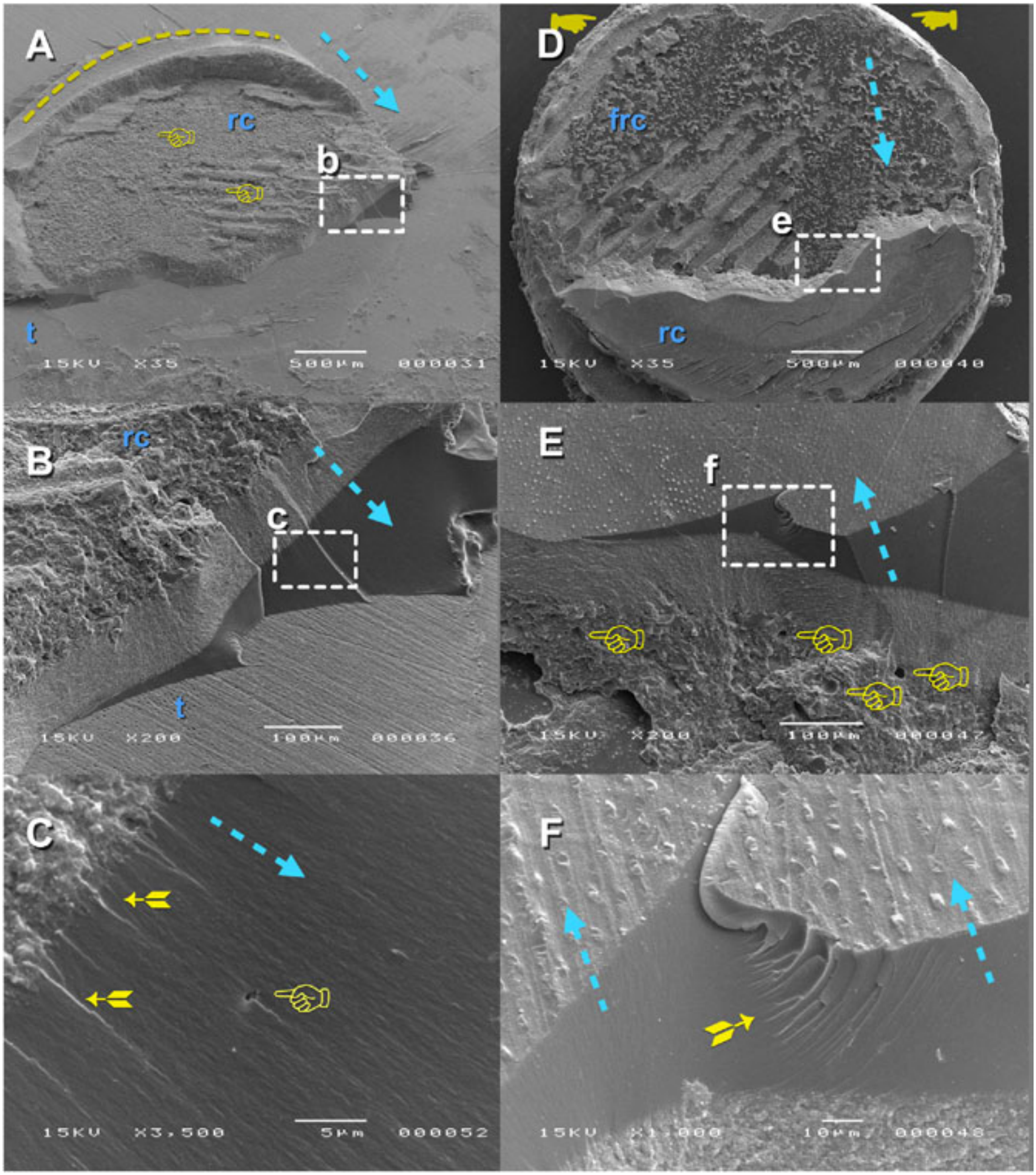

Fig. 3 – SEM micrograph of a mixed failure mode on sample subjected to shear test. (A) Overview of failure on tooth (t) side with resin cement (rc) remnants adhered to dentin. Some porosities (pointers) on the resin cement interface are already visible on low magnification (×35). Wired loop contact location is illustrated by the dotted line and the dotted arrow shows the direction of the fracture. (B) Magnification of (b) showing the propagation of the fracture through resin cement, adhesive and exposed dentin (t). (C) Magnification of (c) showing the presence of Hackles (arrow) which are lines that appears on fracture surface running in the local direction of cracking, separating parallel, but noncoplanar and a Wake-Hackle (pointer) which is a type of Hackle extending from a pore at the crack front in the direction of cracking, when an advancing crack encounters a pore, it is split in two planes and create a step or "tail" between them. (D) Overview of FRC (frc) side with some adhered resin cement. The pointers show wired loop contact and dotted arrow shows the direction of the fracture leading to sample debond. (E) Magnification of (e) where the resin cement fractured, the presence of pores (pointers) in cement.

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Table 1 – Characteristic strength and Weibull modulus of groups with 95% upper and lower confidence limits.

	SC	μTC	μTF	μTT
Upper	10.1	23.5	5.9	4.3
Characteristic strength (MPa)	6.9 B	20.9 A	4.1 B	3.1 B
Lower	4.7	18.6	2.9	2.2
Upper	2.1	6.7	2.2	3.6
Weibull modulus (*m*)	1.4 B	4.5 A	1.5 B	2.2 A,B
Lower	1	3.1	1	1.3

Different uppercase letters indicate significant differences among columns ($p < 0.05$), in the same row. Comparison of characteristic strength between SC and μTC denote statistical difference (nonoverlap between upper and lower confidence limits). Groups μTF and μTT were not significantly different, but both presented significantly lower characteristic strength than μTC. Weibull modulus was significantly higher when μTC was compared to SC. Only fatigue (μTF) significantly reduced the Weibull modulus under μT testing, whereas thermocycling did not.
SC = control specimens tested in shear test; μTC = control specimens tested in microtensile test; μTF = samples subjected to mechanical fatigue and microtensile test; μTT = specimens submitted to thermocycling and microtensile test.

Table 2 – Failure mode in shear and microtensile groups (%).

Groups	FM		
	Mixed	Adhesive (FRC/cement)	Adhesive (dentin/cement)
SC	46	40	13.3
μTC	49.8	45.8	4.3
μTF	53.2	38	8.6
μTT	52.5	32.5	15.0

FM = Failure mode; FRC = fiber-reinforced composite; SC = control specimens tested in shear test; μTC = control specimens tested in microtensile test; μTF = samples subjected to mechanical fatigue and microtensile test; μTT = specimens submitted to thermocycling and microtensile test.

ers with stresses concentrated near the edge of each flaw [33]. An increase in the bonded area may result in an increase in the number of flaws leading to a decrease in bond strength [16,34]. In the present study, bonded area was 7 mm^2 for shear test, and 1 mm^2 for microtensile and SEM micrographs confirmed a larger number of voids within resin cement interface of shear relative to microtensile specimens. Considering that it is the largest flaw, and not the average flaw, that controls the lifetime of a restoration and that there exists great variability in strength-controlling flaws at adhesive interfaces it is very likely that bond-strength data will commonly not fit the Gaussian distribution [30,35,36]. Therefore, a Weibull distribution can be used to predict level of stresses required to interface failure [37].

The Weibull modulus reflects the variability in results, where a higher scatter in data is represented by a lower Weibull modulus (e.g. SC group). Indeed, although normally distributed data may be treated by parametric tests, care should be taken when treating non-normally distributed data to report bond-strength data [36]. The results of Shear Bond Strength test showed a greater scatter the data that reflects on a lower Weibull modulus. Thus, a less homogeneous dataset was found for this group and, strategically, only Microtensile bond strength samples were subjected to aging.

In the present study, SEM fractographic analysis of most samples tested in shear showed that the fractures started at the loading area close to the interface between FRC and resin cement. These findings suggest that the fracture may have started duo to high stress concentration close to the loading area instead of at the cement/dentin interface itself. Indeed, finite element analysis findings indicate the development of non-uniform stress distributions in shear bond test [10,13,38].

On the other hand, microtensile bond strength test provides specimens with cementing surface perpendicular to the tensile force and more homogeneous stress across the interface. Therefore, maximum principal stress values could be much closer to the nominal strength [11–13]. Furthermore, in case of the microtensile bond test, the loading force passes through the tooth substrate and resin composite before reaching the adhesive interface, with subsequent stress concentration in these materials [38,39]. Comparisons between microtensile and shear tests showed that microtensile bond test appears to be more accurate in detecting differences between dental adhesives [38].

Thermocycling and mechanical fatigue tests are mostly used to simulate the clinical environment [36,40,41]. Thermocycling is a combination of thermal stresses and hydrolytic degradation and is a method to simulate temperature

(F) Magnification of (f) showing a series of Twist-Hackles (arrow) which is a Hackle that separates portions of the crack surface, each of which has rotated from the original crack plane in response to a lateral rotation in the axis of principal tension. It shows the local direction of crack propagation (dotted arrow) and that the fracture line had a sudden change of direction, which suggests the non-uniform nature of tensile stress. The presence of these fractographic elements suggests that failure started on wired loop contact area, disrupted the adhesion between FRC and resin cement, extended to fracture the cement layer and to eventually debond the resin cement and dentin interface.

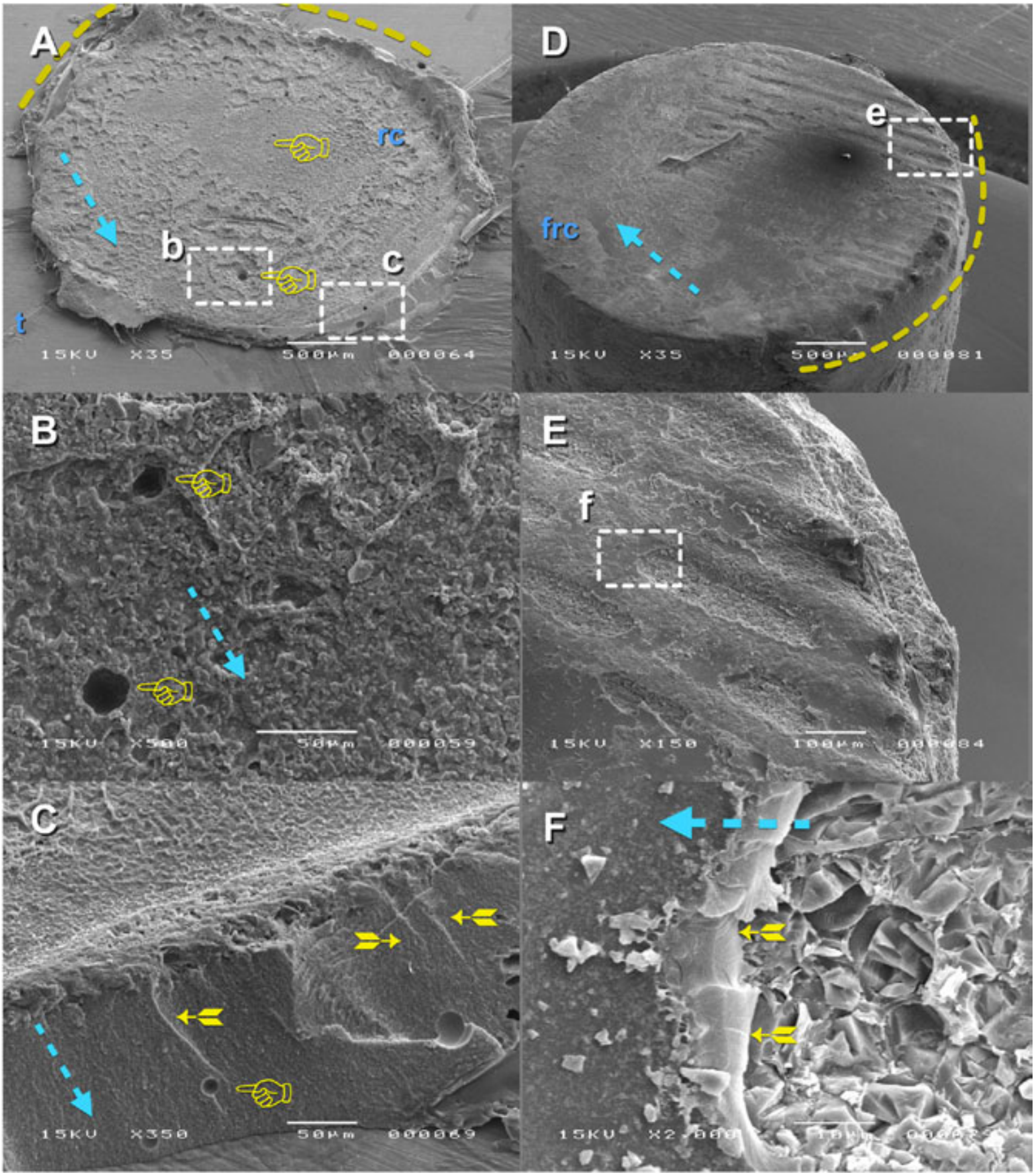

Fig. 4 – SEM image of adhesive failure at the resin cement and fiber-reinforced resin interface on specimen tested in shear. (A) Overview of failure on tooth (t) side where resin cement (rc) remained adhered to dentin. Porosities (pointers) can be observed on resin cement surface (A, B, C). Wired loop contact is illustrated by the dotted curved line and the dotted arrow shows the direction of the interfacial fracture. (B) Magnification of (b) showing the presence of pores on cement surface that was in contact with FRC. (C) Magnification of (c), the presence of Hackles (arrows) and Wake-Hackles (pointer) showing the trajectory of the failure (dotted arrow). (D) An Overview of FRC (frc) side without any resin cement adhered. Dotted curved line illustrates the wired loop contact and dotted arrow points the direction of propagation of the interfacial failure. (E) Magnification of (e) and (F) magnification of (f) showing the presence of Hackles (arrows). The observed fractographic marks suggest that failure started on wired loop contact area and propagated until adhesive interface failure between FRC and resin cement.

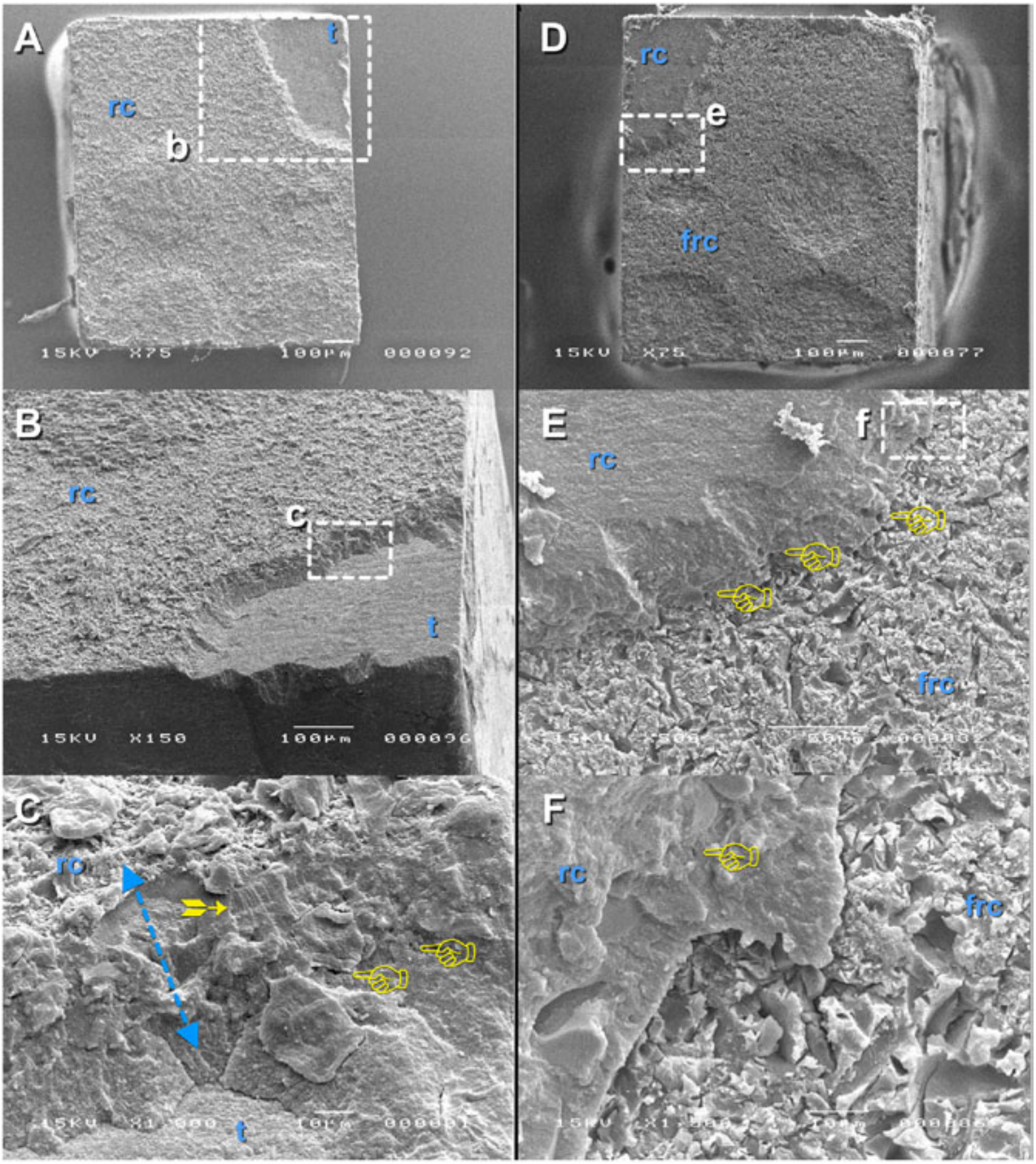

Fig. 5 – SEM micrograph of a mixed failure mode of a microtensile sample. (A) Overview of failure exposing tooth substrate (t) and the resin cement (rc) adhered to dentin. (B) Magnification of (b) shows the region where there was exposure of dentin (t) and the interfacial fracture of the resin cement. (C) Magnification of (c) depicts the presence of hackles (arrow) and voids (pointers) at the resin cement interface. (D) Overview of FRC (frc) side with some resin cement (rc) remnant adhered. (E) Magnification of (e) where the interfacial fracture shifted from the dentin/cement interface towards the FRC/resin cement interface. Some voids (pointers) are observed within the cement. (F) Magnification of (f) shows the interface between FRC and resin cement where the pointer shows a void. The presence of hackles in resin cement at the interfacial fracture region suggests that the direction of fracture (dotted arrow in image C) was in the long axis of the specimen, perpendicular to the tensile force.

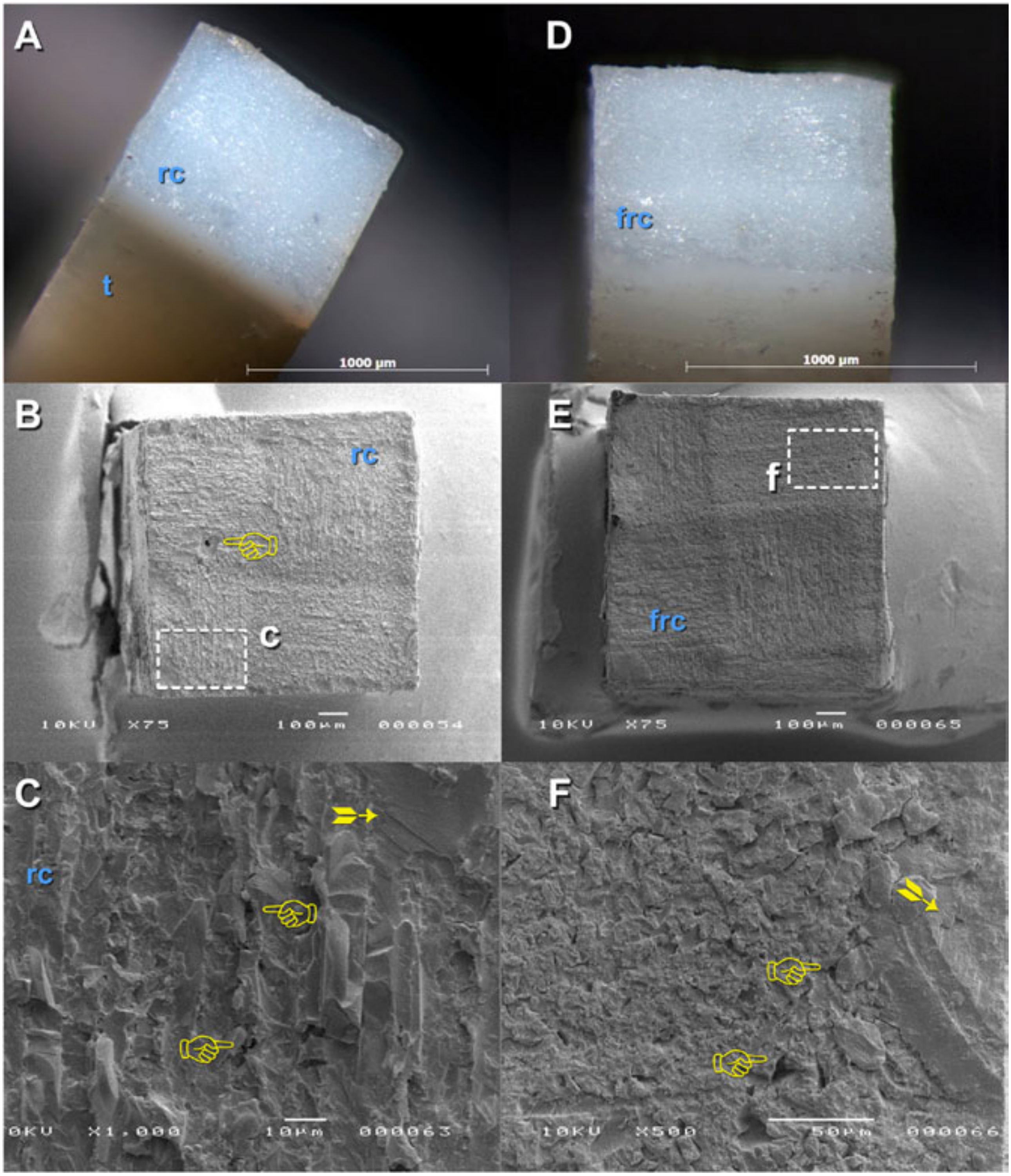

Fig. 6 – Stereomicroscopy and SEM micrographs of adhesive failure at the resin cement and fiber-reinforced resin interface of microtensile sample. (A) Stereomicroscope and (B) SEM overviews of failure on tooth (t) side with resin cement (rc) adhered to dentin. (B) shows a void at the resin cement interface. (C) Magnification of (c) showing the presence of Hackles (arrow) and voids (pointers) at the resin cement interface. (D) Stereomicroscope and (E) SEM micrograph of FRC (frc) side without resin cement (rc) adhered. (F) Magnification of (f) showing the roughness of sandblasted FRC. Some hackles (arrow) at adhesive remnant and voids (pointers) were also observed.

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related breakdown by repeated sudden temperature changes. Mechanical fatigue was used to simulate the exposure of tooth-FRC bond to cyclic subcritical loadings produced during chewing. [40]. Both simulations have caused deterioration of the bond strength as shown previously [42], and as observed in our results, leading us to reject the second null hypothesis.

A previous study showed that the aging induced by thermocycling leads to both contraction and expansion stresses at the tooth/restoration interface due to the different thermal coefficients of the restoration and the tooth structure [40]. Moreover, thermocycling accelerates the chemical degradation of the adhesive interface. Whereas the fatigue test used in this study did not result in damage of the FRC blocks, it did decrease the microtensile bond strength likely due to degradation of bonding interface through flaw size increase caused by fatigue.

Fractographic analysis of samples tested in shear depicted a predominant fracture initiation near the contact area of the wired loop and propagation through cement and adhesive until failure. The presence and location of wake-hackles and twist-hackles indicated that the shear stresses were commonly not concentrated at the bonded interface in a uniform way, and that a bending component of the bonded assembly was likely present during the test.

In terms of method and study design, it is important to clarify the choice of tooth surfaces for the bond procedures for each test. Although the ISO 11405 standard recommends the preparation of buccal surfaces of third molars, preparing the buccal surface for microtensile bond strength tests resulted in inappropriate samples, i.e., dentin portions of the sample were too short and could not be fixed to the movable portion of the test device. Thus, occlusal surface was chosen for adhesive procedures for microtensile test samples and buccal surfaces for shear as recommended by ISO 11405. This could be considered as a drawback of the present study. However, as evidenced by our fractographic analysis, the majority of failures were between the FRC and the cement interface for either testing methods, suggesting that regardless of tooth surface, the bond strength to dentin outperformed the bond strength to FRC.

Regarding ISO 11405 standard, it is important to mention that a new version of this standard was released in 2015. Important guidelines, that are aligned with most of the current literature on the field [25,27,41,43] have been included. Although the present study was first designed based on the previous standard version (2003), care was taken initially regarding sample preparation an sample size and afterwards to adequate statistical treatment of results to currently suggested standards [27].

It is still debated whether pre-test failures should be excluded, included as zero values, or included as greater than zero values when performing the statistical analysis of microtensile data [25,44]. In this study, premature failures were entered in statistical calculations, as their occurrence exclusively in microtensile testing was thought worthy of consideration in the comparison with the shear method. Pre-test failures were entered in calculations by assigning them the lowest value measured in the respective group, following previous recommendations [25].

The tested CAD/CAM fiber-reinforced composite presented a significant decrease on bond strength after aging, regardless of the aging method used, which is expected for mechanical or thermal cycled interfaces. Fractographic analysis was a key tool to enlighten the mechanisms that leads to failure since the majority of them involved the fiber-reinforced composite/cement interface. Although bond strength values decreased after aging, the clinical relevance of these findings warrants future clinical trials on FRC prostheses bonded to teeth, considering that 5-year clinical results did not report FRC/resin cement debonding issues in implant-supported long span reconstructions [45].

5. Conclusion

The results of this study showed that FRC bonded to dentin samples tested in shear compared to microtensile resulted in significantly lower Weibull modulus and characteristic bond strength values when tested immediately. Aging through thermocycling or mechanical fatigue reduced the characteristic bond strength of samples tested in microtensile, relative to baseline with the majority of failures emerging between restoration material and cement interface.

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