



## Micro-shear Bond Strength of Repair System to Different Hybrid Ceramic Materials Using Different Surface Treatment Protocols (In-Vitro study).

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**Objectives:** The purpose of this in-Vitro study was to evaluate the effect of different surface treatments on micro-shear bond strength of different hybrid ceramic materials.

**Material and methods:** Three different CAD/CAM blocks (Vita Enamic, Cerasmart and Nacera hybrid) were cut into 2mm thickness rectangles and divided into 4 groups according to the surface treatment: Control, grinding with diamond stone, airborne abrasion with Al<sub>2</sub>O<sub>3</sub> particles and 9.5% Hydrofluoric acid etching. After surface treatment the surface roughness was measured and scanned with AFM. Silane coupling agent was applied on the treated ceramic surfaces followed by 2 adhesives (monobond and heliobond), Composite micro-tubes were fabricated on the ceramic rectangles. A shear force was exerted to each composite micro-tube at cross head speed of 0.5mm/min until failure occurred and the load of failure was recorded in MPa. Data were collected, tabulated statistically analyzed.

**Results:** Regarding the surface treatments methods, surface roughness scanning and micro-shear bond strength test showed a significant difference between grinding, air abrasion and HF etching groups. Regarding the ceramic materials, surface roughness scanning and micro-shear bond strength test showed a significant difference between Vita Enamic, Cerasmart and Nacera hybrid groups.

**Conclusions:** Different surface treatment methods improved the bond strength of composite resin to different hybrid ceramic material. Airborne abrasion gave superior surface roughness and bond strength among all other surface treatment methods used, irrespective to the ceramic material.

**Keywords:** Repair, Hybrid ceramics, Micro-shear bond strength, surface treatment.

### I. INTRODUCTION

In recent years, ceramics have become popular restorative materials because of their biocompatibility, color stability and their highly esthetic appearance, therefore dental ceramics are usually used for replacement of missing teeth, restoration of damaged teeth and enhancing the natural dentition esthetics.<sup>(1)</sup> Hybrid ceramics consist of a ceramic network which is fully integrated by a polymer network with different proportions varying according to the ceramic type.<sup>(2)</sup> These ceramics were developed with the aim of imitating the natural teeth structure, they have mechanical properties near to those of dental structure as hardness and modulus of elasticity, therefore decrease the wear of the opposite dentition if compared to other types of ceramics.<sup>(3, 4)</sup> However all-ceramic restorations clinically are susceptible to chipping and fracture.<sup>(5)</sup> There is no need to replace a chipped restoration, due to many factors: the cost of replacement, more trauma to the natural dentition and further loss of tooth structure.<sup>(6)</sup> Intraoral repair is considered a minimally invasive procedure which includes adding of a restorative material, may be with a pre-treatment of the chipped restoration.<sup>(7)</sup>

Ceramic surface treatment of the chipped restoration can be done in the procedure of intraoral repair, it includes mechanical, chemical or combination treatments to produce surface irregularities, so improving the adhesion to the ceramic restoration.<sup>(8)</sup> Silane can be used with ceramics to increase the bond strength between the ceramic surface and restorative material, as it may improve the surface energy for the application of adhesives.<sup>(1)</sup>

Micro-shear test has been presented to determine the adhesive bond strength to small areas of substrate.<sup>(9)</sup> This leads to decrease in; surface preparation, amount of adhesive coverage and



specimens number. Also, specimens preparation for the micro-shear bond strength test is easier, several specimens can be prepared without specimen trimming after the bonding process. Micro-shear test is becoming frequently used to permit more uniform distribution of the loading stresses.<sup>(10)</sup> The hypothesis of this study is that different types of surface treatments will affect the micro-shear bond strength of composite resin to CAD/CAM polymer-infiltrated hybrid materials.

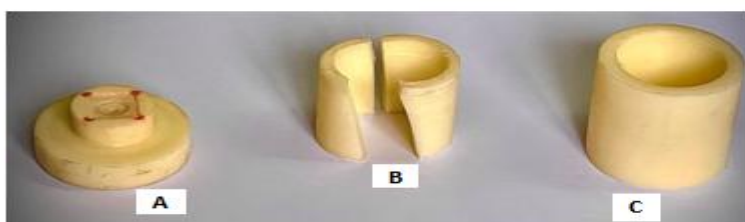
## II. MATERIALS AND METHODS

Three types of polymer-infiltrated hybrid ceramic materials, Vita Enamic (E) (Vita Zahnfabrik - Germany), Nacera Hybrid (N) (Doceram medical ceramics - Germany), and Cerasmart (C) (GC Dental Product - Japan) were used to assess the micro-shear bond strength of resin composite in the repair process. Blocks of the three used ceramic materials were cut into rectangles with 2 mm thickness by micro-saw (Buehler, Isomet 4000 linear precision saw, USA) under water cooling. Specially-designed centralizing devices were used to centrally embed each rectangle in cold cured acrylic resin (Acrostone special tray material cold cure, Egypt). **Figure (1)** The outer surface of each ceramic rectangle was polished under water cooling with polishing paper by CNC machine (Metkon, Forcipol 2V Grinder-Polisher, Kemet, UK) to obtain a standardized specimens' surface finish. Each ceramic material specimens were randomly divided into 4 subgroups according to the surface treatment used; Control (B), Grinding (G), Air particle abrasion (A), and Hydrofluoric acid (F).

Control (B): The ceramic surfaces of this group were kept untouched; no treatment was done to the already finished ceramic material surface. Grinding surface treatment (G): Dry grinding with

a 125  $\mu\text{m}$  grain sized ceramic grinding cylindrical diamond stone (Komet, USA), a very thin layer was removed evenly by applying 10 strokes in one direction over the entire ceramic surface. Air abrasion surface treatment (A): Air abrasion unit (microjato plus, Bio art, Brazil) was used with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles at 2.5 bar pressure for 20 seconds from a distance of 10 mm with the handpiece positioned perpendicular to the surface of ceramic. Hydrofluoric acid surface treatment (F): Hydrofluoric acid etchant (9.5% conc.) (Bisco, Inc. Schaumburg, USA) was applied to the dry ceramic surface and left for 60 seconds, the etchant was suctioned and the ceramic surface was rinsed with water spray for 30 seconds according to manufacturer instructions.

After ceramic surface treatments, representative specimens from each subgroup of each ceramic material were scanned by Atomic Force Microscope (AFM) (Model: Flex AFM3, Switzerland) to detect the surface topography of the ceramic surfaces. A heavy body rubber base impression material (Xilgumsep fluid, Lascod, Italy) was used to create a cylinder of rubber base with 2 mm thickness at the edges, 1 mm thickness at the ceramic rectangle area and 2.5 cm diameter. The inner surface of rubber base cylinder had a depression accommodating each ceramic rectangle that will assist in the fabrication of composite micro-tubes, marks were done at the depression of the rubber base cylinders and the marks were distributed evenly according to the diameter of each rectangle then these marks were drilled with 1 mm diameter cylindrical bur, each resulted hole was 1 mm diameter and the space between each hole and the adjacent was 2 mm, creating a mold that would accommodate the composite micro tubes for the micro-shear test. **Figure (2)**



**Figure 1:** Specially-designed centralizing devices

- A: Teflon base
- B: Splinted Teflon mold
- C: Teflon cylinder



**Figure 2:** Rubber base mold

One thin coat of silane coupling agent (Bisco Porcelain primer, USA) was applied to each treated ceramic surface and left for 30 seconds. A

thin layer of Monobond was added on the ceramic surface and left for 60 seconds then air dried, and a thin layer of Heliobond was applied and cured with



a light curing unit according to the repair kit manufacturer recommendations. (Ceramic Repair N, Ivoclar Vivadent AG, Liechtenstein).

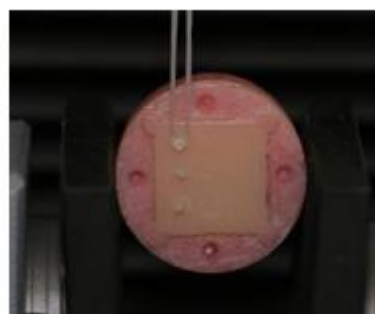
The previously fabricated rubber base cylinders were realigned over their blocks and the resin composite material (Tetric-N-Ceram, Ivoclar Vivadent AG, Liechtenstein) was condensed into the holes and light cured. Each subgroup of each ceramic material contained 20 specimens of composite micro-tubes (n=20). **Figure (3)** The



**Figure 3:** Composite micro-tubes

The failure mode was examined under a stereo microscope (Olympus SZ61, Munster, Germany), failure modes were classified as; adhesive (failure at composite-ceramic bonded interface), cohesive (failure at the composite material) or mixed (adhesive + cohesive). Observations were performed from all tested

micro-shear bond strength test was performed by Universal Testing machine (Bluehill Universal, Instron, Norwood, USA). A fishing wire was hooked around the resin composite micro-tube at the ceramic-composite interface and attached to the upper moving arm of the testing machine. A shear force was exerted to each cylinder specimen at 0.5mm/min cross head speed until failure occurred, the load of failure was recorded in MPa. **Figure(4)**

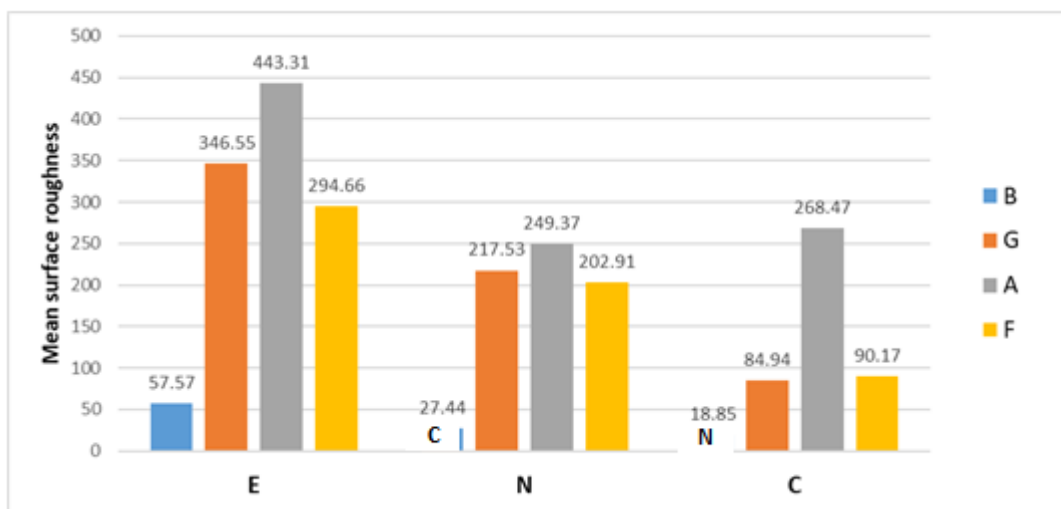


**Figure 4:** Micro-shear bond strength test

specimens and all obtained data were collected, tabulated and subjected to statistical analysis.

### III. RESULTS

**Surface roughness results:** Overall surface roughness (nm) among the ceramic materials (E, N, C) and surface treatment methods (B, G, A, F) were shown in **Figure 5**.



**Figure 5:** Comparison between the mean surface roughness of the studied ceramic materials groups

Regarding the ceramic materials, in Vita Enamic groups, the highest mean surface roughness value was found in group EA (443.31±51.82 nm) and the lowest value was found in group EB (57.57±6.24 nm). One-Way ANOVA test revealed a

statistically significant difference (P<0.001) between all Vita Enamic groups.

In Cerasmart groups, the highest mean surface roughness value was found in group CA (249.37±30.09 nm) and the lowest value was found



in group **CB** (27.44±3.25 nm). One-Way ANOVA test showed a statistical significant difference between group **CB** and groups **CG**, **CA** and **CF** and between groups **CA** and **CF**. While there was no significant difference between groups **CG** and **CF**, and no significant difference between groups **CG** and **CA**.

In Nacera hybrid groups, the highest mean surface roughness value was found in group **NA** (268.47±43.17 nm) and the lowest value was found in group **NB** (18.85±1.79 nm). One-Way ANOVA test showed a statistical significant difference between group **NB** and groups **NG**, **NA** and **NF** and between group **NA** and groups **NG** and **NF**. While there was no significant difference between groups **NG** and **NF**.

Regarding the surface treatment methods, the highest mean surface roughness value in control groups was found with Vita Enamic group (**EB**)(57.57±6.24 nm), while the lowest value was found with Nacera Hybrid group (**NB**)(18.85±1.79 nm), there was statistical significant difference between all studied groups. The highest mean surface roughness in grinding groups was found with Vita Enamic group (**EG**) (346.55±34.59 nm), while the lowest value was found with Nacera Hybrid group (**NG**)(84.94±5.08nm), there was a statistical significant difference between all studied

groups. The highest mean surface treatment value in air abrasion groups was found with Vita Enamic group (**EA**)(443.31±51.82 nm), while the lowest value was found with Cerasmart group (**CA**)(249.37±30.09 nm), there was a statistical significant difference between group **EA** and groups **CA** and **NA**. While there was no significant difference between groups **CA** and **NA**. The highest mean surface roughness value in hydrofluoric acid groups was found with Vita Enamic group (**EF**)(294.66±26.58 nm), while the lowest value was found with Nacera Hybrid group (**NF**)(90.17±10.84 nm), there was statistical significant difference between all groups.

Two-Way ANOVA test was used to evaluate the impact of changing the ceramic material and different surface treatment on surface roughness. It indicated that changing the ceramic material and surface treatment had a statistical significant difference on the surface roughness of the ceramic materials (P<.001).

#### Micro-shear bond strength test:

Statistical analysis including mean and standard deviation of the overall micro-shear bond strength in (MPa) among the ceramic materials and surface treatment methods were shown in **Table (1)**.

Microshear bond strength(MPa)	B N=20	G N=20	A N=20	F N=20	Test significance
<b>E</b>	26.50±4.14	40.46±5.56 <sup>A</sup>	57.47±5.35 <sup>a</sup>	40.11±4.81 <sup>A</sup>	F=128.68 P<0.001*
<b>C</b>	15.92±4.32	36.12±5.50 <sup>A</sup>	40.74±5.0 <sup>B</sup>	38.29±6.32 <sup>AB</sup>	F=74.14 P<0.001*
<b>N</b>	12.08±3.11	23.23±2.85 <sup>A</sup>	51.55±5.55 <sup>a</sup>	23.62±2.99 <sup>A</sup>	F=393.52 P<0.001*
	F=73.46 P<0.001*	F=69.44 P<0.001*	F=50.81 P<0.001*	F=68.05 P<0.001*	

Similar superscripted capital letters denote non-significant difference between groups through the same row and similar superscripted small letters denote non-significant difference between groups through the same column

F: One-Way ANOVA test \*statistically significant

The results of Micro-shear bond strength test followed the same pattern of surface roughness measurements, regarding the surface ceramic materials; In Vita Enamic groups; the highest mean micro-shear bond strength value was found in group **EA**, while the lowest value was found in group **EB**. One-Way ANOVA test revealed a

statistical significant difference (P<0.001) between group **EB** and groups **EG**, **EA** and **EF**. While there was no significant difference between groups **EG** and **EF**.

In Cerasmart groups; the highest mean micro-shear bond strength value was found in group **CA**, while the lowest mean value was found in group **CB**. One-Way ANOVA test showed a statistical significant difference between group **CB** and groups **CG**, **CA** and **CF** and between groups **CG** and **CA**. While there was no significant difference between groups **CG** and **CF** and between groups **CA** and **CF**.





In Nacera hybrid groups; the highest mean micro-shear bond strength value was found in group **NA**, while the lowest value was found in group **NB**. One-Way ANOVA test revealed a statistical significant difference between group **NB** and groups **NG**, **NA** and **NF** and between group **NA** and groups **NG** and **NF**. While there was no significant difference between groups **NG** and **NF**.

Regarding the surface treatment methods, the highest mean micro-shear bond strength value in control groups was found with Vita Enamic group **EB**, while the lowest value was found with Nacera Hybrid group **NB**. The highest mean micro-shear bond strength value in grinding groups was found with Vita Enamic group **EG**, while the lowest value was found with Nacera Hybrid group **NG**. The highest mean micro-shear bond strength value in air abrasion groups was found with Vita Enamic group **EA**, while the lowest value was found with Cerasmart group **CA**. The highest mean micro-shear bond strength value in hydrofluoric acid groups was found with Vita Enamic group **EF**, while the lowest value was found with Nacera Hybrid **NF**. One-Way ANOVA test showed a statistical significant difference ( $P < 0.001$ ) between all studied surface treatment methods groups.

Two-Way ANOVA test was used to evaluate the impact of changing the ceramic materials and different surface treatment methods on micro-shear bond strength of the composite micro-tubes. It indicated that changing the ceramic material and surface treatment method had a statistical significant difference on the micro-shear bond strength of the composite micro-tube ( $P < 0.001$ ).

#### Failure mode analysis:

The results of mode of failure analysis for all ceramic groups with different surface treatment methods showed predominance of adhesive failure (50-90%), followed by mixed failure (5-40%) and the lowest failure mode among all ceramic groups was the cohesive failure within the composite micro tubes. (0-15%).

#### IV. DISCUSSION

All-ceramic restorations are susceptible to chipping clinically, relying on the reason and extent of chipping in ceramic restoration, repairing intraorally with composite resin can be recommended as minimally invasive, cost effective and simple alternative to extraoral repair.<sup>(11)</sup> Clinical application of different surface treatments and the use of repair system are required for intraoral repair to optimize the bond of the

composite resin material onto the ceramic surface.<sup>(12)</sup>

This study was made to evaluate the intra oral reparability of ceramic-resin hybrid materials that combine the favorable characteristics of ceramics and composite. The purpose of developing these ceramics was to mimic the natural dentition, they have mechanical properties close to those of dental structure as hardness and modulus of elasticity, thus decrease the wear of opposing teeth when compared to other ceramic types.<sup>(13)</sup> Ceramic materials used in this study were Vita Enamic, Nacera hybrid and Cerasmart.

Blocks of the three used ceramic materials were cut into rectangles by micro-saw to decrease the material loss and gain the maximum number of rectangles from each block. Specially-designed centralizing devices were used to centrally embed each rectangle in cold cured acrylic resin to give standardized acrylic block containing ceramic rectangle prominent by 1 mm, each material had its specially-designed centralizing device because the three materials had different dimensions.

Mechanical interlocking is the most important aspect in increasing the bond strength of repaired ceramic restorations, Therefore, many surface treatment methods were used on the ceramic surfaces to increase the surface area thus increasing the surface roughness which improve the mechanical interlocking.<sup>(14)</sup> Surface treatments that involve grinding using diamond bur creates micro-retentive surface structure thus increasing the surface area and wettability. Air abrasion using 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles and 2.5 bar air pressure, these parameters were chosen to avoid superficial cracks and defects in the material surface and to keep the surface treatment homogenous.<sup>(15)</sup>

Acid etching using 9.5% hydrofluoric acid is carried out, as it can create a roughened surface in most acid sensitive polymers and ceramics.<sup>(16)</sup> These surface treatment methods are easily applicable and commonly used surface treatments methods in intra-oral repair of ceramic restorations.<sup>(2, 8)</sup> Control group where no surface treatment was applied, is used as a base line. In this study, Atomic Force Microscope (AFM) was used to assess surface roughness, as this technology demonstrated efficacy in evaluation of micro-irregularities on hard surfaces.<sup>(17)</sup>

A rubber base impression material was used to fabricate molds for the composite micro-tubes as there is no adhesion between the rubber base material and the composite so the molds can be removed easily keeping the composite micro-tubes intact. The surface roughening was followed by applying silane coupling agent, which is the



most commonly used primer to enhance the chemical adhesion between composite and ceramic surfaces.<sup>(18)</sup> Adhesive system should be used to achieve chemical bond between the ceramic surfaces and composite resin in the repair process, ceramic repair system which contains two types of adhesives monobond and heliobond and Tetric N Ceram composite was used in the present study as it is suitable for intra oral repair of chipped restorations also it gave high bond strength values in previous study.<sup>(19)</sup>

Micro-shear bond strength test is done by exerting a force parallel to the bonding interface, the micro-shear bond strength (in MPa) is calculated by dividing the maximum load (in N) to the surface area (in mm<sup>2</sup>) of the resin composite.<sup>(20)</sup> The pattern of fracture was determined by analyzing the specimen under a Stereo-microscope to evaluate the mode of failure at the interface area.

According to the results of this in-Vitro study the hypothesis which is; different types of surface treatments affect the bond strength of composite resin to polymer-infiltrated hybrid ceramic materials was accepted as the surface treatments generally enhanced the surface roughness and micro-shear bond strength of resin composite to polymer-infiltrated hybrid ceramic materials when compared to untreated material surfaces.

#### Surface roughness results:

Regarding the surface treatment method, the highest surface roughness values in Vita Enamic, Cerasmart and Nacera Hybrid were found in air abraded ceramic surfaces by Al<sub>2</sub>O<sub>3</sub> particles, this may be because air abrasion is based on throwing of the particles, that was accelerated by air pressure against the substrate surface, so the energy generated by this impact intensity roughens the substrate surface giving an irregular and porous surface topography.<sup>(21)</sup> The lowest surface roughness values in Vita Enamic, Cerasmart and Nacera Hybrid were found in untreated control surfaces, may be because the surfaces are polished and have a lower surface area when compared to the treated surfaces.

Regarding the ceramic materials, the highest surface roughness values in Control, Grinding, Air abrasion and HF groups was found with Vita Enamic ceramic material this may be due to high filler content in Vita Enamic (86% feldspar ceramic) when compared with Cerasmart (71% silica and barium glass) and Nacera hybrid (50% nano glass).

#### Micro-shear bond strength test results:

The bond strength test results showed the same pattern of surface roughness in relation to the surface treatment as air abrasion surface treatment showed the highest bond strength values through all ceramic materials used in this study, this is due to increased ceramics surface roughness that improves the interlocking between resin composite and substrate and increased surface energy that enables optimal wetting of the silane thus enhancing chemical bond.<sup>(21)</sup> Grinding and HF etching revealed significantly lower bond strength values than air particle abrasion surface treatment. The lower bond strength values of HF etching may be because HF removes the glass matrix keeping only the polymer component, thus the polymer alone at the interface could result in weaker bond strength. While the other surface treatments create a rough surface keeping both the polymer and the glass matrix.<sup>(22)</sup> Control groups, where no surface treatment was made, showed the lowest bond strength values due to decreased surface roughness compared to treated ceramic surfaces resulting in less mechanical interlocking and less wettability of the adhesive system.

Surface treatment of ceramic surfaces showed improvement in the repair bond strength of composite resin to polymer infiltrated hybrid ceramic materials, which is in agreement with previous studies.<sup>(1, 13, 14)</sup> In this study, Micro-shear bond strength test showed the lowest bond strength values with untreated control groups while the highest bond strength values was found with air particle abrasion surface treatment groups and these findings were in agreement with results of **Stawarczyk et al.**<sup>(14)</sup> and **Şişmanoğlu et al.**<sup>(21)</sup> although they compared between different hybrid ceramic materials and used different bond strength test methods. In contrast to this study's findings, **Güngör et al.**<sup>(13)</sup> showed that bond strength of composite resin to hybrid ceramic material was relatively lower after air abrasion when compared to grinding with diamond bur, which may be because they used different bond strength test method, different air abrasion bar pressure and less air abrasion time than this study. Also, **Silva et al.**<sup>(22)</sup> showed that grinding gave higher bond strength values than air borne abrasion, which may be due to the use of different diamond bur grain size (180 µm) and different Al<sub>2</sub>O<sub>3</sub> particle size (45 µm) that may affect the results of bond strength tests.

#### The limitations of this study were:

Different adhesives was not used in this study; it has been reported that using of different universal adhesives affects repair bond strength



values.<sup>(23)</sup> Bond strength was not evaluated after the effect of fatigue loading and long-term aging on repaired hybrid ceramics with composite resin.

## V. CONCLUSION

**Under the limitations of this In-Vitro study, it could be concluded that:**

1. Different methods of surface treatment enhanced the surface roughness of polymer infiltrated hybrid ceramic materials and improved the micro-shear bond strength of resin composite to ceramic material.
2. Air particle abrasion gave the highest surface roughness and micro-shear bond strength among the other surface treatment methods, while the lowest surface micro shear bond strength was seen in untreated control groups for all studied ceramic materials.
3. Vita Enamic ceramic material gave the highest surface roughness and micro-shear bond strength through all surface treatment methods used in this study.

**Under the limitations of this study, it could be recommended to:**

1. Use air particle abrasion as a surface treatment prior to the ceramic repair process.
2. More studies are needed to assess the long-term aging and fatigue loading effects on the repaired hybrid ceramic and composite resin system.

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