

Effect of surface conditioning and adhesive strategies on the repair bond strength of resin matrix ceramics

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ARTICLE INFO

Keywords:

Resin matrix ceramic
Hybrid ceramic
Repair
Resin composite

ABSTRACT

Objectives: To evaluate the effect of different surface pretreatment protocols on the repair bond strength and integrity of resin composite to three different resin matrix ceramics (RMCs).

Material and Methods: Three RMCs - Cerasmart (CS), Grandio blocs (GB) and Katana Avencia (KA) - were subjected to one of three mechanical surface treatments: polishing, diamond bur roughening, or sandblasting. These were randomly assigned to four conditioning groups: 10-MDP and silane-containing adhesive (Clearfil Universal Bond Quick), 10-MDP-based adhesive (G-Premio Bond), 9% buffered hydrofluoric acid (HF) plus silane, and a 10-MDP and silane-free adhesive (Heliobond). A nanohybrid composite (Reflectys) was applied as the repair material. Specimens were sectioned into sticks and subjected to micro-tensile bond strength (μ TBS) testing using a universal testing machine. The surface treatments were compared by one-way ANOVA and post-hoc tests for different cases of the variables μ TBS and surface roughness.

Results: For CS, the combination of sandblasting and 10-MDP plus silane-containing adhesive yielded the highest μ TBS (46.42 ± 3.12 MPa), with no significant difference observed between polishing and bur roughening. Similarly, GB showed significantly higher μ TBS when sandblasted and treated with 10-MDP plus silane adhesive (42.01 ± 5.06 MPa). However, for KA, the most effective protocol was the combination of sandblasting and 9% buffered HF with silane (48.31 ± 8.46 MPa).

Conclusions: Repair bond strength is highly dependent on the specific RMC used. No single universal mechanical surface treatment or conditioning protocol was found to be optimal across all materials. Clinicians should utilise individualised repair protocols - specifically 10-MDP and silane-containing adhesives for CS and GB, and HF and silane for KA - to achieve maximum repair bond strength.

Clinical Significance: When repairing RMC restorations chairside, clinicians should identify the specific material brand, whenever possible, as the chemical composition of the RMC dictates whether a universal adhesive or a traditional HF and silane protocol will provide the most durable immediate repair.

1. Introduction

The use of traditional dental ceramics has long been favoured for their excellent aesthetics, biocompatibility, and chemical stability [1,2]. However, their low fracture toughness, inherent brittleness and high surface hardness often result in catastrophic fractures or significant wear of the opposing dentition [3,4]. To address these limitations, resin matrix ceramics (RMCs) - also termed hybrid ceramics - were developed to

combine the advantageous features of ceramics and the mechanical flexibility of resin composites [5,6].

According to the American Dental Association (ADA) nomenclature, hybrid ceramics are defined by a predominant inorganic refractory content (>50% by weight) within an organic polymer matrix [7]. Based on their microstructure, RMCs are classified into three distinct categories: (1) resin nanoceramics, (2) glass-ceramics within an interpenetrating polymer network (IPN), and (3) zirconia-silica ceramics

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<https://doi.org/10.1016/j.jdent.2026.106968>

Received 26 May 2026; Received in revised form 5 August 2026; Accepted 8 August 2026

Available online 8 August 2026

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within an IPN [8]. By combining a ceramic network with a polymer matrix, these computer-aided design and computer-aided manufacturing (CAD-CAM)-processable materials achieve a Young's modulus similar to human dentin, enhancing their millability and potential for intraoral repair [6,9].

Despite their optimised mechanical performance, RMCs remain susceptible to fatigue and fracture under functional masticatory loads [10]. When a RMC restoration exhibits localised defects, intraoral repair using resin composites is often the preferred treatment of choice [11] in an attempt to prolong the clinical service of existing RMC restoration. Contrary to complete replacement, repair is minimally invasive, cost-effective, and preserves healthy tooth structure [12,13]. However, the success of such repairs is complicated by the aging process; prolonged exposure to the oral environment - including thermal changes and chemical degradation - can alter the material's surface integrity and compromise the adhesive interface [14–16].

Effective bonding requires a synergistic approach of mechanical surface treatment and chemical conditioning [17]. While techniques such as sandblasting, diamond bur roughening, and hydrofluoric acid (HF) etching are used to increase surface area and mechanical interlocking, chemical adhesion is typically mediated through universal adhesives and silane coupling agents [18–23]. Recent evidence suggests that while silane alone may be insufficient, its integration into universal adhesive systems may significantly enhance bond durability [24,25].

At present, there is still some debate as to the best repair protocol for the various RMCs and it is therefore difficult for clinicians to select a repair protocol to achieve the best clinical outcome for a particular RMC material. Thus, currently, a standardised "gold standard" protocol for the different types of RMC repair remains elusive.

Therefore, this *in vitro* study aimed to evaluate and compare the microtensile bond strength (μ TBS) of various mechanical surface treatments and surface conditioning strategies for the repair of three distinct RMCs with a nanohybrid composite. The null hypotheses tested were that there were no statistically significant differences in repair bond strengths between (i) the mechanical surface treatments used in the various repair protocols, and (ii) the type of adhesive/chemical treatment applied for RMC repair with a nanohybrid resin composite ($\alpha = 0.05$).

2. Materials and methods

2.1. Specimen preparation

The description, composition and manufacturer details of the materials used in this study are summarised in Table 1. A total of eighteen blocks [six Cerasmart blocks (CS) (GC Dental Products, Leuven, Belgium), six Grandio blocs (GB) (Voco GmbH, Cuxhaven, Germany), and six Katana Avencia (KA) blocks (Kuraray Noritake Dental, Tokyo, Japan)] were sectioned using a low-speed diamond internal annulus saw microtome (Leitz 1600, Leitz, Wetzlar, Germany). The sectioned blocks were sliced into smaller blocks measuring in the case of CS 7 mm length x 12 mm width x 10 mm height and in the case of GB and KA 7.25 mm length x 14.5 mm width x 10 mm height.

All sliced blocks were subjected to an accelerated ageing process in a thermal cycling machine (SD Mechatronik Thermocycler THE-1100, Germany) for 5000 cycles at 5–55 °C with a dwell time of 30 s. Subsequently, the top of the sectioned surfaces were polished with wet 500-grit, 1000-grit and 1200-grit silicon carbide discs using a polishing machine (Struers LaboPol35, Struers A/S, Rodovre, Denmark) at 300 rpm for 30 s and then cleaned for 3 × 5 min in an ultrasonic bath (Elmasonic S40H, Elma Schmidbauer GmbH, Singen, Germany) containing purified water to clean the cut surface from any contamination.

2.2. Surface treatment methods

The CS blocks (n = 12), GB (n = 12), and KA blocks (n = 12) were

Table 1

The RMCs and repair protocol materials used in the study.

Material (Code)	Manufacturer	Components	LOT
Cerasmart (CS)	GC Dental Products, Leuven, Belgium	Organic part: Bis-MEPP, UDMA, DMA Inorganic part: barium glass (300 nm), SiO ₂ (20 nm) Filler content: 71 wt%	2103236
Grandio blocs (GB)	Voco GmbH, Cuxhaven, Germany	Organic part: 14% UDMA, DMA Dimethacrylates Inorganic part: glass ceramics, silica Filler content: 86 wt%	2142307
Katana Avencia (KA)	Kuraray Noritake Dental, Tokyo, Japan	Organic part: UDMA, methacrylate monomers Inorganic part: SiO ₂ (40 nm), Al ₂ O ₃ (20 nm) Filler content: 62 wt%	000629
Clearfil Universal Bond Quick adhesive	Kuraray Noritake Dental Inc., Japan	Bis-GMA, MDP, HEMA, hydrophilic amide monomer, filler, ethanol, water, NaF, photo initiators, chemical polymerization, accelerator, silane coupling agent, others	CK0443
G-Premio Bond	GC, Japan	10-MDP, MTDP, 4-MET, methacrylic acid ester, silica, catalyst, photoinitiators, acetone, water	2211031
Ultradent Porcelain Etch	Ultradent, USA	9% buffered hydrofluoric acid	BLF87
Ultradent Porcelain Silane	Ultradent, USA	Methacryloxypropyltrimethoxy silane < 10%, isopropyl alcohol < 95%	BL2TS
Heliobond adhesive	Ivoclar Vivadent, Liechtenstein	Bis-GMA 59.5 wt%, TEGDMA 39.7 wt%, CQ stabilizers and catalysts 0.8 wt%	Z024NN
Reflectys	Itena Clinical, Paris, France	High filler load of 80 wt% in a 20% resin matrix, utilizing barium aluminosilicate and fumed silica	DF1534A1

randomly divided, using randomisation tables, into four equal groups, each containing three specimens. Three custom-made silicon mold retention molds were fabricated using these specimens. Each mold contained a rectangular recess at the centre. The recess in the CS mold was 7 mm length x 12 mm width x 18 mm depth. The recesses in both the GB and KA molds were 7.25 mm length x 14.5 mm width x 18 mm depth. The custom silicone molds were utilised exclusively to standardise the resin composite build-up process. The variations in the internal dimensions of the mold cavities reflected the inherent size differences of the manufacturer-supplied CAD-CAM blocks. Specifically, the CS mold recess measured 7 mm x 12 mm, while the GB and KA molds were fabricated to 7.25 mm x 14.5 mm to accommodate their larger manufactured dimensions. Each mold was individually tailored to the halved ceramic segments to ensure a precise and stable fit during the incremental layering of the Reflectys composite.

Three specimens of each RMC material were subjected to (i) sandblasting from a distance of 10 mm, at a pressure of 2–3 bar, held for 10 s, using 50 μ m aluminium oxide powder, MicroEtcher (Danville, USA), (ii) roughening with a diamond bur [20 μ m, ISO 504, 858C 010 (Meisinger, Germany)] and (iii) surface polishing without any additional surface preparation. Following surface treatment each block was cleaned with acetone for 3 × 5 min in an ultrasonic bath (Elmasonic S40H, Elma Schmidbauer GmbH, Singen, Germany) and placed back into their silicon molds. Each group of the CS (C1–C12), GB (G1–G12), and KA (K1–K12) specimens received the allocated surface conditioning treatments and adhesive strategies according to manufacturers' instructions (Fig. 1).

Group C1, G1, K1: one thin layer of Clearfil Universal Bond Quick

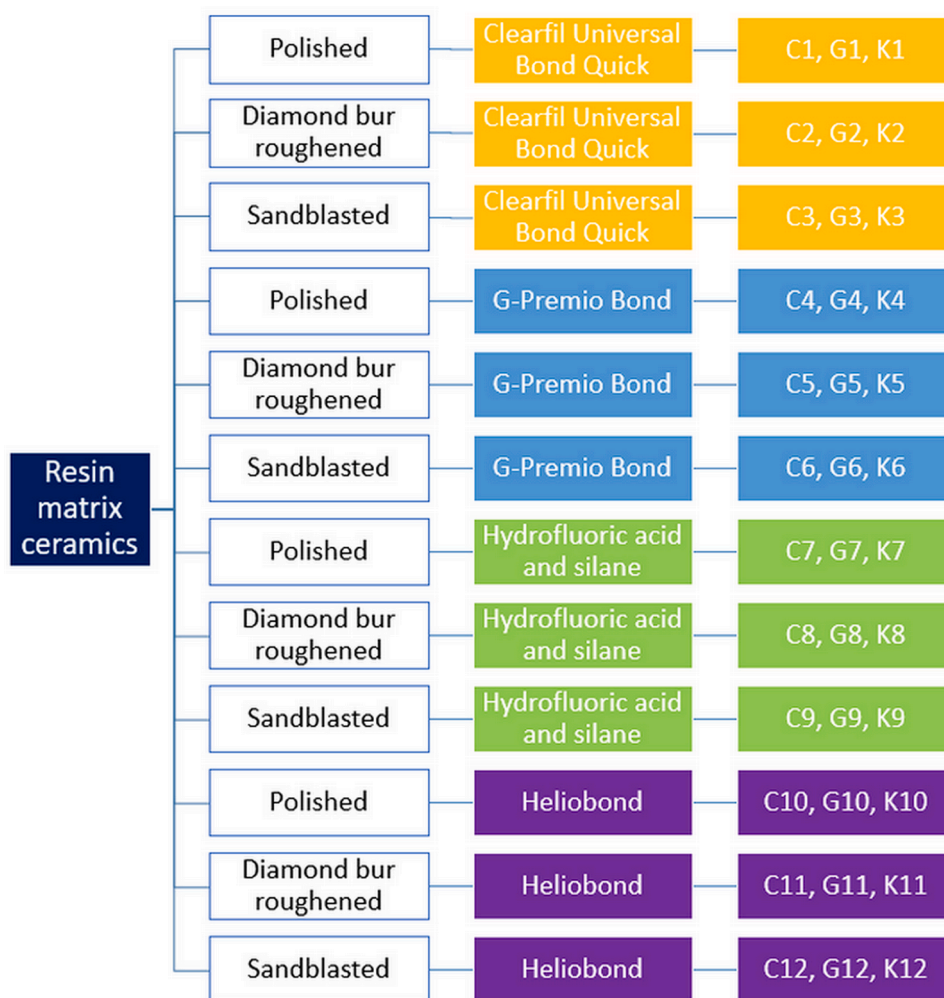


Fig. 1. Summary of mechanical surface treatments and conditioning methods for the specimens groups in the study, where C1-C12 relate to CS, G1-G12 relate to GB and K2-K12 relate to the KA groups.

adhesive (Kuraray Noritake Dental Inc., Japan) applied to the polished surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C2, G2, K2: one thin layer of Clearfil Universal Bond Quick adhesive (Kuraray Noritake Dental Inc., Japan) applied to the diamond bur roughened surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C3, G3, K3: one thin layer of Clearfil Universal Bond Quick adhesive (Kuraray Noritake Dental Inc., Japan) applied to the sandblasted surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C4, G4, K4: one thin layer of G-Premio Bond adhesive system (GC, Japan) applied to the polished surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C5, G5, K5: one thin layer of G-Premio Bond adhesive system (GC, Japan) applied to the diamond bur roughened surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured

for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C6, G6, K6: one thin layer of G-Premio Bond adhesive system (GC, Japan) applied to the sandblasted surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C7, G7, K7: application of 9% buffered HF (Ultradent Porcelain Etch, USA) to the polished surface of the RMC block for 90 s, washed with water and then acetone for 90 s, dried with oil-free air/water syringe for 5 s, followed by the application of silane (Ultradent Porcelain Silane, USA) to the surface with a disposable applicator for 60 s.

Group C8, G8, K8: diamond bur roughening of surface of the RMC block followed by the application of 9% buffered HF (Ultradent Porcelain Etch, USA) for 90 s, washed with water and then acetone for 90 s, dried with oil-free air/water syringe for 5 s, and application of silane (Ultradent Porcelain Silane, USA) to the surface with a disposable applicator for 60 s.

Group C9, G9, K9: sandblasting of surface of the RMC block and application of 9% buffered HF (Ultradent Porcelain Etch, USA) for 90 s, washed with water and then acetone for 90 s, dried with oil-free air/water syringe for 5 s, followed by application of silane (Ultradent Porcelain Silane, USA) to the surface with a disposable applicator for 60 s.

Group C10, G10, K10: one thin layer of Heliobond adhesive (Ivoclar Vivadent, Lichtenstein) applied to the polished surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured

for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C11, G11, K11: one thin layer of Heliobond adhesive (Ivoclar Vivadent, Lichtenstein) applied to the diamond bur roughened surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

Group C12, G12, K12: one thin layer of Heliobond adhesive (Ivoclar Vivadent, Lichtenstein) applied to the sandblasted surface of the RMC block with a disposable applicator and circular brushing motion for 10 s, waited 10 s, dried with oil-free air/water syringe for 5 s and light cured for 20 s (Bluephase 20i, Ivoclar Vivadent, Lichtenstein).

2.3. Resin nanohybrid composite application

A nanohybrid resin composite (Reflectys, Itena Clinical, Paris, France) was applied to the conditioned RMC surfaces in 2-mm increments until a total build-up height of 7 mm was achieved. Each layer was light-polymerized for 40 s using a high-intensity LED curing unit (Bluephase 20i, Ivoclar Vivadent, Lichtenstein) according to the manufacturer's instructions. Following the build-up procedure, the specimens were removed from the silicon molds and stored in a controlled environment at room temperature ($25 \pm 1^\circ\text{C}$) for 24 h prior to sectioning.

The composite-RMC blocks were mounted onto a flat polymer support using a self-adhesive resin cement (G-Cem One, GC, Tokyo, Japan) to facilitate stable sectioning. The blocks were then sectioned into longitudinal beams of 1 mm width $\times$ 1 mm length $\times$ 14 mm height dimension using a precision hard tissue microtome (Leitz 1600, Leitz, Wetzlar, Germany) under constant water cooling. A minimum of 20 specimens was obtained for each experimental group.

Following sectioning, the specimens were stored in distilled water at for 24 h to ensure standardized conditions before μTBS testing.

2.4. Microtensile bond strength testing

Prior to testing, the exact cross-sectional dimensions of each specimen were measured using a digital caliper (DC54150, DML digital micrometers, Sheffield, England) to determine the precise interfacial bonding area (mm^2). Each specimen was then individually aligned and fixed to a custom microtensile testing jig using a cyanoacrylate adhesive.

Tensile loading was performed using a universal testing machine (Instron 5544, Norwood, MA, USA) equipped with a 2 kN load cell. The test was conducted at a constant crosshead speed of 1 mm/min until failure occurred. The maximum load at fracture (F) was recorded in Newtons (N), and the microtensile bond strength (σ in MPa) was calculated according to the following equation:

$$\sigma = F/A$$

where F is the load at failure (N) and A is the pre-measured bonding area (mm^2).

2.5. Failure mode analysis

Following microtensile testing, the fractured surfaces of all specimens were examined using a stereomicroscope (Olympus SZ61, Tokyo, Japan) at 25 times magnification to determine the mode of failure. Failures were visually classified into one of the following three categories:

1. **Adhesive failure:** Failure occurring entirely at the interface between the RMC and the repair composite.
2. **Cohesive failure:** Failure occurring entirely within the RMC or the repair composite.
3. **Mixed failure:** A combination of adhesive and cohesive failure, where remnants of the bonding agent or composite remained on the RMC surface.

The distribution of failure modes was recorded for each experimental group and expressed as a percentage of the total number of specimens tested.

2.6. Surface characterization: profilometry and scanning electron microscopy (SEM) analysis

To evaluate surface topography and morphology, three specimens of each RMC material (CS, GB, and KA) were sectioned into plates of 1 mm height using a hard tissue microtome (Leitz 1600, Leitz, Wetzlar, Germany). To simulate clinical aging, all specimens were subjected to thermocycling (SD Mechatronik Thermocycler THE-1100, Germany) for 5000 cycles between 5°C and 55°C , with a dwell time of 30 s time in each bath. Following aging, the surfaces were standardized by polishing with silicon carbide (SiC) abrasive discs (500-grit, 1000-grit and 1200 grit; Struers LaboPol35, Struers A/S, Rodovre, Denmark) at 300 rpm for 30 s under water cooling. Specimens were then cleaned in an ultrasonic bath (Elmasonic S40H, Elma Schmidbauer GmbH, Singen, Germany) with distilled water for 3×5 min to remove any surface contaminants.

The specimens from each material were then randomly assigned to one of three surface conditioning groups:

1. **Polishing (Control):** No further treatment after the 1200-grit finish.
2. **Sandblasting:** Surfaces were air-abraded with $50\ \mu\text{m}$ aluminium oxide powder using a MicroEtcher (Danville Materials, Carlsbad, CA, USA) at a pressure of 2–3 bar from a distance of 10 mm for 10 s.
3. **Diamond bur roughening:** Surfaces were manually roughened using a fine-grit diamond bur (grit size, ISO 504, 858C 010, Meisinger, Neuss, Germany).

These prepared surfaces were subsequently analysed using a profilometer for surface roughness (Ra) and SEM for morphological assessment.

2.7. Surface topography and morphological analysis

2.7.1. Scanning electron microscopy

To characterise the surface morphology resulting from different conditioning methods, representative specimens from each group were sputter-coated with a 50-nm layer of gold (Au) using a sputter coating unit (Bio-Rad SEM Sputter Coating Unit PS3, Microscience Division, West Chester, USA). The surfaces were then visualised using a field-emission scanning electron microscope (Hitachi S-4300, Hitachi Science Systems, Ltd., Tokyo, Japan) operating voltages ranging from 5 to 15 kV get more information about the different surface morphologies of the RMCs. Micrographs were captured at various magnifications to examine the topographic features at scales of $10\ \mu\text{m}$ and $50\ \mu\text{m}$.

2.7.2. Surface profilometry

Quantitative surface roughness measurements were performed using a contact profilometer (Ambios Technology XP-1, Santa Cruz, CA, USA). A diamond stylus with a tip radius of $2.0\ \mu\text{m}$ was used to scan the surfaces at a speed of with a constant contact force of $0.5\ \text{mm/s}$ speed with a constant contact force of 1 mg. Ten measurements ($n = 10$) were conducted at different locations for each specimen to ensure a representative assessment of the surface texture. The following roughness parameters were recorded:

- Ra: Arithmetical mean deviation of the profile.
- Rq: Root mean square deviation of the profile.
- Rp: Maximum profile peak height.
- Rv: Maximum profile valley depth.

2.8. Statistical analysis

Mean values of μTBS and surface roughness parameters were

compared among the different RMC groups. Homogeneity of variances was assessed using Levene's test. Depending on the outcome, either a one-way analysis of variance (ANOVA) or Welch's robust ANOVA was performed. Where significant differences were identified, post hoc pairwise comparisons were conducted using Tukey's honestly significant difference (HSD) test following one-way ANOVA or Tamhane's T2 test following Welch's ANOVA, as appropriate. These statistical methods are considered robust to moderate departures from normality for sample sizes comparable to those used in the present study.

Descriptive statistics and all inferential analyses were performed using IBM SPSS Statistics for Windows, Version 31.0 (IBM Corp., Armonk, NY, USA). A significance level of $\alpha = 0.05$ was adopted for all statistical tests.

3. Results

3.1. Microtensile bond strength (μ TBS)

The μ TBS values (Mean SD) for all experimental groups are summarised below, categorised by the RMC substrate (Table 2.).

3.1.1. Cerasmart blocks

For CS specimens, the adhesive system significantly influenced the bond strength. The highest μ TBS overall was achieved in the sandblasted group (C3) treated with Clearfil Universal Bond Quick (46.42 $\pm$ 3.12 MPa); however, no significant differences were observed between the polishing (C1) and diamond bur roughening (C2) protocols within this adhesive group (Fig. 2A) by the Welch ANOVA ($p = 0.668$).

When treated with G-Premio Bond, the diamond bur roughening group (C5) yielded the highest values (26.22 $\pm$ 4.57 MPa), followed by sandblasting (C6), while the polished group (C4) showed significantly lower bond strength than the other two (16.00 $\pm$ 4.14 MPa, Tukey gave for both cases $p < 0.001$). In contrast, for the 9% HF - silane group only the sandblasted and the diamond roughened cases showed significant differences (Tamhane, $p = 0.016$) at level 5%, with the diamond bur group (C8) group reaching 21.94 $\pm$ 4.03 MPa. For the Heliobond groups, sandblasting (C12) was significantly more effective (29.62 $\pm$ 5.06 MPa, Tukey $p < 0.001$) than the other two surface treatments (Fig. 2A).

3.1.2. Grandio blocs

In the GB groups, sandblasting group consistently emerged as the most effective surface conditioning method. Within the Clearfil Universal Bond Quick groups, sandblasting (G3) resulted in the highest μ TBS (42.01 $\pm$ 5.06 MPa), demonstrating a significant difference compared to all other treatments (Tamhane gave for both case $p < 0.001$). Diamond bur roughening (G2) performed significantly better than polishing (G1) ($p = 0.001$) (Fig. 2B).

For the remaining adhesive strategies (G-Premio Bond, HF/silane, and Heliobond), sandblasting (G6, G9, and G12) also produced the highest values within each respective group (18.04 $\pm$ 3.00 MPa, 17.38 $\pm$ 1.70 MPa, and 14.87 $\pm$ 2.20 MPa respectively). For both HF -silane and Heliobond, the sandblasted group was significantly superior to the

polished and bur-roughened counterparts (Tamhane gave for all cases $p < 0.001$) (Fig. 2B).

3.1.3. Katana Avencia blocks

The KA specimens showed high sensitivity to both mechanical and chemical treatments. In the Clearfil Universal Bond Quick group, sandblasting (K3) achieved significantly higher values (46.75 $\pm$ 8.11 MPa) compared to bur roughening (K2), and polishing (K1) cases (Tamhane gave for both $p < 0.001$). Similarly, with G-Premio Bond, sandblasting (K6) was the most effective protocol (31.06 $\pm$ 2.46 MPa), followed by bur roughening (K5).

Notably, the highest absolute bond strength for KA was recorded in the 9% HF + silane group following sandblasting (K9), reaching 48.31 $\pm$ 8.46 MPa and being significantly different from the other two means pf surface treatments (Tukey gave for both cases $p < 0.001$). In the Heliobond group, although sandblasting (K12) showed the highest mean value (28.27 $\pm$ 8.14 MPa), only the sandblasted and polished comparison provided statistically significant difference (Tamhane, $p = 0.043$) (Fig. 2C).

3.2. Summary of peak bond strengths

Across all tested RMCs, sandblasting combined with specific adhesive strategies consistently yielded the highest μ TBS values as set out below:

- CS: The highest bond strength was achieved using Clearfil Universal Bond Quick on sandblasted surfaces (Group C3: 46.42 $\pm$ 3.12 MPa), which was significantly superior to all other adhesive combinations for this material.
- GB: Similarly, the sandblasted group treated with Clearfil Universal Bond Quick (Group G3: 42.01 $\pm$ 5.06 MPa) demonstrated the significantly highest μ TBS values.
- KA: This material exhibited the highest absolute bond strengths in the study. Peak values were observed following sandblasting when paired with either 9% HF and silane (Group K9: 48.31 $\pm$ 8.46 MPa) or Clearfil Universal Bond Quick (Group K3: 46.75 $\pm$ 8.11 MPa), with no statistically significant difference between these two "top-tier" protocols.

3.3. Failure analysis

The failure modes of all specimens subjected to microtensile testing were characterized using stereomicroscopy. The distribution of failure patterns (adhesive, cohesive, and mixed) across the three RMC materials is summarized in Fig. 3.

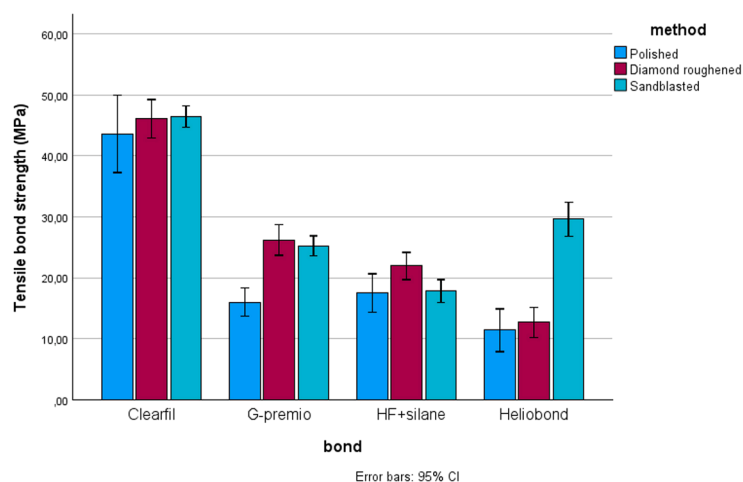
3.3.1. Cerasmart blocks

For CS, the highest incidence of cohesive failure (all within the repair composite) was observed in the groups treated with Clearfil Universal Bond Quick. Specifically, the sandblasted group (C3) exhibited a cohesive failure rate of 66.66%, followed by diamond bur roughening (C2) at

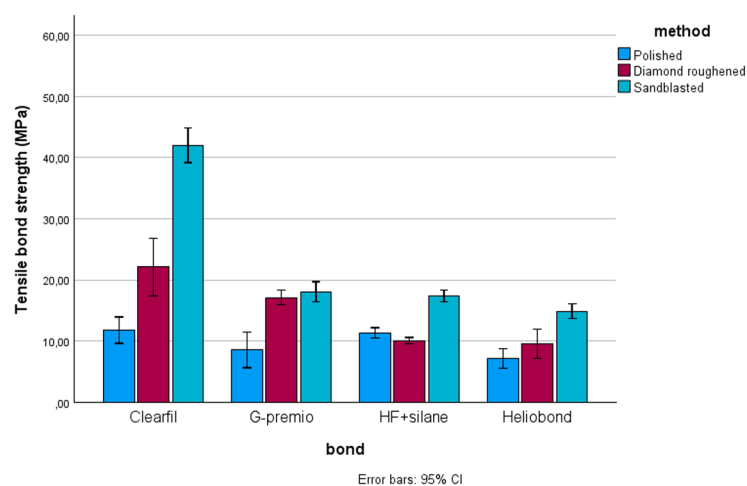
Table 2
Summary of the μ TBS results (MPa) for all RMC experimental groups.

Resin matrix ceramic	Surface conditioning	Adhesive			
		Clearfil Universal Bond Quick	G-Premio Bond	9% buffered hydrofluoric acid and silane	Heliobond
Cerasmart	Polishing	43.61 $\pm$ 11.48	16.00 $\pm$ 4.14	17.48 $\pm$ 5.74	11.38 $\pm$ 6.35
	Diamond bur roughening	46.10 $\pm$ 5.70	26.22 $\pm$ 4.57	21.94 $\pm$ 4.03	12.67 $\pm$ 4.50
	Sandblasting	46.42 $\pm$ 3.12	25.22 $\pm$ 2.93	17.83 $\pm$ 3.36	29.62 $\pm$ 5.06
Grandio blocs	Polishing	11.76 $\pm$ 3.88	8.52 $\pm$ 5.25	11.33 $\pm$ 1.47	7.14 $\pm$ 2.56
	Diamond bur roughening	22.11 $\pm$ 8.48	17.12 $\pm$ 2.23	10.06 $\pm$ 0.90	9.51 $\pm$ 4.28
	Sandblasting	42.01 $\pm$ 5.06	18.04 $\pm$ 3.00	17.38 $\pm$ 1.70	14.87 $\pm$ 2.20
Katana Avencia	Polishing	2.61 $\pm$ 1.75	8.02 $\pm$ 6.81	32.82 $\pm$ 6.16	21.54 $\pm$ 3.97
	Diamond bur roughening	30.41 $\pm$ 9.79	25.69 $\pm$ 3.02	36.68 $\pm$ 6.31	23.55 $\pm$ 9.37
	Sandblasting	46.75 $\pm$ 8.11	31.06 $\pm$ 2.46	48.31 $\pm$ 8.46	28.27 $\pm$ 8.14

A) Cerasmart blocks



B) Grandio blocs



C) Katana Avencia blocks

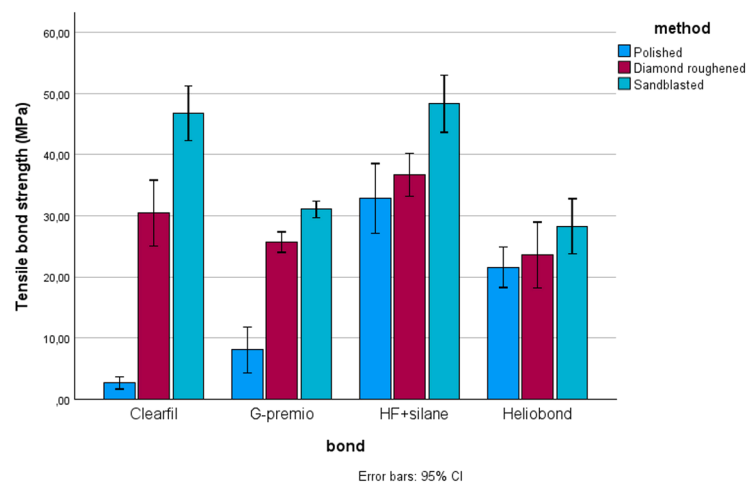


Fig. 2. Mean microtensile bond strength (μ TBS) values (MPa) of RMCs subjected to various mechanical surface protocols (polishing, diamond bur roughening, or sandblasting) and conditioning strategies (Clearfil Universal Bond Quick, G-Premio Bond, 9% buffered HF and silane, or Heliobond). Data are presented for (A) CS, (B) GB, and (C) KA. Error bars represent standard deviations.

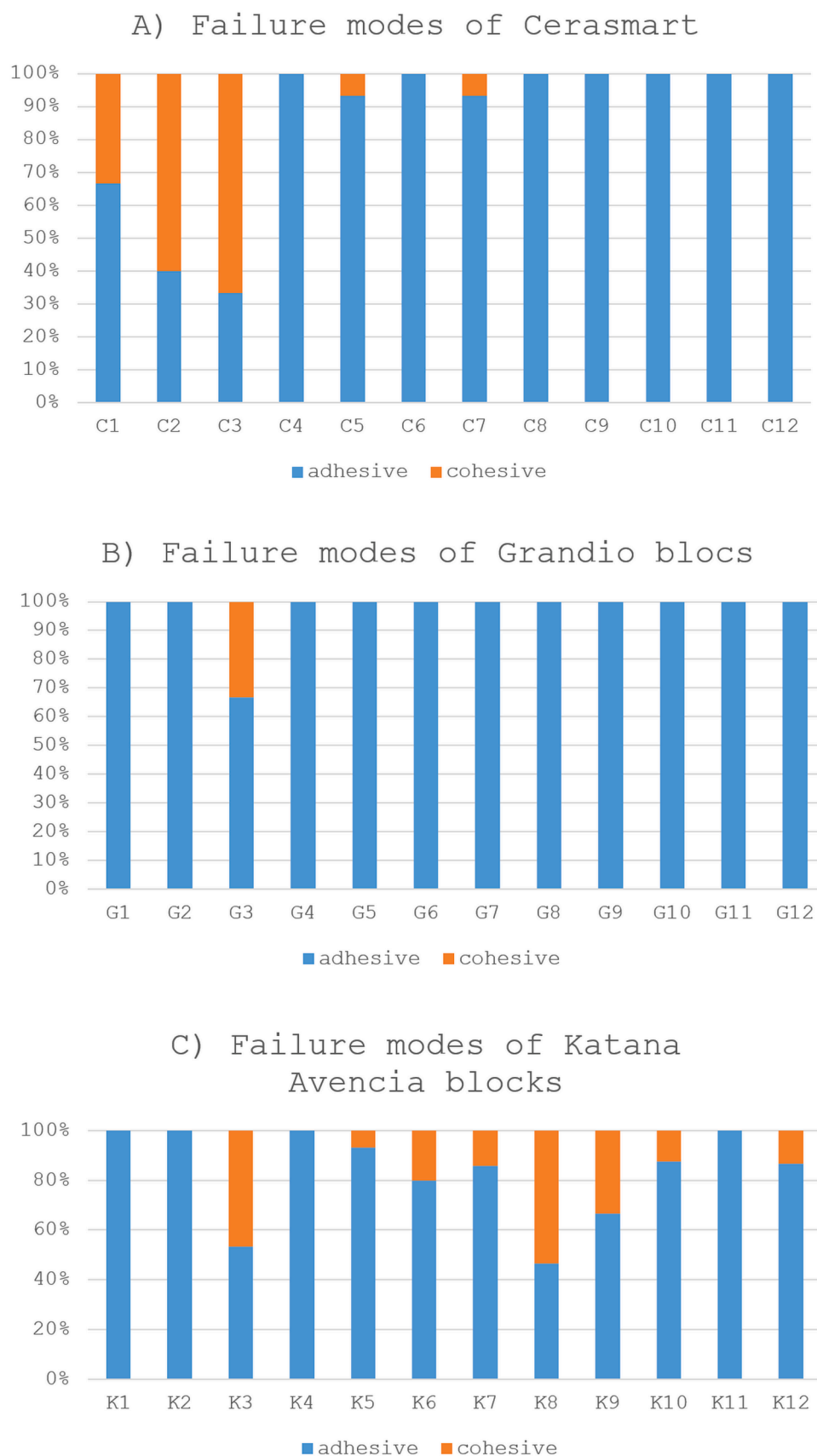


Fig. 3. Distribution of failure modes (adhesive, cohesive, and mixed) for the three RMCs following various surface conditioning and adhesive strategies. (A) CS; (B) GB; (C) KA. Mechanical treatments included polishing, diamond bur roughening, and sandblasting. Adhesive protocols included Clearfil Universal Bond Quick, G-Premio Bond, 9% buffered HF + silane, and Heliobond.

60%, and polishing (C1) at 33.33% (Fig. 3A). There was a notable absence of mixed and adhesive failures.

3.3.2. Grandio blocs

In the GB groups, the failure was predominantly adhesive across most surface conditioning and adhesive combinations. The only exception was the sandblasted group treated with Clearfil Universal Bond Quick (G3), which demonstrated a cohesive failure rate of 33.33% (Fig. 3B). This corresponds with G3 having the significantly highest μ TBS value for this material.

3.3.3. Katana Avencia blocks

KA specimens displayed a diverse range of failure modes. High rates of cohesive failure were recorded in the sandblasted groups treated with Clearfil Universal Bond Quick (K3, 46.66%) and 9% HF + silane (K9, 33.33%). Interestingly, the diamond bur roughened group treated with HF + silane (K8) showed the highest cohesive failure rate for this material at 53.33%. The sandblasted group treated with G-Premio Bond (K6) also exhibited cohesive failure in 25% of cases (Fig. 3C).

A higher prevalence of cohesive failures is noted in the high-bond-strength groups (e.g. C3 and K3).

3.4. Scanning electron microscopy (SEM) analysis

Representative SEM micrographs (magnification x 8000) (Fig. 4) revealed distinct topographical alterations resulting from the different surface conditioning protocols across all three RMCs as follows:

- **Polished surfaces:** Exhibited a relatively smooth and uniform morphology, characterized only by fine, unidirectional scratches produced by the sequential SiC paper finishing.
- **Diamond bur roughened surfaces:** Demonstrated a high degree of irregularity with deep, multi-directional linear grooves and evidence of localized surface chipping. This treatment increased the macroscopic surface area but appeared more aggressive than polishing.
- **Sandblasted surfaces:** Showed the most significant morphological transformation. The surfaces appeared highly porous and micro-retentive, with a "crater-like" appearance resulting from the impact of the particles. This treatment effectively exposed the inorganic filler particles by selectively removing the superficial polymer matrix, providing a superior foundation for micromechanical interlocking.

A transition is noted from relatively smooth surfaces in the polished groups to the highly irregular, micro-retentive "crater-like" morphology in the sandblasted groups (C, F, I), which facilitates superior micro-mechanical interlocking.

3.5. Surface roughness analysis (Profilometry)

Surface roughness (Ra) values for all RMCs and surface conditioning protocols are presented in Table 3. Across all RMCs, sandblasting consistently produced the highest surface roughness values compared with diamond bur roughening and polishing. Overall, significant differences in mean Ra values were observed among the three surface treatments (Welch ANOVA, $p < 0.001$). Pairwise comparisons were performed using Tamhane's T2 test, and the results are presented in Table 3.

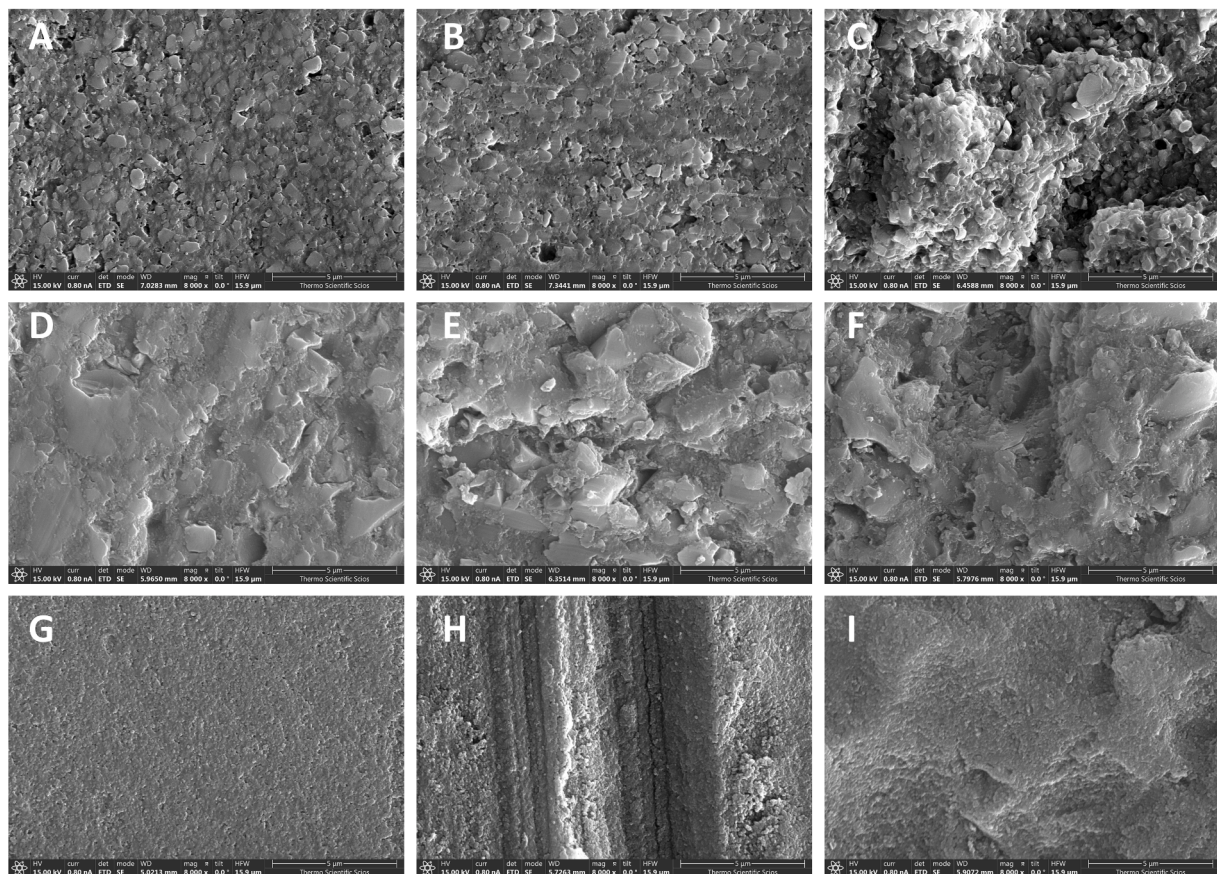


Fig. 4. Representative SEM micrographs of RMC surfaces at 8000 x magnification (scale bar = 5 μ m). CS: (A) polished, (B) diamond-roughened, and (C) sandblasted surfaces. GB: (D) polished, (E) diamond bur roughened, and (F) sandblasted. KA: (G) polished, (H) diamond bur roughened, and (I) sandblasted surfaces.

Table 3

Surface roughness parameters (Mean, Standard Deviation - nm) of RMCs following different surface conditioning protocols.

Material	Surface treatment	Ra		Rq		Rp		Rv	
		mean	std deviation	mean	std deviation	mean	std deviation	mean	std deviation
Cerasmart	Polishing	675.65	266.74	792.58	295.01	1230.00	351.11	-2058.86	746.99
	Diamond bur roughening	1218.15	518.45	1517.81	670.99	3176.92	1009.92	-4361.18	1820.22
	Sandblasting	3988.59	1810.89	4631.91	1914.81	7539.97	3232.21	-11,000.06	3045.62
Grandio blocs	Polishing	283.021	45.75	332.48	42.55	799.68	99.83	-900.37	144.96
	Diamond bur roughening	835.40	80.02	1046.27	100.18	2926.59	341.79	-3488.75	319.39
	Sandblasting	4975.66	2408.50	5855.74	2725.73	11,555.66	4909.63	-9717.19	5817.89
Katana Avencia	Polishing	190.60	39.67	232.83	46.86	557.67	97.74	-769.18	144.93
	Diamond bur roughening	806.58	268.30	1024.86	441.35	2107.42	520.32	-3075.56	2056.70
	Sandblasting	2200.92	562.97	2652.61	634.68	6130.31	1204.86	-7905.9	1797.53

For CS, sandblasted surfaces exhibited a mean Ra value of 3988.59 ± 1810.89 nm, which was significantly higher than both the diamond bur roughened (1218.15 ± 518.45 nm) and polished (675.65 ± 266.74 nm) groups ($p = 0.020$ and $p < 0.001$, respectively). In addition, diamond bur roughening produced significantly greater surface roughness than polishing ($p = 0.033$).

Cerasmart: Sandblasted surfaces exhibited a mean Ra of 3988.59 ± 1810.89 nm, which was significantly higher (Tamhane, $p = 0.02$ and $p < 0.01$, respectively) than both the diamond bur roughened (1218.15 ± 518.45 nm) and polished groups (675.65 ± 266.74 nm). Whereas the diamond bur roughened and polished surfaces also show a significant difference in the mean values (Tamhane, $p = 0.033$).

Grandio blocs: The highest absolute roughness values in this study were observed in the sandblasted GB group (4975.66 ± 2408.50 nm). This was significantly higher than the diamond bur roughened (835.40 ± 80.02 nm) and polished surfaces (283.02 ± 45.75 nm), furthermore, the latter two are also show significant difference in the mean (Tamhane test gave for all pairs $p \leq 0.001$).

Katana Avencia: Sandblasting produced a mean of (2200.92 ± 562.97 nm), significantly exceeding other treatments ($p < 0.001$). In contrast to the other materials, for KA, the difference between diamond bur roughened (806.58 ± 268.30 nm) and polished surfaces (190.60 ± 39.67 nm) was statistically significant (Tamhane test gave for all pairs $p < 0.001$).

4. Discussion

Notwithstanding the widely accepted view that mechanical and chemical strategies are required for achieving high repair bond strength when repaired with a resin composite [26–31], evidence about how best to perform a state-of-the-art repair for the various RMCs remains evolving. The objective of this study was to evaluate the influence of various mechanical and chemical surface conditioning strategies on the repair bond strength of three distinct RMCs (CS, GB, and KA) and a nanohybrid resin composite.

In the present study, the CS groups treated with Clearfil Universal Bond Quick (C1–C3) demonstrated the highest μ TBS values, irrespective of surface treatment. Clearfil Universal Bond Quick is a universal adhesive containing 10-MDP and silane, a combination that may promote both chemical interaction and improved wetting of the treated RMC substrate. The phosphate functional group of 10-MDP can form chemical bonds with oxide-containing surfaces, a mechanism well established for ceramics such as zirconia and lithium disilicate, where phosphate groups react with surface oxides to produce durable interfacial bonding [32].

In the present study, the favourable performance observed for CS may also relate to its material-specific characteristics, including microstructure and filler characteristics. CS contains an inorganic filler phase (approximately half barium glass) [33], and its reported particle size (≈ 300 nm) may contribute to the greater roughness observed in our profilometric analysis compared with other RMCs subjected to the same treatments. Overall, enhanced micromechanical retention combined

with improved wettability and chemical coupling (silane) may explain the consistently high μ TBS values obtained for CS when an MDP/silane-containing adhesive was used (Fig. 5).

Our findings are broadly consistent with those of Doğan et al. [28] who reported similar μ TBS values for sandblasted CS treated with a comparable protocol (40.79 ± 6.67 MPa versus 46.43 ± 3.13 MPa in the present study), although their polished (12.37 ± 2.40 MPa) and diamond-roughened (24.38 ± 3.95 MPa) groups yielded lower values than ours. Such differences may reflect methodological variation (e.g., surface-preparation parameters, ageing protocols, or cement/composite systems), reinforcing that both the substrate and protocol details influence final bond strength.

Surface treatment remains a key determinant of bonding effectiveness. Sandblasting is widely used to increase surface area and surface energy by creating microroughness and partially removing the smear layer, thereby improving mechanical interlocking and bonding performance [34–36]. However, inappropriate sandblasting parameters (pressure, particle type, distance, and duration) can damage RMC surfaces and compromise mechanical integrity [37]. For repair of CAD-CAM materials, aluminium oxide sandblasting is commonly considered the most suitable abrasive for airborne-particle abrasion [19, 20], and our results support its effectiveness. In agreement with Şismanoğlu et al. [21], sandblasted CS generally exhibited higher roughness and improved bond strength compared with less aggressive surface treatments.

Notably, in some CS groups using the same adhesive system (C5 and C8), diamond-bur roughening produced higher μ TBS values than sandblasting (C6 and C9). The literature is mixed on the relative performance of diamond roughening versus airborne abrasion. Erdemir et al. [38] reported that although bur preparation may generate high roughness, tensile strength can remain lower than that achieved after sandblasting, while Bankoğlu Güngör et al. [22] found that bur roughening produced both higher surface roughness and higher bond strength for RMCs. These discrepancies likely reflect differences in bur type and grit, load and direction of preparation, subsequent cleaning protocols, and the interaction of each surface morphology with the chemistry of the adhesive/cement system.

With respect to chemical pretreatment, HF etching followed by silanisation did not yield the highest values for CS in the present study, which aligns with the material's filler chemistry. Güngör et al. reported μ TBS values of 13.64 ± 0.95 MPa following polishing, ageing, HF etching and silanisation, whereas our comparable group achieved 17.49 ± 5.75 MPa [22]. One explanation for lower HF effectiveness is that a substantial proportion of CS's inorganic phase is barium glass, which may be less reactive to HF than silica-rich phases [33], limiting the degree of etching-derived microporosity and subsequent micro-mechanical retention.

The role of silane remains nuanced. While some studies suggest that silane alone may not significantly improve bond strength [23], universal adhesives incorporating functional monomers can enhance bonding, and the most favourable outcomes have been reported when silane is used in conjunction with a universal adhesive [24]. This may be

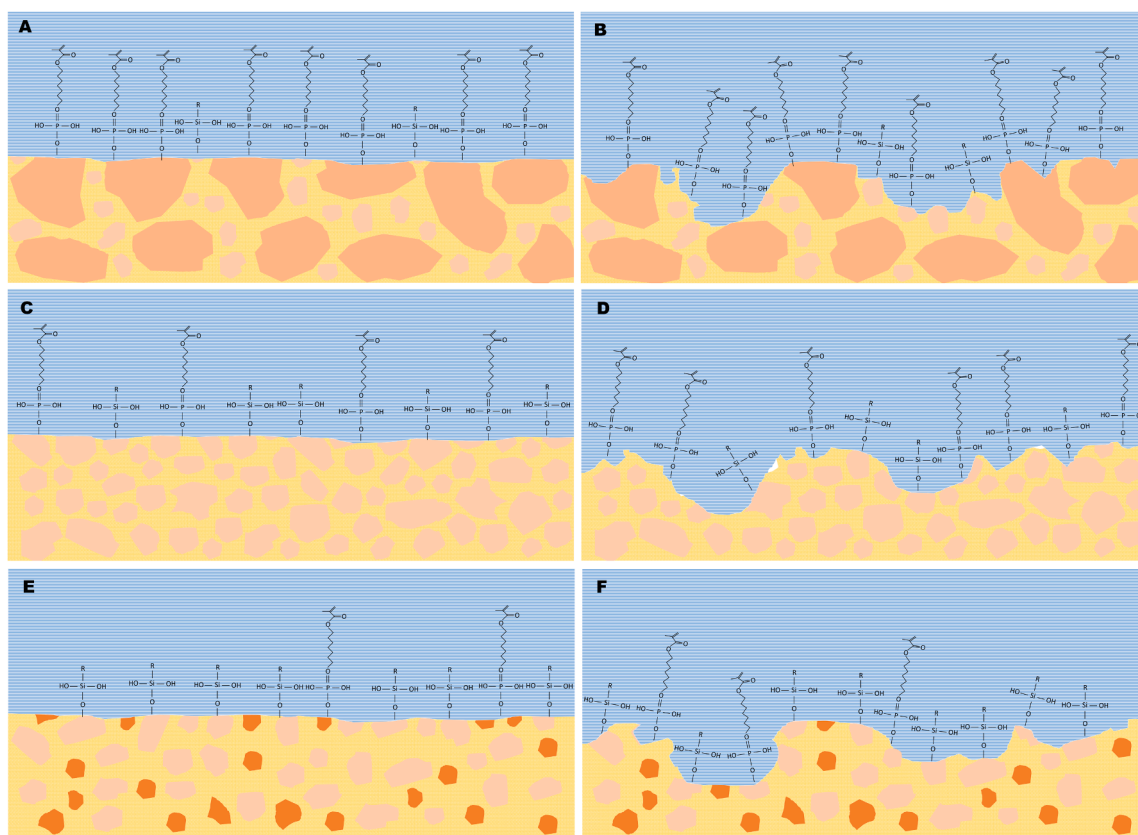


Fig. 5. Schematic representation of polished and roughened surfaces of the RMCs after application of an MDP- and silane-containing adhesive: CS -polished (A), roughened (B); GB - polished (C), roughened (D); KA - polished (E), roughened (F).

attributable to multiple factors: acidic conditions facilitating initial reactions, and the hydrophobic nature of 10-MDP contributing to improved resistance to hydrolytic degradation, while phosphate ester groups support adhesion to hydroxylated surfaces of non-glass ceramics [39–41]. In this context, the consistently strong performance of MDP and silane-containing adhesive protocols supports the clinical value of combining mechanical and chemical strategies.

For GB, the highest μ TBS values were observed in the sandblasted group treated with Clearfil Universal Bond Quick (G3). More generally, sandblasted groups across adhesive protocols (G6, G9, G12) tended to outperform polished or diamond roughened counterparts, suggesting that for this material, surface roughening via airborne abrasion is a particularly influential determinant of interfacial strength. This pattern implies that the GB surface benefits substantially from increased surface area and microretentive topography, which may be critical for reliable bonding.

For KA, the highest μ TBS values were achieved with HF etching followed by silanisation (K7–K9). In addition, the combined use of sandblasting and Clearfil Universal Bond Quick (K3) also resulted in favourable bond strengths. These findings emphasise that, beyond mechanical pretreatment, chemical modification can significantly influence bonding performance, and that the effect of HF differs from airborne abrasion. HF selectively dissolves susceptible filler phases, producing microporosity and a microretentive surface that can enhance resin infiltration and micromechanical interlocking [42–44]. Importantly, the optimal HF concentration is material dependent. Prior research has shown that HF etching reduces silicon content to varying degrees across RMCs and that KA may require a higher HF concentration (3.5%) to achieve higher bond strength compared with other blocks (CS 2%, Shofu Block HC 3%, Vita Enamic 0.5%) [33]. In the present study, the comparatively strong response of KA to HF + silane may reflect a higher proportion of HF-reactive silica-containing phases relative to CS,

where a substantial fraction of the inorganic content is barium glass [33]. Despite the favourable bond strengths observed following HF etching, its clinical use - particularly for intraoral repair procedures - requires considerable caution because of its highly corrosive and cytotoxic properties [45,46]. Therefore, meticulous isolation using a rubber dam is essential to protect the surrounding soft tissues and should be regarded as mandatory whenever HF is applied intraorally [47]. Furthermore, when repair procedures involve adjacent dentine surfaces, inadvertent contact of HF with dentine should be avoided, as HF causes dentine demineralisation and has been shown to reduce subsequent adhesive bond strength [48].

More broadly, adhesion to RMCs may be influenced by differences in microstructure, filler particle size and composition, and the organic-inorganic ratio [49]. Increasing inorganic content and larger filler particles may enhance silane coupling efficacy [49]. As a bifunctional coupling agent, silane can co-polymerise with methacrylate groups in the resin cement and interact with the inorganic phase via hydroxyl groups, while also improving surface wetting and facilitating resin penetration into microporosities created by mechanical or chemical pretreatment [50]. These mechanisms are consistent with our observation that protocols integrating surface roughening and silane + MDP-based chemistry produced the most favourable bonding outcomes overall. It is also noteworthy that the high rate of cohesive failure in the Clearfil-treated groups (especially C3 and K3) indicates that the adhesive bond strength exceeded the cohesive strength of the RMC material itself.

Based on our findings, both null hypotheses were rejected, as surface treatment and adhesive protocol significantly influenced the bond strength between RMCs and the nanohybrid composite repair material. Differences among materials likely reflect variations in composition, microstructure and manufacturing technology. Overall, sandblasting combined with an adhesive containing silane and 10-MDP produced

consistently high μ TBS values across RMCs, supporting the use of combined mechanical and chemical conditioning strategies for repair procedures.

5. Limitations of the study and future investigations

This study has several limitations. Firstly, it was conducted *in vitro* and therefore cannot fully reproduce the complex intraoral environment (saliva, pH fluctuations, biofilm, and functional occlusal loading), which may influence both bonding durability and failure patterns. Secondly, the specific thermocycling protocol utilised to simulate thermal ageing relates to immediate (short-term) repair bond strength under *in vitro* conditions. Thus, the study findings do not relate to long-term durability of the repaired adhesive interface and, consequentially, clinical implications should be interpreted accordingly. Future studies should incorporate prolonged water storage, cyclic mechanical loading, and longer-term ageing protocols to evaluate the long-term durability of the repair interface. Thirdly, only a single nanohybrid composite resin was used as the repair material; different repair resin composites (with varying viscosity, filler content and monomer chemistry) may yield different bond strengths and limit generalisability. Fourthly, airborne-particle abrasion was performed using one parameter set (50 μ m aluminium oxide, 2–3 bar, 10 mm distance, 10 s), and outcomes are known to be sensitive to these variables; therefore, the findings may not translate directly to other sandblasting protocols. Furthermore, only the tested adhesives/conditioning protocols were evaluated; other universal adhesives, dedicated primers (separate silane/MDP), or alternative systems could perform differently. Finally, the Profilometry and SEM performed describe topography, but they do not provide data on surface energy/wettability (contact angle) or chemical composition changes, which limit the mechanistic interpretation. Future *in vitro* studies should incorporate longer-term ageing, cyclic loading, a broader range of repair materials and surface-conditioning parameters and employ measurements to assess surface energy and wettability. In terms of clinical research, future clinical studies are necessary to determine the long-term longevity of these repair protocols in a dynamic intraoral environment.

6. Conclusion

Within the limitations of this *in vitro* study, both mechanical surface pretreatment and adhesive strategy significantly influenced the immediate repair bond strength of RMCs. The optimal repair protocol was material dependent, indicating that no single conditioning strategy is universally applicable to all RMCs. Sandblasting consistently produced the highest bond strengths and, when combined with a 10-MDP- and silane-containing universal adhesive, yielded the most favourable immediate repair bond strengths for Cerasmart and Grandio blocs. In contrast, Katana Avencia demonstrated the highest immediate bond strength following sandblasting combined with HF etching and silanisation. These findings suggest that repair protocols should be tailored to the specific RMC whenever possible. However, further studies incorporating long-term ageing and mechanical fatigue are required to confirm the durability and clinical relevance of these repair strategies.

Funding

This research was supported by the Ministry of Culture and Innovation of Hungary through the National Research, Development and Innovation Fund, under grant agreement [TKP2021-\(TKP2021- EGA funding scheme\)](#).

CRediT authorship contribution statement

Gábor Suta: Writing – original draft, Visualization, Resources, Methodology, Investigation, Conceptualization. **Tünde Radics:** Writing – review & editing, Visualization, Resources. **József M. Gáll:** Writing –

review & editing, Visualization, Formal analysis. **Attila Csík:** Writing – review & editing, Visualization, Validation, Resources, Investigation. **Igor R. Blum:** Writing – review & editing, Investigation. **Csaba Hegedűs:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Given his role as Editorial Board member, Igor Blum is not involved in the peer review of this article and had no access to information regarding its peer review. Full responsibility for the editorial process for this article was delegated to another journal editor.

Acknowledgements

None.

Data availability: The data supporting the findings of this study are available from the corresponding author upon request.

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