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In Vitro Evaluation of Shear Bond Strength and Mode of Failure of the Interface between an Indirect Composite Bonded to Fiber-Reinforced Composite Substructures

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Abstract

Purpose: Failures of fixed partial dentures (FPDs) fabricated with fiber-reinforced composites (FRCs) have been attributed to veneering fractures. The aim of the present study was to investigate the shear bond strength and mode of failure between an indirect composite and FRC substructures.
Material and Methods: SR Adoro indirect composite was bonded to the following substructures: (a) flat surface made of unidirectional glass fibers (group A), (b) retentive sticks made of unidirectional glass fibers (group B), (c) flat surface made of fiber net (group C), (d) retentive sticks made of fiber net (group D), (e) nickel-chromium dental alloy (control, group E). For every group, 13 specimens were fabricated. All specimens were hydrothermocycled (5000 cycles, 5°C/30sec, and 55°C/30sec). A bond test was performed in a testing machine at a 0.5 mm/min crosshead speed according to ISO 10477. The failure mode was determined by examination of the fractured surfaces under an optical microscope. Selected specimens were examined with scanning electron microscope and with energy dispersive spectroscopy for compositional determination. The morphology (flat-sticks) and the type (unidirectional-net) of fibers on the bond strength were estimated.
Results: The mean shear bond strength was significantly different between groups E and A ($p = 0.044$), and groups A and B ($p = 0.010$). All FRC specimens showed cohesive failure. Group E showed predominantly adhesive failure. The bond strength was higher when sticks or fiber nets were used.
Conclusions: Fiber nets and retentive sticks increase the shear bond strength between FRCs and indirect composite. *Clinical implications:* In FPDs, the morphology and type of FRC substructures might influence the shear bond strength between the FRC substructure and the indirect veneering composite. With the proper design of these substructures, the number of veneering fractures may be decreased.

The continuously growing demand for more-esthetic restorations has led to the development of new, metal-free prosthetic dental products. Metal-free fixed partial dentures (FPDs) may be fabricated either by all-ceramic systems or by fiber-reinforced composites (FRCs).

The use of FRCs in dentistry has been described in the literature for at least 30 years. FRCs consist of fibers embedded in a resinous matrix providing satisfactory mechanical properties.[1]

The reinforcing properties of FRCs are influenced by several factors, such as the type and quantity of the fibers, their impregnation by the resinous matrix, their adhesion to it, and their orientation. Theoretically, unidirectional fibers reinforce the composite by 100% in one direction (anisotropic mechanical properties), whereas a fiber net reinforces the composite by 50% or 25% in two directions.

As most FRCs are translucent materials, they exhibit good esthetics based on their "shine-through" effect.[2] Their indications include endodontic dowels,[3] splints,[4] crowns, denture bases,[1] prosthodontic frameworks on implants,[5] and fixed prosthodontic restorations.[6,7]

FRCs are used as substructures beneath the newer hybrid particulate filler composite veneering materials.[8] Several

commercial FRC products have been introduced for clinical use. The most common include glass fibers preimpregnated with monomer or polymer systems. A number of clinical reports[9-12] indicate that the most common failures of these restorations are due to veneer fractures.[13,14] These failures can be attributed to improper framework design,[15-17] high bending tendencies of the framework materials,[18] and insufficient bond strength between framework and veneering material.[13,14,19] Although several attempts have been made to increase the bond between the FRC substructure and the veneering composite resin, this issue is still of great concern.[20-22] In a previous paper,[23] the shear bond strength of a light-cured veneering composite bonded to FRC substrates was tested.

Consequently, the aim of this study was to investigate the shear bond strength of a commercial indirect composite bonded to different FRC substructures and to characterize the mode of failure of the fractured surfaces. The null hypothesis was that the different types of FRC substructures do not affect the shear bond strength between them and the composite material. The mode of failure is expected to be the same for all combinations of FRC substructures and veneering composite.

Materials and methods

The composition of the materials used is described in Table 1. An indirect composite material (SR Adoro) was bonded to five FRC substructures. According to the manufacturers, SR Adoro is a laboratory resin able to bond successfully to both base metal alloys and FRC substructures. 4all is a nickel-chromium alloy proposed by the manufacturers to be used, among other materials, with SR Adoro. According to the literature, fiber direction influences the bond strength between a laboratory composite and an FRC framework.[22] In Vectris Pontic and Vectris Frame, the fiber directions are specific. So, the use of these products permits specimens with specific and reproducible fiber directions.

For the purpose of the present study, 65 specimens were fabricated and divided into five equal groups as follows: In group A, SR Adoro was placed on an FRC substructure with a flat surface made of unidirectional glass fibers (Vectris Pontic) (Fig 1A). In group B, SR Adoro was bonded to an FRC substructure with a surface made of unidirectional glass fibers (Vectris Pontic) containing retentive rectangular sticks, 0.5×0.5 mm^2 in cross section and 10 mm in length, positioned parallel to each other at a distance of 0.5 mm (Fig 1B). In group C, SR Adoro was placed on an FRC substructure with a flat surface of glass fiber net (Vectris Frame) (Fig 1C). In group D, SR Adoro was bonded to an FRC substructure of glass fiber net (Vectris Frame) with retentive sticks similar to those in group B (Fig 1D). Group E (control group), consisted of specimens where SR Adoro was bonded to a cast dental Ni-Cr alloy (4all).

For the fabrication of the specimens of group A, three 10 mm long pieces of unpolymerized FRC Vectris Pontic were pressed by a transparent polyester sheet (Mylar) and a glass plate into a silicon mold, moistened with Vectris Glue (Ivoclar Vivadent) for the FRC substructures to form rectangles of $10 \times 10 \times 1$ mm^3. The mold was placed in a Vectris VS1 oven (Ivoclar Vivadent) for 10 minutes under vacuum, for the thermo-photo polymerization of the material according to the manufacturer's instructions.

For the specimens of group B, a translucent impression was made as described below. A metallic jig with dimensions $10 \times 10 \times 1$ mm^3 was cast containing retentive sticks (10 mm long and 0.5×0.5 mm^2 in cross section) in one of the surfaces. The retentive sticks were positioned parallel at a distance of 0.5 mm to each other. The jig was placed into the silicon mold used previously with the retentive sticks looking upward. A separator was applied both on the silicon mold and the metallic jig, and an impression of the retentive sticks was taken with a translucent vinyl(poly siloxane) material (Transil, Ivoclar Vivadent). The translucent impression was fitted exactly on the silicon mold. The translucent impression was used to press three pieces of unpolymerized FRC Vectris Pontic 10 mm long into the silicon mold. The long axes of the fibers were placed parallel to the long axes of the retentive sticks. The material was then polymerized in the Vectris VS1 oven for 10 minutes.

Group C specimens were prepared by pressing two 10 mm long pieces of unpolymerized FRC Vectris Pontic with a transparent polyester sheet (Mylar) and a glass plate into the silicon mold moistened with Vectris Glue. Then the mold was placed in the Vectris VS1 oven for 10 minutes under vacuum. After polymerization, a Vectris Frame was pressed on to the polymerized Vectris Pontic with the Mylar sheet and the glass plate. The specimens were placed in the Vectris VS1 oven for 10 minutes for additional polymerization according to manufacturer's instructions.

Group D specimens were prepared by pressing with the translucent impression two 10 mm long pieces of unpolymerized FRC Vectris Pontic into the silicon mold moistened with Vectris Glue and then polymerizing them in the Vectris VS1 oven for 10 minutes under vacuum. After polymerization a Vectris Frame was pressed onto the polymerized Vectris Pontic using the translucent impression, followed by additional polymerization for 10 minutes in the same oven.

The excess material was removed from the FRC substructure of the specimens of groups A to D with carbide burs. The specimens were carefully air abraded with 100 μm Al_2O_3 particles at 1 bar according to the manufacturer's recommendations. The residues were removed by tapping, and Vectris wetting liquid (Ivoclar Vivadent) was applied immediately afterward. The liquid was allowed to set for 60 seconds.

Group E castings were rectangles $10 \times 10 \times 1$ mm^3, with retention beads (200 μm to 300 μm in diameter) in one of the flat surfaces, which was air braded with 100 μm Al_2O_3 particles. All residue was removed by tapping it off, and a bonding agent (SR Link, Ivoclar Vivadent) was applied all over the surface and allowed to set for 3 minutes.

An adhesive tape with a 5 mm diameter inner hole was placed on the specimen to define the bonding area. A thin layer of SR Adoro liner was applied on the specimens of groups A to D. The liner was polymerized for 20 seconds using a halogen light-curing unit (Trilight, 3M ESPE, Seefeld, Germany). Two layers of opaquer were applied to the specimens of group E. Each layer was polymerized for 20 seconds using the light-curing unit. The specimens were polymerized afterward in a Targis Power Upgrade furnace (Ivoclar Vivadent) for 11 minutes.

Table 1 Materials used

Materials	Composition	Manufacturer	Lot no.
4all (Ni-Cr base metal alloy)	Ni: 61 wt% Cr: 25.7 wt% Mo: 11 wt% Si: 1.5 wt%	Ivoclar Vivadent, Schaan, Liechtenstein	J14779
SR Adoro Opaquer (Low-viscosity laboratory resin)	55 wt% dimethacrylates (Bis-GMA, TEGDMA, UDMA) and 43 wt% inorganic fillers	Ivoclar Vivadent, Schaan, Liechtenstein	F15584
SR Adoro dentin A3,5 (high-viscosity laboratory resin)	17–19 wt% dimethacrylates (UDMA); 64–65 wt% inorganic fillers	Ivoclar Vivadent, Schaan, Liechtenstein	G26586
SR Adoro liner (low-viscosity laboratory resin)	48 wt% dimethacrylate (Bis-GMA, TEGDMA, UDMA); 51 wt% inorganic fillers	Ivoclar Vivadent, Schaan, Liechtenstein	F40868
SR Link	Bonding agent based on a phosphoric ester	Ivoclar Vivadent, Schaan, Liechtenstein	H 15063
Vectris Pontic (unidirectional glass fibers)	64–66 wt% glass fibers and 30–32 wt% dimethacrylate	Ivoclar Vivadent, Schaan, Liechtenstein	F94028
Vectris Frame (glass fiber net)	49–51 wt% glass fibers and 44–46 wt% dimethacrylate (Bis-GMA and TEGDMA)	Ivoclar Vivadent, Schaan, Liechtenstein	F94027
SR Gel	Glycerine, silicon dioxide, and aluminum oxide	Ivoclar Vivadent, Schaan, Liechtenstein	G28777
SR Adoro Thermo Guard	Diethylene glycol, water, inorganic filler, synthetic fibers	Ivoclar Vivadent, Schaan, Liechtenstein	F41798
Vectris Glue	38–40 wt% dimethacrylate, 59–61 wt% inorganic filler (barium glass filler and silicon dioxide)	Ivoclar Vivadent, Schaan, Liechtenstein	E58546
Transil	Vinyl(poly siloxane)	Ivoclar Vivadent, Schaan, Liechtenstein	G09604

The inhibition layer was removed from all the specimens. SR Adoro dentin was condensed through a tubular mold of celluloid (5 mm diameter, 3 mm height), forming two layers of 1.5 mm in height. Every layer was photo cured for 20 seconds. SR Gel was applied on the entire surface of specimens of groups A, B, C, and D as well as to the SR Adoro surface of the specimens of group E. SR Adoro Thermo Guard was also applied on the metal surface of the specimens of group E. All specimens were polymerized in a Targis Power Upgrade furnace for 25 minutes.

The finished specimens were checked and adjusted to the predetermined dimensions. They were then hydrothermocycled for 5000 cycles in baths of 5°C and 55°C, remaining in each bath for 30 seconds with a 2 second dwell time. Shear strength testing was performed using a universal testing machine (Tensometer 10, Monsanto, Akron OH) exerting a constant load applied at the interface between veneering and substructure materials at a 0.5 mm/min crosshead speed according to ISO 10477. The direction of the fibers and the sticks were put randomly to the applied load.

The fractured surfaces of the specimens (the FRC substructure surface and the veneering composite surface) were examined under an optical microscope (ZEISS Stemi 2000-C, Toronto, Canada) at ×16 magnification to determine the mode of failure. Selected areas of the fractured surfaces were examined in a scanning electron microscope (Quanta 200, FEI, Hillsboro, OR), and subjected to energy dispersive x-ray spectroscopy (EDS) (Sapphire CDU, Edax Int., Mahwah, NJ) to analyze the elementary composition of the remaining material. The failure mode was determined arbitrarily according to the sum of the percentage of the remaining indirect composite on the FRC substructure surface plus the percentage of the remaining fibers of the FRC material on the indirect composite surface (%IC + F). Particularly, if the sum of these percentages was less than 25%, the failure mode was considered adhesive, and if it was more than 75%, the failure mode was cohesive. On the other hand, when the sum was between 25% and 50%, the failure mode was predominantly adhesive, and when it was between 50% and 75%, the failure mode was predominantly cohesive. In the specimens of the control group, because no metal was detached on the veneering composite surface, the mode of failure was determined according to the percentage of the remaining indirect composite on the metal substructure surface (cohesive when it was higher than 75%, adhesive when it was lower than 25%, predominantly adhesive when it was between 25% and 50%, and predominantly cohesive when it was between 50% and 75%). A software analysis program (Image J, Wayne Rasband, National Institute of Health, Bethesda, MD) was used to calculate these percentages.

For comparison of the shear strength among the five groups, one-way ANOVA was conducted along with Tukey's *post hoc* test. In addition, the effect of the surface morphology (flat surface and surface with sticks) and fiber type (unidirectional fibers and fiber net) on the shear bond strength was investigated among the four fiber-reinforced groups by using two-way factorial ANOVA. All statistical tests were carried out at a significance level of 0.05. Statistical analysis was performed using SPSS for Windows (Rel. 15.0.0. 2006. SPSS Inc., Chicago, IL).

Results

The mean bond strengths and standard deviations of the five groups are presented in Table 2. Representative images of the

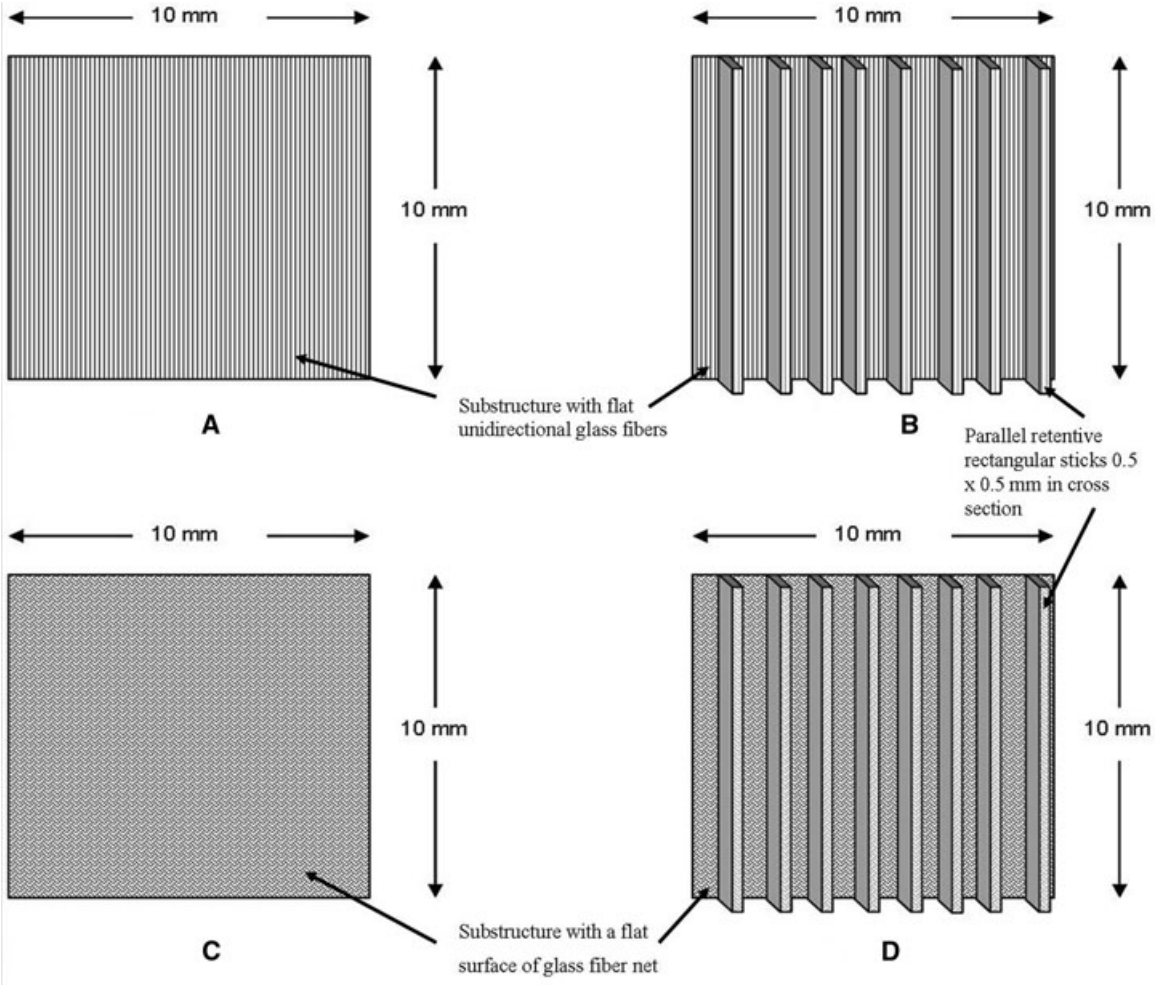

Figure 1 Schematic illustration of the FRC substructure of groups A to D.

Table 2 Mean shear bond strength (MPa) and standard deviation of the groups tested

Groups	Mean ± SD
Group A	11.83 ± 4.2^{a}
Group B	20.86 ± 8.2^{b}
Group C	$16.66 \pm 8.9^{a,b}$
Group D	$16.74 \pm 5.8^{a,b}$
Group E	19.29 ± 5.6^{b}

The same superscript letters indicate no statistically significant differences between the tested groups.

fractured surfaces of the specimens of groups B and D in the optical and scanning electron microscopes, as well as their elemental analysis are presented in Figures 2 and 3. EDS revealed that the surfaces with fibers contained Mg and Ca, whereas the veneering composite areas did not. The failure mode of all specimens belonging to the FRC groups was cohesive (IC + F > 75%), and the failure mode of the control group was predominantly adhesive.

One-way ANOVA of bond strength results revealed significant differences among the tested groups ($p = 0.015$). More specifically, the mean shear bond strength was statistically different between groups E and A ($p = 0.044$), as well as between groups A and B ($p = 0.010$). Two-way ANOVA revealed that the effect of FRC morphology (flat surface and surface with sticks) of the specimens on the bond strength was statistically significant ($p = 0.024$), whereas that of fiber type (unidirectional fibers and fiber net) was not ($p = 0.856$); however, the interaction between surface morphology and fiber type was statistically significant ($p = 0.026$). When unidirectional glass fibers were used, the strength was significantly affected by the surface. More specifically, the strength was statistically greater ($p = 0.002$) when sticks were used on Vectris Pontic (20.86 MPa ± 8.20), comparable to the use of flat Vectris Pontic surfaces (11.83 MPa ± 4.15). There was no statistically significant difference when the fiber net was used ($p = 0.978$).

Discussion

SR Adoro is a modern indirect resin composite. It consists of an aromatic-aliphatic urethane dimethacrylate monomer and a high percentage of inorganic fillers (65 wt%) in the nanoscale

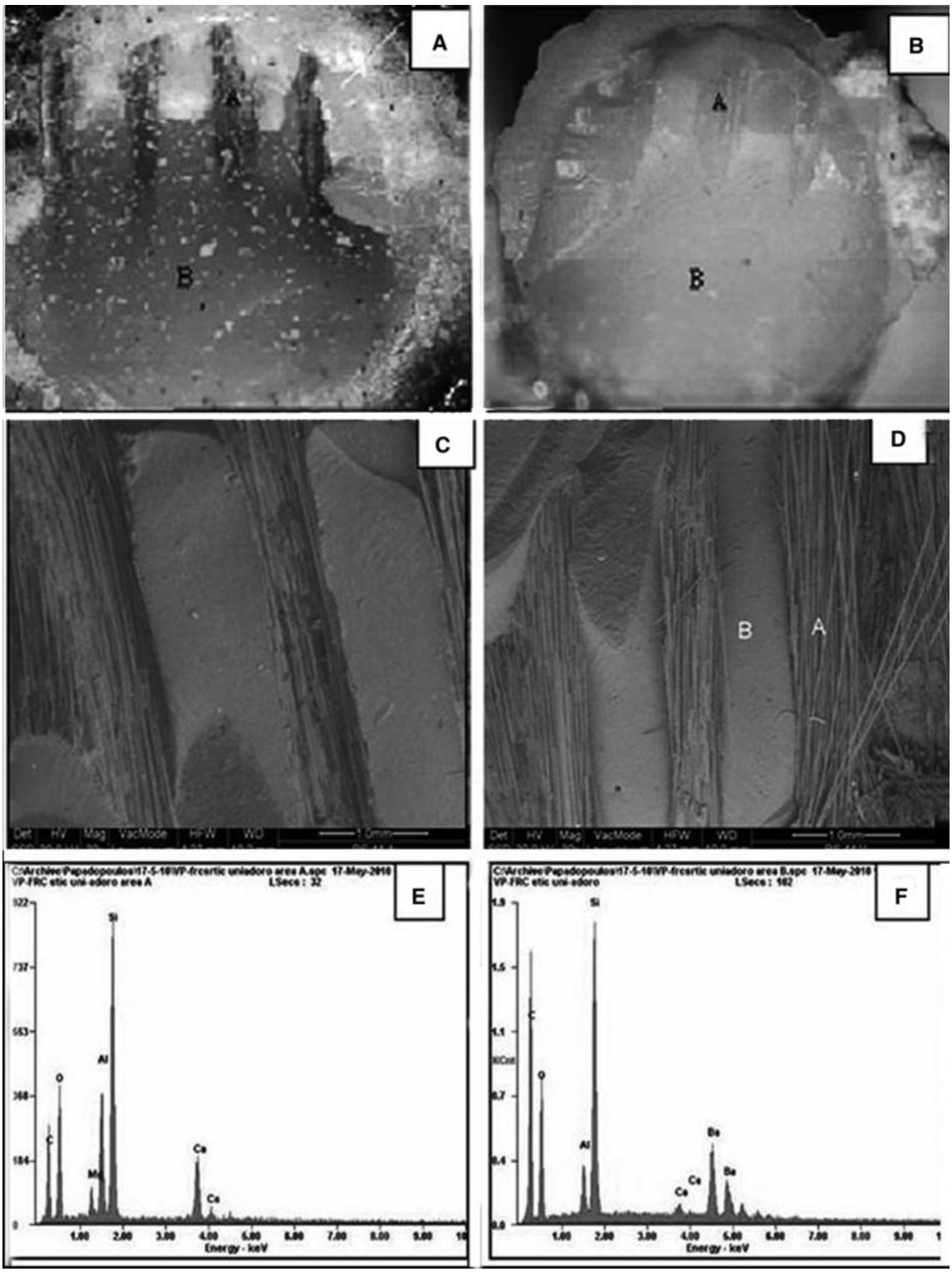

Figure 2 Optical microscope (A, B), SEM (C, D), and EDS (E, F) images of a specimen of group B at substructure (A, C, E) and indirect composite (B, D, F) fractured surfaces. Unidirectional fibers (B) interlocked into the retentive sticks (A) on both the indirect composite surface and the FRC substructure fractured surfaces can be seen.

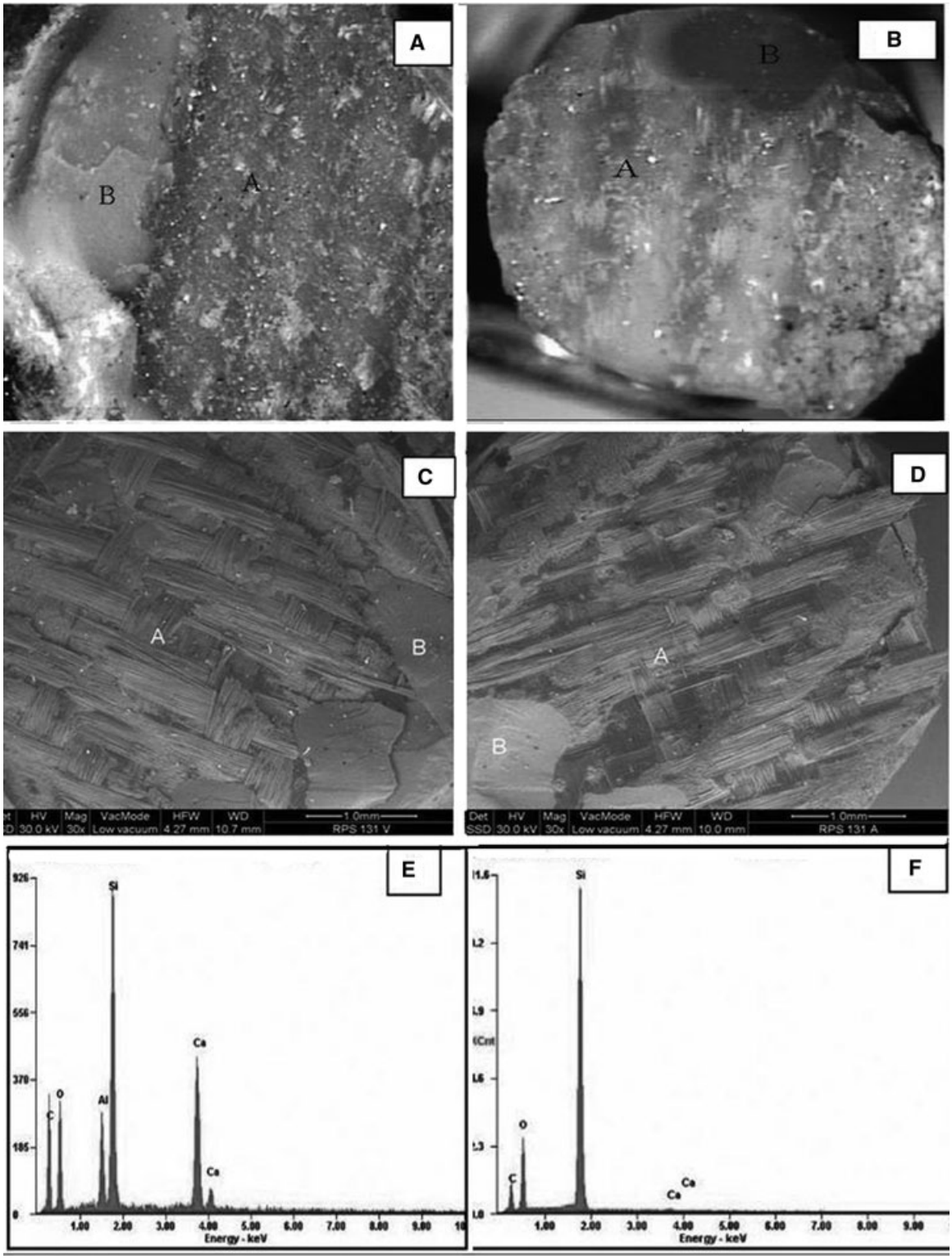

Figure 3 Optical microscope (A, B), SEM (C, D), and EDS (E, F) images of a specimen of group D at substructure (A, C, E) and composite (B, D, F) fractured surfaces. The texture of the fiber net (A) and the morphology of the retentive sticks (B) can be seen.

range that, according to the manufacturers, improves its mechanical and physical properties.[25] SR Adoro can be bonded successfully to base metal alloys using a chemical bonding agent based on a phosphoric ester with a methacrylate function (SR Link bonding system) and small beads (200 μm to 300 μm in diameter) for micromechanical retention. Metal-composite bonding agents and especially agents based on phosphoric esters, provide adequate bond strength and reduce microleakage at the metal/resin interface, preventing discoloration of the veneering composite.[26] Vectris, on the other hand, consists of E-glass fibers embedded in an organic polymer matrix (dimethacrylate monomer). E-glass fibers contain MgO and CaO, which may be prone to hydrolysis.

The bond between SR Adoro and the Vectris FRC substructure involves a resin/resin bond and a glass/resin bond. The resin/resin bond exists between the polymer matrix of the Vectris FRC (Bis-GMA) and that of the SR Adoro (urethane dimethacrylate, UDMA). The matrices of both materials (SR Adoro, Vectris) are compatible, and so a stronger resin/resin bond can be made. The uncured composite material remaining on the surface of the FRC (inhibited layer), which reacts chemically with the monomers in the resin, is mainly responsible for this bond. On the other hand, the highly cross-linked polymer matrix of Vectris might inhibit a strong resin/resin bond. Furthermore, as the polymer matrix of Vectris is already polymerized, it might be difficult to be dissolved by the matrix of SR Adoro liner (Bis-GMA, TEGDMA, UDMA). The glass/resin bond is formed between the glass fibers and the resin matrix, through silane agents.[27] Particularly, silane forms a covalent bond at the glass surface and in turn demonstrates a functional methacrylate group, which may co-polymerize with the methacrylate of the resin matrix. In addition, silane agents increase the wettability of the surface of the glass fibers, permitting resin to penetrate them.

All the FRC specimens exhibited a cohesive mode of failure, indicating that the FRC-indirect composite bond was quite strong. This finding is in accordance with the null hypothesis stating that the mode of failure was expected to be the same for all FRC groups. Several factors might have participated in the strengthening of the bond between Vectris FRC and SR Adoro composite. Chemical similarities between the polymer matrices of the Vectris FRC and SR Adoro may have improved the bond. On the other hand, the use of a Mylar sheet and the translucent impression during the polymerization process do not permit oxygen to inhibit radical polymerization of the FRC, allowing for a very thin inhibited layer to remain on the surface. Additionally, air abrasion might also have increased the bond strength by increasing the roughness of the FRC substructure.[28] Other investigators presume that air abrasion of the FRC substructure decreases the shear bond strength by removing the inhibited layer.[14] Furthermore, the SR Adoro-Vectris system uses a low viscosity resin (SR Adoro liner) that might penetrate into the FRC substrate (intermediate resin, IMR) and produce a stronger interpenetrating polymer network (IPN). According to many studies, the IPN considerably increases the veneering composite/FRC bond strength.[19,29,31] As all FRC specimens had a cohesive and not adhesive failure mode, the veneering composite/FRC bond was not the problem. Further investigation is necessary to establish whether the weakest link was the veneering composite or the substructure material, or both.

The mode of failure of the control group was predominantly adhesive. When the fractured surfaces of the FRC specimens (the FRC substructure surface and the veneering composite surface) were examined under the optical microscope, fibers detached from the FRC substructure and attached to the composite fractured surface were noted. On the other hand, there was no metal detected on the composite fractured surface of the control group. So, metal substructure is more stable than the FRC substructure. This fact might also explain the cohesive mode of failure of the FRC specimens.

According to the results of the present study, the bond strength of the Ni-Cr base alloy specimens (group E) was significantly higher only when compared to group A (flat surface and unidirectional fibers). When sticks or fiber net were used, FRCs behaved almost identically to the base alloy, as no statistical difference was found between groups B to E. This finding was in contrast with the null hypothesis in which the different types of the FRC substrates were not expected to play any role in the shear bond strength.

The mean shear bond strength of the metal-composite combination recorded in this study (19.29 MPa) is in accordance with other studies.[32-34] According to Matsumura et al[35] the resin-metal shear bond strength must exceed 10 MPa to ensure clinically satisfactory results. The most recently developed FRCs, although not well documented clinically, seem to be a reliable alternative to the cast alloy substrates for fixed prosthodontics when sticks or fiber nets are used, as far as their bond to the indirect composite is concerned. On the other hand, the metal-composite and FRC composite bond strengths did not exceed the metal-ceramic bond strength, which, according to ISO 9693 for metal-ceramic dental restorative systems, must be not less than 25 MPa.[36]

The exact mechanism of shear bond enhancement when sticks and fiber net are used is not known. It is possible that sticks increase the FRC surface that bonds to the indirect composite. They might also interlock the composite resin and subsequently increase the shear strength. The fiber net might increase the bonding surface through the irregularities of its frame.

In one study[22] the effect of fiber orientation on bond strength between an indirect composite and FRCs was investigated. Higher bond values were achieved when glass fibers were perpendicular and longitudinal to the applied load than when they were transversal. In the present study, the load applied on the specimens was in a random direction to the orientation of the fibers and of the retentive sticks of the substructure, to more closely imitate clinical conditions, because fiber orientation is not taken into consideration in the FRC manufacturer's recommendations on the preparation of the FRC substructure. That might be the reason for the somewhat high standard deviation of the bond strengths of the specimens. Other studies of similar materials have presented a high standard deviation.[37,38] Nevertheless, retentive sticks significantly increased the shear bond strength when used with unidirectional fibers, while the use of retentive sticks with fiber net did not provide a statistically significant increase to the veneering composite/FRC shear bond strength.

Unidirectional fibers exhibit high strength when subjected to transverse forces; therefore, it has been proposed that pontics of FPDs should be fabricated using unidirectional fibers.[39] In those cases, retentive sticks may also be used to increase the FRC/indirect composite shear bond strength. Further investigation is needed to establish how the sticks should be oriented in a real FPD substructure according to the direction of the occlusal forces.

Several studies have investigated the bond strength between FRC and resin composite in various applications. A wide range of bonding values has been recorded under different experimental conditions. Particularly, Waki et al[14] recorded shear bond strength values varying between 3.0 ± 1.3 MPa and 17.2 ± 5.8 MPa. In another study by Keski-Nikkola et al[38] values ranged between 10.8 ± 4 MPa and 21 ± 4 MPa. Lassila et al[22] found bonding values at a higher level, between 24.8 ± 6.3 and 44.8 ± 3.4 MPa. In the previous studies, different materials and methodology were used, making a direct comparison impossible.

In the present study, an estimation of the bond strength of a veneering composite and an FRC substructure was undertaken in vitro. In vivo studies of applied forces are more complicated, as the directions of the forces applied are more difficult to control. In addition, the modulus of elasticity of the two materials does not factor in the bond strength in shear testing, whereas it is an important factor in clinical conditions. It is therefore not possible to directly translate the result of this in vitro study to the in vivo situation. The results of this study, however, do provide guidance in terms of the properties of these materials tested. Alterations of the orientation of the fibers and of the morphology of the FRC substructure can probably provide better bond strengths of the veneering composite/FRC substructure interface and so the FRC prosthetic restoration may function in the mouth for a longer period of time with no veneering fracture problems. Further investigation is needed.

Conclusions

The following conclusions may be derived:

1. All FRC specimens exhibited a cohesive mode of failure.
2. When sticks or fiber net was used in FRC substructures, the bond strength between FRC and indirect composite was statistically similar to the bond strength between Ni-Cr cast dental alloy and the indirect veneering composite.

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