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Shear Bond Strength of Different Repair Systems to Titanium After Water Aging

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Clinical Relevance

Among the repair systems evaluated, those which use the tribochemical silica-coating procedure can be considered good options for repairing exposed titanium surfaces. Furthermore, the Cojet system's failure mode and stable behavior after water storage seems to indicate its use for a titanium surface repair technique.

SUMMARY

This study evaluated the shear bond strength (SBS) and stability of commercially pure titanium (CP Ti)/repair material interfaces promoted by different repair systems. One hundred CP Ti cast discs were divided into five repair system groups: 1) Epricord (EP); 2) Bistite II DC (BT); 3) Cojet (CJ); 4) Scotchbond Multi-Purpose Plus (SB) (control group); and 5) Cojet Sand plus Scotchbond Multi-Purpose Plus (CJSB). The specimens were stored in distilled water for 24 hours at 37°C, thermal cycled (5000 cycles, 5°-55°C) and stored under the same conditions for either 24 hours or six months (n=10). SBS was tested and the data were analyzed by two-way analysis of variance (ANOVA) and Tukey test (α=.05). Failure mode was determined with a stereomicroscope (20×). The repair system, storage time, and their interaction significantly affected the SBS ($p<0.001$). At 24 hours, CJSB exhibited the highest SBS value, followed by CJ. At six months, these two groups had similar mean SBS ($p>0.05$) and higher means in comparison to the other groups. For both storage times, BT

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presented the lowest SBS, while the EP and SB groups did not differ significantly from one another ($p>0.05$). There were no significant differences in SBS between the storage times for the groups EP and CJ ($p>0.05$). The groups BT, SB, and CJSB showed 100% adhesive failure, irrespective of storage time. The CJSB group showed the highest SBS at both storage times. At six months, the CJ group exhibited a similar SBS mean value when compared to the CJSB group. Water storage adversely affected the groups BT, SB (control group), and CJSB. Considering SBS values, stability, and the failure mode simultaneously, the CJ group showed the best CP Ti repair performance.

INTRODUCTION

Metal-ceramic tooth- or implant-supported prostheses are still widely used in oral rehabilitations.[1,2] Different metal compositions can be used to manufacture their frameworks, such as NiCr, NiCrTi, AgPd, CoCrMo, and Ti, with clinically satisfactory results. However, fracture or chipping of the ceramic veneer is a potential problem for these restorations,[3] with these occurrences reported as the second most likely cause for their replacement, after dental caries.[4] According to Libby and others,[5] failure resulting from porcelain fracture has been reported to range from 2.3% to 8.0%.

In certain clinical situations, a simple repair technique may reestablish the esthetics and function of a compromised restoration, avoiding the replacement of the fractured metal-ceramic prosthesis, which would increase the cost and time required.[6] Moreover, this procedure is not conservative, possibly increasing the risk of trauma to the tooth during removal of the restoration.[7]

In general, to repair fractured restorations, composite resins are employed. For this purpose, some commercially available composite resins have surface treatment protocols defined by their manufacturers.[8] The goal of these surface treatments is to provide both micromechanical retention and chemical bonding between the composite resin and the substrate.[9,10]

However, when complete deveneering of porcelain results in extensive metal exposure, the repair procedure is a potential clinical challenge,[11] especially in titanium frameworks. Despite its excellent biological and mechanical properties, when the oxide layer of the commercially pure titanium (CP Ti) surface is mechanically removed (by airborne-particle abrasion during a repair procedure), an unstable oxide layer is formed simply by contact with oxygen.[12] This oxide layer restricts the bonding of resin-based materials to titanium;[13] therefore, it is necessary to investigate the efficacy of different materials and their respective protocols on titanium surface repair.

Thus, the purpose of this *in vitro* study was to evaluate early adhesive bonding and stability of CP Ti/repair material interface provided by different repair systems. The null hypothesis to be tested was that all repair systems could provide statistically similar adhesive bonding and durability.

MATERIALS AND METHODS

One hundred discs (9.0 mm wide and 3.0 mm thick) were cast in CP Ti Grade 2 (RMI Co, Niles, OH, USA) using an Ar-arc casting machine (EDG Equipamentos e Controles Ltda, São Carlos, SP, Brazil). The Rematitan Plus (Dentaurum JP Winkelstroeter KG, Ispringen, Germany) phosphate investment was used according to the manufacturer's instructions. The CP Ti discs were embedded in polyvinyl chloride (PVC) tubes (20.0 mm in diameter and 27.0 mm in length) containing polymethyl methacrylate (PMMA) autopolymerizing acrylic resin (Jet, Artigos Odontológicos Clássico, São Paulo, SP, Brazil). A polisher (Metaserv 2000, Buehler UK Ltd, Coventry, UK) was used to smooth all specimen bonding surfaces with silicon carbide sandpapers (120-, 220-, and 320-grit). The specimens were divided into five groups according to the repair systems (n=20): 1) Epricord (EP); 2) Bistite II DC (BT); 3) Cojet (CJ); 4) Scotchbond Multi-Purpose Plus (SB) (control group); and 5) Cojet Sand plus Scotchbond Multi-Purpose Plus (CJSB). Table 1 summarizes the sequence of materials and procedures used in each repair system group according to the manufacturer's specifications.

Airborne-particle abrasion was performed for 20 seconds with an air abrasion unit (Basic Classic, Renfert GmbH, Hilzingen, Germany), at 0.24 MPa air pressure. For this procedure, the specimens were mounted in a special holder, which allowed a 90° angle and a distance of 10 mm from the surface of the specimen to the blasting tip. All specimens were ultrasonically cleaned in distilled water for 10 minutes.

The bonding agents (metal primer, silane, and adhesive) were applied with a disposable brush in a single layer. The dual-cured resin cement Bistite II DC (paste-paste), used as opaque material, and

Table 1: *Sequence of Materials and Procedures Used in Each Repair System Group*

Repair Systems (Manufacturer)	Sequence of Material Application
Epricord (EP) (Kuraray Co Ltd, Osaka, Japan)	. Airborne-particle abrasion with 50 μm Al_2O_3 particles
	. Alloy Primer (wait 60 s)
	. Epricord Opaque Primer (wait 60 s). Epricord Body Opaque
	. Epricord Dentin composite resin
Bistite II DC (BT) (Tokuyama Dental Corp, Tokyo, Japan)	. Airborne-particle abrasion with 50 μm Al_2O_3 particles
	. Bistite II DC resin cement
	. Estelite Σ composite resin
Cojet (CJ) (3M ESPE AG, Seefeld, Germany)	. Airborne-particle abrasion with 30 μm silica-modified Al_2O_3 particles (Cojet Sand)
	. Espe-Sil silane (wait 30 s)
	. Sinfony opaque
	. Visio-Bond adhesive
	. Z100 composite resin
Scotchbond Multi-Purpose Plus (control group) (SB) (3M ESPE, St Paul, MN, USA)	. Airborne-particle abrasion with 50 μm Al_2O_3 particles
	. Scotchbond phosphoric etchant by 15 s (rinse and dry)
	. Adper Scotchbond Multi-Purpose Plus adhesive
	. Masking Agent opaque
	. Z100 composite resin
Cojet Sand plus Scotchbond Multi-Purpose Plus (CJSB) (3M ESPE, St Paul, MN, USA)	. Airborne-particle abrasion with 30 μm silica-modified Al_2O_3 particles (Cojet Sand)
	. RelyX Ceramic Primer silane (wait 60 s)
	. Adper Scotchbond Multi-Purpose Plus adhesive
	. Masking Agent opaque
	. Z100 composite resin

Sinfony opaque (powder-liquid) of Cojet were proportioned by weight and mixed according to the manufacturer's instructions. Opaque agents were applied to the treated CP Ti surfaces using a custom-made metal matrix (4.0 mm in diameter circular aperture and 0.3 mm in thickness), which was placed on the surface of the specimen by attaching a centralizing ring to the PVC tube.

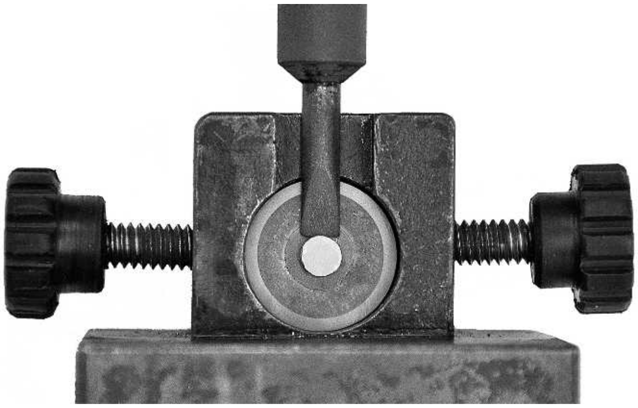

Figure 1. *Shear bond strength test apparatus.*

To apply the composite resins, a custom-made metal split matrix with a circular aperture (4.0-mm internal diameter and 2.0-mm thickness) was positioned on the surface of the specimen using the centralizing ring attached to the PVC tube. The fluid resins of the adhesive systems, Visio-Bond and Adper Scotchbond Multi-Purpose Plus, were light-cured for 20 seconds, while the opacifying agents and the dual-cured resin cement were light-cured for 40 seconds each. The composite resins were polymerized for 60 seconds (40 seconds with the matrix in place and 20 seconds after the metal matrix was removed). This procedure was performed with a visible light-curing unit (Curing Light XL3000, 3M ESPE, St Paul, MN, USA) and an intensity of approximately 550 mW/cm^2, which was assessed with the same radiometer (DMC Equipamentos Ltda, São Carlos, SP, Brazil) prior to each use.

After preparation, all specimens were stored in distilled water at 37°C for 24 hours before thermal cycling between 5°C and 55°C for 5000 cycles, with a 30-second dwell time. After thermal cycling, 10 specimens from each group were stored in distilled water at 37°C for 24 hours before the shear bond strengths were determined. The remaining 10 specimens in each group were stored under the same conditions for 6 months to evaluate the stability of the repair systems.

After water storage, using a mechanical testing machine (810 Material Test System, MTS Systems Corp, Eden Prairie, MN, USA), each specimen was placed in a metal apparatus and subjected to a shear bond strength (SBS) test (Figure 1). A knife-edge blade running at a 0.5 mm/min crosshead speed was used to direct a uniaxial compressive load to the specimen as closely as possible to the resin/metal interface until the materials' debonding was observed. The force (N) required to fracture the specimen was divided by the bonding surface area in order to obtain the shear bond strength values (MPa).

Table 2: *Two-way ANOVA*

Source of Variation	SS	*df*	MS	F	*p*
Repair system	2487.16	4	621.79	272.92	<0.001
Storage time	183.90	1	183.90	80.72	<0.001
Repair system × storage time	33.52	4	8.38	3.68	<0.01
Residual	205.05	90	2.28		
Total	2909.63	99			

Each specimen was examined under a stereomicroscope (M80, Leica Microsystems Ltd, Heerbrugg, Switzerland) at 20× magnification, and the digital images were captured and analyzed by imaging software (Leica Application Suite EZ, Leica Microsystems Ltd). A single calibrated observer recorded the failure mode as adhesive failure between titanium and resin, cohesive failure of the resin, or mixed failure (a combination of both). For this classification, the adhesive area was divided into quadrants,[14] and the predominant mode of failure was observed for each one. Failure was classified as adhesive or cohesive if either of these modes were predominate in three or more quadrants, and classified as mixed if two quadrants presented adhesive failure and the other two, cohesive failure.

The data were analyzed by two-way analysis of variance (ANOVA) and when there was a significant difference among the means, the Tukey (HSD) post-hoc test (α=0.05) was applied.

RESULTS

The results from the two-way ANOVA (Table 2) indicated that the repair system ($p<0.001$), storage time ($p<0.001$), and interaction between these variables significantly affected the SBS ($p<0.01$). Table 3 shows the mean SBS values (MPa), standard deviations for each group, and the statistical groupings identified with the Tukey HSD test.

Comparison of the repair systems showed that for 24 hours of storage time, CJSB exhibited the highest SBS value, followed by the CJ group. However, for a

Table 3: *Mean Shear Bond Strength Values (MPa), Standard Deviations (±) and Statistical Results**

Repair Systems	**Storage Times**	
	24 Hours	**Six Months**
EP	13.0 ± 2.0 Ac	11.2 ± 1.3 Ab
BT	8.1 ± 1.2 Ad	4.8 ± 0.4 Bc
CJ	18.3 ± 2.2 Ab	17.4 ± 1.9 Aa
SB (control group)	13.4 ± 1.4 Ac	10.1 ± 1.3 Bb
CJSB	22.7 ± 1.4 Aa	18.6 ± 1.2 Ba

** Different uppercase letters indicate significant differences in row (p<0.05). Different lowercase letters indicate significant differences in columns (p<0.05).*
Abbreviations: BT, Bistite II DC; CJ, Cojet; CJSB, Cojet Sand plus Scotchbond Multi-Purpose Plus; EP, Epricord; SB, Scotchbond Multi-Purpose Plus.

storage time of six months, these two groups showed similar mean SBS values ($p>0.05$) and higher values than the other groups. For both storage times, BT presented the lowest SBS, while the EP and SB groups did not differ significantly from one another ($p>0.05$).

The storage time decreased the SBS mean values for the groups BT, SB, and CJSB.

Table 4 lists the predominant failure mode of the studied repair systems at 24 hours and six months of storage. Figures 2 and 3 illustrate the predominant modes of failure observed for each group at 24 hours and six months of storage, respectively.

DISCUSSION

In the present study, the repair systems CJ, BT and EP were investigated. These systems, considering their different purposes, are also indicated for metal-ceramic prostheses repair. The group SB was used as a control, given that it is a widespread and largely available system in the literature. In the group CJSB, which was proposed to improve the efficacy of the SB group (control group), the steps of airborne-particle abrasion with 50 μm Al_2O_3 particles and acid etching (done in SB group) was replaced by silica-modified Al_2O_3 particles (Cojet Sand which was used in the Cojet group) followed by silane application (CJSB group).

At 24 hours and six months, the CJSB group exhibited the highest SBS values; however, at six months, the CJ group did not differ significantly from the CJSB group. In these groups, the metallic substrate was abraded with silica-modified Al_2O_3 particles (Cojet Sand) and then treated with silane (RelyX Ceramic Primer in the CJSB group and Espe-Sil in the CJ group). Thus, the Cojet Sand particles driven onto the CP Ti surface under pressure provided micromechanical retention and deposition of a silica layer, which causes the surface to be more chemically reactive to the silanes applied afterwards.[15,16] The results of the present study are in agreement with the literature, since this procedure, known as tribochemical silica-coating, has been highly effective for bonding resin-based materials to different substrates.[3,6,17] Lee and others[18] ob-

Table 4: *Percentage of Failure Modes of the Studied Repair Systems*

Groups	**24 Hours**			**Six Months**		
	Adhesive	**Cohesive**[a]	**Mixed**[a]	**Adhesive**	**Cohesive**[a]	**Mixed**[a]
EP	20	30	50	60	10	30
BT	100	—	—	100	—	—
CJ	30	60	10	30	60	10
SB	100	—	—	100	—	—
CJSB	100	—	—	100	—	—

[a] *All cohesive failures occurred in the opaque layer.*
Abbreviations: BT, Bistite II DC; CJ, Cojet; CJSB, Cojet Sand plus Scotchbond Multi-Purpose Plus; EP, Epricord; SB, Scotchbond Multi-Purpose Plus.

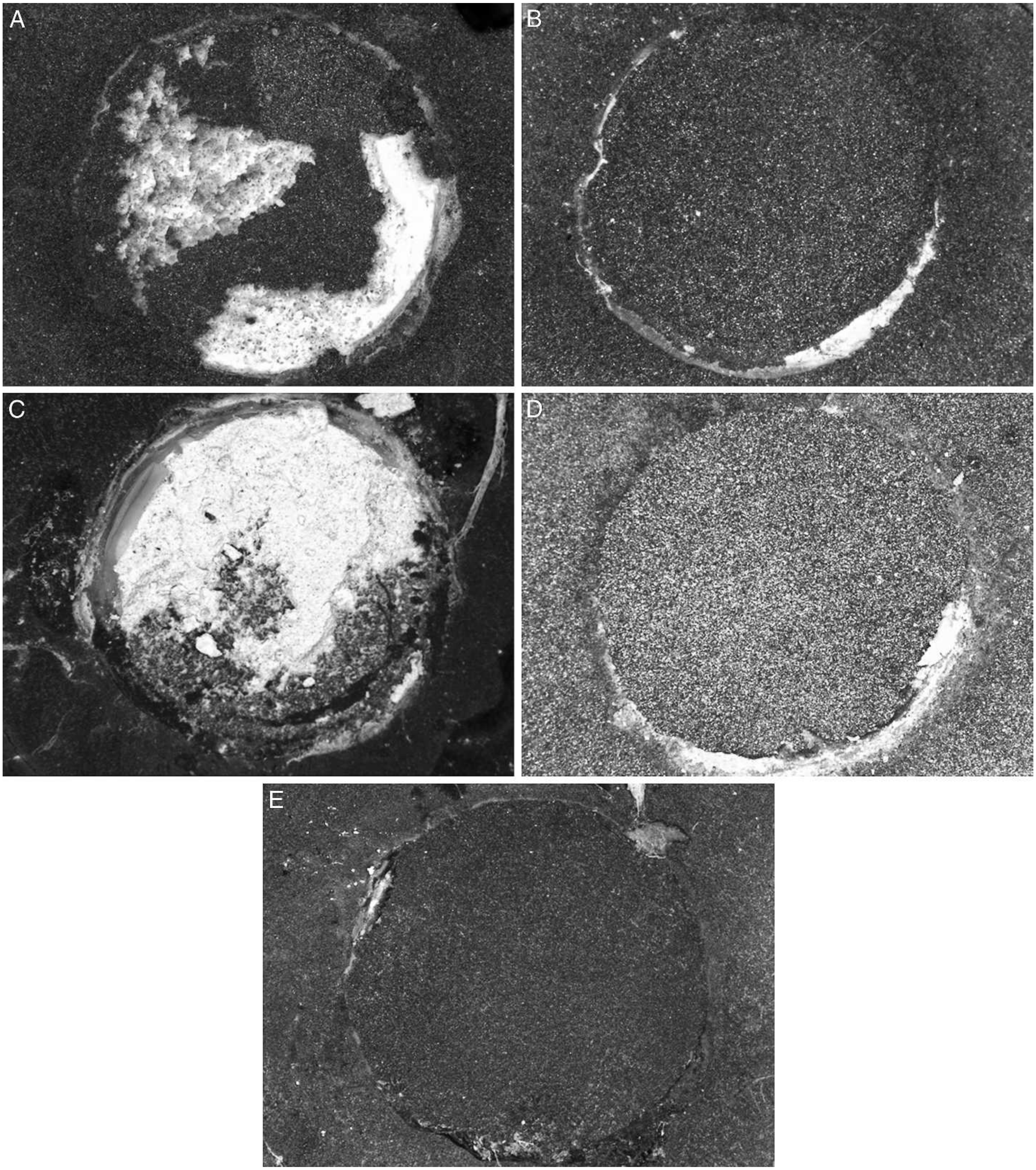

Figure 2. *Microscopic image (20×) of predominant modes of failure observed for each group at 24 hours. (A): Mixed failure - EP group. (B): Adhesive failure - BT group. (C): Cohesive failure - CJ group. (D): Adhesive failure - SB group. (E): Adhesive failure - CJSB group.*

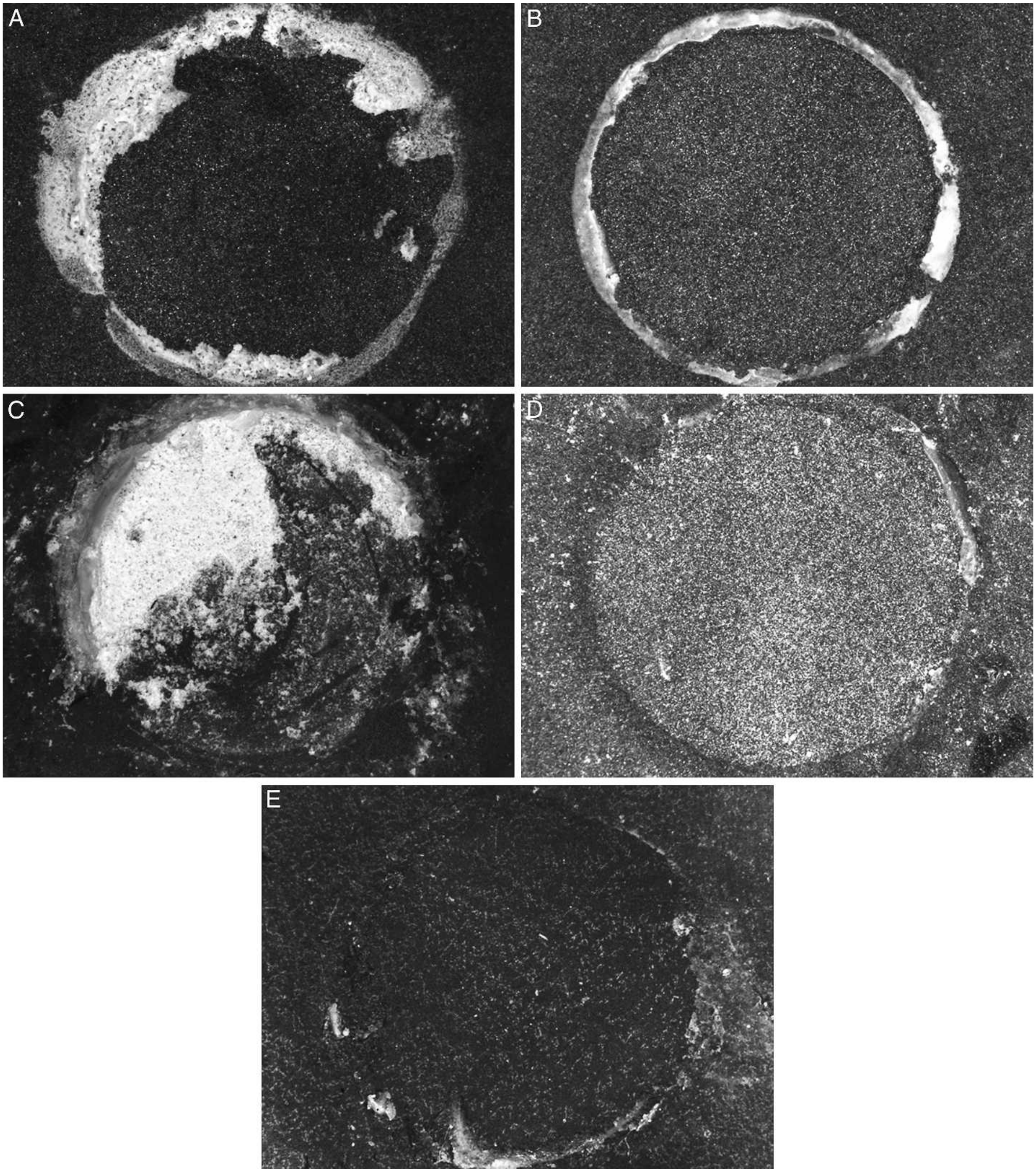

Figure 3. *Microscopic image (20×) of predominant modes of failure observed for each group at six months. (A): Adhesive failure - EP group. (B): Adhesive failure - BT group. (C): Cohesive failure - CJ group. (D): Adhesive failure - SB group. (E): Adhesive failure - CJSB group.*

served higher efficacy of a method used for silica-coating (Rocatec system; 3M ESPE AG) followed by silane Espe-Sil, when compared with airborne-particle abrasion with 250 μm Al_2O_3 particles on the bond strength at the Ti-6Al-4V alloy/composite resin interface. Santos and others[14] and Haneda and others[19] evaluated the efficacy of some repair systems used in the present study and found that abrasion with silica-modified Al_2O_3 particles followed by silane application promoted the highest SBS values at the interface resin-based material/NiCr alloy.

An important aspect that should be discussed is the difference of the predominant failure mode observed in the CJSB and CJ groups. The CJSB group presented 100% adhesive failure at 24 hours and at six months, while the CJ group exhibited a predominant cohesive failure of the opaque layer at both storage times. This failure mode indicates that the bond strength between the repair system and the CP Ti substrate in the CJ group was higher than the cohesive strength of the opacifying agent (Sinfony). It is important to highlight that the Masking Agent opaque used in the CJSB group is a single paste material and its organic phase is composed of triethylene glycol dimethacrylate (TEGDMA) and bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) monomers, while the Sinfony opaque used in the CJ group is a powder-liquid material and is composed of methyl methacrylate (MMA). Özcan and Kumbuloglu[3] investigated the effect of composition, viscosity and thickness of four opaque agents on the bond strength of composite resin to CP Ti. According to these authors,[3] MMA-based opaque agent seems to adhere better to CP Ti than the one based on TEGDMA. This fact may possibly explain the continuation of SBS mean value at six months in the CJ group. On the other hand, the lower adhesion capacity of the opaque agent based on TEGDMA (CJSB group) may have been responsible for the decrease in SBS value after six months and for the predominance of adhesive failure, at both storage times. Moreover, the authors[3] also comment that MMA monomer is not sufficiently polymerized in the presence of oxygen. This fact may explain the predominance of cohesive failure presented by the CJ group.

The control (SB) and EP groups exhibited similar SBS values at 24 hours and six months. However, these groups provided significantly lower SBS mean values than those of the CJ and CJSB groups and higher values than the BT group. In the EP group, airborne-particle abrasion with Al_2O_3 particles was performed, followed by application of Alloy Primer and Epricord Opaque Primer. These metal primers contain the 10-methacryloyloxydecyl dihydrogen phosphate (MDP) adhesive monomer, which is able to establish chemical bonds to metal oxides of base metal surfaces and to copolymerize with the monomers of the resin-based materials. Thompson and others[15] comment that the bond strength promoted by airborne-particle abrasion with Al_2O_3 particles followed by application of phosphoric acid primers (MDP) is generally lower than that of a tribochemical silica-coating/silane association.

However, several studies[20-23] indicate that the MDP monomer is effective on the bond between titanium and resinous materials. This observation may explain the fact that at 24 hours the EP group showed 50% mixed failure and 30% cohesive failure in the opaque layer, indicating that the bond between opaque agent and CP Ti provided by both Alloy Primer and Epricord Opaque Primer was more effective than the mechanical strength of the opaque material. On the other hand, although for the EP group there was no significant difference between both storage times, the increase of adhesive failure from 20% to 60% may indicate that a possible hydrolysis of water degradable chemical bonds took place.

In contrast to the other groups, the control group (SB) is based only on micromechanical retention provided by airborne-particle abrasion with Al_2O_3 particles, as no material in this group is capable of establishing chemical bonds with the metal substrate. In this group, the adhesive was used after airborne-particle abrasion to increase the wettability of CP Ti by the Masking Agent opaque. Possibly, the absence of chemical bonds between repair material and CP Ti explains the 100% adhesive failure at both storage times and the significant decrease of SBS after six months. Santos and others[14] and Haneda and others[19] also verified, in NiCr alloy, no significant difference between Scotchbond Multi-Purpose Plus and Clearfil SE Bond (Kuraray Co Ltd), whose repair protocol is the same as that of Epricord, which was used in the present study.

Finally, the BT group showed the lowest mean SBS values at both storage times. In this group, the Bistite II DC resin cement is applied to the alumina abraded surface. This cement is used as an opaque material and contains the 11-methacryloyloxundecan 1,1-dicarboxylic acid (MAC-10) monomer, which like the MDP monomer, provides chemical bonds between the repair material and metallic oxides present in base metal alloys. However, MAC-10 monomer seems to be less effective than MDP monomer, according to some authors.[20,24–27] Moreover, in this group, an intermediate bonding agent was not used, which could increase the metal surface wettability by the resin cement, which presents with an apparent high viscosity. These two factors may explain, for both storage times, the lower performance of this group (BT) when compared to the others, the high predominance of adhesive failure (100%) and, finally, the decrease of SBS after six months. These results were also observed by Santos and others[14] and Haneda and others.[19]

The results of the present study indicate the superiority of using the tribochemical silica-coating system followed by silane application in the production of metal-ceramic prosthesis repairs. The CJSB group proposed in the present study, despite undergoing a significant decrease in SBS at six months, was the only one that presented significantly higher SBS at both storage times. The CJ group, in which abrasion with silica-modified Al_2O_3 particles was also performed, exhibited SBS that was statistically similar to the SBS of the CJSB group only at six months. The CJ group did not show a decrease in SBS after water storage, indicating a higher stability in comparison to the CJSB group, which was also confirmed by the predominance of cohesive failure of opaque layer at both storage times.

Further studies to evaluate other factors that could exert a great influence on the bond strength of metal/repair system interfaces, such as long-term water storage and dynamic fatigue loading, as well as long-term clinical studies, should be conducted to establish the reliable behavior of these repair materials under clinical conditions.

CONCLUSION

Within the limitations of this study, the results indicated that the silica-coated groups (CJ and CJSB) showed the highest SBS mean values at 24 hours and six months, while the BT group showed the lowest SBS. The EP and SB (control group) groups did not differ statistically at both storage times. The BT, SB (control group), and CJSB groups exhibited a decrease in SBS after six months, while the SBS values of the EP and CJ groups were not affected by water storage. Considering SBS values, stability, and failure mode, the CJ group showed the best performance in repairing CP Ti.

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