

RESEARCH AND EDUCATION

Bond strength of additively manufactured composite resins to dentin and titanium when bonded with dual-polymerizing resin cements

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ABSTRACT

Statement of problem. Additively manufactured composite resins for definitive restorations have been recently introduced. The bond strength between these composite resins and different substrates has not been extensively studied.

Purpose. The purpose of this in vitro study was to measure the shear bond strength (SBS) between additively manufactured composite resins and dentin and titanium substrates and compare those with the SBS between subtractively manufactured polymer-infiltrated ceramic and the same substrates (dentin and titanium), when different dual-polymerizing resin cements were used.

Material and methods. One hundred and eighty cylinder-shaped specimens (Ø5×5 mm) were prepared from 3 materials recommended for definitive restorations: an additively manufactured composite resin (Crowntec [CT]); an additively manufactured hybrid composite resin (VarseoSmile Crown Plus [VS]); and a subtractively manufactured polymer-infiltrated ceramic (Enamic [EN]) (n=60). Specimens were randomly divided into six subgroups to be cemented to the two substrates (dentin and titanium; n=30) with 1 of 3 resin cements (RelyX Universal, Panavia V5, and Variolink Esthetic DC) (n=10). The restoration surface to be bonded was treated according to the respective manufacturer's recommendations. Dentin surfaces were treated according to the resin cement (Scotchbond Universal Plus Adhesive for RelyX Universal, Panavia V5 Tooth Primer for Panavia V5, and Adhese Universal for Variolink Esthetic DC), while titanium surfaces were airborne-particle abraded, and only the specimens paired with Panavia V5 were treated with a ceramic primer (Clearfil Ceramic Primer Plus). SBS was measured in a universal testing machine at a crosshead speed of 1 mm/min. Failure modes were analyzed under a microscope at ×12 magnification. Data were analyzed by using 2-way analysis of variance and Tukey honestly significant difference tests ($\alpha=0.05$).

Results. When SBS to dentin was considered, only restorative material, as a main factor, had a significant effect ($P<0.001$); EN had the highest SBS ($P<0.001$), while the difference in SBS values of CT and VS was not significant ($P=0.145$). As for SBS to titanium, the factors restorative material and resin cement and their interaction had a significant effect ($P<0.001$). Within each resin cement, EN had the highest SBS to titanium ($P<0.001$), and within each restorative material, Variolink resulted in the lowest SBS ($P\leq 0.010$). Overall, EN and RelyX were associated with the highest SBS to titanium ($P\leq 0.013$). Mixed failures were predominant in most groups.

Conclusions. Regardless of the substrate or the resin cement used, the subtractively manufactured polymer-infiltrated ceramic had higher shear bond strength than the additively manufactured composite resins. The SBS of the additively manufactured composite resins, whether bonded to dentin or titanium, were not significantly different from each other. Regardless of the restorative material, Variolink DC resulted in the lowest SBS for titanium surfaces. (J Prosthet Dent 2023;■:■-■)

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Clinical Implications

RelyX Universal and Panavia V5 may be recommended to cement tooth-supported or titanium-based implant-supported restorations fabricated by using the additively manufactured composite resins.

Computer-aided design and computer-aided manufacturing (CAD-CAM) technologies have led to additive and subtractive manufacturing methods and diversified restorative materials.^{1,2} Given the advantages of additive manufacturing, such as the ability to fabricate complex geometries with less material waste,³ materials that can be additively manufactured⁴ have been marketed. Among these materials, composite resins indicated for definitive indirect restorations have become popular, and these materials can be used for tooth- or implant-supported restorations.^{2,5} Manufacturers categorize these materials either as composite resins^{2,3} or hybrid composite resins that comprise ceramic particles.⁶ Hybrid materials are also among those that can be subtractively manufactured. One such material, containing 86 wt% ceramic and 14 wt% acrylate polymer network (urethane dimethacrylate and triethylene glycol dimethacrylate),⁷⁻¹⁰ combines the advantages of composite resins and ceramics.^{11,12} This polymer-infiltrated ceramic is indicated for both tooth- and implant-supported restorations.¹³

A durable bond between the fixed indirect restoration and the cement is critical for clinical longevity and stability.¹⁴⁻¹⁶ Restorative material and cement may affect the bond strength.¹⁷ Even though several types of cement have been proposed for definitive cementation, resin cements have enhanced mechanical properties¹⁸ and better clinical longevity for indirect restorations.¹⁹ Dual-polymerizing resin cements were introduced to overcome the difficulty of light polymerizing beneath thick indirect restorations²⁰ and combine the advantages of light polymerizing and autopolymerizing resin cements.²¹

Among the studies on the properties of additively manufactured definitive composite resins,^{2,3,5,6,22-29} only Graf et al⁶ focused on their bonding properties. They investigated the effect of different surface treatments on the pull-off bond strength of crowns fabricated by using an additively manufactured definitive composite resin. However, the crowns were cemented on polymer-based tooth-shaped preparations. Studies on the bond strength between additively manufactured composite resins and clinically relevant substrates like dentin and titanium would broaden the knowledge on their applicability. Therefore, the present study aimed to compare the bond strength between additively manufactured

composite resins or subtractively manufactured polymer-infiltrated ceramic and dentin or titanium when bonded with different dual-polymerizing resin cements. The null hypothesis was that the type of restorative material and resin cement would not affect the bond strength to dentin and titanium.

MATERIAL AND METHODS

Cylinder-shaped specimens (Ø5×5 mm) were fabricated from two additively manufactured composite resins (Crowntec; Saremco Dental AG, CT and VarseoSmile Crown Plus; Bego GmbH, VS) and one subtractively manufactured polymer-infiltrated ceramic (Enamic; Vita Zahnfabrik, EN) indicated for definitive restorations (Table 1). Sixty specimens were fabricated from each material and divided into six subgroups depending on the resin cement and the substrate. The number of specimens in each subgroup was determined from an a priori power analysis ($\alpha=.05$, $1-\beta=95\%$, and effect size of $f=0.493$), which deemed nine specimens per group adequate.³⁰ Ten specimens per subgroup were fabricated to increase the statistical power.

For the fabrication of additively manufactured specimens (CT and VS), a cylinder-shaped standard tessellation language (STL) file with the Ø5×5-mm dimensions was designed in a software program (Meshmixer v3.5.474; Autodesk Inc). The STL file was imported into a nesting software program (Composer v1.3; ASIGA) and placed vertically on the build platform. After generating supporting structures automatically, this design was duplicated for standardization, and specimens were manufactured by using a digital light processing-based 3-dimensional (3D) printer (MAX UV; ASIGA). After fabrication, CT specimens were cleaned with a 96% alcohol-soaked (95% Ethanol Absolut; Grogg Chemie AG) cloth to remove residual resin,³¹ while VS specimens were cleaned in an ultrasonic bath containing reusable ethanol solution, followed by thorough cleaning in an ultrasonic bath containing fresh ethanol (95%).³² After cleaning, all specimens were dried with an air syringe. CT specimens were then placed in a xenon polymerization device (Otoflash G171; NK Optik) and polymerized under nitrogen oxide gas atmosphere (4000 lighting exposures), which was followed by airborne-particle abrasion of the specimen surfaces with 50- μm glass beads (Roll-oblast; Renfert) at 0.15-MPa and removing the supports with a cut-off wheel (Keystone Cut-off Wheels; Keystone Industries).³¹ Supports of VS specimens were removed first by using the same cut-off wheel, and specimen surfaces were airborne-particle abraded with 50- μm glass beads at 0.15 MPa until the whitish layer that appeared after cleaning had disappeared. Thereafter, the VS specimens were placed in the xenon polymerization device and polymerized under nitrogen oxide gas

Table 1. Materials used

Material	Chemical Composition
Crowntec (CT) (additively manufactured composite resin)	Esterification products of 4,4'-isopropylphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, pyrogenic silica, initiators. Total content of inorganic fillers: 30-50 wt%
Enamic (EN) (polymer-infiltrated ceramic)	14 wt % methacrylate polymer (urethane dimethacrylate and triethylene glycol dimethacrylate) and 86 wt % fine-structure feldspar ceramic network
VarseoSmile Crown Plus (VS) (additively manufactured hybrid composite resin)	Esterification products of 4,4'-isopropylphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, methyl benzoylformate, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide. Total content of inorganic fillers: 30-50 wt%
RelyX Universal (dual-polymerizing self-adhesive resin cement)	Bisphenol A derivative free dimethacrylate monomers, phosphorylated dimethacrylate adhesion monomers, photoinitiators, amphiphilic redox initiators, radiopaque fillers, rheological additives, pigments
Variolink Esthetic DC (dual-polymerizing resin cement)	Urethane dimethacrylate, 1,10-decandiol dimethacrylate, a-dimethylbenzyl hydroperoxide, initiators, stabilizers, pigments and inorganic fillers of ytterbium trifluoride, spheroid mixed oxide
Panavia V5 (dual-polymerizing adhesive resin cement)	Bisphenol A glycidyl methacrylate, triethylene glycol dimethacrylate, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, Colloidal silica, silanated aluminum oxide filler, d-camphorquinone, pigments
Scotchbond Universal Plus Adhesive (universal adhesive)	10-methacryloyloxydecyl dihydrogen phosphate, 2-hydroxyethyl methacrylate, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane
Adhese Universal (universal adhesive)	10-methacryloyloxydecyl dihydrogen phosphate, methacrylated carboxylic acid polymer, 2-hydroxyethyl methacrylate, bisphenol A glycidyl methacrylate, 1,10-decandiol dimethacrylate, water, ethanol, highly dispersed silicon dioxide, initiators and stabilizers
Panavia V5 Tooth Primer (self-etching primer)	10-methacryloyloxydecyl dihydrogen phosphate, 2-hydroxyethyl methacrylate, hydrophilic aliphatic dimethacrylate, accelerators, water
Clearfil Ceramic Primer Plus (universal prosthetic primer)	3-methacryloxypropyl trimethoxysilane, 10-methacryloyloxydecyl dihydrogen phosphate, ethanol

atmosphere (3000 lighting exposures).³² Specimens were fabricated one day before being bonded to their respective substrate and kept in a light proof box in an incubator at 37 °C. For the fabrication of subtractively manufactured specimens (EN), a cylinder-shaped STL (Ø5×10 mm) was designed by using the same design software program and imported into a nesting software (PrograMill CAM V4.2; Ivoclar AG). A 5-axis milling unit (PrograMill PM7; Ivoclar AG) was used to subtractively manufacture cylinder-shaped specimens, which were

then cut in half by using a precision cutter (Vari/cut VC-50; Leco Corp) to obtain specimens of the target dimension.

Dentin specimens were produced from sound-extracted permanent human molars obtained from a pooled biobank. The local ethical committee considers pooled biobanks as irreversibly anonymized and waives the necessity for ethical approval. The molars were cleaned with a scaler and curette and then ground parallel to the occlusal surface to the center of the coronal dentin (Struers Labo-Pol 21 #220 and #500; Struers). The ground dentin surface was examined to ensure that it had no residual enamel and no pulp opening. Subsequently, the roots were removed by using a water-cooled diamond saw (IsoMet Low Speed Saw; Buehler), and the molars were then embedded in autopolymerizing acrylic resin (Paladur; Kulzer GmbH) using stainless-steel molds. The dentin specimens were stored in the refrigerator (4 °C in tap water) after the polymerization of acrylic resin. Titanium specimens (Colado CAD; Ivoclar AG) were fabricated by using a cylinder-shaped STL file, which was processed similarly to the STL file generated for EN specimens to produce Ø9×3-mm titanium disks, which were also embedded in autopolymerizing acrylic resin. After the acrylic resin had polymerized, the titanium specimens were stored under ambient conditions.

The randomization function of a software program (Excel; Microsoft Corp) was used to randomly divide all dentin and titanium specimens into three groups based on the dual-polymerizing resin cement (RelyX Universal; 3M ESPE, Variolink Esthetic DC; Ivoclar AG, and Panavia V5; Kuraray Noritake) to be used. Dentin specimens and resin cements were removed from the refrigerator at least an hour before cementation. The bonding surfaces of CT and VS specimens were airborne-particle abraded by using 50-µm aluminum oxide (Al₂O₃) particles (Cobra; Renfert) for 10 seconds with 0.2-MPa pressure and 10-mm distance, steam-cleaned for 5 seconds from a distance of 100 mm (Minivapor 93; Effegi Brega), and dried with oil-free air. Bonding surfaces of EN specimens were wet-ground for 5 seconds on silicon carbide abrasive papers (Struers Labo-Pol 21 #500; Struers), which was followed by hydrofluoric acid etching (IPS Ceramic Etching Gel; Ivoclar AG) for 60 seconds. Specimens were then steam-cleaned for 10 seconds from a distance of 30 mm, dried with oil-free air, and treated with a ceramic primer (Clearfil Ceramic Primer Plus; Kuraray Noritake) that was gently air-dried for 5 seconds.

Dentin surfaces were wet-ground for 5 seconds on silicon carbide abrasive paper (Struers Labo-Pol 21 #500; Struers) to obtain a standardized smear layer and air-dried. The dentin surface was then treated with the adhesive recommended for each of the three resin cements (Scotchbond Universal Plus Adhesive; 3M for RelyX Universal, Panavia V5 Tooth Primer; Kuraray Noritake

Table 2. Descriptive statistics of shear bond strength to dentin

Resin Cement	Restorative Material		
	CT	VS	EN
RelyX Universal	8.52 ±1.59 ^a	8.71 ±1.23 ^a	11.84 ±1.74 ^a
Panavia V5	7.45 ±1.92 ^a	10.04 ±2.67 ^a	12.63 ±1.56 ^a
Variolink DC	8.16 ±2.31 ^a	8.03 ±1.23 ^a	10.66 ±1.43 ^a
Total	8.04 ±1.95 ^a	8.93 ±1.96 ^a	11.71 ±1.73 ^b
<i>P</i> *	—	<.001	—

CT, Crowntec; EN, Enamic; VS, VarseoSmile Crown Plus. Different superscript lowercase letters indicate significant differences in rows. Total values derived from pooled data of each restorative material. *P* value of main factor restorative material indicated as *P** (*P*<.05).

for Panavia V5, and Adhese Universal; Ivoclar AG for Variolink Esthetic DC). The adhesives were applied with a microbrush and left undisturbed for 20 seconds before being gently air dried for 5 seconds. The adhesive (Adhese Universal; Ivoclar AG) was then light polymerized for 5 seconds by using a light-emitting diode (LED) polymerization unit (Bluephase; Ivoclar AG) in high power mode. Throughout the study, the light power density was verified periodically with a radiometer (Bluephase Meter; Ivoclar AG) to be at least 950 mW/cm². Titanium surfaces were wet ground for 5 seconds on silicon carbide abrasive paper (Struers Labo-Pol 21 #500; Struers) and air dried. The surfaces were then airborne-particle abraded by using 50- μ m Al₂O₃ particles for 20 seconds with 0.2-MPa pressure at a 10-mm distance and steam-cleaned for 10 seconds from a distance of 30 mm. Surfaces to be bonded with Panavia V5 were finally treated with a ceramic primer (Clearfil Ceramic Primer Plus; Kuraray Noritake) according to the manufacturer's instructions and immediately air dried for 5 seconds.

The cementation procedure was similar for all restorative material-resin cement combinations, and all pretreatments were performed by a single operator (D.Y.). The resin cements were applied on the treated surfaces of the CT, VS, or EN specimens by using the refillable syringes of the respective manufacturers, and the specimens were then brought into contact with the substrate surface with the aid of a brass holder. The specimen was pressed against the substrate surface by a brass rod, which applied a constant force of 2 N.³⁰ Resin cements were light polymerized for 3 seconds at each of the 2 opposite sides of the cemented restorative material cylinders, and excess cement was removed with a scalpel (Surgical Scalpel Blade #11; Swann-Morton). The specimen complex was then light polymerized for a total of 40 seconds, 10 seconds at each of four sites around the circumference, aiming at the base of the cylinders. Ten minutes after the start of mixing the resin cements, the complex was removed from the brass holder and stored in tap water at 37 °C. After 24 hours, the bonded specimens were subjected to a shear bond strength (SBS) test by using a wire

Table 3. Descriptive statistics of shear bond strength to titanium

Resin Cement	Restorative Material			Total	<i>P</i> **
	CT	VS	EN		
Relyx Universal	9.20 ±1.71 ^B	9.70 ±1.41 ^B	22.46 ±2.19 ^C	13.79 ±6.48 ^C	—
Panavia V5	9.11 ±2.16 ^B	7.58 ±3.02 ^B	20.21 ±3 ^C	12.30 ±6.31 ^b	<.001
Variolink DC	4.04 ±0.82 ^A	4.25 ±0.72 ^A	9.96 ±1.35 ^B	6.08 ±2.95 ^a	—
Total	7.45 ±2.93 ^a	7.18 ±2.97 ^a	17.55 ±5.95 ^b	—	—
<i>P</i> *	—	<.001	—	—	—

CT, Crowntec; EN, Enamic; VS, VarseoSmile Crown Plus. Different superscript uppercase letters indicate significant differences between restorative material-resin cement pairs, while different superscript lowercase letters indicate significant differences between restorative materials or resin cements. Total values derived from pooled data of each restorative material or each resin cement *P* value of main factor restorative material indicated as *P** and *P* value of main factor resin cement indicated as *P*** (*P*<.05).

loop attached to a universal testing machine (Zwick Z1.0 TN; Zwick) at a crosshead speed of 1 mm/min. The wire loop was positioned at the base of the cemented restorative material cylinders in contact with the substrate. The operator (D.Y.) who performed the cementation procedures performed the SBS tests. The maximum force at debonding was recorded (N), and the SBS (MPa) was calculated by using the formula: SBS (MPa)=Maximum force (N)/Bonding area (mm²).

The bonding area was equal to the diameter of the luted cylinders of restorative material (5 mm). Each substrate surface was then evaluated by using a microscope (M420; Leica) that was integrated with a light source (CLS 150X; Leica) and a fiber optic illuminator (Intralux 150H; Volpi) under $\times 12$ magnification for failure analysis. Failure modes were categorized as: Type 1: Adhesive failure at the substrate surface; Type 2: Adhesive failure between the resin cement and restorative material; Type 3: Cohesive failure in the substrate; Type 4: Cohesive failure in the resin cement; Type 5: Cohesive failure in the restorative material; or Type 6: Mixed failure (simultaneous presence of 2 of the Types 1 to 5 failure modes).¹⁷

Considering that the data were normally distributed according to the Shapiro-Wilk test, 2-way analysis of variance tests with restorative material and resin cement as main factors were used to analyze SBS data within each substrate. Further analyses were conducted by using Tukey honestly significant difference tests. A statistical analysis software program (IBM SPSS Statistics, v23; IBM Corp) was used for all analyses ($\alpha=.05$).

RESULTS

The SBS results for dentin are presented in Table 2. The factor restorative material had a statistically significant effect on SBS (*P*<.001), whereas neither the factor resin cement (*P*=.064) nor the interaction between the 2 main factors (*P*=.082) had a significant effect. Among the tested restorative materials, EN had the highest SBS (*P*<.001), while CT and VS had lower SBS values that were statistically similar (*P*=.145).

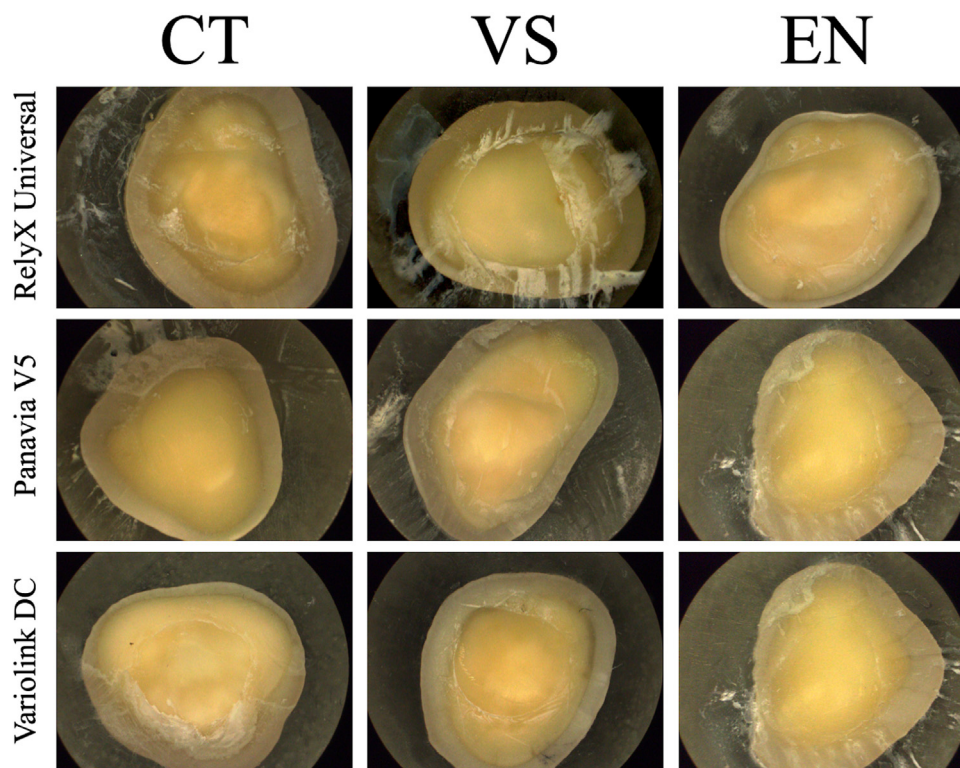


Figure 1. Representative microscope images (original magnification $\times 12$) showing failures of each restorative material-resin cement pair bonded to dentin. CT, Crowntec; EN, Enamic; VS, VarseoSmile Crown Plus.

The SBS results for titanium are presented in Table 3. The factors restorative material and resin cement and the interaction between the main factors had a significant effect ($P < .001$). Among the tested restorative material-resin cement pairs, EN had the highest SBS when cemented with RelyX and Panavia ($P < .001$), whereas CT and VS cemented with Variolink had the lowest SBS ($P \leq .004$). All restorative materials had significantly lower SBS when Variolink was used ($P \leq .010$), while Panavia and RelyX led to statistically similar SBS within each restorative material ($P \geq .229$). The differences among the other restorative material-resin cement pairs were not significant ($P \geq .170$). Regardless of the resin cement, EN had the highest SBS among the restorative materials ($P < .001$), while CT and VS had statistically similar SBS ($P = .856$). RelyX had the highest and Variolink had the lowest SBS ($P \leq .013$).

Figures 1 and 2 show representative images of the dominant failure mode observed for each restorative material-resin cement pair for each substrate, and Table 4 summarizes the failure modes observed. For most of the groups, failures were predominantly mixed (Type 6). On the dentin surfaces, the main part of the fracture surface within these mixed failures consisted of cohesive failure, while on the titanium surfaces, adhesive failure dominated those mixed failures.

DISCUSSION

This in vitro study evaluated the bond strength of two additively manufactured composite resins to dentin and titanium when bonded with three different dual-polymerizing resin cements compared with that of a subtractively manufactured polymer-infiltrated ceramic. The restorative material type affected the bond strength to both substrates, whereas the resin cement type affected the bond strength only to titanium. Therefore, the null hypothesis was partly rejected.

The subtractively manufactured polymer-infiltrated ceramic EN had significantly higher bond strengths than the two additively manufactured composite resins to both dentin and titanium, regardless of the resin cement used. EN was used as a control group since it has been clinically established as a material with a polymer network base for the fabrication of definitive prostheses,¹⁰ which is also the indication for CT and VS. The better bond strength to dentin of EN over that of more composite resin-like materials was also reported by Cekic-Nagas et al,¹¹ who attributed this difference to microstructural differences among the tested materials. Graf et al⁶ compared the pull-off bond strengths of EN and VS crowns bonded to a glass fiber-reinforced high-performance polymer with dentin-like behavior and reported the significantly higher bond strengths of EN,

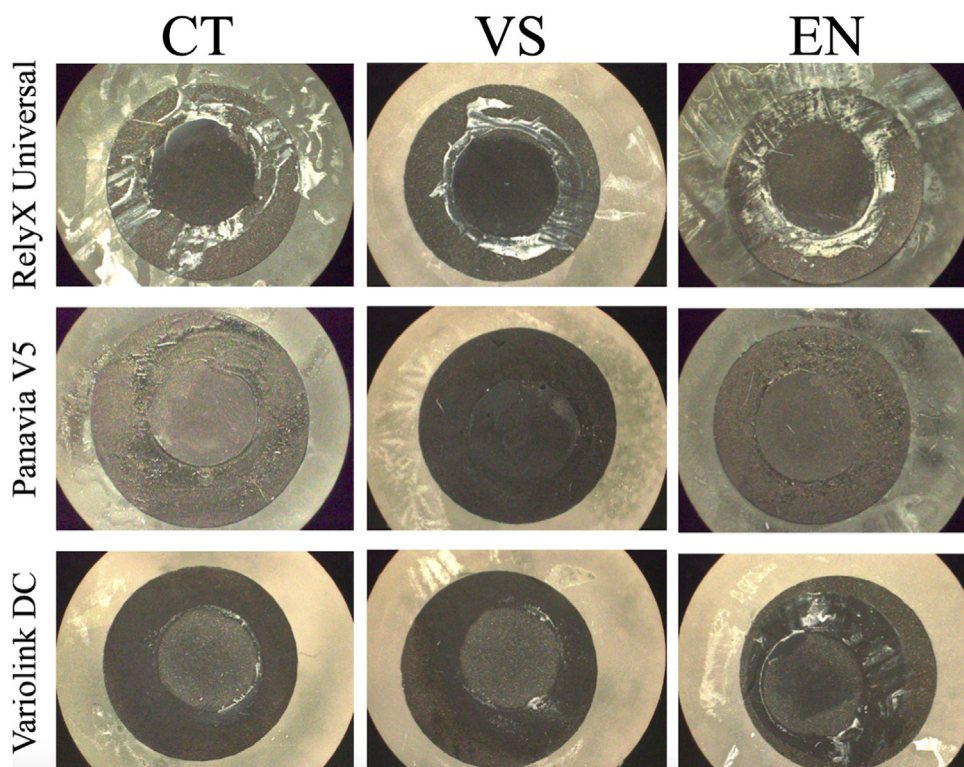


Figure 2. Representative microscope images (original magnification $\times 12$) showing failures of each restorative material-resin cement pair bonded to titanium. CT, Crowntec; EN, Enamic; VS, VarseoSmile Crown Plus.

Table 4. Failure mode within each substrate (D: dentin, T: titanium; $n=10$). Type 1: Adhesive failure at the substrate surface; Type 2: Adhesive failure between resin cement and restorative material; Type 3: Cohesive failure in substrate; Type 4: Cohesive failure in resin cement; Type 5: Cohesive failure in restorative material; Type 6: Mixed failure

Failure Mode	CT						VS						EN						
	RelyX		Panavia		Variolink		RelyX		Panavia		Variolink		RelyX		Panavia		Variolink		
	D	T	D	T	D	T	D	T	D	T	D	T	D	T	D	T	D	T	
Type 1	–	–	–	1	–	6	–	–	–	2	–	5	–	–	–	–	–	–	–
Type 2	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
Type 3	1	–	–	–	–	–	–	–	–	–	–	–	3	–	5	–	–	–	–
Type 4	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
Type 5	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
Type 6	9	10	10	9	10	4	10	10	10	8	10	5	7	10	5	10	10	10	10

CT, Crowntec; EN, Enamic; VS, VarseoSmile Crown Plus.

which was consistent with the findings of the present study. One possible explanation for the better bonding performance of EN over CT and VS in the present study would be a higher degree of conversion from the controlled and optimized polymerization of the blocks from which the specimens were manufactured.⁸ Statistically similar bond strength results of CT and VS support this hypothesis and might indicate a potential bond-deteriorating effect of residual monomer at the restorative material-resin cement interface. Even though CT and VS specimens were postpolymerized in a xenon polymerization device following the manufacturer's

recommendations, longer light polymerization may increase their degree of conversion and lead to higher bond strength, a possibility that should be further investigated. Another potential reason for the better bonding performance of EN over CT and VS may have been the higher filler content of EN, as there is a positive correlation between the filler content of resin-based materials and bond strength.³³ An additional explanation may be the surface treatments. In accordance with the manufacturers' instructions, CT and VS were airborne-particle abraded, while EN, having the highest ceramic content of the tested materials, was etched with hydrofluoric acid

and subsequently treated with a ceramic primer that comprised phosphate monomers. A previous study on the SBS of different CAD-CAM ceramic-glass polymers also reported higher bond strengths for EN compared with materials with lower ceramic content, even though they were also acid etched.¹² Therefore, it may be speculated that EN would have had higher bond strength than CT and VS, even if they had been treated with hydrofluoric acid and ceramic primer. Nevertheless, this hypothesis needs to be supported by studies that investigate the effect of surface treatments on the bond strength of the additively manufactured composite resins tested in the present study. In addition, evaluations of how the pretreatments affect the surface topography should be carried out to improve the understanding of the bonding of these composite resins. The SBS values of EN measured in dentin are within the range of the values reported previously,⁹ in which five different resin cements were tested. The facts that in the previous study some resin cements resulted in a slightly higher SBS than in the present study may have been because of differences in the resin cements tested and in the pretreatment of EN.

Even though the type of resin cement did not significantly affect the bond strength to dentin, it did affect the bond strength to titanium in that Variolink DC led to significantly lower bond strength for all three restorative materials. Considering that all adhesives used in the present study contained the phosphate monomer 10-methacryloyloxydecyl dihydrogen phosphate, which enhances bond strength, it seems more likely that this difference was associated with the chemical compositions of the resin cements. Thus, variations in the choice and proportion of monomers^{34,35} and in the type and load of fillers^{16,18} influence the degree of conversion and mechanical properties of resin cements, and therefore also polymerization shrinkage and stress and ultimately bond strength.^{36,37} Likewise, the type and concentration of photosensitizers determine not only the degree of conversion but also the polymerization rate and thereby the polymerization stress.^{38,39}

Limitations of the present study included that no aging was performed before bond strength testing. Bond strength values may decrease clinically as restorative materials are subjected to frequent intraoral thermal changes, and aging has been reported to reduce the bond strength of some resin cements.¹² The resin cements were chosen because of their chemical similarities; other types of resin cement may lead to different results. All additively manufactured specimens were fabricated by using the same printer, and bond strengths may have differed if other additive manufacturing technologies had been used. An SBS test was preferred in the present study, as no additional, and possibly traumatic, processing was required after cementation,

which might have stressed the bond and/or led to pre-test failures. However, the SBS test might generate localized high-stress areas that could lead to an under-estimation of the bond strength;⁴⁰ thus, the results of the present study should be confirmed with other bond strength tests. Future studies should also elaborate on the findings of the present study, investigating how fabrication parameters such as layer thickness or build angle affect the bond strength of additively manufactured definitive composite resins. In addition, studies on the fracture resistance and survival of restorations fabricated by using tested composite resins and cemented to different substrates with resin cements are needed to clarify the indications and limitations of these materials.

CONCLUSIONS

Based on the findings of the present study, the following conclusions were drawn:

1. Bond strength to dentin and titanium was affected by the additively and subtractively manufactured materials tested, while the resin cement type only affected bond strength to titanium.
2. Regardless of the substrate and the resin cement, the additively manufactured composite resins tested had lower bond strength than that of the subtractively manufactured polymer-infiltrated ceramic.
3. Among the resin cements tested, Variolink DC led to the lowest bond strength between the restorative materials and titanium.

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