

In vitro Bonding of Prosthodontic Adhesives to Dental Alloys

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In vitro tensile bond strengths were determined for three adhesive cements and two resin-bonded bridge cements to two alloys, each prepared by two methods: sandblasted Ni-Cr-Be alloy (I), electro-etched Ni-Cr-Be alloy (II), sandblasted Type IV gold alloy (III), and tin-plated Type IV gold alloy (IV). Storage conditions of 24 hours at 37°C and 30 days at 70°C were evaluated. The highest bond strengths were obtained for the electro-etched Ni-Cr-Be alloy, and all bond failures were cohesive. At both 24 hours and 30 days, the adhesive cements had the highest bond strengths to the other alloy/surface preparations (I, III, and IV). The adhesive cements usually failed cohesively under these conditions, whereas the resin-bonded bridge cements failed adhesively at the cement-alloy interface. Storage for 30 days at 70°C caused average decreases of 30%, 5%, 15%, and 32% for alloy/surface preparations I to IV, respectively.

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Introduction.

The metal-reinforced resin-bonded bridge (Maryland bridge), as described by Tanaka *et al.* (1979) and Livaditis and Thompson (1982), depends on the electrolytic etching of a Ni-Cr-Be alloy to achieve mechanical bonding between the resin cement and the alloy. Yamashita and Yamami (1982) proposed treatment of the metal surface by sandblasting with 50- μ m aluminum oxide to achieve bonding between oxides formed on the alloy surface and an adhesive cement. Strong bonds were obtained with oxides of nickel, chromium, cobalt, and tin. These results suggested that tin-plating of Type IV gold alloy might allow it to be used as a substrate for a bonded bridge.

The purpose of this study was to determine *in vitro* tensile bond strengths of three adhesive cements and two resin-bonded bridge cements to two alloys, each with two surface preparations: sandblasted Ni-Cr-Be alloy, electro-etched Ni-Cr-Be alloy, sandblasted Type IV gold alloy, and tin-plated Type IV gold alloy. Storage conditions of 24 hours at 37°C and 30 days at 70°C were evaluated.

Materials and methods.

Five commercial resin cements, as described in Table 1, were evaluated. Three cements were adhesive cements (A, P, and S) and two were resin-bonded bridge cements (C and K).

The Ni-Cr-Be and Type IV gold alloys used are also described in Table 1. Specimens (8 × 8 mm square and 1.5 mm in thickness) of each alloy were prepared by a lost-wax technique. The Ni-Cr-Be and Type IV gold alloys were invested (Hi Temp, #04766040X, Whip-Mix Corp., Louisville, KY; and Luster Cast, #111183 1316, Sybron/Kerr, Romulus, MI, respectively).

Two different surfaces of the Ni-Cr-Be alloy were prepared — sandblasted (I) and electro-etched (II). The sandblasted Ni-

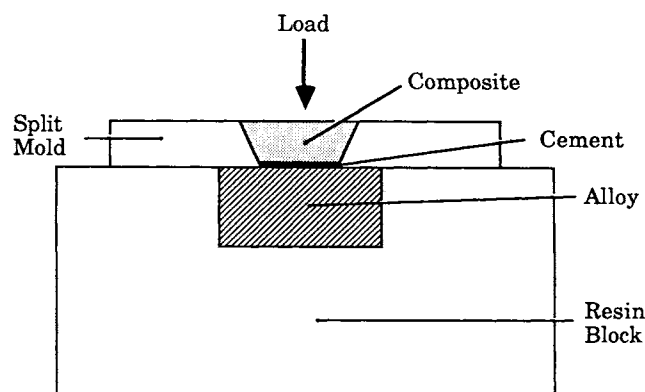


Fig. 1—Assembly used to prepare bond strength specimens.

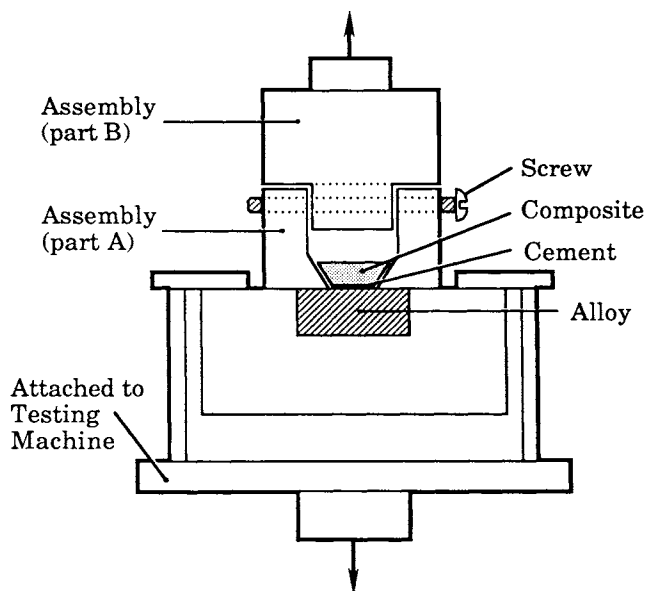


Fig. 2—Assembly used to test bond strength specimens in tension.

Cr-Be surfaces were prepared with 50- μ m-diameter Al₂O₃ particles (Comco, Inc., Burbank, CA). The electro-etched Ni-Cr-Be surfaces were prepared following manufacturer's instructions by: (a) sandblasting the area to be etched with 50- μ m Al₂O₃ particles, (b) etching for three minutes in a solution of 10% by volume H₂SO₄ with methanol (55 mL H₂SO₄, 60 mL CH₃OH, and 500 mL distilled water) using magnetic stirring and a current of 56 milliamps, and (c) cleansing in an ultrasonic bath for 10 minutes with 18% hydrochloric acid. The etched surfaces were then examined under magnification.

Two different surfaces of the Type IV gold alloy were prepared — sandblasted (III) and tin-plated (IV). The sandblasted Type IV gold surfaces were prepared with 50- μ m-diameter Al₂O₃ particles. The tin-plated Type IV gold surfaces were prepared (Kura Ace plater, Model 100, Kuraray Co., Ltd., Osaka, Japan) following manufacturer's instructions.

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TABLE 1
PRODUCTS, BATCH NUMBERS, AND MANUFACTURERS OF PRODUCTS TESTED

| Code Product | Batch Number | Manufacturer |
|---|---|--|
| Adhesive cements: | | |
| A ABC | base - Aug. 1987 catalyst - Aug. 1987 etchant - 6121 | Vivadent Schaan, Liechtenstein |
| P Panavia Ex | powder - PN-0404 liquid - PL-1011 etchant - EL023 Oxyguard- OG-170 | Kuraray Co., Ltd. Osaka, Japan |
| S Super-Bond C & B | polymer - 50301 monomer - none catalyst - 517 etchant - 50701 | Sun Medical Co., Ltd. Kyoto City, Japan |
| Resin-bonded bridge cements: | | |
| C Conclude | paste A - 5018 paste B - 5BC resin A - 5M1 resin B - 5B1 etchant - 3R1 | 3M Dental Products St. Paul, MN 55144 |
| K Kerr Maryland Bridge Cement | base - 61122 catalyst - 61122 resin base - 61125 resin catalyst - 61125 etchant - 61135 | Sybron/Kerr Romulus, MI 48174 |
| Ni-Cr-Be alloy: Rexillium III | 041785-52 | Rx Jeneric Gold Co., Inc. Wallingford, CT 06492 |
| Type IV gold alloy: Jelenko Type IV Gold | 1871-022686 | J.F. Jelenko & Co. Armonk, NY 10504 |

TABLE 2
BOND STRENGTH OF ADHESIVE AND RESIN-BONDED BRIDGE CEMENTS TO Ni-Cr-Be ALLOY, AND TYPE IV GOLD ALLOY.

| Condition | Tensile Bond Strength kg/mm ² | | | | |
|----------------------------------|--|-------------|-------------|-------------|-------------|
| | A | P | S | C | K |
| Ni-Cr-Be Alloy (Sandblasted) | | | | | |
| 24 hours | 1.58 (0.28) | 2.02 (0.18) | 2.45 (0.54) | 1.02 (0.22) | 1.44 (0.23) |
| 30 days | 0.76 (0.15) | 1.04 (0.14) | 2.54 (0.22) | 0.53 (0.18) | 1.33 (0.29) |
| Ni-Cr-Be Alloy (Electro-etched) | | | | | |
| 24 hours | 2.01 (0.53) | 2.23 (0.33) | 2.80 (0.27) | 1.92 (0.21) | 2.58 (0.60) |
| 30 days | 1.84 (0.09) | 2.54 (0.08) | 2.81 (0.39) | 1.89 (0.45) | 1.74 (0.49) |
| Type IV Gold Alloy (Sandblasted) | | | | | |
| 24 hours | 0.89 (0.13) | 1.12 (0.22) | 2.24 (0.59) | 1.01 (0.12) | 0.96 (0.31) |
| 30 days | 0.79 (0.20) | 1.36 (0.26) | 2.18 (0.27) | 0.53 (0.19) | 0.62 (0.23) |
| Type IV Gold Alloy (Tin-plated) | | | | | |
| 24 hours | 1.62 (0.26) | 1.88 (0.08) | 2.60 (0.42) | 0.59 (0.14) | 1.31 (0.20) |
| 30 days | 0.50 (0.12) | 1.33 (0.26) | 2.54 (0.09) | 0.62 (0.13) | 0.43 (0.16) |

*Mean value of five replications with standard deviation in parentheses.

The assembly used to prepare bond strength specimens is shown in Fig. 1. Adhesive tapes with a 5-mm hole were placed on the alloy surfaces to limit the area to be bonded. Inverted truncated cones (Barakat and Powers, 1986) of a polymerized composite resin (P-30, 3M Co., St. Paul, MN) were bonded to each substrate with each of the cements under a pressure of 0.075 kg/mm² applied for seven minutes. Eight minutes later, the bonded samples were stored in distilled water at 37°C for 24 hours or at 70°C for 30 days.

The cements were mixed according to manufacturers' instructions as follows: Cement A was mixed with equal volumes of base and catalyst pastes. A primer (Contact Cement, Vivadent, Schaan, Liechtenstein) was used in conjunction with cement A for all samples. Cement P was mixed to a powder-liquid ratio of 3.1/1 and was used in conjunction with a coating

(Oxyguard) to inhibit air during polymerization. Cement S was mixed by means of a brush technique. Cements C and K were mixed with equal volumes of base and catalyst pastes and were used in conjunction with their self-cured enamel bonding agents.

The assembly used to test bond strength is shown in Fig. 2. The samples were debonded in tension in a testing machine (Instron, Model TT-BM, Instron Corp., Canton, MA) at a cross-head speed of 0.05 cm/min, as described by Barakat and Powers (1986). Bond strengths were calculated as the load at failure, divided by the nominal area (19.6 mm²) of the cemented sample. Locations of failure were determined by optical microscopy and were recorded as within the cement (cohesive failure) or at the cement-alloy interface (adhesive failure). No adhesive failures at the cement-composite interface or cohesive failures within the alloys were observed.

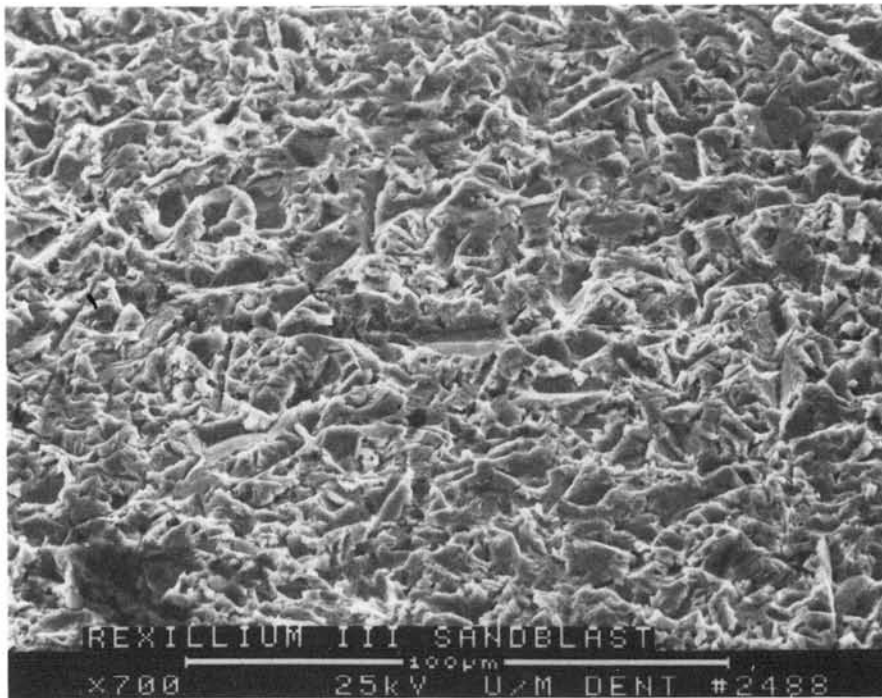


Fig. 3—Scanning electron photomicrograph of sandblasted surface of Ni-Cr-Be alloy (magnification 700X).

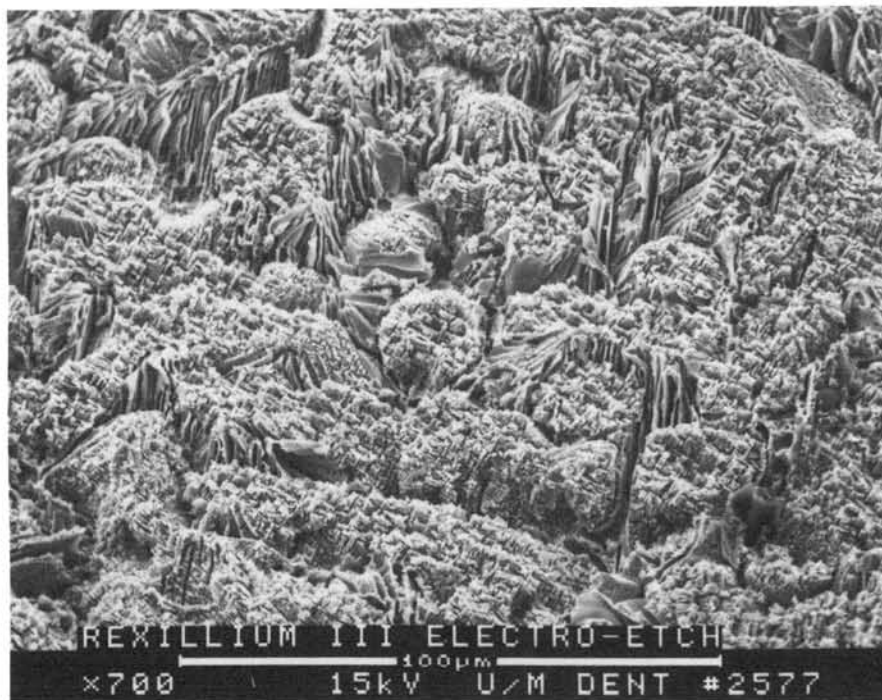


Fig. 4—Scanning electron photomicrograph of electro-etched surface of Ni-Cr-Be alloy (magnification 700X).

Five replications were tested for each group of five cements, two alloys, two surface preparations, and two storage conditions. Mean values and standard deviations were calculated. Data were analyzed by a four-factor analysis of variance (Dalby, 1968), and means were compared with a Tukey multiple comparison interval (Guenther, 1964).

Results.

Mean values and standard deviations of the bond strength data are shown in Table 2. The Tukey interval at the 95% level

of confidence for comparisons of means between two alloys, two surface preparations, and two storage conditions was 0.08 kg/mm². The Tukey interval for comparisons among the five cements was 0.18 kg/mm². The types of bond failures are listed in Table 3.

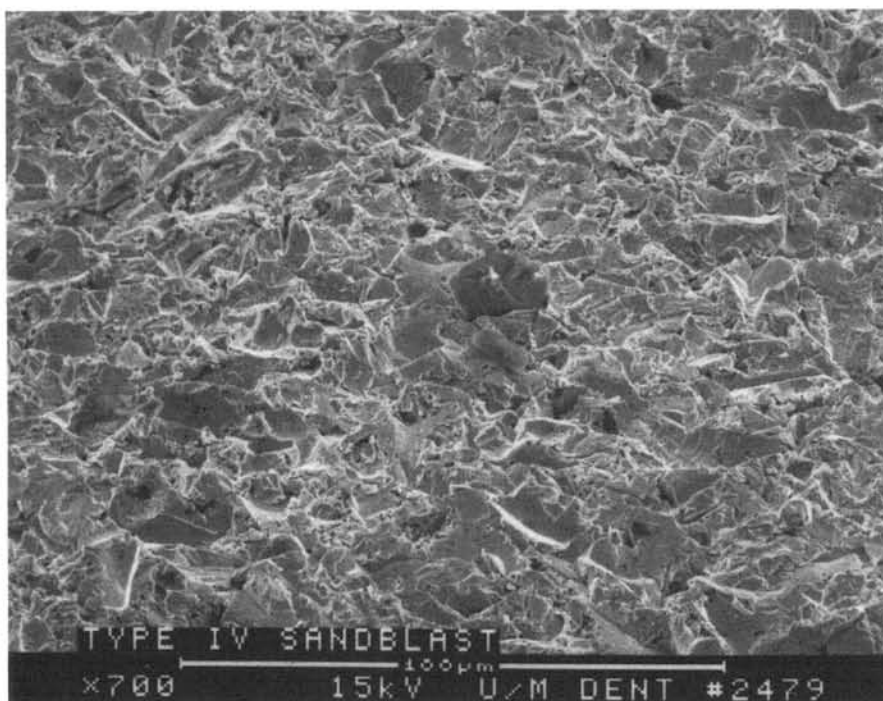
The factors of alloy, surface preparation, storage condition, and cement and their interactions were all statistically significant. For all of the cements at both storage conditions, the highest bond strengths were obtained for the electro-etched Ni-Cr-Be alloy. All bond failures for these conditions were cohesive. Bond strengths to the electro-etched Ni-Cr-Be alloy at

TABLE 3
TYPE OF BOND FAILURE OF ADHESIVE AND RESIN-BONDED BRIDGE CEMENTS TO Ni-Cr-Be ALLOY AND TYPE IV GOLD ALLOY

| Condition | A | P | S | C | K |
|----------------------------------|-----------|----------|----------|----------|----------|
| Ni-Cr-Be Alloy (Sandblasted) | | | | | |
| 24 hours | cohesive* | cohesive | cohesive | adhesive | adhesive |
| 30 days | adhesive | cohesive | cohesive | adhesive | adhesive |
| Ni-Cr-Be Alloy (Electro-etched) | | | | | |
| 24 hours | cohesive | cohesive | cohesive | cohesive | cohesive |
| 30 days | cohesive | cohesive | cohesive | cohesive | cohesive |
| Type IV Gold Alloy (Sandblasted) | | | | | |
| 24 hours | cohesive | cohesive | cohesive | adhesive | adhesive |
| 30 days | adhesive | adhesive | cohesive | adhesive | adhesive |
| Type IV Gold Alloy (Tin-plated) | | | | | |
| 24 hours | cohesive | cohesive | cohesive | adhesive | adhesive |
| 30 days | adhesive | cohesive | cohesive | adhesive | adhesive |

*Determined from five replications.

Fig. 5—Scanning electron photomicrograph of sandblasted surface of Type IV gold alloy (magnification 700X).



both 24 hours and 30 days were always larger than those to the sandblasted Ni-Cr-Be alloy. Bond strengths to the tin-plated Type IV gold alloy at 24 hours were larger than those to the sandblasted Type IV gold alloy, except for cement C. Bonding to sandblasted Ni-Cr-Be alloy at 24 hours was greater than bonding to sandblasted Type IV gold alloy, except for cement C.

Overall, the highest bond strengths at both 24 hours and 30 days were obtained with the adhesive cements. At 24 hours, bond failures for the adhesive cements were cohesive, whereas those for the resin-bonded bridge cements were always adhesive. One adhesive cement (S) had the highest values of bond strength and showed little change after storage at 70°C. The two resin-bonded bridge cements were least affected by storage at 70°C when bonded to the electro-etched surface of the Ni-Cr-Be alloy.

Storage for 30 days at 70°C caused average decreases of 30%, 5%, 15%, and 32% for alloy/surface preparations I to IV, respectively. Bond strengths to the electro-etched surface of the Ni-Cr-Be alloy were least affected by the 30-day, 70°C storage condition, and bond failures remained cohesive for all cements. The adhesive cements (especially P and S) were less

affected by the 70°C storage than were the two resin-bonded bridge cements. In two instances, the bond strength of cement P improved by 14 to 21% after storage at 70°C. Bond failures of cements P and S remained cohesive after storage at 70°C, whereas those of cements A, C, and K changed to adhesive for alloy/surface preparations I, III, and IV.

Scanning electron photomicrographs of typical surfaces of sandblasted Ni-Cr-Be alloy, electro-etched Ni-Cr-Be alloy, sandblasted Type IV gold alloy, and tin-plated Type IV gold alloy before bonding are shown in Figs. 3 to 6, respectively. The alloy surfaces that debonded adhesively were similar in appearance to those shown in Figs. 3 to 6. The alloy surfaces that debonded cohesively were covered with cement.

Discussion.

The higher bond strengths of the adhesive cements compared with the resin-bonded bridge cements result from the adhesive components of the adhesive cements in addition to mechanical bonding. The adhesive components are probably organic esters such as methacryloxyethylphenyl phosphate or 4-

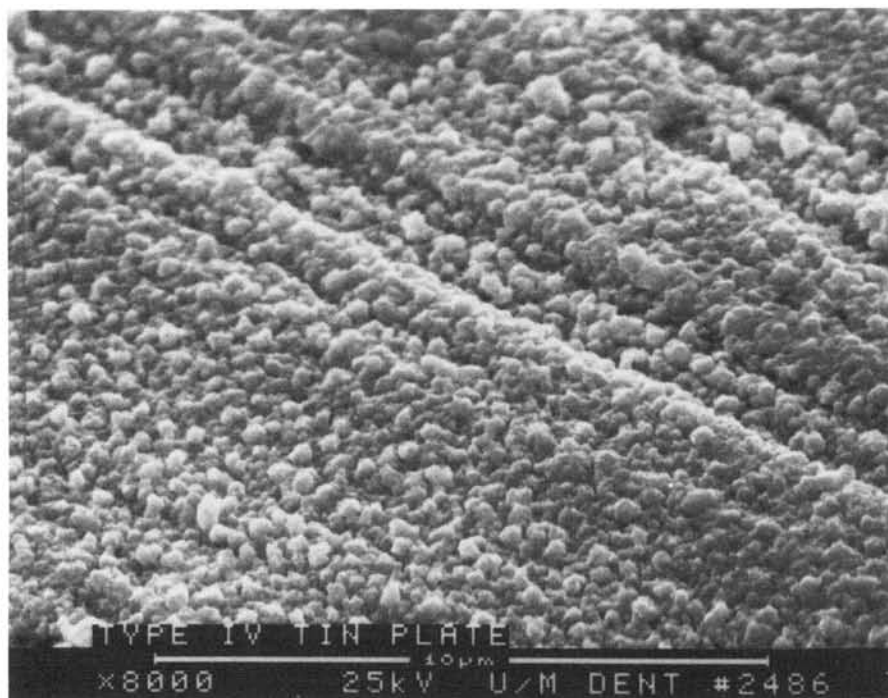


Fig. 6—Scanning electron photomicrograph of tin-plated surface of Type IV gold alloy (magnification 8000X).

methacryloxyethyl-trimellitic anhydride, which bond chemically to metal oxides. Good penetration is expected from all three adhesive cements, since cement A uses a resin primer and cements P and S are powder/liquid systems.

The better bonding of the adhesive cements to the sandblasted Ni-Cr-Be alloy than to the sandblasted Type IV gold alloy suggests that the adhesive components have a higher affinity to the oxides available at the surface of the Ni-Cr-Be alloy than to tin oxide. Tin-plating of the gold alloy did improve its bond strength compared with the sandblasted surface, but only to a level similar to that of the sandblasted Ni-Cr-Be alloy. Tin-plating may also have increased the surface area available for bonding.

The two resin-bonded bridge cements (C, K) bonded better to the electro-etched Ni-Cr-Be alloy than to the other alloy/surface preparations. Scanning electron microscopy suggests that the area of the electro-etched surfaces is larger and more irregular than that of the other alloy/surface preparations. The higher bond strength of cement K compared with that of cement C may be the result of higher penetration of cement K into the surface irregularities. High penetration would be favored by a cement with low viscosity and high surface tension and by an alloy surface with high wettability.

In vitro bond strengths of adhesive cements P and S to non-etched dentin and to non-etched and etched enamel were greater than bond strengths of the resin-bonded bridge cements C and K (Powers *et al.*, in press). These adhesive cements also bonded

better to the alloys than to tooth structure under all conditions. These results suggest that the use of adhesive cements P and S with sandblasted Ni-Cr-Be and type IV gold alloys may be clinically acceptable.

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