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**Original**

# Influence of storage conditions and effect of metal priming agents on bond strength of resin-modified glass ionomers to gold alloy

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**Abstract: This study evaluated the influence of water storage conditions and the effect of metal priming agents on bond strength and durability of four luting agents joined to gold alloy. Disk specimens were cast from a gold alloy (Degudent U), and the surfaces were ground flat with abrasive paper. Three surface conditions employed were: unprimed, primed with Alloy Primer, and primed with Metaltite. Three resin-modified glass ionomers (RMGIs; Vitremer Luting Cement, Fuji Lute, and Xeno Cem Plus) and a resin adhesive (Super-Bond C&B) were used for bonding the gold alloy. Unprimed specimens bonded either with Fuji Lute or with Super-Bond C&B were immersed in water at 5, 37, and 55°C for 7 days, or subjected to thermocycling (5,000 cycles; 5°C, 1 min and 55°C, 1 min). In addition, specimens were bonded with 12 combinations comprising three surface conditions and four luting agents, and thermocycled for 20,000 cycles. Shear bond strengths were then determined and analyzed statistically. Thermocycling was useful for evaluation of the bonding durability of RMGIs. Application of two metal priming agents combined with RMGIs considerably enhanced the bond strength to the gold alloy. (J Oral Sci 51, 21-28, 2009)**

Keywords: bonding; gold alloy; primer; resin-modified glass ionomer; thione.

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## Introduction

The use of resin-modified glass ionomers (RMGIs) for bonding noble metal alloys has increased substantially. This trend is probably attributable to improvement of material properties as well as the spreading application of collarless porcelain-fused-to-metal restorations, especially in the anterior maxillary region. Seating restorations and fixed partial dentures incorporating RMGI materials are now practicable due to improved bond strength (1,2), adequate sealing ability (3), and acceptable cement thickness (4).

Vallittu and Forss (1) reported that RMGI cements seemed to have better bonding properties than conventional glass ionomer cements. Swartz et al. (2) thereafter confirmed that microabraded and tin-plated alloy luted with a RMGI exhibited strong bonding that had 73% of the bond strength of specimens luted with resin adhesive. However, Piwowarczyk et al. (5) reported that the bond strengths of zinc-phosphate, glass ionomer, and RMGI cements were inferior to those of resin adhesives when stored in water for 14 days followed by thermocycling. In addition, Burmann et al. (6) reported that bond strength of glass ionomer to gold alloy was inferior to that of composite adhesive.

Polymerizable monomers are being incorporated into RMGI compositions. This has led to application of metal priming agents in combination with RMGIs for bonding of casting alloys. Although a number of papers have reported the bonding of noble metal alloys with priming agents and adhesive resins (7-10), only limited information is available about the effect of priming agents on the bond strength of RMGIs to noble alloys (11,12).

The current study evaluated the influence of water storage conditions on the bond strength and durability of a representative RMGI luting agent. Also, bond strengths of three RMGIs and a resin adhesive to gold alloy were determined with the aim of evaluating the effect of metal priming agents on bonding durability.

## Materials and Methods

### Materials

Information on the materials is summarized in Table 1. A gold alloy for metal-ceramic restorations (Degudent U, DeguDent GmbH, Hanau, Germany) was used as the substrate material. Two single-liquid metal priming agents (Alloy Primer, Kuraray Medical Inc., Tokyo, Japan; and Metaltite, Tokuyama Dental Corp., Tokyo, Japan) were selected. Both priming agents contained an organic sulfur compound; 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione (VTD) in Alloy primer, and 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6) in Metaltite.

Table 1 Materials assessed

| Material | Abbreviation | Component | Lot number | Composition |
|---|---|---|---|---|
| Gold alloy | | | | |
| Degudent U | | | 10011271 | Au 77.3, Pt 9.8, Pd 8.9, Others 4.0 (mass%) |
| Metal priming agents | | | | |
| Alloy Primer | | | 00156A | VTD, MDP, Acetone |
| Metaltite | | | 01403 | MTU-6, Ethanol |
| Luting agents | | | | |
| Resin-modified glass ionomers (RMGIs) | | | | |
| Vitremer Luting Cement | VL | Powder | 20041030 | Fluoroaluminosilicate glass |
| | | Liquid | 20041030 | Water, HEMA, Copolymer of acrylic and itaconic acids |
| Fuji Lute | FL | Powder | 0711071 | Fluoroaluminosilicate glass |
| | | Liquid | 0711051 | Polyalkenoic acid, HEMA, Water, UDMA (13) |
| Xeno Cem Plus | XC | Powder | 385022 | Fluoroaluminosilicate glass, Photochemical initiators (14) |
| | | Liquid | 385022 | UDMA, HEMA, Polyalkenoic acid, Chemical initiators (14) |
| Resin adhesive | | | | |
| Super-Bond C&B | SB | Initiator | RL43 | TBB |
| | | Monomer | RK1 | 4-META, MMA |
| | | Opaque Ivory powder | RF1 | PMMA |

HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate;
4-META: 4-methacryloyloxyethyl trimellitate anhydride; MMA: Methyl methacrylate;
MTU-6: 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate; PMMA: Poly (methyl methacrylate);
TBB: Tri-*n*-butyl borane; UDMA: Dimethacryloxyethyl 2,2,4- (or 2,4,4-) trimethylhexamethylene diurethane;
VTD: 6- (4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione

Three RMGIs (Vitremer Luting Cement, VL, 3M ESPE, Seefeld, Germany; Fuji Lute, FL, GC Corp., Tokyo, Japan; and Xeno Cem Plus, XC, Dentsply-Sankin K.K., Tokyo, Japan) and a resin adhesive (Super-Bond C&B, SB, Sun Medical Co., Ltd., Moriyama, Japan) were selected as the luting agents. These RMGIs consist of powder and liquid. Each powder includes fluoroaluminosilicate glass. The liquid of VL consists of 30-40% copolymer of acrylic and itaconic acids modified with pendant methacrylate, 25-35% 2-hydroxyethyl methacrylate (HEMA), and 30-40% water. The liquid of FL consists of 22-24% polyalkenoic acid, 35-40% HEMA, 25% water, and 5-7% dimethacryloxyethyl 2,2,4- (or 2,4,4-) trimethylhexa-methylene diurethane (UDMA) (13). The liquid of XC contains UDMA, HEMA, and polyalkenoic acid, instead of water (14). The SB system is an acrylic resin adhesive consisting of initiator, monomer liquid, and powder.

## Influence of water temperature and thermocycling on bond strength

A total of 80 pairs of disks (10 mm and 8 mm in diameter by 2.5 mm thick) were cast from the gold alloy. All disks were ground with #1,500 silicon carbide abrasive paper and cleaned with acetone. A piece of tape 50 μm thick with a circular hole 5 mm in diameter was positioned on the surface of 10-mm-disks to define the bonding area. Ten sets of 8 specimens were then divided into 2 groups. Five sets were bonded with FL, which was spatulated according to the manufacturer's instructions. The other 5 sets were bonded with SB using brush-dip technique. Each luting agent was applied on the each size of disks and then bonded. Immediately after bonding, a constant vertical load of 5.0 N was applied to the bonded specimens for 30 min. Then the specimens were immersed to 37°C distilled water for 24 h. Each of 5 sets was divided into 5 groups. A control group was immediately evaluated by shear bond test. Three groups of each luting agent were stored at 5, 37, and 55°C distilled water for 7 days, respectively. The remaining group was thermocycled between 5°C and 55°C for 5,000 cycles (approximately 7 days; TC).

## Effect of priming agents on bond strength

Ninety-six disk pairs (12 sets of 8 specimens) were prepared, and they were divided into 12 combinations of 3 surface conditions and 4 luting agents. The 3 surface conditions were; 1) unprimed, 2) priming with Alloy Primer, and 3) priming with Metaltite. Four luting agents were VL, FL, XC, and SB. After priming or unpriming, the specimens were bonded with one of the 4 luting agents according to the manufacturer's instructions. The bonded specimens were immersed in distilled water at 37°C for 24 h, and subsequently thermocycled 20,000 cycles between 5°C water and 55°C water with a 1-min dwell time per bath.

## Bond strength test and scanning electron microscopic observation

After storage under each of the conditions, shear bond strength was measured with a mechanical testing device (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. Debonded surfaces were observed through an optical microscope (8×; SZX9, Olympus Corp., Tokyo, Japan), and the failure modes were classified into the following three categories; adhesive failure, cohesive failure within the luting agent, and a combination of adhesive and cohesive failure. Selected specimens from the control and the TC groups were observed with a scanning electron microscope (S-4300, Hitachi High-Technologies Corp., Tokyo, Japan) with an accelerating voltage of 15 kV.

## Statistical analyses

Equality of variance of the shear bond strength results was primarily analyzed by Levene test and F test. When the Levene test and F test did not show equality of variances, the Steel-Dwass test was performed to evaluate the effect of water temperature and thermocycling, priming agents, and the four luting agents. Mann-Whitney *U*-test was used to evaluate the difference in bond strengths between two groups. Significance level was set at 0.05 for all analyses.

# Results

Levene test and F test run on the bond strength results revealed that several groups did not exhibit equality of variance. The Steel-Dwass test was therefore applied to evaluate the influence of water temperature, thermocycling, priming agents, and luting agents on bond strength. In addition, the Mann-Whitney *U*-test was used for comparison of bond strengths between FL and SB luting agents under various storage conditions.

Table 2 shows the shear bond strength results as well as statistical categories of FL and SB for various storage conditions. Bond strength of FL varied from 2.2 MPa to 11.7 MPa. Although the bond strength of FL deteriorated significantly during storage in water, the reduction rate was not dependent on storage temperature (category b). Application of thermocycling considerably reduced the bond strength of FL (category c). Unlike FL, the bond strength of SB was not negatively affected after 7 days under all conditions. However, a significant difference was found between the 7-day group (stored at 5°C, category d) and the thermocycled group (category e). Mann-Whitney *U*-test revealed that bond strengths of FL were significantly

Table 2 Influence of storage conditions on bond strength (MPa) of two luting agents to gold alloy

| | | FL | | SB | | Difference between FL and SB* |
|---|---|---|---|---|---|---|
| 37°C, 24 h | Median | 11.7 | | 19.6 | | |
| | Mean | 11.7 | a | 20.9 | d, e | Significant, $P = 0.021$ |
| | SD | 2.4 | | 9.2 | | |
| 5°C, 7 days | Median | 4.8 | | 19.1 | | |
| | Mean | 5.2 | b | 19.9 | d | Significant, $P = 0.001$ |
| | SD | 1.3 | | 6.1 | | |
| 37°C, 7 days | Median | 6.5 | | 11.1 | | |
| | Mean | 6.3 | b | 11.9 | d, e | Significant, $P = 0.002$ |
| | SD | 1.9 | | 3.1 | | |
| 55°C, 7 days | Median | 6.9 | | 11.1 | | |
| | Mean | 6.9 | b | 11.0 | d, e | Significant, $P = 0.005$ |
| | SD | 1.5 | | 2.4 | | |
| TC, 5,000 | Median | 2.5 | | 8.0 | | |
| | Mean | 2.2 | c | 9.3 | e | Significant, $P = 0.001$ |
| | SD | 1.4 | | 3.2 | | |

*, Mann-Whitney *U*-test
TC, Thermocycling (1 cycle = 5°C, 1 min and 55°C, 1 min); SD, Standard deviation
Identical letters in the vertical columns indicate that the values are not statistically different (Steel-Dwass grouping, $P > 0.05$).

Table 3 Failure modes of FL-alloy and SB-alloy debonded specimens

| | 37°C, 24 h | | 5°C, 7 days | | 37°C, 7 days | | 55°C, 7 days | | TC, 5,000 | |
|---|---|---|---|---|---|---|---|---|---|---|
| | FL | SB | FL | SB | FL | SB | FL | SB | FL | SB |
| A | 8 | 8 | 8 | 7 | 8 | 8 | 8 | 8 | 8 | 6 |
| C | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| AC | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 2 |

A, Adhesive failure; C, Cohesive failure; AC, Combination of adhesive and cohesive failures

lower than those of SB under all storage conditions.

Table 3 shows the results of failure mode analysis. The majority of specimens were classified as showing adhesive failure. Several specimens in the SB groups, i.e., stored at 5°C for 7 days and subjected to 5,000-thermocycling, showed a combination of adhesive and cohesive failure.

Table 4 shows the post-thermocycling bond strengths of the 4 luting agents joined to primed and unprimed gold alloy disks. Bond strength after 20,000-thermocycling varied from a minimum of only 0.2 MPa to a maximum of 42.2 MPa. For all of the 4 luting agents, the effect of the 2 priming agents was apparent. Specifically, bond strengths of the 4 luting agents (categories h, k, m, and p) to unprimed gold alloy were significantly lower than to primed gold alloy. The priming effect of Metaltite was comparable to that of Alloy Primer in combination with XC (category l), whereas Metaltite generated superior strength of bonding to Alloy Primer when the agent was used together with VL, FL, and SB (categories f, i, and n). The statistical categories of post-thermocycling bond

Table 4 Effect of priming agents on post-thermocycling bond strength (MPa) of four luting agents to gold alloy

| Priming | | Unprimed | | Alloy Primer | | Metaltite | |
|---|---|---|---|---|---|---|---|
| VL | Median | 5.3 | | 12.6 | | 31.8 | |
| | Mean | 5.3 | h | 12.0 | g | 30.4 | f |
| | SD | 1.5 | | 2.3 | | 2.7 | |
| FL | Median | 0.4 | | 31.0 | | 41.3 | |
| | Mean | 0.4 | k | 30.8 | j | 41.9 | i |
| | SD | 0.2 | | 3.9 | | 3.6 | |
| XC | Median | 0.2 | | 30.5 | | 31.8 | |
| | Mean | 0.2 | m | 30.8 | l | 29.0 | l |
| | SD | 0.1 | | 5.4 | | 6.3 | |
| SB | Median | 3.6 | | 35.5 | | 42.6 | |
| | Mean | 2.9 | p | 34.1 | o | 42.2 | n |
| | SD | 2.0 | | 4.3 | | 1.8 | |

Identical letters in the same line indicate that the values are not statistically different ($P > 0.05$).

strength under identical priming conditions are shown in Fig. 1. The shear bond strength in the unprimed group bonded with VL and SB (category q) was higher than that of FL and XC (category r). For the groups primed with Alloy Primer, the bond strength was lower for VL (category t) than for the other 3 groups (category s). The shear bond strength of FL or SB with Metaltite (category u) was higher than that of VL or XC with Metaltite (category v).

Table 5 summarizes the failure modes of 20,000-thermocycled specimens. All of the unprimed specimens showed adhesive failure. Three combinations – Metaltite-FL, Alloy Primer-XC, and Alloy Primer-SB – exhibited either adhesive failure or adhesive-cohesive failure. All of the Metaltite-SB specimens showed a combination of adhesive-cohesive failure after application of 20,000-thermocycling.

Figure 2 shows a scanning electron micrograph of a ground alloy specimen before bonding. Scratches generated by the abrasive paper can be seen. Debonded surfaces of the unprimed-FL combination are shown in Figs. 3 and 4. Remnants of FL can be seen on both surfaces of the specimens stored in water for 24 h and those subjected to 5,000-thermocycling. Debonded surfaces of the unprimed-SB combination are presented in Figs. 5 and 6. Splintered resin remnants can be detected on the surface of the specimen stored in water for 24-h (Fig. 5). After application of thermocycling, the surface appearance was similar to that of the unprimed-FL specimen (Fig. 6).

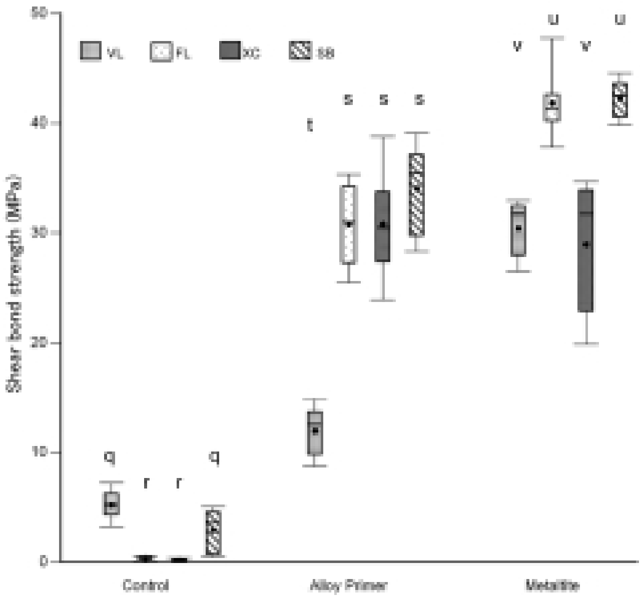


Fig. 1 Post-thermocycling bond strength of four luting agents to gold alloy under different priming conditions. Identical letters indicate that the bond strengths were not significantly different (Steel-Dwass grouping, $P > 0.05$).

## Discussion

The present study evaluated the effect of water storage on the bond strength to gold alloy of FL. As shown in Table 2, the reduction in bond strength of FL was remarkable after 7 days of immersion in water at 37°C. Also, the

Table 5 Failure modes of 20,000-thermocycling specimens

| | VL | | | FL | | | XC | | | SB | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | UP | AP | MT | UP | AP | MT | UP | AP | MT | UP | AP | MT |
| A | 8 | 8 | 8 | 8 | 8 | 4 | 8 | 4 | 8 | 8 | 4 | 0 |
| C | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| AC | 0 | 0 | 0 | 0 | 0 | 4 | 0 | 4 | 0 | 0 | 4 | 8 |

UP, Unprimed; AP, Alloy Primer; MT, Metaltite
A, Adhesive failure; C, Cohesive failure; AC, Combination of adhesive and cohesive failures

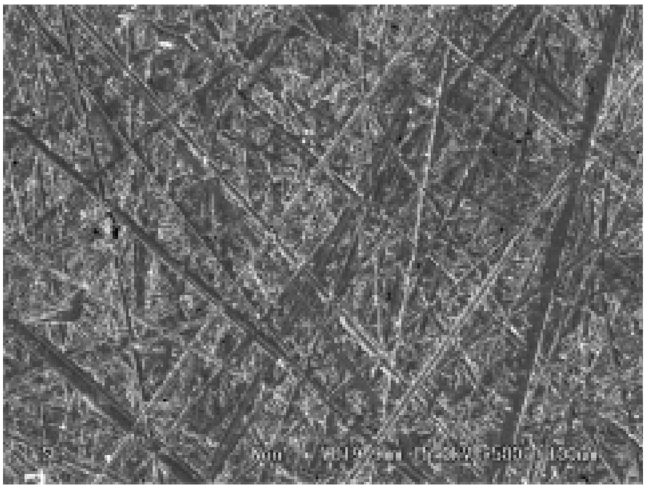

Fig. 2 Scanning electron micrograph of the gold alloy ground with #1,500 SiC abrasive paper.

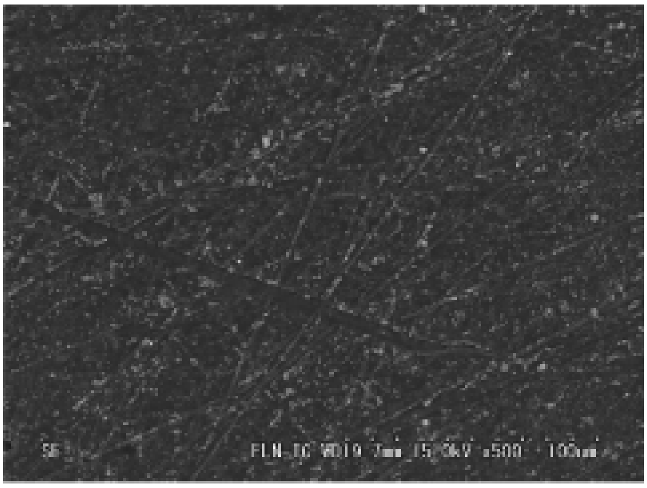

Fig. 4 Debonded surface of the unprimed FL specimen (5,000-thermocycling).

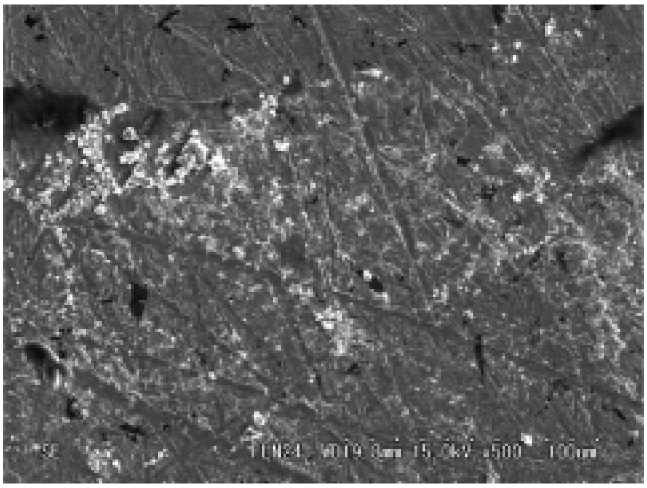

Fig. 3 Debonded surface of the unprimed FL specimen (24-h water stored).

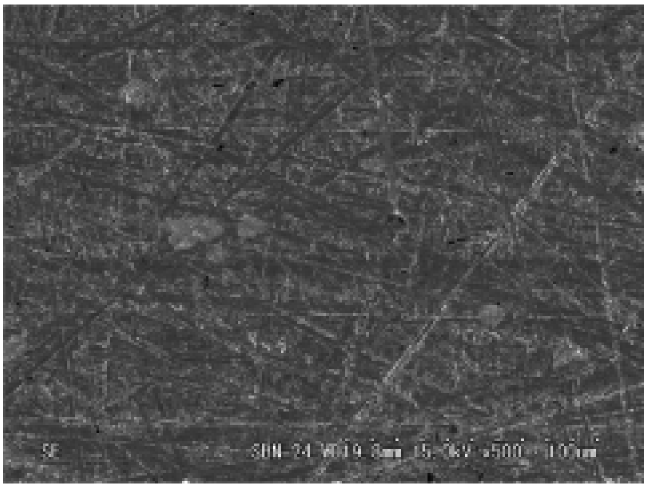

Fig. 5 Debonded surface of the unprimed SB specimen (24-h water stored).

reduction of FL bond strength was not dependent upon water temperature. These results suggest that 7-day immersion in water is sufficient for evaluating the durability of bonding to the gold alloy of a RMGI. However, no reduction in the bond strength of SB was apparent after 7 days of immersion in water, indicating that additional

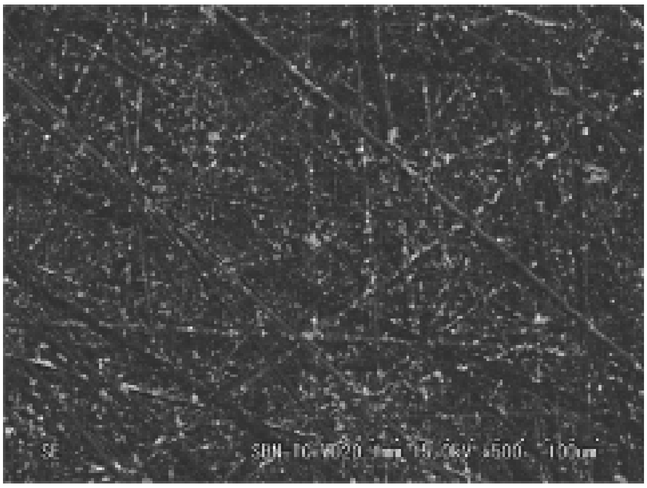


Fig. 6 Debonded surface of the unprimed SB specimen (5,000-thermocycling).

accelerated aging was required to evaluate the durability of the bond. The present study therefore used thermocycling (15) in water baths in combination with bond strength tests to evaluate the long-term durability of adhesive systems. This study employed 5,000-thermocycling, which required 7 days of immersion in water, before shear bond testing of SB. The mean bond strength of SB was reduced from 20.9 MPa (24-h, 37°C) to 9.3 MPa (5,000-thermocycled). These values, however, were not significantly different from each other. The number of thermocycles was therefore set at 20,000 (10) to evaluate the durability of the bond of the three RMGIs and SB combined with metal priming agents.

Thermocycling is applicable to the interface between an adhesive resin and an adherend with a different coefficient of thermal expansion. The greater the difference in the coefficient of thermal expansion of the two substrates, the reduction in bond strength of the interface will be higher (16). Therefore, it may be beneficial for researchers not to use metal-to-metal bonded specimens or ceramic-to-ceramic bonded specimens to accelerate reduction in bond strength at the adhesive interface (11,17).

The effect of metal priming agents on post-thermocycling bond strength to gold alloy was evaluated using 3 surface conditions and 4 luting agents. Two priming agents considerably increased the bond strength of the 4 luting agents, probably due to the fact that polymerizable monomers were incorporated into the 4 luting agents, and that the 2 priming agents contain different thione compounds, each of which is capable of bonding to copper and noble metals (9,18). The mechanism of bonding of Alloy Primer and Metaltite to noble metals is considered to be a chemical interaction between the thione monomer and noble metal elements (19,20). Further investigation is required to clarify the type of chemical interaction, if any, that forms between thione and noble metal elements. Overall, the results suggest that application of two metal priming agents with thione monomer combined with RMGIs considerably enhances the bond strength to gold alloy.

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