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# Evaluation of thiouracil-based adhesive systems for bonding cast silver-palladium-copper-gold alloy

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**Abstract:** This study aimed to evaluate the effect of adhesive systems based on a thiouracil monomer on bonding to silver-palladium-copper-gold (Ag-Pd-Cu-Au) alloy (Castwell M.C.12). Disk specimens were cast from the alloy and then air-abraded with alumina. The disks were bonded using six bonding systems selected from four primers and three luting materials. Shear bond strengths were determined both before and after thermocycling. Bond strength varied from 2.7 MPa to 32.0 MPa. Three systems based on a thiouracil monomer (MTU-6) showed durable bonding to the alloy, with post-thermocycling bond strengths of 22.4 MPa for the Metaltite (MTU-6) primer and Super-Bond, a tri-*n*-butylborane (TBB) initiated resin, 9.0 MPa for the Multi-Bond II resin, and 8.1 MPa for the Metaltite and Bistite II system. It can be concluded that a combination of thiouracil-based primer and TBB initiated resin is effective for bonding Ag-Pd-Cu-Au alloy. (J Oral Sci 52, 405-410, 2010)

**Keywords:** Ag-Pd-Cu-Au alloy; bonding; thiouracil; tri-*n*-butylborane.

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## Introduction

Application of adhesives for bonding restorations and

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fixed partial dentures (FPDs) has increased substantially. This trend is attributed to the development of adhesive functional monomers compatible with various substrates, as well as improvements in the properties of adhesive systems. Currently employed dental adhesives contain various functional monomers designed especially for intraoral application. These monomers can be categorized into acids (1), thiols or thiones (2-4), and others.

Silver-palladium-copper-gold (Ag-Pd-Cu-Au) alloys with 12 mass% gold are frequently used in Japan for cast restorations, FPDs, and the frameworks of removable dentures, because restorative and prosthodontic treatments employing Ag-Pd-Cu-Au alloys are covered by the Japanese health insurance system. A number of bonding systems for noble metal alloys, including Ag-Pd-Cu-Au alloy, have been developed. Representative systems applicable for bonding noble metal alloys consist of a single liquid primer containing a sulfur compound, and a resin-based luting or bonding material (5-7). Among the single liquid primers used for noble metals, the primer Metaltite (Tokuyama Dental Corp., Tokyo Japan), which contains a thiouracil compound (6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate, MTU-6), has been used effectively for bonding indirect composites to noble alloys (6,8), bonding denture base resin to metal frameworks (9), and cementing restorations and fixed partial dentures (7,10-13). In addition, a number of clinical reports have confirmed the durability of the bonding obtained with the Metaltite adhesive system for seating resin-bonded FPDs made of Ag-Pd-Cu-Au alloy (14-18).

One approach for excluding the primer is to incorporate a thiouracil compound (MTU-6) into the cement

composition. Amano et al. (19) reported the effectiveness of such a material (Ionotite F, Tokuyama Dental Corp.) for bonding Ag-Pd-Cu-Au alloy. Although an acrylic resin containing MTU-6 is currently on the market, no information is available about its characteristics of bonding to various substrates. The present study evaluated the bonding characteristics of thiouracil-based adhesive systems joined to cast Ag-Pd-Cu-Au alloy.

## Materials and Methods

### Materials

A Ag-Pd-Cu-Au alloy (Castwell M.C.12, GC Corp., Tokyo, Japan) designed for cast restorations and denture frameworks was selected as the substrate material. Four primers (Metalite, Multi-Bond II Primer, Tokuyama Dental Corp.; Super-Bond Liquid, V-Primer, Sun Medical Co., Ltd., Moriyama, Japan) were assessed as the bonding promoter. All primers were of the single-liquid type, and contained an adhesive functional monomer MTU-6, methacrylate-phosphate, 4-methacryloyloxyethyl trimellitate anhydride (4-META), and 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione (VTD), respectively (Table 1). Two methyl methacrylate (MMA)-based acrylic resins (Multi-Bond II, Tokuyama Dental Corp.; Super-Bond C&B Opaque, Sun-Medical Co., Ltd.) and a composite material (Bistite II, Tokuyama Dental Corp.) were used as luting materials. The Multi-Bond II resin has a powder-liquid composition, and the liquid contains a borate compound and MTU-6. The Super-Bond

resin comprises three components: a tri-*n*-butylborane (TBB)-based initiator, monomer liquid, and powder. The Bistite material consists of two pastes, in which a photo-initiator is included, and is therefore categorized as a dual-polymerized luting material. Information about the materials is summarized in Table 1.

### Shear bond test

The Ag-Pd-Cu-Au ingot was cast in a cristobalite investment material (Cristoquick 20, GC Corp.) using a high-frequency induction heating inhalation-pressure casting machine (Argoncaster-C, Shofu Inc., Kyoto, Japan). A total of 132 disk specimens (10 mm in diameter; 2.5 mm in thickness) were prepared from the alloy, and were divided into six sets of 22 disks (groups 1 through 6; Table 2). All the disks were ground with 800-grit silicon-carbide abrasive paper. The alloy surfaces were subsequently air-abraded with alumina (50-70  $\mu\text{m}$  grain size; Hi-Aluminas, Shofu Inc.) using an airborne-particle abrader (Jet Blast II, J. Morita Corp., Suita, Japan) for 10 s with an air pressure of 0.2 MPa. The distance of the orifice from the disk surface was 20 mm.

After air-abrasion, a piece of double-coated tape with a circular hole, 5 mm in diameter, was positioned on each disk specimen to define the bond area. The disks, except for group 6, were subsequently primed with one of the four materials shown in Table 2. A stainless steel ring (SUS 303; 6 mm inside diameter, 2 mm height, and 1 mm wall thickness) was placed so as to surround the opening area.

Table 1 Materials assessed

Material / Trade name	Manufacturer	Lot number	Composition (mass %)
Ag-Pd-Cu-Au casting alloy			
Castwell M.C.12	GC Corp.	602021, 707201	46 Ag, 20 Pd, 20 Cu, 12 Au
Primer			
Metalite	Tokuyama Dental Corp.	25	MTU-6, Ethanol
Multi-Bond II Primer	Tokuyama Dental Corp.	4	Phosphate monomer, UDMA, Acetone, Catalyst
Super-Bond Liquid	Sun Medical Co., Ltd.	RT3	MMA, 4-META
V-Primer	Sun Medical Co., Ltd.	RL1	VTD, Acetone
Luting material			
Multi-Bond II	Tokuyama Dental Corp.	2	Liquid: Borate catalyst, MMA, UDMA, HEMA, MTU-6
		301	Powder: PolyMMA, Catalyst
Super-Bond C&B Opaque	Sun Medical Co., Ltd.	RT3	Liquid: MMA, 4-META
		SF1	Powder: PolyMMA, Titanium oxide
		RT13	Initiator: TBB, TBB-O
Bistite II	Tokuyama Dental Corp.	10068	A: NPGDMA, Bis-MPEPP, Silica-zirconia B: Silica-zirconia, MAC-10, BPO, <i>dl</i> -camphorquinone

UDMA, Urethane dimethacrylate; VTD, 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione; MTU-6, 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate; MMA, Methyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; TBB, Tri-*n*-butylborane; TBB-O, Partially oxidized tri-*n*-butylborane; NPGDMA, Neopentyl glycol dimethacrylate; Bis-MPEPP, 2,2-bis[4(methacryloxyethoxy) phenyl] propane; MAC-10, 11-(methacryloyloxy) 1,1-undecane dicarboxylic acid; BPO, Benzoyl peroxide.

Table 2 Adhesive systems assessed

Group	Primer	Luting material
Group 1	Multi-Bond II Primer	Multi-Bond II
Group 2	Metaltite	Super-Bond C&B Opaque
Group 3	Super-Bond Liquid	Super-Bond C&B Opaque
Group 4	V-Primer	Super-Bond C&B Opaque
Group 5	Metaltite	Bistite II
Group 6	none	Bistite II

The 22 rings for group 1 were filled with the Multi-Bond II resin using the brush-dip technique. The 66 rings for groups 2, 3, and 4 were filled with the Super-Bond C&B Opaque resin using brush-dip technique. The 44 rings for groups 5 and 6 were filled with two layers of the Bistite II composite. Each layer was light-exposed with a handheld light-polymerization unit (Optilux 501, sdsKerr Corp., Orangé, CA, USA) for 20 s. The light source power density was confirmed to be 400 to 500 mW/cm<sup>2</sup> using a dental radiometer (Optilux Radiometer, sdsKerr Corp.). Thirty minutes after preparation, the specimens were stored in water at 37°C for 24 hours before being tested for bond strength. This state was defined as the 0 thermocycle. One-half of the specimens (11 specimens in six groups) were tested at the 0 thermocycle. The remaining half of the specimens (11 specimens in six groups) were subsequently thermocycled between cold water at 5°C and hot water at 55°C for 20,000 cycles with a 1-minute dwell time per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan).

Each specimen was placed in a steel mold and seated in a shear-testing jig. Shear bond strengths were then determined with a mechanical testing machine (Type 5567, Instron Corp., Canton, MA, USA) at a cross-head speed of 0.5 mm/minute. 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: A, adhesive failure at the alloy-luting material interface; C, cohesive failure within the luting material; and AC, combination of adhesive and cohesive failures. Typical specimens after surface preparation and debonding were observed with a scanning electron microscope (S-4300, Hitachi High-Technologies Corp., Tokyo, Japan) at an accelerating voltage of 15 kV.

### Statistical analysis

The average shear bond strength and standard deviation were calculated for each group. The results were primarily analyzed by Kolmogorov-Smirnov test for evaluation of the normal distribution (SPSS 15.0, SPSS Inc., Chicago,

IL, USA). Then the Levene test was also run on the results of shear bond testing to reveal any equality of variance (SPSS 15.0). When the results of the Kolmogorov-Smirnov test did not show a normal distribution in at least one category, and the Levene test did not show homoscedasticity, the results were analyzed by Kruskal-Wallis test (SPSS 15.0). Based on the results of the Kruskal-Wallis test, Steel-Dwass non-parametric multiple comparisons (Kyplot 4.0, KyensLab Inc., Tokyo, Japan) were applied to compare the difference among the six adhesive systems for each thermocycling condition. Differences between the pre- and post-thermocycling bond strengths were analyzed using the Mann-Whitney *U*-test. The level of statistical significance for all tests was set at  $P < 0.05$ .

### Results

The Kolmogorov-Smirnov test run on the bond strength results showed a normal distribution for all groups. The Levene test applied to the pre- and post-thermocycling groups, however, did not show equality of variance for both groups. The Kruskal-Wallis test showed that the  $\chi^2$  value was 57.88 for the pre-thermocycling group and 59.76 for the post-thermocycling group. The *P* value was <0.001 for both the pre- and post-thermocycling bond strengths. The pre- and post-thermocycling results were therefore analyzed separately using Steel-Dwass multiple comparisons. In addition, the difference between pre- and post-thermocycling bond strengths within an identical group was analyzed using the Mann-Whitney *U* test.

The results for shear bond strength are summarized in Table 3. Pre-thermocycling average bond strengths varied from a minimum of 5.2 MPa to a maximum of 32.0 MPa, and were categorized into five groups (categories a-e). Among the pre-thermocycling groups, group 1 (Multi-Bond II Primer and Multi-Bond II) and group 2 (Metaltite and Super-Bond C&B Opaque) showed the highest bond strength (category a), whereas group 6 (unprimed Bistite II) showed the lowest bond strength (category e). Post-thermocycling average bond strengths varied from 2.7 MPa to 22.4 MPa, and were again categorized into five groups (categories f-j). Among the post-thermocycling groups, group 2 (Metaltite and Super-Bond C&B Opaque) recorded the greatest bond strength (category f), followed by group 4 (V-Primer and Super-Bond C&B Opaque; category g), and then groups 1 (Multi-Bond II Primer and Multi-Bond II) and 5 (Metaltite and Bistite II) (category h). Comparison between the pre- and post-thermocycling results revealed that the bond strength in four groups (groups 1-4) was significantly reduced by application of thermocycling ( $P < 0.05$ ), whereas the bond strength in two groups (groups 5 and 6) was improved after



Table 3 Shear bond strength in MPa

Group	0 thermocycle				20,000 thermocycles				M-W	Post-TC / Pre-TC
	Median	IQR	Mean	SD	Median	IQR	Mean	SD		
Group 1	32.5 a	4.7	32.0	2.7	8.9 h	0.5	9.0	0.8	S	28.1%
Group 2	28.9 a,b	1.1	29.0	0.5	22.2 f	2.7	22.4	1.8	S	77.2%
Group 3	28.1 b	2.3	27.6	2.1	2.7 j	0.7	2.7	0.4	S	9.8%
Group 4	25.7 c	2.9	25.0	1.6	18.9 g	4.1	19.5	3.1	S	78.0%
Group 5	6.8 d	0.9	7.2	0.8	8.3 h,i	1.2	8.1	1.2	S	113.0%
Group 6	5.2 e	1.8	5.2	1.3	6.7 i	1.3	6.9	0.6	S	133.0%

$n = 11$ ; IQR, Interquartile range; SD, Standard deviation; Identical lower case letters indicate that the values are not significantly different. (Steel-Dwass comparison;  $P > 0.05$ ); M-W, Abbreviation "S" indicates that the difference between pre- and post-thermocycling bond strengths is significant. (Mann-Whitney  $U$  test;  $P < 0.05$ ); TC, Thermocycling.

Table 4 Failure modes after shear bond testing

Group	0 thermocycle			20,000 thermocycles		
	A	AC	C	A	AC	C
Group 1	0	11	0	0	11	0
Group 2	0	11	0	0	7	4
Group 3	1	10	0	8	3	0
Group 4	0	11	0	0	8	3
Group 5	4	7	0	0	9	2
Group 6	11	0	0	10	1	0

A, Adhesive failure at the alloy-luting material interface;  
C, Cohesive failure within the luting material;  
AC, Combination of adhesive and cohesive failures.

thermocycling ( $P < 0.05$ ).

The results of failure mode analysis are summarized in Table 4. None of the specimens showed cohesive failure before application of thermocycling. A combination of adhesive and cohesive failures was detected for all specimens in groups 1, 2, and 4, whereas adhesive failure was observed for all specimens in group 6. After the thermocycling, the number of specimens showing adhesive failure increased in group 3. However, specimens showing cohesive failure were observed in groups 2, 4 and 5 after application of thermocycling.

Figure 1 shows the alloy after the surface had been air-abraded with alumina. A complex relief pattern generated by the alumina particles can be seen. Figures 2-4 show the debonded surfaces of the alloy after it had been bonded with the three thiouracil-based adhesive systems. Although three specimens were subjected to thermocycling, remnants of the resin materials are evident on all specimens.

## Discussion

Comparison of bonding to Ag-Pd-Cu-Au alloy between Multi-Bond prototype and Super-Bond C&B Opaque has been evaluated using the materials Metaltite and V-Primer (12). The Multi-Bond prototype consists of a MMA-

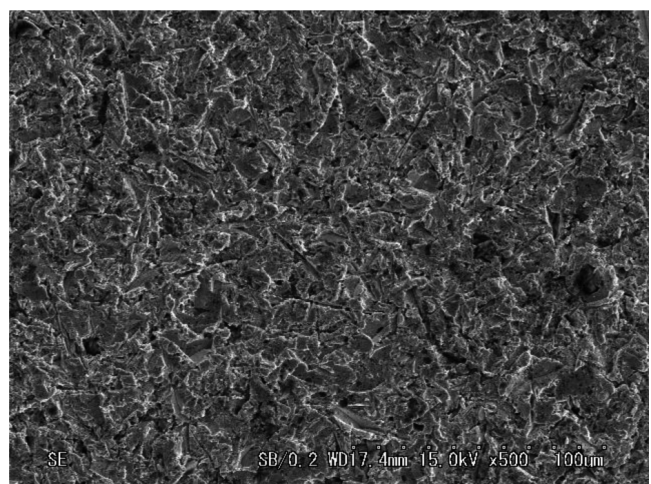


Fig. 1 Scanning electron micrograph of Ag-Pd-Cu-Au alloy air-abraded with alumina (original magnification,  $\times 500$ ).

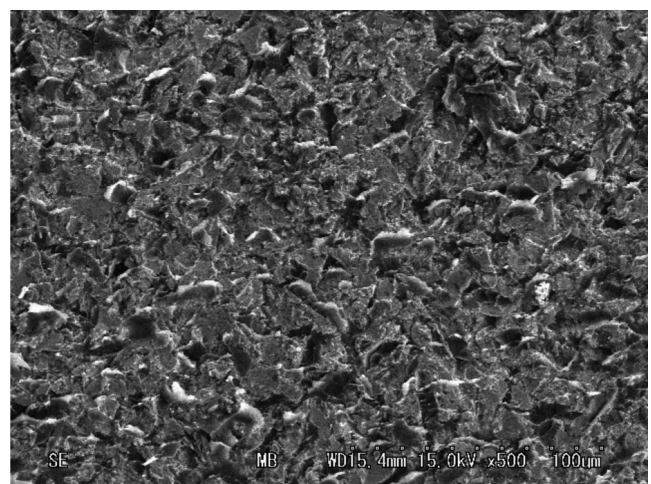


Fig. 2 The debonded surface of the thermocycled group 1 specimen (Multi-Bond II Primer and Multi-Bond II; original magnification,  $\times 500$ ).

polyMMA system with a carboxylic monomer in the liquid

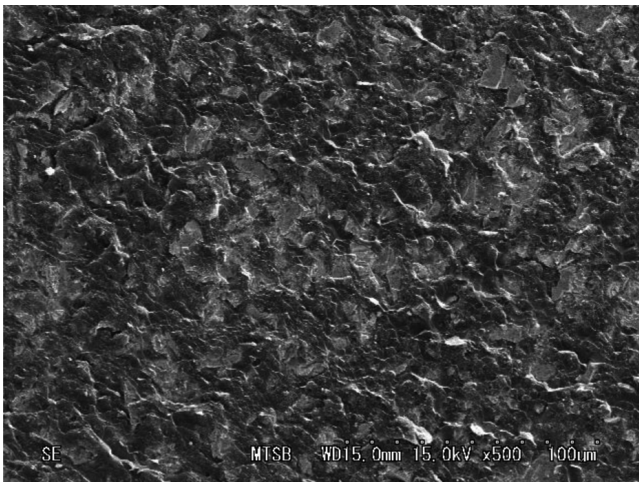


Fig. 3 The debonded surface of the thermocycled group 2 specimen (Metaltite and Super-Bond C&B Opaque; original magnification,  $\times 500$ ).

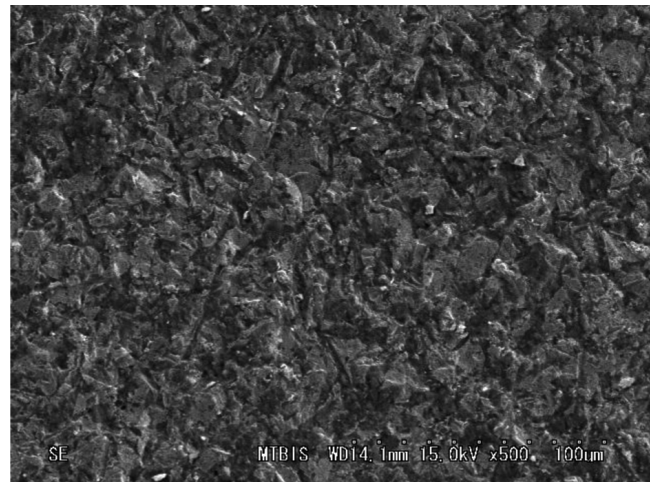


Fig. 4 The debonded surface of the thermocycled group 5 specimen (Metaltite and Bistite II; original magnification,  $\times 500$ ).

component, and a benzoyl peroxide-tertiary amine redox initiator. Clinicians are therefore required to apply the Metaltite or V-Primer material before bonding Ag-Pd-Cu-Au alloy with the Multi-Bond prototype. The difference between the Multi-Bond prototype and Super-Bond C&B Opaque can be evaluated simply, because the unprimed and primed conditions are identical for both resins. The results (12) have shown that the reduction in bond strength after thermocycling is comparable for both resins when the alloy is unprimed. However, the reduction in bond strength is more remarkable for the Multi-Bond prototype when the alloy is primed with either the Metaltite or the V-Primer material.

In the new formulation of Multi-Bond II, 1) MTU-6 is added to the monomer liquid component, 2) a methacrylate-phosphate is added to the primer, and 3) the carboxylic monomer has been eliminated from the bonding system. Based on this information, the current project evaluated the bonding performances of six bonding systems. As shown in Table 3, the pre-thermocycling bond strength of the Multi-Bond II resin was equivalent to, or higher than that of the Super-Bond C&B resin, and the majority of specimens in groups 1 through 4 showed AC-type failure at the 0 thermocycle. This was probably due to the difference in 24-hour mechanical strength of the two resins. Specifically, the Super-Bond resin contains a greater amount of initiator in the liquid component (approximately 8%) (20), than the Multi-Bond resin (less than 5%). If excessive TBB derivative were to act as a plasticizer during and after the setting reaction, the strength of the polymerized Super-Bond resin would be reduced.

On the other hand, TBB-initiated resin tends to exhibit

more durable bonding than other acrylic resins, when the substrate is properly primed. One of the reasons may be gradual improvement of monomer conversion and reduction of contraction stress at the substrate-resin interface. The difference in bond strength between the Multi-Bond prototype and Super-Bond Materials joined to Ag-Pd-Cu-Au alloy (12) or titanium alloy (21), as well as the results of the present study for post-thermocycling groups 1 and 2, supports this speculation.

The Bistite II composite showed a considerably lower pre-thermocycling bond strength than the two acrylic resins. Unlike the two acrylic resins, however, the bond strength of the Bistite composite increased after application of thermocycling. These results suggest that it is difficult to evaluate the proper bonding and setting behavior of the material because the bond strength is increasing somewhat after application of thermocycling. Also, the standard deviation was greater for the Bistite material than for the acrylic resins. Due to the high viscosity of paste-type composite material, it may be more difficult for the Bistite II material to penetrate into an alumina-abraded alloy surface than for the acrylic resins.

It is evident that, for the Super-Bond C&B resin, two thione-based primers are effective for bonding the alloy, and Metaltite exhibited a better bonding performance than V-Primer (categories f and g). Within the limitations of the present experiment, it can be concluded that the thiouracil monomer MTU-6 is effective for bonding to Ag-Pd-Au-Cu alloy, and that application of the Metaltite primer in combination with the Super-Bond C&B resin generates the most durable bonding to the alloy.

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