

Effect of three metal priming agents on the bond strength of adhesive resin cement to Ag-Zn-Sn-In alloy and component metals

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The aim of this study was to investigate the effect of three metal priming agents on the bond strength of adhesive resin cement to Silver-Zinc-Tin-Indium (Ag-Zn-Sn-In) alloy and pure Ag, Zn, Sn, and In. The specimens were air-abraded with alumina and then primed with one of three metal priming agents: V-Primer, Estenia Opaque Primer, or Alloy Primer. The metal disks were bonded with adhesive resin cement (Super-Bond Bulk-mix technique). Shear bond strengths ($n=10$ /group) were determined before and after 50,000 thermocycles for Ag-Zn-Sn-In alloy as well as after 5,000 thermocycles for pure Ag, Zn, Sn and In. For Ag-Zn-Sn-In alloy, the post-thermocycling bond strength of the Alloy Primer group was significantly higher than that of the other primers. It can be concluded that Alloy Primer containing both the vinyl-thione monomer (VBATDT) and hydrophobic phosphate monomer (MDP) is effective for bonding Ag-Zn-Sn-In alloy and pure Ag, Zn and Sn.

Keywords: Bond strength, Adhesive resin cement, Silver-Zinc-Tin-Indium alloy, Metal priming agent

INTRODUCTION

The use of a cast post and core has been a widely used technique in dentistry to restore extensively damaged teeth. Cast post and cores have an acceptable long-term survival time of more than 10 years¹. Dental practitioners are still favorably using cast posts and cores²⁻⁴. Silver-based casting alloys without gold, such as silver-tin-zinc (Ag-Sn-Zn), silver-indium (Ag-In) or silver-zinc-tin-indium (Ag-Zn-Sn-In) alloys, are frequently used for cast post and cores as an alternative to gold alloys because of their lower cost compared with gold alloys⁴⁻⁶. It was shown that the mold filling capability of Ag-Zn-Sn-In alloy is superior to that of silver-palladium-copper-gold alloy (Ag-Pd-Cu-Au alloy) with a conventional centrifugal casting machine⁷. However, there have been clinical failures; the most common cause of failure has been loss of retention of the post and core from the root due to deficiencies in bonding at the metal-cement interface^{8,9}. Posts and cores fabricated from semi-precious alloy had a higher risk of failure of posts and cores than high-gold-content alloys⁹. Therefore, the bonding between alloys and luting cement plays an important role in the longevity of the prosthesis¹⁰⁻¹². Deficiencies in bonding at the metal-resin interface can become a significant clinical problem from not only loss of retention but also from microleakage, secondary caries and tooth fracture, which could ultimately lead to periapical pathosis^{6,8-11,13-15}. Increasing microleakage from debonding causes bacterial contamination through the tooth-crown interface after restoring the tooth.

Numerous investigations have been performed to observe the bond strength of luting agents to the cast post and cores¹⁶⁻¹⁹. The adhesive resin cement appeared

to have a significant strengthening effect on the retention of cast post and cores¹⁷⁻¹⁹. Regarding bonding for dental metal alloys, the combined use of the resin cement and metal priming agent is generally effective²⁰⁻²⁵. The currently available metal priming agents can be categorized into three types, those for (1) precious metal alloys; (2) base metal alloys; and (3) both precious and base metal alloys. Matsumura *et al.*^{20,21} reported that the combined use of thiol-based metal priming agents for precious metal alloy and adhesive resin cement effectively bonded to precious metal alloys, except the Ag-In-Zn alloy. Shimizu *et al.*²² concluded that airborne particle abrasion with alumina was effective for bonding to Ag-Zn-Sn-In alloy, but the effects on the bond durability of the metal priming agents was not clear.

Little information is available about the relationship between the use of metal priming agents and the resultant bond strength with Ag-Zn-Sn-In alloys. In particular, comparison of functional monomers contained in the metal priming agents has not been performed for bonding Ag-Zn-Sn-In alloy and its component metals. The purpose of the present study was to investigate the effect of three metal priming agents on the bond strengths of adhesive resin cement to Ag-Zn-Sn-In alloy and its component metals. The null hypothesis was that there would be no difference between the three metal priming agents in terms of the bond strength of adhesive resin cement to Ag-Zn-Sn-In alloy and its component metals.

MATERIALS AND METHODS

Preparation of the specimens for Ag-Zn-Sn-In alloy

Table 1 lists the materials used in this study. Figure 1

Table 1 Materials used for this study

Material	Manufacturer	Lot number	Composition
Alloy			
Ag-Zn-Sn-In alloy (Miro Bright)	GC, Tokyo, Japan	—	Ag 72%, Zn 13%, Sn 9%, In 6%
Pure metal			
Pure Silver (pure Ag)	The Nilaco, Tokyo, Japan	AG-403558	Ag 99.98, mass%
Pure Zinc (pure Zn)	The Nilaco	ZN-483551	Zn 99.5, mass%
Pure Tin (pure Sn)	The Nilaco	SN-443551	Sn 99.9, mass%
Pure Indium (pure In)	The Nilaco	IN-203558	In 99.99, mass%
Metal priming agent			
V-Primer	Sun Medical, Moriyama, Japan	GV1	VBATDT, acetone
Estenia Opaque Primer	Kuraray Noritake Dental, Osaka, Japan	CG0003	MDP, monomer solvent
Alloy Primer	Kuraray Noritake Dental	C40023	MDP, VBATDT, acetone
Adhesive resin cement			
Super-Bond Catalyst	Sun Medical	FW23F	Tri- <i>n</i> -butylborane (TBB)
Super-Bond clear powder	Sun Medical	GV5	Polymethyl methacrylate (PMMA)
Super-Bond Liquid	Sun Medical	FX2	4-META in methyl methacrylate

VBATDT: 6-(4-vinylbenzyl-*n*-propyl)amino-1,3,5-triazine-2,4-dithione

MDP: 10-methacryloyloxydecyl dihydrogen phosphate

4-META: 4-methacryloyloxyethyl trimellitate anhydride

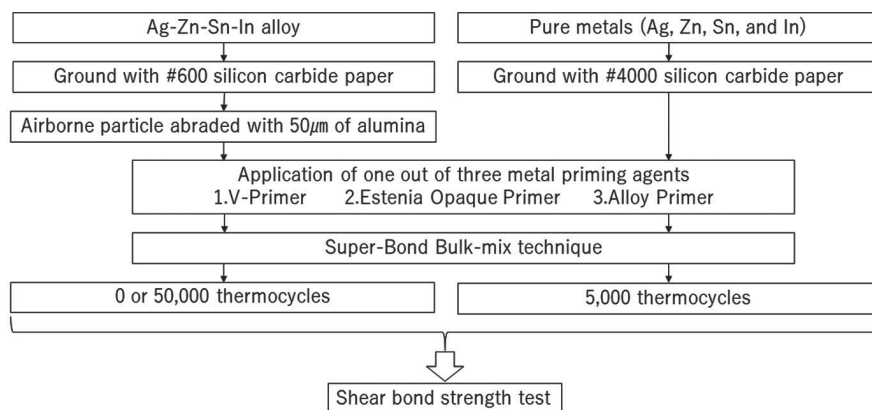


Fig. 1 Flow diagram of preparing bonded specimens for shear bond strength test.

shows a flow diagram of preparing bonded specimens for shear bond strength test. Disk-shaped wax patterns (10.0 mm in a diameter and 2.5 mm thick) were cast with a Ag-Zn-Sn-In alloy (Miro Bright, GC, Tokyo, Japan) in a cristobalite investment material (Cristobalite Micro II, GC) using a conventional spring-driven centrifugal casting machine (Kerr Centrifico casting machine, Kerr

Manufacturing, Romulus, MI, USA). Specimens were embedded in an autopolymerizing resin (Palapress vario, Heraeus Kulzer, Hanau, Germany) with acrylic rings. The disk surfaces were ground with up to 600-grit silicon carbide paper under running water. The polished surfaces were then airborne particle abraded with 50 µm of alumina (Aluminous Powder WA 360, Pana

Heraeus Dental, Osaka, Japan) for 15 s using a grit blaster (Duostar Z2, Bego, Bremen, Germany). The air pressure was 0.5 MPa with the nozzle positioned at a 90° angle approximately 5 mm from the specimen surface. After airborne particle abrasion, the metal surface were cleaned with a non-oil compressed air system (ACP-50, RYOBI LIMITED, Nagoya, Japan) to remove the excess alumina powder particles.

A Teflon ring (5.0 mm inner diameter) and masking tape with a circular hole were placed to define the bonding area. Three metal priming agents (V-Primer, Estenia Opaque Primer, and Alloy Primer) were separately applied to the metal surfaces using a sponge pellet according to the manufacturers' instructions. Each metal priming agent contained at least one functional monomer in the solvent. The adhesive luting cement (Super-Bond Bulk-mix technique, Sun Medical, Moriyama, Japan) was mixed according to the manufacturer's instructions and applied inside the Teflon ring on each specimen using a micro syringe (Super-Bond micro syringe, Sun Medical). After the adhesive luting cement was set, the masking tape and Teflon ring were removed as gently as possible, and the specimen was completed. All specimens were immersed in 37°C water for 24 h. Afterwards, half of the specimens were placed in a thermocycling apparatus (Thermal Shock Tester TTS 1, Thomas Kagaku, Tokyo, Japan) and thermocycled for 50,000 cycles in water between 5 and 55°C with a dwell time of 1 min at each temperature. The remaining half of the specimens were not thermocycled.

Each specimen was positioned in a shear bond testing jig. The shear bond strengths were determined using a universal testing machine (Autograph AGS-J, Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min.

Preparation of the specimens for pure metals (Ag, Zn, Sn, and In)

In addition, pure metal disks (Ag, Zn, Sn, and In) were cut from plates using a cutting saw. To measure the bond strength of pure metals (Ag, Zn, Sn, and In), the specimens were also fabricated as described in the above section. The disk surfaces were ground with up to 4000-grit silicon carbide paper under running water. Airborne-particle abrasion with alumina was not performed on all pure metal specimens because our aim was to evaluate the functional monomers in the priming agents for pure metals. The shear bond strengths were determined after thermocycling for 5,000 cycles. The bond strength of specimens that underwent spontaneous debonding was defined as 0 MPa.

Analysis of the fracture surface

After shear bond testing, the type of fracture surface was observed using an optical microscope at 30× magnification. Failure modes were categorized into the following three categories: A (adhesive failure at the metal-resin interface), C (cohesive failure at the inside of resin), or M (a mixture of adhesive and cohesive failure).

Statistical analysis

The means and standard deviations (S.D.) for the shear bond strengths ($n=10/\text{group}$) were calculated using a computer program (IBM SPSS Statistics version 22, IBM, Armonk, NY, USA). All data were analyzed with the Levene test to evaluate the homoscedasticity, and with analysis of variance (ANOVA) and the Newman-Keuls post hoc test at a significance level of $\alpha=0.05$ with computer software.

RESULTS

For Ag-Zn-Sn-In alloy, the result of the shear bond test before and after thermocycling are summarized in Fig. 2. The data was analyzed as having homoscedasticity. Two-way ANOVA showed that there were significant differences between the metal priming agents ($p<0.0001$) and thermocycling conditions ($p<0.0001$). There was also a significant interaction between the metal priming agents and thermocycling ($p<0.0001$) for shear bond strengths. Therefore, the bond strengths were analyzed by one-way ANOVA and the Newman-Keuls *post hoc* test.

Before thermocycling, application of V-Primer yielded significantly higher shear bond strengths than Estenia Opaque Primer ($p<0.05$), and there was no significant difference between V-Primer and Alloy Primer. After thermocycling, the shear bond strengths of all groups were significantly reduced with 50,000 thermocycling ($p<0.05$). The mean shear bond strength of Alloy Primer was the greatest (13.1 MPa), which was followed by Estenia Opaque Primer (5.6 MPa) and V-Primer (1.6 MPa). The bond strengths of Alloy Primer were significantly higher than those of V-Primer and Estenia Opaque Primer.

For pure metals (Ag, Zn, Sn, and In), the results of the shear bond test after thermocycling are summarized in Table 2. All the specimens of the pure In groups

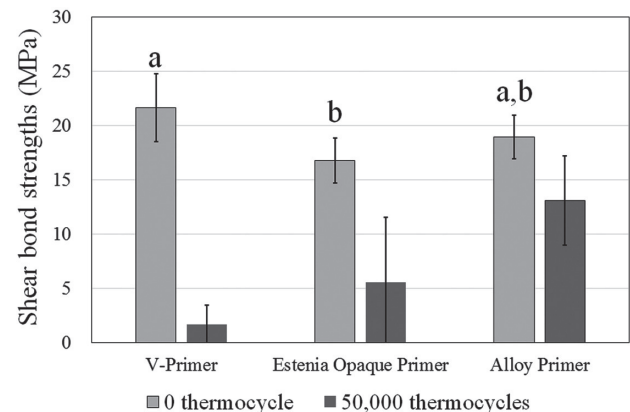


Fig. 2 Shear bond strengths (MPa) of Ag-Zn-Sn-In alloy before and after 50,000 thermocycles. Categories with the same letter were not significantly different ($p>0.05$).

Table 2 Statistical analysis of shear bond strengths (MPa) of pure Ag, Zn, Sn, and In after 5,000 thermocycles

Metal	Primer	Bond strength MPa (SD)	Grouping
Ag	Alloy Primer	22.4 (3.1)	a
Ag	V-Primer	19.0 (4.7)	b
Zn	Alloy Primer	17.0 (6.1)	b, c
Zn	Estenia Opaque Primer	15.5 (2.9)	c
Sn	Alloy Primer	7.0 (5.2)	d
Sn	Estenia Opaque Primer	6.8 (2.5)	d
Sn	V-Primer	1.1 (1.7)	e
Zn	V-Primer	0.3 (0.4)	e
Ag	Estenia Opaque Primer	—	—
In	V-Primer	—	—
In	Estenia Opaque Primer	—	—
In	Alloy Primer	—	—

SD: standard deviation.

Identical letters indicate that the values are not significantly different ($p>0.05$).

Table 3 Failure modes of Ag-Zn-Sn-In alloy before and after 50,000 thermocycles

Thermocycles	0			50,000		
	A	M	C	A	M	C
V-Primer	9	1	0	10	0	0
Estenia Opaque Primer	10	0	0	10	0	0
Alloy Primer	10	0	0	10	0	0

A: Adhesive failure at the metal-resin interface;

C: Cohesive failure at the inside resin;

M: Mixture of adhesive and cohesive failures.

Table 4 Failure modes of pure Ag, Zn, Sn, and In after 5,000 thermocycles

Metal	Primer	Adhesive	Mixture	Cohesive
Ag	Alloy Primer	10	0	0
	V-Primer	9	1	0
	Estenia Opaque Primer	10	0	0
Zn	Alloy Primer	10	0	0
	V-Primer	10	0	0
	Estenia Opaque Primer	10	0	0
Sn	Alloy Primer	10	0	0
	V-Primer	10	0	0
	Estenia Opaque Primer	10	0	0
In	Alloy Primer	10	0	0
	V-Primer	10	0	0
	Estenia Opaque Primer	10	0	0

A: Adhesive failure at the metal-resin interface;

C: Cohesive failure at the inside resin;

M: Mixture of adhesive and cohesive failures.

and pure Ag group with Estenia Opaque Primer were spontaneously debonded prior to 5,000 cycles. Therefore, the data without pure In groups and pure Ag group with Estenia Opaque Primer were analyzed by one-way ANOVA and the Newman-Keuls *post hoc* test. The Newman-Keuls *post hoc* test divided the results into 5 groupings: (a)–(e). Pure Ag conditioned with Alloy primer exhibited the greatest bond strength (22.4 MPa). For pure Zn and pure Sn, there were no significant differences in the shear bond strength between Alloy primer and Estenia Opaque Primer.

The modes of failure of all specimens are presented in Tables 3 and 4. Microscopic observation of debonded surfaces revealed that most specimens had adhesive failure. Some of Ag-Zn-Sn-In alloy specimens after thermocycling showed brownish discoloration at the adhesive-failed metal surface (Alloy Primer; 2 out of 10, Estenia Opaque Primer; 7 out of 10, and V-Primer; 10 out of 10). The pure metals did not show discoloration at the debonded specimen surface.

DISCUSSION

This study evaluated the adhesive characteristics of three single-liquid metal priming agents in terms of the bonding durability between Ag-Zn-Sn-In alloy and adhesive resin cement. The thermocycling test is one of the most important accelerated aging methods to evaluate the bonding durability of metal-bonded resin interface²⁶. According to Shimizu *et al.*²², the effects on the metal priming agents for precious metal alloys were not clear for the bond durability after 20,000 thermocycles of adhesive resin cement to Ag-Zn-Sn-In alloy. Therefore, three metal priming agents were evaluated to improve the bonding durability of adhesive resin cement to Ag-Zn-Sn-In alloy before and after 50,000 thermocycles. Additionally, pure metals (Ag, Zn, Sn, and In) were used to evaluate of the effects of each metal priming agent to the metallic elements constituting Ag-Zn-Sn-In alloy. This alloy chemically contains Ag 72 mass%, Zn 13 mass%, Sn 9 mass%, and In 6 mass% according to the manufacturer's product information. The surfaces of pure metals were finished into flat surfaces with 4000-grit silicon carbide papers to exclude as much as possible the influence of mechanical retention and to focus only on the efficacy of each adhesion functional monomer. As a result of this study, there were difference among the three metal priming agents in terms of the bond strength of adhesive resin cement to Ag-Zn-Sn-In alloy and its component metals, and the null hypothesis was rejected.

Three metal priming agents applied in this study contain two kinds of the functional monomer. The 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer of Estenia Opaque Primer and Alloy Primer is classified as a phosphate ester monomer, and the 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione (VBATDT) of V-Primer and Alloy Primer is classified as a triazine dithione derivative monomer. It is suggested that the improvement of the bond strength

to base metal alloys with MDP is based on chemical bonding with the metal oxide formed on these alloys^{20,26}. For the bonding mechanism of VBATDT to precious metals, it is considered to be a chemical interaction between sulfur and precious metal elements²⁷. As shown in Table 2, conditioning with V-Primer or Alloy Primer considerably improved the bond strength to pure Ag in this study. Suzuki *et al.*²⁷ reported that the Raman and infrared reflection absorption spectroscopic evaluations indicated that the VBATDT is primarily chemisorbed onto the Au, Ag and Cu surfaces. It is speculated that the sulfur atom of VBATDT adsorbed the Ag surface after the application of metal priming agents. The shear bonding test with pure Ag showed that the effect of Alloy primer was superior to that of V-primer, although both primers contain the VBATDT monomer. This result agreed with a previous investigation that the combined use of VBATDT and MDP yielded statistically superior bond strengths to pure Ag compared with VBATDT alone²⁷. According to Suzuki *et al.*²⁸, the roles of MDP in the metal primer are presumably to form a monolayer with appreciable durability and to promote polymerization with resin monomers at the interface between the metal and resin. It is also possible that the differences between the two primers in terms of the VBATDT monomer concentration affect the bonding of adhesive resin cement to pure Ag. Alloy Primer and Estenia Opaque Primer effectively bonded to pure Zn and Sn. It is well known that these two metals (Zn and Sn) easily form oxides; therefore, these oxides could effectively work to improve the bond strength of resin cement to these metals. It is speculated that the MDP monomer adsorbed the metal oxide after the application of metal priming agents^{20,26}. All specimens in the pure In group debonded prior to 5,000 cycles. According to Goto *et al.*²⁹, the small addition of In exhibited a remarkable increase in the bond strength of resin cement to Ag-Pd-Au-Cu alloy. However, the current study clearly revealed that both VBATDT and MDP monomers were ineffective for bonding pure In.

Thermocycling was used as an accelerated aging test to evaluate of the bonding durability of each bonding system^{20,21}. As shown in Fig. 2, thermocycling considerably decreased the bond strengths to Ag-Zn-Sn-In alloy. The results indicate that the thermocycling aging test is essential for evaluating the bond durability of adhesive resin cement to Ag-Zn-Sn-In alloy^{22,30}. The post-thermocycling shear bonding test showed that Estenia Opaque Primer was superior to V-Primer. According to the manufacturer's safety data sheet information, Estenia Opaque Primer contains benzoyl peroxide. There is a possibility that the addition of benzoyl peroxide as accelerator of resin polymerization could affect the curing process and improve the bond durability. Matsumura *et al.*^{20,21} reported that the bond durability of Ag-In-Zn alloy was scarce after thermocycling and they speculated that the reason was that the corrosion resistance of Ag-In-Zn alloy was insufficient at the adhesive interface. It is known that the dental silver based alloys are susceptibility to

corrosion and tarnish in the oral environment³¹). Silver-based casting alloys such as Ag-Zn-Sn-In alloy contain a small amount of indium to improve their tarnish resistance of this alloy³²). In the present study, visual inspection of Ag-Zn-Sn-In alloy revealed that some alloy specimens exhibited surface brownish discoloration of the debonded metal surface after thermocycling. The lower the bond strength after thermocycling was, the larger the number of surface brownish discoloration was. This suggests that poor bonding of Ag-Zn-Sn-In alloy can be explained by the lack of corrosion resistance of the alloy at the interface²¹).

As shown in Fig. 2, the post-thermocycling shear bonding test showed that Alloy Primer was superior to V-Primer and Estenia Opaque Primer even though all metal priming agents contain the functional monomer. These results indicated that the bonding behavior of Ag-Zn-Sn-In alloy may differ from that of the other precious metal alloys^{20,21}). Generally, VBATDT monomer is likely the most effective metal priming agent against precious metal alloy²⁴). However, the combined use of VBATDT and MDP effectively enhanced the bond durability to Ag-Zn-Sn-In alloy with adhesive resin cement, which could be why there was a synergistic effect of both VBATDT for Ag and MDP for Sn and Zn. Namely, this result could be caused by a twofold reason: the effect of appropriate concentration of VBATDT on bonding and the positive effect of MDP on polymerization when both functional monomers coexisted^{27,28}). Further investigation is necessary to adequately explain this phenomenon and confirm the validity of these conclusions. In addition, longitudinal clinical investigations will be required to determine the clinical performance.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

1. The VBATDT monomer in Alloy Primer and V-Primer was effective for bonding pure Ag. The MDP monomer in Alloy Primer and Estenia Opaque Primer considerably improved the bond strength to pure Zn and Sn, while neither the VBATDT monomer nor the MDP monomer was effective with pure In.
2. The combined treatment of VBATDT and MDP with Alloy Primer effectively enhanced the bond durability to Ag-Zn-Sn-In alloy with adhesive resin cement.

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