

레진 시멘트와 코발트 크롬 합금의 미세인장결합강도에 다양한 프라이머들이 미치는 영향

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Effects of primers on the microtensile bond strength of resin cements to cobalt-chromium alloy

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Purpose: The aim of this study is to evaluate the effects of various primers on the microtensile bond strength (μ TBS) of resin cements to cobalt-chromium (Co-Cr) dental casting alloy. **Materials and methods:** Four adhesive primers (Universal primer, Metal primer II, Alloy primer, and Metal/Zirconia primer) and two resin cements (Panavia F2.0, G-CEM LinkAce) were tested. One hundred fifty Co-Cr beams were prepared from Co-Cr ingots via casting (6 mm length \times 1 mm width \times 1 mm thick). The metal beams were randomly divided into ten groups according to the adhesive primers and resin cements used; the no-primer groups served as the control ($n = 15$). After sandblasting with aluminum oxide (125 μ m grain), the metal and resin cements were bonded together using a silicone mold. Prior to testing, all metal-resin beams were examined under stereomicroscope, and subjected to the μ TBS test. The mean value of each group was analyzed via one-way ANOVA with Tukey's test as post hoc ($\alpha = .05$) using SPSS software. **Results:** The mean μ TBS of all groups was ranged from 20 to 28 MPa. There is no statistically significant difference between groups ($P > .05$). Mixed failure, which is the combination of adhesive and cohesive failures, is the most prevalent failure mode in both the Panavia F2.0 and G-Cem LinkAce groups. **Conclusion:** The μ TBS of all tested groups are relatively high; however, the primers used in this study result in no favorable effect in the μ TBS of Panavia F2.0 and G-Cem LinkAce resin cement to Co-Cr alloy. (*J Korean Acad Prosthodont* 2019;57:95-101)

Keywords: Bond strength; Metal alloy; Resin cement; Primer

Introduction

Resin luting cement was developed for the cementation of esthetic materials such as all-ceramic and indirect-composite restorations. In comparison with conventional luting cements, resin luting cements have improved the cementation of base metal alloys due to their lower solubility, better wear resistance, and marginal closure,^{1,2} and it is widely used in dentistry.³ The use of self-adhesive resin cement, which combines the advantages of conventional and adhesive luting

agents, is increasing since the self-adhesive resin cement is more user-friendly and less technique-sensitive than conventional resin cements.⁴

However, because of the low chemical affinity of resin cement to metal alloys, surface treatments are recommended to achieve a more durable bonding.⁵⁻⁷ A variety of surface treatments have been studied in an effort to improve bond strength, including mechanical and chemical bonding as well as combinations of both. The application of several functional monomers is considered as one of the most ef-

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fective chemical treatments to enhance the physicochemical bonding of resin cements to metal alloy.^{8,9}

There are various adhesive primers currently used in dentistry, and each primer contains functional monomers such as 4-methacryloyloxyethyl trimellitate anhydride (4-META), 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate (MTU-6), and methacryloyloxyalkyl thiophosphate derivatives (MEPS), that increase the retention of resin to a metal surface. However, determining the most effective functional monomer for bonding and the effects of primer application on bond strength remain in debate.^{5-7,10,11}

Although macrotests such as tensile and shear bond tests are commonly used in studies to analyze metal-resin bond strength, these studies also have their limitations.¹² To overcome some limitations of macrotests, Sano *et al.*¹³ used microtensile bond tests, which are considered more appropriate for the evaluation of bond strength since they allow a more uniform distribution of stress, reduce cohesive failure, and provide a more realistic measurement of bond strength at the adhesive interface. Cobalt-Chromium (Co-Cr) metal alloy specimens are difficult to fabricate for microtensile bond tests, and as a result are not widely reported in the literature at this time.

The purpose of this study is to evaluate the effects of various primers on the microtensile bond strength of resin cements to a Co-Cr dental casting alloy.

Materials and methods

In this study, we used Co-Cr alloy, four adhesive primers and two resin cements. Specific information concerning the materials utilized in this study is presented in Table 1. One hundred fifty Co-Cr metal beams (6 mm long, 1 mm wide, and 1 mm thick) were casted. After trimming and polishing the beams, the bonding surfaces were sand-blasted with aluminum oxide (125 μ m grain) for 5 seconds at 80-psi pressure. The distance from the nozzle to the metal surface was 1.5 cm. The metal beams were cleaned using an ultrasonic cleaner for 1 minute, and were then randomly divided into ten groups according to primer and resin cement; the no primer (NP) group served as the control (n = 15, Table 2). A silicone mold with rectangular cavities (12 mm long, 1 mm wide, and 1 mm thick) was used to fabricate the metal-resin beams (Fig. 1) and the resin cement was applied to the rest part of the mold. All materials were applied and handled according to the manufacturer's instructions. After light-curing, all speci-

Table 1. Materials used in this study (information provided by manufacturers)

Materials	Components	Lot no.	Manufacturer
Remanium 2001	Co Cr Mo W 63 23 7 4.3 (%)	247	Dentaurum GmbH & Co. KG, Ispringen, Germany
PANAVIA F 2.0	Paste A: BPEDMA, MDP, DMA Paste B: Al-Ba-B-Si glass/ silica containing composite	00035B	Kuraray Co., Tokyo, Japan
G-CEM LinkAce	UDMA; phosphoric acid ester monomer; 4-META; water; dimethacrylates; silica powder	0611091	GC Dental Industrial Co., Tokyo, Japan
Universal primer	MTU-6	004/005	Tokuyama Dental Co., Tokyo, Japan
Metal primer II	1% MEPS, 99% methyl methacrylate	1307022	GC Dental Industrial Co., Tokyo, Japan
Alloy primer	MDP, VBATDT, 98.5% acetone	00445B	Kuraray Co., Kurashiki, Japan
Metal/Zirconia primer	Phosphonic acid methacrylate monomer	R60214	Ivoclar Vivadent AG, Schaan/Liechtenstein

BPEDMA = bisphenol-A-polyethoxy dimethacrylate; DMA = dimethacrylate; UDMA = urethane dimethacrylate; MTU-6 = 6-methacryloyloxyhexyl 2-thiouracil-5-carboxylate; MEPS = methacryloyloxyalkyl thiophosphate derivatives; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; VBATDT = 6-4-vinylbenzyl-n-propyl amino-1,3,5-triazine-2,4-dithione

Table 2. Number of specimens per group

Groups	Panavia	G-cem
No primer (NP)	N = 15	N = 15
Universal primer (UP)	N = 15	N = 15
Metal Primer II (MP)	N = 15	N = 15
Alloy Primer (AP)	N = 15	N = 15
Metal Zirconia Primer (MZP)	N = 15	N = 15

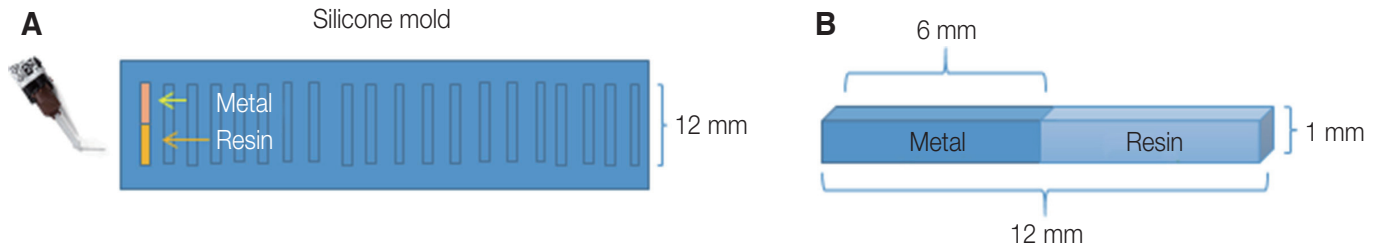


Fig. 1. Fabrication of specimen. (A) A silicone mold, (B) The metal-resin specimen.

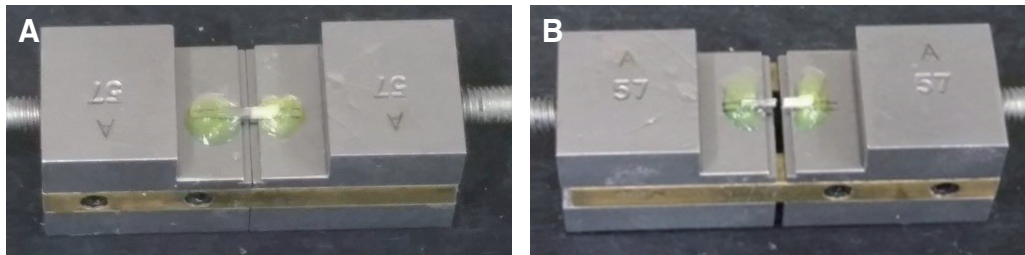


Fig. 2. (A) Metal-resin beams were individually attached via an active gripping method to the flat grips steel fixture of the microtensile tester using LI-BOND PEN adhesive. (B) The metal-resin specimens were loaded under tension (crosshead speed: 1.0 mm/min).

mens were allowed to completely set for 24 hours; the metal-resin beams were then stored in distilled water at 37°C for 24 hours before conducting the microtensile bond test.

Prior to testing, all metal-resin beams were studied under a stereomicroscope (Damisystem, TaeShin BioScience, Namyangju, Korea) at $\times 30$ magnification for flaws, bubbles, or excess resin on the specimen's bonding interface; specimens with defects were excluded. An active gripping method was used by attaching each specimen to the flat grip steel fixture of the microtensile tester (Micro Tensile Tester, BISCO, Schaumburg, IL, USA) using a light-cured adhesive (LI-BOND PEN, DFS Diamon, Riedenburg, Germany). The metal-resin beams were loaded under tension at a cross-head speed of 1.0 mm/min using the microtensile tester machine (Fig. 2). Bond strength values were calculated using the formula, $\sigma = L / A$, where 'L' is the load at failure (N) and 'A' is the adhesive area (mm^2). The mode of failure of each specimen was determined using a stereomicroscope.

The mean of each group was analyzed via one-way ANOVA with microtensile bond strength (μTBS) as the dependent variable, primer treatment as the independent factor, and Tukey's test as post hoc ($\alpha = .05$) using SPSS software (SPSS ver. 22.0, IBM, Chicago, IL, USA).

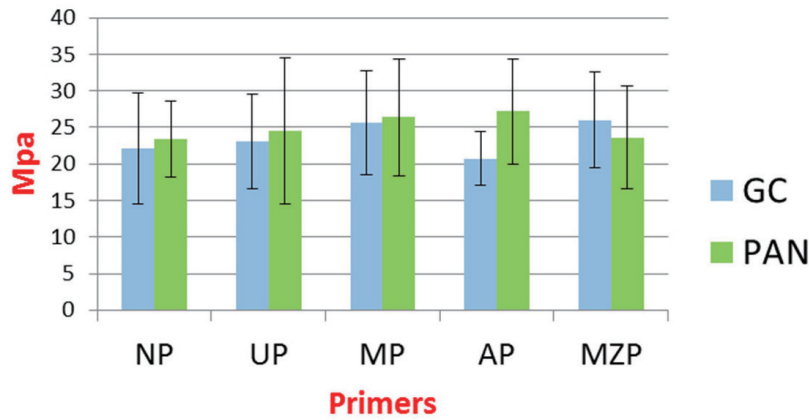
Results

The mean μTBS of all groups was ranged from 20 to 28 MPa (Fig. 3). Panavia F2.0 groups and G-Cem LinkAce groups showed no significant difference in bond strength. In the comparison among groups when primers were used, no statistically significant differences were observed ($P > .05$).

No pre-fabrication failures were found via the fractographic analysis after bonding of the resin cement to the metal alloy in all groups. Mixed failure, which is the combination of adhesive and cohesive failures, is the most prevalent failure mode in both the Panavia F2.0 and G-Cem LinkAce groups (Fig. 4).

Discussion

Traditionally, experimental designs for macrotests were used to compare bond strength to metal alloys in primer systems; however, because larger bonding areas lead to a higher possibility of error and may consequently reduce bond strength,¹⁴ microtests were developed.¹³ Microtensile bond testing is considered more appropriate for



	NP		UP		MP		AP		MZP	
	GC	PAN	GC	PAN	GC	PAN	GC	PAN	GC	PAN
Mean	22.09	23.37	23.03	24.57	25.59	26.41	27.22	20.75	26.00	23.61
(SD)	(1.97)	(1.34)	(1.67)	(2.58)	(1.84)	(2.07)	(1.86)	(0.97)	(1.70)	(1.81)

Fig. 3. Mean and standard deviation of μ TBS results. (GC: G-CEM LinkAce, PAN: Panavia F2.0, NP: no primer, UP: universal primer, MP: metal primer, AP: Alloy primer, MZP: metal/zirconia primer).

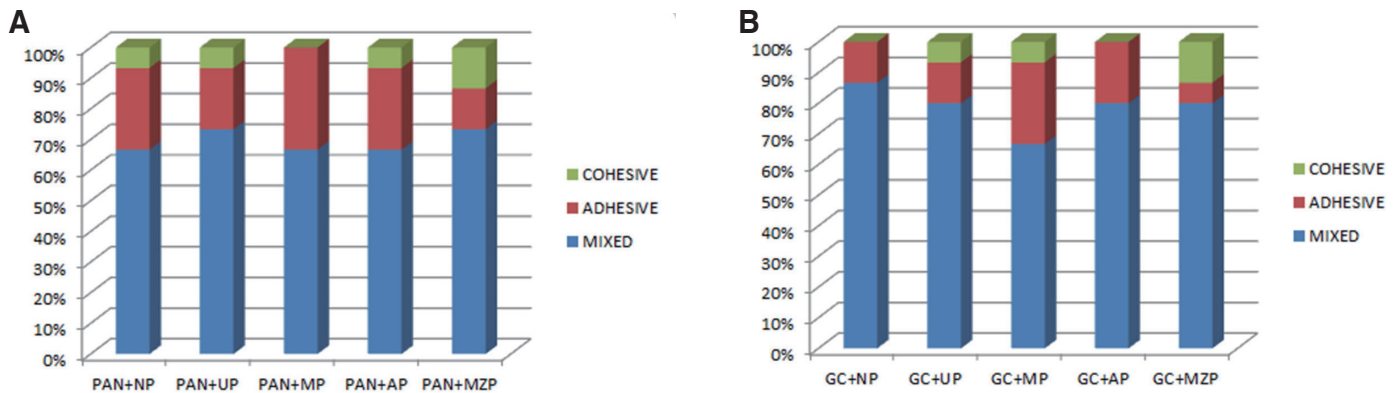


Fig. 4. The percentage of mode of failure in the Panavia F2.0 group (A) and G-Cem group (B). (GC: G-CEM LinkAce, PAN: Panavia F2.0, NP: no primer, UP: universal primer, MP: metal primer, AP: Alloy primer, MZP: metal/zirconia primer).

the evaluation of bond strength than microshear bond testing, since it allows a more uniform distribution of stress, reduces cohesive failure, and provides a more realistic measurement of the bond strength of the adhesive interface.^{15,16} However, cutting of the specimen for microtensile bond testing is difficult and the vibrations from cutting the specimen using a water-cooled diamond saw during microtensile bond tests may create microcracks on the periphery,¹⁷ and these microcracks may result in high levels of pre-testing failures.¹⁸ Therefore, most studies on the bonding of metal alloy to resin cement use microshear bond tests, in which specimens can be fabricated without cutting.¹⁶ The specimen in this study was fabricated by casting a

small-diameter metal beam and bonding it after fabrication, so that a microtensile test could be performed without the cutting.

In this study, we evaluate two adhesive systems: a conventional resin cement, Panavia F2.0, and a self-adhesive resin cement, G-Cem LinkAce. The 10-MDP, contained in Panavia F2.0, is a monomer mainly used as an etching monomer result from the function of dihydrogenphosphate group, and it has a quite hydrophobic property due to a long carbonyl chain render.¹⁹ The MDP has a coupling mechanism by: (i) dihydrogen phosphate group which presents great chemical bonding with Co-Cr alloy²⁰ and (ii) the polymerizable methacryloyl group which is essential for copolymerizing the MDP

monomer and the resin cement. The 4-META, which is included in the G-Cem LinkAce, is a monomer synthesized in the late 1970s.²¹ When mixing the 4-META with water, the hydrolysis reaction will occur and change 4-META to 4-MET and subsequently the esterification of 4-MET would promote adhesion.¹⁹ The chromium in the Co-Cr alloy produces a thin surface layer of chromic oxide at room temperature that can enhance the chemical bond between the Co-Cr alloy and 4-META.²²

However, in this study, the bond strength of the G-Cem group showed no significant difference with the Panavia F2.0 group. The μ TBS obtained for all groups were more than 20 MPa, similar to previous studies,²³ which is clinically acceptable.²⁴

The effect of a primer on bond strength varies with primer type. However, the use of metal primers for increasing the bond strength of non-precious alloys to resin cement remains controversial. According to Yoshida *et al.*,⁵ the bond strength between resin cement and sandblasted casting alloy was significantly higher when the metal primer was applied due to the affinity of some functional monomers to the oxide layer of base metal alloys. In addition, one recent study reported the tested primer significantly improved the tensile and shear bond strength of the resin cement to metal alloys.²⁵

In contrast, according to Di Francescantonio *et al.*,¹⁰ the use of alloy primer between metal alloy and resin cements did not increase the bond strength for most cementing systems tested. In the present study, although four primers with different functional monomers were applied to Co-Cr alloy, we observed no significant increase in bond strength in any of the four primer groups, which coincides with previous studies.^{6,8} It can be assumed that sandblasting increases the surface irregularities of the alloy and improves mechanical bond strength, and when the primer is applied, some of these surface irregularities may be filled. This mechanism can effect total microtensile bond strength, which may explain the results of this study.⁶

Other factors that may influence the durability of resin bonding and that were not evaluated in this study include pH changes, dynamic fatigue loading, thermocycling, and the various components of the resin cement and primer. Therefore, careful interpretation in the clinical application of these results is suggested and further *in vitro* research and standardized studies must be conducted to confirm the efficacy of the tested systems.

Conclusion

Within the limitation of this study, the μ TBS of all tested groups are relatively high; however, the primers used in this study showed no favorable effect on the adhesive bonding of Panavia F2.0 and G-Cem LinkAce resin cement to Co-Cr alloy.

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레진 시멘트와 코발트 크롬 합금의 미세인장결합강도에 다양한 프라이머들이 미치는 영향

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목적: 레진 시멘트와 코발트 크롬 합금 간의 미세인장결합강도에 다양한 프라이머들이 미치는 영향을 평가하기 위한 것이다.

재료 및 방법: 본 실험에서는 4개의 프라이머(Universal primer, Metal primer II, Alloy primer, and Metal/Zirconia primer)와 2개의 레진 시멘트(Panavia F2.0, G-CEM LinkAce)를 사용하였고, 길이 6 mm, 폭 1 mm, 두께 1 mm의 150개의 코발트 크롬 빔들이 캐스팅 과정을 통해 제작되었다. 150개의 코발트 크롬 빔들을 프라이머와 레진 시멘트의 종류에 따라 프라이머 처리를 하지 않은 대조군을 포함하여 10개 그룹으로 나누었다. 금속과 레진시멘트를 산화알루미늄($125\ \mu\text{m}$ 크기)으로 샌드블라스팅 처리한 후 실리콘 틀을 이용하여 접착시켰다. 실험 전에, 입체현미경(stereomicroscope)을 이용하여 모든 금속-레진 빔들을 검사하였고, 미세인장결합강도 실험을 시행하였다. 통계적인 평가에는 one-way ANOVA와 Tukey's test를 사용하였다.

결과: 모든 그룹들의 평균 미세인장결합강도는 20 - 28 MPa이었으며, 그룹 간에 통계적으로 유의한 차이는 없었다. Panavia F2.0과 G-CEM LinkAce 그룹 모두에서 접착 파절과 응집 파절이 동시에 나타나는 혼합 파절이 가장 흔하게 일어난 파절 양태였다.

결론: 모든 실험군들의 미세인장결합강도는 상대적으로 높았지만, 본 실험에서는 프라이머의 사용이 Panavia F2.0 및 G-CEM LinkAce 레진시멘트와 코발트 크롬 합금 사이의 미세인장결합강도를 증가시키지 않았다. (*대한치과보철학회지* 2019;57:95-101)

주요단어: 결합 강도; 금속 합금; 레진 시멘트; 프라이머

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