https://doi.org/10.4047/jkap.2017.55.3.264

표면처리에 따른 금속과 간접복합레진간의 전단결합강도 비교연구

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Comparison of shearbond strength between metal and indirect resin according to the different conditionings

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Purpose: The purpose of this study is to investigate the differences in bond strength of four different indirect composites to the gold alloy and Ni-Cr alloy according to type of metal surface treatment after water storage. **Materials and methods:** Type IV gold alloy and Ni-Cr alloy were used for casting alloy while four types of indirect composite resins (Gradia, Tescera Sinfony and in;joy) were used in this study. Metal specimens were produced by casting and total of 240 specimens (60 specimens per one indirect composite group) were prepared. After bonding indirect composite resin and undergoing 24 hours of polymerization, customized jig was attached to the metal specimen and shear bond strength were measured using universal testing machine. Also, differences in shear bond strength before and after water storage for 240 hours were also measured. **Results:** In the measurement of shear bond strength according to the metal surface treatments, bead group showed high strength followed by loop and flatting group (P<.05). After being stored in water bath for 240 hours, Gradia showed statistically significant high bond strength compared to other indirect composite resins in all groups (P<.05). **Conclusion:** Shearbond strength was found to be different according to type of metal surface treatment and type of metal used after storage in water. Further studies need to be developed for clinical practices as three are still problems of microleakage, stain or wear. *(J Korean Acad Prosthodont 2017;55:264-71)*

Keywords: Shear bond strength; Indirect composite resin; Metal; Gold alloy; Ni-Cr alloy

Introduction

The restorations of function and aesthetic in dental treatment are very important subjects. For this, many aesthetic and restorative materials have been developed and commercialized. Among these materials, dental porcelain is the most widely used material as porcelain fused metal crown or all ceramic crown shows excellent aesthetics and bio-compatibility.

However, the disadvantages of these porcelain-based restorative materials are that it takes more time, requires skillful techniques and is physically vulnerable. Also, its sub metal's melting point must be higher than the porcelain's burning temperature and it may cause excessive wear to the opposing dentition.[1,2] To overcome the drawbacks, indirect composite resins have been introduced since the 1980's. The first generation resin products were rarely applied to clinical practice due to low contents of inorganic filling materials which resulted in weak properties. Since the 1990's, the second generation products, in which inorganic material contents were increased and the polymerization was improved, have been used. These products have been further improved compared to the first generation products in terms of wear-resistance, fracture strength and color stability.[3]

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Article history: Received February 20, 2017 / Last Revision June 1, 2017 / Accepted June 19, 2017

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pISSN 0301-2875, eISSN 2005-3789

Indirect composite resin may be applicable to inlay, onlay, laminate, full coverage restoration, metal-resin restoration and etc. The resin may minimize the wear of antagonist teeth,[4] prevent less transformation during polymerization, show better color expression than porcelain and support better maintenance in case of fracture.[5] However, indirect composite resin, when compared to porcelain, had problems such as stain, plaque accumulation and wear and had weaker bond strength to metal surface, especially when comparing to porcelain fused crown.[6] Largely, there are two methods to increase the bond strength between resin and metal; mechanical method and chemical method. The mechanical methods contain beads, nail head, mesh, dovetails, loop, microcrystals, particle roughened surface, porous metal coating, sandblasting, electrolytic etching and etc,[7] while the chemical methods contain silica-coating, tin-plated, metal surface agent and etc. Generally, the factors affecting the bond strength between resin and metal include types of metal,[8] metal surface treatment methods[9] and types of resin[10] and the factors finally affecting the bond strength. The previous studies used each factor for the measurement of bond strength but few studies have been designed for the effect of combined factors on the shear bond strength.

This study used four commercially available indirect composite resins such as Tescera (Bisco Inc., Schaumburg, IL, USA), Sinfony (3M ESPE, Seefeld, Germany), in:joy (Denstply, Hanau, Germany) and Gradia (GC Co., Tokyo, Japan), attached them to precious alloy (Type IV gold alloy) and non-precious alloy (Ni-Cr alloy) according to the manufacturers' instruction and transformed the metal surface to the maintenance form of flatting, bead and loop in order to evaluate the difference of bond strength within each indirect composite resin. In addition, this study was intended to find out any differences in shear bond strength before and after storage under water for 240 hours.

Materials and methods

Manufacturing alloy specimens

For this study, Type IV gold alloy (DM46 Woori dongmyung Co. Seoul, Korea) and Ni-Cr alloy (VeraBond 2, Aalbadent, Cordelia, CA, USA) were used for casting alloy, while four kinds of indirect composite resins such as Tescera (Bisco Inc., Schaumburg, IL, USA), Sinfony (3M ESPE, Seefeld, Germany), in:joy (Denstply, Hanau, Germany) and Gradia (GC Co., Tokyo, Japan) were used (Table 1). Alloy specimens were manufactured in a size of 10 mm × 10 mm × 2 mm (W × H × T) as 240 pieces of square flat shape for four kinds; 30 gold alloy specimens and 30 Ni-Cr alloy, 60 each for four kinds. The surfaces on which indirect composite resin is attached were three kinds such as flatting, bead and loop; the flatting group (10 mm × 10 mm × 2 mm wax pattern) was ground with No.800 and No.1200 sandpaper after investing, quenching and melting. The bead group was attached onto the same wax pattern in a size of 7 mm in diameters, making a single layer using 200 ㎛ acrylic beads (VENEER-LOCK, Geo. Taub Prod. & Fusion Co. Inc., Jersey City, NJ, USA). The loop group was manufactured by attaching four of semi-circular acrylic pattern with 1 mm diameter to the same wax pattern diagonally into the 7 mm circle. This study intended to exclude any possible errors which may occur due to the direction of specimens. Ultrasonic cleaning was done for every metal specimen for 15 minutes after casting. Every metal specimens were subject to sandblasting with 50 ㎛ aluminum oxide and 60 psi pressure for 15 seconds 10 mm away vertically to the metal plane. Then, the ultrasonic cleaning was conducted for 15 minutes and dried (Fig. 1).

Resin build-up

Using 7 mm diameter and 3 mm high Teflon ring, four kinds of indirect composite resins were prepared in order of metal primer, opaque resin and indirect resin according to the manufacturers' instruction; it was then polymerized using the curing unit of each manufacturer (Table 1).

Measuring the shear bond strength

To measure the shear bond strength by using Instron (Instron 3366, Instron Corp., Norwood, MA, USA), 120 specimens were polymerized for 24 hours and the remaining 120 specimens were stored in a water bath filled with distilled water with 37 ± 1°C for 10 days. Then, they were fixed onto a fabricated jig and a load was applied to the same direction closed to the adhesive plane of specimens and indirect composite resin by using a blade tool. At that moment, the crosshead speed was 1.0 mm/min while the measurement of shear

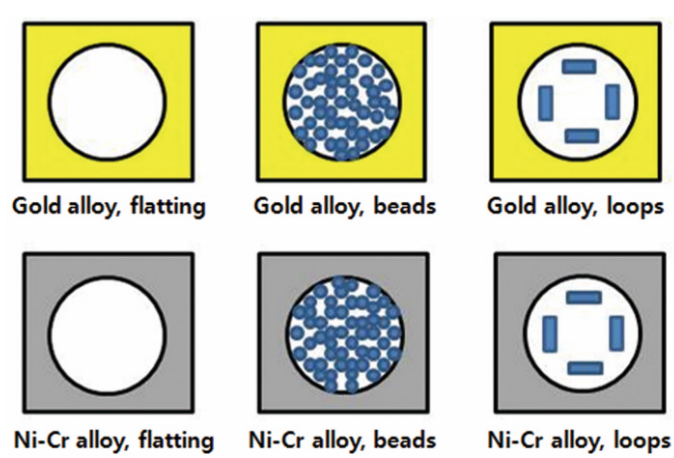

Fig. 1. Prepared gold alloy and Ni-Cr alloy specimens.

Table 1. The composite veneering resins used in this study

Product Name	Polymerization method	Composition	Curing unit	Manufacturer
Gradia	Light + Heat (70°C) oxygen barrier	UDMA, EDMA (75 wt% filler: Ceramic, Prepolymer, SiO_2)	LABOLIGHT LV-III (GC Co., Tokyo, Japan) for 5 minutes	GC, Japan
Sinfony	Light + Heat (40°C) with vacuum	HEMA, Octahydro-4,7-methano-1H-indenediyl-bis(methylene-diacrylate), (50 wt% filler 0.5 - 0.7 ㎛: Sr-Ba-Al-Si glass, pyrogenic silica)	Pre-polymerization: Visio Alfa (3M ESPE) for 5 s End-polymerization: Visio Beta Vario (3M ESPE) for 15 minutes under vacuum	3M ESPE, Germany
Tescera	Light + Heat & Aqua (130°C and 70 psi) oxygen scavenger	Bis-GMA, UDMA (70 wt% silica)	TESCERA ATL (Bisco) light cup for 5 minutes Heat cup for 15 minutes	Bisco, USA
in:joy	Light + Heat (70°C)	inorganic monofiller (55 wt% filler)	ENTERRA (Denstply) for 10 minutes	Denstply, Germany

bond strength adopted the applied max value while the resin was being separated from the metal. The fixation part of the jig was manufactured not to have any gap from a specimen and firmly fixed using screws to prevent any movement (Fig. 2).

Statistical analysis

For the statistical analysis, SPSS 19.0 program (SPSS Inc., Chicago, IL, USA) was used. After calculating the mean and standard deviation of each group, they were analyzed at 5% significance level by using two-way analysis of variance in order to compare the shear bond strength depending on the type of resin and metal surface type. Also, to verify any significant difference among groups, a multiple comparison test, Bonferroni test was conducted at 5% significance level. In addition, to compare the shear bond strength depending on metal, paired t-test was done at 5% significance level.

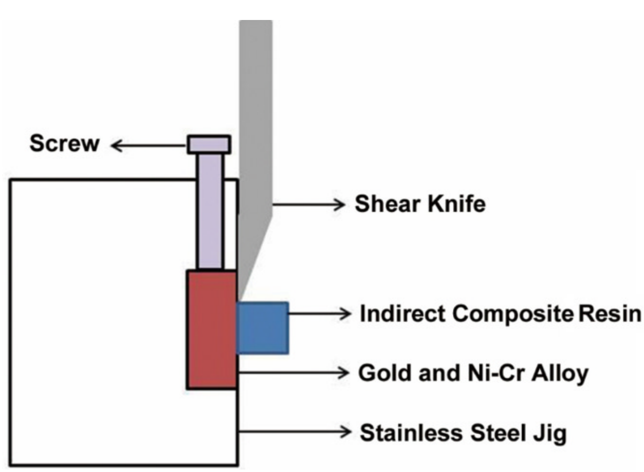

Fig. 2. Schematic diagram for measuring shear bond strength.

Results

Shear bond strength

To obtain the shear bond strength, the surface area was calculated by the diameter of specimen (7 mm), which was converted to MPa. Then, the mean and standard deviation of each group were calculated and the difference of bond strength among resins and the difference depending on the surface types were statistically processed with two-way ANOVA, which were summarized in Table 2, Fig. 3 and Fig. 4. To verify which group has significant difference, post hoc bonferroni comparison test was performed; in the bond strength measured before storing in water bath, Tescera showed significantly higher bond strength ($P < .05$) in every metal specimen than any other indirect composite resin. After storing in water bath for 240 hours, Gradia group showed significantly higher shear bond strength than other resin groups ($P < .05$). In the bond strength depending on the surface type, bead group showed significantly higher strength in every group before and after storage in water bath ($P < .05$), followed by loop group and flatting group. To compare the difference of inter-metal shear bond strength within each indirect composite resin, independent two sample t-test was executed and gold alloy in the bead group of Gradia, Sinfony and in:joy showed significantly higher bond strength than Ni-Cr alloy group ($P < .05$). In Gradia, flatting group and loop group showed higher strength than gold alloy, while the remaining groups did not show any significant difference.

Pattern of fracture

The fracture pattern on the contact surface with resin in the measurement of shear bond strength was shown in Fig. 5 and Fig. 6. Flatting group resulted in adhesive failure on the surface in every specimen while bead and loop groups showed complex failures in which adhesive failure and cohesive failure were co-exist-

Table 2. Shear bond strength (MPa) groups for incubation and metal and surface; Bonferroni tests of four indirect composite resins

Incubation	Metal	Resin	Surface treatment			Bonferroni test
			Flatting	Bead	Loop	
24 hours	Gold alloy	Gradia	7.65 ± 1.15	16.89 ± 1.89	12.94 ± 1.26	
		Tescera	9.48 ± 1.62	19.40 ± 1.11	13.15 ± 1.14	
		Sinfony	12.55 ± 2.58	14.03 ± 1.11	11.50 ± 0.88	
		in:joy	8.59 ± 1.41	14.59 ± 1.95	11.43 ± 2.01	
	Ni-Cr alloy	Gradia	10.68 ± 0.78	14.01 ± 1.19	14.69 ± 0.97	
		Tescera	9.90 ± 1.66	17.22 ± 1.59	14.24 ± 1.68	
		Sinfony	9.97 ± 1.11	14.72 ± 1.02	11.13 ± 0.87	
		in:joy	7.63 ± 1.44	11.27 ± 0.89	11.25 ± 1.10	
						$P < .05$
240 hours	Gold alloy	Gradia*	5.22 ± 1.04	20.61 ± 1.94	13.45 ± 1.15	
		Tescera	3.30 ± 1.25	19.32 ± 2.37	13.22 ± 1.85	
		Sinfony	4.03 ± 0.98	15.36 ± 0.78	13.75 ± 1.34	
		in:joy	3.02 ± 0.59	12.60 ± 1.18	9.08 ± 1.79	
	Ni-Cr alloy	Gradia*	15.60 ± 1.65	21.01 ± 1.63	20.23 ± 1.09	
		Tescera	3.85 ± 0.42	13.25 ± 2.92	13.03 ± 1.18	
		Sinfony	2.72 ± 0.51	17.65 ± 0.74	13.65 ± 1.37	
		in:joy	2.77 ± 0.39	9.99 ± 1.46	9.32 ± 1.22	

Shear bond strength data are presented as mean ± SD. $P = .05$. * Statistically significant

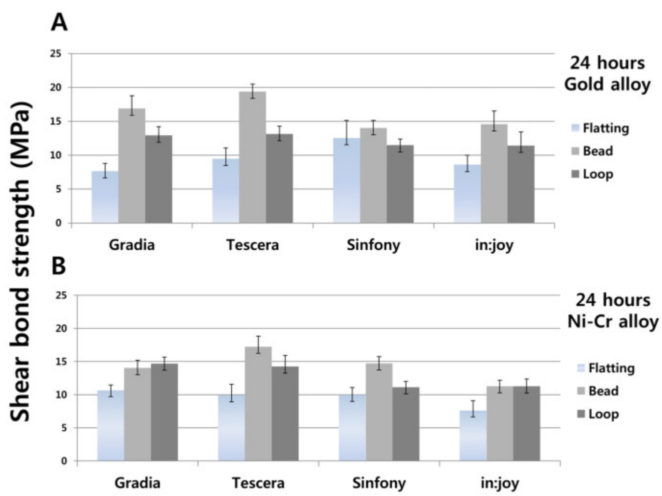

Fig. 3. Mean shear bond strength between metal surface treatments after 24 hours. (A) Mean shear bond strength of gold alloy specimens, (B) Mean shear bond strength of Ni-Cr alloy specimens.

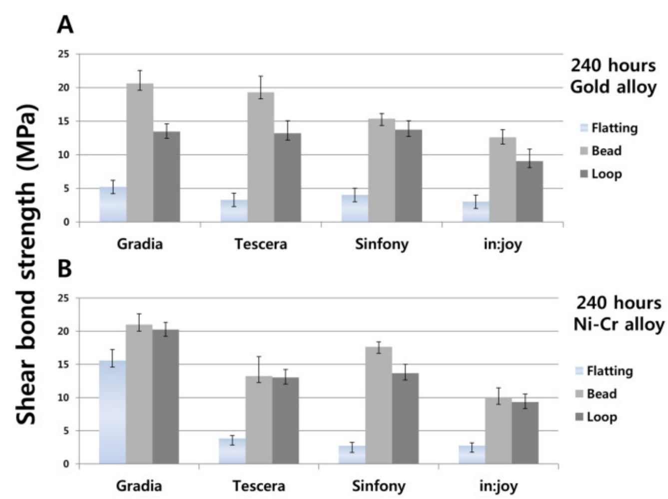

Fig. 4. Mean shear bond strength between metal surface treatments after 240 hours. (A) Mean shear bond strength of gold alloy specimens, (B) Mean shear bond strength of Ni-Cr alloy specimens.

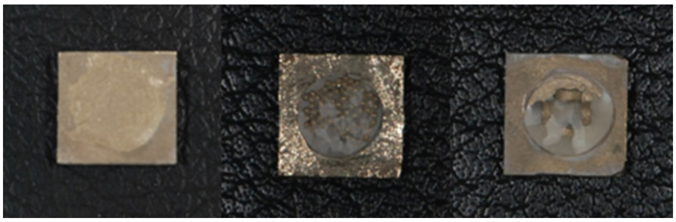

Fig. 5. Gold alloy specimen surfaces after shear bond strength measurement (In order of flatting, bead and loop).

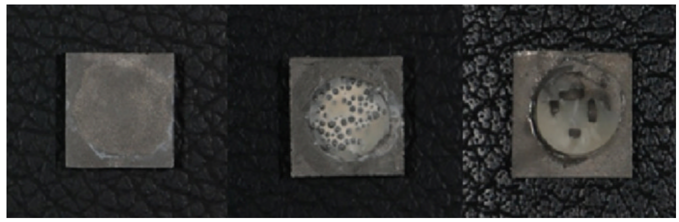

Fig. 6. Ni-Cr alloy specimen surfaces after shear bond strength measurement (In order of flatting, bead and loop).

ed. The bead group especially showed that indirect composite resin was tightly inserted between and among beads while the loop group showed more adhesive failure.

Discussion

In manufacturing dental prosthesis using indirect composite resin, the bond of sub metal structure and resin significantly affects the maintenance of prosthesis, function and aesthetics; it is one of the most important components for the long term prognosis.[11] The bond between indirect composite resin and metal can be classified into mechanical method and chemical method; the currently available indirect composite resins use the above two methods. The mechanical methods include macro retentive methods such as undercut, bead, loop, wire and nailhead 6 and macro retentive methods such as sandblast and acid etching, while the chemical methods include silica coating, metal pre-treatment agent and etc; there are also mixed methods such as tribochemical mechanism.

Traditionally, the bond of indirect composite resin and metal has used mechanical methods, which disadvantageously require more use of opaque to block the color of substructure, as the maintenance structure is increased, despite constantly maintaining the strength and risks of vulnerability to leakage.[2] Tanaka *et al*.[12] reported that using 200 ㎛ bead showed the highest maintenance of resin and might minimize the leakage. This study also used bead and loop as the mechanical methods, in which the size of bead was 200 ㎛ on average and the radius of loop was 500 ㎛, which showed more difference when applied to opaque resin. The present study reflected on the difference of bond strength and actually showed that four kinds of indirect composite resins had higher shear bond strength in the bead than the loop. However, it neglected the number, location and size of bead/loop, so the measurements might vary if the number and arrangement of bead/loop had been differently set. The study by Nicholls and Sheu,[13] showed that bond strength had more difference correlated to the arrangement and volume of the bead by arranging small beads as wide as the diameter.

The present study applied an adhesive as big as 7 mm circles onto the wax pattern to maintain every specimen uniformly since beads could not be arranged in gaps, brings it into contact with beads contained in a bowl, which is so called 'closed packing method'.[14] Although the height of loop was attempted to make as low as possible, its manufacturing was not possible as the loop hole was blocked if it was smaller than 500 ㎛. In addition, if a loop hole is manufactured to be small and the opaque layer blocks the hole, it may decrease the mechanical maintenance of the loop, probably weakening the final bond strength. Meanwhile, looking at the fracture shape, bead was shown to be separated upwards when indirect composite resin was separated higher than the maximum shear bond strength. However, loop showed that at the time of fracture, it was stuck on metal, taking more time to finally separate contrast to the expectation that fracture delay effect of loop would be higher than the bead.

In the study, sandblasting was applied to every specimen using Al_2O_3, with 50 ㎛ size, which was also a kind of mechanical method; especially, microretentive method. In the study by Sarafianou *et al*,[15] they used 250 ㎛ Al_2O_3 and 50 ㎛ Al_2O_3, experimenting the comparison of indirect composite resin's bond strength after the surface treatment of Ni-Cr alloy. As a result, they reported that irrespective of the type of metal pre-treatment agent, a group using 50 ㎛ Al_2O_3 showed higher bond strength. Regarding the chemical adhesive method, metal pre-treatment agents recommended by manufacturers were used. Actually, Tescera One-Step (Bisco Inc., USA), Sinfony Rocatec system (3M ESPE, Germany), Gradia Metal Primer II (GC Co., Japan) were used while in:joy (Denstply, Germany) recommended Rocatec system as they do not have their own metal pre-treatment agent. For instance, Rocatec system applies silicate particles onto metal surface, in which the particles of sandblast contacts with the metal surface, converting kinetic energy to thermal energy on 1 - 2 ㎛ surface and producing silicate layer as the high energy catches silicate particles.[16,17] The silicate layer's coupling action may increase the bond strength of composite resin and metal. In case of Metal Primer II, it contains the functional monomer of MEPS (methacryloyloxyalkyl thophosphate derivatives) when it reacts with metal. When the bond strength is increased and uniformly applied to sandblast-processed metal surface, it becomes polymerized.

Considering that the adhesiveness depends on the type of metals, the surface of non-precious alloy has a higher chemical reaction than its metal oxide layer; showing higher bond strength with resin if used with metal pre-treatment agent containing carboxylic acid derivatives.[18,19] Meanwhile, it is reported that using acidic adhesive monomer will result in lower bond strength[20] in the bond of precious alloy and resin than non-precious alloy. Then the bond strength could be significantly increased as a metal pre-treatment agent contains sulfur derivative monomer. Then, to significantly increase the bond strength, metal pre-treatment agent containing sulfur derivative monomer can be used. Matsumura *et al*.[21] reported that in the comparative experiment of bond strength between resin and gold alloy with four kinds of metal pre-treatment agents, the group using metal pre-treatment agent containing sulfur derivative monomer showed the greatest improved bond strength. In the study by Yamashita *et al*.,[22] they reported that in the experiment with a metal pre-treatment agent containing organic sulfur compounds, might effectively increase the bond strength of resin for gold and silver. A recent study reported that newly synthesized sulfur derivative monomer, dithiooctanoate monomer would be more effective in bonding precious metals than metal pre-treatment agents.[23] The study focused on evaluating the adhesiveness of metal pre-treatment agents produced for indirect composite resin provided by manufacturers, and excluded the types of metal pre-treatment agents. Also, as sandblast, Rocatec system and metal pre-treatment agents were mixed and used; it was not possible to directly compare the effects. But in most groups, it showed that bead groups and loop groups had significantly higher shear bond strength than flatting groups, suggesting that simultaneous use of mechanical and chemical bonds would be more desirable than using a chemical bond. The development of chemical bond between indirect composite resin and metal would yield greater advantages.

The thickness of opaque layer may be reduced, if specific maintenance device to accommodate the huge volume can be used. When the appropriate conditions are met, the life of prosthesis may be extended resulting from increased bond strength and reduced leakage between resin and metal. In addition, it may also help to secure thicker dentin, contributing to better aesthetic restoration. The shear bond strength measured after storing in a water tank for 240 hours showed significant reduction compared to the initially measured strength ($P < .05$), Some in the groups of Gradia and Sinfony, however, showed increased bond strength in the case of 240 hours of water tank storage compared to the initial shear bond strength. In association with this result, Kern and Thompson[24] explained that the reaction resolving the polymerization stress in complex resin owes to changes in temperature during thermal cyclic which increases the bond strength slightly while Moulin *et al*.[25] and Knobloch *et al*.[26] insisted that it is closely related to increases in conversion rate of polymer material as the temperature increases. It may be anticipated that the results can be variable according to the components of resin, polymerization methods or outworn polymerization facility.

In the previous studies reporting the comparative results for the bond strength between porcelain and indirect composite resin, Ciftci *et al*.[27] and Almilhatti *et al*.[28] reported that in the experiment to measure the bond strength for Ni-Cr alloy, the bond strength would not be equal to that of porcelain. However, Petridis *et al*.[29] measured the bond strength for the group bonding gold alloy for porcelain, feldspatic porcelain and the counterpart for three kinds of indirect composite resins before and after thermocycling. He reported that although the bond strength of porcelain was not greater than that of indirect composite resin, they showed similar bond strength; which shows the possibility of the bond between metal and indirect composite resin in the available range in the clinical practice. Nevertheless, further studies need to be developed for clinical practices as it still has the problems of microleakage, stain or wear.

Conclusion

Within the limitation of this study, regardless of the type of metal surface treatments, bead group showed the highest strength followed by loop and flatting group. Also, Gradia showed statistically significant high bond strength compared to other indirect composite resins in all groups. Further studies need to be developed for clinical practices as it still has the problems of microleakage, stain or wear.

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표면처리에 따른 금속과 간접복합레진간의 전단결합강도 비교연구

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목적: 본 연구에서는 4종의 간접복합레진에 대하여 금합금과 Ni-Cr합금에 대한 결합강도의 차이와 각각의 합금에 표면형태를 다르게 하여 일정한 온도의 증류수에 보관 전과 후에 전단결합강도의 차이가 있는지 알아보고자 하였다.
재료 및 방법: 금속 시편을 주조 제작하였는데, 금합금과 Ni-Cr합금의 표면형태를 flatting, bead, loop로 하여 4종의 간접복합레진(Gradia, Tescera Sinfony and in;joy)당 60개씩 총 240개를 준비하였다. 이후 금속표면에 각각의 제조사에서 지시한대로 간접 복합레진을 접착하였다. 간접복합레진의 중합 24시간 후에 만능시험기를 이용하여 전용 지그에 금속시편을 부착 후, 전단결합강도를 측정하였다. 또한 증류수에 240시간 보관 후에 같은 방법으로 전단결합강도를 측정하였다.
결과: 금속의 표면 형태에 따른 전단결합강도 측정에서 4종의 레진 모두 bead군에서 높은 결합강도를 보였고 loop, flatting 순이었다 ($P < .05$). 수조에서 240시간 보관 후에는 Gradia가 모든 군에서 다른 레진 군에 비해 비슷하거나, 통계적으로 유의하게 높은 ($P < .05$) 전단결합강도를 나타내었다.
결론: 금속표면의 형태에 따라, 금속의 종류에 따라, 물속 보관 전후에 따라 금속과 간접복합레진의 결합력의 차이를 나타내었고 추가 실험을 통해 최적의 조합에 따른 임상에서의 적용가능성을 보여주었다. *(대한치과보철학회지 2017;55:264-71)*

주요단어: 전단결합강도; 간접복합레진; 금속; 금합금; Ni-Cr합금

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원고접수일: 2017년 2월 20일 / 원고최종수정일: 2017년 6월 1일 / 원고채택일: 2017년 6월 19일

[a] 이 두 저자는 본 연구에 동일한 기여를 하였음.