Evaluation of Universal Bonding Agent Containing MDP on Zirconia Bonding

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감사의 글

2013년 보존과에서 수련을 시작한 이후, 대학원 수업을 듣기 시작한 일이 엊그제 같은데 어느새 석사 논문을 작성하며 네 번째 학기를 마무리 짓고 있는 것이 신기하게 느껴 집니다. 실험에 대한 대략적인 구상과 다른 기존의 연구에 대하여 조사 하던 중 불의의 교통사고를 당하게 되어 자칫 실험 시작도 하지 못하고 졸업을 유보 해야 할 상황에서, 저를 믿어주시고 끊임없이 지도해 주시어 결국 실험을 잘 마무리 하고 석사 과정을 잘 마무리 할 수 있도록 이끌어 주시고 지도해 주신 박정원 교수님께 특별한 감사의 인사를 드립니다.

또한 논문을 심사해주시며, 실험의 전반적인 방향을 바로 잡아 주시고 실험 재료에 대하여 세심하게 검토해주신 박성호 교수님, 연구 자체의 의미를 다시 생각해보고 자칫 산만해 질 수 있는 연구를 정리할 수 있게 지도해 주신 노병덕 교수님 모두 감사 드립니다.

보존과에 들어와 다양한 임상 경험과 지식을 습득하는 것뿐 아니라, 환자를 대하는 마음 가짐 까지 배울 수 있게 기회를 주시고 이끌어 주신 신수정 교수님, 수련 생활을 함에 있어 간과하기 쉬운 부분을 짚어 주시고 생각해 볼 수 있게 지도해 주신 김선일 교수님께 감사의 인사 드립니다. 또한 부족한 저에게 많은 것을 알려주시고 지도해 주신 이찬영 교수님, 이승종 교수님, 김의성 교수님, 정일영 교수님, 신유석 교수님께 감사를 드립니다. 실험을 하면서 많은 도움을 주신 기공실장님과 회사 관계자 분들, 통계에 많은 도움을 주신 홍정화 선생님께도 감사를 드립니다.

마지막으로 항상 제 뒤에서 사랑과 정성으로 응원해 주시고 도와주시는 부모님과 이 기쁨을 함께 나누고 싶습니다.

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ABSTRACT

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(Directed by Prof. Jeong-Won Park, D.D.S., M.S.D., Ph.D.)

The purpose of this study was to compare the evaluation of the universal bonding agents and their surface treatment methods, sandblasting and zirconia primer application, on Y-TZP zirconia bonding.

60 Y-TZP blocks (20 mm x 80 mm x 5 mm) were produced. Three universal bonding agents (Single Bond Universal(SBU), All-Bond Universal(ABU), GC-Premio Universa(GCU), one MDP containing self-etching agent (Clearfil SE bond(CSE)) and one total etching agent (Optibond FL(OFL)) were tested with/without sandblasting and zirconia primer (Z-Primer plus(ZP)).

Bonding procedure of each group was performed according to the guidance of the manufacturer.

Microshear bond test was carried out using a universal testing machine with cross-head speed of 1.0 mm/min. Peak load to fracture (N) was measured for each specimen.

Data for bonding agent, z-primer, sandblasting were analyzed by 3-way ANOVA. Bonferroni Post-hoc analysis was performed for each z-primer, sandblasting, materials, materials & z-primer, and materials & sandblasting deemed statistically significant.

As a result, the sandblasting and z-primer application increased the bond strength of the Y-TZP and resin composite ($P<0.05$). There are significant differences between SBU and ABU, GCU, ZP, OFL group. Also, there is a significant difference between OFL and CSE group ($P<0.001$).

Following the results of this experiment, generally sandblasting and Z-primer application can increase bond strength between Y-TZP and composite resin. However, SBU showed superior bond strength than other universal bonding agents with or without sandblasting and Z-primer, and it can simplify the clinical procedure.

Key words: MDP, universal bonding, zirconia bonding

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I. Introduction

Yttria-tetragonal zirconia (Y-TZP) is an effective option for dental treatment due to its mechanical strength. Y-TZP has a lot of advantages such as superior esthetics, biocompatibility and strong structure (1, 2). Due to these advantages, Y-TZP is used as core material for ceramic crown and also applied as whole crown, inlay and onlay (1, 2). Moreover, with the development and wide usage of CAD/CAM system, the use of Y-TZP seems to have increased recently (1, 2).

However, obtaining stable cementation of Y-TZP restorations is quite a challenge (3). According to a study by Larsson et al, the most critical factor for the restoration failure of Y-TZP was shortage of retention, accounting for 45% (4). To prevent shortage of retention, the most essential and basic factor is proper preparation of the tooth. Also, the cementation process is important to obtain sufficient retention of the restoration.

Effective cementation of Y-TZP is difficult due to the fact that it is resistant to acid etching unlike other conventional glass ceramics. This is due to the high crystalline structure of Y-TZP and its lack of glass particles (5). Therefore, a mechanical roughening process such as air-abrasion and application of laser can be considered as an useful method (5). While, the effectiveness of the mechanical pre-treatment process for Y-TZP bonding is controversial (6). However, it is essential to note that sandblasting may induce microcracks, which may be a critical degrading factor in terms of the long-term bond strength of Y-TZP restorations. (7, 8)

Another approach to encourage the bond strength between Y-TZP and composite resin is chemical treatment. In the early days, application of silane for Y-TZP was considered as a pre-treatment option. However, unlike conventional glass-ceramic restorations, application of silane for Y-TZP cementation has proven to have no effect (9-11).

An alternative method for promoting bond strength is the application of MDP monomer-containing agents. According to some studies, using agents containing MDP monomer has shown to effectively improve bond strength (12-15). The zirconia primer is one of the bonding agents that contain MDP monomer. It has been introduced as a surface treatment agent due to its ability to enhance the bonding strength between Y-TZP and

resin cement (16). Thereafter, the total bonding procedure for Y-TZP restorations can be summarized as primarily applying the zirconia primer and applying the bonding agent afterwards. However, pre-treating Y-TZP surface with the zirconia primer is a time-consuming process. Many products in the dental field that contain MDP monomer other than zirconia primers have been launched. For example, “universal” and “multi-purpose” adhesives are such products. The MDP monomer-containing bonding agent called universal adhesive can be applied as self-etch or as etch & rinse adhesive. Universal adhesives have been developed to make the clinical procedure more simple and user-friendly. Therefore, to simplify the clinical procedure, application of universal adhesives in a single step can be considered as an alternative treatment option for bonding of Y-TZP restoration. The purpose of this study was to compare the evaluation of the universal bonding agents and their surface treatment methods, sandblasting and zirconia primer application,, on Y-TZP zirconia bonding.

II. Materials & Methods

(1) Specimen preparation

Sixty six Y-TZP (Everest ZS-Ronde, KaVo; Biberach, Germany) blocks (20 mm x 80 mm x 5 mm) were produced and assigned randomly to 11 groups (n = 6), according to the surface treatment and bonding agent used. The products used in this study are listed in the table 1. The products are applied according to the instructions of manufacturer.

Table 1 Material used in this experiment

Product	Composition	Lot	Manufacturer	Instruction for use
Single Bond Universal (SBU)	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™, Copolymer, Filler, Ethanol, Water, Initiators, silane	517709	3M EPSE (Saint Paul, MN, USA)	1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20s. If necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5s until it no longer moves and the solvent has evaporated completely. 3. Light cure
All-Bond Universal (ABU)	MDP, Ethanol, Bis-phenol glycidyl methacrylate (Bis-GMA), HEMA, water, Initiators	1300000367	Bisco (Schaumburg, IL, USA)	1. Apply two separate coats of adhesive, scrubbing the preparation with a microbrush for 10-15 s per coat. Do not light polymerize between coats 2. Evaporate excess solvent by thoroughly air-drying with an air syringe for at least 10 s, there should be no visible movement of the material. The surface should have a uniform glossy appearance. 3. Light cure
OptiBond FL (OFL)	Primer: HEMA, GPDM, PAMM, photo-initiator, ethanol, water. Bonding: TEGDMA, UDMA, GPDM, HEMA, Bis-GMA, filler, photo-initiator	5143463	Kerr (Orange, CA, USA)	1. Apply Primer over enamel and dentin surfaces with a light scrubbing motion for 15 s. Gently air dry for approximately 5 s. 2. Apply adhesive over enamel and dentin uniformly creating a thin coating. Blow to margin or to thin if necessary using a light application of air. 3. Light cure
Clearfil SE Bond (CSE)	Primer: 10-MDP, HEMA, hydrophilic DMA, photo-initiator, aromatic tertiary amine, water. Bonding: 10-MDP; Bis-GMA, HEMA, Hydrophobic DMA, photo-initiator, aromatic tertiary amine, silanated colloidal silica	52258	Kuraray (Tokyo, Japan)	1. Apply Primer to the entire cavity wall with a sponge or a disposable brush tip.Leave it in place for 20 s. 2. Evaporate the volatile ingredients with a mild oil-free air stream. 3. Apply Bond to the entire surface of the cavity with a sponge or a disposable brush tip. 4. Make the bond film as uniform as possible using a gentle oil-free air stream. 5. Light-cure BOND for 10 s with a dental curing unit.

GC universal (GCU)	Acetone, Water, 10-MDP, 4-MET, Methacryloyoxyalkl thiophosphate (MEPS), Ester phosphate monomer, Thio-phosphoric ester monomer	GC (Tokyo, Japan)	
Z-Prime Plus	Biphenyl dimethacrylate, MDP, Ethanol	1400007936	Bisco (Schaumburg, IL, USA)	1. Clean the internal surface of the restoration; rinse and air dry. 2. Apply 1-2 coats of Z-Prime Plus, uniformly wetting the internal surface. Dry with an air syringe for 3-5 s. 3. Proceed with cementation using a luting cement.
Premisa	Prepolymerized filler, Barium glass, Silica filler, Ethoxylated bis-phenol-A-dimethacrylate, Triethylene glycol dimethacrylate (TEGDAMA), Light-cure initiators and stabilizers	Translucent 2955400	Kerr (Orange, CA, USA)	Light-cured

(2) Experimental groups

The prepared specimens were randomly divided into 11 groups and each group was divided into 2 subgroups. The groups tested in this study are showed in Figure 1. Each group consisted of 30 specimens. Each group was divided into two subgroups, based on the presence or absence of sandblasting procedure. For the sand blasting, 50 μm diameter aluminum-oxide particles were blasted perpendicularly into the Y-TZP surface under an air pressure of 3 bar at 10.0 mm distance for 10 seconds (RONDOflex plus 360, KaVo; Biberach, Germany).

Figure 1. Experimental design of this experiment

(3) Bonding procedure

The application of primer and bonding agent was done according to the manufacturers' instructions (Table 1). After application of the bonding agent, the 2 mm height cylindrical translucent molds (TYGON® R-3603 Laboratory Tubing; 0.8 mm - diameter, Sanit Gobain performance Plastic, Maime Lakes, FL, USA) were located on the site in which the bonding agent had been applied and light-cured by LED curing unit (Elipar S10, 3M ESPE, St. Paul, MN, USA) providing 1200 mW/cm^2 for 40 seconds. Resin was filled into the cylindrical translucent mold by a resin applicator and packing was performed with dycal applicator. After resin filling, the cylindrical translucent mold was light-cured by LED curing light providing 1200 mW/cm^2 for 40 seconds. After light curing, the cylindrical translucent mold and excess bonding agent was removed by a #11 blade (Paragon® Sterile stainless surgical blades, LANCE PARAGON LTD., Sheffield, S6 2BJ. England). The specimens were stored in air, for one day.

(4) Microshear bond test

Following maturation stage for 24 hours to complete composite resin polymerization, each specimen was attached to the testing device with cyanoacrylate adhesive (ALTECO Korea Inc., Pyungtaek, Korea). Microshear bond test was carried out using a universal testing machine (EZ test, Shimadzu Co., Kyoto, Japan) with cross-head speed of 1.0 mm/min. Shear load was applied to the base of the composite resin cylinder with a thin metal wire (wire-loop method) until bond failure of the specimen occurred (Pashley et al., 1995).

(6) Statistical analysis

The data was statistically analyzed using SAS 9.2 software (SAS Institute, Cary, NC, USA).

Data for 3 factors (material, z—primer, sandblasting) were analyzed by 3-way ANOVA. post-hoc analysis was performed for each Z-primer, sandblasting, and materials deemed statistically significant. Furthermore, analysis was performed for interactions between material and z-primer, and interactions between material and sandblasting. Bonferroni method was applied for post-hoc analysis.

III. Results

The mean and standard deviation of microshear bond strength is listed in table 2.

3 factors (materials, sandblasting, Z-Primer) was analyzed by 3-way ANOVA in table 3. According to the results, there were significant differences for each factors - bonding materials, sandblasting, Z-Primer.

The post-hoc analysis was taken for the terms that represent the significant differences.

The post-hoc analysis results for materials are depicted on table 2, with significant difference between each group. As seen on figure 2, there was significant difference between the SBU group and Z group, OFL group, CSE group, and GCU group. Furthermore, there was significant difference between the CSE group and OFL group.

Sandblasting and Z-primer application showed higher bond strength ($p<0.05$).

According to the post-hoc analysis on relationship between sandblasting and material, sandblasting caused higher bond strength in GCU group and OFL group ($p<0.05$).

According to the post-hoc analysis on relationship between Z-primer and material, Z-primer application attributed to higher bond strength in GCU group, OFL group, and CSE group ($p<0.05$).

Table 2. Mean and standard deviation of microshear bond strength (N, mean ±S.D.) (n=15 for each condition)

Bonding agent	Z-primer		No sandblast	Sandblast	Total mean of bonding agents(n=15 or 30)
Z Primer only			28.98 ± 11.03	32.77 ± 8.52	30.88 ± 9.87^{BC}
Optibond FL	No		22.18 ± 6.66	27.19 ± 8.64	28.67 ± 8.43^{C}
	Yes		30.85 ± 6.93	34.47 ± 6.65	
Clearfil SE Bond	No		25.03 ± 8.31	30.86 ± 10.75	33.35 ± 9.42^{AB}
	Yes		38.61 ± 5.00	38.88 ± 4.61	
Single Bond Universal	No		37.95 ± 7.70	36.38 ± 5.32	37.43 ± 6.65^{A}
	Yes		38.39 ± 6.87	36.99 ± 6.99	
All-Bond Universal	No		32.84 ± 4.68	30.43 ± 5.65	32.34 ± 6.17^{BC}
	Yes		34.00 ± 7.10	32.11 ± 6.99	
G-Premio Universal	No		23.59 ± 4.40	29.26 ± 7.36	29.92 ± 7.48^{BC}
	Yes		31.68 ± 5.27	35.12 ± 7.67	
Total mean of Z-Primer	No	29.57 ± 8.58*	Total mean of sandblasting		
	Yes	34.40 ± 7.62**	31.28 ± 8.78^{ǂ}	33.13 ± 7.93^{ǂǂ}	

Different superscript in total mean of bonding agents means statistically significant different ($p<0.001$).

Different number of "*" means statistically significant different ($p<0.001$).

Different number of "ǂ" means statistically significant different ($p=0.454$).

Table 3. Statistical results of three–way ANOVA

Source	DF	F Value	p - value
Sandblasting	1	6.33	**0.0124**
Z Primer	1	44.66	**<.0001**
Sandblasting * Z Primer	1	1.05	0.3053
Materials	**5**	12.51	**<.0001**
Sandblasting * Materials	5	2.65	**0.0232**
Z Primer * Materials	4	5.66	**0.0002**
Sandblasting * Z Primer * Materials	4	0.43	0.7867

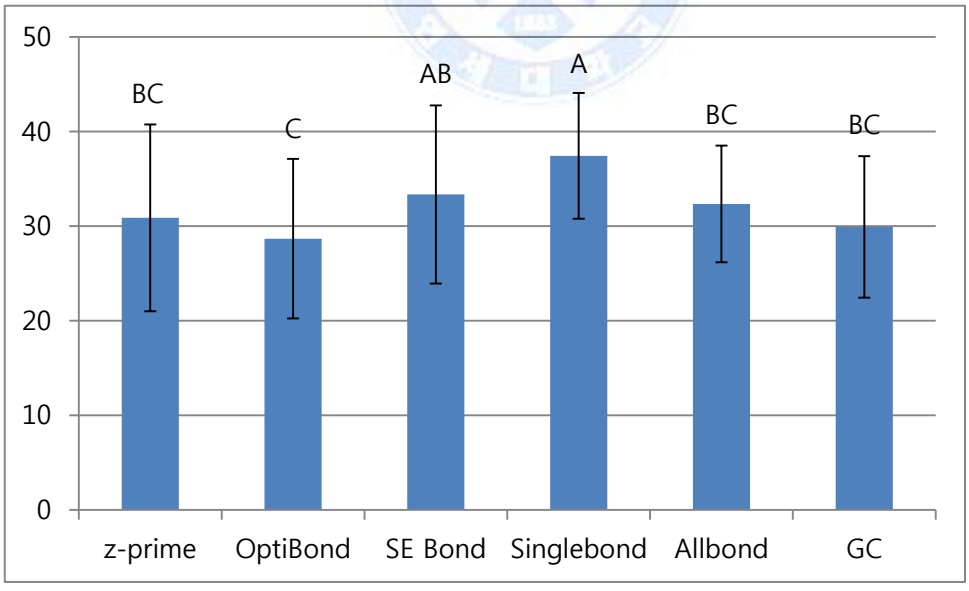

Figure 2. Bonferroni Post-hoc analysis of bonding agents

IV. Discussion

This study evaluated the effect of MDP-containing primer or bonding agent on bond strength between Y-TZP and composite resin. The effectiveness of sandblasting is still in debate. Some studies have claimed that pre-treatment of Y-TZP by using sandblasting has positive effect on enhancing bonding strength (18-20). In these studies, sandblasting the surface of Y-TZP showed increase of bond strength. This may be due to the roughening of the Y-TZP surface, which leads to increased micromechanical retention between Y-TZP and composite resin (10).

In contrast to the fore-mentioned studies, some studies have shown that sandblasting has little or no effect on bonding strength. According to these studies, it was concluded that the sandblasting process showed improvement of initial bond strength, but the effect did not last long. Our study was in accordance with previous studies, showing enhancement of initial bond strength after sandblasting. However, the long-term bond strength was not evaluated in this study. Therefore, long-term evaluation including thermocycling is necessary to measure precise effect of sandblasting on bonding strength between Y-TZP and resin.

In our study, the Z-prime plus was chosen for representing the zirconia primer to improve bond strength between Y-TZP and composite resin. There are many studies that explain the advantage of using zirconia primer for bonding Y-TZP. (13, 14, 21) This study also coincided with other studies that the zirconia primer encourages the bond strength between Y-TZP and resin composite.

In the present study, five bonding agents were used. As negative control, the OptiBond FL was selected due to the fact that it is a conventional 3-step etch-and-rinse system bonding agent that doesn't contain MDP. The result of microshear test in this group showed relatively low bonding strength. When Z-prime Plus (Bisco Inc.) was additionally applied before the application of OptiBond FL, they showed improved bond strength between resin and zirconia similar with other recent study (21).

In this study a zirconia primer has a positive effect on bond strength between Y-TZP and resin. This may be due to the fact that the MDP monomer increases the wettability of Y-TZP, leading to the reduction of the contact angle of the Y-TZP surface, leading to improvement of the bond strength. Moreover, the MDP monomer seems to have an affinity to metal oxides such as zirconium dioxide (ZrO_2) (10, 22). Such properties of the zirconia primer may attribute to the improvement of bond strength between Y-TZP and resin composite.

In addition, the Clearfil SE Bond, a 2-step self-etch bonding agent containing MDP monomer, was tested. The CSE group was a representation of conventional bonding agent with MDP monomer that is not a universal bonding agent. Although it was expected to show relatively high adhesion, the result of the bond strength of this group was not significantly high. Further study is needed for thorough evaluation of such materials.

However, when Z-prime Plus was additionally applied before application of SE bond, the result showed significantly higher bonding strength than non-primed group, similar to that of OFL. This result may be due to the improvement of the bond strength between Y-

TZP and composite resin through the additional MDP application. However, it is necessary to find different aspects of MDP in SE bond and Z-prime plus.

Three universal adhesives (All-Bond Universal, Single Bond Universal, GC Universal) were chosen to compare the bonding effect of universal adhesives, because they are containing MDP and simple to use.

The Single bond universal showed the highest bonding strength. This could be due to the silane and MDP monomer contained in Single bond universal. Some studies issued that silane has no effect on bond strength between Y-TZP and resin composite (9, 11). However, the improved bond strength with Single bond universal in this study may indicate that silane may have a positive effect in increasing the bond strength between Y-TZP and resin composite. Further studies are necessary to confirm this effect. In group SBUZ, additional application of Z-prime plus did not improve the bond strength compared to group SBU. This may indicate that the Single bond universal could substitute zirconia primers in the future, due to the similar properties of the MDP monomer in both Single bond universal and Z-prime plus.

The bond strength of group GCU, applied with GC-universal, showed lower bond strength than the bond strength of the group ABU and SBU. The GC-universal is a universal bonding agent like Single bond universal, and All Bond universal. However, due to the different composition of the GC-universal, the bond strength between Y-TZP and composite resin could be uneven. Similar to other universal bonding agents, the additional application of Z-prime plus improved the bond strength between Y-TZP and composite resin. Further study is necessary to prove the properties.

V. Conclusion

Following the results of this experiment, generally sandblasting and Z-primer application can increase bond strength between Y-TZP and composite resin. However, SBU showed superior bond strength than other universal bonding agents with or without sandblasting and Z-primer, and it can simplify the clinical procedure.

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국문요약

지르코니아 수복물 접착에 있어서

MDP 를 함유한 Universal bonding agent 의 평가

연세대학교 대학원 치의학과

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이번 연구의 목적은 지르코니아 수복물을 접착함에 있어서, universal bonding agent 를 사용하고, sandblasting 과 같은 표면처리 방법을 사용하거나 지르코니아 primer 를 사용하는지 에 따른 영향을 평가하는 것이다.

실험 방법은 60 개의 지르코니아 (20 mm x 80 mm x 5 mm) 블럭을 사용하였다. 3 가지 universal bonding agnet (Single Bond Universal(SBU), All-Bond Universal(ABU), GC-Premio Universal(GCU)) 와 MDP 를 함유하고 self-etching 계열인 Clearfil SE bond(CSE) 와 total-etching 계열인 Optibond FL(OFL) 를 사용하였고, sandblasting 시행 여부와 zirconia primer (Z-Primer plus(Z))

사용여부를 가지고 실험을 진행하였다. 각 군의 bonding agent 는 제조사의 지시를 따라서 사용하였다. 미세전단결합강도 시험은 universal testing machine 을 사용하여 1.0 mm/min 의 속도로 cross-head 를 이동시켜 시행하였다. 파절시의 peak load (N) 를 각각 시편에 대하여 측정하였고, 실험 결과를 bonding agent, zirconia primer, sandblasting 의 3 가지 변수에 관하여 3-way ANOVA 통계적 처리를 시행하였다. 사후 검정은 통계적 유의차가 있다고 나온, zirconia primer, sandblasting, material 각각과, material 과 zirconia primer 그리고 material 과 sandblasting 에 디하여 시행하였다.

결과는 sandblating 과 zirconia primer 를 적용하면 지르코니아 수복물의 결합강도가 증가한다 ($P<0.05$). 또한 SBU 군과 ABU, GCU, Z, OFL 군 사이의 통계적 유의차가 존재하며, OFL 군과 CSE 군 사이에도 통계적 유의차가 존재한다 ($P<0.001$).

이번 실험 결과에 따르면, 지르코니아 수복물 접착에 있어서, 일반적으로 sandblasting 과 zirconia primer 를 적용하는 것은 결합강도에 도움이 된다. 또한 SBU 는 sandblasting 이나, zirconia primer 사용 여부에 관계없이 다른 universal bonding agent 에 비해서 가장 우수한 결합력을 나타내며, 이를 통하여 임상 술식을 간소화 할 수 있을 것으로 생각된다.

핵심이 되는 말: MDP, universal bonding, 지르코니아 수복물 접착