Effect of dentin coating on microleakage and bond strength of adhesive resin cements

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Purpose: This study determined the effect of the application of dentin coating materials on microleakage and shear bond strengths of adhesive resin cements.
Materials and Methods: One hundred and five bovine dentin specimens for dye penetration testing and shear bond testing were ground flat with #600 grit silicon-carbide paper. The specimens were divided into fifteen groups of seven specimens each, and they were subdivided into two dentin-coated (Touch&Bond and Brush&Bond) and non-coated (control) conditions of five groups each. One out of the five groups was subjected to a microleakage test, which was performed by immersing the specimens in 5% methylene-blue for 24 hours. The remaining four groups were used for shear bond test. Shear bond specimens were fabricated by bonding Ni-Cr alloy disks with adhesive resin cements (Panavia F or C&B Metabond) both with and without post-treatment of proprietary dentin conditioners to the coated surfaces. The mean values of microleakage and shear bond strength for each group were statistically analyzed by two-way analysis of variance (ANOVA) and Bonferroni/Dunn test at a 95% confidence level.
Results: The dentin coated specimens showed significantly less dye penetration than the non-coated specimens. For both adhesive resin bonded groups, shear bond strengths of Brush&Bond coated specimens without post-treatment (7.8 MPa for C&B Metabond specimens, 1.5 MPa for Panavia F specimens) significantly increased (16.0 MPa and 21.7 MPa, respectively) through the use of dentin conditioners onto the coated surface.
Conclusion: It was suggested that appropriate post-treatments to the coated surfaces were necessary when adhesive resin cements were used for luting prostheses. **(Int Chin J Dent 2006; 6: 71-78.)**
Key Words: adhesive resin, dentin coating, dentin conditioner, microleakage, bond strength.

Introduction

Dentin exposure occurs when enamel is removed during tooth preparation. In cases of preparation for full coverage crowns, more than a million dentinal tubules are exposed,[1] and this provokes postoperative sensitivity in vital teeth if an appropriate procedure is not employed. Exposed dentin is usually covered with provisional restorations retained by temporary cement.[2] However, these are often subject to problems including microleakage, dislodgement, and even fracture. These contribute to the onset of post-preparation tooth sensitivity and potential pulp damage.[3,4] Therefore, it is important to maintain dentin protection during the provisional phase of treatment so that physical and/or chemical stimuli do not result in postoperative sensitivity. Dentin protection is essential in preventing bacterial infection and maintaining pulp health. In endodontically treated teeth, coronal seal has been shown to be a major factor in the long-term success of endodontic treatment.[5]

Dentin has less resistance to acid attack when compared to enamel; thus, it is more susceptible to caries. Secondary caries after final restoration of a tooth affects dentin surfaces more predominately than enamel margins. Regardless of the pulpal condition of a prepared tooth, it is important to protect dentin from factors that result in a poor prognosis. These include physical and chemical stimuli, microleakage, bacterial infection and the effect of bacterial by-products.

Studies have demonstrated that new dentin coating system and dentin bonding agents used to protect dentin surfaces can exhibit microleakage that extends into dentinal tubules.[6-8] The use of such agents for reducing sensitivity after crown preparation has also been shown to be an effective clinical treatment.[9-13] Some materials applied to coat the dentin surfaces may have adverse effects on the bond of luting agents. Teeth treated with a hydrophilic dentin primer system demonstrated a significant reduction in crown retention when polycarboxylate-based or zinc phosphate cements were used as the luting agents,[14] while the bond strength of crowns cemented to dentin pretreated with dentin bonding agents seemed to depend upon the combination of luting agent and dentin bonding agent.[15]

The purpose of this study was to evaluate the effect of the application of dentin coating material on microleakage and shear bond strength of luting agents to these surfaces.

Materials and Methods

The materials used in this study are presented in Table 1. Touch&Bond and Brush&Bond (Parkell, Farmingdale, NY, USA) were used as dentin coating materials. Both materials contain a 4-META molecule along with other co-monomers and are self-etching dentin bonding systems. Two adhesive resin cements, C&B Metabond (Parkell) and Panavia F (Kuraray Medical, Tokyo, Japan) were used as the luting agents. Dentin Activator (Parkell) with C&B Metabond and ED Primer (Kuraray Medical) with Panavia F were individually used as a post-treatment to coat dentin before luting.

Table 1. Materials used.

Material/Trade name	Component	Manufacturer	Lot number
Dentin coating material			
Brush&Bond	4-META, MMA, Polyacrylates	Parkell	L-200 200208, S-060
Touch&Bond	4-META, MMA, UDMA	Parkell	L-990006, S-990706
Cement			
C&B Metabond	4-META, MMA, TBBO	Parkell	P, ER1; Liq, EL1; Cat, EK22
Panavia F	MDP, Bis-GMA, Filler	Kuraray Medical	61175
Dentin conditioner			
Dentin Activator	Citric acid, Ferric chloride	Parkell	1091
ED Primer	HEMA, 5-NMSA, MDP	Kuraray Medical	A, 00148A; B, 00033A
Temporary cement			
Freegenol Temporary Pack	Zinc oxide, Rosin	GC Corp.	270351
Metal adhesive primer			
Alloy Primer	MDP, VBATDT	Kuraray Medical	0105AA

One hundred nineteen extracted bovine anterior teeth were used. Their roots and pulps were removed, and the orifices of pulp chambers were sealed with C&B Metabond after conditioning the dentin using Dentin Activator to prevent dye penetration and contaminant from that surface. The teeth were stored in 37°C distilled water for 24 hours. The labial surfaces of all tooth crowns were ground flat with a #400 silicon-carbide paper under water irrigation to expose dentin, and polished with a #600 grit paper.

The flow chart of specimen fabrications is presented in Fig. 1. One hundred five bovine teeth sealed with C&B Metabond were randomly selected for dye penetration testing and shear bond testing. The teeth were divided into three groups, including two dentin-coated (Touch&Bond and Brush&Bond) groups and a non-coated group of 35 specimens each to serve as a control. Group 1 was used for Touch&Bond. The material was applied to the ground dentin surfaces for 20 s, air-dispersed, and photo-polymerized for 5 s. The second layer of Touch&Bond was applied, gently air-dried and polymerized for 10 s. Group 2 was used for

Brush&Bond. The Brush&Bond was applied to the surface for 20 s, gently air-dried and photo-polymerized for 10 s. After the photo-polymerization, the superficial oxygen-inhibited layer was removed with an alcohol swab. Group 3 (control) received no dentin coating.

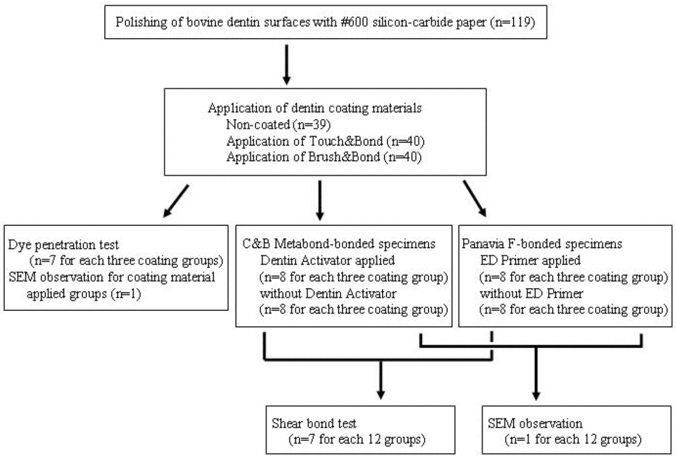


Fig. 1. Design for fabricated specimens.

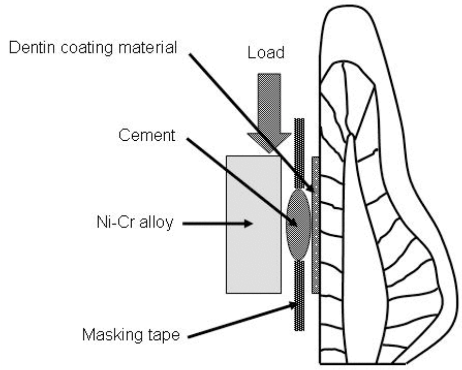


Fig. 2. Schematic illustration of shear bond test.

In addition, Group 1, 2, and 3 were subdivided into five groups of seven specimens each. One out of the five groups was subjected to a microleakage test and the remaining four were used for shear bond test.

To evaluate dye penetration evaluation the specimens for the microleakage test were immersed in a 5% solution of methylene blue at room temperature for 24 hours after the dentin surfaces were treated. They were then removed from the dye solution, received superficial dye removal, and allowed to dry at room temperature for one hour. The specimens were sectioned labio-lingually with a diamond disk and observed by a light microscope under 40x magnification. The dye penetration was measured at the five deepest points for each specimen. In short, the extent of dye penetration was determined at 35 points for each condition. The values for each specimen were averaged, and the data were statistically analyzed with analysis of variance (ANOVA), followed by Bonferroni/Dunn test at a 95% confidence level to compare individual groups.

To evaluate shear bond strengths the remaining 12 groups were used, including four groups each of two dentin-coated and non-coated conditions. For shear bond specimen fabrication the area to apply dentin coating materials was limited to a diameter of 10 mm with the use of masking tape. After coating, a temporary cement, Freegenol Temporary Pack (GC Corp., Tokyo, Japan), was then applied to the coated surfaces using a 10 mm diameter plastic plate. The specimens were then stored in 37°C water for 1 hour to set the temporary cement. After this water storage period, the temporary cement was mechanically removed and the surface was cleaned with an alcohol swab. The masking tape was placed onto the coated surface so that the adhesive area was limited to 5 mm in diameter.

Coated dentin specimens were randomly divided into two groups to be luted with C&B Metabond and Panavia F. In addition, C&B Metabond and Panavia F specimens were divided into two groups. The dentin conditioner, (Dentin Activator or ED Primer) was applied to the coated dentin as post-treatment for one group. The other group received no post-treatment. After the post-treatment, metal rods were luted with two resin cements to measure shear bond strengths. The metal rods (10 mm diameter and 3 mm height) were cast in a Ni-Cr alloy

(Rexillium II, Jeneric/Pentron, Wallingford, CT, USA). The disk surfaces were polished with a #600-grit paper, then air-abraded with 50 µm alumina. In the case of Panavia F, Alloy Primer (Kuraray Medical) was also applied to the metal surfaces after they had been air-abraded.

After each surface treatment, cast disks were cemented directly to coated dentin surfaces according to manufacturers' directions. Non-coated specimens served as a control group for each cement. After cement excess was carefully removed with a brush, the specimens were stored at room temperature for 1 hour. They were then placed in 37°C water and stored for an additional 24 hours before shear bond testing. The bond strengths were determined with a universal testing machine at 1.27 mm/minute crosshead speed (Fig. 2). Seven specimens were tested for each condition of shear bond testing. The mean values of each group were statistically analyzed with two-way ANOVA, with the conditions of dentin coating materials and post-treatments for respective cements as independent factors. Differences among the groups were analyzed by a Bonferroni/ Dunn test at a 95% confidence level.

After determining shear bond strengths, both tooth and metal surfaces were observed by scanning electron microscope (SEM) to analyze the failure patterns. Fourteen teeth were used for observation of cross-sectional surfaces by SEM. The 12 types of specimens for shear bond testing and two specimens which were only treated with each coating material to dentin were prepared for observation by SEM. Completed specimens were embedded with an auto-polymerized acrylic resin and cross-sectioned with a rotary diamond disk. They were then treated with 6 N hydrochloric acid (HCl) for 30 s and rinsed with distilled water to remove the inorganic phase. This is followed by 10% sodium hypochlorite (NaOCl) for 5 minutes to remove the organic components of the dentin using a technique described in the literature.[16] This permits better observation of the dentin coating materials that penetrated into dentinal tubules. Specimens were dried and gold/platinum sputtered for SEM observation.

Results

The results of dye penetration are presented in Table 2. The dye reached to the pulp chambers in all the control specimens. When dentin-coating materials were applied, the depths of dye penetration significantly decreased as compared to the control ($p<0.05$). The value of Brush&Bond specimens averaged 0.08 µm, while that of Touch&Bond specimens was 0.16 µm. There was no significant difference between Brush&Bond and Touch&Bond ($p>0.05$). SEM observations revealed that dentin surfaces were completely covered with a layer of each dentin coating material.

Table 2. Results of dye penetration for coating materials.

Dentin coating material	Dye penetration	SD (mm)	Statistical category
Non-coated (Control)	1.38	0.34	a
Touch&Bond	0.16	0.09	b
Brush&Bond	0.08	0.03	b

Statistical category: Identical letters indicate that they are not statistically different ($p>0.05$).

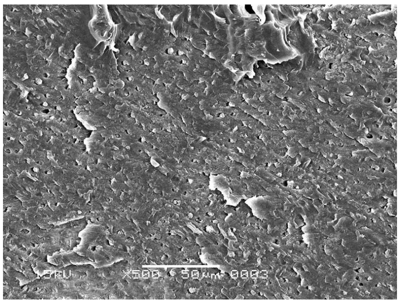
For shear bond strength measurement, the results of two-way ANOVA indicated that significant differences were found between coating materials ($p<0.0001$) and treatment to coated dentin ($p<0.0001$). In addition, significant interaction between coating materials and treatment to coated dentin ($p<0.0001$) were obtained. The results of shear bond testing and the destruction sites during the shear bond tests are presented in Table 3. Electron micrographs of failure site are presented in Fig. 3.

Table 3. Shear bond strengths, standard deviations in MPa, statistical categories, and failure modes.

	C&B Metabond								Panavia F							
	Without DA				With DA				Without ED				With ED			
	Mean	SD	SC	FM	Mean	SD	SC	FM	Mean	SD	SC	FM	Mean	SD	SC	FM
Non-coated	2.0	1.4	c	CD	32.4	5.6	f	CD	2.3	0.4	g	CD	10.0	2.4	h	CD
Touch&Bond	9.7	4.4	d	CC	11.4	2.8	d, e	CC	1.3	0.6	g	CC	14.3	2.6	h	CO
Brush&Bond	7.8	3.0	c, d	CC	16.0	1.4	e	CC	1.5	0.9	g	CC	21.7	5.1	I	CO

DA, Dentin Activator; ED, ED Primer, SD, Standard deviation.
SC, Statistical category; Identical lower case letters indicate that they are not statistically different (p>0.05). The C&B Metabond group and the Panavia group were not statistically compared in this table due to different surface preparation procedures.
FM, Failure mode; CC: failure occurred at the interface between cement and coating; CO: cohesive failure occurred at both cement and coating; CD, failure occurred at the interface between cement and dentin.

When the specimens were luted with C&B Metabond, the shear bond strengths (SBS) of coated specimens (11.4 MPa for Touch&Bond and 16.0 MPa for Brush&Bond) were significantly lower than Non-coated specimens (32.4 MPa) treated with Dentin Activator. These results are shown in Table 3 and existed despite the dentin coating material and/or post-treatment with Dentin Activator ($p<0.0003$). The Brush&Bond coated group treated with Dentin Activator demonstrated significantly higher SBS compared with the group without Dentin Activator ($p<0.0001$). Scanning electron micrograph of the destruction was presented Fig. 3-a when the specimen was luted with C&B Metabond after applying Dentin Activator. This photograph illustrates the exposed dentin surface. When Touch&Bond or Brush&Bond was applied to dentin surfaces, the failure mainly occurred at the cement/coating interface (Fig. 3b). Dentin surfaces were covered and protected with a layer of each dentin coating material.



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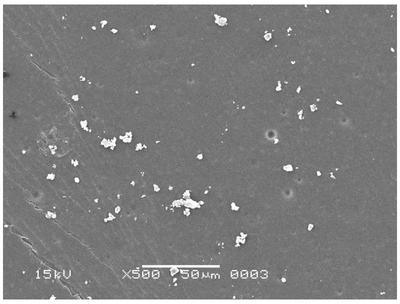

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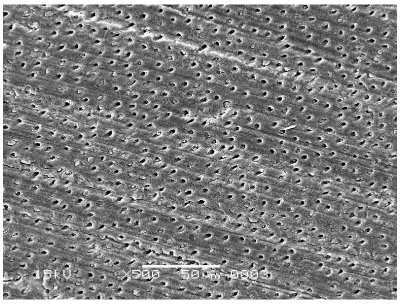

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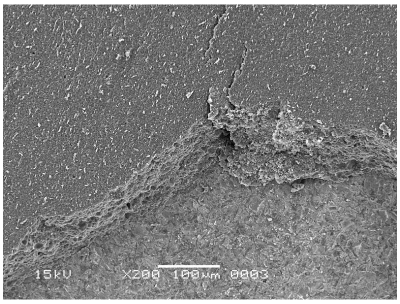

3d

Fig. 3a.
Debonded dentin surface of C&B Metabond-bonded specimen pretreated with Dentin Activator. Debonding occurred dentin/C&B Metabond interface.
Fig. 3b.
Debonded dentin surface of C&B Metabond-bonded specimen coated with Brush&Bond followed by pretreated with Dentin Activator. Debonding occurred at C&B Metabond/Brush&Bond interface.

Fig. 3c.
Debonded dentin surface of Panavia F-bonded specimen pretreated with ED Primer. Debonding occurred at dentin/Panavia F interface.
Fig. 3d.
Debonded Ni-Cr alloy surface of Panavia F-bonded specimen coated with Brush&Bond followed by pretreatment with ED Primer, showing cohesive/adhesive failure.

When ED primer was not applied to coated surfaces before cementing, SBS of Panavia F ranged from 1.3 to 1.5 MPa, and specimen failure occurred entirely at the cement/coating interface. However, when Brush&Bond

coated surfaces were treated with ED Primer, the SBS of Panavia F significantly increased compared to the control with ED Primer ($p<0.0001$) as shown in Table 3. The failure of control occurred at cement/dentin interface (Fig. 3c). The failures of the Brush&Bond coated specimens with ED Primer mainly occurred at the cement/coating interface and partially at the coating/dentin interface. For Brush&Bond/ED Primer/Panavia F, the failure at the coating/dentin interface showed a greater area (Fig. 3d) compared to the groups of Touch&Bond/ED Primer/Panavia F.

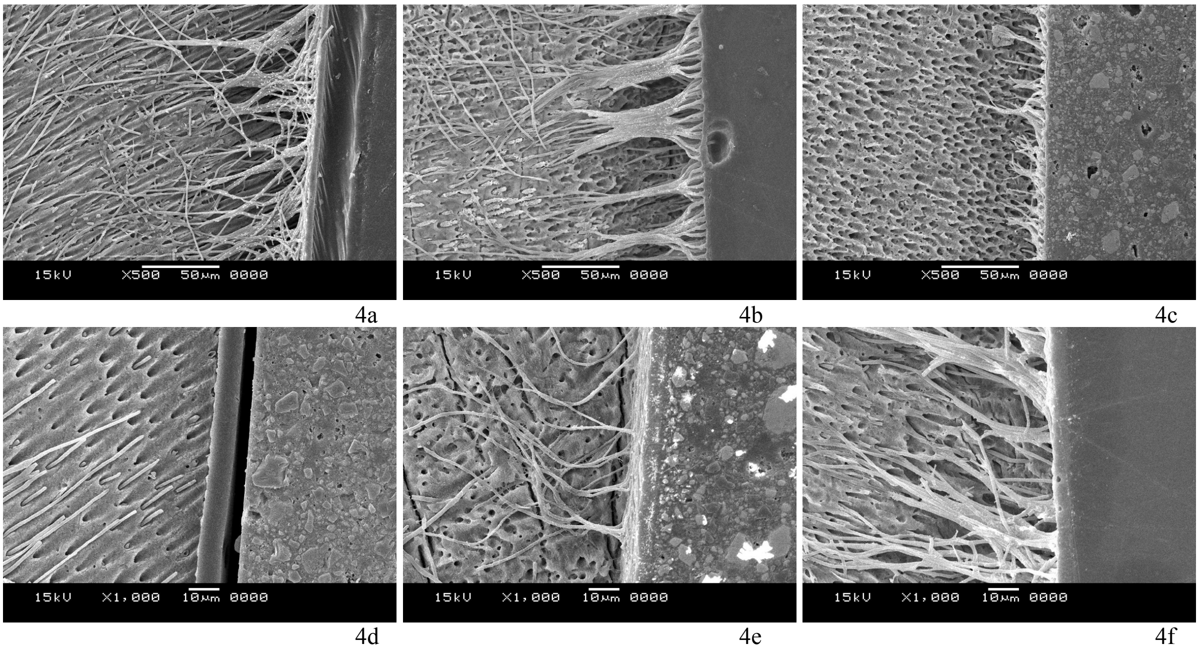

Fig. 4. Cross-sectional surfaces: **4a,** Brush&Bond; **4b,** C&B Metabond with Dentin Activator; **4c,** Panavia F with ED Primer; **4d,** Panavia F without ED Primer on Brush&Bond coated dentin surface; **4e,** Panavia F with ED Primer on Brush&Bond coated dentin surface; **4f,** C&B Metabond with Dentin Activator on Brush&Bond coated dentin surface.

Cross-sectional micrographs are presented in Fig. 4. The SEM for Brush&Bond (Fig. 4a) and Dentin Activator/C&B Metabond (Fig. 4b) showed longer and greater numbers of resin tags compared to ED Primer/Panavia F (Fig. 4c). When specimens were cemented to the coated surfaces with Panavia F without ED Primer, crack formation was observed between cement/coating interface (Fig. 4d). However, cracks could not be seen when ED Primer was used (Fig. 4e). When specimens were luted to the coated surfaces with C&B Metabond, cracks could not be found regardless of the treatment of Dentin Activator (Fig. 4f).

Discussion

Sound tooth enamel has an inherent capacity to resist unfavorable stimuli. When dentin is exposed during dental treatment, the danger of pulpal involvement increases as the tooth becomes more susceptible to bacterial infection and other problems. Therefore, dentin protection plays an important role in maintaining the health of restored teeth. The results of the microleakage test demonstrated that the dentin coating materials used in this study were successful in protecting exposed dentin from dye penetration. This suggests that these materials could be effective in protecting exposed dentin from unfavorable stimuli in the clinical situation and acquiring good prognoses. The results of the shear bond tests demonstrated that the failure pattern of bonded luting agents was material or post-treatment dependent. The variation of shear bond strengths according to procedures was illustrated in Table 3.

The SBS of tested resin cements appeared to be affected by pretreatment of the coated surface and the cement used. The coated specimens demonstrated significantly lower SBS compared to non-coated specimens when C&B Metabond was used. The specimens failed in shear predominantly at the cement/coating interface. This might be due to the weak bond of the cement to the coated surface. Higher bond strength might be expected if a superficial unpolymerized layer existed on the coated surface; however, in this study, the oxygen-inhibited layer was completely removed to better simulate clinical conditions. The surface was not only swabbed with alcohol but also further contaminated by the use of temporary cement. Therefore, the improvement of a coated surface appears to be the key to achieve better bonding. Shear bond strengths were improved by applying Dentin Activator to the coated surfaces compared to untreated specimens, especially those coated with Brush&Bond. It is speculated that there are two possibilities for this improvement. The first is the cleansing effect of citric acid, and the other is promotion of polymerization by ferric chloride in the Dentin Activator for C&B Metabond.[17] However, it is suspected that bond strengths between C&B Metabond and coating surface are low, as the failure occurred in cement/dentin coating materials in the micrograph (Fig. 3b). C&B Metabond could hardly be observed on the coated surface.

When the specimens were bonded using Panavia F, SBS was dramatically improved by applying ED Primer onto the Brush&Bond coated surfaces as compared to that of no post-treatment. It is speculated that interfacial polymerization of Panavia F was enhanced by the catalyst in ED Primer. The failure can be seen at coating material/dentin interface. From this result, it is suspected that ED Primer has a capacity to generate a chemical bonding between coating material and Panavia F. In fact, Panavia F exhibited very poor bond strength to coated surfaces when they were not treated with ED Primer. This poor performance might be attributed to interfacial separation between cement and dentin coating materials (Fig. 4d) due to polymerization shrinkage of Panavia F. Therefore, the post-treatment using ED Primer is essential when Panavia F is used.

When considering dentin coating as a protection for exposed dentin during provisionalization, the site of debonding becomes a critical issue. It is better to have the debonding at the coating/cement interface rather than at the coating/dentin interface. In that way, even if the bond fails, dentin remains protected by the adhesive coating. Shear bond strength of the resin cements significantly improved by using dentin conditioner. The balance of bond strength to dentin substrate is important, as the interfaces could possibly be deteriorated over time in clinical situations due to masticatory stresses.[18] In the present study, no attempts of stress-fatigue were made to the interfaces. Further investigations are suggested.

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