

Tensile and shear bond strengths of a stainless steel used in orthodontic brackets bonded to bovine enamel using two types of resin cement

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The purpose of this study was to examine the relationship between the bond strength of stainless steel with two types of resin cements (MMA- and composite-based) on bovine enamel depending on the directionality of the applied force. The specimens were either placed in water or subjected to thermal cycles (TC), and the shear or tensile bond strengths (SBS or TBS) were determined. The SBS showed significantly greater than the TBS for both types of cement, and the SBS and TBS for composite-based cement had larger than MMA-based one. No significant difference in SBS was observed in the cements even after being subjected to TC. Cohesive failures of the cement and bovine enamel in the composite-based group, while adhesive failures were observed in MMA-based one. Consequently, the direction of the force at both cements affected the retention of stainless steel, and MMA-based cement was preferred when prioritizing less enamel damages.

Keywords: Adhesive resin cement, Orthodontic bracket, Thermal cycle, Bond strength, Stainless steel

INTRODUCTION

In multi-bracket systems used for orthodontic treatment, brackets are attached to the enamel surface with adhesive resin cement to transmit orthodontic forces from the wire and enable three-dimensional tooth movement. There are two methods of attaching brackets to the enamel surface: the direct method developed in the 1960s^{1,2)} and the indirect method in recent years³⁾. However, bracket materials have changed over time from precious metals to stainless steel, and this transition was due to Dr. Edward Angle's invention of the edge-wise appliance in 1925. Technological advancement and a better understanding of science have facilitated the development of materials that are used nowadays that reduce metallic allergies and improve esthetic appearances. Ceramics, such as alumina and zirconia, and polymers, such as polyethylene terephthalate and polyurethane, are available as esthetic bracket options. Ceramic brackets are brittle with high frictional resistance to wires, while polymer brackets have low strength. Thus, metallic brackets with superior mechanical strength and low frictional resistance with wires are often preferred over aesthetic brackets.

Following acid treatment at enamel surface, multi-bracket appliances are placed on the tooth surface using adhesive resin cement. The appliances are exposed to external factors such as orthodontic forces and temperature changes and are not removed for several years during treatment. Appliance bond strength must be able to withstand not only acting forces on the teeth from the external environment but also be easy enough to remove after treatment without damaging the enamel⁴⁻⁶⁾.

Various types of adhesive resin cement have been developed to bond prosthodontic restorations to abutment materials such as dentin, resin composite, and silver alloys. When the abutment material is dentin, a decline in retention and bond strength from humidity and aging is expected and must be accounted for. However, the bond strength of adhesive resin cement to enamel has been reported to be adequate due to lower water and organic contents in dentin^{7,8)}. *In-vitro* tests have been used to evaluate the effects of oral temperature and water content on the bond strength and retention of multi-bracket appliances with adhesive resin cement⁹⁻¹¹⁾.

During orthodontic treatment, forces applied to multi-bracket appliances are shear forces parallel to the tooth surface and tensile forces perpendicular to the tooth surface during removal¹²⁾. Methods for bonding brackets of different compositions to the tooth structure and crown restorative materials have been evaluated^{9,11,13,14)}; however, there are limited studies that depict the relationship between bond strength and the direction of the force applied on the bracket or assess damage to the tooth surface during removal.

The objective of this study was to determine the bond strength of multi-bracket appliances on tooth surfaces depending on the direction of the applied force and the resulting damage to the tooth surface. This study used two types of orthodontic adhesive resin cement to attach multi-bracket appliances to respective tooth surfaces. Shear and tensile bond strengths were evaluated because similar multi-bracket appliances are subjected to shear forces during treatment and tensile forces during removal. Accelerated degradation tests using thermal cycling were performed to determine the effects of material aging on bond strength. The null hypotheses

were as follows: (1) the type of adhesive resin cement does not affect the bond strength of stainless steel as an alternative to bracket, (2) there is no difference between the shear and tensile forces that are applied to stainless steel, and (3) the bond strength between stainless steel and adhesive resin cement is not affected by accelerated aging tests using thermal cycling.

MATERIALS AND METHODS

Preparation of specimen

Eighty bovine mandibular anterior teeth were cut at the cement-enamel junction, and the crowns were used. Each crown was embedded in an epoxy ring (EX ring, 1-inch, Refine Tech, Kanagawa, Japan) with auto-polymerized epoxy resin (Scandiplex, Fritsch Japan, Kanagawa, Japan). After the resin was cured, the bovine enamel surface was polished with a series of abrasive paper to 600 grit to prepare a flat enamel surface. Use of bovine teeth for bond test were approved by the Institutional Ethics Committee of Iwate Medical University (approval number: #02-004 and #04-002).

In the preliminary experiment, the composition of a stainless steel bracket was confirmed to be equivalent to SUS304 as shown in Table 1; therefore, stainless steel (StSt: SUS304) was used for this study. An StSt rod (SUS304: MEGASUS, Hyogo, Japan) with a diameter of 4.0 mm was cut into 2.5 mm thickness for the shear test and 6.2 mm for the tensile test. The end surfaces were evened using a lathe. The lathed surface was blasted with 50 μm alumina particles (Hi-alumina, Shofu, Kyoto, Japan), ultrasonically washed with acetone (Acetone, FUJIFILM Wako Pure Chemical, Osaka, Japan), and distilled water for 10 min each, and prepared for bonding to bovine enamel.

Two types of adhesive resin cement were used in this study: a methyl methacrylate (MMA)-type (MCP: MCP bond, Sun Medical, Shiga, Japan), and a composite-type

(UB: Universal bond UB, GC Orthory, Tokyo, Japan). The composition and code are listed in Table 2. The StSt specimens were bonded to bovine enamel using resin cement according to the manufacturer's instruction. For MCP specimens, the bovine enamel surfaces were etched (35% phosphoric acid, Surface reactor red, Sun Medical) for 30 s, washed with tap water, and air-dried. The poly(methyl methacrylate) powder, MMA liquid, and catalyst were mixed using a brush, and the paste was applied to the StSt specimen and pressed onto the etched bovine enamel substrate. The excess cement was removed, and the specimen was left for 6 min. For UB specimens, the bovine enamel surface was etched using etchant (GC Orthory) for 30 s, resin cement was applied to the StSt specimens, then pressed to the bovine enamel substrate and cured by the light irradiation (PEN Bright, Shofu) for 20 s from two directions. All specimens were immersed in distilled water at 37°C for 7 days. For the shear test, 40 specimens were subjected to thermal cycles to investigate the effects of aging on the bond strengths of the resin cement. Accelerated aging was implemented by putting the specimens through 5 or 10 \times 10³ thermal cycles of alternate immersion in 5°C and 55°C water for 30 s each.

Bond strength

Bond strengths were evaluated using shear and tensile tests on a universal test machine (EZ-LX: SHIMADZU, Kyoto, Japan). For the shear test, a load was applied parallel to the bonded surface at a cross-head speed of 1.0 mm/min, and the shear bond strength (SBS) was calculated from the maximum load. For the tensile test, a load was applied perpendicular to the bond surface at a cross-head speed of 3.0 mm/min until the StSt specimens detached from the bovine enamel substrate. The tensile bond strength (TBS) was calculated from the maximum load.

After the shear and tensile tests, the fractured

Table 1 The composition of one metal brackets and SUS304 used in this study, determined by FE-EPMA*¹ (mass%)

	Fe	Cr	Ni	Mn	Si	Others (<0.5 mass%)
Metal bracket* ²	70.5	18.7	8.3	1.4	0.6	Cu, Mo
Specimen (SUS304)	69.3	20.8	7.2	1.7	0.5	Cu

Standard error: ± 1.5 mass% for Fe, ± 0.7 mass% for Cr and Ni, ± 0.3 mass% for Mn

*¹ field emission electron probe microanalyzer (JXA-8530F, JEOL, Tokyo, Japan), *² Metal Bracket (Dentsply Sirona, Tokyo, Japan)

Table 2 Code, category, and composition of adhesive resin cements used in this study

Code	Category	Product (Company)	Composition
MCP	MMA-type	MCP bond (Sun Medical)	methyl methacrylate, aromatic amine, 4-methacryloxyethyl trimellitate anhydride (4-META), poly (methyl methacrylate), benzoic peroxide (BPO)
UB	Composite-type	Universal bond UB (GC Orthory)	methacrylate ester, glass filler, phosphoric ester monomer (MDP), initiator (photo polymerization)

surfaces of all specimens were observed with a digital microscope (UM12, MicroLinks Technology, Kaohsiung, Taiwan) to classify fracture modes. The fracture modes were classified into four categories: category 1 was a cohesive failure of resin cement, category 2 was an adhesive failure at the interface between resin cement and bovine enamel, category 3 was a mixed failure of cohesive and adhesive failures observed in approximately equal proportions, and category 4 was a cohesive failure of the bovine enamel. Typical surfaces representing cohesive and adhesive fractures were observed using a field-emission scanning electron microscope (FE-SEM; SU8010, Hitachi High-Technologies, Tokyo, Japan) with an accelerating voltage at 10 kV. The specimens were coated with osmium using OsO₄ plasma in an osmium coater (Osmium Plasma Coater OPC60A, Filgen, Aichi, Japan).

Statistical analysis

The bond strengths were statistically analyzed using a statistical software (Bell curve for excel, Social Survey Research Information, Tokyo, Japan). First, the normality of the bond strength was tested using the Shapiro-Wilk test. Then a two-way analysis of variance (ANOVA) was followed by Tukey's multiple comparison tests based on the cement type and bond strength test method. The SBS after the accelerated aging were statistically analyzed by two-way ANOVA followed by Tukey's multiple comparison test based on cement type and the number of thermal cycles. The fractured modes after the bond test were analyzed by χ^2 and residual tests. The significant level in all tests was set at 5% ($\alpha=0.05$).

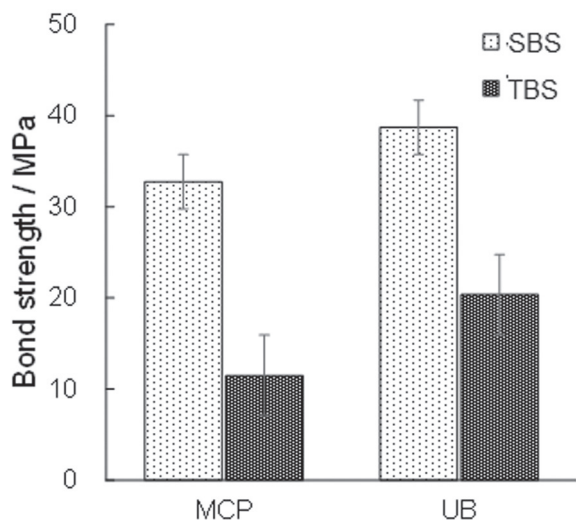


Fig. 1 Bond strengths of StSt specimens on bovine enamel with two-types of adhesive resin cement (MCP and UB) via shear and tensile tests.

RESULTS

Bond strengths

The SBS and TBS (mean±standard deviation) of StSt specimens bonded to bovine enamel with MCP and UB are shown in Fig. 1. For MCP, the mean values for SBS and TBS were 32.8 and 11.5 MPa, respectively, while mean values for SBS and TBS were 38.7 and 20.4 MPa, respectively. The TBS was 30–50% lower than the SBS for both types of cement. The Shapiro-Wilk test showed normality in all groups. Two-way ANOVA and Tukey's multiple comparison test showed significant differences between the bond strengths of the resin cement for each testing method ($p<0.05$), indicating that UB had larger SBS and TBS than MCP.

The change in SBS after thermal cycling for each adhesive resin cement is shown in Fig. 2. Two-way ANOVA revealed no significant differences in bond strength based on the interaction or the number of thermal cycles ($p>0.05$); however, a significant difference was detected between the types of cement ($p<0.05$).

Observation of fractured surface

Figure 3 shows the typical digital microscopic images showing cohesive failure of resin cement (category 1) and adhesive failure between bovine enamel and StSt specimen (category 2). The category 1 in fracture mode was that residual resin cements were observed on both the bovine enamel and StSt specimen (Figs. 3a and b). Whereas the cement was not observed on the bovine enamel, the residual resin cement was observed on the opposite of StSt specimen (Figs. 3c and d), and then categorizing to category 2. The classification of fracture modes is shown in Fig. 4. In the shear bond test, fracture modes under categories 1 and 3 were observed for specimens bonded with MCP, while only category 1 was observed for specimens bonded with UB (Fig. 4a). In the

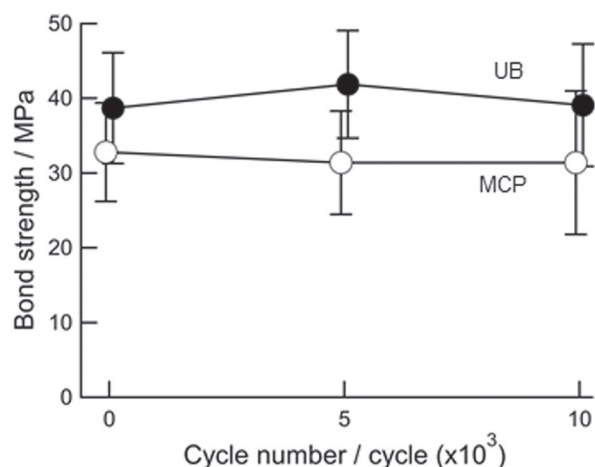


Fig. 2 Changes in shear bond strength of StSt specimens on bovine enamel with two-types of adhesive resin cement (MCP and UB) by thermal cycling (5×10^3 and 10×10^3).

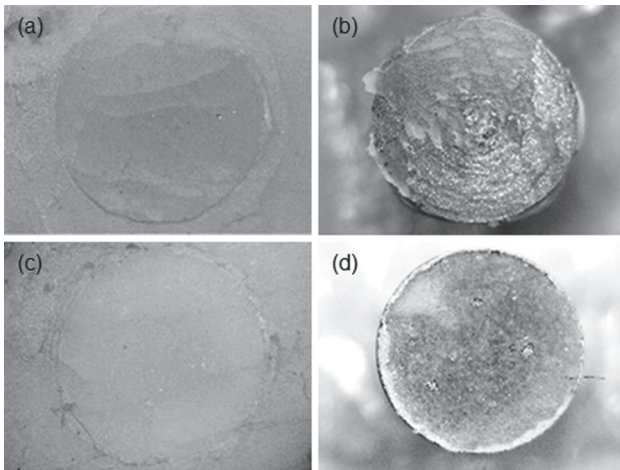


Fig. 3 Typical digital microscopic images after shear bond test of cohesive failure of bovine enamel (a) and opposite StSt specimen (b), and adhesive failure of bovine enamel (c) and opposite StSt specimen (d).

tensile bond test, fracture modes of all categories were observed for MCP, while mostly category 1 was observed for the UB. When the specimens were subjected to thermal cycling, there was a decrease in the proportion of category 1 with an increase of category 3 for MCP ($p < 0.05$, χ^2 and residual tests). Mainly category 1 was observed for the UB even after thermal cycling (Fig. 4b).

Typical SEM images of the fractured surface of the bovine enamel and StSt specimens bonded to UB and MCP after 7-day immersion and TC are shown in Fig. 5. Cohesive failure of resin cement was observed on both bovine enamel and StSt specimen for MCP and UB after 7-day immersion in water, which displayed similar microscopic images (Figs. 5a, b, d and e). In specimens that showed adhesive failure at the interface between bovine enamel and cement, enamel rods were observed on the bovine enamel (Fig. 5c), and mirrored shapes of enamel rod-like structures were observed in the resin cement of the StSt specimens for MCP (Fig. 5f).

DISCUSSION

Test methods

Multi-bracket appliances transmit force from the wire to the tooth leading to mesiodistal or intrusive-extrusive movements. The transmitted force is a shear force parallel to the adhesive tooth surface. Furthermore, when removing the multi-bracket appliances at the end of treatment, a load perpendicular to the adhesive surface is applied. When the direction of the force applied to the multi-bracket appliance was considered, the bond strength was smaller when the force applied was perpendicular to the tooth surface (brackets removal force) than when the force applied was parallel (orthodontic forces)¹⁵. Therefore, this study conducted shear and tensile tests to examine bond strengths with

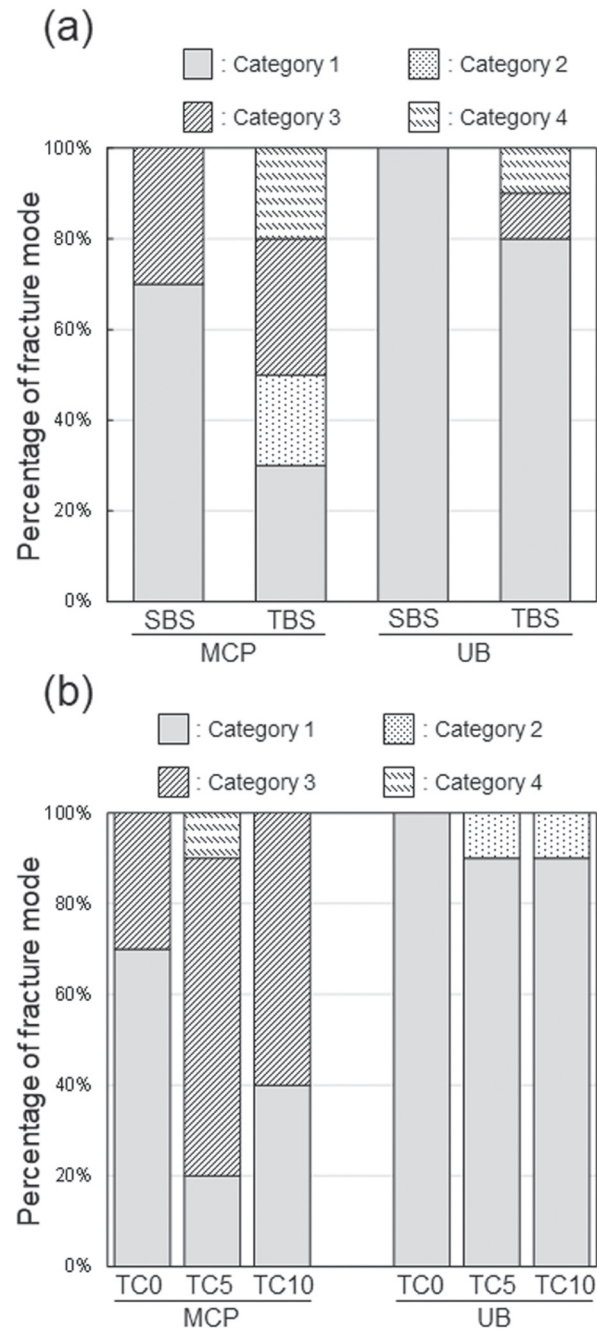


Fig. 4 Classification of failure mode after shear and tensile bond tests bond to two-types of adhesive resin cement (MCP and UB) (a), and after thermal cycling test (b).

horizontal and vertical loads on the tooth surface.

In this study, StSt specimen to SUS304, was used instead of the actual metallic brackets because of its similar composition as listed in Table 1. The bracket base is shaped to achieve optimal interlocking force between the cement and tooth surface, and this shape affects the shear stress acting on the boundary between the structures^{12,16,17}. In other words, it is difficult to

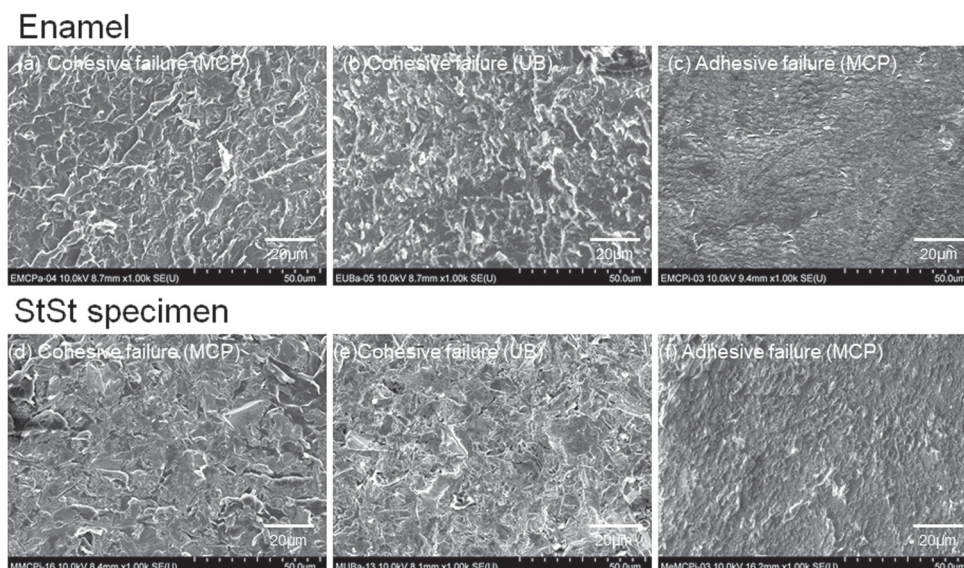


Fig. 5 Typical SEM images of cohesive failures in resin cement and adhesive failure between enamel and stainless steel via shear bond test. (a) cohesive failure of MCP on bovine enamel and (d) on opposite StSt specimen for 7-day immersion in water, (b) cohesive failure of UB on bovine enamel and (e) on opposite StSt specimen for 7-day immersion in water, (c) adhesive failure of MCP on bovine enamel and (f) on opposite StSt specimen after TC

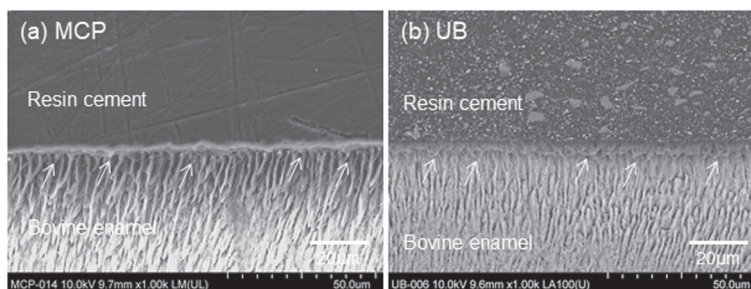


Fig. 6 Cross-sectional SEM images of interface between bovine enamel and resin cement. White arrows indicate resin tag in enamel. (a) MCP, (b) UB

unidirectionally determine the bond strength of bracket because the adhesive surface area and curvature vary depending on the tooth the appliance is placed. Therefore, in this study, rod-shaped specimens of SUS304 were used to simplify the testing process.

The substrate used in this study was bovine mandibular anterior teeth. The structure and composition of bovine enamel are similar to human enamel^{16,18}, and reports have suggested no significant difference in bond strength between the two¹⁹. Since the surface roughness of adherents is known to affect bond strengths²⁰, the bovine teeth used in this study were polished to 600-grit with water-resistant abrasive paper and acid-etched with phosphoric acid. Therefore, the bond strengths derived in this study were considered similar tendency to the bond strengths between the human enamel and multi-bracket appliances made of a stainless steel.

Shear and tensile bond strengths

StSt specimens were bonded to bovine enamel using MMA-based and composite-based resin cements, and the bond strengths were determined using shear and tensile tests. In both the tests, UB showed higher bond strength than MCP. This result is consistent with previous reports on the bond strength of various adhesive resin cement²¹. Moreover, the SBS was significantly greater than the TBS for both types of cement. Therefore, the null hypotheses that (1) the type of resin cement does not affect the bond strength of StSt that (2) there is no difference between the shear and tensile forces applied to StSt were rejected.

For fracture modes, adhesive failure between the bovine enamel and cement was observed in the MCP, but cohesive failures of the cement were observed in the UB. Factors that may have influenced these results include

the strength of the cement and adhesive monomers and the effects of pretreatment^{20,22}. Since the composite-based cement contains inorganic fillers as a reinforcing material, it possesses a higher elastic modulus and strength than the MMA-based cement²⁰. In this study, resin tags confirmed the effect of acid-etching on the cross-sectional SEM images of both the cements (Fig. 6). The phosphoric acid used for pretreatment makes the bovine enamel surface rough, and the cement applied to the rough bovine enamel surface becomes mechanically interlocked. In addition, the types and concentrations of adhesive monomers in each cement are varied. The UB was applied as a paste, and the MCP was applied in a powder-liquid form using the brush stacking method. The flowability and adhesive monomer penetration differed between the UB as paste and the MCP as powder/liquid, but no significant difference was observed in the formation of resin tags. When the failure mode is cohesive failure, the thickness and strength of the cement is known to affect the bond strength. In the literature, the effects of cement strength on the bond strength reported to increase as the cement thickness increases^{17,23}. Since the composite-based cement, UB, is higher compressive strength than the MMA-based cement, MCP, the strength of the resin tag was higher, and the resulting bond strength between the cement and bovine enamel was also significantly greater. That is, the horizontal force was applied to the bovine enamel surface, the larger the strength of cement, UB, by itself could be caused the greater the resistance to the force. The resin penetration into the rough bovine enamel surface after etching and the strength of the cement itself influenced the bond strength.

In this study, the bovine enamel surface was roughened by acid-etching, but the resin tags were almost vertical to the surface layer (Fig. 6). Since the direction of the load applied during the shear test was perpendicular to the resin tag and more resistant compared to the load applied during the tensile test, which is parallel to the resin tag, the SBS was significantly higher than TBS.

Accelerating aging test

Adequate bond strength of multi-bracket appliances is required in the oral cavity to withstand orthodontic forces, temperature changes, humidity, and mastication. There are concerns for resin-based materials that their functions are declined due to deterioration caused by water absorption²⁴. This study conducted an aging test using thermal cycles to accelerate the deterioration of the adhesive materials.

Ten-thousand thermal cycles corresponded to one year of stress in the oral environment^{25,26}, and previous reports indicated that the bond strength decreased between 7,000 to 10,000 cycles²⁶, that is, the thermal cycle may replicate the degradation of adhesive materials over a one-year period in oral cavity. Therefore, the SBS was evaluated in this study after subjecting the specimens to 5 and 10×10^3 thermal cycles. As a result, no decrease in the bond strength was observed for both resin types of cement even after 10×10^3 thermal cycles.

Therefore, the null hypothesis (3) was accepted that the bond strength between stainless steel and adhesive resin cement is not affected by accelerated degradation tests using thermal cycles.

As shown in Fig. 2, the bond strength of UB showed large values even after thermal cycling. After bond tests, the adhesive resin cements were observed on partially or whole surfaces of StSt specimens, indicating that the adhesive resin cements were adequate bonding to stainless steel. Analysis of the fracture surface indicated that in the early stages of thermal cycling (after 7 days of immersion), the rates of adhesive and mixture failures were higher in the MCP than in UB. Furthermore, the fracture surface of specimens after thermal cycling and shear test showed that the proportion of adhesive failure increased in MCP (Fig. 4). The adhesive failure occurred between the bovine enamel and cement and not between the cement and StSt specimen. Since it is a well-known fact that deterioration occurs in resin-based materials after water absorption²⁴, the effects of thermal cycling were more significant on the fracture surface than on the bond strength. In other words, the bond strength between the resin cement and StSt was greater than that of resin cement and bovine enamel. The cement's resin components (adhesive and polyfunctional monomers) were hydrolyzed and caused deterioration at the enamel-cement interface.

The results showed that the 10×10^3 thermal cycles undertaken in this study did not affect the bond strength of resin cement. If the thermal cycling at 10×10^3 cycles indicates a period of 1 years, the bond strength of resin in the oral cavity is adequate against environmental impacts such as humidity and mastication.

Clinical implication

Orthodontic appliances, such as bracket and band, require a bond strength that can withstand orthodontic forces during treatment but can be easily removed after treatment without damaging the enamel. The adhesive surface of the multi-bracket appliances comes in various designs to improve the mechanical interlocking force of the resin cement^{12,17}. In addition, since multi-bracket appliances are curved to fit the tooth surface, the cement is expected to have sufficient strength and bond strength. In this study, there was no adhesive failure between the cement and StSt specimen, indicating that all adhesive relationships were between the resin cement and enamel.

The fracture modes after the tensile test showed a cohesive failure of the bovine enamel in some specimens when the composite-type resin cement was used. Since tensile stress occurs during removal, this outcome indicates the possibility of enamel damage during the removal of multi-bracket appliances. The composite-type resin cement, UB, used in this study was a light-cure type cured by light irradiation. Recently, there have been efforts to develop high-power light irradiators to shorten the curing time even more, so the setting time is to be controlled easy. Since the MMA-type cement takes longer to cure by chemical polymerization, the

composite-type cement is used more often for bonding multi-bracket appliances due to its efficiency.

After treatment completion, the multi-bracket appliances are removed from the tooth surface using resin-removing pliers and burs. Since the tooth structure may be damaged during removal, there have been efforts to improve the multi-bracket appliances base and resin cement to minimize the damage⁷⁾. From the results of this study, the bond strength of MMA-type and composite-type cements to the tooth structure is sufficient for orthodontic treatment. The bond strength of composite-type resin cement was higher than that of MMA-type resin cement, and it bonded firmly to the bovine enamel, demonstrated by the number of cohesive fractures. However, large bond strength between the composite-type resin cement and human enamel during orthodontic treatment may be damaged to the enamel when multi-bracket appliances are removed. In addition, cement removal on composite-type resin cement takes longer than the MMA-based resin cement, which displayed more adhesive failure, and since the resin cement remained on the tooth surface, the risk of human enamel damage during removal was greater²¹⁾. The chair time and usability during placement and removal of the multi-bracket appliances should be the criteria when selecting the final adhesive material.

CONCLUSIONS

In this study, two types of orthodontic adhesive resin cement (MMA-based and composite-based) were used to bond StSt the metal used to fabricate multi-bracket appliances, to bovine enamel and evaluated using shear and tensile tests. As a result, the following conclusions were obtained.

1. The SBS and TBS of UB was greater than those of MCP.
2. The SBS was greater than the TBS in both types of resin cement.
3. When bonding with MCP, adhesive failures were observed after tensile test. For UB, cohesive failures of the bovine enamel and resin cement were observed after the tensile test.
4. There was no decrease in bond strength for both types of resin cement after the accelerated aging test involving 10×10^3 thermal cycles. However, the proportion of adhesive failures increased when bonding with MCP.

Based on the findings, when bonding StSt brackets on the human enamel surface, in addition to the resin cement's usability and initial bond strength, it is important to consider the ratio of residual cement and damage to enamel at the removal.

ACKNOWLEDGMENTS

This study was supported by the following: a grant from Japanese Orthodontic Society, a grant from the JSPS KAKENHI Grant Number 20K10015 (S.T.). The SEM observation were performed in Technical Support Center

for Life Science Research, Iwate Medical University. This work was performed under the Inter-University Cooperative Research Program of the Institute for Materials Research, Tohoku University (Proposal No. 202012-CRKEQ-0027 and 202112-CRKEQ-0021). The authors would like to thank Mr. Issei NARITA from Institute for Materials Research, Tohoku University for supporting measure of composition of brackets and stainless steels using EPMA.

CONFLICT OF INTEREST

The authors have no conflict of interest.

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