Development of an Easy-debonding Orthodontic Adhesive using Thermal Heating

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We produced experimentally a new bonding material that consisted of a mixture of a base resin (4-META/MMA-TBB resin adhesive) and thermoexpandable microcapsules for safe, easy debonding. Microcapsules in the base resin would start expansion at 80℃, leading to a remarkable decrease in bond strength. Stainless steel brackets were bonded to bovine permanent mandibular incisors using bonding materials containing the microcapsules at different contents. After thermal cycling or heating, the shear bond strength of the brackets was measured. Shear bond strength of the bonding materials containing 30−40 wt% microcapsules decreased to about one-third or one-fifth that of the base resin on heating. Heating the brackets for eight seconds increased the temperature in the pulp chamber by 2℃, which should not induce pulp damage.

Results obtained suggested that the new bonding material should prove useful for removing brackets easily at the time of bracket debonding without any pain or enamel cracks, while maintaining the bonding strength during active orthodontic treatment.

Keywords : Debonding, Orthodontic adhesive, Microcapsules

INTRODUCTION

Recently, many bonding materials have been developed for use in orthodontic treatment. The improvement of orthodontic adhesives, etching agents, and primers allows the bonding strength to remain stable in all situations. Studies showed that the shear bond strength of orthodontic brackets bonded with recent bonding materials exceeded 6-8 MPa, thereby rendering sufficient strength for orthodontic treatment[1-4]. This guideline was based on a report by Reynolds whereby a minimum bond strength of 5.9-7.9 MPa allowed satisfactory clinical performance and successful clinical bonding in orthodontic treatment[5].

Although it is important that the brackets used for orthodontic treatment be strongly bonded so that they do not detach, excessive bond strength can sometimes cause pain in the tooth or enamel cracks when the brackets are removed at the end of the active treatment[6-8].

To reduce the pain and clinical incidence of irreversible enamel surface damage, several methods of debonding brackets have been suggested. These methods include debonding brackets using ultrasonic instrumentation[9,10], electrothermal heating[11-13], or laser irradiation[14-16]. However, there are some problems with these methods. Debonding using ultrasonic instrumentation is slow[10]; debonding using electrothermal heating does not decrease the bond strength sufficiently; and laser irradiation is expensive. Consequently, we experimentally produced an adhesive comprising a mixture of base resin and thermoexpandable microcapsules for easy debonding.

Microcapsules were used because they expand in the adhesive on heating, such that the physical bonding properties of the adhesive will be markedly reduced.

This study examined the decrease in bonding strength on heating of orthodontic brackets bonded using an adhesive resin containing thermoexpandable microcapsules. We also tested bond durability and temperature rise in the pulp chamber on heating for clinical applications of this bonding material.

MATERIALS AND METHODS

Bonding materials for easy debonding
The base bonding material used in this experiment was 4-META/MMA-TBB resin adhesive (Orthomite SuperBond, Sun Medical, Tokyo, Japan). This adhesive consisted of both polymer and monomer components, whereby the polymer mixed easily with microcapsules to produce particles of uniform size. While SuperBond kept to a minimum the risk of enamel cracks−being more flexible than other orthodontic resin adhesives, the bond strength of brackets bonded with SuperBond was high enough to cause pain on the teeth when the brackets were removed[6,7]. With a view to decreasing the bond strength, different amounts of thermoexpandable microcapsules (Matsumoto Microsphere F-36D, Matsumoto Yushi-Seiyaku, Osaka, Japan) were mixed with the base bonding material.

The microcapsules used in this study had particle sizes of 10-20 μm, and expansion started at a temperature of 80℃. Maximum volumetric expansion was about 70 times. Microcapsules in the content

range of 10 to 40 wt were mixed with Orthomite SuperBond polymer and used for the experiments.

Features of thermoexpandable microcapsules
Thermoexpandable microcapsules are harmless within the human body and expand 4-5 times in diameter (*i.e.*, the volume increases by 50-100 times) on heating. The expanding agents they contain are volatile organic compounds such as isobutene, pentane, petroleum ether, hexane, heptane, low-boiling-point halogenated hydrocarbon solvent, or methylsilane. The capsules are covered with a membrane polymer that consists of thermoplastic resin composed of copolymers such as vinylidene chloride, acrylic acid ester, or methacrylic acid ester. When the microcapsules are heated above the softening point of the membrane polymer and the vapor pressure of the expanding agent rises, they expand by 50-100 times in volume.

Tooth specimens and bracket bonding
Two hundred freshly extracted bovine permanent mandibular incisors were collected from a slaughterhouse. The criteria for tooth selection included an intact labial enamel with no cracks caused by the extraction forceps and no caries. The teeth were divided randomly into 20 groups of 10 specimens each for each testing variable.

Soft tissues were removed from the teeth. After separating the crown from the root, the pulp was extirpated and the crown stored in distilled water at 5 until further use. Then, the crown was embedded in self-curing acrylic resin (Tray Resin, Shofu, Kyoto, Japan) for easy placement in the testing machine.

The labial surface of each incisor was polished with waterproof #400 and #600 sandpapers. The enamel surfaces were rinsed with water and dried with an oil-free air stream.

One operator bonded 200 stainless steel maxillary central incisor brackets with a 0.018-in slot (New DynaLock, 3M Unitek, Monrovia, CA) as follows. The enamel surface was etched for 30 seconds, rinsed, and dried according to the manufacturer's instructions. After placing an adhesive on the bracket base and positioning the bracket at the center of the treated enamel surface, the bracket was firmly seated. Excess adhesive was then removed with a dental probe without disturbing the bracket. After the samples were stored for 30 minutes at room temperature, the specimens were immersed in distilled water at 37 for 24 hours. The average bracket base surface area was determined to be 16.3 mm^2.

Thermal cycling and heating the brackets
For bond durability test, the specimens were subjected to continuous thermal cycling for 1,000 cycles between 4 and 60 water baths with a 30-second dwell time in each bath before shear bond strength measurement.

Brackets in the heating groups were heated from the top with a heater (Ultra Five Heater, Hakko, Nagano, Japan) for eight and 10 seconds, and then cooled immediately with water (Fig. 1). Heating temperature was set at 300 .

Shear bond strength measurement
All samples were tested in a shear mode on a universal testing machine (Instron, Canton, MA). For shear testing, the specimens were secured in the lower jaw of the machine so that the bracket base of
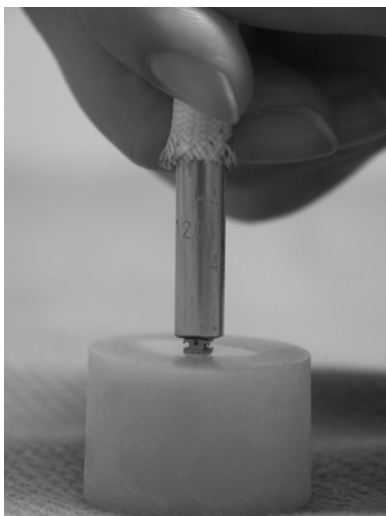

Fig. 1 Brackets in the heating groups were heated from the top with a heater for 8 and 10 sec.
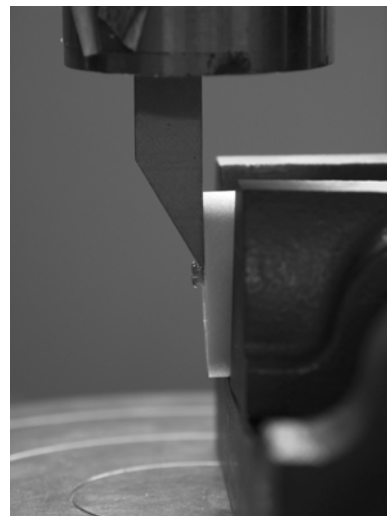

Fig. 2 Specimens were stressed in an occulusogigival direction at a crosshead speed of 1 mm/min

the sample paralleled the direction of the shear force. The specimens were stressed in an occlusogingival direction at a crosshead speed of 1 mm/min (Fig. 2).

Measurement of temperature rise in the pulp chamber
To evaluate temperature rise in the pulp chamber when the brackets were heated for several seconds, five fresh human permanent first premolars extracted for orthodontic treatment were used. Protocol for this experiment was reviewed and approved by the Nihon University Department of Dentistry Ethics Committee. The extracted teeth were stored in distilled water at 4　　until use. Brackets were bonded to the teeth with bonding material containing 30 wt　microcapsules. Each tooth was drilled with an air turbine from the lingual cementoenamel junction toward the labial bonded bracket. Then, the 0.5-mm-diameter sensor head of a K-type thermocouple (Okazaki Manufacturing, Kobe, Japan) was placed in contact with the inner surface of the pulp wall, facing the labial surface where the bracket was bonded. After heating for five, eight, or 10 seconds, the heated bracket was cooled with water immediately. Temperature of the inner surface of the pulp wall was measured at a room temperature of 24± 1　, and measurement was performed five times for each of the three heating times.

Statistical analysis
Appropriate statistical analyses of the results were performed on all data sets using the program SPSS (Chicago, IL). Descriptive statistics that included the mean and standard deviation (SD) were calculated for each of the 20 groups. Two-way analysis of variance (ANOVA) was used to determine whether there was an interaction between the presence or absence of heating and the percentage of microcapsules, and between the presence or absence of thermal cycling and the percentage of microcapsules. Scheffé's test for multiple comparisons was used to determine whether significant differences existed among the various groups. Significance for all statistical tests was predetermined at $P<0.05$.

RESULTS

Figure 3 shows the descriptive statistics for the shear bond strength of each group before and after heating. Two-way ANOVA showed that there was a synergistic effect between the presence or absence of heating and the percentage of microcapsules.

Among the unheated groups, the shear bond strength of the group containing 40 wt microcapsules was significantly lower at 0.7 times that of the base resin ($P<0.01$). Between the heated and unheated groups, the bond strengths of the heated groups were significantly lower than those of
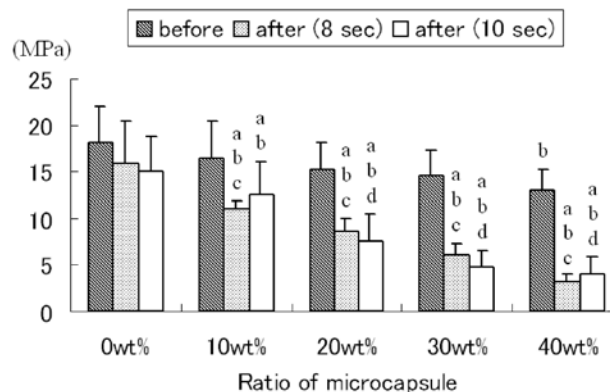

Fig. 3　Comparison of shear bond strengths before and after heating for 8 and 10 seconds. Bond strength decreased significantly on heating for 8 or 10 sec, and this decrease was dependent on the percentage of microcapsules.
　a:　Bond strength of the heated group was significantly lower than that of the unheated group in the respective 10, 20, 30, and 40 wt　groups.
　b:　Significant difference from the control group (before, 0 wt　).
　c:　Significant difference from 0 wt　group after heating for 8 seconds.
　d:　Significant difference from 0 wt　group after heating for 10 sec.

the unheated groups in the respective 10, 20, 30, and 40 wt　groups ($P<0.05$). In the groups heated for eight seconds, the bond strengths of all the groups containing microcapsules were significantly lower at 0.2-0.6 times that of the base resin ($P<0.01$). In the groups heated for 10 seconds, the bond strengths of the groups containing 20, 30, and 40 wt microcapsules were significantly lower at 0.2-0.6 times that of the base resin ($P<0.01$). At this juncture, it should be mentioned that heating for five seconds was barely sufficient to expand the microcapsules (data not shown).

Figures 4(a)-(c) represent the typical stereomicroscope images of tooth surface and bracket base after 8-second heating in the 0, 20, and 40 wt　groups respectively. In 0 wt　group (Fig. 4(a)), interfacial peeling between the tooth surface and adhesive was found. In 20 wt　group (Fig. 4(b)), cohesive failure in the adhesive and interfacial peeling between the tooth surface and adhesive were found. In 40 wt group (Fig. 4(c)), cohesive failure in the adhesive and interfacial peeling between the adhesive and bracket base were found.

Figure 5 shows the descriptive statistics for the shear bond strength of each group after 1,000 thermal cycles. There was no synergistic effect betweenthe presence or absence of thermal cycling and the percentage of microcapsules according to two-way ANOVA. Bond strengths of the groups
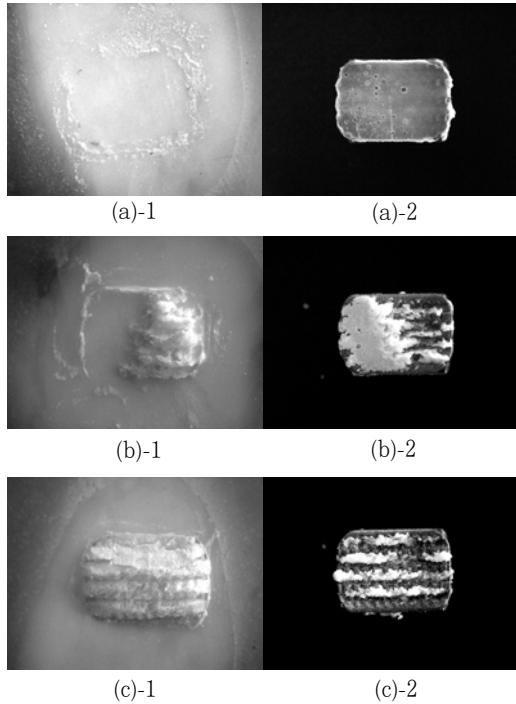
(a)-1 (a)-2

(b)-1 (b)-2

(c)-1 (c)-2

Fig. 4 Typical streomicroscope images of tooth surface
(1) and bracket base (2) after 8-sec heating.
  a: In 0 wt group, only interfacial peeling be-
     tween tooth surface and adhesive occurred.
  b: In 20 wt group, cohesive failure in the ad-
     hesive and interfacial peeling between tooth
     surface and adhesive occurred.
  c: In 40 wt group, cohesive failure in the ad-
     hesive and interfacial peeling between adhe-
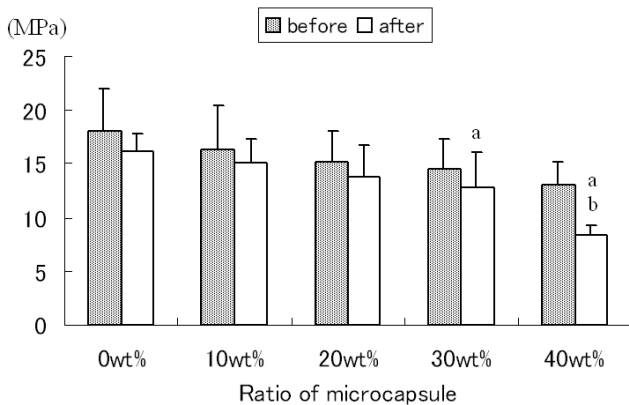   sive and bracket base occurred.

Fig. 5 Comparison of shear bond strengths before and
after thermal cycling.
  a: Significant difference from 0 wt group
      after thermal cycling.
  b: Significant difference between before and
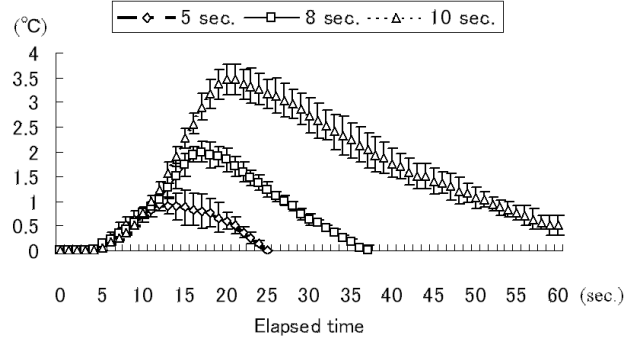   after thermal cycling at the same wt .

Fig. 6 Temporal changes in the temperature of the pulp
chamber. Temperature rise in the pulp chamber
ranged between 1 and 3.5 on heating for 5 to 10
sec.

containing 30 and 40 wt microcapsules were signifi-
cantly lower at 0.8 and 0.5 times that of the base
resin respectively ($P > 0.05$). Moreover, within the 40
wt group, the bond strength after thermal cycling
was only 0.6 times of that before thermal cycling.

Figure 6 shows the temperature increase in the
pulp chamber when the brackets were heated with a
heater. Temperature of the pulp chamber increased
by 1, 2, and 3.5 after heating for five, eight, and 10
seconds respectively. The temperature returned to
baseline within 25 seconds on heating for five seconds
and within 37 seconds on heating for eight seconds,
but still did not return to baseline within 60 seconds
on heating for 10 seconds.

DISCUSSION

In this study, we found that heating decreased the
bond strength of the orthodontic brackets signifi-
cantly when bonded with bonding materials contain-
ing microcapsules. Bond strength of the heated
groups was significantly lower than that of the un-
heated group in the 10, 20, 30, and 40 wt groups
respectively ($P < 0.05$). On heating for eight seconds,
the bond strength decreased to 60.4, 47.8, 33.6, and
17.6 that of the unheated conventional adhesive
(SuperBond) in the 10, 20, 30, and 40 wt groups re-
spectively. Similarly, on heating for 10 seconds, the
bond strength decreased to 69.5, 42.4, 26.5, and 22.3
that of the control in the 10, 20, 30, and 40 wt
groups respectively. These findings suggested that
the bond strength of the brackets bonded using this
bonding material decreased significantly after heat-
ing for eight to 10 seconds, and this decrease was de-
pendent on the percentage of microcapsules. It was
likely that the internal stress of the bonding mate-
rial, induced by the expansion of microcapsules on
heating, collapsed the linear structure of the material
whereby this collapse led to cohesive failure in the
bonding material. Indeed, the stereomicroscope

images of 40 wt% group revealed cohesive failure in the adhesive and interfacial peeling between the adhesive and bracket base after debonding (Fig. 4(c)). It should be mentioned that conditions of the tooth in Fig. 4(c) will not induce enamel cracks.

For the 30 wt% group, bond strength decreased to about one-third on heating compared to the base resin and unheated groups. Nevertheless, one of these values was 7 MPa, which was still higher than the minimum bond strength (5.9 MPa) recommended for orthodontic treatment by Reynolds[5]. Therefore, debonding might still be difficult. For the 40 wt% group, bond strength decreased to about one-fifth on heating and was less than 5.9 MPa. Therefore, debonding would likely be much easier compared to normal debonding and should not cause pain or enamel cracks. Mimura *et al.* reported that SuperBond expanded until 80°C, after peaking at 60°C[14]. Indeed, the bond strength of the unheated 40wt% group was significantly lower at 72% that of the unheated base resin group, although it was still about 13 MPa which should be sufficient for bonding in clinical use as reported by Reynolds[5].

To test bond durability, specimens were subjected to 1,000 thermal cycles between 4°C and 60°C. This is equivalent to a period of about two years and eight months, on the assumption that a rapid temperature change happens once daily. In the 40 wt% group, thermal cycling significantly decreased the bond strength by 35% to only 8.43±0.83 MPa. Nevertheless, this was still higher than the minimum bond strength (5.9 MPa) required for orthodontic treatment. However, note that bond strength decreases with treatment time in orthodontics.

The microcapsules used in this study began to expand at 80°C. This temperature should be sufficiently high to avoid expansion in daily life. Plant *et al.* reported that it was impossible to take coffee hotter than 68°C into the mouth[17], while coffee at 55-67°C is drinkable[18]. In this study, brackets were heated from the top of the bracket wings with the heater set to 300°C. On this note, temperature rise in the pulp chamber when microcapsules were expanded must be taken into consideration. The maximum temperature rise in the pulp chamber of the first premolars was 0.93±0.15, 2.00±0.20, and 3.47±0.31°C with heating for five, eight, and 10 seconds respectively. Zach and Cohen reported that 85% of the tissue recovers when the pulp temperature rose to 5.5°C above the body temperature in a study of Macaca monkeys, while only 40% recovered with a rise of 11°C[19]. Moritz and Henriques also reported that the extent of pulp tissue damage depended on both the size and duration of heat accumulation in the pulp chamber[20]. Since the temperature rise in the pulp chamber of premolars on heating for 10 seconds was only about 3.5°C, pulp damage might not occur.

However, it must be highlighted that the enamel and dentin layers at the center of the tooth crown in incisors are thinner than in premolars[21]. On this note, pulp temperature might increase more in incisors than in premolars. Based on the results in this study, it seemed that there were no significant differences in bond strength between heating for eight and 10 seconds. Most probably, the higher increase in pulp chamber temperature on heating for 10 seconds was due to a slightly prolonged heating time. In light of these findings for 8- and 10-second heating, a heating time of eight seconds might be better for avoiding damage to the pulp.

Heating temperature of the heater was set at 300°C in this study. For clinical application, it might mandate an improvement or modification to the heater design such that it is covered with a low heat conductance material (such as silicon rubber) to prevent any thermal injury to the oral soft tissues.

Results of the present study should be leveraged and used to develop a new bonding material for clinical orthodontics so that no pain or enamel cracks would be encountered during bracket debonding. In particular, it was reported that debonding of ceramic brackets caused more pain and enamel cracks[22-26]. With a view to solving these problems, in progress now are experiments that further examine the bond strength of ceramic brackets bonded with this new bonding material.

CONCLUSIONS

Within the limitations of the present study, the following conclusions were drawn:

Bond strength of the new bonding material containing 30-40 wt% microcapsules in conventional orthodontic adhesive (SuperBond) decreased to about one-third or one-fifth of SuperBond alone on heating for eight seconds. Although thermal cycling decreased the bond strength by 7.8-35.4% in all the bonding materials, the resultant bond strength was still greater than the minimum bond strength necessary for orthodontic treatment.

Since heating the brackets for eight seconds increased the pulp chamber temperature by 2°C for a short period, eight seconds seemed to an optimal heating duration without causing pulp damage.

These results thus demonstrated that the new bonding material should facilitate bracket removal during bracket debonding without pain or enamel cracks.

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