# [Original] Matsumoto Shigaku 23:97~105, 1997 key words: Adhesives – Bond strength – 4-META MMA resin – Rebonding – Bis-GMA resin

# Shear Bond Strength of Brackets Rebonded with 4-META MMA Resin

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#### Summary

The purpose of this study was to determine the difference of the shear bond strength between new and rebonded brackets with a chairside cleaning method proposed by the authors. A preliminary study was carried out to examine the optimal cleaning time for both 4-META MMA resin (Superbond, Sun Medical, Kyoto, Japan) and Bis-GMA resin (Concise, 3M, Monrovia, California, USA) using acetone or chloroform. The 4-META MMA resin was completely removed by acetone (13 min.) and by chloroform (8 min.), while the Bis-GMA resin was not removed by either. The resin remnant on the bracket base was stained with alcohol-based ink and analyzed quantitatively under a microscope attached to a CCD camera and a computer analyzer. Stainless steel brackets with foil mesh base designs were bonded to twenty extracted human premolars with the 4-META MMA resin. All specimens were stored in water at 37°C for 24 hours, and thermocycled 120 times from 4°C to 60°C before shear bond strength testing. Specimens were stressed to bond failure using an Autograph AG-5000D testing machine (Shimadzu, Kyoto, Japan). The bases of the debonded brackets were inspected for resin according to an adaptation of the adhesive remnant index (ARI) system. After recording of the shear bond strengths, the brackets were cleaned and rebonded to the same teeth and tested once more. The results revealed that the rebonding of the brackets even twice following the original bonding had no significant effect on the bond strength value. The capacity for rebonding of the same bracket by an easy and fast cleaning method is an advantage of the 4-META MMA resin over the Bis-GMA resin.

## Introduction

The reuse of the same debonded orthodontic bracket would be beneficial in reducing waste and costs for both orthodontists and patients, especially in cases where bond failure occurs or a bracket repositioning is required at chairside. Rebonding of the same bracket would be the best choice were a practical, simple and fast chairside method of bracket cleaning available.

Several reconditioning methods have been described as a means of eliminating the residual adhesive remnant from the bracket base in a so-called recycling process<sup>1~5)</sup>. The main purpose of a recycling process is to remove the bonding material completely from the bracket base without damaging the finer mesh or distorting its dimensions and also without altering the physical properties of the brackets structure<sup>1.3,6)</sup>. The bracket specifications, such as torque and tip, should be preserved. Furthermore, appropriate bracket sterilization should be employed<sup>7)</sup>. Basically, the two main commercial methods employ either thermal or chemical processes to remove the remnant bonding material. While the thermal process utilizes heat, the chemical recycling process employs solvents<sup>1,2)</sup>.

However, the heat utilization during the thermal recycling process is not indicated for metallic brackets. It is etablished that heat exposure beyond 650°C has irreversible and highly deleterious effects on the metallic microstructure<sup>1,3,8)</sup>. At temperatures between 400°C to 900°C, a chromium carbide precipitate is formed resulting in partial disintegration of the alloy. This leads to weakness of the metallic structure and alteration of properties such as hardness and corrosion resistance and loss of metal annealing<sup>1,5,7)</sup>. In addition, heating brackets to high temperatures in order to burn out the bonding material remnants results in an unaesthetic brown-black appearance<sup>5,9)</sup>.

The chemical method uses solvents stripping the adhesive from the bracket with ultrasonic cleaning at temperatures below 100°C, followed by 250°C for sterilization purposes and a flash eletropolishing procedure. Nevertheless, the compositions of the solvents developed by individual recycling companies are not identified. Buchman (1980)<sup>1)</sup> considered the chemical reconditioning process the most desirable method in terms of not altering the mechanical properties of the metallic bracket.

However, the full set of commercial bracket reconditioning processes, using either heat or chemical solvents, is not practical for performance at chairside. This is particularly true when an improper bracket position may necessitate removal and appropriate rebonding at the correct position or when bonding failure occurs.

Although clinical and laboratory studies have been reported on the bond strength of commercially recycled orthodontic brackets, there appears to be little guidance regarding the fate of previously debonded brackets that have been rebonded with 4-META MMA resin.

The purpose of this study was to determine the bond strength of brackets bonded with 4-META MMA resin prior to and after the cleaning method proposed by the authors. The timing of the resin remnant removal was included in the first part of the study in order to determine an optimal cleaning time. It is also our intention to elaborate a practical protocol for bracket cleaning at chairside.

# **Material and Methods**

Adhesive remnant study. The first part of the investigation consisted of bonding 200 stainless steel brackets with foil-mesh base design to 11-mm-diameter metallic spheres with smooth surfaces

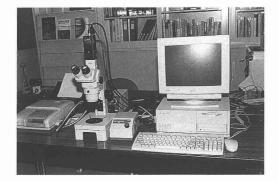


Fig. 1: Computer image analyzer

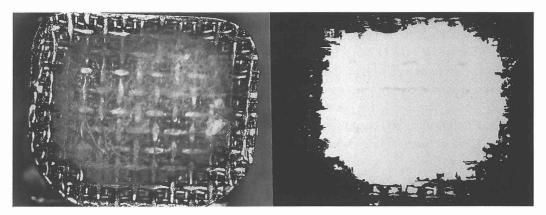


Fig. 2: Bracket base stained with alcohol-based ink

to provide a homogeneous and standard resin thickness. The brackets were bonded to the spheres with either 4-META MMA resin (n=150) or Bis-GMA resin (n=50).

Following debonding, all bracket bases were placed separately into small glass bins each contains 1 ml of acetone or chloroform solutions. The bins were than placed into the ultrasonic cleaner machine for 1 to 15 minutes. After every minute one bin was removed, and the brackets were rinsed with 70% alcohol and distilled water in order to remove all traces of solvent. All brackets were then placed into the alcohol-based ink for stain purposes.

*Computer image analyzer*. The resin remnant on the bracket base was stained with alcoholbased ink (Sakura color products, Japan) for 5 minutes and analyzed quantitatively under a microscope linked to a CCD camera and a computer image analyzer (IP-500PC, Asahi Chemical, Tokyo, Japan) (Fig. 1). The alcohol-based ink was used as a stain marker to facilitate the assessment of the amount of the 4-META MMA resin remnant (Fig. 2). After recording of the resin remnant percentage on each bracket base, the mean values of the adhesive remnant ratio were graphed.

*Brackets and adhesives.* For the second part of the experiment conventional premolar stainless steel brackets with foil-mesh base design (A-Company, San Diego, California, USA), commonly used in clinical practice, were selected. The bonding agent employed in this experiment was only the 4-META MMA resin, since the Bis-GMA resin was not removed by the solvents.

# **Bonding tests**

For this study, a sample of 20 non-carious extracted maxillary premolars which had been stored in 70% ethyl alcohol were collected. The teeth were carefully examined and any carious, damaged or malformed specimens were excluded. No evidence of defective enamel on the buccal surfaces was found on viewing under a  $\times$  10 magnifying lens (SZH-111, Olympus, Tokyo, Japan). The teeth were embedded in die self-cure acrylic blocks with the buccal surfaces exposed above the resin and parallel to the face of the block. All blocks were allocated a number so that each bracket would be correctly identified and rebonded to the original tooth. Before bonding, the buccal tooth enamel surface was polished for 20 seconds using a rubber cup and a slurry of pumice and water. Etching was performed using 37% orthophosphoric acid for 60 seconds. The teeth were then rinsed for 20 seconds under cold water to remove all traces of orthophosphoric acid and dried under an oil-free air jet. After drying, the brackets were bonded to the buccal surfaces with 4-META MMA resin. All teeth were bonded in accordance with the manufacturer's instructions. Although according to Deguchi et al.  $(1996)^{12}$  the brush-on technique is clinically used for the 4-META MMA resin, the polymer (0.07 g) was mixed with four drops of monomer and one drop of catalyst in the dispensing dish, so that the testing conditions would be standardized. During all stages of bonding the brackets, all materials were handled in the summer room with a controlled temperature of 23°C and at 60% humidity. After bonding was completed, all specimens were stored at 37°C in distilled water for 24 hours and thermocycled 120 times from 4°C to 60°C prior to the bond strength testing. According to Mogi<sup>13)</sup>, this is equivalent to a 3-month period of storage of the 4-META MMA resin in 37°C water.

*Bond strength testing.* The bonding strength testing was carried out with a testing machine. The acrylic blocks were placed in the device, and the direction of the force application was oriented parallel to the tooth buccal surface. The load was applied 1 mm from the bracket resin interface by the testing machine in such a way that the shearing force acted on the bracket/enamel interface. The cross-head speed of 1 mm per minute was used, and the force required to debond the brackets was recorded in kilogram force.

Adhesive Remnant Index. The extent of residual adhesive resin on the bracket bases was visually inspected under a stereoscopic microscope at  $\times$  17.5 magnification. The amount of resin remaining on the bracket base was determined and classified with a modification of the Adhesive Remnant Index (ARI)<sup>14,16)</sup> as follows: (0) no adhesive remaining; (1) less than half remaining; (2) half or more than half remaining; and (3) all of the adhesive remaining.

*Rebonding used brackets*. After recording of shear bonding values and the modified ARI classification, the debonded brackets were cleaned using the method proposed by the authors. Each debonded bracket was placed in a glass bin containing 1 ml of chloroform, ultrasonically cleaned with 8 minutes and then rinsed with 70% alcohol to remove all traces of solvent. All 20 teeth were cleaned with both hand scalers and low-speed rotary instruments, pumiced, and washed to restore the visually clean enamel appearance for the following first rebonding. The cleaned brackets were rebonded following the protocol described above and were submitted to the shear bond strength test. Since the blocks were numbered and the debonded brackets were stored separately, it was possible to compare the performance of the same bracket/tooth combinations when bonded and then debonded. After the results recorded, the protocol was repeated for the second rebonding.

Statistical analysis. Student's t test was used to compare the each data.

#### Results

Adhesive remnant study. The results of the first part of the experiment with the mean values for the resin remnant ratio on the bracket base following by chemical cleaning are shown in Figure 3. The 4-META MMA resin was completely removed from the bracket base using both chloroform (8 min.) and acetone (13 min.) while the Bis-GMA resin was not removed.

*Bond strength.* The initial mean shear bond strength of the new brackets and rebonded brackets are illustrated graphically in Figure 4. After following the first and the second rebonding through the cleaning method proposed by the authors there was no significant difference in the shear bond strength compared to that of the new brackets.

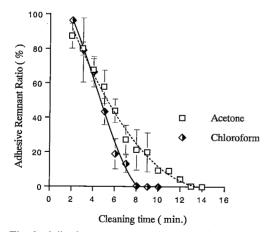
Adhesive Remnant Index. The ARI was of recorded following the initial debonding procedure mainly to access the sites of fractures. The percentage distribution of the ARI is given in Figure 5. It showed wide variation among the new and rebonded brackets, with most of the bracket bases having over 70% of the adhesive still remaining on the surface. No significant difference was found between the ARI values for the first and second rebonded bracket strength. However a significant difference was observed for the new and rebonded brackets.

#### Discussion

This investigation was carried out in two parts, the first of which was intended to determine the optimal cleaning time for the resin remnant on the bracket base using one of two solvents. The second part investigated the shear bond strength of both new and rebonded brackets cleaned by the same method used in the first part.

*Optimal cleaning time*. In the investigation of the optimal cleaning time with chloroform and with acetone to remove the 4-META MMA resin and the Bis-GMA resin remnant from the bracket base, only the 4-META MMA resin could be completely removed, with either chloroform or acetone solvent. The Bis-GMA resin was not removed from the bracket base using these solvents.

O'Brien et al.<sup>15</sup> proposed a method of quantifying the amount of resin remnants using a



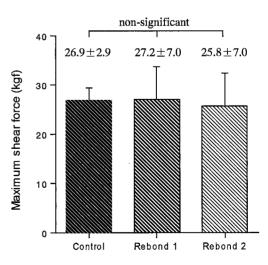


Fig. 3: Adhesive remnant ratio at each cleaning time with acetone and with chloroform. The graph was generated using the computer image analyzer. Data are mean values, with vertical bars denoting standard deviations.

Fig. 4: Shear bond strength values with standard deviations of new control and rebonded brackets.

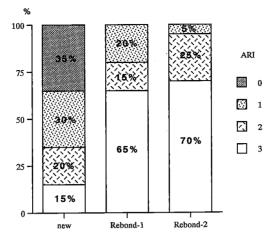


Fig. 5: Percentage frequency distribution of the adhesive remnant index (ARI) for new brackets and following initial and repeat debonding.

projection microscope and a computer. The remnants were examined and traced onto high quality tracing paper and then, the area under each tracing was calculated and expressed as a percentage of the bracket base. In our study, through use of the image analyzer, it was possible to quantify the amount of resin remnant with high precision. The use of the alcohol-based ink was effective in identifying all traces of resin remnant on the bracket base, even those located inside the meshes.

Metallic spheres of 11 mm diameter were used in order to obtain a standard and homogeneous thickness of resin on the base of the debonded brackets. Using spheres instead of cylinders made the bonding procedure easier, since the bonding could be performed everywhere in the sphere superfine structure without changing the resin thickness.

There have been many attempts to develop a fast chairside method of reconditioning the debonded bracket. Lew and Djeng (1990)<sup>10</sup> reported a chairside method of recycling in which the ceramic brackets were heated until they turn cherry red to eliminate the bonding material. Lew *et al.*<sup>11</sup>, later investigating the shear bond strength of these rebonded brackets, observed a decrease in the bond strength of approximately 40%.

Regan *et al.*  $(1993)^{9}$  described a chairside recycling method where the bonding material on the debonded brackets was removed with a bur or merely roughened and bonded with a chemically cured adhesive. Gaffey *et al.*  $(1995)^{16}$  investigated the shear/peel bond strength of electrothermally debonded ceramic brackets treated with either heat and silane coupling agent and observed a significant decrease in bond strength for all rebonded brackets. More recently, Egan *et al.*  $(1996)^{17}$  evaluated the bond strength of brackets rebonded and coated, without removing the initial bonding material, using a plastic conditioner and adhesion booster. The mean bond strength produced with no conditioner was not significantly different from that of the initial bonding, and the use of the plastic conditioner did not improve the rebond strength.

In light of the failure to find any significant difference in the shear bond strength of new and rebonded brackets, there appears to be some advantage in employing the 4-META MMA resin as a bonding material. In addition, the time consumed for the complete resin remnant removal using either chloroform (8 min.) or acetone (13 min.), followed by adequate alcohol rinse to remove all traces of solvent, seems to be proportional to the chairside time consumed for the tooth preparation,

such as scaling, pumicing, and etching for new rebonding.

The shear bonding testing. In the second part of this study we evaluated the shear bond strength of both the new and rebonded metallic brackets. The results of the experiment accorded with those of previous studies demonstrating that cleaning used stainless steel brackets by chemical means does not significantly alter their shear bond strength<sup>1,8,15)</sup>.

The standard deviations were greater for the brackets after both the first (7.0) and second (7.2) rebonding compared to the control (2.9). This could have been caused by variations in the adhesive thickness, voids produced by the bonding procedure, the degree and extent of etching on the tooth surface, and variations of the curvature on the buccal surface of the teeth. However, O'Brien *et al.* (1988)<sup>15)</sup> found no correlation between the site of separation and the shear bond values. Since each block was allocated a number and the debonded brackets were stored separately, it was possible to compare the performance of the same bracket/tooth combinations when bonded and then debonded.

Mascia and Chen (1982)<sup>6)</sup> and Egan *et al.* (1996)<sup>17)</sup> failed to demonstrate significant difference in bond strength among teeth that had been reused for bonding. Gaffey *et al.* (1995)<sup>16)</sup> reported a reduction in the shear bonding strength of brackets rebonded to bovine teeth; nevertheless the brackets were not bonded to the same teeth. Bonding failure occurred in the majority of the rebonded bracket samples (70% after the first and 65% after the second rebonding) at the enamel/ resin interface. This finding is in accordance with that cited by Egan *et al.* (1996)<sup>17)</sup>. The predominance of enamel/resin failure following the initial bonding can be attributed to the intrinsic property of the 4-META MMA resin of adhesion to metal and modifications at the enamel surface<sup>9)</sup>. The potential for rebonding of the same bracket by an easy and fast cleaning method is among the advantages of the 4-META MMA resin over the Bis-GMA resin.

# Conclusions

Based on the results of this study we conclude that :

- 1. The 4-META MMA resin is easily removed from the bracket base using either chloroform or acetone, whereas the Bis-GMA resin is not removed at all. The 4-META MMA resin presents the advantage of affording rebonding of the same bracket by an easy and fast cleaning method.
- 2. For the 4-META MMA resin the time consumed in the complete resin removal using chloroform (8 min.) was shorter than that using acetone (13min.).
- 3. Alcohol-based ink seems to useful stain marker for the 4-META MMA resin remnant.
- 4. There is no difference in the shear bond strength between new and rebonded brackets using 4-META MMA resin. Successive rebonding following the original bonding appears to have no significant effect on the bond strength.
- 5. Using a glass bin containing 1 ml of chloroform placed in an ultrasonic cleaner for 8 minutes appears to be a fast and practical cleaning method for the 4-META MMA resin remnant.

The findings of this study suggest that the use of 4-META MMA resin is indicated for allowing an easy rebonding by an easy and fast cleaning method. However, the extrapolation from laboratory data to clinical situations should always be done with care.

# Acknowledgments

The authors gratefully acknowledge the suggestions and advice of the staff of the Department of Orthodontics, Matsumoto Dental University School of Dentistry.

Special thanks to Dr. Y. Yoshikawa, Dr. T. Kojima and Dr. A. Sakatoku for their faithful

assistance and to Mr. H. Pine for proof reading this paper.

This study was partly supported by grants from Nagano prefecture.

#### Appendix

This paper was presented at the XI Meeting of Brazilian Health Field Professionals in Japan (held in Osaka University Faculty of Dentistry, on February 8, 1997)

### References

- Buchman, D. J. (1980) Effects of recycling on metallic direct-bonding orthodontic brackets. Am. J. Orthod. 77: 654-668.
- 2) Machen, D. E. (1993) Orthodontic bracket recycling. Am. J. Orthod. Dentofac. Orthop. [Comments]. 104: 618–619.
- 3) Hixson, M. E., Brantley, W. A., Pincsak, J. J. and Conover, J. P. (1982) Changes in bracket slot tolerance following recycling of direct-bond metallic orthodonic appliances. Am. J. Orthod. 81: 447-454.
- 4) Oliver, R. G. and Pal, A. D. (1989) Distortion of edgewise orthodontic brackets associated with different methods of debonding. Am. J. Orthod. Dentofac. Orthop. **96**: 65–71.
- 5) Wheeler, J. J. and Ackerman, R. J. Jr. (1983) Bond strength of thermally recycled brackets. Am. J. Orthod. 83: 181-186.
- 6) Mascia, V. E. and Chen, S. R. (1982) Shearing strength of recycled direct-bonding brackets. Am. J. Orthod. 82: 211-216.
- Maijer, R. and Smith, D. C. (1986) Biodegradation of the orthodontic bracket system. Am. J. Orthod. Dentofac. Orthop. 90: 195–198.
- 8) Regan, D., Van Noort, R., O'Keefe, C. (1990) The effects of recycling on the tensile bond strength of new and clinically used stainless steel orthodontic brackets: an in vitro study. Br. J. Orthod. 17: 137–145.
- 9) Regan, D., Le Masney, B. and Van Noort, R. (1993) The tensile bond strength of new and rebonded stainless steel orthodontic bracket. Eur. J. Orthod. 15: 125-135.
- 10) Lew, K. K. K. and Djeng, S. K. (1990) Recycling ceramic brackets. J. Clin. Orthod. 24: 44-47.
- Lew, K. K. K., Chew, C. L. and Lew, K. M. (1991) A comparison of shear bond strengths between new and recycled ceramic brackets. Eur. J. Orthod. 13: 306-310.
- 12) Deguchi, T., Ito, M., Obata, A., Koh, Y., Yamagishi, T. and Oshida, Y. (1996) Trial production of titanium orthodontic brackets fabricated by metal injection molding (MIM) with sintering. J. Dent. Res. 75: 1491-1496.
- Mogi, M. (1982) Study on the application of 4-META/MMA-TBB resin to orthodontics. J. Jpn. Orthod. Soc. 41: 260-271. (in Japanese, English abstract )
- Artun, J. and Bergland, S. (1984) Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am. J. Orthod. 85: 333-340.
- 15) O'Brian, K. D., Watts, D. C. and Read, M. J. F. (1988) Residual debris and bond strength is there a relationship? Am. J. Orthod. Dentofac. Orthop. 94: 222-230.
- 16) Gaffey, P. G., Major, P. W., Glover, K. Grace, M. and Koehler J. R. (1995) Shear/peel bond strength of repositioned ceramic brackets. Angle Orthod. 65: 351–358.
- 17) Egan, F. R., Alexander, S. A. and Cartwrigth, G. E. (1996) Bond strength of rebonded orthodontic brackets. Am. J. Orthod. Dentofac. Orthop. 109: 64-70.

# 抄録:4-META MMA レジンで接着した矯正用ブラケットの再接着時における圧縮剪断荷重について

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本研究では、撤去したブラケットに付着した接着剤を、著者らが考案した臨床的方法で除去し、同一 歯面に再接着させた場合の圧縮剪断力の比較を行った.まず、予備実験としてアセトンとクロロホルム を用い、接着剤は4-META MMA レジンと Bis-GMA レジンを用いて理想的な接着剤撤去時間を求め た.4-META MMA レジンにおいては、アセトンでは13分以内で、クロロホルムでは8分でレジン除去 が可能であったが、Bis-GMA レジンでは撤去は不可能であった.ブラケットベースに残留した接着性レ ジンは、アルコールインクで染め出し、実体顕微鏡に接続した CCD カメラからコンピューターアナライ ザー装置に接続し、ブラケットベース表面の残留レジンのブラケットに占める割合を測定した.

メッシュ状のベース面を持つステンレススチール製のブラケットを、4-META MMA レジンで20本 のヒト抜去小臼歯に接着した。37℃の水中に24時間浸漬した後、4 ℃と60℃120回のサーマルサイクリン グを行い、圧縮剪断試験を行った。圧縮剪断試験後、ブラケット表面の接着剤残留指数(ARI)を記録し た、剪断試験後に、ブラケットの接着剤を予備実験で得られた方法で除去し、接着剤除去後の同一歯に 再び接着し、圧縮剪断試験を行った。2 回再接着を行った際の圧縮剪断荷重は、1 回目の新鮮な歯面お よびブラケットでの接着時の剪断荷重と有意差はなかった。また、4-META MMA レジンは Bis-GMA レジンよりもより簡単で、しかも同一ブラケットの再接着を早く行うことが可能であった。