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Shear bond strength of brackets bonded to amalgam with different intermediate resins and adhesives

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SUMMARY The aims of this study were to compare, *in vitro*, the shear bond strength (SBS) of stainless steel orthodontic brackets bonded to silver amalgam with the use of three different intermediate resins and two different adhesives, and to evaluate bond failure mode. Forty-five amalgam specimens were divided into three equal groups. In groups 1 and 2, the brackets were bonded with Unite (3M Unitek) using Reliance Metal Primer (RMP; Reliance Orthodontic Products) and Power Bond™ OLC (PB OLC; Ortho Organizers Inc.) as intermediate resins, respectively. In group 3, Resinomer and One-Step Plus (OS+; Bisco Inc.) were used. Thirty bovine teeth served as the controls to test bracket bonding to acid-etched enamel with Unite and Resinomer–OS+. After thermocycling from 10 to 50°C 1000 times, all samples were tested for SBS. Bond failure sites were classified using a modified adhesive remnant index (ARI) system. Data were analyzed with one-way analysis of variance, *post hoc* Tukey multiple comparison and chi-square tests.

The results showed that the mean SBS to amalgam surfaces were significantly lower than those to etched bovine enamel ($P < 0.001$). There were no statistically significant differences in mean SBS between the amalgam bonding groups ($P > 0.05$). For the ARI, significant differences were found between the amalgam- and enamel-bonding groups ($P < 0.001$).

The mean SBS of stainless steel orthodontic brackets bonded to amalgam surfaces with RMP, PB OLC, OS+ intermediate resins and Unite and Resinomer adhesives was significantly lower than to etched bovine enamel. Bond failure occurred at the amalgam–adhesive interface regardless of the adhesive system and without damage to the amalgam restoration.

Introduction

The progress in technical improvements of orthodontic bonding has lead to a decrease in banding of posterior teeth. A study on orthodontic trends revealed that molars and premolars were banded less routinely than in the past (Keim *et al.*, 2002). On the other hand, some orthodontic patients, especially adults, often have buccal amalgam restorations on their posterior teeth. In such cases, successful bonding of orthodontic attachments to amalgam surfaces is a challenge for orthodontists. This clinical problem led to the investigation of bonding to amalgam and the results of these studies revealed that different procedures are needed for improved amalgam bonding. These include surface treatment and the use of intermediate resins and adhesives which chemically bond to metals (Zachrisson and Buyukyilmaz, 1993; Zachrisson *et al.*, 1995; Jost-Brinkmann *et al.*, 1996; Gross *et al.*, 1997; Buyukyilmaz and Zachrisson, 1998; Jost-Brinkmann and Bohme, 1999; Sperber *et al.*, 1999; Harari *et al.*, 2000).

Different surface treatment procedures, such as roughening with a diamond bur (Zachrisson *et al.*, 1995; Harari *et al.*, 2000, Skilton *et al.*, 2006), sandblasting (Zachrisson *et al.*, 1995; Buyukyilmaz and Zachrisson, 1998; Sperber *et al.*, 1999, Skilton *et al.*, 2006), Ga–Sn liquid application (Gross *et al.*, 1997), and chemical corrosion (Sperber *et al.*, 1999), have been introduced for effective bonding on non-enamel surfaces. Sandblasting, the most common method used for surface preparation, creates scratch-like irregularities that increase bond strength. It was also noted that air abrasion increased the surface area of Co–Cr and Ni–Cr alloys leading to improved adhesion to resins containing 4-methacryloxyethyl trimellitate anhydride (4-META; Atta *et al.*, 1990). In addition to mechanical retention, bonding on metal has the advantage of chemical adhesion. Therefore, adhesives chemically bonded to amalgam such as 10-methacryloyloxydecyl dihydrogen phosphate bis-GMA resins or 4-META are recommended for this purpose (Zachrisson and Buyukyilmaz, 1993; Gross *et al.*, 1997).

4-META is a coupling agent which increases adhesion to enamel, composite resins (Atsuta *et al.*, 1982), and dental alloys by chemically bonding to the oxidized surface of non-precious metals (Tanaka *et al.*, 1981). When standard orthodontic adhesives, which lack the ability of chemical bonding, are preferred, they may be used with intermediate resins to enhance bond strength (Zachrisson and Buyukyilmaz, 1993; Zachrisson *et al.*, 1995; Jost-Brinkmann *et al.*, 1996; Buyukyilmaz and Zachrisson, 1998). In amalgam bonding, one of the most commonly used intermediate resins containing 4-META is Reliance Metal

Primer (RMP; Reliance Orthodontic Products, Itasca, Illinois, USA; Gross *et al.*, 1997, Buyukyilmaz and Zachrisson, 1998). Its effectiveness in increasing bond strength of orthodontic brackets to dental metals was shown by Buyukyilmaz and Zachrisson (1998).

Another booster for bonding to acrylic, composite, metal, or enamel surfaces is Power Bond™ OLC which is a light-cured bonding conditioner (PB OLC; Ortho Organizers Inc., San Marcos, California, USA). However, its effectiveness in bonding orthodontic attachments to amalgam has not been investigated.

Resinomer (Bisco Inc., Schaumburg, Illinois, USA) is a fluoride releasing, low-viscosity resin composite containing diarylsulfone dimethacrylate (DSDM). DSDM is a monomer which forms strong micromechanical as well as chemical bonds to all dental metals. According to the manufacturer, the principal uses of this dual cured, multipurpose resin ionomer are bonding to amalgam restorations, cementation of metal-based restorations and bonding orthodontic brackets. Resinomer is intended to be used with fourth- or fifth-generation adhesives such as One-Step Plus (OS+; Bisco Inc.). OS+, containing biphenyl dimethacrylate and hydroxethyl methacrylate, is designed to bond composite to dentine, enamel, and cast metals and set amalgam. In the literature, no data are available on the efficacy of this adhesive and intermediate resin system in orthodontic bonding to amalgam surfaces.

The aims of this study were to compare, *in vitro*, the shear bond strength (SBS) of orthodontic brackets bonded to silver amalgam with the use of three different intermediate resins (RMP, PB OLC, and OS+) and the use of two different adhesives [Unite (3M Unitek, Monrovia, California, USA) and Resinomer] and to evaluate bond failure mode using a modification of the Adhesive Remnant Index (ARI).

Materials and methods

A cavity, (width 6 mm, length 7 mm, axial depth 2 mm) with retention grooves at the base was prepared in a prototype acrylic tab fabricated from self-cure acrylic (Meliodent; Bayer Dental, Leverkusen, Germany). The impressions of this prototype tab were obtained using polyvinylsiloxane impression material (Speedex; Coltène, Whaledent, Altstatten, Switzerland). Self-cure acrylic was poured into the impressions to fabricate 45 uniform rectangular cavities in acrylic tabs. A commonly used lathe-cut non-gamma 2 amalgam, ANA 2000 (Nordiska Dental AB, Angelholm, Sweden) was condensed into the cavities and burnished with hand instruments. After 24 hours, the amalgam specimens were polished with brown and green rubber points (Shofu Inc., Kyoto, Japan) and stored in water at 37°C for an additional 48 hours. Before bonding, the amalgam surfaces were treated with aluminium oxide sandblasting with 50 μm abrasive powder (Korox 50; Bego, Bremen, Germany) in a microetcher (Danville Engineering, San Ramon, California, USA) at approximately 7 kg/cm^2 of air pressure for 3 seconds from a distance of 10 mm and thoroughly rinsed and dried. Mandibular stainless steel incisor brackets (Generus Roth; GAC International Inc., Bohemia, New York, USA) with an average bracket base surface area of 9.8 mm^2 were bonded to the sandblasted amalgam surfaces by the same orthodontist (DG). The single mesh base of the brackets was microetched by the manufacturer.

For bonding, a conventional orthodontic bonding adhesive (Unite) and an amalgam bonding adhesive (Resinomer) were used according to the manufacturers' instructions. Three intermediate resins, RMP, PB OLC, and OS+, enhancing bond strength to metal surfaces or amalgam were also used. In group 1, the brackets were bonded to amalgam with Unite and RMP, in group 2 with Unite and PB OLC and in group 3 with Resinomer and OS+ (Table 1). For each specimen in group 1, one drop of RMP was dispensed onto a mixing pad and a brush was visibly wetted with RMP. One coat of RMP was applied to the sandblasted amalgam and allowed to dry for 30 seconds. In group 2, two coats of PB OLC were applied to the amalgam surface. After the second coat, the surface was air dried with compressed air for 10 seconds and light cured for 20 seconds. All light curing was performed with a light-emitting diode (Starlights; Mectron S.p.a., Carasco, Italy). The brackets were bonded with Unite in groups 1 and 2 and the adhesive was allowed to set for 4 minutes. In group 3, OS+ was applied, air dried for 10 seconds and light cured for an additional 10 seconds. Resinomer was then used to bond brackets and allowed to self-cure for 6 minutes.

Thirty bovine permanent mandibular incisors were used as the controls. In group 4, the brackets were bonded to bovine enamel with Unite and in group 5 with Resinomer and OS+ (Table 1). In this study, all primary mandibular teeth were excluded because bond strengths to primary and permanent bovine teeth differ (Oesterle *et al.*, 1998). The teeth were stored in a solution of 70 per cent (w/v) ethyl alcohol and were not autoclaved. Prophylactic treatment was performed with pumice powder paste (Isler Pomza; Isler Dental, Ankara, Turkey), then rinsed with an air–water syringe for 10 seconds, and dried with air. Before bonding, the enamel was etched for 30 seconds with 38 per cent phosphoric acid gel solution (Etch-Rite; Pulpdent Corporation, Watertown, Massachusetts, USA), rinsed for 20 seconds with water, and dried with an air–water syringe. For each group, 15 brackets were bonded with either Unite or Resinomer and OS+. Following complete curing of the materials at room temperature, the teeth were embedded in acrylic blocks using a mounting jig to align the facial surface of the tooth and the base of the bracket perpendicular to the base of the mould. All the samples were stored in water at 37°C for 24 hours.

All amalgam and bovine teeth specimens were thermocycled 1000 times from 10 to 50°C, 15 seconds in

Table 1 Groups and description of test design.

Group	Bonding surface	Adhesive	Intermediate resin
1	Amalgam	Unite	Reliance Metal Primer
2	Amalgam	Unite	Power Bond OLC
3	Amalgam	Resinomer	One-Step Plus (OS+)
4	Enamel	Unite	—
5	Enamel	Resinomer	OS+

each bath, and 10 seconds travelling between two baths at room temperature.

After thermocycling, debonding was performed with a shearing force using a testing machine (LR50K; Lloyd Intruments Ltd, Fareham, Hants, UK; Figure 1a,b). Each specimen was orientated such that the labial surface of the tooth or the amalgam surface was parallel to the force during the shear strength test. A 50 kg tension cell was used at a crosshead speed of 1 mm/minute. The force to bond failure was recorded electronically, measured in Newtons (N) and converted into megapascals (MPa) with the following equation: Shear strength (MPa) = debonding force (N)/ bracket base area (mm^2) and 1 MPa = 1 N/mm^2.

After debonding each bracket, the tooth and amalgam surface was examined and classified according to the ARI (Årtun and Bergland, 1984). ARI scores range from 0 to 3 (score 0 = no adhesive left on the tooth, score 1 = less than half of the adhesive left on the tooth, score 2 = more than half of the adhesive left on the tooth, and score 3 = all adhesive left on the tooth, with a distinct impression of the bracket mesh).

Statistical analysis

Statistical calculations were performed with GraphPad Prisma V.3 program for Windows (GraphPad Software Inc., San Diego, California, USA). In addition to standard descriptive statistical calculations [mean and standard deviations (SDs)], one-way analysis of variance was used for comparison of the bond test groups. For evaluation of the differences between the groups, a *post hoc* Tukey multiple comparison test was utilized. A chi-square test was used to evaluate ARI data. Statistical significance level was established at $P < 0.05$.

Results

The means and SDs of groups are given in Table 2. When the results were statistically evaluated, the SBS of the first three groups, in which orthodontic bonding to amalgam was performed, were significantly lower than groups 4 and 5 ($P < 0.001$; Table 2). However, there were no statistically significant differences between the amalgam-bonding groups in terms of SBS ($P > 0.05$). For the enamel bonding groups, the mean SBS of brackets bonded with Unite (22.11 MPa) exceeded the mean SBS of brackets bonded with Resinomer and OS+ (19.46 MPa), which was found to be statistically significant ($P < 0.01$).

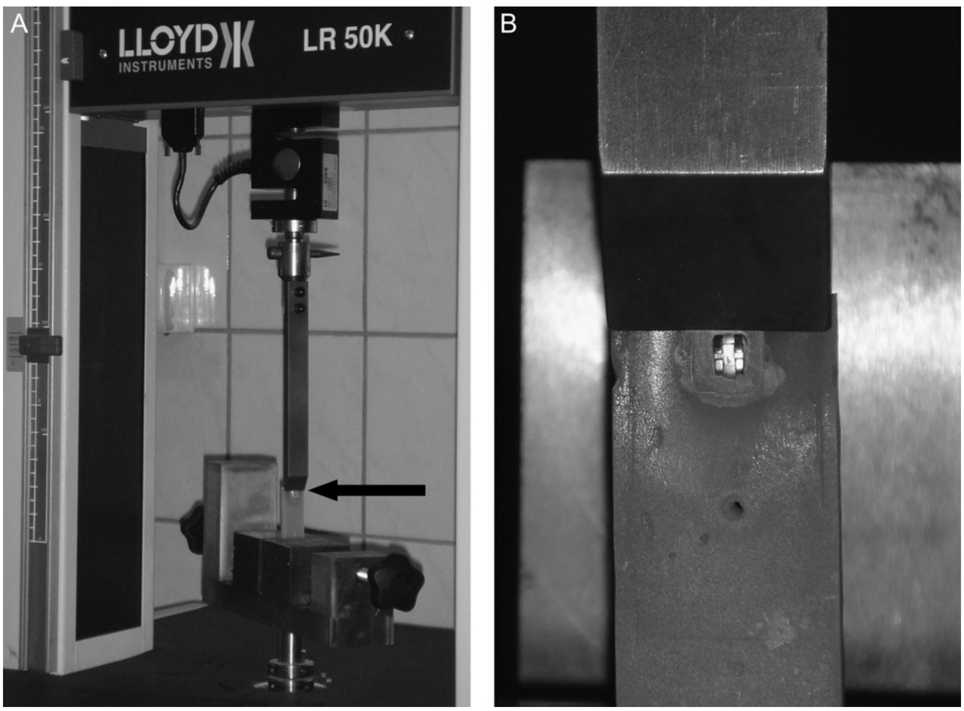

Figure 1 (A) Testing machine with a shear blade (arrow) and acrylic tab mounted on the machine. (B) The shear blade is resting on the bracket bonded to the amalgam.

Table 2 Shear bond strength [megapascals (MPa)], standard deviations (SD), and results of one-way analysis of variance and *post hoc* Tukey's comparison test for groups.

	n	Mean (MPa)	SD
Group 1	15	7.15	1.44*
Group 2	15	5.99	1.26*
Group 3	15	6.41	2.16*
Group 4	15	22.11	1.93*†
Group 5	15	19.46	2.87*†
			$F = 389.4$, $P = 0.0001$

*$P < 0.001$ (groups 1,2,3 < groups 4,5; *post hoc* Tukey comparison test).
†$P < 0.01$ (group 5 < group 4; *post hoc* Tukey comparison test).

Table 3 shows the bonding failure types and percentages. Comparison of the ARI scores ($\chi^2 = 77.59$) indicated that bracket failure mode was significantly different between the amalgam- and enamel-bonding groups ($P < 0.001$). In the amalgam groups, bond failure occurred at all times at the adhesive–amalgam interface. In the enamel groups, three types of bond failure were identified, with the bonding material mostly remaining on the enamel.

Discussion

Bonding orthodontic attachments to artificial tooth surfaces is often challenging and may require modified bonding procedures. As recommended by many investigators (Zachrisson *et al.*, 1995; Gross *et al.*, 1997; Buyukyilmaz and Zachrisson, 1998; Sperber *et al.*, 1999), in the present study, sandblasting with 50 μm aluminium oxide was carried out immediately before bonding to improve adhesion and all specimens were abraded for 3 seconds as air abrasion for more than 4 seconds has been found to be unnecessary (Jost-Brinkmann *et al.*, 1996). Bonding to amalgam restorations involves modification of the metal surface by sandblasting or diamond bur roughening (Graber *et al.*, 2005). Air abrasion with Al_2O_3 leads to improved retentive surfaces than roughening with a diamond bur (Zachrisson *et al.*, 1995). When the effect of different particle size was evaluated, no obvious differences were found between 50 and 90 μm abrasives (Buyukyilmaz and Zachrisson, 1998).

In the present investigation a crosshead speed of 1 mm/minute was chosen because this velocity has been used in most amalgam-bonding studies (Zachrisson *et al.*, 1995, Jost-Brinkmann *et al.*, 1996, Buyukyilmaz and Zachrisson, 1998, Sperber *et al.*, 1999). Furthermore, in a recent study (Klocke and Kahl-Nieke, 2005), it was shown that crosshead speed variation between 0.1 and 5 mm/minute did not seem to influence debonding force measurements or failure mode of brackets bonded to enamel with a composite adhesive.

This *in vitro* study compared the mean SBS of brackets bonded to dental amalgam with different adhesive and intermediate resin combinations. The mean SBS of brackets bonded to amalgam or to enamel was also compared. The results suggest lower mean SBS with amalgam than with enamel, in accordance with the findings of others (Zachrisson *et al.*, 1995; Buyukyilmaz and Zachrisson, 1998; Harari *et al.*, 2000). The mean SBS of the brackets bonded to sandblasted amalgam ranged from 5.99 to 7.15 MPa, in contrast to those bonded to bovine enamel which ranged from 19.46 to 22.11 MPa. Even though there is no universally accepted minimum clinical bond strength, Reynolds (1975) stated that bond strengths ranging from 5 to 8 MPa would be adequate for clinical success. In this *in vitro* study, the mean SBS obtained with various adhesive and intermediate resins in bonding to amalgam seem to be clinically adequate. The superficial oxide layer, present on the metallic surfaces, may be responsible for the high adhesive strength of base metals such as amalgam (Jost-Brinkmann *et al.*, 1996). Because all of the amalgam surfaces in the present study were polished and sandblasted prior to bonding, the enhancer effect of such an oxide layer appears to be minimized. On the other hand, because the oxide layer may be present on old amalgam restorations, the bond strength might be even higher in the oral environment. When using a surface treatment approach besides sandblasting or roughening with a bur, the preserved oxide layer may increase overall bond strength in bonding to amalgam. Clinical investigations are needed to validate the effectiveness of the abovementioned strategies in bonding to amalgam.

In the oral cavity, orthodontic adhesives are routinely subjected to thermal changes and such temperature variations introduce stresses in the adhesive that might influence bond strength. Although some authors have

Table 3 Percentage of adhesive remnant index (ARI) remaining on the enamel/amalgam after debonding.

	Group 1		Group 2		Group 3		Group 4		Group 5		
	n	%	n	%	n	%	n	%	n	%	
ARI 0	15	100.0	15	100.0	15	100.0	0	0.0	0	0.0	
ARI 1	0	0.0	0	0.0	0	0.0	1	6.7	1	6.7	
ARI 2	0	0.0	0	0.0	0	0.0	14	93.3	13	86.7	$\chi^2 = 77.59$
ARI 3	0	0.0	0	0.0	0	0.0	0	0.0	1	6.7	$P = 0.0001$

claimed that the bond strength of composite resin to amalgam is minimally affected by thermocycling (Gross *et al.*, 1997; Buyukyilmaz and Zachrisson, 1998), it has also been shown that the bond strength values of no-mix adhesives reduce following thermocycling (Arici and Arici, 2003). It also decreases the bond strength between resin composite and amalgam (Ozcan *et al.*, 2006). Therefore, in the present study, thermocycling was performed to simulate the oral environment and to eliminate misleading high-bonding values. All amalgam and bovine tooth specimens were thermocycled from 10 to 50°C because it has been reported that the maximum intraoral thermal variation ranges from 18.9 to 48.4°C at silver amalgam restorations (Michailesco *et al.*, 1995).

The strongest bonds to amalgam were obtained with RMP and Unite although there were no statistically significant differences between the amalgam bonding groups ($P > 0.05$). 4-META, the principal molecule present in RMP, is believed to form hydrogen bonds with the oxygen and hydroxyl groups in the metal oxide layer, indicating chemical bonding (Buyukyilmaz and Zachrisson, 1998). In that study where the effectiveness of intermediate resins was evaluated in addition to several other parameters, a higher mean bond strength (10.9 ± 3.5 MPa) was obtained for RMP and a concise group bonded to lathe-cut amalgam. This may be due to the use of different adhesives and tensile rather than SBS testing (Buyukyilmaz and Zachrisson, 1998).

Resinomer yielded comparable bond strengths to RMP in the amalgam groups, whereas in the enamel groups, the mean SBS of brackets bonded with Resinomer and OS+ was higher than the mean SBS of brackets bonded with Unite. This multipurpose resin ionomer and related adhesive (OS+) can be safely used for bonding to amalgam restorations as well to bonding orthodontic brackets. Furthermore, this fluoride-releasing adhesive might be advantageous for orthodontic bonding to enamel in patients with caries susceptibility.

A bovine tooth model was used in this study because the enamel of bovine incisor teeth has been shown to be histochemically similar to human enamel (Nakamichi *et al.*, 1983). Bovine teeth have also been shown to be possible substitutes for human teeth in either dentine or enamel bond testing (Reis *et al.*, 2004). According to Nakamichi *et al.* (1983), adhesion to enamel showed no statistically significant difference between human and bovine teeth. On the other hand, bond strength to bovine enamel has been shown to be weaker than to human enamel (Oesterle *et al.*, 1998). Therefore, the results and the clinical relevance of the present study should be interpreted with caution. The orthodontic bond strengths of the investigated adhesives to bovine enamel might be weaker than to human enamel. Thus, higher bonding values might be achieved clinically.

Orthodontic bond studies should evaluate not only bond strength values but also the location of the bond failure (Oilo, 1993). There are two basic and controversial points of view concerning bond failure mode. According to the first, bond failure at the bracket–adhesive interface or within the adhesive is more desirable than at the adhesive–enamel interface to avoid enamel fracture at the time of debonding (Britton *et al.*, 1990; Bishara *et al.*, 1998). On the other hand, it has also been suggested that a reduced amount of remnant adhesive on the enamel is clinically beneficial because this requires less clean-up after debonding (Sinha *et al.*, 1995; Jost-Brinkmann *et al.*, 1996). In the present study, for all the amalgam bonding groups, ARI scores were 0, indicating a purely adhesive failure at the resin–amalgam interface with no amalgam breakage, in agreement with the findings of Zachrisson *et al.* (1995). On the other hand, in an investigation where bonding to Adlloy-treated amalgam was evaluated, bond values were comparable with bonding to etched enamel (Gross *et al.*, 1997). However, high bond values resulted in fractures within the amalgam during debonding. Consequently, adequate orthodontic bond strength to enamel may be excessive for amalgam.

Conclusion

1. The mean SBS of stainless steel orthodontic brackets bonded to amalgam surfaces with RMP, PB OLC, OS+ intermediate resins, and Unite and Resinomer adhesives was significantly lower than to etched bovine enamel.
2. Bond failure occurred at the amalgam–adhesive interface regardless of the adhesive system and without damage to the amalgam restoration.

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