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The effect of early static loading on the *in vitro* shear/peel bond strength of a 'no-mix' orthodontic adhesive

E. Ching*, P. A. Cook*, N. L. Bubb** and D. J. Wood**
*Orthodontic Department and **Biomaterials Unit, Leeds Dental Institute, UK

SUMMARY This study addressed the question of whether shear and tensile loads applied 15 minutes after bonding metal brackets to enamel affected the shear/peel bond strength of the adhesive.

Ninety standard 0.022-inch stainless steel edgewise premolar mesh-backed brackets were bonded using a no-mix chemical-cured adhesive to 90 teeth, which had been prepared in a standardized manner. After 15 minutes three groups of 30 teeth were subjected to the following regimes: no applied load, tensile static load of 0.77 N (78 g), and shear static load of 0.77 N.

After 14 days storage in 100 per cent relative humidity at 37°C, the shear/peel strength of the adhesive bond was measured using a purpose built jig mounted on a universal testing machine. Shear/peel bond strengths were analysed using Weibull statistics.

The Weibull moduli of the three groups indicated that the adhesive performed consistently despite early static loading. Characteristic strengths were 9.22, 9.27, and 9.05 MPa for the control, tensile, and shear groups, respectively. The findings indicate that static loads (such as tying in of archwires) can be placed on brackets 15 minutes after cementation, without a clinically significant reduction in bond strength of the tested adhesive.

Introduction

It is generally accepted that the bond strength of a setting composite resin adhesive increases with time due to continued polymerization of the resin under the bracket base. Early bond strengths increase at an exponential rate in the first few minutes after cure, slowing down to a gradual rate leading to a steady state (Braem *et al*., 1987; Horn and James, 1993; Chamda and Stein, 1996). However, clinically there is no definite indication of when the plateau stage is reached, although some studies have been carried out to investigate the rate of cure of composite adhesives (Horn and James, 1993; Chamda and Stein, 1996). Millett and Gordon (1994) stated that 'as most orthodontists activate the appliances in the mouth from 10–15 minutes after bonding, the initial bond strength of orthodontic attachments is very important ...'

Few *in vitro* studies have investigated and compared the consequences of loading stresses on a setting adhesive. Ireland and Sherriff (1996) found that a mill recessed bracket bonded with Concise when subjected to a static mass of 78 g (0.77 N) 1 hour after bonding produced a significantly higher shear bond strength than that of a bracket which had not been subjected to prior loading.

The authors repeated their experiment with a no-mix adhesive, Right-On, bonding mesh-based brackets to extracted premolar teeth and found no significant differences in bond strengths. A retrospective analysis of bond failure rates in 60 patients who underwent orthodontic treatment was also conducted. It compared a group of 30 patients, in whom the archwire was tied in 1 hour after bonding, with another group whose brackets were left unloaded for at least a week. Their results suggested that it was safe to tie in the archwires on the same visit as bracket placement, 1 hour after cementation, as there was no difference in bond failure rates (Ireland and Sherriff, 1997).

A search of the literature reveals very few clinical studies showing direct comparisons of bond failure rates between groups where archwires were inserted immediately after cementation and those where the wires were tied in on a separate visit. However, certain articles quote clinical percentage failure rates of an adhesive system and also record the time interval when the first archwire was tied in. It can, therefore, be deduced from those studies that the percentage failure rate quoted represents the clinical failure rate of an adhesive system that has experienced early loading. However, the lack of standardization between studies does not permit direct comparison. Zachrisson (1977) evaluated failure rates of brackets, where the archwires were inserted immediately after bonding. He conducted a long-term evaluation of 705 brackets bonded with Concise in 46 children who underwent edgewise orthodontic treatment. The average treatment time was 17 months. The failure rates were 4–10 per cent for the anterior teeth and first premolars. Gorelick (1977) tied in his archwires 5–10 minutes after cementation. He tested 549 bonded attachments with Concise and observed them for a 12-month period. The minimum failure rate was 4 per cent on the upper anterior teeth and the maximum 7 per cent on the lower premolars. These percentages compare favourably with the *in vivo* bond failure rate of 7.9 per cent recommended by Zachrisson (1985) as the acceptable standard for clinical performance, indicating that the placement of archwires soon after bonding has little detrimental effect on the strength of the setting cement. Both studies used Concise. However, there was no comparison with a control group.

Hence, it appears that the initial bond strength of a setting adhesive and the effect of loading on that strength play a role in determining timing of archwire placement. It was thus the aim of this study to investigate the effect of shear and tensile static loading 15 minutes after bracket placement on the strength of a setting adhesive.

Materials and methods

Testing was carried out where possible following the recommendations of Fox *et al.* (1994). Extracted human premolars were collected from patients under 18 years of age and soft tissue remnants were removed. All teeth had sound enamel and were caries free. Ninety teeth were collected, 45 mandibular and 45 maxillary premolars. Each tooth had one appropriate 0.022-inch stainless steel edgewise premolar mesh backed bracket (Dyna-Lock, 3M Unitek, Bracknell, UK) bonded to its buccal aspect in a standardized manner using a no-mix chemically-cured adhesive, Unite (3M Unitek). To ensure the correct plane of debond, brackets with zero tip and torque were used. Bonding was carried out at an ambient temperature of 24°C using the following protocol:

(1) oil-free prophylaxis with a polishing cup and pumice;
(2) 30-second wash and 30-second dry using a 3-in-1 syringe;
(3) 30-second etch with 37 per cent phosphoric acid;
(4) 30-second wash and dry until frosty white in appearance;
(5) following the manufacturer's instructions, application of primer to enamel;
(6) brackets were seated with firm pressure to minimize the thickness of the resin film;
(7) excess adhesive removed from the periphery of the attachment base using a dental probe.

Fifteen minutes after bond-up, three groups of 30 specimens were subjected to one of the following loading regimes, as shown in Figure 1:

(1) control, i.e. no applied mass;

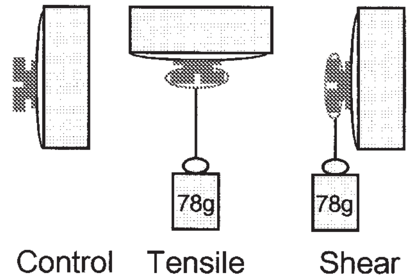

Figure 1 Control, tensile, and shear regimes.

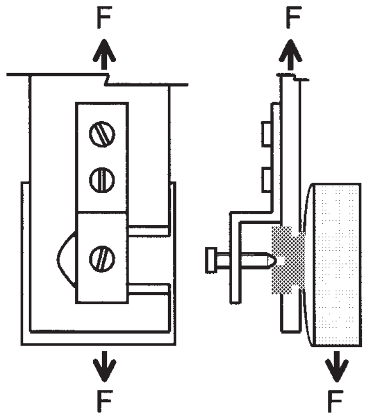

Figure 2 Debonding jig with bracket *in situ*, indicating direction of shear force (F).

(2) static tensile mass of 78 g was attached via nylon thread using elastics positioned in a normal manner;
(3) static shear mass of 78 g was attached via nylon thread looped over the tie wings of the bracket in an attempt to minimize the tensile component of the applied load.

After 14 days the brackets were debonded from the teeth in shear using a universal testing machine (LR10K, Lloyd Instruments Ltd., Fareham, UK; NAMAS certified 990072) fitted with a jig (Figure 2), modified from that used by Littlewood and Redhead (1998). In effect, a lap joint was formed which minimized the peel element of the shear/peel failure. A cross-head speed of 0.1 mm/min and a 500 Newton (± 0.05 per cent) load cell were used. Maximum loads to failure were recorded in Newtons and converted to MPa by dividing the load by the nominal surface area of the bracket.

The site of bond failure was categorized using the adhesive remnant index (ARI) (Årtun and Bergland, 1984) as follows:

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.
Score 3 All adhesive left on the tooth, with distinct impression of the bracket mesh.

Results

Data were analysed using Weibull statistics, which takes into account the fact that it is the lowest, rather than the mean bond strengths that govern failure (Fox and McCabe, 1992). The Weibull distribution is based on the following equation:

$$P_f = 1 - \exp\left[-\frac{(\sigma - \sigma_u)^m}{\sigma_o}\right]$$

where P_f is the probability of failure, σ is stress, σ_u is threshold stress at which the first bond failure occurs (assumed to be zero), σ_o is the characteristic level of stress, and m is the Weibull modulus. A high value of m indicates less scatter of results and, therefore, a more predictable material.

Weibull plots for the three groups are shown in Figure 3. Table 1 gives the values obtained for Weibull moduli, characteristic strengths and

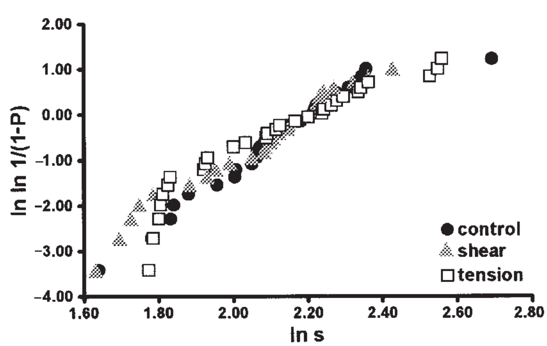

Figure 3 Weibull distribution of data.

Table 1 Weibull moduli and characteristic strengths.

Group	Weibull modulus	Characteristic strength (MPa)	Correlation coefficient
Control	5.27	9.22	0.93
Tensile	4.45	9.27	0.89
Shear	4.99	9.05	0.97

Table 2 Summary of bond failure sites as characterized by ARI.

	Score			
	0	1	2	3
Group				
Control (n = 30)	15	15	0	0
Tensile (n = 30)	13	15	2	0
Shear (n = 30)	11	18	1	0

correlation coefficients, and Table 2 summarizes the bond failure sites according to the ARI.

Discussion

Whilst orthodontic adhesives are required to produce a strong and durable bond to withstand both occlusal and orthodontic forces, they must permit bracket removal at the end of treatment without enamel damage. These aspects require an adhesive with an extensive, but finite amount of strength.

The 'optimum' forces required for orthodontic tooth movement depend on the type of movement desired. Generally, those required for tipping, rotating, and extruding teeth are light, ranging from 50 to 75 g (0.49–0.74 N). For uprighting tooth roots, 75–125 g (0.74–1.23 N) is recommended and for bodily movement of teeth, 100–150 g (0.98–1.47 N; Proffit and Fields, 1993). In this experiment, masses of 78 g (loads of 0.77 N) were chosen to reproduce the experimental masses used by Ireland and Sherriff (1996, 1997). The use of the 78-g masses were justified by the authors as being 'approximately midway in the optimal force range for the various types of tooth movement … most of which might occur during initial alignment.'

The bond strengths obtained in this study compare favourably with results obtained by other authors. Willems *et al*. (1997) tested 22 commercially used orthodontic adhesives and found that, with the exception of Unite, no-mix adhesives did not produce high bond strengths. A characteristic strength of 7.6 MPa was obtained for Unite, which is less than the values obtained for each group in this experiment (9.22, 9.27, 9.05 MPa). One explanation of this finding is that the jig successfully minimized the peel element of failure in this study; however, the different testing conditions in each study must also be considered.

Comparing Weibull moduli between the three test groups in this experiment, the control group was found to have a slightly higher Weibull modulus (5.27) than the tensile (4.99) and shear group (4.45). These differences were so small they are likely to be insignificant clinically. Certainly, Fox *et al*. (1991), when investigating Right-On and Direct found bond failure rates to be similar and yet their respective Weibull moduli were 4.82 and 6.78.

Characteristic bond strengths of 9.22, 9.27, and 9.05 MPa for control, tensile, and shear groups, respectively, differ only by a maximum of 0.22 MPa, and are once again likely to be clinically insignificant. The values suggest that the 'stressed' adhesive will perform as well as 'unstressed' adhesive since a force of equivalent magnitude is needed to fracture the bond in both situations. Generally, bond failures in all groups occurred at the cement-enamel interface, with the vast majority of failures being clean or having less than half of the adhesive remaining on the tooth surface. Since a failure rate of 7.9 per cent has been quoted by Zachrisson (1985) as being clinically acceptable, stresses corresponding to this rate were calculated for each test group. Bond strengths at 7.9 per cent failure were 5.74, 5.49, and 5.29 MPa for control, shear, and tensile test groups, respectively. The differences in these values are again unlikely to be clinically significant.

In orthodontics, there is no consensus concerning the minimum recommended time required for a setting adhesive to remain undisturbed before tying in the archwire. Several authors have attempted to propose suitable time intervals, ranging from 5 minutes (Tavas and Watts, 1979), to 30 minutes (Thanos *et al*., 1979), to 24 hours after cementation (Greenlaw *et al*., 1989). The results of this study indicate that it is 'safe' to load the bracket 15 minutes after cementation using Unite. Clinical studies are needed to test the hypothesis that these *in vitro* results can be applied directly to the clinical situation of tying in archwires.

Conclusions

The findings of this *in vitro* study indicate that a static load (such as tying in an archwire) applied in tension or shear 15 minutes after bonding has no effect on the characteristic strength and Weibull modulus of Unite orthodontic adhesive.

Address for correspondence

Dr D. J. Wood
LDI Biomaterials Unit
Division of Restorative Dentistry
Leeds Dental Institute
Leeds LS2 9LU, UK

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