European Journal of Orthodontics 34 (2012) 493–497
doi:10.1093/ejo/cjr051
Advance Access Publication 19 April 2011

© The Author 2011. Published by Oxford University Press on behalf of the European Orthodontic Society.
All rights reserved. For permissions, please email: journals.permissions@oup.com

Influence of light dose on bond strength of orthodontic light-cured adhesives

Shoko Yoshida, Yasuhiro Namura, Maki Matsuda, Ayano Saito and Noriyoshi Shimizu
Department of Orthodontics, Nihon University School of Dentistry, Tokyo, Japan

Correspondence to: Yasuhiro Namura, Department of Orthodontics, Nihon University School of Dentistry, 1-8-13 Kandasurugadai, Chiyoda ward, Tokyo 101-8310, Japan. E-mail: namura-y@dent.nihon-u.ac.jp

SUMMARY Although the polymerization reaction in light-cured orthodontic adhesive continues for some time after light irradiation, it is unclear whether insufficiently irradiated adhesive develops sufficient bond strength. This *in vitro* study examined the maturation of bond strength after exposure of a variety of light doses. Large metal brackets were bonded to the enamel of 288 bovine mandibular incisors by irradiation at two light intensities (200 and 400 mW/cm^2) and for three exposure times (3, 5, and 10 seconds) using three orthodontic adhesives (TB, OP, and BOB). Shear bond strengths and adhesive remnant indices (ARIs) were determined immediately (T1) and 24 hours after bonding (T2; $n = 8$ in each group). Comparisons were made using the Kruskal–Wallis H-test, the Bonferroni-corrected Mann–Whitney U-test, and the Yates-corrected chi-square test. Bond strengths of the adhesives that showed maturation at low light intensity (200 mW/cm^2) increased by 1.4- to 2.0-fold in 24 hours. An increase in exposure time increased bond strength more than did an increase in light intensity for most orthodontic adhesives. With an exposure time of 3 seconds at 200 mW/cm^2, the ARI scores of TB and OP differed significantly between T1 and T2. Thus, the most acceptable procedure when applying low-dose light intensity to a bracket before the placement of a wire is to increase the exposure time and/or wait for sufficient maturation of bond strength.

Introduction

Adhesive systems for orthodontic brackets have been developed to obtain sufficient bond strength and maximize convenience. However, despite advances in adhesive technology, bond failure may still occur without proper handling. Care is required during bonding (Zachrisson and Büyükyilmaz, 2005) with respect to environmental conditions (Rikuta *et al.*, 2008) to achieve successful bonding and to minimize bond failure during treatment.

Most currently used orthodontic adhesives are light cured (Krishnaswamy and Sunitha, 2007). Such light-curing systems allow sufficient time for bracket positioning and cure the adhesive on demand, which can help prevent contamination saliva. However, light exposure may be of low intensity in areas that are difficult to reach with the tip of the lighting device, such as the molars. This may result in insufficient curing depth and leads to the possibility of bond failure (Wang and Meng, 1992).

Some light-cured resins have a period of bond strength maturation that peaks at 24 hours after irradiation (Oesterle and Shellhart, 2008). Yamamoto *et al.* (2006) reported that bond strength measured immediately after the recommended light irradiation increased by 1.6- to 1.8-fold in 24 hours. Although irradiation of high-cured adhesives at recommended light intensity generates sufficient bond strength with maturation, the degree to which the bond strength of an adhesive irradiated at low light intensity is compensated for by maturation remains unknown. Our study thus compared the maturation of adhesives cured at low light intensity with that of adhesives cured at recommended light intensity and examined whether sufficient maturation occurred after short exposure times.

Materials and methods

Transbond XT paste (components: Bis-GMA, TEGDMA, silane-treated quartz, amorphous silica, etc.; lot No. 5RL) and Transbond Plus self-etching primer (components: methacrylate ester derivative, water, etc.; lot No. 228883A; 3M Unitek, Monrovia, California, USA; abbreviation: TB), Beauty Ortho Bond (primer components: solvent, water, phosphoric acid monomer, etc.; lot No. 120502; paste components: Bis-GMA, TEGDMA, S-PRG filler; lot No. 120503; Shofu, Kyoto, Japan; abbreviation: BOB), and Orthophia LC (primer components: phosphoric acid monomer, etc.; lot No. 070522; paste components: Bis-GMA, TEGDMA, silica filler, agent amplifying the radical, etc.; lot No. 070425; Tokuyama Dental, Tokyo, Japan; abbreviation: OP) were used in this experiment. These orthodontic light-cured adhesive systems were used with tooth surface treatments based on a self-etching primer. All the self-etching primers contain phosphate acid ester monomer, the tooth surface treatment agents of TB and BOB are sixth-generation single-application bonding systems and OP is a seventh-generation single-application bonding system in which all components are mixed together in a single flask (Pithon *et al.*, 2010). A large metal bracket (New Dyna-lock; 3M Unitek) with a surface area of 15.26 mm^2, which was measured at the sides of the bracket base with digital slide calipers (NTD12P-15C; Mitutoyo, Kawasaki, Japan), was used in this study. We used an Eliper Freelight (3M ESPE Dental Products, St Paul, Minnesota, USA) or an Ortholux XT (3M Unitek) adjusted to a light intensity of 200 or 400 mW/cm^2, the light output was measured with a light tester (Jet Light; J. Morita, Osaka, Japan).

This study used 288 bovine mandibular incisors (2–3 years old) instead of human teeth because they are readily obtainable and consistent. After removing the roots of the teeth with a low-speed Proxxon Mini Band Saw (Proxxon, Niersbach, Germany), the pulp chamber of each tooth was filled with cotton to avoid penetrating the embedding media. The labial surfaces of the incisors, which were exposed to facilitate the bonding procedure, were positioned level with and parallel to the margin of the mounting mould. Each tooth crown was mounted in self-curing acrylic resin (Tray Resin II; Shofu) to expose the labial surface and placed immediately in tap water to reduce the temperature increase caused by polymerization. The final finish was accomplished by grinding the labial surface of the crown on wet 600 grit silicone carbide paper to achieve a flat surface. After ultrasonic cleaning with distilled water to remove excess debris, the surfaces were washed and dried with oil-free compressed air.

A mixed solution of the self-etching primer was applied to the enamel surface with a disposable applicator for 3–5 seconds according to the manufacturer's instructions, and the surface was dried with oil-free compressed air. The composite adhesive was applied to the base of the metal bracket and pressed firmly onto the tooth surface. Excess paste was removed from around the border of the bracket base, and the adhesives were light cured for 3, 5, or 10 seconds from each interproximal side.

The samples (n = 8 in each group) bonded under each irradiation condition were tested at two time points (T1, immediately after irradiation and T2, after storage for 24 hours in distilled water at 37°C) with a universal testing machine (5567; Instron, Norwood, Massachusetts, USA) in shear mode at a crosshead speed of 1.0 mm/min. Shear bond strength values were calculated based on the peak load at failure divided by the bracket area. All tests were performed at 23 ± 1°C and relative humidity of 50 ± 5%.

Means, standard deviations, and median values of bond strength were calculated. The data were analysed using the Kruskal–Wallis *H*-test, followed by the Mann–Whitney *U*-test with Bonferroni's correction.

After the shear bond strength test, each specimen was examined under an optical microscope to identify the location of bond failure. The residual adhesive on each tooth was assessed according to the adhesive remnant index (ARI; Årtun and Bergland, 1984). Quantitative analysis of the residual adhesive on the tooth surface was performed visually. Each specimen was given an ARI score according to the amount of adhesive remaining on the tooth surface, as follows: 0, no adhesive remaining; 1, <50% adhesive remaining; 2, ≥50% adhesive remaining; and 3, all adhesive remaining with a distinct impression of the bracket base. The differences in the distribution of ARI scores were analysed using the chi-square test with Yates correction.

Results

The shear bond strengths of each adhesive system are shown in Figures 1 and 2. All bond strengths were dependent on exposure time and light intensity, and all bond strengths at T2 were greater than at T1 under the same experimental conditions. Increased exposure time produced higher bond strength than increased light intensity, and 24 hours maturation produced greater increases in bond strength than increased exposure time. At 200 mW/cm^2 irradiation, all bond strengths of TB and OP were significantly greater at T2 than at T1 ($P < 0.05$). The bond strength of adhesives that showed maturation increased by 1.4- to 2.0-fold in 24 hours with 5 and 10 seconds irradiations and 6.5- to 14.2-fold with 3 seconds irradiation. However, no significant difference in bond strength was detected in BOB between T1 and T2 for 5 and 10 seconds irradiations at 200 mW/cm^2, and the time course for BOB at 200 mW/cm^2 was markedly flatter than at 400 mW/cm^2. The bond strength of all adhesives irradiated at 400 mW/cm^2 increased by 1.8- to 4.0-fold in 24 hours.

The ARI scores of all adhesive systems are shown in Table 1. Increased exposure time resulted in increased residual adhesive on the teeth. Significant differences between T1 and T2 ($P < 0.05$) were observed only for TB and OP with a 3 seconds exposure time at 200 mW/cm^2. No significant difference was found between light intensities or between T1 and T2 for the other conditions.

Discussion

We examined whether maturation of bond strength occurred with irradiation at low light intensity. After a 3 seconds exposure time at 200 mW/cm^2 (T1), the adhesive pastes remained viscous. Additionally, bond strengths were markedly low; they were significantly lower than those at 400 mW/cm^2. We observed a 6.5- to 14.2-fold increase in bond strength with maturation. However, the bond strength achieved under these conditions (3 seconds exposure at 200 mW/cm^2) is unrealistic for clinical use because the brackets would not withstand masticatory forces.

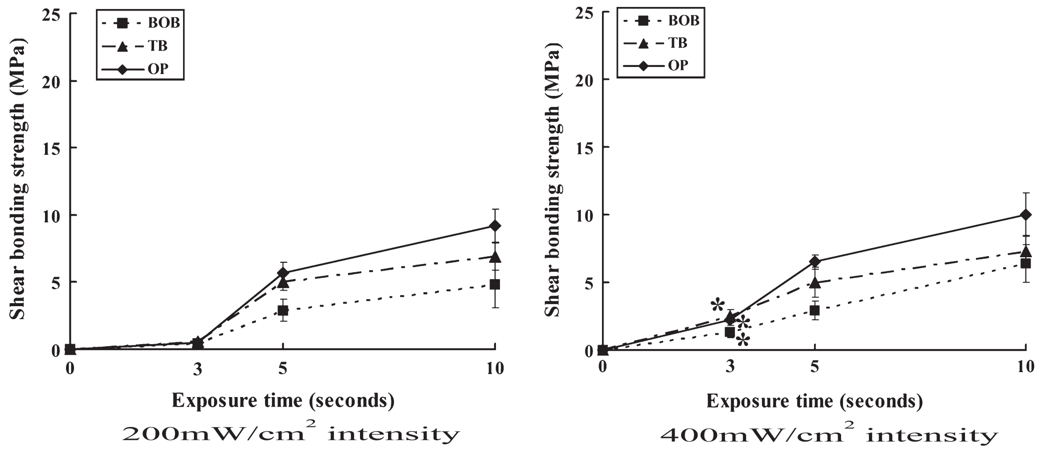

Figure 1 Time courses of changes in shear bond strength for each adhesive in T1. An asterisk indicates that significant difference ($P < 0.05$) in bond strengths of the same adhesive with the same exposure time is detected between light intensities (200 and 400 mW/cm^2).

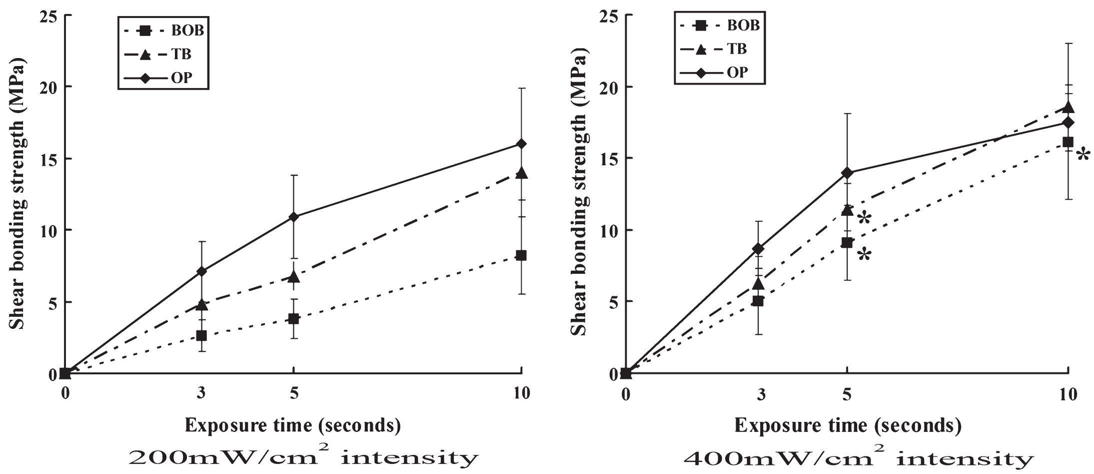

Figure 2 Time courses of changes in shear bond strength for each adhesive in T2. An asterisk indicates that significant difference ($P < 0.05$) in bond strengths of the same adhesive with the same exposure time is detected between light intensities (200 and 400 mW/cm^2). No significant difference in bond strength was detected in BOB between T1 and T2 for 5 and 10 seconds irradiations at 200 mW/cm^2.

After 5 and 10 seconds exposure times, no significant difference in bond strength was detected between light intensities at T1. Bond strength did not differ significantly between T1 and T2 for BOB at 200 mW/cm^2 but did differ significantly at 400 mW/cm^2. These results indicate an unacceptable maturation of bond strength in BOB when it is cured at low light intensity. On the other hand, bond strengths of OP and TB matured significantly (1.4- to 2.0-fold) over 24 hours. When only a low light dose can be applied to a location, such as for posterior molars or impacted teeth which are bonded under surgical conditions, the clinician should wait for 24 hours before applying any orthodontic force or tension to prevent bracket failure. The OP paste is a resin composite mixed with an initiator that is supplied with a commercial resin composite for restorative dentistry (Estelite Flow Quick; Tokuyama Dental), and this amplified exposure of ordinary base components, such as Bis-GMA or filler, to radicals. Uno *et al.* (2006) performed micro-hardness measurements using the nano-indentation method and reported that the curing properties of Estelite Flow Quick were enhanced by inclusion of an initiator that amplified radical production. Our results were consistent with these findings; bond strength in the orthodontic adhesive system containing the initiator was double that of BOB at low light intensity after 24 hours.

The results of the present study indicate that the bond strengths of all adhesives depended on light exposure time i.e. longer irradiation times were associated with high bond strength of the bracket. Silta *et al.* (2005) investigated the capabilities of the latest generation of light-emitting diodes by evaluating bond strength and reported significant differences among light-curing units and polymerization

Table 1 Adhesive remnant index (ARI) of each adhesive system at T1 and T2.

Exposure time (s)	Light intensity (mW/cm^2)	Adhesive score	T1												T2											
			BOB				TB				OP				BOB				TB				OP			
			0	1	2	3	0	1	2	3	0	1	2	3	0	1	2	3	0	1	2	3	0	1	2	3
3	200		1	1	6	0	0	1	7	0	0	0	8	0	7	1	0	0	2	6	0	0*	7	1	0	0*
	400		5	1	1	1	1	2	3	2	5	0	2	1	8	0	0	0	3	5	0	0	8	0	0	0
5	200		4	4	0	0	3	3	2	0	3	5	0	0	8	0	0	0	8	0	0	0	8	0	0	0
	400		7	1	0	0	7	1	0	0	8	0	0	0	8	0	0	0	8	0	0	0	8	0	0	0
10	200		6	2	0	0	0	2	6	0	6	2	0	0	8	0	0	0	8	0	0	0	8	0	0	0
	400		8	0	0	0	4	3	1	0	6	2	0	0	8	0	0	0	8	0	0	0	7	0	0	1

ARI scores: 0, no adhesive left on tooth surface; 1, less than 50% of adhesive left on tooth surface; 2, more than 50% of adhesive left on tooth surface; and 3, all adhesive left on the tooth surface.
*Significant difference in ARI scores of the same adhesive at the same intensity and exposure time between T1 and T2.

times. Staudt *et al.* (2005) reported that bond strength was dependent on light power density. We cured the adhesives using a curing unit adjusted to an intensity of 200 mW/cm^2 (low intensity) and another unit with an intensity of 400 mW/cm^2 (ordinary intensity), and our results indicated that bond strengths were greater at ordinary intensity than at low intensity, although most differences were not statistically significant.

Grandhi *et al.* (2001), Webster *et al.* (2001), Zeppieri *et al.* (2003), Swanson *et al.* (2004), and Fjeld and Ogaard (2006) have cited Reynolds' (1979) suggestion that a minimum bond strength of 60–80 kg/cm^2 (5.9–7.8 MPa) is adequate for clinical use. The bond strength of orthodontic adhesives is affected by the location of the debonding force (Klocke and Kahl-Nieke, 2005) and the testing method used (Powers *et al.*, 1997), and simply comparing Reynolds' suggestion with experimental results may not be appropriate for clinical use. It should also be noted that the bond strengths determined in this experiment cannot be equated with those on human teeth because the strength of brackets bonded to bovine teeth may be somewhat lower than that of brackets bonded to human teeth (Oesterle *et al.*, 1998). We used TB and BOB, which are sixth-generation tooth surface conditioners, and OP, which is a seventh-generation product (Pithon *et al.*, 2010), as experimental materials. With the TB self-etching primer, which has been used in many studies, Öztoprak *et al.* (2007) reported a mean bond strength of 13.76 MPa under conditions of 72 hours storage in water, and Santos *et al.* (2010) reported a mean bond strength of 125.8 N under conditions of 24 hours thermal cycling. Our T1 bond strength data for BOB (6.4 MPa) and TB (7.3 MPa) at 400 mW/cm^2 intensity were comparable to those reported by Yamamoto *et al.* (2006), who used similar experimental conditions (6.7 and 6.5 MPa, respectively). As these bond strengths are the values for bonding that is performed according to the manufacturers' instructions, which are considered relevant to clinical practice. The results of the present study also indicated that the bond strength of BOB with irradiation at 400 mW/cm^2 for 10 seconds achieved a clinically acceptable value at 24 hours due to bond strength maturation. However, bond strength did not differ significantly between T1 and T2 with irradiation at 200 mW/cm^2. It has also been reported that bond strength peaks at 24 hours and subsequently decreases (Oesterle and Shellhart, 2008). The bond strength of BOB with low-intensity irradiation is not likely to be able to withstand orthodontic treatment during 1.5 years or more.

We used the ARI to evaluate the differences for each adhesive after debonding. Fox *et al.* (1994) reported that the site of bond failure provides information about the quality of the bond between the adhesive and the tooth and between the adhesive and the bracket base. In the present study, ARI scores differed significantly between T1 and T2 only for TB and OP at 200 mW/cm^2 light intensity and 3 seconds exposure time. Retief (1974) reported that enamel fractures can occur with bond strengths as low as 13.5 MPa, and failure between the bracket base and the adhesive is desirable in case of high bond strengths so enamel fractures are avoided. However, a number of researchers (e.g. Al Shamsi *et al.*, 2006; Cal-Neto *et al.*, 2006; Faltermeier *et al.*, 2007; Scougall-Vilchis *et al.*, 2007; Montasser *et al.*, 2008) have found less residual adhesive on the enamel when self-etching primers were used, which is considered with this study. Most ARI scores indicated failure between the adhesive and the tooth, and this can be explained by our use of self-etching primers and ground bovine teeth.

Conclusions

The bond strengths of adhesives TB and OP increased 1.4- to 2.0-fold in 24 hours at low light doses: this was due to maturation of those adhesives. But there was no such increase for BOB. An increase in exposure time to light increased bond strength more than an increase in light intensity for most orthodontic adhesives. The results of this *in vitro* study suggest that increasing the exposure time and/or waiting for sufficient maturation of the adhesive are potential ways of increasing bond strengths for brackets which are in locations where it is difficult for the light to reach.

Funding

This study was supported, in part, by a grant from Dental Research Centre, Nihon University School of Dentistry in 2007–2008; a Grant-in-Aid for Young Scientists (B) (20791593).

References

Al Shamsi A, Cunningham J L, Lamey P J, Lynch E 2006 Shear bond strength and residual adhesive after orthodontic bracket debonding. Angle Orthodontist 76: 694–699

Årtun K, Bergland S 1984 Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. American Journal of Orthodontics 85: 333–340

Cal-Neto J P, Carvalho F, Almeida R C, Miguel J A 2006 Evaluation of a new self-etching primer on bracket bond strength in vitro. Angle Orthodontist 76: 466–469

Faltermeier A, Behr M, Mussig D 2007 A comparative evaluation of bracket bonding with 1-, 2-, and 3-component adhesive systems. American Journal of Orthodontics and Dentofacial Orthopedics 132: 144.e1–144.e5

Fjeld M, Ogaard B 2006 Scanning electron microscopic evaluation of enamel surfaces exposed to 3 orthodontic bonding systems. American Journal of Orthodontics and Dentofacial Orthopedics 130: 575–581

Fox N A, McCabe J F, Buckley J G 1994 A critique of bond strength testing in orthodontics. British Journal of Orthodontics 21: 33–43

Grandhi R K, Combe E C, Speidel T M 2001 Shear bond strength of stainless steel orthodontic brackets with a moisture-insensitive primer. American Journal of Orthodontics and Dentofacial Orthopedics 119: 251–255

Klocke A, Kahl-Nieke B 2005 Influence of force location in orthodontic shear bond strength testing. Dental Materials 21: 391–396

Krishnaswamy N R, Sunitha C 2007 Light-emitting diode vs. halogen light curing of orthodontic brackets: a 15-month clinical study of bond failures. American Journal of Orthodontics and Dentofacial Orthopedics 132: 518–523

Montasser M A, Drummond J L, Evans C A 2008 Rebonding of orthodontic brackets. Part I, a laboratory and clinical study. Angle Orthodontist 78: 531–536

Oesterle L J, Shellhart W C 2008 Effect of aging on the shear bond strength of orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 133: 716–720

Oesterle L J, Shellhart W C, Bellanger G K 1998 The use of bovine enamel in bonding studies. American Journal of Orthodontics and Dentofacial Orthopedics 113: 514–519

Pithon M M, dos Santos R L, Ruellas A C, Sant'Anna E F 2010 One-component self-etching primer: a seventh generation of orthodontic bonding system? European Journal of Orthodontics 32: 567–570

Powers J M, Kim H B, Turner D S 1997 Orthodontic adhesives and bond strength testing. Seminars in Orthodontics 3: 147–156

Retief D H 1974 Failure at the dental adhesive-etched enamel interface. Journal of Oral Rehabilitation 1: 265–294

Reynolds I R 1979 A review of direct orthodontic bonding. British Journal of Orthodontics 2: 171–179

Rikuta A *et al.* 2008 Influence of environmental conditions on orthodontic bracket bonding of self-etching systems. Dental Materials Journal 27: 654–659

Scougall-Vilchis R J, Yamamoto S, Kitai N, Hotta M, Yamamoto K 2007 Shear bond strength of a new fluoride-releasing orthodontic adhesive. Dental Materials Journal 26: 45–51

Silta Y T, Dunn W J, Peters C B 2005 Effect of shorter polymerization times when using the latest generation of light-emitting diodes. American Journal of Orthodontics and Dentofacial Orthopedics 128: 744–748

Staudt C B, Mavropoulos A, Bouillaguet S, Kiliaridis S, Krejci I 2005 Light-curing time reduction with a new high-power halogen lamp. American Journal of Orthodontics and Dentofacial Orthopedics 128: 749–754

Swanson T, Dunn W J, Childers D E, Taloumis L J 2004 Shear bond strength of orthodontic brackets bonded with light-emitting diode curing units at various polymerization times. American Journal of Orthodontics and Dentofacial Orthopedics 125: 337–341

Uno S, Morigami M, Sugizaki J, Yamada T 2006 Curing characteristics of a flowable resin composite "Estelite Flow Quick". Adhesive Dentistry 24: 130–135(in Japanese)

Wang W N, Meng C L 1992 A study of bond strength between light- and self-cured orthodontic resin. American Journal of Orthodontics and Dentofacial Orthopedics 101: 350–354

Webster M J, Nanda R S, Duncanson M G Jr, Khajotia S S, Sinha P K 2001 The effect of saliva on shear bond strengths of hydrophilic bonding systems. American Journal of Orthodontics and Dentofacial Orthopedics 119: 54–58

Yamamoto A, Yoshida T, Tsubota K, Takamizawa T, Kurokawa H, Miyazaki M 2006 Orthodontic bracket bonding: enamel bond strength vs. time. American Journal of Orthodontics and Dentofacial Orthopedics 130: 435.e1–435.e6

Zachrisson B U, Büyükyilmaz T 2005 Bonding in orthodontics. In: Graber T M, Vanarsdall R L, Vig K W (eds). Orthodontics: current principles techniques, 4th edn. Elsevier Mosby, St Louis, pp. 579–659