The effects of primer precuring on the shear bond strength between gold alloy surfaces and metal brackets

Won-Jun Shon*, Tae-Woo Kim**, Shin-Hye Chung* and Min-Ho Jung**
*Departments of Conservative Dentistry and **Orthodontics, School of Dentistry and Dental Research Institute, Seoul National University, Seoul, Korea

Correspondence to: Dr Min-Ho Jung, Private practice, 3rd Floor, Tae-nam Building, 72-3 Cham-won Dong, Seo-cho Gu, Seoul 137-909, Korea. E-mail: Fortit@chol.com

SUMMARY The objective of this study was to investigate the effects of precuring of primer coated on bracket bases on the strength of bonds between metal brackets and gold alloy. Square type III gold alloy plates were sandblasted with 30 m silicon dioxide. After silica coating, excessive particles were removed gently with air. Silane was then applied, and maxillary central incisor metal brackets were bonded to each conditioned alloy surface with Transbond XT. Half of the specimens were precured at the bracket base after primer coating and the other half was not precured before bonding to the alloy surface. After bracket positioning, samples were cured using a light emitting diode (LED) for 40 seconds. Shear bond strengths were tested and adhesive remnant index (ARI) was evaluated after 1 hour and 24 hours. The primer precuring and 24 hours group exhibited highest bond strength (12.53 MPa) and the no precuring and 1 hour group showed lowest bond strength (5.58 MPa). Precured groups showed lower ARI scores. Due to the shallow curing depth of LED light and inhibition of transillumination at the metal surface, primer precuring at the bracket base is required for secure bracket bonding on gold alloy surfaces using LED curing units.

Introduction

The proportion of adults in orthodontic patients is more than 20 per cent in the USA (Keim *et al.*, 2008) and in some metropolitan areas, the percentage is reaching more than 60 per cent (Jung, 2009). For this reason, the need to bond orthodontic brackets onto various alloys has increased. In adult patients, metallic surfaces are commonly encountered on the labial surfaces of molars and lingual surfaces of anterior teeth. To obtain proper bond strength on such surfaces, pretreatment of the alloy surface is required prior to bracket bonding, and surface roughening is a prerequisite for achieving sufficient bracket-to-alloy bonding (Jost-Brinkmann and Böhme, 1999; Zachrisson, 2000).

Silane coupling agents are widely accepted as adhesion promoters in dentistry and enhance bond strength by promoting a chemical bond between resin monomer and metal (Pleuddemann, 1982) or silica (Sung and Kang, 2009; Trakyali *et al.*, 2009). Silane molecules react with carbon double bonds on the monomers in resin composites during free radical polymerization (Gladwin and Bagby, 2000).

A recently introduced technique based on silicoating enhances mechanical retention by sandblasting, as well as chemicophysical bonding between composite resin and alloy with a silane coupling agent (Nergiz *et al.*, 2004). Metal surfaces are abraded with 30 m grain size aluminium oxide modified with silicic acid with an intraoral sandblaster. The particles form a reactive silica layer on the substrate. Silane must then be applied to allow for chemical bonding with the adhesive resin (Hansson, 1990; Jung 2005). Silica

coatings are used in many dental applications including intraoral repair of fractured ceramic restorations involving metal exposure (Schneider *et al.*, 1992; Özcan and Niedermeier, 2002), bracket bonding at the metal surface (Nergiz *et al.*, 2004), and treatment of ceramic bracket bases for rebonding (Toroglu and Yaylali, 2008).

Light emitting diode (LED) might provide a shallower depth of cure than conventional halogen lights (Stahl *et al.*, 2000), but this depth is adequate for bonding orthodontic brackets to the enamel surface (Bishara *et al.*, 2003; Krishnaswamy and Sunitha, 2007). Greenlaw *et al.* (1989) has suggested that free radicals are produced where light exposure is available and diffusion of these free radicals takes time to polymerize the resin under the bracket base. Because curing light cannot travel through alloy or metal and the amount of free radicals produced and degree of conversion seem to be much less when bonding on the metal surface than on the enamel surface.

We assumed that precuring on the base of the metal bracket would produce free radicals at the entire bracket base and increase the bond strength between bracket and metal surface. The objective of this study was to investigate the effects of bracket base precuring on the bond strength between metal brackets and gold alloy.

Materials and methods

A total of 30 square-shaped type III gold alloy (Au 50 per cent, Pd 5 per cent, Ag 32.5 per cent, Cu 11.45 per cent,

Argen Co., San Diego, California, USA) plates 1.5 mm thick and 10 mm wide and long were used in this study. Alloy plates were embedded in cold curing acrylic (Leocryl, Leone, Sesto Fiorentino, Italy) and placed in acrylic rings (Taejin Acyrilic, Seoul, Korea). Each plate was oriented so that its surface would be parallel to the force during the shear bond test. The specimens were then prepared for testing using different surface-conditioning methods.

For the pilot study, all the alloy plates were sandblasted with an intraoral sandblaster (Air-Flow Handy II, EMS Corp., Dallas, Texas, USA) filled with 30 m silicon dioxide (Cojet-Sand; 3M ESPE, Seefeld, Germany). Following the manufacturer's instructions, the abrasive was applied vertically to the plates from a distance of 10 mm with 2.5 bar pressure for 15 seconds. After silicoating, excessive particles were removed gently with air. Silane (ESPE-Sil; 3M ESPE) was then applied to the specimens and air-dried for 1 minute.

After surface conditioning, maxillary central incisor metal brackets (item number: 017-875, Victory, 3M Unitek, Monrovia, California, USA) were bonded to each conditioned alloy surface with Transbond XT (3M Unitek). The average surface area for the bracket base was 10.56 mm^2. Thin uniform coat of Transbond XT primer was applied to the bracket base and then resin adhesive was applied. The bracket was then positioned on the prepared alloy surface with sufficient pressure to squeeze out excess adhesive, which was removed carefully.

The first 10 brackets were light cured (Ortholux LED curing light; 3M Unitek) for 20 seconds and the second and third group of ten brackets were cured for 30 and 40 seconds, respectively. The manufacturer recommends holding the light 1–2 mm above the bracket and to cure from the mesial and distal edges for equal amounts of time on each side. A minimum light intensity of at least 2000 mW/cm^2 was verified by a curing radiometer (Demetron 100; Demetron Research, Danbury, Connecticut, USA).

After bonding, the specimens were stored in water at 37°C for 24 hours before bond strength testing. Shear bond strengths (SBSs) were determined with a universal testing device (LF Plus; Ametek, Albany, New York, USA). For shear testing, the specimens were secured in the lower jaw of the machine and the bracket base of the sample was oriented parallel to the direction of the shear force. The specimens were stressed with a crosshead speed of 1 mm/minute. The maximum load necessary to debond was recorded.

Although some researchers showed that 20 seconds of LED curing was adequate in the orthodontic bonding procedure (Swanson *et al.*, 2004), 20 and 30 seconds groups showed too low bond strength [average SBS were 1.12 ± 0.47 and 2.43 ± 1.02 MPa, which were significantly lower than SBS (12.18 ± 2.39 MPa) of 40 seconds group] considering that the clinically recommended minimum

bond strength is 6–8 MPa (Reynolds, 1975). Therefore, 40 seconds of curing time was chosen for the main experiment.

After bond strength testing, all the alloy surfaces were polished. Polished specimens were cleaned for 10 minutes in an ultrasonic bath (Bransonic, Ultrasonic Cleaner, Shelton, Connecticut, USA) containing ethylacetate and air-dried with oil-free air.

Because two factors (primer procuring and test timing) should be evaluated, we assigned alloy plates to four groups. Twelve alloy plates were treated as previously described and the brackets were bonded as the pilot study. After 40 seconds of photopolymerization, the specimens were stored in water at 37°C for 1 hour using a thermostatic chamber and then SBS was measured. Other 12 specimens were prepared in same manner and stored in water at 37°C for 24 hours. SBS testing was also completed for these samples.

In third and fourth group, the preparation methods of the specimens and SBS measurements were as the same as in first and second group except for the inclusion of the thin coating of Transbond XT primers on the bracket base and LED light curing for 10 seconds before adhesive application and bracket positioning were done. Table 1 shows the characteristics of surface-conditioning methods and test timing.

After debonding, all samples were examined under ×10 magnification to assess adhesive remnants on alloy surface using the adhesive remnant index (ARI) system (Bishara *et al.*, 1999). The ARI scale has a range of 5–1. The ARI scores were also used as a more complex method of defining the site of bond failure between the substrate, the adhesive, and the bracket base. The ARI assessment and image capture were done with the Zeiss OPMI 111 microscope (Mednet Locator Inc., Memphis, Tennessee, USA).

After sandblasting with Co-jet and after silane application, specimens were sputter-coated with carbon evaporation (SCD-005; Leica Microsystems, Wetzlar, Germany) and examined with a ×1000 magnification by using a scanning electron microscope (SEM; JEOL JSM-6380, Akishima, Japan).

The language R was used to perform the data analysis. After checking the normality assumption and the equality

Table 1 Shear bond strength (SBS) (MPa, mean ± standard deviation) of the specimens after 1 or 24 hour.

Surface conditioning	Time (h)	N	SBS (MPa)	Significance
No precuring	1	12	5.58 ± 2.10^A	No precuring < primer
	24	12	8.98 ± 2.44^B	precuring*
Primer precuring	1	12	11.18 ± 3.18^C	1 < 24 h*
	24	12	12.54 ± 2.45^C	

*$P < 0.001$ significant result after two-way analysis of variance; the same superscripts indicate the homogenous subsets after Student–Newman–Keuls multiple comparisons test ($P < 0.05$).

of variance, two-way analysis of variance (ANOVA) was implemented to deduce the existence of significant influential factors upon the SBS among the two variables: primer precuring and testing time. Differences among the groups were assessed using Student–Newman–Keuls multiple comparisons test. The Fisher's exact test was used to examine whether there were differences in the ARI between the groups.

Results

Table 1 displays the mean and standard deviation values of SBS associated with surface-conditioning methods and test timing. The SBS value of each group was 5.58, 8.98, 11.18, and 12.53 MPa respectively. Two-way ANOVA revealed significant differences ($P < 0.05$) between groups, depending on the surface-conditioning method. There was no interaction effect and significantly greater SBSs were observed in primer precuring groups. The precuring and 24 hour storage group showed highest SBS. The 1 hour and 24 hour group did not show statistically significant differences when the bracket base was precured. Figure 1 shows distribution of the SBS values in relation to precuring and time factor.

The no precuring and 1 hour group showed significantly higher ARI score in the pair-wise Fisher's exact tests than primer precuring groups (Table 2). In this group, more than half of the specimens (7/12) showed small amount of uncured resin adhesive remnants on alloy surface or bracket base macroscopically. Surface characteristics of the specimens are shown in Figure 2 and an uncured portion of the adhesive can be observed. All the uncured portion is located around the centre of the alloy surface or bracket base (Figure 2B).

SEM photographs show gold alloy surfaces after polishing and after silicoating procedure (Figure 3). After silane application, the surface looks similar but irregularities seem to be lessened.

Discussion

When a metal bracket is bonded to an alloy surface, the transillumination effect (Cheng *et al.*, 1989) cannot occur. So if the conventional light curing method (curing from the mesial and distal direction) is used in this situation, free radicals are produced only at the periphery where light exposure is available. For this reason, some of the specimens in the no precuring and 1 hour group showed uncured adhesive remnant at the central portion as in Figure 2B. Such a surface characteristic showed insufficient light penetration between metal bracket base and gold surface. In the precuring and 1 hour group, no specimen showed uncured resin remnants. Incomplete polymerization has been associated with bonding failures and inferior physical properties of light-cured composite materials.
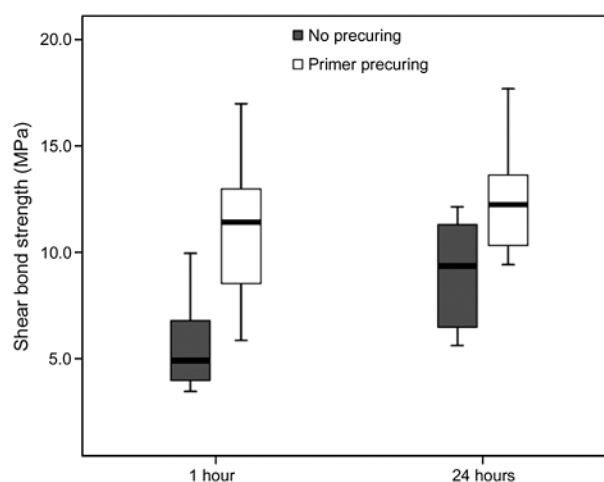

Figure 1 Box plot distribution of the shear bond strength by the precuring and time factors.

Table 2 Frequency distribution of the adhesive remnant index (ARI) scores.

Surface conditioning	Time (h)	N	ARI scores*				
			1	2	3	4	5
no precuring	1[a]	12	0	2	6	4	0
	24[ab]	12	0	2	8	2	0
primer precuring	1[b]	12	3	3	5	1	0
	24[b]	12	5	4	3	0	0

The same superscripts indicate no statistically significant difference between the groups after pair-wise Fisher's exact tests.
*ARI scores: 5 = no composite remained on the specimen; 4 = less than 10 per cent of composite remained on the specimen surface; 3 = more than 10 per cent but less than 90 per cent of the composite remained on the specimen; 2 = more than 90 per cent of the composite remained on the specimen; 1 = all the composite, with an impression of the bracket base, remained on the specimen.

Bradburn and Pender (1992) recommended precuring on the mesh base of the metal bracket to increase bond strength. Bracket base precuring can increase the amount of free radicals on the bracket base and SBS can be increased by this procedure in alloy-bracket bonding. In our experiment, 10 seconds of precuring could significantly increase the bond strength.

Conventional bonding procedures at the enamel surface using QTH and Transbond XT did not show significant bond strength increases during 30 minutes to 24 hours after curing (Minick *et al.*, 2009) and when using plasma arc light and Transbond APC also showed no statistically significant bond strength increases during 30 minutes to 24 hours after curing (Oesterle *et al.*, 2001). Our results also showed nonsignificant bond strength improvement between 1 and 24 hours in the base precuring group but a significant
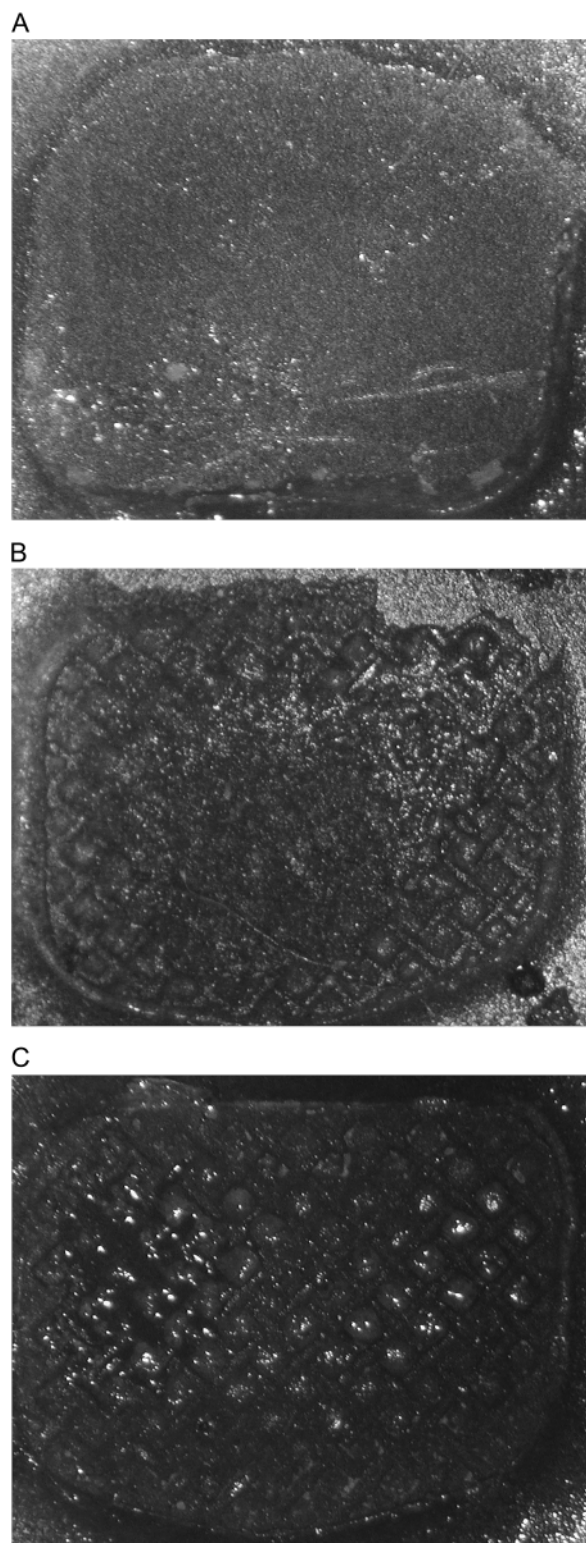
Figure 2 Specimens' surface after bond strength testing. A: a specimen in group 2, adhesive remnant index (ARI) score 4; B: a specimen in group 1, ARI score 3, uncured adhesive at the centre; C: a specimen in group 4, ARI score 1.

increase was found in the no precuring group. Even in the precuring groups, there was a mild increasing tendency as time goes by and it seems to be related to the diffusion
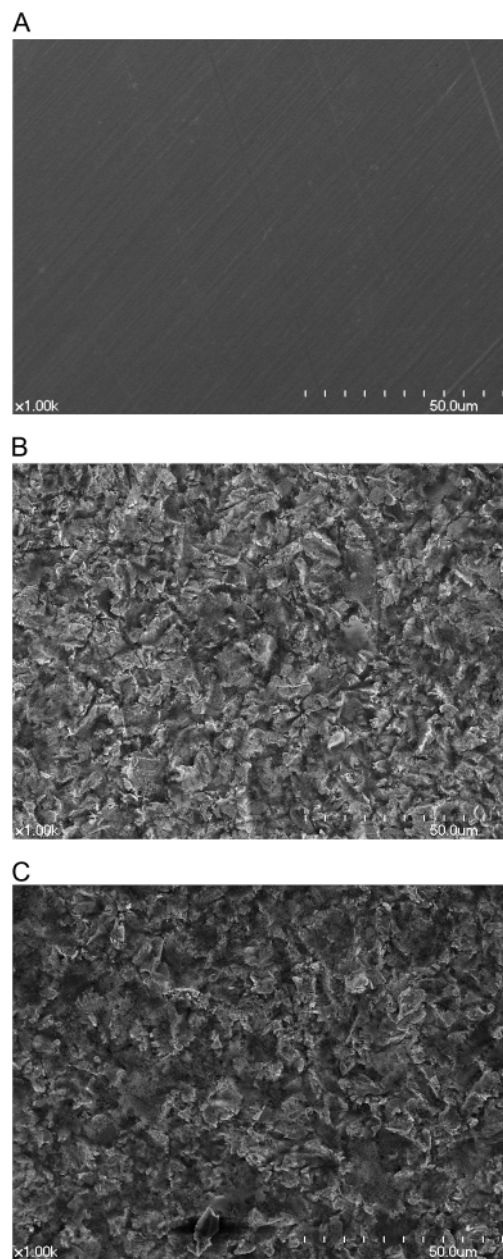

Figure 3 Scanning electron micrographs of specimens at ×1000 magnification. After polishing (A), after sandblasting (B) and after silane application (C).

of free radicals and increased polymerization with time (Greenlaw *et al.*, 1989).

The application of a silicoating to an alloy is an important advance in adhesive bonding of composites to metal because silica coating can reduce the importance of the alloy composition and its oxide formation which is critical in classical bonding procedure using metal primer (Schneider *et al*., 1992; Jung *et al*., 2010). But there are some difficulties in using it. The limited shelf life (Jung, 2005), the mess created by sand in the mouth, and

sensitivity to humidity are the main difficulties. In humid conditions, silanized interfaces seem to be unstable, and the silane bond was found to deteriorate under atmospheric moisture (Nergiz *et al.*, 2004). So using a rubber dam is usually recommended during the silica coating procedure and it seems to be beneficial to keep drying the silanized surface using air.

The primer precuring groups showed a tendency to lower ARI scores than those of the no precuring groups. That means, the adhesive resin showed stronger adhesion to the alloy surface after primer precuring. Because weakest bond between bracket base and alloy surface is usually found in the adhesive–alloy interface, this type of failure site is more favourable for stronger bonding.

After silicoating procedure, a lot of minor irregularities and sharp edges can be seen in the SEM photograph (Figure 3B). After silane application (Figure 3C), surface irregularities seem to be lessened and edges become round. It seems to be the effect of silane coating.

It should be emphasized that there are differences between *in vitro* and *in vivo* bond strengths. Direct transfer of this value to clinical situations is not universally accepted since bond strength can be influenced by many factors (Zachrisson, 2000). Even though the clinical relevance of *in vitro* studies is limited, such studies are essential in testing recently developed products to set up clinical guidelines.

Conclusions

Precuring of the bracket base may significantly increase the SBS of metal brackets to gold alloy surfaces. Primer precuring is required for secure metal bracket bonding on alloy surfaces using LED curing units.

Acknowledgement

References

Bishara S E, Ajlouni R, Oonsombat C 2003 Evaluation of a new curing light on the shear bond strength of orthodontic brackets. Angle Orthodontist 73: 431–435

Bishara S E, VonWald L, Olsen M E, Laffoon J F 1999 Effect of time on the shear bond strength of glass ionomer and composite orthodontic adhesives. American Journal of Orthodontics and Dentofacial Orthopedics 116: 616–620

Bradburn G, Pender N 1992 An in vitro study of the bond strength of two composites used in the direct bonding brackets to molars. American Journal of Orthodontics and Dentofacial Orthopedics 102: 418–426

Cheng L, Ferguson J W, Jones P, Wilson H J 1989 An investigation of the polymerization of orthodontic adhesives by the transillumination of tooth tissue. British Journal of Orthodontics 16: 183–188

Gladwin M, Bagby M 2000 Direct polymeric restorative materials. In: McGrew L (ed). Clinical aspects of dental materials. Lippincott Williams & Wilkins, Philadelphia, pp. 55–56

Greenlaw R, Way D C, Galil K A 1989 An in vitro evaluation of a visible light-cured resin as an alternative to conventional resin bonding systems. American Journal of Orthodontics and Dentofacial Orthopedics 96: 214–220

Hansson O 1990 Strength of bond with Comspan Opaque to three silicoated alloys and titanium. Scandinavian Journal of Dental Research 98: 248–256

Jost-Brinkmann P G, Böhme A 1999 Shear bond strengths attained in vitro with light-cured glass ionomers vs composite adhesives in bonding ceramic brackets to metal or porcelain. Journal of Adhesive Dentistry 1: 243–253

Jung M H 2005 Direct bonding with composite resin; review. Journal of Korean Foundation for Gnatho-Orthodontic Research 7: 61–117

Jung M H 2009 Current trends in orthodontic patients in private orthodontic clinics. Korean Journal of Orthodontics 39: 36–42

Jung M H, Chung S H, Shon W J 2010 Shear bond strength between gold alloy and orthodontic metal bracket using LED curing light. Korean Journal of Orthodontics 40: 27–33

Keim R G, Gottlieb E L, Nelson A H, Vogels D S 3rd 2008 2008 JCO study of orthodontic diagnosis and treatment procedure: part 2 breakdowns of selected variables. Journal of Clinical Orthodontics 42: 699–710

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

Minick G T, Oesterle L J, Newman S M, Shellhart W C 2009 Bracket bond strengths of new adhesive systems. American Journal of Orthodontics and Dentofacial Orthopedics 135: 771–776

Nergiz I, Schmage P, Herrmann W, Ozcan M 2004 Effect of alloy type and surface conditioning on roughness and bond strength of metal brackets. American Journal of Orthodontics and Dentofacial Orthopedics 125: 42–50

Oesterle L J, Newman S M, Shellhart W C 2001 Rapid curing of bonding composite with a xenon plasma arc light. American Journal of Orthodontics and Dentofacial Orthopedics 119: 610–616

Özcan M, Niedermeier W 2002 Clinical study on the reasons for and location of the failures of metal-ceramic restorations and survival of repairs. International Journal of Prosthodontics 15: 299–302

Pleuddemann E P 1982 (ed). Nature of adhesion through silane coupling agents. In: Silane Coupling Agents, Plenum Press, New York, pp. 111–139

Reynolds I R 1975 A review of direct orthodontic bonding. British Journal of Orthodontics 2: 171–178

Schneider W, Powers J M, Pierpont H P 1992 Bond strength of composites to etched silica-coated porcelain fusing alloys. Dental Materials 8: 211–215

Stahl F, Ashworth S H, Jandt K D, Mills R W 2000 Light emitting diode (LED) polymerisation of dental composites: flexural properties and polymerisation potential. Biomaterials 21: 1379–1385

Sung J Y, Kang K H 2009 Shear bond strength of rebonded ceramic brackets. Korean J Orthodontics 39: 234–247

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

Toroglu M S, Yaylali S 2008 Effects of sandblasting and silica coating on the bond strength of rebonded mechanically retentive ceramic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 134: 181e1–181e7

Trakyali G, Malkondu Ö , Kazazoğlu E, Arun T 2009 Effects of different silanes and acid concentrations on bond strength of brackets to porcelain surfaces. European Journal of Orthodontics 31: 402–406

Zachrisson B U 2000 Orthodontic bonding to artificial tooth surfaces: clinical versus laboratory findings. American Journal of Orthodontics and Dentofacial Orthopedics 117: 592–594