File ID	125275
Filename	Chapter 6: The influence of dynamic fatigue loading on the separate components of the bracket-cement-enamel system
Version	Final published version (publisher's pdf)

SOURCE (OR PART OF THE FOLLOWING SOURCE):

Type	Dissertation
Title	A better understanding of orthodontic bracket bonding
Author	T.J. Algera
Faculty	Faculty of Dentistry
Year	2009
Pages	127

FULL BIBLIOGRAPHIC DETAILS:
http://dare.uva.nl/record/294720

CHAPTER 6

The influence of dynamic fatigue loading on the separate components of the bracket-cement-enamel system

Am J Dent 2008 21:239-243

6.1 Abstract

The aim of this study is to evaluate the influence of cyclic loading and the type of adhesive on the shear bond strength of the bracket-cement-enamel bond.

The materials studied are: Transbond XT (a Bis-GMA resin composite cement, 3M-Unitek), Fuji Ortho LC (a resin modified glass ionomer cement, GC), and Fuji IX Fast (a conventional glass ionomer cement, GC). The Shear Bond Strength (SBS) and the Shear Bond Fatigue Limits (SBFL) are determined after 72 hours storage in 37 ºC water for the cement itself, the button-cement interface, the cement-enamel interface, and the bracket-cement-enamel system. The SBFL is determined with the aid of the 'staircase method' at 10,000 cycles. The results are analyzed using ANOVA and Tukey HSD *post hoc* test ($p < 0.05$).

ANOVA shows significant differences between the SBS of the materials. Fatigue is observed in all substrate combinations, with the exception for the Fuji IX Fast cement-enamel and the Fuji Ortho LC bracket-cement-enamel combinations.

Using SBS alone for evaluating and predicting the bond strength properties of the bracket-cement-enamel system can give interpretation failures, because materials providing high initial strength do not always show the best fatigue resistance.

6.2 Introduction

Bonding of mesh based brackets to teeth is an important step in an orthodontic treatment with fixed appliances. The bracket-cement-enamel system has several variables that have to be controlled during the bonding and debonding procedures. Resin composite is the material of first choice nowadays because of their good and predictable bonding properties. However, the material also has some disadvantages such as the micro mechanical invasive adhesion to enamel and their ability to host bacteria. Glass ionomer based cements can be a valuable alternative, because of their fluoride releasing property and their ability to bond chemically to enamel.(1) However, the bond strength of conventional glass ionomers seems too weak for clinical use.(2)

The *in vitro* bond strength of a bracket to a tooth has been studied extensively. These studies are difficult to compare due to large variation in the design of the test, substrate, type and size of the bracket, and the cement used. Nevertheless, according to the literature, the recommended bond strength of the bracket-cement-enamel system varies from 2.9 to 10.0 MPa.(3-6) In contrast to the numerous *in vitro* studies only a few *in vivo* shear bond strength studies are reported. Pickett *et al.* compared the *in vitro* shear strength with the *in vivo* shear strength after an average of 23 months of treatment. The *in vitro* shear bond strength was 12.8 (3.1) MPa, while the *in vivo* value was only 5.5 (2.2) MPa. Saliva, acid, masticatory forces, the *in vivo* bonding procedure, and forces of the orthodontic treatment were suggested as possible explaining factors for the reduced bond strength.(4)

Various studies showed a negative effect of water and saliva on the strength of composites, resin modified glass ionomer cements (RMGICs), and conventional glass ionomer cements (GICs).(7-9) Beside this deterioration of the cements as a result of water or saliva, the bracket-cement-enamel system is also exposed to repeated mechanical loading and stresses induced by temperature changes.(10) As a result of the ever changing balance between functional forces and the resistance of the fixed appliance, jiggling forces have a weakening effect on the bond strength. These forces can initiate micro-cracks inside the brittle adhesive, concentrated around defects located at the surfaces or inside the material. Propagation of these cracks is a slow process that weakens the system further. Failure occurs often when a peak stress acts on the bracket. Various studies showed the effect of repeated mechanical loading on the strength of composites and enamel and dentine bonding systems. To our knowledge only one study based on repeated mechanical loading in relation to the bracket-cement-enamel system is reported.(11)

The purpose of this study was to investigate the effect of repeated mechanical loading on the bracket-cement-enamel system using three different types of cement, e.g. resin composite, RMGIC, and GIC. Therefore the initial shear bond strength (SBS) and the shear bond fatigue limit (SBFL) after 10,000 cycles were measured for each of these cements. In addition to this the initial shear bond strength and shear bond fatigue limit of the cement-enamel, cement-only, and button-cement combinations were determined.

6.3 Materials and Methods

Material handling

The three materials used in this study are listed in Table 6.1 by product name, manufacturer, cement type, batch number, and expiry date. All cements were handled according to the manufacturer's instructions. Fuji IX Fast was applied to air dried enamel without performing a conditioning step. Before bonding with Fuji Ortho LC conditioning took place with 10% polyacrylic acid (GC Dentin Conditioner, GC Corp., Tokyo, Japan) for 20 s. The conditioner was rinsed where after air drying took place. Prior to bonding with Transbond XT, 35% phosphoric acid (Ultradent Products, South Jordan, Utah, USA) was applied on the enamel for 30 s, followed by rinsing, air drying, and application of adhesive primer (3M Unitek, Monrovia, Ca, USA). Curing of the light activated components was performed with an Elipar Trilight curing unit (3M-ESPE Dental Products, Seefeld, Germany).

Table 6.1 Cements used in this study.

Material	Manufacturer	Cement type	Batch nr	Exp. Date
Fuji IX Fast	GC Corporation Tokyo, Japan	Conventional glass ionomer cement	0506083	2007-06
Fuji Ortho LC	GC Corporation Tokyo, Japan	Resin modified glass ionomer cement	0309253	2005-09
Transbond XT	3M-Unitek, Monrovia, Ca, USA	Resin composite	3 JF	2006-10

Specimen preparation

The initial shear bond strength (SBS) and shear bond fatigue limit (SBFL) were determined on four different types of specimens, e.g. cement-enamel, bracket-cement-enamel, cement-only, and bracket-cement. A schematical representation of the specimens is shown in Figure 6.1. Cylindrical cores of enamel (6.0 mm in diameter) were cut from incisors of 2 years old bovines, using a water-cooled, diamond coated, trepan drill. These cylinders were bonded with composite in 2.0 mm thick stainless steel plates, with the enamel surface aligned slightly above the plate surface. The enamel was ground by hand on 240 grit, water cooled abrasive paper, until the surface was flush with the stainless steel plate. Hereafter finishing took place up to 1200 grit, ensuring a standardized enamel surface.

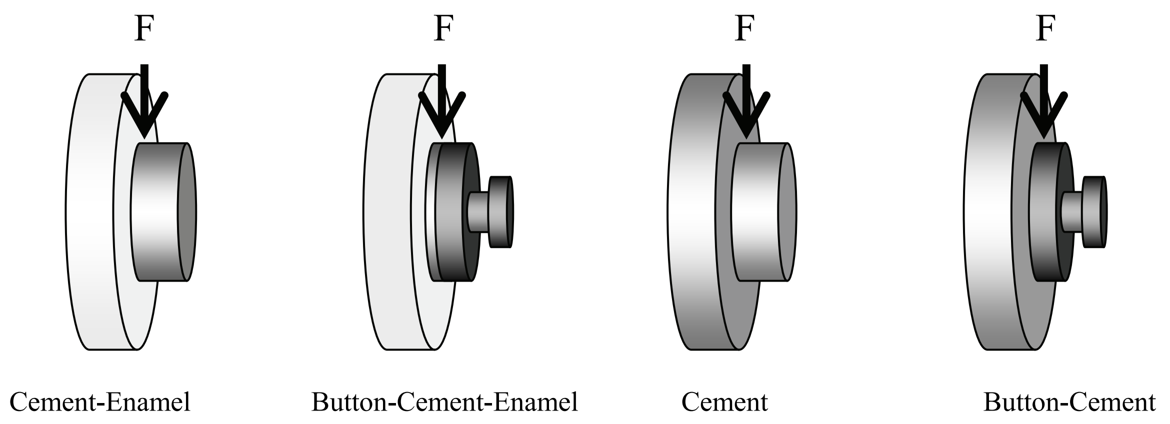

Figure 6.1 Schematically representation of the four different specimens tested. The arrows indicate the location of force application during testing.

When preparing the bracket-cement-enamel specimens the cements were applied to round mesh based stainless steel buttons (Direct Bond, Ortho Organizers Inc. San Marcos, Ca, USA). Immediately hereafter this combination was firmly pressed against the enamel surface. To standardize the procedure the brackets were placed with the aid of a polyether impression material mould (Impregum F, 3M ESPE, Seefeld, Germany). Because of the flat surface of the impression material around the bracket, no cement could cover the top surface of the bracket base. The brackets had a diameter of 3.5 mm and a bonding area of 9.6 mm^2.

For the cement-enamel specimens a cylindrical mould, with an inner diameter of 2.0 mm and a height of 2.0 mm, was placed on the enamel surface. The cylinder was filled with cement, covered with a polyester strip.

The cement-only specimens were prepared with the same mould. The mould was placed on top of the stainless steel plate at the centre of a cylindrical hole (Ø 6.0 mm). The existing lumen was filled at one tempo with one of the adhesives (Figure 6.1).

A similar procedure was followed for the bracket-cement group preparation. The same mould which was used to hold the bracket for the bracket-cement-enamel specimen preparation was positioned on top of the stainless steel plate at the centre of the cylindrical hole (Ø 6.0 mm) and filled with one of the cements. All specimens were stored for 72 hours in tap water of 37 °C.

Shear bond strength (SBS) testing

After storage the specimens were transferred to a test fixture. This fixture consisted of a cylindrical cavity in a brass block, with shallow, diametrically opposed grooves that were machined to fit precisely a two plate assembly; the specimen and the jig for applying the shear force (see Figure 6.2). The SBS of eight specimens was tested in a universal testing machine (Hounsfield H109KM universal testing machine, Redhill, UK) at a crosshead speed of 1 mm/min.

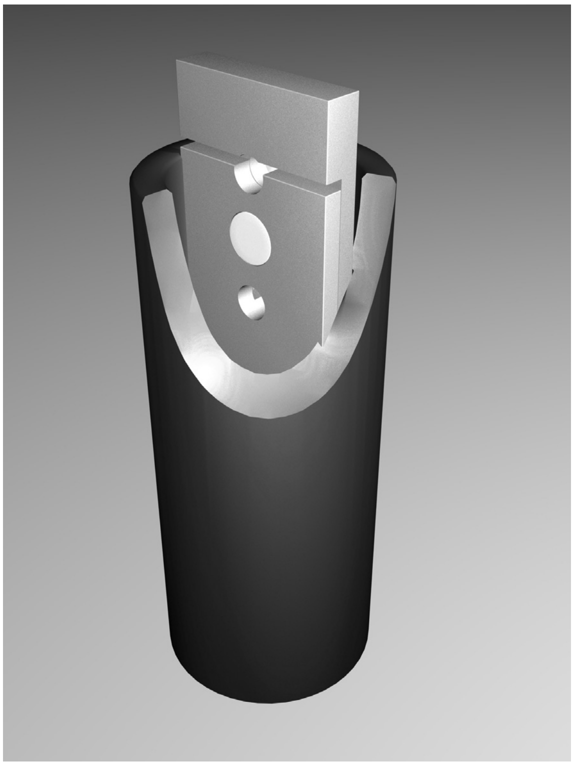

Figure 6.2 Schematical representation of the test fixture. The two-plate specimen assembly fits precisely in the diametrically opposed grooves. Therefore movement of the jig was only possible in one direction, ensuring a pure shear force.

Determination of the shear bond fatigue limit (SBFL)

A twelve-station fatigue testing apparatus utilizing air pressure to regulate and deliver the force was employed for load cycling.(12) The SBFL of the load tests were determined for 10,000 cycles (13) at a frequency of 1.0 Hz and a 50% duty cycle. The load cycling of the specimens (n = 20 per group) was carried out with the same test configuration as the SBS test. The 'staircase' method was used to determine the SBFL.(14, 15) The first test of each series started at 50% of the previously determined SBS of each cement. The maximum applied stress of the following tests was determined by the result of each previous test: failure or non-failure. When a specimen did not fail, the next specimen was put to the test at a stress level one increment higher than the previous test. In case of failure the next test was determined at one increment lower than the one that failed. Increments 5% up or down from the first performed fatigue test were used. The SBFL and its standard deviation were determined using Equation 1 and 2, respectively.

$$SBFL = X_0 + d\left(\frac{\Sigma i n_i}{\Sigma n_i} \pm 0.5\right) \qquad (1)$$

$$SD_{SBFL} = 1.62d\left(\frac{\Sigma n_i \Sigma i^2 n_i - (\Sigma i n_i)^2}{(\Sigma n_i)^2} + 0.029\right) \qquad (2)$$

In this Equation X_0 is the lowest stress level considered in the analysis and d is the fixed stress increment. In Equation 1, the negative sign is used when the analysis is based on failures; otherwise the positive sign is used. The lowest stress level considered is designated as $i = 0$, the next as $i = 1$, and so on. The number of failures or non-failures at the given stress level is represented by n_i.

Statistical analysis

Means and standard deviations of the SBS and SBFL were computed, and compared using one-way ANOVA and Tukey post hoc tests at a significance level of 0.05 using SigmaStat 3.1 (SPSS Inc, Chicago, USA). The statistical difference between the SBS and SBFL were analyzed with an independent t-test.

6.4 Results

The mean SBS results and standard deviations are summarized in Table 6.2. The SBS of the bracket-cement-enamel specimens was significant higher for Transbond XT compared to Fuji IX Fast and Fuji Ortho LC. The SBS of the latter two cements was not significantly different. The SBS of the cements increases from Fuji IX Fast < Fuji Ortho LC < Transbond XT and the same trend was observed for the bracket-cement specimens, although the SBS was remarkable higher compared to the cohesive strength of the cement alone. The bonding to enamel, e.g. the cement-enamel specimens, showed again an increase for the SBS from Fuji IX Fast < Fuji Ortho LC < Transbond XT.

Table 6.2 The SBS (MPa) with the standard deviations in parenthesis. Statistical differences were calculated using one-way ANOVA and the Tukey HSD *post hoc* analysis ($p < 0.05$).

	Cement-Enamel (MPa)	Bracket-Cement-Enamel (MPa)	Cement (MPa)	Bracket-Cement (MPa)
Fuji IX Fast	4.9 (1.5)	14.2 (5.0)^{aA}	17.1 (2.9)^{AB}	23.0 (5.7)^{B}
Fuji Ortho LC	15.8 (4.1)^{C}	15.3 (3.2)^{aC}	26.2 (4.3)	34.9 (4.4)
Transbond XT	31.8 (11.4)^{D}	23.7 (6.5)	36.4 (7.7)^{D}	45.7 (3.7)

Equal capital characters indicate statistical equality within the material (horizontal).
Equal small characters indicate statistical equality between the materials (vertical)

Table 6.3 The SBFL (MPa) after 10,000 cycles with the standard deviations in parenthesis. Statistical differences were calculated using one-way ANOVA and the Tukey HSD *post hoc* analysis ($p < 0.05$).

	Cement-Enamel (MPa)	Bracket-Cement-Enamel (MPa)	Cement (MPa)	Bracket-Cement (MPa)
Fuji IX Fast	5.9 (8.8)^{aA}	8.0 (2.9)^{A}	8.5 (1.9)^{A}	10.0 (0.3)^{A}
Fuji Ortho LC	10.2 (4.5)^{abB}	11.7 (5.2)^{cBC}	13.9 (1.9)^{CD}	16.2 (0.8)^{D}
Transbond XT	14.7 (6.3)^{bE}	14.3 (2.7)^{cE}	19.6 (0.7)^{F}	19.2 (4.2)^{F}

Equal capital characters indicate statistical equality within the material (horizontal).
Equal small characters indicate statistical equality between the materials (vertical).

The data points, the calculated average and standard deviation of the SBFL at 10,000 cycles of Fuji Ortho LC for the four different types of specimens are graphically represented in Figure 6.3. For the SBFL the standard deviations of all three cements are summarized in Table 6.3. The SBFL of the bracket-cement-enamel specimens was significant stronger for Transbond XT and Fuji Ortho LC compared to Fuji IX Fast. The SBFL of the cement increase from Fuji IX Fast < Fuji Ortho LC < Transbond XT and the same trend was observed for the bracket-cement specimens. In contrast to the SBS values there are no significant differences between the bracket-cement and the cohesive strength of the cement alone. The ranking of the SBFL for the cement-enamel specimens was the same as the SBS values.

Table 6.4 shows the ratios between the SBS and SBFL. Fatigue was observed for all materials except for Fuji IX Fast cement-enamel specimens and Fuji Ortho LC cement specimens.

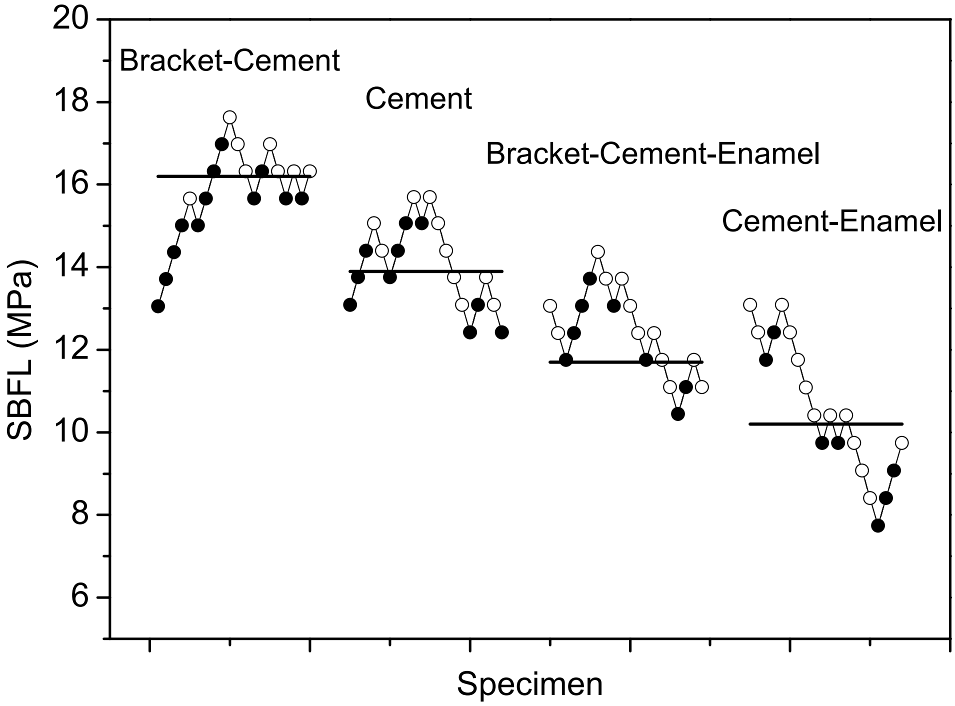

Figure 6.3 This graph shows the staircase results of Fuji Ortho LC for the four variables. Black dots indicate that the specimen did not fail whilst the white dots represent failure. From these results the SBFLs were calculated which are represented in the graph by the black lines.

Table 6.4 The ratios between the SBS and SBFL. The SBS and SBFL were not significantly different for the numbers marked with an asterisk ($p < 0.05$).

	Cement-Enamel	Bracket-Cement-Enamel	Cement	Bracket-Cement
Fuji IX Fast	1.20*	0.56	0.50	0.43
Fuji Ortho LC	0.65	0.76*	0.53	0.46
Transbond XT	0.46	0.60	0.54	0.42

6.5 Discussion

Shear and peel forces are major forces acting on the bracket-cement-enamel system during function. For that reason most *in vitro* research is focused on the SBS of the bracket-cement-enamel system. The mode of fracture is generally evaluated together with the adhesive remnant index (ARI score). The ARI score is used to assess the amount of resin left on the enamel and bracket surfaces after debonding, identifying the weakest link in the bracket-cement-enamel system. In this study not only the SBS and the SBFL of the bracket-cement-enamel system were evaluated, but also the separate combinations, cement-enamel, cement-only, and bracket-cement. The separate combinations were studied to identify the weakest link of the bracket-cement-enamel system with regard to SBS and SBFL.

The SBS of Transbond XT was significantly greater than those of the Fuji Ortho LC and Fuji IX Fast. This finding agrees with several other publications, where brackets bonded with resin composite and pre-treated with 37% phosphoric acid resulted in higher SBS compared to RMGIC or GIC bonded brackets (16-19). Most failure patterns show equal distributions of cement on the bracket and tooth surface or debond from the bracket base. In general the composite resin breaks cohesively or adhesively from the bracket base. The low SBS of the enamel-cement interface of Fuji Ortho LC and Fuji IX Fast can be explained by the weak bond strength of the cement-enamel interface or the different geometry in the test set up (see below). The SBS of this interface is significantly lower (see Table 6.2) than the cohesive SBS of the cement and the adhesive SBS of the cement-button interface, giving apparently rise to debonding from the enamel surface. In contrast, composite resins, like Transbond XT, seem to debond through the cement or at the bracket interface. In this study the SBS of cement-enamel was not significantly different compared to the cohesive strength of the material (see Table 6.2), which can explain the cohesive failure and the different debonding pattern for the composite resins bonded brackets.

The SBS of bracket-cement-enamel specimens cemented with Fuji IX Fast (4.9 MPa) was significantly lower than the SBS of cement-enamel specimens (14.2 MPa). A possible explanation for this is the geometry and combination of materials of the tested specimens. Due to the low Young's modules of the cement, (ca. 10 GPa) loading of the cement-enamel specimens resulted in deformation of the cement cylinder. This gave rise to tensile stress at the top of the cement-enamel surface and compressive strength at the bottom of the cement cylinder. In case of the bracket-cement-enamel specimens the applied load was more homogeneously distributed, because of the stiffer (ca. 200 GPa) stainless steel bracket on top of the cement.

Because of the complex shape and therefore the unknown stress distribution inside a bracket or button it is hard to quantify these effects. Nevertheless, it has been shown that the position of loading on the bracket (20) and the type of bracket (21) can give significant differences in SBS. For example, Klocke *et al.* found that loading close to the bonding surface gave a significant higher SBS 22.7 (4.2) MPa compared to loading at the outer surface of the bracket on the wings 9.4 (3.0) MPa.(20) Finite element analysis based on the experimental data could prove the above mentioned reasoning.

The SBS is generally used to evaluate the bonding systems in the laboratory. In principle, comparison between specimens or methods is justified, but extrapolation of these results in the clinical situation is not straightforward. For example, based on the SBS of this study (see Table 6.2) and one found in the literature (22), the bond strength of GIC is comparable to the SBS of clinically often used RMGIC. Therefore, one can tentatively conclude that GICs provide sufficiently high SBS to retain brackets under clinicical conditions. Nevertheless, clinical reports show high failure rates for GICs (up to 50%) within an average orthodontic treatment period.(2) Apparently the SBS is not adequate enough for evaluation of the bracket-cement-enamel system.

As mentioned earlier, Pickett *et al*. showed that the *in vitro* SBS of brackets bonded with Transbond XT of 12.8 (3.1) MPa reduced to 5.5 (2.2) MPa over time. Several reasons were given as possible factors to explain this reduction in bond strength.(4) A deteriorating effect of water on GICs (23) as well as on resin composite based cements (9) has been observed in various studies. Previous experiments showed a decrease of the mechanical strength of resin based materials up to 20%.(15) Long-term storage over 180 days showed that there were no clinical relevant increases or decreases in SBS for a resin composite and RMGICs in a bracket-cement-enamel system.(21) Apparently, the masticatory forces and the forces of the orthodontic treatment give rise to such an amount of fatigue that the bond strength of cements, bonded to the enamel or bracket, is reduced with 50% to 60%. Lohbauer *et al*. (24) found a similar decrease in strength for restorative composites in 4-point bending fatigue tests using the staircase method.

In this study the staircase method was used to determine the SBFL. Although there is some concern about this type of fatigue testing, because of its non-conservative approach to the outcomes and lack of lifetime prediction capacity (10), it is the method of first choice in this study.(25) The SBFL's of the specimens in this study were only 65% to 42% from the initial SBS, except for the Fuji IX Fast cement-enamel and Fuji Ortho LC bracket-cement-enamel specimens. The latter specimens did not show a significant effect of fatigue at 10,000 cycles. No significant differences were observed in the Fuji IX Fast specimens, cement-enamel, bracket-cement-enamel, cement-only, and bracket-cement. Apparently the strength of these specimens is determined by the cohesive strength of the cement if the specimens are cyclically loaded. Also the cohesive strength of Fuji Ortho LC was not significantly different from the bracket-cement and bracket-cement-enamel specimens. On the other hand the SBFL of Fuji Ortho LC was marginally lower than the SBFL of the bracket-cement and bracket-cement-enamel specimens. For these results it seems that the reduced strength level can be explained by a slow crack growth mechanism in the cement due to the brittle nature and the low cohesive strength of these materials. In brittle materials flaws have a predominant effect on the fatigue characteristics and these materials do not show a clear relationship between fatigue life and stress.(25) The resin based cement, Transbond XT, showed a different type of behaviour. The SBFL of the cement and bracket-cement were significantly higher than the SBFL of the cement-enamel and the bracket-cement-enamel specimens. It seems that the cement-enamel interface was the weakest link in this group. It is noteworthy that Erickson *et al.* found a reduction of 57% of the bond strengths for enamel-composite specimens using Single Bond as bonding system.(12) A SBS of 25.3 MPa was reported, which decreased to 14.6 MPa after 100,000 cycles. Interestingly, Titley *et al.* reported that the ARI score shifted from mainly adhesive at the bracket-side after 24 hours to mainly adhesive at the enamel-side after 180 days for the stainless steel brackets, but the SBS did not decrease.(21) It was beyond the scope of this paper to study these effects by means of fractographics, which will be a topic for further research. In contrast to the Transbond XT and Fuji IX Fast specimens, the bracket-cement-enamel bonding of Fuji Ortho LC did not show a significant reduction of the SBFL compared to the SBS. Synergy of the good fatigue resistance of the cement-enamel bonding of the glass ionomer content and on the other hand the high SBFL of the resin part might be an explanation for these remarkable results.

In the literature, the recommended bond strength of the bracket-cement-enamel system varies as mentioned from 2.9 to 10.0 MPa.(3-6) Most of these data are based on short-term SBS or tensile measurements. Pickett *et al.* (4) showed that the *in vitro* shear bond strength of 12.8 (3.1) MPa was reduced to 5.5 (2.2) MPa *in vivo*. This study shows that the decreased *in vivo* SBS is mainly due to fatigue induced by dynamic loading. Based on the *in vitro* SBS one could tentatively conclude that GICs provide sufficiently high strength to retain brackets under clinical conditions. This was in contrast to the clinical reports, where high debonding values (50%) for GICs were observed. Evaluation based on the SBFL results shows that Fuji Ortho LC and

Transbond XT are not significantly different, but both are significantly stronger than Fuji IX Fast. These findings represent the clinical observations where both the RMGIC and resin composite cements show a failure rate of ca. 5% and the GICs 50%. Based on the results of the bracket-cement-enamel system of Fuji Ortho LC a SBFL of at least 11 MPa is required as the recommended bond strength. Furthermore, it shows that using SBS alone for evaluation lead to misinterpretation, because materials that provide high initial strengths do not obviously reveal the best fatigue resistance.

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