The influence of cyclic shear fatigue on the bracket-adhesive complex

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Meiner lieben Familie gewidmet

Inhaltsverzeichnis

1 Zusammenfassung

Ein häufiges Problem in der Kieferorthopädie ist der Verlust von Brackets während der Behandlung. Intraoral auftretende Kräfte sind üblicherweise kleiner als die Haftfestigkeiten, die in *in vitro*-Studien für den Bracket/Adhäsiv/Zahnschmelz-Verbund angegeben werden und treten wiederkehrend auf. Die Wahrscheinlichkeit ist groß, dass ein Versagen als eine Folge von zyklischer Ermüdung auftreten kann. Zusätzliche isolierte Belastungen auf einzelne Verbünde können zu einem Versagen der Klebung führen, insbesondere wenn das System vorher durch die mechanische Wechsellast ermüdet wurde.

Das Ziel dieser Studie war es, den Einfluss der Ermüdung auf den Bracket/Adhäsiv-Verbund in Abscherversuchen zu ermitteln.

Brackets mit laserstrukturierter Basis (Discovery®, Dentaurum) und mit Netzbasis (Ultra-Minitrim®, Dentaurum) wurden auf flachen silanisierten Edelstahlplatten mit einem Zwei- und einem Vier-Komponenten chemisch härtenden Kunststoff geklebt (No-Mix Bonding System, Dentaurum sowie Concise™, 3M Unitek). Die Proben wurden in destilliertem Wasser bei 37°C für 3 Tage gealtert. Ein Teil der Proben wurde als Kontrollgruppe benutzt, um die Haftfestigkeit bei Abscherversuchen zu ermitteln. Der zweite Teil wurde in eine Materialprüfmaschine Zwick 1445 (Zwick GmbH & Co) nach der Stufenmethode über 1000 Lastzyklen einer mechanischen Dauerlast ausgesetzt. Die überlebten Proben wurden abgeschert und deren Haftfestigkeit wurde mit der der Kontrollgruppe verglichen. Die Ermüdungsgrenze und deren Verhältnis zu der Haftfestigkeit wurden berechnet. Die Verteilung der Kunststoffreste auf den Bracketbasen wurde am Rasterelektronenmikroskop bei 25-facher Vergrößerung visuell und quantitativ beurteilt.

Die Ermüdung über 1000 Zyklen bei Scherbelastung zeigte einen uneinheitlichen Einfluss auf den Bracket/Adhäsiv-Verbund, der stark von den verwendeten Materialkombinationen abhing. Ermüdete Proben zeigten eine 8 % höhere Scherhaftfestigkeit in Materialgruppe A (Bracket Discovery® / Adhäsiv No-Mix) und eine 10 % niedrigere Scherhaftfestigkeit in Materialgruppe D (Bracket Ultra-Minitrim® / Adhäsiv Concise™) verglichen mit den nicht ermüdeten Proben. In den Materialgruppen B (Bracket Ultra-Minitrim® / Adhäsiv No-Mix) und C (Bracket Discovery® / Adhäsiv Concise™) wurden keine statistisch signifikanten Unterschiede zwischen der Scherhaftfestikeit der ermüdeten und nicht ermüdeten Proben festgestellt. Das Verhältnis von Haftfestigkeit und Ermüdungsgrenze war ca. 60 % bei den Materialkombinationen A, B und C und ca. 67 % bei der Materialkombination D, was auf ein besseres Ermüdungsverhalten der Gruppe Ultra-Minitrim® / Concise™ hindeutet. Unter den nicht ermüdeten Proben zeigte die laserstrukturierte Basis von Discovery® eine ca. 59 % höhere Scherhaftfestikeit als die Netz-

basis von Ultra-Minitrim®. Der Vier-Komponenten-Kunststoff Concise™ zeigte ca. 66 % höhere Haftfestigkeit als der Zwei-Komponenten-Kunststoff No-Mix.

Auch auf die Verteilung der Kunststoffreste zeigte die Ermüdung einen stark variablen Einfluss, je nach Art des untersuchten Frakturmodus. Unabhängig von den getesteten Materialkombinationen zeigte die Ermüdung den gleichen Einfluss auf die Fläche der günstigen Frakturart (Summe aus Bruch des Verbunds zwischen Bracket und Kunststoff sowie kohäsiver Frakturfläche), jedoch konnte eine statistische Signifikanz nicht immer nachgewiesen werden. Es zeigte sich kein statistisch signifikanter Unterschied der Fläche dieser günstigen Frakturart zwischen den ermüdeten und den nicht ermüdeten Proben. Die ermüdeten Proben, die 1000 Zyklen nicht überlebt hatten, zeigten jedoch eine signifikant größere Fläche der günstigen Frakturart in Materialgruppen B, C und D im Vergleich zu den Proben, die 1000 Zyklen überlebt hatten. In der Gruppe D war diese Fläche größer auch im Vergleich zu den nicht ermüdeten Proben. Die bevorzugte Frakturart, die kohäsive Fraktur, war ebenfalls ermüdungsabhängig, jedoch war der Einfluss in den Materialgruppen stark schwankend. In den Materialgruppen A und B wurde kein signifikanter Einfluss der Ermüdung gefunden. Ermüdete Proben zeigten signifikant höhere kohäsive Frakturflächen in der Gruppe D und die nicht überlebten Proben zeigten eine signifikant kleinere Fläche als die überlebten. Im Gegensatz dazu zeigten die nicht überlebten Proben in der Gruppe C eine signifikant größere Fläche als die überlebten. Der Einfluss der Bracketbasis auf die Verteilung der Kunststoffreste bei den nicht ermüdeten Proben war materialabhängig aber gleich für beide Frakturarten. Das Bracket Ultra-Minitrim® zeigte eine signifikant höhere günstige und bevorzugte Frakturart als das Bracket Discovery® in Verbindung mit dem Adhäsiv Concise™ und keinen Unterschied mit dem Adhäsiv No-Mix. Der Einfluss des Adhäsives war materialabhängig und uneinheitlich. Beide Adhäsive zeigten keinen signifikanten Unterschied in Bezug auf die günstige Frakturart mit beiden Brackets. Die bevorzugte Frakturart war signifikant kleiner für den Kleber Concise™ in Kombination mit dem Bracket Ultra-Minitrim®. Beim Bracket Discovery® wurden keine Unterschiede gefunden.

Zusammenfassend lässt sich feststellen, dass die Ermüdung einen variablen Einfluss auf die Scherhaftfestigkeit und die Restkunststoffverteilung auf den Bracketbasen hatte. Der Einfluss der Ermüdung war stark materialabhängig. Die Verteilung der Kunststoffreste auf den nicht ermüdeten Proben war ebenfalls materialabhängig. Nur die Scherhaftfestigkeit der nicht ermüdeten Proben zeigte ein einheitliches Verhalten für alle getesteten Kombinationen. Die Stufenmethode bietet ein einfaches und reproduzierbares experimentelles Protokoll für die Standardisierung von Ermüdungsversuchen, wenn die Höhe der zyklischen Belastung in der Nähe der Ermüdungsgrenze liegen soll.

2 Introduction

Bonding brackets to teeth (Figure 1) has been a common procedure in orthodontics for many decades.

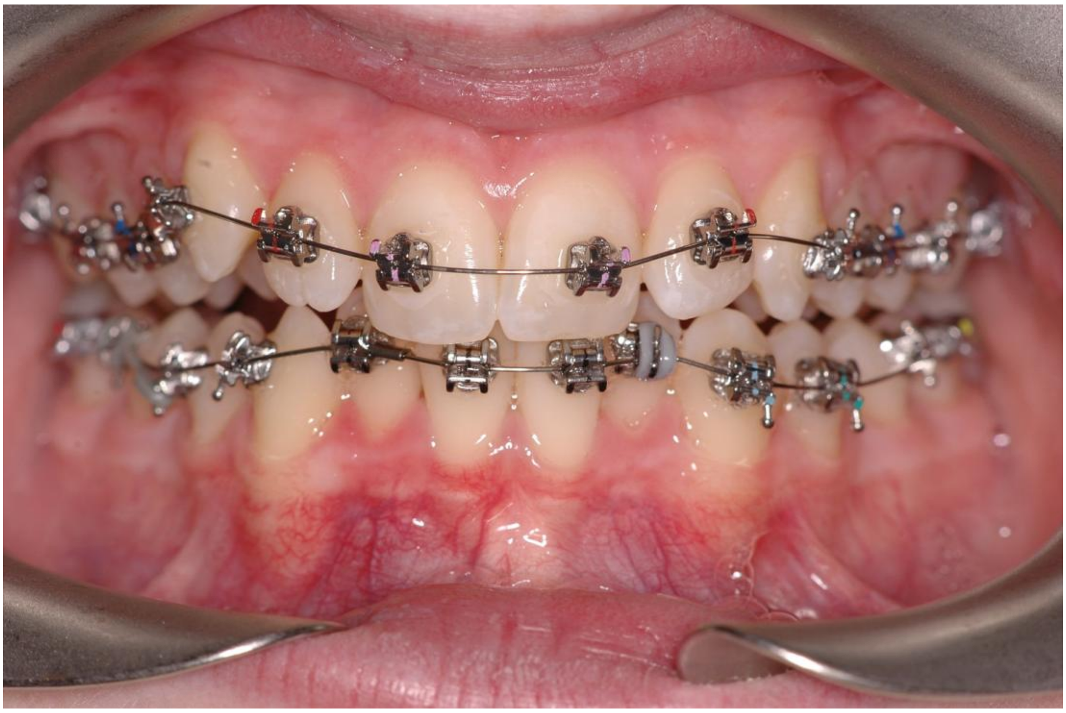

Figure 1. Example of a multibracket appliance immediately after bonding.

Bracket bond strength depends on a number of factors. Some of them are the bracket material and the surface geometry of its retention base, the bonding material and bonding procedure, the conditioning of the surfaces to be bonded, the loading mode, the temperature, the ageing and fatigue of all materials involved.

A commonly encountered problem is the bond failure of brackets during treatment. The frequency of this has been investigated by many authors and has been found to vary between 0.5% and 16%. Various factors can contribute to the likelihood of a bond failure, including operator technique, patient behaviour, variation in the enamel surface and bracket properties.

The mechanical properties of composites have been extensively researched in the laboratory, but the clinical situation has not been accurately replicated. Some researchers have suggested that failure is the result of changes that occur because of exposure to the oral environment. They found differences between bond strength test results after storage in sterile water and the oral environment with reduced bond strength *in vivo* compared with the *in vitro* results (Murray and Hobson, 2003).

Biodegradation is the result of a combination of disintegration and dissolution in saliva, chemical and physical degradation, wear caused by chewing food, erosion by the food itself, and bacterial activity (Oilo, 1992). The simulation of clinical conditions is a difficult task. The controlled debonding procedures using testing machines do not allow a precise comparison with the failure pattern occurring *in vivo*. For example shear or tensile tests are not comparable to the debonding procedure recommended by the manufacturers, where special pliers are used. Further, the wide range of tensile or torque loads transferred to the system from mastication or orthodontic mechanics are not taken into consideration (Eliades and Brantley, 2000).

High shear bond strengths of the bracket-adhesive complex have contributed to the acceptance of each system tested. However, the forces applied in the oral environment are more likely to be of a cyclical nature, well below the ultimate shear strengths reported in *in vitro* studies. The failure over time is much likely to be the result of fatigue. The discrepancy between *in vitro* and *in vivo* results underline the difficulty of predicting the *in vivo* behaviour based on results of *in vitro* bond strength tests alone. Shear fatigue test data, however, could provide a better insight into the long-term *in vivo* behaviour of a dental bonding system (Ruse et al., 1995).

The nature of *in vivo* loading is characterised by cyclic and low-magnitude forces. Isolated or initial powerful impacts are seldom. Nevertheless, powerful impacts may occur after some time, i.e. after the system has been cyclic-loaded, and lead to failure. Failure may also be the result of cyclic low forces without sudden impact.

One approach in resolving the soundness of *in vitro* derived data could be the fatigue of the adhesive-bracket system. This can be accomplished through exploring the total-life tolerance of the system to a low-magnitude, cyclic, mechanical stress. Standard tests measure the strength at a sudden and powerful impact, although the system was not constructed to resist high loading (Eliades and Brantley, 2000). Also, if fatigue testing shows a mean decrease in bond strength, then one can infer that the actual debonding force needed to remove orthodontic brackets after treatment will be less than what static bond strength tests report because static tests are done without fatiguing the bracket before debonding.

Fatigue of materials provides a broad variety of complex mechanistic processes for scientific investigation. Implications of fatigue failure encompass many aspects of our lives. Considerations of fatigue failure are intimately tied to the structural integrity in engineering components. However, the mechanistic and scientific basis for the study of fatigue is essential for many reasons: The macroscopic fatigue crack appearance is comparable to the microstructural dimension of materials. Scientific knowledge of failure mechanisms is required for improving the fatigue resistance by optimising the microstructural characteristics of a material. Characteristic features observed on the fatigue fracture surfaces during "post-mortem" analyses are linked to the microscopic mechanisms of failure and the macroscopic rates of crack advance.

Another approach for understanding the clinical behaviour of the bracket-adhesive system is the finite elements analysis. Some issues regarding research methodology and interpretation of the findings of bond strength experiments have been thus clarified (Katona, 1997).

Some aspects of the properties of the bracket-adhesive complex may be clarified by *in vitro* approaches but the actual performance of the system can only be illustrated in the environment where it was intended to function. Clinical studies focusing on the failure rate in a controlled environment under identical conditions may target the examined variable. However a definition of research protocols fulfilling all the conditions is nearly impossible (Eliades and Brantley, 2000).

3 Review of the literature

3.1 Materials

3.1.1 Adhesives

An adhesive is a substance capable of holding materials together. The first adhesives tried in orthodontics were the epoxies, which were excellent bonding agents. Their inability to provide resistant bonds in the oral environment has allowed the acrylics to emerge, despite their poorer chemical affinity. The present generation of acrylics, based upon cross linking monomers, derives its performances from its toughness and resistance to the mouth conditions (Matasa, 1989).

Composite resins

The most widely used adhesives are composite resins. In its material sense, a composite can be anything that consists of two or more distinctive phases. The most common resins in orthodontics consist of a dispersion of fillers (the discontinuous phase, made of round or irregular shaped particles, fibres, whiskers) in a binder or matrix (the continuous and usually softer phase). Matrices are the weak link: under load, these are the first to crack and craze, breaking down at strains far lower than the fibres can endure. However, by keeping the reinforcing fibres together in the right orientation and position and by transferring and distributing the load, matrices protect the fibres against chemical attack, rendering composites remarkable for their strength and endurance. Equally important as the nature of the matrix and filler is their interface: related defects or weaknesses can severely limit the performance of the composite. In the absence of affinity, composite's components do not behave as a whole and are easily disassembled. For this reason, whenever it is necessary and possible, go-betweens, coupling or keying agents are used, such as silanes.

Composite resins are considered the standard adhesives in contemporary orthodontics. They are based on matrices of Polymethylmethacrylate (PMMA) or Bisphenol-A-glycidyldimethacrylate (Bis-GMA). Further developments led to products such as Trimethylhexanurethan-dimethacrylate (UDMA), Triethylenglykol-dimethacrylate (TEG-DMA), Bismethacryl-oxy-ethoxy-phenylpropan (Bis-EDMA) or Ethoxy-bisphenoldi-

methacrylate (Ethoxy Bis “A“ DIMA). The filler particles usually consist of Quartz, Ceramics or Siliciumdioxide. Composites are considered brittle materials.
Since presenting the acid etch technique (Buonocore, 1955; Silverstone, 1975) much research has been undertaken to improve bonding to enamel. Newest developments have led to simplified bonding protocols with one-component adhesives and self-etching primers. It is widely accepted that resin-based adhesives bond to enamel only by mechanical interlocking and no chemical affinity (Diedrich, 1981). Further, the acrylics used in dentistry have a relatively poor affinity to metal and ceramics. Their bond is based primarily on mechanical interlocking, thus being proportional to the roughness/undercuts of the substrate. To enhance this, etching of both teeth and bracket bases are desirable. On the contrary, a smooth base, resulting from abrasion or electro-polishing, will sharply decrease the bond strength. The use of wetting agents, which allow the oil-loving adhesive to penetrate the oil-hating/water-loving substrates, is equally beneficial. Some substances, like the silanes, titanates and zirconates act not only as wetting agents, but contribute also to chemical bonding (Matasa, 1989).

Glass ionomer cements

Glass ionomer cement was introduced to dentistry in the 1970s (Wilson and Kent, 1972) and was later popularised in orthodontics (White, 1986). It adheres chemically to enamel, dentin, nonprecious metals and plastics and does not require acid-etching of the tooth surface. Another advantage is that glass ionomers release fluoride for at least 12 months and have the ability to absorb fluoride, allowing them to act as rechargeable fluoride reservoirs. Glass ionomer cements proved to have lower bond strength (Bishara et al., 1999a; Haydar et al., 1999; Jahnig and Henkel, 1990) than composite resins. However, they serve as a good bonding agent for ceramic brackets due to their lower bond strength, since ceramic brackets usually show an excessive bond strength to teeth, when bonded with composites (Haydar et al., 1999).

Compomers

Combinations of glass ionomer cements and composite resins into hybrid materials called polyacid-modified resin composites or compomers have been introduced with claims that they combine the advantages of both materials. They set initially with photo-polymerisation and then gradually through diffusion of water into the set material. When set, they do not exhibit the typical properties of glass ionomer cements, such as chemical bonding and fluoride rechargeability. Resin reinforced glass ionomer cements have been reported to have a similar clinical failure rate to composite resins when bonded onto teeth soaked with saliva (Cacciafesta et al., 1998). Other investigators found compomers and resin-reinforced glass ionomer cements to have greater shear bond strength than composites (Millett et al., 2001).

Bond strength and crack formation

Internal stresses are the sources of many adhesive failures. Such stresses are the result of shrinkages, poor homogenisation or uneven affinity to the substrates. Almost all polymerisations occur with a decrease in volume (the only exception known is that of cyclic monomers, which open in the process of polymerisation). To reduce shrinkage, high molecular weight monomers as well as fillers have to be used. To reduce general unevenness, good mixing/stirring of the adhesive is compulsory, as well as an overall preparation of the substrate surfaces. It is known that bonding does not occur on the whole surface being treated, but only on few selected centres of high energy (Matasa, 1989).

Air masks bonding centres on the substrates, and, if embedded in the adhesive, it decreases its cohesion. It also acts as a polymerisation inhibitor, oxygen being a free radical interceptor (Matasa, 1989).

Although crack nucleation is still not well-understood, this event generally occurs in regions of stress concentration, such as scratches or grain boundaries on the surface, or voids in the interior. According to the Griffith theory, brittle materials such as glasses and ceramics contain pre-existing flaws formed during processing (Suresh, 1998). These flaws, which may be present on the surface or within the volume of the specimen, serve as nuclei for cracks. The sizes of these cracks and the critical stress intensity factor define the fracture strength for these materials: At stresses exceeding the fracture stress, cracks propagate catastrophically, while at stress levels below the fracture stress, cracks can heal.

In fibre-reinforced materials, cracks do not propagate for long within the matrix before reaching the fibre interface. There, the crack may bifurcate and travel for considerable distances along the interface. The angular distribution of stresses, determining crack propagation direction at the crack tip in composites, is determined by microstructure, not by the direction of the applied load. Similarly, the speed of crack propagation is determined not simply by the stress intensity, but also by microstructure: The strength gradient at the interface between matrix and filler will determine the crack-growth rate rather than the crack propagation rate determined for the matrix alone. Following local matrix and interface failure surrounding a dispersed fibre, the fibre itself ruptures. Then, load is transferred to neighbour fibres, which in turn rupture. After a critical density of single-fibre failures is attained, the fracture of the body takes place (Baran et al., 2001). Therefore, optimal strength properties are obtained in composites where the particle/matrix adhesion is good (Spanoudakis and Young, 1984).

Within an evaluation of composite restorative materials classified according to the filler particle size, observation of the fracture surface revealed inter- and intra-particle fracture for the small particle and hybrid composites. Microfilled composites fractured between and through pre-polymerised particles (Drummond, 1989).

Highly filled resin composites deform little under function. The cracks move through the matrix-phase and along the filler particles, resulting in a more localised destruction and in an inherent crack arresting or retarding mechanism (Braem et al., 1994a). Composites with higher filler volumes show an increased fracture strength and elastic modulus (Ferracane et al., 1998). Composites with the highest filler content exhibit the highest flexural strength, flexural modulus and hardness, but the maximum fracture toughness is obtained at approximately 55% of filler volume (Kim et al., 2002).

It has been reported that for hybrid composites, increasing the volume fraction of filler particles would heighten the probability of increasing the local energy for crack growth. In other words, an advancing crack would need more energy to move through the particles. In hybrid composites, the direction of crack growth continually changes as it encounters particles. Consequently, the rate of crack growth decreases and more particles are sheared because of higher volume fraction of filler particles (Aghadazeh Mohandesi et al., 2007).

Apart from the type of filler content, the composition of resin matrix also influences the mechanical properties of composites. Varying the relative amounts of UEDMA, Bis-GMA and TEGDMA has a significant effect on the mechanical properties of the resin composition. The replacement of Bis-GMA and TEGDMA by UEDMA is instrumental for the increase in both tensile and flexural strengths (Asmussen and Peutzfeldt, 1998) or the compressive strengths and fatigue strengths (Aghadazeh Mohandesi et al., 2007) of the matrix. Substitution of Bis-GMA by TEGDMA increases tensile, but reduces flexural strength (Asmussen and Peutzfeldt, 1998). Composites which utilise a Bis-GMA/TEGDMA blend exhibit a higher degree of conversion and higher strength when the quantity of TEGDMA is increased (Asmussen, 1982), which was confirmed by other studies (Aghadazeh Mohandesi et al., 2007; Drummond, 1989). Thus, by selecting specific combinations of these components, it may be possible to design composites with properties that are tailor made to specific applications.

The properties of the matrix, the filler, and the filler volume fraction determine the failure properties of the composite at high crack velocities, while at low velocities the stability of the filler/matrix interface is the controlling factor (Montes and Draughn, 1987). Debonding at the filler-matrix interface occurs in particulate-reinforced composites at low static stresses, producing a rough fracture surface, while at higher applied stresses that cause higher crack velocities, the crack propagates through filler and matrix, resulting in a smooth fracture surface (Cantwell et al., 1988).
The attachment of the adhesive to the bracket material is the bottleneck of the direct-bonding procedure; the bracket base-cement interface is the weakest point in orthodontic bonding (Dickinson and Powers, 1980; Faust et al., 1978; Fowler et al., 1992; Ireland and Sherriff, 1994; Keizer et al., 1976).
However one should take into consideration that the diversity in structure and composition among composites, glass ionomers, and resin-reinforced glass ionomers does not allow findings from one product to be extrapolated to other similar products.

3.1.2 Brackets

Brackets are attachments bonded on the teeth and used to apply forces for orthodontic tooth movement. They evolved as a replacement of bands in the 1960s and 1970s, offering a more hygienic and aesthetic approach in orthodontic appliances. Several materials are used for orthodontic attachments: Plastic, metal (steel, titanium, gold) and ceramic brackets are currently available. To improve the aesthetics, there has been a trend to reduce the size of the bracket and its base.
Although resins bond well to an etched dry enamel substrate by mechanical interlocking, they do not adhere to stainless steel. Bracket base retention mechanisms can be mechanical or chemical or a combination of both.
Crystal-like formation of the rough bracket bases results in retention of the resin. The rheological properties of the adhesive as well as the size of the pores, grooves or the mesh influence the amount of retention of the adhesive. Furthermore, variations of the surface morphology lead to fluctuations in adhesive thickness (Eliades et al., 1991).
Bracket design, such as wing design or the dimensions of the bracket may contribute to misalignment of load application, thus making the system more vulnerable to failure (Eliades and Brantley, 2000) while smooth bracket bases contribute to a homogeneous load application (Eliades et al., 1991).
The effective surface area of the base in contact with the adhesive depends on the morphology of the base, i.e. rougher bases have larger surfaces. Bracket base treatments

such as microetching to increase surface area have been shown to enhance adhesion, leading to an increase in bond strength (MacColl et al., 1998; Sharma-Sayal et al., 2003; Willems et al., 1997). Sandblasting resulted in a reduction of failure rate and an increase of survival time under fatigue (Millett et al., 1993).

Mechanical retention such as mesh, laser structured or hook shaped bases currently exist. The initially perforated bracket bases have been replaced by foil-mesh bases. Mesh pad, which is the system most commonly used, has been known for many decades (Newman, 1965) and its improvement has been the objective of many research projects and manufacturers. Having acceptable static and cyclic bond strengths the mesh-foil based metal bracket has remained the most efficient and useful bracket system ever designed. It has excellent fatigue resistance and moderate bond strength because of metal deformation that prevents enamel fracture (Soderquist et al., 2006). There are contradicting results in research upon the effect of the size of the mesh: Coarser mesh gauzes have been reported to increase mechanical retention (Reynolds and von Fraunhofer, 1976; Sharma-Sayal et al., 2003). It is probable that the wider mesh allows a more efficient and complete penetration of the adhesive, resulting in significantly higher bond strengths. Yet other investigators proved finer meshes to result in greater shear bond strength when using lightly filled composites (Maijer and Smith, 1981). Tensile bond strength was independent of nominal area and mesh size of the bases in another study (Dickinson and Powers, 1980). Double-mesh bases showed similar bond strength compared to single-mesh bases (Bishara et al., 2004).

A new approach to improve mechanical retention of brackets has been the use of the Nd:YAG laser to melt and evaporate the metal and as a result burn hole-shaped retentions into the base (Sernetz and Binder, 1997). The laser-structured bracket bases showed higher tensile bond strength and improved fatigue behaviour than simple foil mesh bases (Sorel et al., 2002).

A bracket base provided with communicating channels or undercuts will allow the adhesive to gradually push aside the air, while bases which can entrap it are prone to give weaker bond (Matasa, 1989). Finally some authors pointed out weld spots on the mesh base of brackets to be disadvantageous (Dickinson and Powers, 1980; Maijer and Smith, 1981).

3.2 Bond strength

3.2.1 Factors affecting bond strength

Beyond the material properties and the substrate geometry (see chapter 3.1), there are various factors which influence the bond strength of orthodontic attachments:

Test mode

Strength testing of direct bonded orthodontic bracket systems is commonly performed with tensional, shear peel, or torsional loads. In general, the results of these tests are reported as an average stress that is computed by dividing the experimentally measured force at failure by the area of the bracket base. The average value, obtained in this manner, implies an evenly distributed stress field. Finite element analysis showed however that the stress distribution within the bracket-adhesive complex is inhomogeneous. It has been shown, that the maximum stress developed in the orthodontic bonding system under tensile loading may be five times greater than the average stress (Katona, 1997). The site of failure may arise from crack initiation caused in areas of higher stress compared with others, which is not taken into consideration in the traditional assumption of homogeneous stress (Eliades and Brantley, 2000).

The three loading modes produce very different non-uniform stress field patterns; the average stress does not adequately characterise bond strength. Comparing results from different loading modes has been proved to be inappropriate in a finite element analysis (Katona, 1997) and after *in vitro* testing (Katona and Long, 2006).

Material properties are different in tension and compression (Beatty and Pidaparti, 1993) and failure in compression is fundamentally different from failure in tension and shear; flaws grow catastrophically in tension and shear but damage accumulates until some material-specific proportion of cracks join to cause bulk failure when compression loading is studied (Baran et al., 1998).

However, a tensile stress must be present for a crack to grow (Reid et al., 1990). Tensile forces are not evident during mastication or cyclic compression, which means that cracks are arrested under these conditions. Nevertheless, when a loading mode is compressive, the load applied in a vertical direction that seeks to promote crack growth may cause a tensile stress component to be formed in the lateral direction. On this account, the tendency for crack generation and subsequent crack growth is higher in a porous structure (Aghadazeh Mohandesi et al., 2007).

Previous findings revealed no significant differences in bond strength between a tensile test and a shear test. However, the shear test produced more true adhesive failures; thus it may be preferable to use a shear test for adhesion testing (Fowler et al., 1992). Other investigators found shear strengths to be lower than flexural strengths and that the Weibull shape parameter obtained from shear data differed for some dental resin materials from that obtained in flexure (Baran et al., 1998). Some researchers found a 50% greater shear bond strength compared to tensile bond strength for both glass ionomers and composites (Jahnig and Henkel, 1990).

Interestingly, fibre-reinforced composites generally show lower fatigue resistance in compression than in tension, because of cooperative buckling of adjacent fibres and matrix shear (Suresh, 1998).

The validity of comparing results is affected by the testing configuration. The applied force generates moments depending on the distance of the force application vector from the centre of resistance. This parameter should be carefully taken into consideration, as it can complicate the extrapolation of conclusions about the failure incident (van Noort et al., 1989).

Recently, a technical specification was published with the intention to standardise as far as possible different laboratory procedures and substantiate the effect or quality of bonding between a dental material and tooth structure (DIN Deutsches Institut für Normung e.V., 2002).

Aging

The main factors that distinguish the oral cavity from *in vitro* media are the presence of complex oral flora and their by-products, as well as the accumulation of plaque on the material. The multifaceted intraoral environmental milieu cannot be simulated with the currently available *in vitro* research methodologies, most of which involve exposure of materials to various electrolytes, artificial saliva, water, and other media. Retrieval analyses can contribute to solving this problem by investigating the performance of the material in the environment in which it was intended to function. Retrieval analysis has been used for decades for biomedically applied materials but only recently was applied to orthodontic materials research (Eliades and Bourauel, 2005).

Soaking in water is known to affect the filler-matrix interface and it is expected that this phenomenon would not only weaken but also embrittle the material by increasing the volume flaw population. It leaches out filler elements, induces filler failures and filler-matrix debonding and reduces the strength of the matrix material. Debonded fillers may act as stress concentrators, which significantly multiply the number of potential crack growth sites.

The changes due to water sorption are clearly reflected in dental restorative materials: Liquid sorption shows a profound effect on characteristic flexural and shear strengths in dental composite materials (Baran et al., 1998). In another study, both the initial flexural strength and the flexural fatigue limit decreased after water sorption (Braem et al., 1994b). Water sorption affects mechanisms of crack propagation in dental composites, with dry specimens fracturing in a stick-slip mode, while soaked specimens exhibit stable slow crack growth, with the filler/matrix interface offering less resistance to crack growth after water sorption (Montes and Draughn, 1987).

In another study with a microfill composite, ageing in saline or distilled water at 37°C for 12 months showed no significant difference in the flexural strength (Drummond and Savers, 1993). The duration of soaking either for 24 hours or for 30 days does not seem to affect the mechanical retention of attachment bases to adhesives (Lopez, 1980).

Time

The bond strength of a composite resin increases with time due to continued polymerisation. 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. (Bishara et al., 1999b; Bishara et al., 2002; Braem et al., 1987; Chamda and Stein, 1996; Evans et al., 2002; Liu et al., 2004; Sharma-Sayal et al., 2003; Wendl and Droschl, 2004). Similar findings apply to glass ionomer cements (Wilson and McLean, 1988).

Few *in vitro* studies investigated the bond strength of the adhesive-bracket complex after early loading within the first hour. They found that early static loading (such as tying in an archwire) of a setting resin had no influence or even increased the shear bond strength of the composite-bracket complex (Ching et al., 2000; Ireland and Sherriff, 1997).

Temperature

In the oral environment, dental restorative materials are exposed to temperatures ranging from 10 to 50 degrees C. The temperature conditions of the oral environment can significantly affect the mechanical properties of composite dental restorative materials. Ultimate strength was shown to decrease linearly with increasing temperature. Elastic modulus and yield strength decrease sigmoidally with increasing temperature. In the clinically significant temperature range, ultimate strength decreases by 14%, the decrease in Young's modulus is either 6 or 11%, and the yield strength decreases by 45% (Draughn, 1981).

Because orthodontic adhesives are routinely subjected to thermal changes in the oral cavity, it is important to determine whether such temperature variations introduce stresses in the adhesive that might influence bond strength. The recommendation of the International Organisation for Standardisation about thermocycling is to expose specimens between 5°C and 55°C for 500 cycles, the exposure to each bath has to be 20 seconds and the transfer time between the two baths 5–10 seconds. Following thermocycling, studies consistently show a weaker strength of all kinds of dental adhesives compared to nonthermocycled specimens (Bishara et al., 2003; Bishara et al., 2007; Jassem et al., 1981).

Another aspect is that in fatigue testing, increasing the frequency of cycling may cause significant temperature increases and changes in the mechanical behaviour of the specimen tested.

Gap width

Gap width of a bonded joint is significantly correlated with the static and dynamic fatigue load. It has been shown that the median gap width of 0.15 mm yielded the highest static resistance of single-lap joints. Both the smaller gap of 0.01 mm and the larger gap of 0.5 mm showed significantly lower static fracture strengths. Dynamic load on the 0.01 mm bond led to only a 5% reduction in bonding strength, in contrast to the 0.15 and 0.5 mm bond, which led to a 15% reduction (Fenske et al., 2003). There seems to be an ideal gap width of 0.1mm – 0.2mm for static loading, whereas decreasing the width of the bonding layer increases the resistance to dynamic loading (Fenske et al., 2003; Wiskott et al., 1999).

Loading rate

Loading velocity during testing may have an influence on bond strength, although the data in the literature are contradicting.

The cross-head speed of the loading plate in shear testing is usually set at 0.5 mm/min for consistency (Eliades et al., 1991; Kao et al., 1995), although this value does not match any clinical conditions. *In vivo* bracket failures usually occur at much higher impact velocities, where viscoelastic properties of the adhesive are mostly absent. In a re-

cent study the chewing velocity was found to be 81 – 100 mm/sec (4860 – 6000 mm/min) and the chewing frequency 1.03 – 1.2 Hz (Buschang et al., 2000). The upper limit of chewing frequency is considered to be 2 Hz (Braem et al., 1994a).
An increased loading speed results in decreased bond strength, probably due to the induction of a stiff body response and elimination of the viscoelastic properties of the resin (Eliades et al., 2004). Changing the crosshead speed of the Zwick machine during shear bond testing from 5.0 to 0.5 mm/min increased shear bond strength by approximately 57 % and also decreased the ratio of the standard deviation to the mean value by half, from 66 % to 33 % in another study (Bishara et al., 2005).
Other researchers found no significant differences in shear bond strength for resins bonded to enamel, and cohesive vs. adhesive failure modes to be similar after testing at various cross head speeds.
For dentin, samples tested at 0.5, 1.0, and 5.0 mm/min showed significantly higher shear bond strength than the 0.1 and 10.0 mm/min specimens. Samples tested at 0.5 mm/min demonstrated strikingly better cohesive vs. adhesive results (Lindemuth and Hagge, 2000). Other investigators found higher shear bond strength and a more cohesive failure mode at higher cross head speeds in a resin-dentin test protocol (Hara et al., 2001). In another resin-dentin research protocol no significant difference was found among the same adhesive systems with different crosshead speeds tested (Yamaguchi et al., 2006).

3.3 Fatigue

The word “fatigue” originated from the Latin expression “fatigare” which means “to tire”. It has become a widely accepted terminology in engineering vocabulary for the damage and failure of materials under repeated application of stresses or strains that are below the ultimate stress, or even the yield stress of the material. Fatigue cracking is one of the primary damage mechanisms of structural components. The facts, that the original bulk design strengths are not exceeded and the only warning sign for an impending fracture is an often hard to see crack, makes fatigue damage especially dangerous.
The first study of metal fatigue is believed to have been conducted around 1829 by the German mining engineer *W. A. J. Albert* (Albert, 1838).
Wöhler conducted systematic investigations of fatigue failure during the period 1852-1869 in Berlin. He observed that the strength of steel railway axles subjected to cyclic load was lower than their static strength. His studies involved bending, torsion and axial

loading fatigue tests on railway axles for the Prussian Railway Service and components in small machines. His work (Wöhler, 1860) led to the characterisation of fatigue behaviour in terms of stress amplitude-life (S-N) curves and to the concept of fatigue “endurance limit”.

Failure may manifest itself as fracture, loss of compliance, or as wear, and is often influenced by environmental factors. The mode of stress or strain application may be static (remaining constant with time), dynamic (applied at some constant rate), or cyclic (stress or strain magnitude varying with time) (Baran et al., 2001).

Occasionally, a crack may initiate at a fault just below the surface. Eventually the cross sectional area is so reduced that the component ruptures under a normal service load, but one at a level which has been satisfactorily withstood on many previous occasions before the crack propagated. The final fracture may occur in a ductile or brittle mode depending on the characteristics of the material.

The elapsed time before failure depends on the magnitude of the applied stress or strain. However, for some materials, a lower limit of stress or strain exists below which the material may be said to possess infinite life.

During fatigue, defects in an engineering component may nucleate in an initially undamaged section and propagate in a stable manner until catastrophic fracture ensues. The progression of fatigue damage by this way can be classified in five stages (Suresh, 1998):

1. Substructural and microstructural changes which cause nucleation of permanent damage.
2. The creation of microscopic cracks.
3. The growth and coalescence of microscopic flaws to form “dominant” cracks, which may eventually lead to catastrophic failure (from a practical standpoint, this stage of fatigue generally constitutes the demarcation between crack initiation and propagation).
4. Stable propagation of the dominant macrocrack.
5. Structural instability or complete fracture.

The nucleation of microdefects and the rate of advance of the dominant fatigue crack are influenced by a wide range of factors. The primary differences among different research philosophies rest on how the crack initiation and propagation are measured. A major obstacle in developing life prediction models for fatigue lies in the definition for crack initiation. While material scientists regard the nucleation of micrometer-size flaws and roughening of fatigued surfaces as the crack inception stage of fatigue failure, practicing engineers tend to relate the resolution of their detection equipment (usually a fraction of a millimetre) with the initial crack size used for the experimental design. Within this broad range lies a variety of definitions for crack nucleation. The total fatigue life is defined as the sum of the number of cycles to initiate a fatigue crack and the number of cycles to propagate it to some final crack size (Suresh, 1998).

3.3.1 Factors affecting fatigue

Beyond the factors affecting bond strength (see chapters 3.1 and 3.2), there are various factors which influence the bond strength and the total life of orthodontic attachments under fatigue conditions.

In order for fatigue cracks to initiate, three basic factors are necessary. First, the loading pattern must contain minimum and maximum peak values with large enough variation or fluctuation (Figure 2). The peak values may be in tension or compression and may change over time but the reverse loading cycle must be sufficiently great for fatigue crack initiation. Secondly, the peak stress levels must be of sufficiently high value. If the peak stresses are too low, no crack initiation will occur. Thirdly, the material must experience a sufficiently large number of cycles of the applied stress. The number of cycles required to initiate and grow a crack is largely dependant on the first two factors (NDT Resource Center, 2001). In addition to these three basic factors, there are a host of other variables, such as stress concentration, corrosion, temperature, overload, microstructure, and residual stresses which can affect the propensity for fatigue.

Since fatigue cracks generally initiate at a surface, the surface condition of the component being loaded will have an effect on its fatigue life. Surface roughness is important because it is directly related to the level and number of stress concentrations on the surface. The higher the stress concentration the more likely a crack is to nucleate. Smooth surfaces increase the time to nucleation. Notches, scratches, and other stress risers decrease fatigue life. Surface residual stress will also have a significant effect on fatigue life. Compressive residual stresses from machining, cold working, heat treating will oppose a tensile load and thus lower the amplitude of cyclic loading (NDT Resource Center, 2001).

Increase in molecular weight has a significant effect on the resistance to fatigue crack growth (Suresh, 1998). The *Young's* modulus is a measure of resistance to deformation and related to the volumetric filler content: the higher the filler content, the higher the modulus and thus the higher the resistance to deformation. The cracks move along the filler particles, resulting in a more localised destruction and in a crack retarding mechanism (Braem et al., 1989). Microfilled materials with low filler content are thus more prone to mechanical deformation. Consequently, they deform more extensively under loading, increasing strain in the resin matrix. When the Young's modulus of the resin becomes too high, the delicate equilibrium between brittleness, tensile strength and compressive strength is disturbed. This may lead to brittle failure. It is possible that the

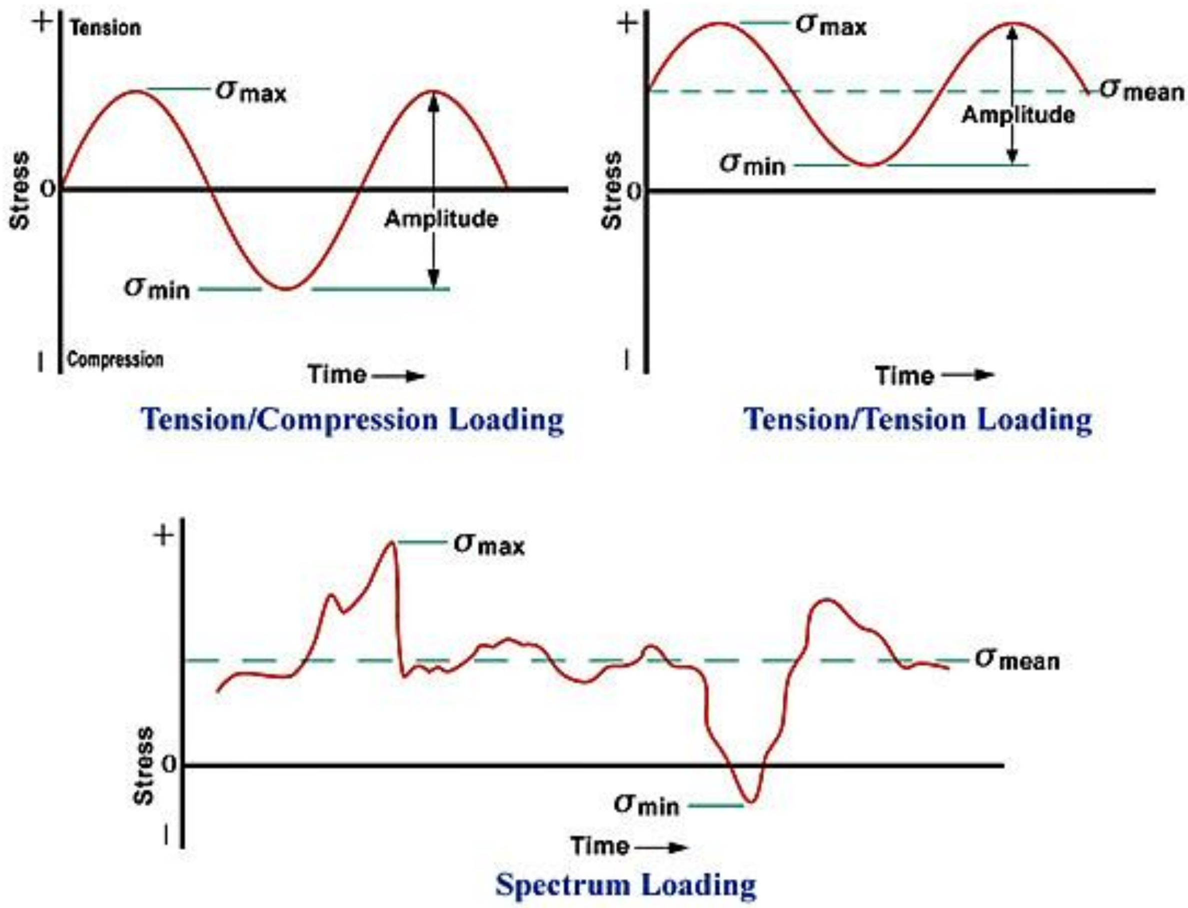

Figure 2. The figure shows several types of loading that could initiate a fatigue crack. The upper left figure shows sinusoidal loading going from a tensile stress to a compressive stress. For this type of stress cycle the maximum and minimum stresses are equal. Tensile stress is considered positive, and compressive stress is negative. The figure in the upper right shows sinusoidal loading with the minimum and maximum stresses both in the tensile realm. Cyclic compression loading can also cause fatigue. The lower figure shows variable-amplitude loading, which might be experienced by a bridge or airplane wing or any other component that experiences changing loading patterns. In variable-amplitude loading, only those cycles exceeding some peak threshold will contribute to fatigue cracking (NDT Resource Center, 2001).

Young's modulus of these resin composites has become too high, resulting in a brittle material that cannot withstand repetitive impact forces (Braem et al., 1994a).

Glass fibre reinforcements increase the fatigue resistance of composite resin and acrylic resin by acting as the stress-bearing component and by activating crack-stopping or crack-deflecting mechanisms. Factors affecting mechanical properties include the types of fibres used, the direction and pattern design of the fibre reinforcement, and uniform pre-impregnation (wetting) of the fibre with resin. Silanised glass fibres are often used because of the well-documented good adherence of treated glass fibres to the polymer matrix, thereby facilitating stress transfer from matrix to fibre and improved aesthetics. Because the mechanical properties of fibre composites depend on the direction of fibres in the polymer matrix, the reinforcing effects of continuous unidirectional fibres are anisotropic, in contrast to woven fibres, which reinforce in two directions, providing orthotropic mechanical properties (Baran et al., 2001).

In lay-ups containing a large number of fibres in the loading direction, fatigue at high applied stresses tends to be dominated by fibre properties, while at low applied stresses, matrix-related damage mechanisms have time to initiate and develop. Lay-ups containing few or no fibres in the load direction have matrix-dependent damage mechanisms occurring regardless of the applied stress level (Dyer and Isaac, 1998).

A crack will grow only if its opening changes cyclically. This requires that the loading cycle be at least partly tensile. It is not obvious that tensile loading should occur in dental restorations during mastication. Yet even when the loading conditions appear to be compressive and thus crack closing, some regions of tensile stress almost invariably exist, making fatigue feasible (Reid et al., 1990).

All the facts mentioned above lend support to the view that fatigue in dental composites expresses itself in various forms, largely depending on the materials' properties which, in turn, are principally dependent on the ratio of filler fraction to matrix-phase, giving improved mechanical behaviour together with an increase in filler fraction. The presence of defects such as air bubbles may cause stress concentration and favour catastrophic failure. Changes in the matrix-phase will have a drastic effect on the behaviour of restorations under stress, since it is the matrix-phase that is increasingly strained during the loading cycles. All the factors that interfere adversely or beneficially with the curing process, such as curing light intensity and curing time, will therefore play an important role in the life-expectancy of restorations (Braem et al., 1994a).

3.3.2 Testing methods

Fatigue research employs two major approaches (Suresh, 1998):

1. Total-life approaches
2. Defect-tolerant approaches

Using different experimental designs for the same materials, research data can be contradicting, as the degree to which the role of crack initiation and growth are incorporated in the calculation vary. Optimising materials for improved resistance to both crack initiation and propagation would require a trade-off between the recommendations of the two approaches (Suresh, 1998).

Defect-tolerant approaches

Defect-tolerant approaches, by assuming pre-existing flaw in all engineering components, define the “useful” fatigue life as the number of fatigue cycles or time to propagate the dominant crack from the initial size to some critical dimension. The flaw is determined by nondestructive flaw detection techniques (i.e. visual, dye-penetrant, x-ray, or ultrasonic, magnetic and acoustic emission methods). These approaches focus primarily on the resistance to fatigue crack growth.
If growth rate data are of interest, reference can be made to several standard procedures, such as the American Society for Testing and Materials (ASTM) specifications. In these tests, standard specimens (with or without a pre-crack) are loaded, and the rate of crack advance / crack propagation is described as a function of the difference in stress intensity.

Total-life approaches

Total-life approaches characterise the total fatigue life (initiation and propagation of cracks until failure) as a function of variables such as stress range, strain range, mean stress and environment. The number of stress/strain cycles necessary to induce failure in initially uncracked specimens is estimated under controlled stress/strain amplitudes. The initiation of dominant cracks can take up to 90 % of the cycles of the total fatigue life. These approaches focus mainly on the initiation of fatigue cracks (Figure 3).

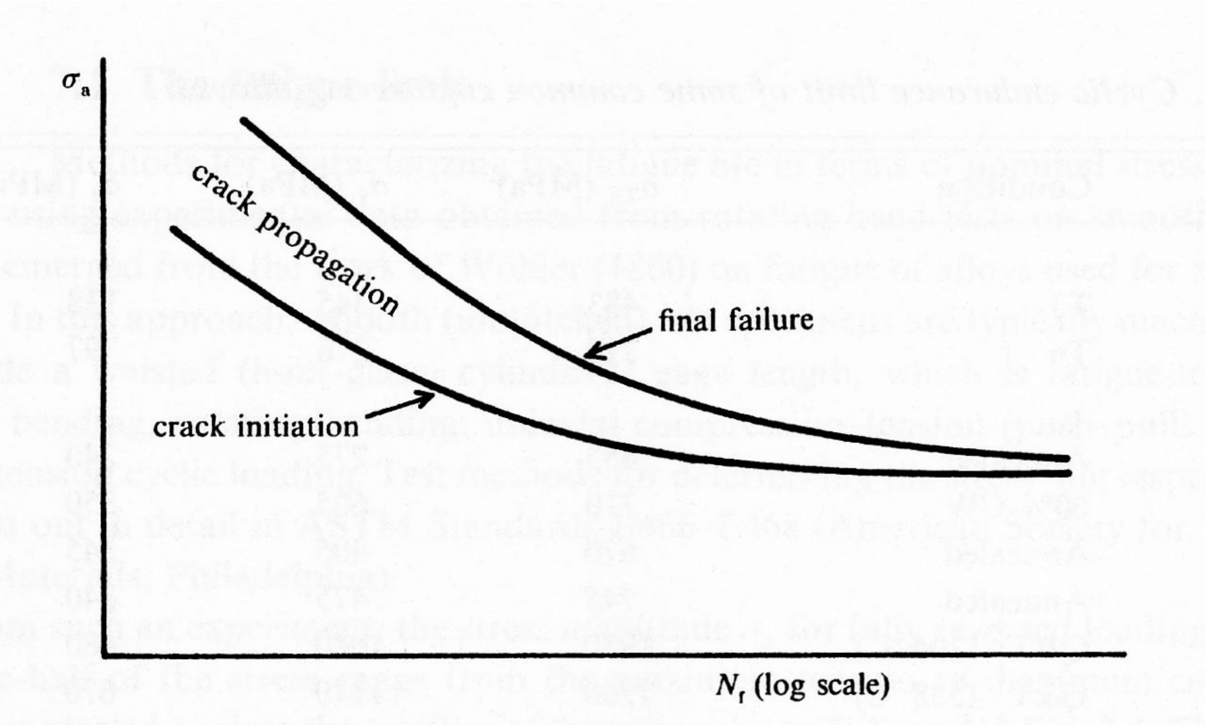

Figure 3. Stress amplitude for fatigue loading as a function of the number of cycles to failure. Contributions of crack initiation and crack propagation processes to total fatigue life in a nominally smooth specimen (Suresh, 1998).

This concept alone cannot offer a quantitative description of the intrinsic resistance of the material to fatigue. This information can be obtained only if there exists a thorough understanding of the micromechanisms of failure. Subtle changes in the microstructure and the environment can lead to drastic alterations in the extent of cyclic damage and fatigue life (Suresh, 1998).

Two of the most common experimental methods in the total-life approach are the continuous and the staircase method. With the continuous method a series of tests can be performed at various levels of stress and the numbers of cycles survived can be observed (Asmussen and Jorgensen, 1982). The staircase method is used to determine the stress levels at which a material can survive for a preset number of stress cycles and gives a value of fatigue limit for that number of cycles (Dixon and Mood, 1948; Draughn, 1979).

Continuous method

The stress life approach to fatigue was first introduced by *Wöhler* (Wöhler, 1860). The outcomes are presented as plots of the magnitude of a cyclical stress (S) against the logarithmic scale of cycles to failure (N) (Figure 3). In the S-N curve, also known as Wöhler curve, a log scale is almost always used for N. The data is obtained by cycling specimens until failure. S-N curves can be for mean life or any probability of failure and require a lot of data to be gathered.

The first test is carried out at a stress level slightly below the fracture stress measured in initial strength tests. The first specimen is tested at a high peak stress where failure is expected in a fairly short number of cycles. The test stress is decreased for each succeeding specimen and the number of fatigue cycles to failure is noted. This is done until one or two specimens do not fail in the specified numbers of cycles, which is usually at least 10^7 cycles. The highest stress at which a runout (nonfailure) occurs is taken as the fatigue threshold (endurance / fatigue limit) (NDT Resource Center, 2001).

Since the amplitude of the cyclic loading has a major effect on the fatigue performance, the S-N relationship is determined for one specific load amplitude. The amplitude is expressed as the R ratio value, which is the minimum peak stress divided by the maximum peak stress: $R = (\sigma\, min)/(\sigma\, max)$ (Figure 2). It is most common to test at an R ratio of 0.1 but families of curves, with each curve at a different R ratio, are often developed (NDT Resource Center, 2001).

Staircase method

Another technique for obtaining sensitivity data has been developed and used in explosives research in 1943 at the Explosives Research Laboratory, Bruceton, Pennsylvania (Dixon and Mood, 1948). The technique consists of choosing an initial level for testing a specimen and a succession of stress levels above and below the initial level. After observing whether it survived or failed, the test is repeated with a fresh specimen. Stress applied in each succeeding test is increased or decreased by a fixed amount, depending on whether the previous stress resulted in failure or success. The testing interval should be approximately equal to the standard deviation. This condition will be well enough satisfied if the interval actually used is between 0.5 and 2 σ (standard deviation). At the end of the tests, the results are analysed to give a value of fatigue limit for the number of fatigue cycles used.

The primary advantage of this also called “up and down method” is that it automatically concentrates testing near the mean thus increasing the accuracy of the mean estimation. For a given accuracy this will require fewer tests than testing groups of equal size at preassigned levels. A minimum of fifteen specimens is required for accurate data analysis (Dieter, 1961). Another advantage is the relative simple statistical analysis compared to the ordinary method. In some experiments though there is the obvious disadvantage of having to test each specimen separately instead of a large group simultaneously.

3.3.3 Fatigue limit

The fatigue limit is the maximum completely reversed stress for which it is assumed that the material will never fail regardless of the number of cycles. The significance of the fatigue limit is that if the material is loaded below this stress, then it will not fail, regardless of the number of times it is loaded.

It is a useful property of steel that when the stress level falls below a certain value the specimen is effectively never likely to fail. It is generally recognised that most ferrous materials exhibit a well-defined fatigue limit of about three-fourths of the material's yield stress.

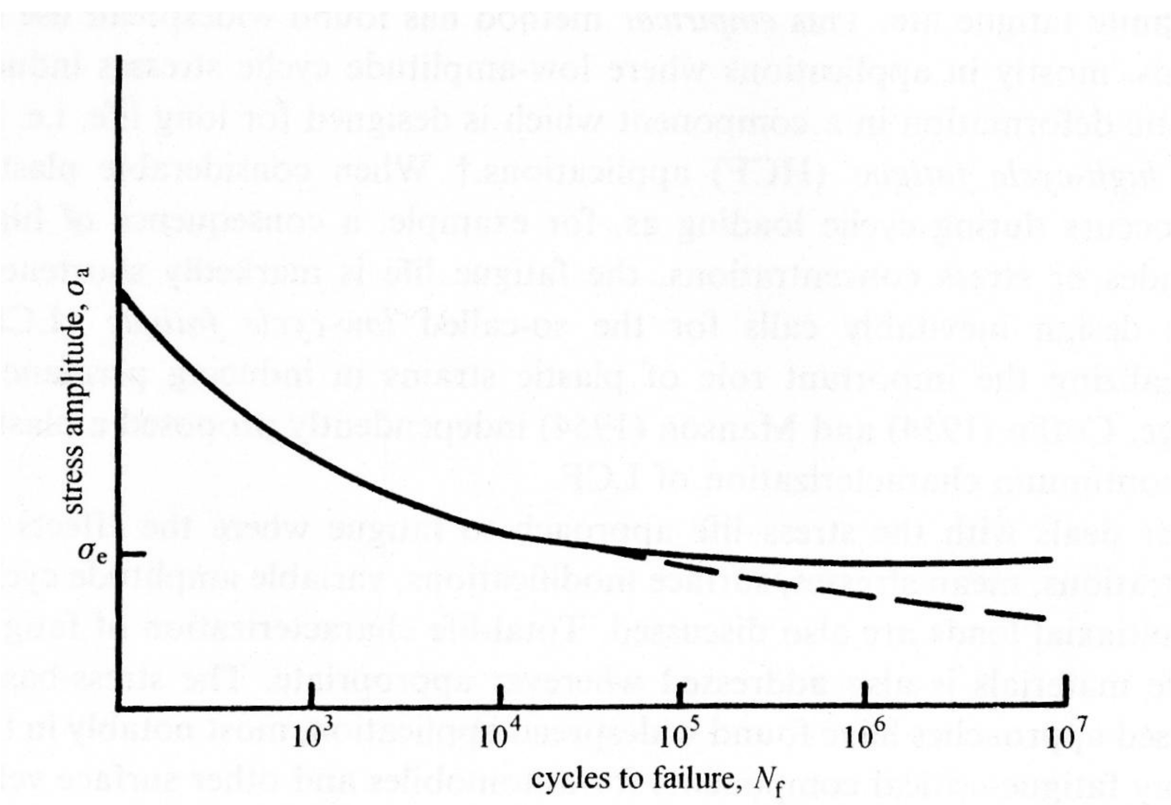

Figure 4. Typical S-N diagram showing the variation of stress amplitude for fatigue loading as a function of the number of cycles to failure for ferrous (continuous line) and nonferrous alloys (dashed line). Unlike ferrous alloys, nonferrous alloys do not generally exhibit a fatigue limit (σ_e) and the stress amplitude continues to decrease with increasing number of cycles. An endurance limit for such cases is defined as the stress amplitude which the specimen can support for at least 10^7 cycles (Suresh, 1998).

Most nonferrous metals (for example aluminium) and polymeric materials do not exhibit well-defined fatigue limits below which the material can be exposed to an infinite number of stress cycles. They exhibit a continually falling curve and the usual indicator of fatigue strength is to quote the stress below which failure will not be expected in less than a given (very large, at least 10^7) number of cycles. This is referred to as the endurance limit (Figure 4). For these materials the test is usually terminated after about 10^8 or 5×10^8 cycles. However, the fatigue and endurance limit are usually used as synonyms in the literature.

The fatigue limit is a variable, a “critical dose”, which cannot be measured in practice. This situation arises in many fields of research because in true sensitivity experiments it is not possible to make more than one observation on a given specimen since this gets altered. A common procedure in experiments of this kind is to divide the specimens into several groups and test each group at a different level. The data consist of the numbers affected and not affected at each level and can be statistically analysed (Bliss, 1935). The number of cycles required for a material to fail at a certain stress is the fatigue life.

In most dental adhesives, no fatigue limit is found. In flexural fatigue testing of dental resins for up to 10^6 cycles no fatigue limit was seen and the data had a linear relationship. At low levels of stress a tendency was demonstrated for microfilled resins to show higher fatigue strength than other types of resinous materials. (Asmussen and Jorgensen, 1982). Cyclic compression and 3-point bending testing for up to 10^4 cycles revealed no fatigue limit in that range, the data had a linear relationship and the slopes for compression and bending data were similar; more brittle materials showed no relationship between stress level and lifetime (McCabe et al., 1990). Shear and flexural fatigue testing of dental resins showed linear relationship and no fatigue limit was found for up to 250,000 cycles (Baran et al., 1998). Cyclic bending fatigue studies of resins used as matrix materials in dental composites revealed no fatigue or endurance limits (Baran et al., 1998).

Other compression fatigue tests showed however a fatigue limit (Draughn, 1979; 1988). In an evaluation of adhesive systems in combination with a hybrid composite bonded to dentin, the specimens showed a fatigue limit of 49 – 86 % compared to the initial strength after thermocycling and tensile fatigue for 5,000 cycles according to the staircase method. The fatigued specimens tended to fracture in a more cohesive mode than the nonfatigued (Frankenberger et al., 1999). A comparison among new hybrid restorative materials, conventional glass ionomers, and light-cured resins showed that the flexural fatigue limit (determined by the staircase method) of hybrid restorative materials was comparable with that of micro-filled composites (Gladys et al., 1997).

Fatigue ratio

The fatigue ratio is the ratio of fatigue limit and ultimate strength of the nonfatigued specimens.

The fatigue ratio of composite resins is reported to be constant, meaning that composite resins with higher bond strength have also higher fatigue limits (Aghadazeh Mohandesi et al., 2007; Draughn, 1979). Other investigators reported a varying fatigue ratio, depending on the filler content of the resin (Brandao et al., 2005) or its chemical structure (Aquilino et al., 1991). In a study about the fatigue of the bracket-adhesive complex on bovine teeth, the fatigue ratio varied between 68 and 92% depending on the bracket base design (Soderquist et al., 2006).

Materials providing high initial strengths do not automatically reveal the best fatigue resistance values (Lohbauer et al., 2003) and neither the elastic properties nor strength data were accurate predictors of their fatigue behaviour (Braem et al., 1995; Gladys et al., 1998). The development of high performance materials depends on a delicate balance between the type, size, shape and concentration of filler particles, as well as on the critical formation of the organic phase.

3.3.4 Fatigue behaviour

Fatigue testing of dental materials reveals two types of behaviour. In type 1 behaviour – the classic fatigue behaviour – there is a clear relationship between fatigue life and fatigue stress, i.e. the fatigue life decreases with increasing applied fatigue stress (McCabe et al., 1990). The data can fit a power law – straight line, no apparent fatigue limit – or a hyperbolic law, implying differences between low and high cycle fatigue, as well as a fatigue limit (Figure 4). Physically, the transition from low cycle to high cycle fatigue is related to competition between propagation of surface cracks and bulk microcracks. The presence of a fatigue limit indicates that two mechanisms or two flaw populations are active. A straight line indicates that only one type of flaw is responsible for failure (Baran et al., 1998).

For type 2 behaviour no relationship exists between fatigue life and fatigue stress. The failure occurs at a level of stress below the ultimate strength of the material, but the values of fatigue life appear to be distributed randomly when several specimens of the same material are tested. It is likely that, for these materials, resistance to fatigue is primarily dependent upon the presence or absence of flaws such as porosities. When a stress in the region of the fatigue limit is used, specimens with no flaws above a critical size are likely to survive indefinitely, while those with flaws above a critical size fail rapidly (McCabe et al., 1990).

A value of fatigue limit can be calculated in both cases. For type 1 materials with data following a power law, there is a fatigue limit for each value of testing cycles. If the relationship follows a hyperbolic law, then there is one stress – one fatigue limit – below which no fracture will occur, even at high cycle fatigue (Figure 4). Type 2 materials are characterised by one fatigue limit which is independent of fatigue life (survival time) and is the same for all values of testing cycles (Figures 5 and 6). For the dental materials, it appears that brittle materials such as dental plaster and heavily filled composites are likely to exhibit type 2 behaviour, whereas less brittle materials, such as more lightly filled composites, are more likely to exhibit type 1 behaviour (McCabe et al., 1990).

For type 1 behaviour, testing should be carried out over a range of stresses and number of cycles in order to characterise the relationship between the two properties. The method of continuous cycling to failure at varying stresses by testing about 30 specimens would be appropriate (Figure 5). If staircase testing is carried out in order to de-

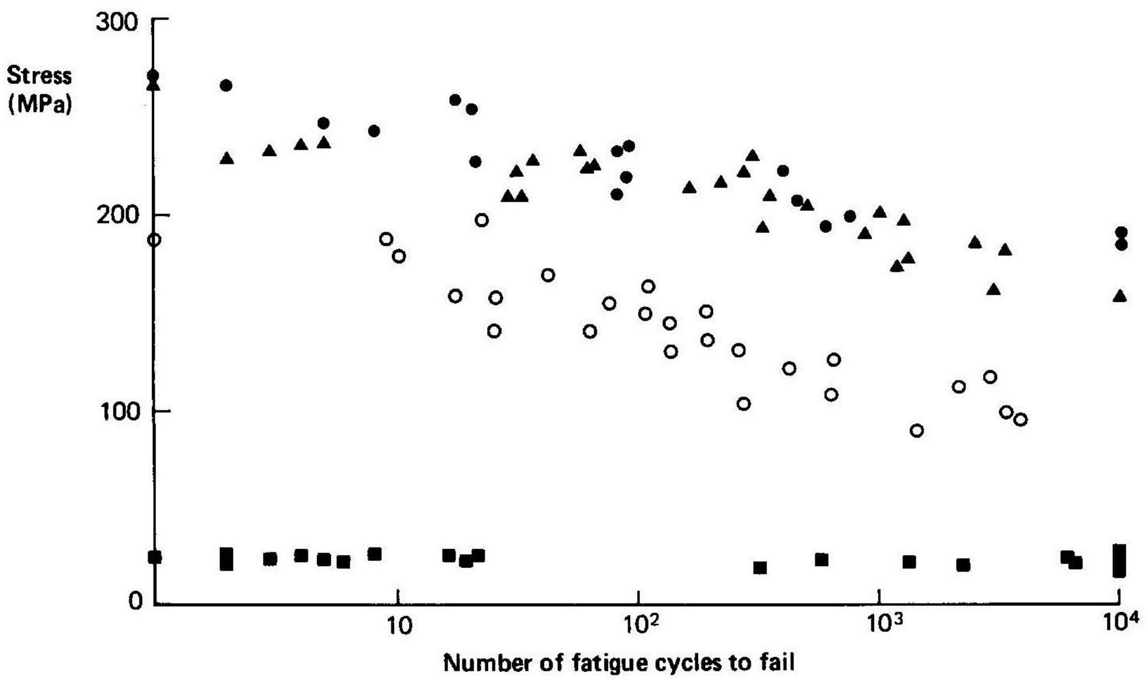

Figure 5. Applied stress vs. number of fatigue cycles to fail. Data collected by the continuous-cycling-to-fail method. The squares represent fatigue behaviour type 2 and the circles and triangles fatigue behaviour type 1 (McCabe et al., 1990).

termine a fatigue limit, the testing should be carried out at a number of preselected fatigue life values (number of fatigue cycles). For each value of fatigue life about 15 specimens would be necessary. This requires the use of more specimens compared to the continuous method but gives data in which fatigue stress and fatigue life are better correlated (Figure 6). The measurement of a single fatigue limit value at one selected number of test cycles may not be a satisfactory way of evaluating type 1 materials. Such a method would only be valid in comparing two materials with similar fatigue behaviour, i.e. if the better performing material at high stresses also performs better at low stresses. If the fatigue behaviours are too different, the curves of two materials would intersect and cross. This would mean that at high stresses one material performs better than the other and at low stresses the position is reversed (McCabe et al., 1990).
If the character of a material is established as type 2, staircase testing can be adopted in any case for future evaluation (McCabe et al., 1990).

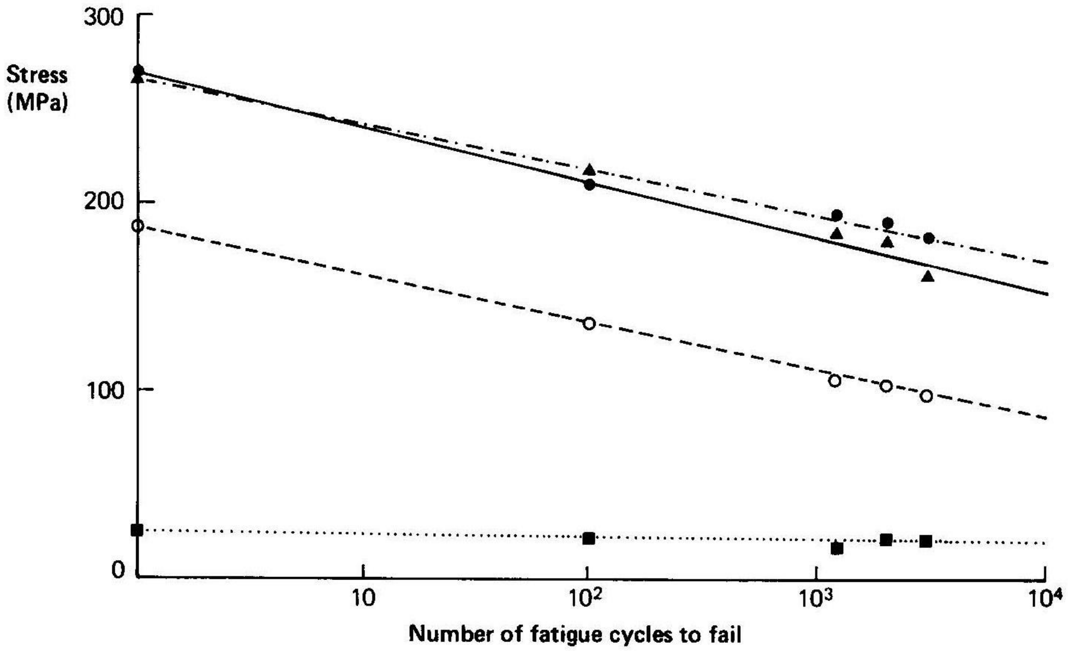

Figure 6. Applied stress vs. number of applied fatigue cycles to fail. Each experimental point results from tests of 17 specimens using staircase testing. The squares represent fatigue behaviour type 2 and the circles and triangles fatigue behaviour type 1 (McCabe et al., 1990).

3.3.5 Fatigue in composites

Although metal fatigue is well described and mathematically documented, this is not the case for resin composites. No other class of materials has such great fatigue strength, even when compared to their high initial strength, like composite materials (Reifsnider, 1980).

Composites typically consist of high-modulus, brittle reinforcing fibres or particles dispersed in a quasi-brittle polymer matrix. Maximum strengthening of these engineered materials occurs when load is transferred from the matrix to the reinforcing phase. Depending on the type of reinforcement (e.g. particulate or fibrous), the direction of load application, the strengths of the various phases, and the interfacial strength, several mechanisms may participate in fatigue-induced damage of composite materials. These include matrix cracking, matrix deformation, void formation, multidirectional cracking, filler debonding, and filler failure. Consequently, scatter in fatigue data for composites is greater than in monolithic materials, where typically a single damage mechanism is presumed to be active. The choice of dominant mechanism is also influenced by the mode of load application: In cyclic fatigue, voids are more likely to form at the fibre-matrix interface than during monotonic loading (Horst and Spoormaker, 1997).

When sufficient microcrack damage has accumulated via the mechanisms described above, a macrocrack is initiated, and its presence changes the compliance of the bulk composite. This change in compliance is often useful in defining fatigue life, since the load-bearing capacity of the composite structure deteriorates well before actual failure through the specimen; strength vs. cycles to failure (S/N) data can exhibit more than one change in compliance (Dyer and Isaac, 1998).

Final failure can take place over a wide variation in final crack sizes; at higher applied stresses, short cracks are responsible for failure, while at low stresses, long cracks are responsible for failure (Suresh, 1998).

There is a concern that, because of the wide variety of fatigue damage modes occurring within composites, crack growth rate measurements are not an appropriate design approach for predicting lifetimes of composites. There usually are many cracks which form in the matrix and in the reinforcement. The propagation rates and directions of these microcracks are continuously modified during the fatigue process as a result of changes in the distribution of internal stresses. In addition, theories of crack propagation were developed for isotropic materials. In composites, the reinforcement materials have not "one" strength, but rather a statistical strength distribution further accentuated by the fact that particulate fillers are typically not monodisperse, and fibre diameters vary. Therefore, the size effect needs to be considered in dealing with the strength of the reinforcing phase (Reifsnider, 1980).

Correlation of strength and fatigue data

Increasing the toughness of the matrix material improves the lifetime and fatigue strength of the composite. Factors improving strength should also improve fatigue resistance. However the literature shows weak correlations between the ultimate and fatigue strength of dental materials.

Some authors showed weak correlations between monotonic flexure strength and resistance to fatigue loading of composites and an acrylic resin for provisional and definitive restorations. Because fatigue tests were considered more pertinent than monotonic tests as to their predictive value, it was concluded that flexure strength data alone may not provide relevant information for long-term clinical performance. The material's resistance to fatigue loading should also be determined (Scherrer et al., 2003).

Initial fracture strengths of some restorative resin composites did not correlate with a clinically more relevant fatigue loading (Lohbauer et al., 2005).

Influence of fatigue on adhesive failure

In vitro fatigue resistance of dental materials has been studied using compressive, flexural and tensile tests in which the load regimen differed between load-controlled and strain-controlled tests. Few studies have compared the bond strength of fatigued and nonfatigued specimens. Most authors describe only the bond strength, under which the specimens fail during fatigue, a value corresponding to the fatigue limit (Aghadazeh Mohandesi et al., 2007; Drummond and Savers, 1993; Soderquist et al., 2006).

The available data about the behaviour of composite restorative materials after cyclic fatigue is not uniform. Most fatigue studies showed no decrease of the bond strength after cyclic loading (Staninec et al., 2008; Williamson et al., 1993). Another study revealed an influence of fatigue depending on the chemical structure of the adhesive, i.e. decreased bond strength of a fatigued 4-Meta-adhesive and no influence on a Bis-GMA-system (Aquilino et al., 1991). The only fatigue study about the bracket-adhesive complex comparing fatigued and nonfatigued specimens showed an influence of fatigue depending on the magnitude of the cyclic loading (Moseley et al., 1995).

3.4 Data analysis

3.4.1 Bond strength

Force vs. stress measurement

Strength testing is commonly reported as an average stress that is computed by dividing the experimentally measured force at failure by the area of the bracket base. This evaluation may be misleading. Stress distribution within the bracket-adhesive complex is mainly inhomogeneous. Rough bracket bases and fluctuations of the resin thickness contribute to an inhomogeneous load application. Increasing the roughness of the bracket base leads to a larger overall surface (Eliades et al., 1991).
Measuring the bond strength in terms of mean stress leads to values which have little to do with the actual inhomogeneous stress distribution in the adhesive. Measuring the mean stress can be a proper way to evaluate the effectiveness of the bracket base against strength testing. The effectiveness of the base is usually irrelevant to the properties of the bracket as a whole, i.e. the force at which the system fails. A clinician is interested in the critical force of failure and not the assumed mean stress. Nevertheless a less effective base can be compensated by increasing its area and vice versa. This is supported by some authors, who proved the independence of the bond strength from the nominal area of bracket bases (Dickinson and Powers, 1980).

Statistics

The bond strength values obtained by tensile or shear testing generally show large coefficients of variation, i.e. 20-50 %, and should be tested statistically by an appropriate method. If the variation is above 50 %, a thorough inspection of the overall procedure is recommended.
Ultimate bond strength and fatigue bond strength results should be based on sound statistical methods and a sufficient number of specimens. If the data are normally distributed, a mean, standard deviation and coefficient of variation can be calculated. Means can be compared by analysis of variance (ANOVA). A mean value signifies an expectation of 50 % failure.
Sample sizes of less than 10 specimens per group are likely to not follow a normal distribution, which is a fundamental assumption for the use of statistical tests such as

ANOVA. Research papers reporting mean bond strength values derived from groups containing less than 10 specimens have been strongly criticised (Fox et al., 1994).

The use of pairwise multiple comparison tests in the post hoc analysis of ANOVA, such as Tukey, Duncan and Student-Newman-Keuls analysis, can more clearly distinguish the difference between pairs. However they should be wisely used, as they can yield different results (Eliades and Brantley, 2000).

S-N curves can be formed for mean life or any probability of failure. Therefore, the use of probability of failure, calculated from the Weibull distribution function, provides a suitable means of comparing many materials and can be also used when the results from adhesion testing are not normally distributed. The stress to give 10 % failure ($P_{f\,10}$) and that to give 90 % failure ($P_{f\,90}$) are convenient ways of characterising the strength of a bond. A minimum of 15 specimens is required in each group for the application of Weibull statistics (DIN Deutsches Institut für Normung e.V., 2002). The Weibull distribution incorporates the Weibull modulus, which stands for consistency of a material, even distribution of defects and less scatter of results. A good quality material is considered to have a Weibull modulus >10.

When using the staircase technique, the mean fatigue limit and its standard deviation for a specific number of cycles are calculated within a relatively simple statistical analysis (Dixon and Mood, 1948; Draughn, 1979).

3.4.2 Fractography

Fractography is the study of fractured surfaces. “Post-mortem” analyses of fatigue failures often involve tracing the origin of fatigue failure via microscopic features present on the fracture surfaces, such as “clam shell” markings and striations. These can provide valuable information about the location where fracture initiated as well as about the magnitude of loads imposed upon the failed component. A fundamental knowledge of the link between the characteristic features observed on the fatigue fracture surfaces, the microscopic mechanisms of failure and the macroscopic rates of crack advance is vital to the success of such post-mortem analyses (Suresh, 1998). Scanning electron microscopy is an ideal means of evaluating fractography (Retief, 1974).

The presence of air voids results in inhomogeneous fracture resistance. Consequently, crack initiation sites are at the air voids with subsequent crack growth. Large porosity has a detrimental effect on fatigue life too. The presence of air voids promotes heterogeneous fatigue crack initiation and early crack growth. A staggering fatigue life reduction of 98 % because of a large air void was shown, thus justifying the importance of proper layering of composites during the manufacturing process. Initial stress concentrations at pores decreases flexural strength, lowers resistance to fatigue, and increases wear (Aghadazeh Mohandesi et al., 2007).

It is also noteworthy that fatigue cracks may initiate from defects and impurities – especially those located in areas of high stress. Large defects would seem to be pivotal in reducing fatigue life since they could significantly reduce the fatigue crack initiation period by promoting heterogeneous crack initiation (Aghadazeh Mohandesi et al., 2007).

Fractographic data of dental resins after flexure and shear fatigue testing showed that a single flaw population is responsible for fracture initiation. All specimens showed fracture initiation at a surface flaw, in a tensile portion of the surface or at an edge. Liquid sorption, the presence of low amounts of filler and the stress level (and the number of cycles to failure) did not affect the site where fracture initiated. Fracture surface morphology was however visibly affected by liquid sorption and the number of cycles to failure (Baran et al., 1998). For PMMA bone cement, it was reported that all fatigue cracks initiate at internal pores and that porosity, pore size, and pore size distribution affected crack initiation and fatigue behaviour (James et al., 1992).

Yet, there is also criticism on fractographic studies. Failure analyses intending to provide inferences about the strength of the individual components of the bonding system based on their interfacial fracture characteristics, should be questioned (Eliades et al., 1993). The site of failure may arise from crack initiation caused by higher stresses compared with other areas, which is not taken into consideration in the traditional assumption of homogeneous stress (Eliades and Brantley, 2000). The failure pattern depends on many other factors except the strength of the adhesive. The interactions between the components of the bracket-adhesive system should be considered in a failure mode analysis. Crack initiation and propagation can be influenced by neighbour structures. For example, the microscopic and macroscopic structure of a ceramic bracket covered with a silane layer can reinforce the adhesive layer, which fractures at higher values than the resin-etched enamel, resulting in crack growth into the enamel and tooth damage (Eliades et al., 1993).

Mechanics of fatigue crack growth in polymers

Fatigue striations

Fractographic data of fatigued surfaces of polymers reveal microscopic striations. These striations are formed due to periodic opening and closing of the tip of an advancing crack under cyclic loading – a mechanism analogous for the striations observed under reversed cyclic loading. Each striation spacing represents the crack growth per cycle. Therefore, the fatigue crack growth rate may be approximately estimated by a fractographic study of the fracture surface (Aghadazeh Mohandesi et al., 2007).
Under compressive fatigue at 10 Hz the striation spacing of dental composites varies between 0.8 μm to 1.2 μm and the rate of fatigue crack growth varies between $0.8 \times 10{-}3$ to $1.2{\times}10{-}3$ mm/cycle (Aghadazeh Mohandesi et al., 2007).

Discontinuous growth bands

These growth bands correspond to a single burst of fatigue crack advance after every several hundred fatigue cycles. They resemble striations, but their spacing is larger. Interpretations of their formation centre on the conception that crazing over many cycles causes the crack to jump suddenly. During cyclic loading, the accumulation of fatigue damage results in a gradual increase in stress around the crack tip. Even though this remains stationary, crazing (fine cracking) is formed. When the stress reaches a critical value, crack growth occurs suddenly within the craze. Most of the fatigue lifetime of a polymer is spend in discontinuous crack growth.

Combined effects

In some polymeric materials, discontinuous crack growth occurs as a consequence of crazing ahead of the crack tip and shear banding at 45° above and below the crack plane. The resultant crack tip process zone has the shape of the Greek letter ε (epsilon) and is called epsilon discontinuous crack growth. It is usually observed at low stress intensity (difference of minimum and maximum stress in a cycle) and with short fatigue cracks. The successive growth, termination and regeneration of epsilon regions produce complex crack shapes and very long fatigue crack lifetimes.

Shear bands

These are bands at 45° above and below the crack plane (loading direction) and they usually derive from an epsilon discontinuous growth band. In the next stages of fatigue, some of them join together and begin to propagate through the material in a direction that is perpendicular to the maximum tensile stress.
Increase of the applied stress intensity or test temperature cause the microscopic fracture mode in polymers to change in the following sequence: discontinuous growth bands formed by crack tip crazing → epsilon discontinuous growth bands formed by the combined effects of crazing and shear banding→ shear bands (Suresh, 1998).

Characteristic appearance of fatigue cracks

Under continued stress, the cracks continue to propagate as the matrix is weakened by their presence and start to connect. Eventually, the growth of one crack or a few of the larger cracks will dominate over the rest of the cracks. With continued cyclic loading, the growth of the dominate crack or cracks will continue until the remaining uncracked section of the component can no longer support the load. At this point, the fracture toughness is exceeded and the remaining cross-section of the material experiences rapid fracture. This rapid overload fracture is the last stage of fatigue failure (NDT Resource Center, 2001).
The crack growth mechanisms eventually lead to bigger shapes, like complex tree-like shapes or clamshell markings. The characteristic appearance of fatigue fractures reflects the initiation site and the progressive development of the crack front, culminating in an area of final overload fracture. A fatigue fracture will have two distinct regions: One being smooth or burnished as a result of the rubbing of the bottom and top of the crack; the second is granular, due to the rapid failure of the material. Clamshell-like markings, often referred to as beach markings because of their resemblance to the ridges left in the sand by retreating waves, are caused by arrests in the crack front as it propagates through the section. They are found if the fatigue has been interrupted and may contain thousands of striations (Figure 7).
Both the smooth surfaces and the clamshell markings are found at the side from where the crack initiated. The opposite side to the initiation site is usually the final region of ductile fracture. Sometimes there may be more than one initiation point and two or more cracks propagate. This produces features with the final area of ductile fracture being a band across the middle. This type of fracture is typical of double bending where a com-

ponent is cyclically strained in one plane or where a second fatigue crack initiates at the opposite side to a developing crack in a component subjected to reverse bending. Some stress-induced fatigue failures may show multiple initiation sites from which separate cracks spread towards a common meeting point within the section.

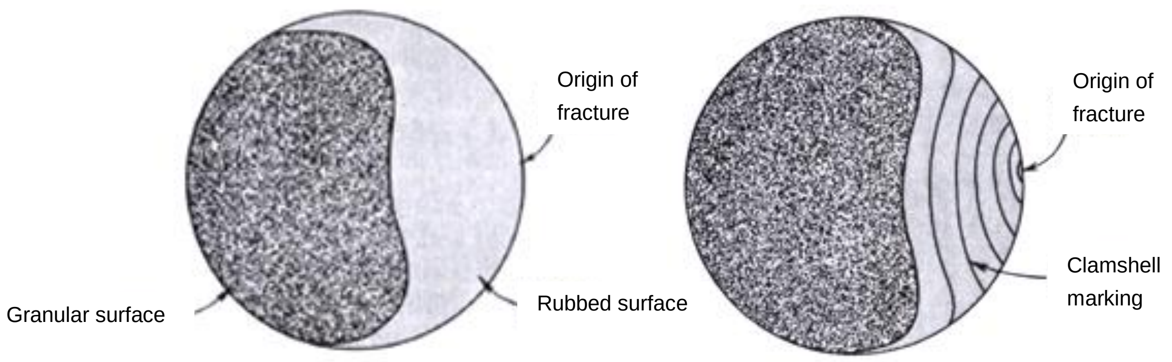

Figure 7. Two distinct regions of fatigue fractures. The crack initiates on the right and propagates to the left creating smoothed surfaces or clamshell markings. When the material fails, a granular surface on the left side is produced (Shawn, 1997).

Fracture mode of bonding processes

The fracture is “adhesive” or “interfacial” when debonding occurs between the adhesive and the adherent. In most cases, the occurrence of “interfacial” fracture for a given adhesive goes along with smaller fracture toughness.

The strongest bonding is achieved when the bond is “cohesive,” that is the adhesive remains after debonding in almost equal proportions on both substrates. In the case of an ideal affinity between substrates and adhesives, the toughness of the last one becomes the limiting factor (Matasa, 1989). The crack may propagate in the centre of the layer or near an interface. For this last case, the “cohesive” fracture can be said to be “cohesive near the interface”. Most quality control standards consider that a “good” adhesive bonding must be “cohesive”.

The fracture type is called “mixed” if the crack propagates at some spots in a “cohesive” and in others in an “interfacial” manner. “Mixed” fracture surfaces can be characterised by a certain percentage of “adhesive” and “cohesive” areas. The “alternating crack path” fracture type occurs if the cracks jump from one interface to the other. This type of fracture appears in the presence of tensile pre-stresses in the adhesive layer.

A major goal in bracket-adhesive-technique is a compromising solution between a weak bond strength leading to a high clinical failure rate and too strong bonding with a high risk for enamel detachment during debonding.

Furthermore, a smooth tooth surface after debonding is desirable, because the necessary cleaning time is reduced. However, if the adhesive remains entirely on the bracket base and the debonding procedure is uncontrolled, the risk for enamel detachment is increased. In these cases it should be investigated, if enamel particles remain on the adhesive. If true, research should aim in changing the bonding protocol, the adhesive specifications or the debonding procedure, so that the fracture line remains in the adhesive. The whole enamel surface should be covered with a thin coat of adhesive, leaving us with a good compromise between good clinical performance and a low chairside time for the orthodontist. Some authors suggest debonding by fracturing the adhesive with a sharp cutter (Caspersen, 1977; Diedrich, 1980) and others by pressing the bracket wings together (Oliver, 1988).

Adhesive Remnant Index – ARI

The ARI is a system for evaluating the amount of adhesive left on the tooth or the corresponding bracket surface after debonding. The possible scores are: 0, no adhesive left on the surface; 1, < 50% of the adhesive left on the surface; 2, > 50% of the adhesive left on the surface; 3, all adhesive left on the surface (Artun and Bergland, 1984). The ARI has been used by many investigators to help standardise bond failure analysis. The ARI may oversimplify the very complex issues of bond failure analysis, but it does allow statistical analysis and cross-study comparisons. A review of the literature reveals that although many investigators use an ARI system for their project, they often modify the criteria, the numbering system, or both.

Enamel detachment index – EDI

The EDI is an approach used to assess the quantity of detached enamel remaining on the bracket bases after debonding. The possible scores are: 0, no enamel detachment; 1, less than 10 % enamel detachment; 2, more than 10 % but less than 30 % enamel detachment (Sorel et al., 2000). A correlation between ARI and EDI showed that the possibility of enamel detachment increases when more adhesive remains on the bracket after debonding (Sorel et al., 2002).

4 Purpose of the study

Fatigue in dentistry is mainly described for materials used in restorations. There are only few investigations on fatigue of brackets bonded on a substrate (Moseley et al., 1995; Soderquist et al., 2006).

The goal of this study was to describe the effect of fatigue on the bracket-adhesive complex by standardising as many factors known to influence shear strength and fatigue shear strength as possible.

This was done in two ways. Firstly the shear strength of nonfatigued and fatigued specimens was compared. Then the fracture surfaces were examined for different fracture modes between the nonfatigued and fatigued specimens.

The only varying factors in this investigation were the materials used. If affecting the influence of fatigue, they were pointed out and their influence on shear strength and visual fracture characteristic was presented.

5 Materials and methods

5.1 Brackets and adhesives

Brackets with relatively flat bases were preferred, so as to have a quite uniform adhesive layer thickness. Therefore brackets intended to be bonded on lower incisors were chosen.

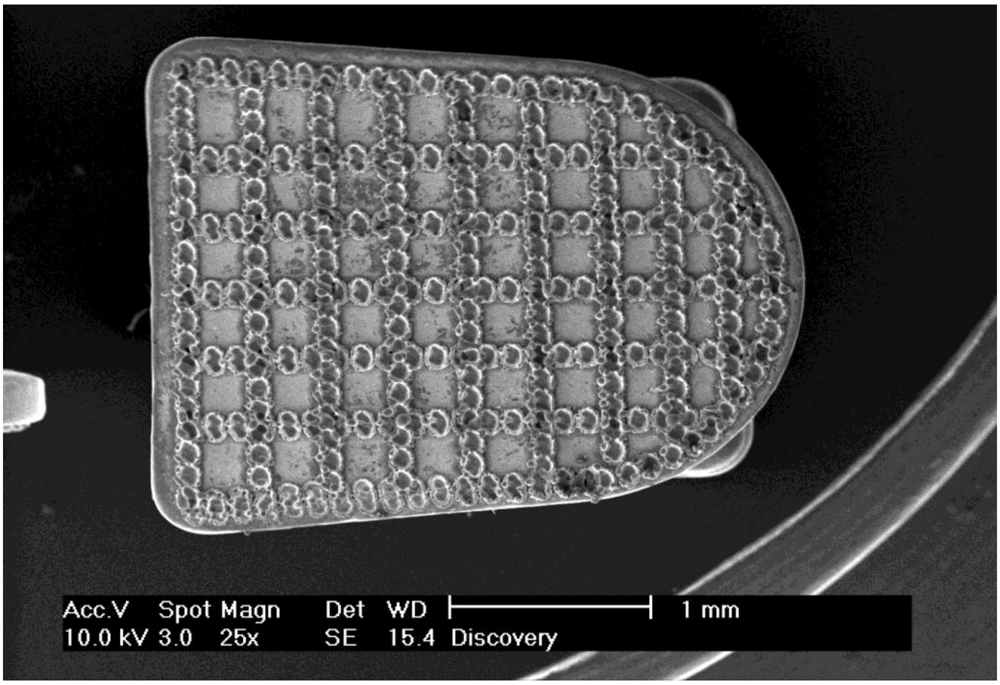

Figure 8. Bracket Discovery®: Laser structured base. SEM-image. 25x magnification.

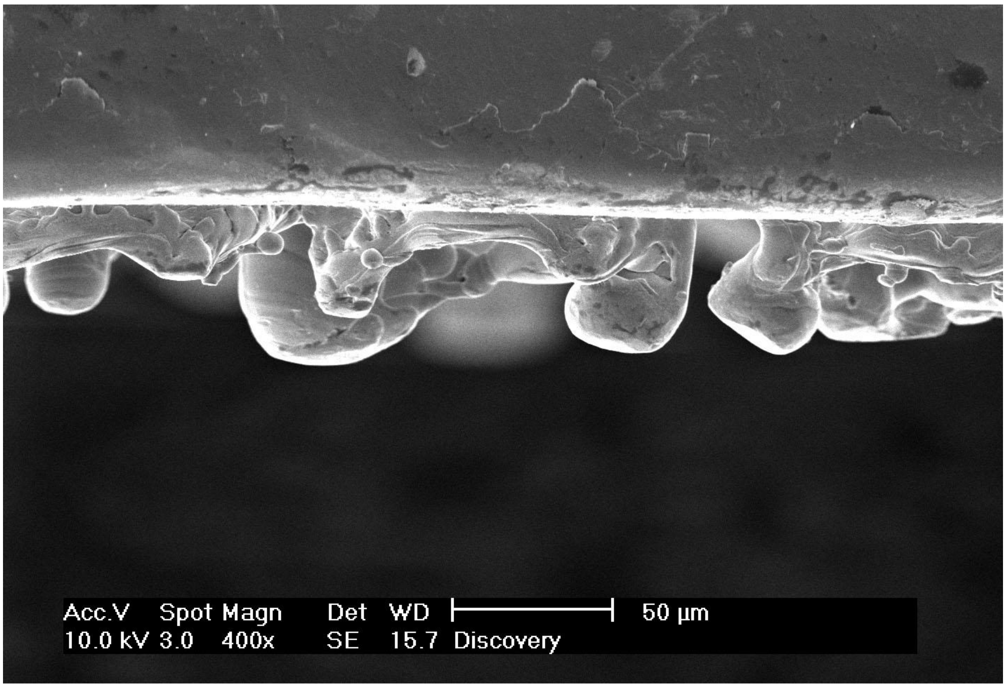

Figure 9. Bracket Discovery®: Laser structured base (profile). SEM-image. 400x magnification.

In order to compare different bases, one type with a laser structured base (Discovery®, Dentaurum J. P. Winkelstroeter KG, Figures 8 and 9) and one with a foil mesh base (Ultra–Minitrim®, Dentaurum J. P. Winkelstroeter KG, Figures 10 and 11), were selected. The comparison of the two bases makes clear, that the laser structured base has more irregularities and undercuts and thus a bigger surface.

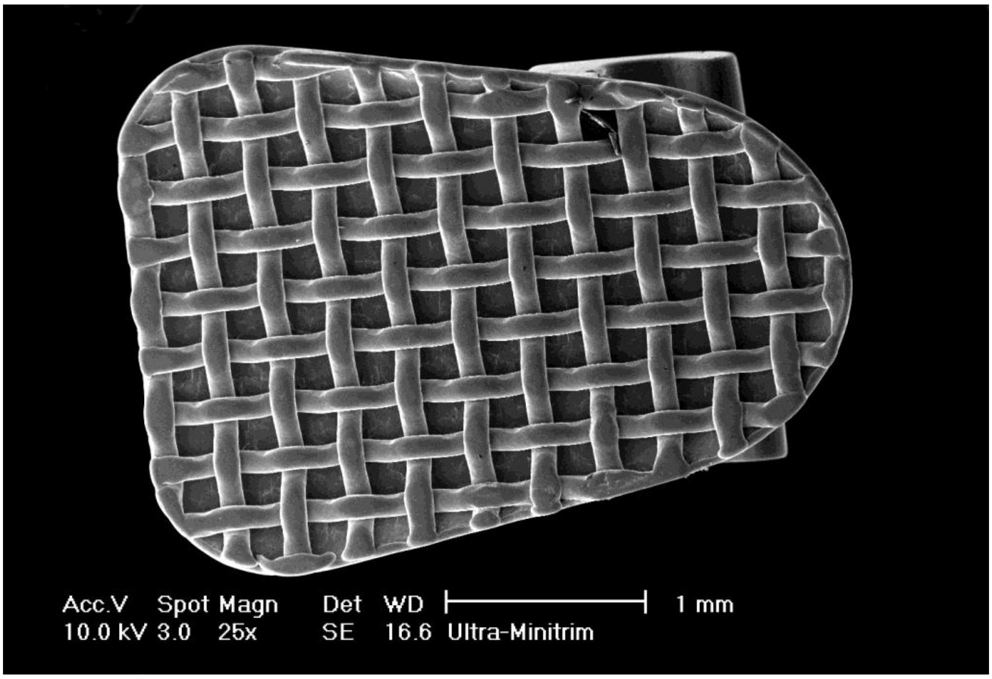

Figure 10. Bracket Ultra-Minitrim®: Foil mesh base. SEM-image. 25x magnification.

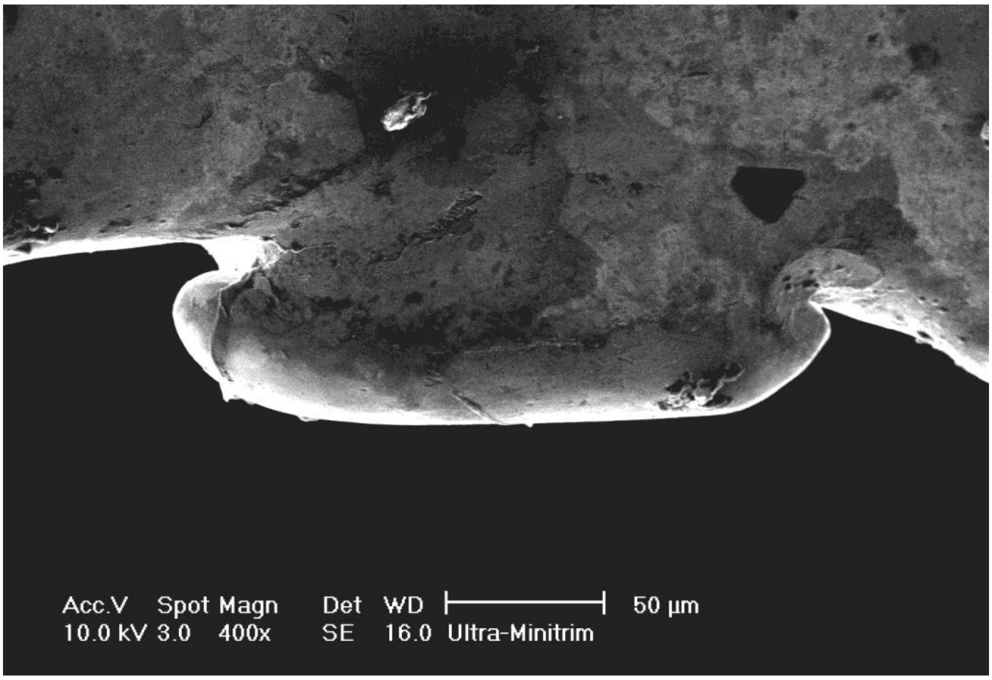

Figure 11. Bracket Ultra-Minitrim®. Foil mesh base (profile). SEM-image. 400x magnification.

	No-Mix			Concise™		
	Adhesive	**Activator**	**Average**	**Paste A/B**	**Resin A/B**	**Average**
Filler particle size	0.04 – 23.7 µm		0.04 – 23.7 µm	9 µm		9 µm
Filler particles [% by wt]	72.5-75.5	0	37	70-85	0	38.75
Bis-GMA [% by wt]	9.3	7.9	8.6	10-20	40-50	30
TEGDMA [% by wt]	13.2	71.5	42.35	1-10	40-50	25.25

Table 1. Constitution of the used composite resins.

In order to compare different adhesives, one two-component (No-Mix Bonding System, Dentaurum J. P. Winkelstroeter KG) and one four-component chemically-curing adhesive (Concise™, 3M Unitek) were chosen, both containing Bis-GMA and TEGDMA monomers. The constitution is presented in Table 1.

No-Mix is polymerised by placing a thin coat of activator fluid on the bracket base and the surface to be bonded to and adhesive on the bracket onto the activator coating. By pressing the bracket on the surface, the adhesive is squeezed and activated from both sides. Concise™ is polymerised by separate mixing of paste A / B and resin A / B and then by mixing them together.

Stainless steel flat plates of 5 mm thickness and 50 mm diameter were cut from a stainless steel cylinder (Remanit 4404, type 316L 2, Edelstahl Witten-Krefeld GmbH) to facilitate a standardised substrate to which the brackets were bonded.

5.2 Specimen preparation

The surfaces of the steel plates were prepared according to the “Rocatec®-System” (3M ESPE, 2001). First they were cleaned and roughened by blasting with 110 µm aluminium oxide sand (high-purity aluminium oxide, Rocatec Pre) for 10 seconds at 2.8 bar. This activated the surface and created a uniform pattern of surface roughness which is ideal for ensuring of microretentive anchorage of the resin. The microblasted surface

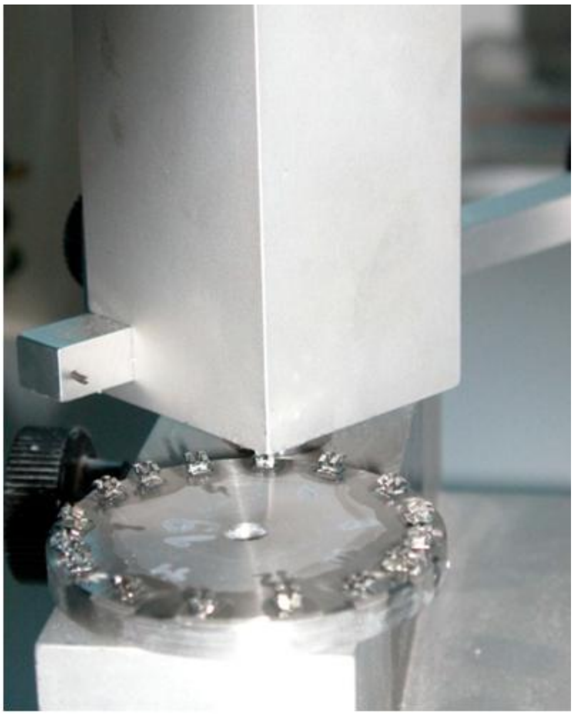

Figure 12. A vertically gliding weight was used to apply a compressing force on each bracket.

was tribochemically coated by spraying with silica-modified aluminium oxide (Rocatec Plus) for 15 seconds at 2.8 bar. This consists of the sand described above (Pre) coated with a thin layer of SiO_2 (silica or silicon dioxide, 110 µm Al_2O_3+ SiO_2 = Rocatec Plus).

Apart from ceramicising the surface, the impact of the particles also causes a certain amount of abrasion. The affected surfaces of the substrate and grit in the atomic and molecular ranges are excited to such an extent that a so-called triboplasma forms. The SiO_2 is impregnated into the surface up to a depth of 15 µm and at the same time fused to the surface in islands. The coated surfaces had to be conditioned in order to be able to create a bond with the resin by silanisation with a dual molecule silane fluid (3M ESPE Sil), which was left to dry out for 5 minutes. This can react with the inorganic silicatised surface at one end and with any organic methacrylated monomer system (MMA, Bis-GMA, etc.) at the other. The resulting anchorage roughly corresponds to the chemical bonding of silanised fillers in composite.

The brackets were bonded to the silanised plates using the adhesives according to the manufacturer instructions. A special device with a vertically gliding weight was used to achieve the same force of 4 N for pressing the brackets on the plates (Figure 12).

Material Combination	**A**	**B**	**C**	**D**
Bracket type	Discovery®	Ultra-Minitrim®	Discovery®	Ultra-Minitrim®
Adhesive type	No-Mix	No-Mix	Concise™	Concise™

Table 2. The analysed material combinations of brackets and adhesives.

Four combinations of materials which resulted from two bracket types and two adhesive types according to Table 2 were analysed. The same bracket and adhesive type was used for 2 metal discs resulting in 8 discs bonded with 15 - 17 brackets each. The brackets were placed circumferentially on the discs in order to be easily reached by the cross head of the testing machine.

Each disc was stored in distilled water at 37°C for 3 days. The temperature was adjusted by a special aquarium heating device and was controlled by an electronic thermometer (Figure 13).

Figure 13. Aging of the specimens in a pot of 37°C distilled water containing an aquarium heating device and an electronic thermometer.

5.3 Testing procedure

Each material combination was divided into two groups: The first group of brackets, bonded on the first disc, was used as a control to determine the ultimate shear bond strength without any fatigue-procedure. The brackets of the second group, bonded on the second disc, underwent fatigue testing.

The discs were mounted into a mechanical testing machine Zwick 1445 (Zwick GmbH & Co) (Figure 14), which was adjusted for applying a load through a flat steel head (Figures 15, 16 and 17). The movement of the head and the magnitude of the applied force was electronically controlled and monitored.

Figure 14. The universal testing machine Zwick 1445.

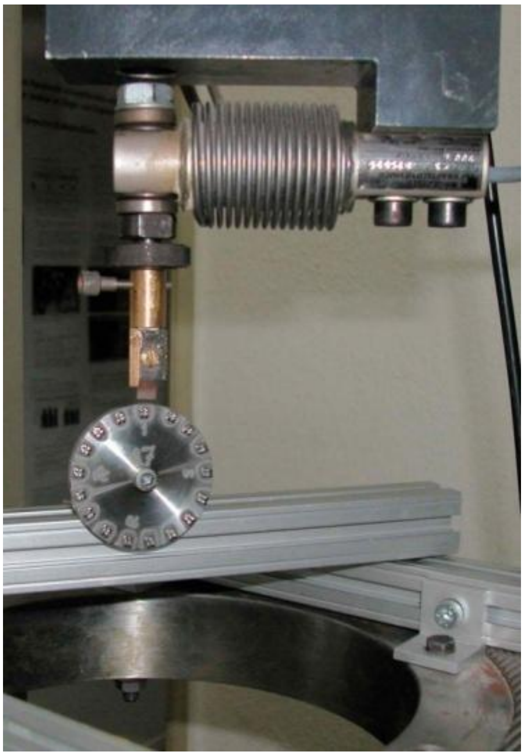

Figure 16. The disc with the bonded brackets fixed in the testing machine under the cross head.

Figure 15. Close up of the cross head and the tested specimen.

The head was adjusted to apply the force near the base of the bracket, i.e. the distance between the force vector and the centre of resistance was kept as small as possible (Figure 17). This was done in order to avoid great rotational moments, which would lead to a more tensile fracture mode.

The brackets of the second disc had to undergo 1000 cycles of shear sawtooth loading with the minimum and maximum stresses both above zero. The tests were performed according to the staircase method (Dixon and Mood, 1948; Draughn, 1979). The fatigue testing started at a load near the expected fatigue limit, which was estimated at approximately 60 % magnitude relative to the ultimate shear strength as determined

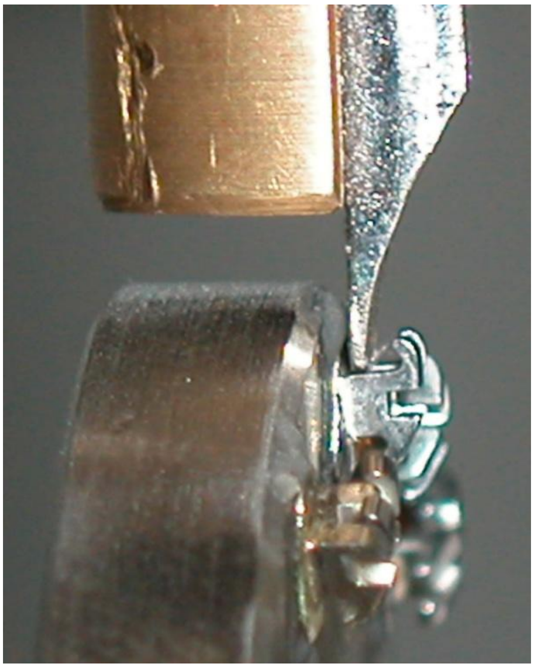

Figure 17. Close up profile of the cross head applying a force near the base of the bracket.

from the first disc, in accordance to previous work (Draughn, 1979). The fatigue testing was carried out at a mean frequency of 0.13 Hz, i.e. 8.06 cycles per minute. This relative small frequency was used because of limitations in the set up of the testing machine. The cross head speed was adjusted at 5 mm / min.

In the case of the specimen not failing within the prescribed number of 1000 stress cycles, the stress for the second specimen was increased by a fixed increment of approximately 5 % of the expected fatigue limit, which was expected to lie between 0.5 and 2 σ. If failure occurred, the stress for the next specimen was decreased. The procedure of increasing the maximum stress by 5 % following a test in which no failure occurred and decreasing the stress by the same increment following a failure was continued for each succeeding specimen through the whole disc.

The survived fatigued specimens of the second disc were subjected to shear strength testing at a cross head speed of 1 mm / min. Comparisons between the values taken for fatigued and nonfatigued specimens were made to extrapolate the effect of fatigue on the shear bond strength.

5.4 Data analysis

5.4.1 Shear strength

The information obtained from the experiment was saved in files on the controlling computer connected to the testing machine. The data were imported into the software Microsoft Excel 2007 (Microsoft Corporation), which was used for graphically presenting the staircase testing and calculating the fatigue limit.

The shear fatigue limit and the standard deviation were calculated according to a statistical method proposed previously (Dixon and Mood, 1948; Draughn, 1979). The mean fatigue limit is given by Equation 1 and its standard deviation by Equation 2.

The shear strength data of all specimens were imported into the software SPSS 16 (SPSS Inc.) for calculating the mean values and performing the statistical analysis.

$$\bar{X} = X_0 + d\,(\frac{A}{N} \pm \frac{1}{2}) \qquad (1)$$

Equation 1. Calculation of the mean fatigue limit $\bar{X}$:
The analysis of the data is based on the least frequent event (failures or nonfailures). The lowest level at which a failure or nonfailure occurs is denoted by $i = 0$, the next $i = 1$, etc. The positive sign is used when the analysis is based on nonfailures and the negative sign when failures are considered.
X_o is the lowest level on which the least frequent event occurs.
d is the increment employed in the sequential tests.
The other constants are defined by: $N = \sum n_i$, $A = \sum i n_i$, $B = \sum i^2 n_i$

$$S = 1.62\,d\left(\frac{NB-A^2}{N^2} + 0.029\right) \qquad (2)$$

Equation 2. Calculation of the standard deviation of the fatigue limit. The formula is an approximation, but is quite accurate when $\left(\frac{NB-A^2}{N^2}\right)$ is larger than 0.3. When the value of $\left(\frac{NB-A^2}{N^2}\right)$ is less than 0.3, more elaborate calculations must be employed (Dixon and Mood, 1948).

5.4.2 Fractography

All the bracket bases were prepared for examination with a scanning electron microscope (SEM, XL 30 W/TMP, Philips Electron Optics). They were cleaned with alcohol, dried and glued on a small flat round stub, which was necessary for mounting them in the SEM. On the stab they were goldcoated with the sputter coater Scancoat Six SEM Sputter Coater (Edwards) to make the surface conductive and ready for visualising.
The bracket bases were examined and photographed under 25x magnification with the scanning electron microscope. The photographs were imported into a CAD/CAM software (MegaCad 4.8b, Megatech Software GmbH) and scaled individually both vertically and horizontally to match the actual dimensions of the brackets. This step was necessary; because of projection errors that occurred by placing the specimens on the SEM stub, i.e. the examined bases were not perpendicular to the electron beam.
After scaling, the surface of the brackets was analysed with the software according to the type of fracture. Three types of fractured surfaces were distinguished depending on

whether the resin remained entirely on the bracket base or not (adhesive fracture) or the fracture line was in the resin, leaving portions of adhesive on the bracket base and the metal disc (cohesive fracture) (Figures 18, 19 and 20). The ratios of the surface fracture mode were calculated and compared between all tested specimens, including the ones that failed during the staircase method.

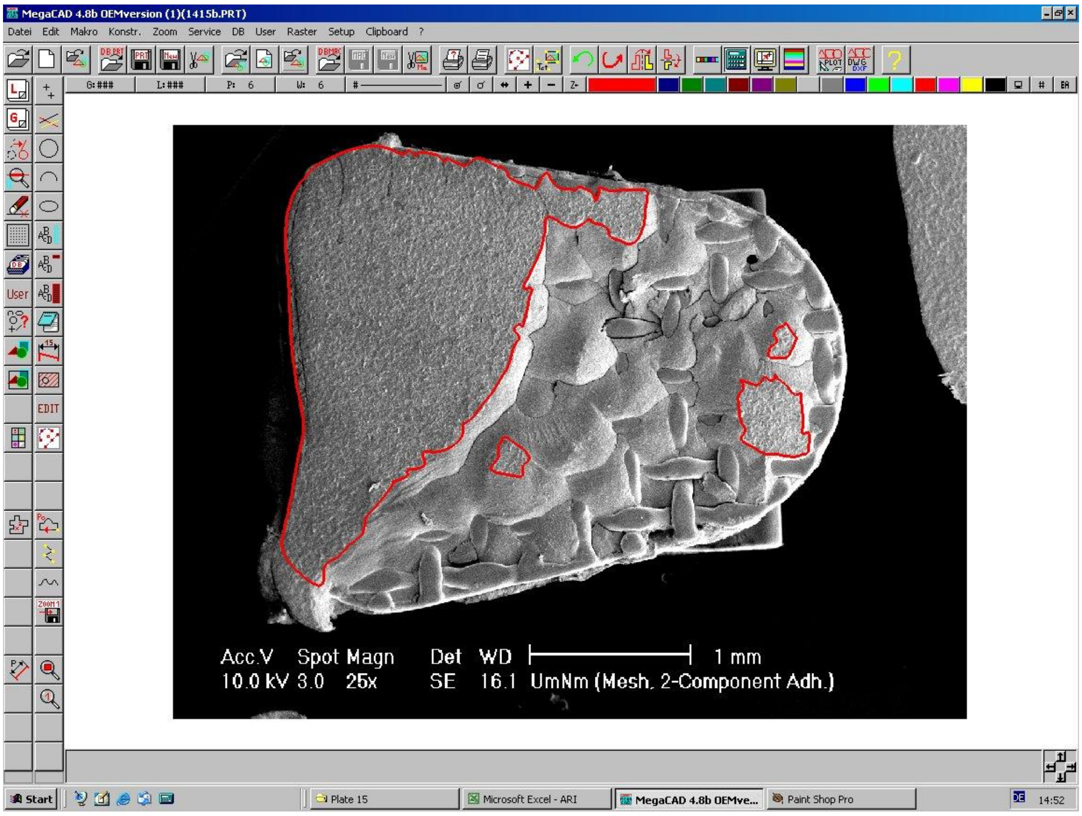

Figure 18. Surface measurement of fracture mode with MegaCad. Setting of the limits of the surface, where the entire adhesive was left on the bracket (adhesive fracture mode between adhesive and disc).

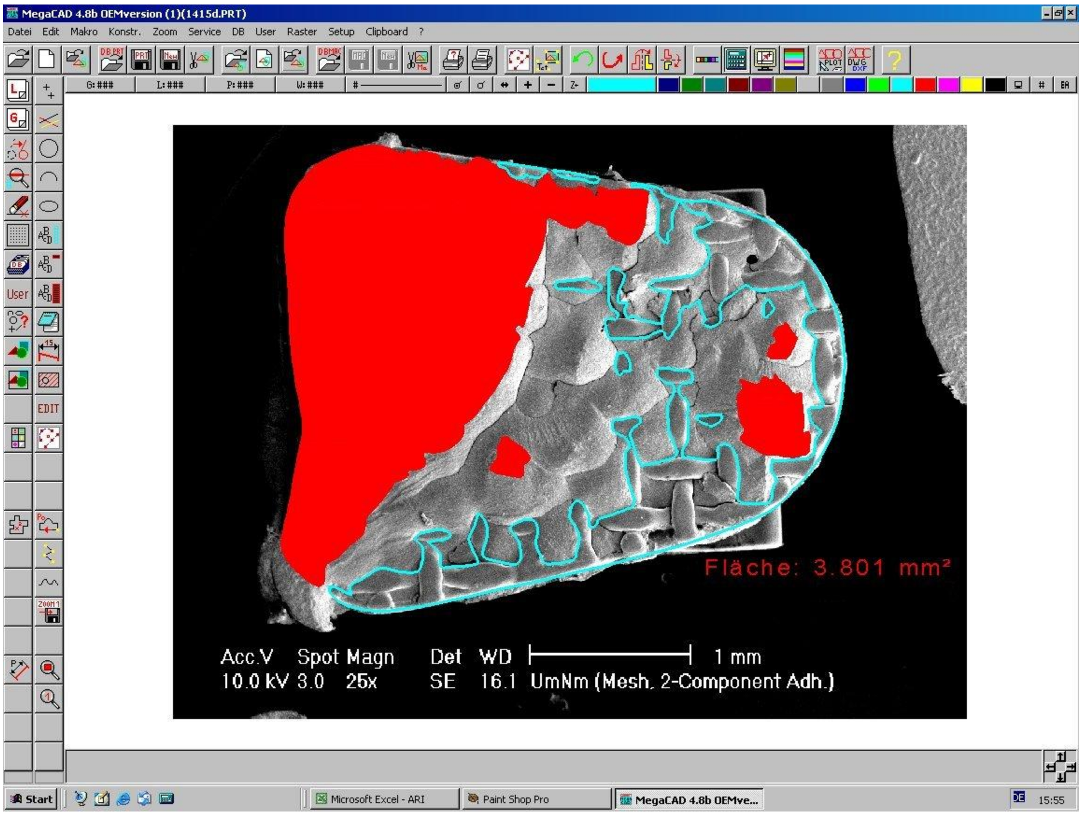

Figure 19. Setting the limits of the surface, where no adhesive is left (adhesive fracture mode between bracket and adhesive).

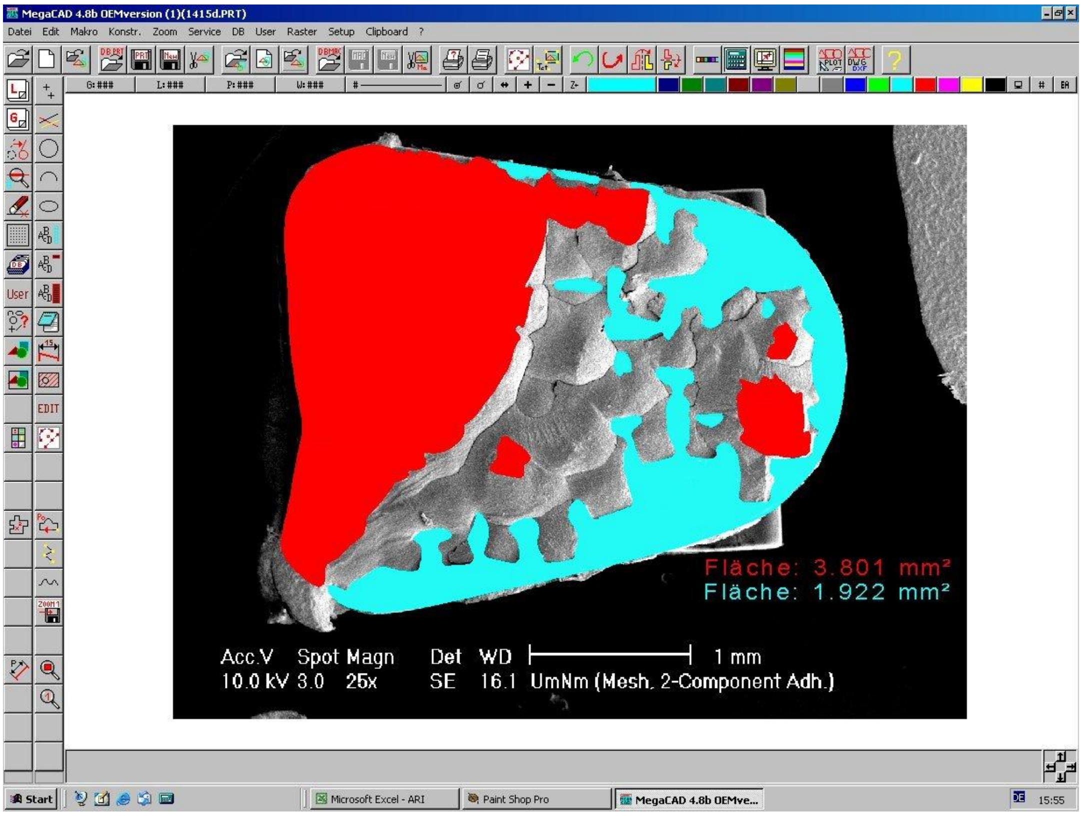

Figure 20. The area on the right represents an adhesive fracture between bracket and adhesive (no adhesive remained on the bracket), the area in the middle represents a cohesive fracture and the area on the left represents an adhesive fracture between adhesive and disc (all adhesive remained on the bracket).

5.4.3 Statistics

Boxplots

The strength and the fractography data were presented as boxplots.

In descriptive statistics, a boxplot (also known as a box-and-whisker diagram) is a convenient way of graphically depicting groups of numerical data through their five-number summaries (the smallest observation, lower quartile (Q1), median, upper quartile (Q3), and largest observation). A boxplot may also indicate which observations, if any, might be considered outliers. The boxplot was invented in 1977 by the American statistician *John Tukey* (Tukey, 1977).

A quartile is any of the three values which divide the sorted data set into four equal parts, so that each part represents 1/4th of the sampled population. The first quartile (Q1) cuts off lowest 25 % of data and indicates the lowest border of the box. The second quartile (Q2) is the median and cuts the data set in half. The third quartile (Q3) cuts off highest 25 % of data and is the upper border of the box. The median lies inside of the box with the presence of a line dividing the box at the median value. The interquartile range (IQR) is obtained by subtracting the first quartile from the third quartile.

Any data observation which lies more than 1.5*IQR lower than the first quartile or 1.5*IQR higher than the third quartile is considered an outlier. The smallest and largest value that is not an outlier is indicated by connecting it to the box with a line or "whisker" and marked clearly using a small line perpendicular to the whisker.

Outliers are marked by open and closed dots. "Extreme" outliers, or those which lay more than three times the IQR lower or higher from the first and third quartiles respectively, are indicated by the presence of an open dot. "Mild" outliers - that is, those observations which lay more than 1.5 times the IQR from the first and third quartile but are not also extreme outliers are indicated by the presence of a closed dot.

Analysis of variance (ANOVA)

In this study a multivariate 3-way analysis of variance (ANOVA) was used for the statistical evaluation of the strength data and the fractographic data. Three factors were defined as “bracket”, “adhesive” and “fatigue”. For the strength data, the dependant variable was the “shear strength”. For the fractographic data, the dependant variable was defined as “favourable fracture” or “cohesive fracture”. The possible values of the factors are presented in Tables 3 and 4. The only difference between the factor values for the strength data and fractographic data evaluation was the value 2 for “fatigue” which was not used for the strength data. The value stands for the specimens, which failed during fatigue. Its influence on shear strength was not investigated, since the strength of the failed-during fatigue specimens was not representative.

Factor	Value	Interpretation
bracket	1	Discovery®
	2	Ultra-Minitrim®
adhesive	1	No-Mix
	2	Concise™
fatigue	0	no fatigue
	1	fatigue

Table 3. ANOVA: Between-Subjects factors for the strength data analysis

Factor	Value	Interpretation
bracket	1	Discovery®
	2	Ultra-Minitrim®
adhesive	1	No-Mix
	2	Concise™
fatigue	0	no fatigue
	1	fatigue
	2	failure during fatigue

Table 4. ANOVA: Between-Subjects factors for the fractographic analysis

Analysis of variance (ANOVA) is used to uncover the main and interaction effects of categorical independent variables (called "factors") on an interval dependent variable. The key statistic in ANOVA is the F-test of difference of group means, testing if the means of the groups formed by values of the independent variable (or combinations of values for multiple independent variables) are different enough not to have occurred by chance. If the group means do not differ significantly then it is inferred that the independent variable(s) did not have an effect on the dependent variable. If the F test shows that overall the independent variable(s) is (are) related to the dependent variable, then multiple comparison tests of significance are used to explore just which values of the independent(s) have the highest impact on the relationship.

Analysis of variance tests the null hypotheses, i.e. that group means do not differ. It is not a test of differences in variances, but rather assumes relative homogeneity of variances. Thus some key ANOVA assumptions are that the groups formed by the independent variable(s) are relatively equal in size and have similar variances on the dependent variable ("homogeneity of variances"). Like regression, ANOVA is a parametric procedure which assumes multivariate normality (the dependent has a normal distribution for each value category of the independent(s)).

Main effects are the unique effects of the categorical independent variables. If the probability of F is less than 0.05 for any independent, it is concluded that the variable does have an effect on the dependent.

Interaction effects are the joint effects of pairs, triplets, or higher-order combinations of the independent variables, different from what would be predicted from any of the independents acting alone. That is, when there is interaction, the effect of an independent on a dependent varies according to the values of another independent. If the probability of F is less than 0.05 for any such combination, we conclude that that interaction of the combination does have an effect on the dependent. The concept of interaction between two independents is not related to the issue of whether the two variables are correlated (Garson, 2008).

There were three independent variables (factors) in this study. Three factors have three main effects and four interaction effects, i.e. three first order interactions (A*B, A*C, B*C) and one second-order interaction (A*B*C). The presence of interactions means, that the influence of one factor depends on another factor, i.e. that the factor influence is not the same for all tested specimens.

In such a case it is wise to test subgroups separately by reducing the possible values to one for all factors except one. If the values for the remaining factor were more than two

(e.g. the factor fatigue with three possible values in the fractographic analysis), then a one-way-ANOVA was performed. In the post hoc analysis the Student-Newman-Keuls test was used after each one-way ANOVA to find the groups of fatigue test mode which differed from each other significantly for each material combination separately. If the tested values for a factor were only two, then the groups were compared with a t-test for statistically significant differences.
The multivariate ANOVA provided therefore the justification for testing the subgroups with the one-way-ANOVA or the t-test.

t-test

The groups were tested for differences with the t-test. A t-test is a statistical hypothesis test in which the test statistic has a Student's t-distribution if the null hypothesis is true. It is used for calculating the statistical significance of the difference between two sample means.
The Student's t-distribution (or also t-distribution) is a probability distribution that arises in the problem of estimating the mean of a normally distributed population when the sample size is small. The derivation of the t-distribution was first published in 1908 by *William Sealy Gosset*, while he worked at a Guinness Brewery in Dublin. He was prohibited from publishing under his own name, so the paper was written under the pseudonym Student. The t-test and the associated theory became well-known through the work of the English geneticist and statistician Ronald Fisher, who called the distribution "Student's distribution".
A null hypothesis is a hypothesis set up to be nullified or refuted in order to support an alternate hypothesis. It is the assumption that no difference exists between the two groups for the variable being compared.
The t-test gives as a result the probability value p, which is defined as the probability of obtaining a result equal to or more extreme than that observed by chance alone, if the null hypothesis is true. In other words, it gives the probability that the results show differences between the compared groups, assuming that the groups are not really different from each other, i.e. the probability that the differences occur by chance.
If the probability value p is lower than a given confidence or significance level α, which is usually set at 0.05, there is less than 5 % probability that the difference occurs by chance. The null hypothesis is rejected and the difference is defined as statistically significant.

6 Results

6.1 Shear strength

The values of the shear strength of all material combinations in relation to the fatigue are presented in Table 5. The first line for each material combination (fatigue = 0) represents the shear strength data obtained from the control group with the nonfatigued specimens. The second line (fatigue = 1) represents the shear strength data which were obtained in shear strength experiments with the fatigued specimens.

The data show an increase of the shear strength after cyclic fatigue in group A (shear strength before fatigue 272 N and after fatigue 293 N) and a decrease in all other groups B (170 N / 166 N), C (450 N / 435 N) and D (283 N / 254 N). The values are also presented as stress [MPa] for better comparability to other studies. The stress values were calculated by dividing the mean shear strength by the bracket surface as given by the manufacturer (Discovery® 8.508mm^2, Ultra-Minitrim® 9.77mm^2).

Groups A / C and B / D consist of the same bracket and A / B and C / D of the same adhesive. Therefore the data show that the groups A / C incorporate the bracket and groups C / D the adhesive which show higher shear strength. These are the bracket Discovery® and the adhesive Concise™.

Material Combination	Fatigue	N	Minimum [N]	Maximum [N]	Range [N]	Mean [N]	Std. Error of Mean [N]	Median [N]	Std. Deviation [N]	Mean [MPa]
A	0	13	247	292	45	272	5	284	18	31.9
	1	8	272	323	51	293	7	299	19	34.5
B	0	15	113	244	131	170	9	168	33	17.4
	1	9	140	185	45	166	6	171	17	17.0
C	0	15	401	494	93	450	7	448	28	52.8
	1	8	367	504	136	435	16	449	46	51.1
D	0	14	244	317	73	283	6	284	22	29.0
	1	9	226	296	70	254	7	245	22	26.0

Table 5. The values of the shear strength [N] and stress [MPa] in relation to the material combinations A-D (A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™) and the fatigue (0 = nonfatigued, 1 = fatigued specimens).

6.1.1 Staircase method

The fatigued specimens underwent cyclic load according to the staircase method. An example plot of the up and down cyclic fatigue of group A is presented in Figure 21. The first specimen was tested at 171 N for 1000 cycles and survived. The load was increased and the second specimen failed at 176 N after 20 cycles (see Figure 22). The load for the next specimen was thus decreased and a failure occurred after 571 cycles (see Figure 22). The load for each test was decreased or increased depending on whether the previous test was terminated with failure or not. The survived specimens represent group A_1 in Table 5.

Figures 22, 23, 24 and 25 show the number of the cycles at which the specimens failed during fatigue in groups A, B, C and D respectively. In material group A the specimens failed either at a very low (relative quickly) or a relative high cycle number. In material group B all the specimens failed at a relative low cycle number. For material groups C and D no clear distribution was found. The bracket Discovery® was capable of surviving more cycles than Ultra-Minitrim®, although it tended to show also more quick failures.

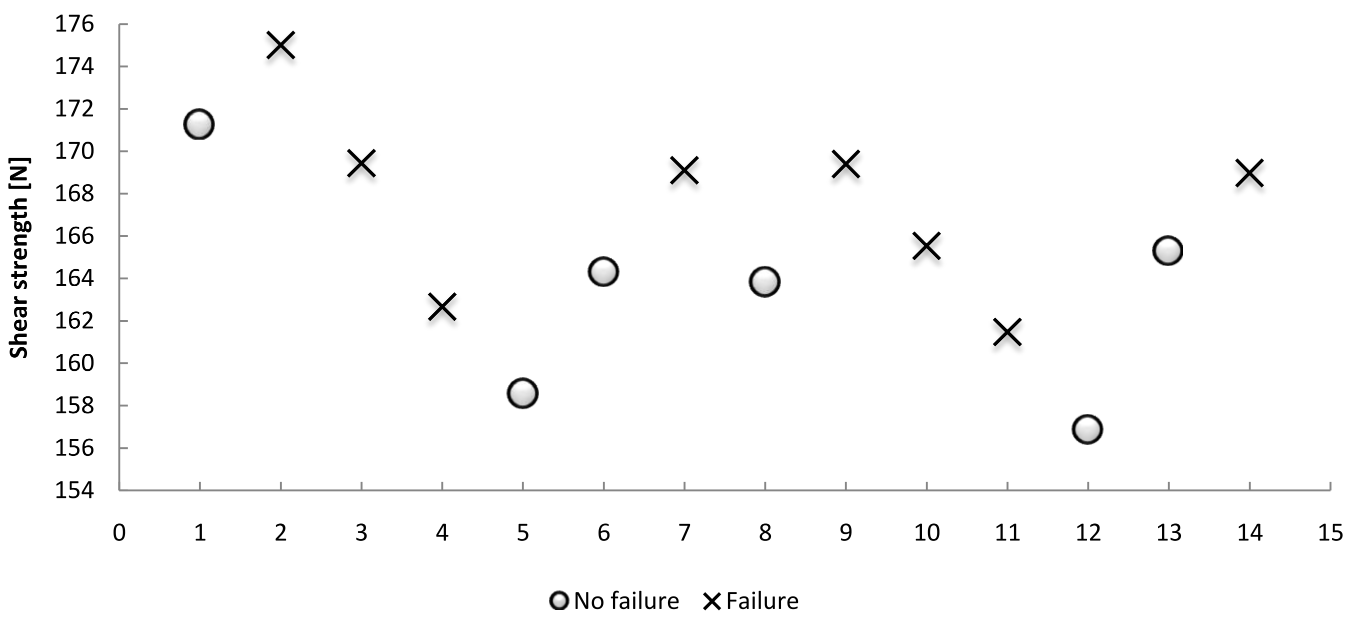

Figure 21. The cyclic fatigue according to the staircase method in material group A. The circles stand for the survived and the x’s for the fractured specimens under fatigue loading of 1000 cycles.

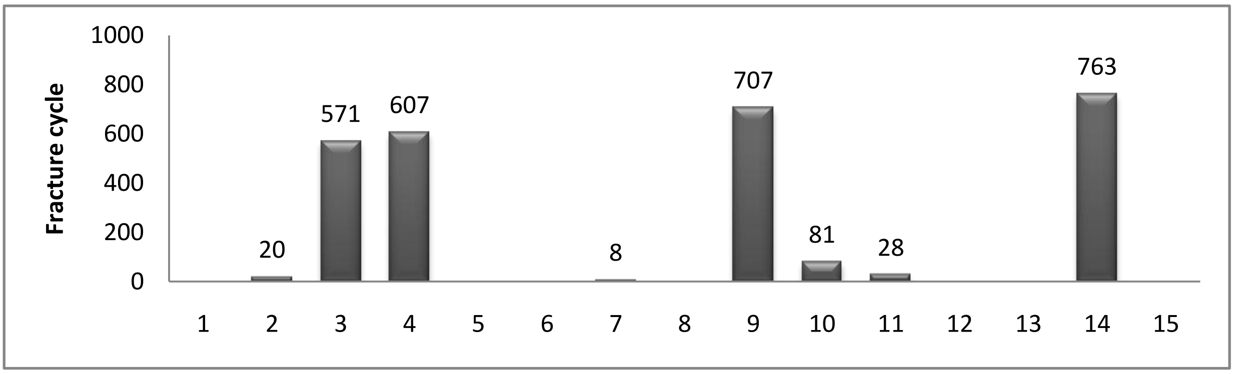

Figure 22. The numbers of fracture cycles of the fatigued specimens in group A (bracket Discovery® / adhesive No-Mix). Missing bars indicate no fracture.

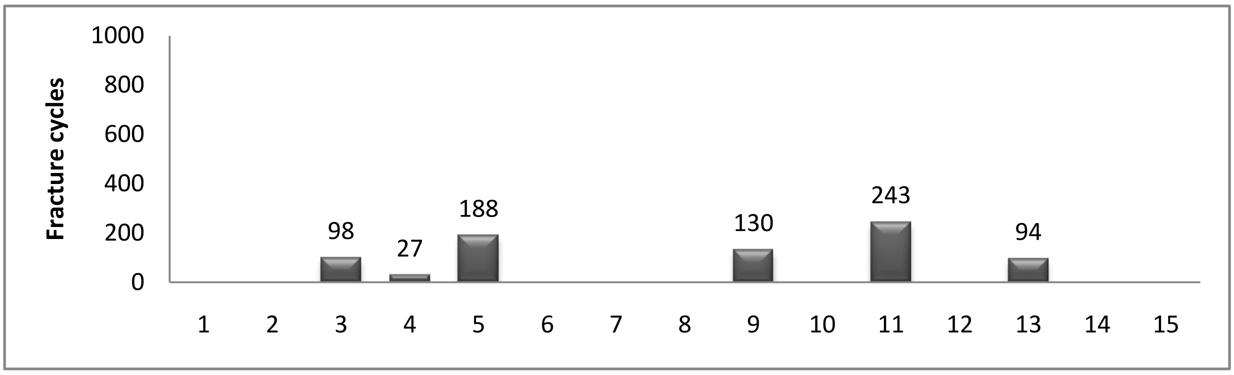

Figure 23. The numbers of fracture cycles of the fatigued specimens in group B (bracket Ultra-Minitrim® / adhesive No-Mix). Missing bars indicate no fracture.

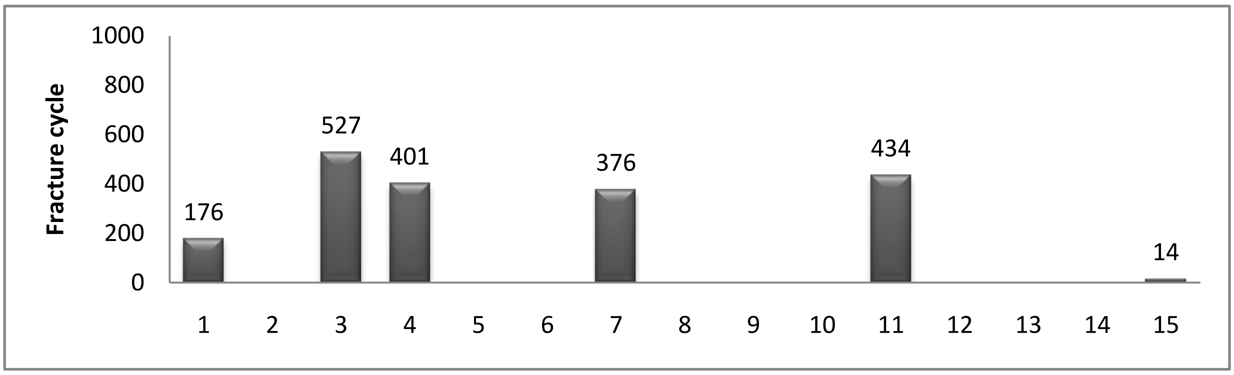

Figure 24. The numbers of fracture cycles of the fatigued specimens in group C (bracket Discovery® / adhesive Concise™). Missing bars indicate no fracture.

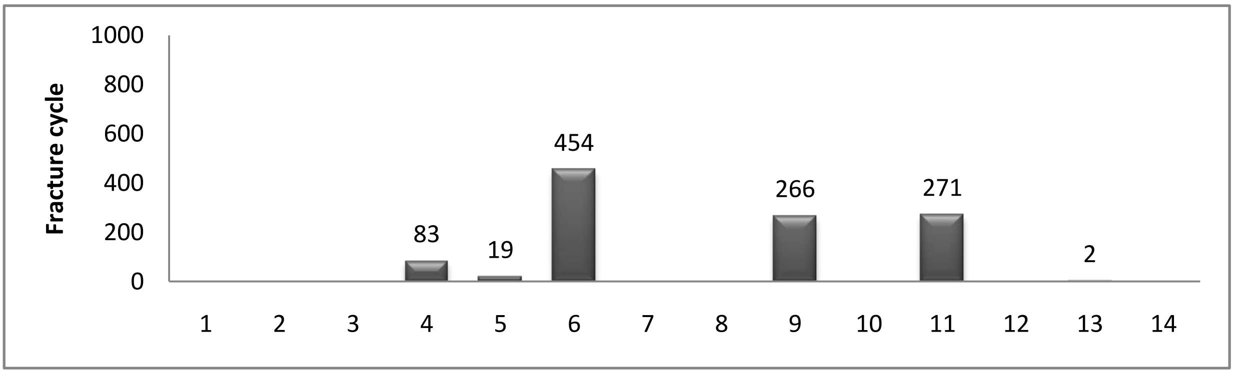

Figure 25. The numbers of fracture cycles of the fatigued specimens in group D (bracket Ultra-Minitrim® / adhesive Concise™). Missing bars indicate no fracture.

6.1.2 Statistical analysis

The test of between-subjects effects in the 3-way analysis of variance (ANOVA, Table 6) revealed two significant main effects (bracket, adhesive) and two first order interactions (bracket * adhesive, adhesive * fatigue). This means that the bracket type has a significant effect on shear strength and that its influence depends on the adhesive type. The adhesive type has a significant effect, which depends on the bracket type and on the occurrence of fatigue. Fatigue has no significant main effect, but the incidence of significant interactions with the adhesive means, that there could be some combination effect.

Dependent variable: Shear strength [N]

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	9.204E5	7	131481.054	182.426	.000
Intercept	7.152E6	1	7.152E6	9923.057	.000
Bracket	440352.609	1	440352.609	610.978	.000
Adhesive	358620.697	1	358620.697	497.577	.000
Fatigue	927.791	1	927.791	1.287	.260
Bracket * Adhesive	18851.833	1	18851.833	26.156	.000
Bracket * Fatigue	2167.974	1	2167.974	3.008	.087
Adhesive * Fatigue	5118.112	1	5118.112	7.101	.009
Bracket * Adhesive * Fatigue	155.108	1	155.108	.215	.644
Error	59820.964	83	720.735		
Total	8.633E6	91			
Corrected Total	980188.345	90			

a. R Squared = .939 (Adjusted R Squared = .934)

Table 6. ANOVA: Tests of between-subjects effects for the strength data analysis

The interactions of all three factors with each other make clear, that there are synergies and neither of them can be evaluated independently. The influence of the factors is not the same in all conditions and therefore cannot be described sufficiently. The factors depend on each other, so the effects should be evaluated for a combination of factors, i.e. for subgroups separately. The results were confirmed by another Analysis of Variance without consideration of the second order interaction (bracket * adhesive * fatigue), which is a useful testing since the second order interaction showed no significant effect in the first analysis.

The subgroups were defined by taking out the possible values for the factors bracket and adhesive and comparing the effect of fatigue for one material combination at a time. The effect of the bracket and the adhesive on the shear strength was also evaluated. Since the comparison was performed between two groups at a time, a t-test was performed.

Effect of the fatigue

The results of the t-tests for the influence of fatigue on the shear strength in the different material groups are presented in Table 7. The table shows the possibility that the differences between the shear bond strength of the nonfatigued and the fatigued specimens in each material group occur by chance. The confidence level was set at 0.05.
Cycling fatigue showed a significant difference in group A and D and no significant effect in group B and C. The shear strength data are graphically presented in Figure 26 and the significance is indicated between the boxplots with an asterisk. In group A the fatigued specimens showed increased shear bond strength of 8 % compared to the non-fatigued. In group D there was a decrease in shear bond strength after fatigue of 10 %.

Material Combination	**A**	**B**	**C**	**D**
p	0.021	0.709	0.428	0.006
Result	Null hypothesis rejected (significant)	Null hypothesis confirmed (non significant)	Null hypothesis confirmed (non significant)	Null hypothesis rejected (significant)

Table 7. t-test results of the effect of fatigue using different material combinations. (α=0.05). A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

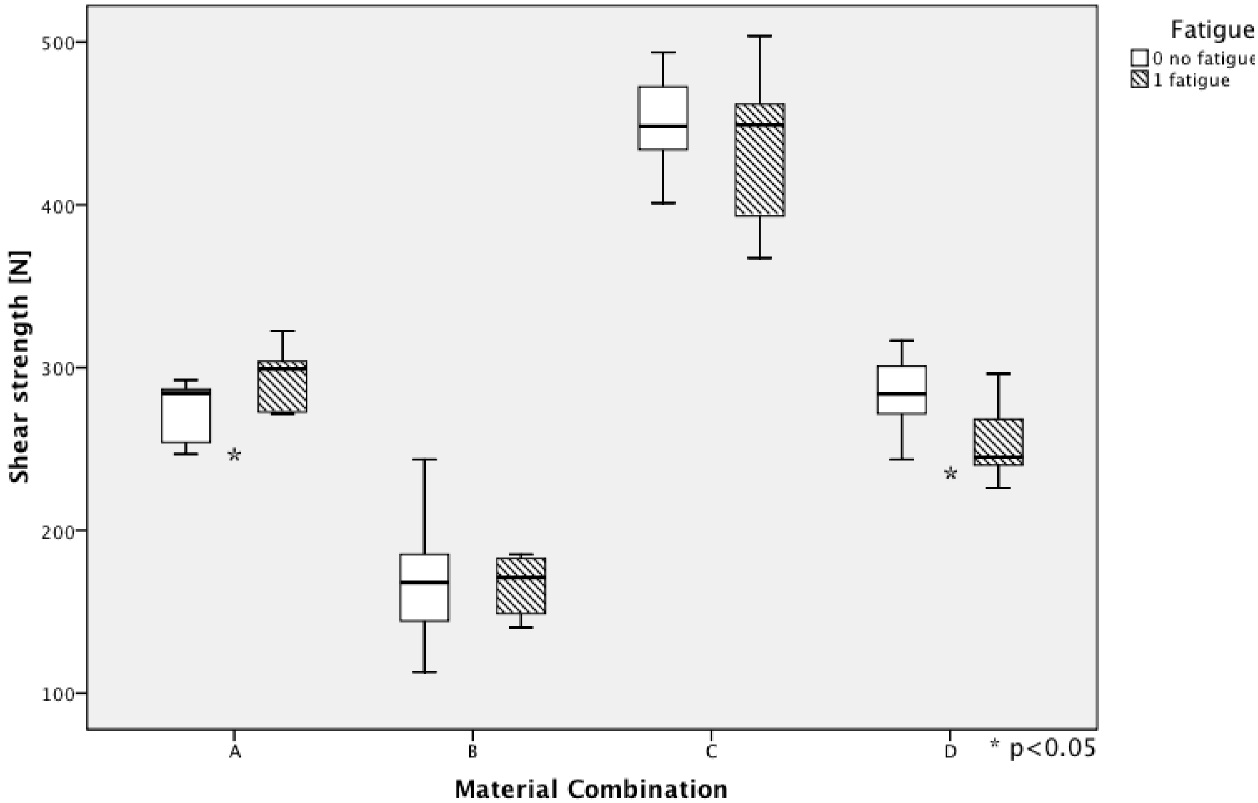

Figure 26. Ultimate shear strength and shear strength after fatigue for each material combination. The asterisk between two groups indicates a statistically significant difference. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

Effect of the bracket type

The difference in shear strength between the two bracket types was tested in the two nonfatigued groups with different adhesives, that is group A_0 with B_0 and C_0 with D_0 (see Table 5, page 60). Two comparisons were made, that is one for each adhesive. The results of the t-tests for the influence of the bracket type on ultimate shear strength depending on the adhesive are presented in Table 8. Figure 27 shows the results graphically as boxplots and the significant differences are marked with an asterisk.

Adhesive type	1	2
p	0.000	0.000
Result	Null hypothesis rejected (significant)	Null hypothesis rejected (significant)

Table 8. t-test results of the effect of the bracket type using different adhesives. α =0.05.
1 = adhesive No-Mix, 2 = adhesive Concise™.

The bracket Discovery® showed with both adhesives statistically significant higher bond strength than the bracket Ultra-Minitrim®. With No-Mix, Discovery® showed 60 % and with Concise™ 59 % higher shear strength respectively (see Table 5, page 60 and Figure 27). Although the ANOVA showed, that the factors bracket and adhesive depended on each other, i.e. their influence was not always the same, the results showed, that the bracket type had nearly the same influence when used with any of the adhesives.

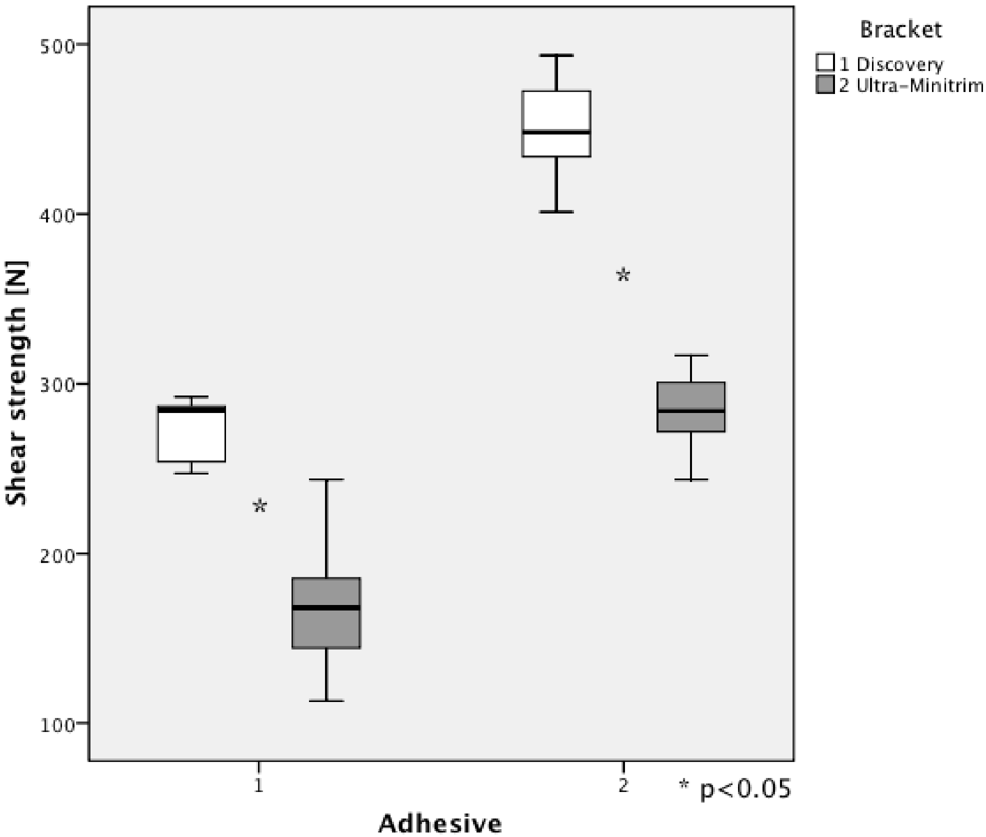

Figure 27. The ultimate shear strength of the two brackets for each adhesive. The asterisk between two groups indicates a statistically significant difference. 1 = adhesive No-Mix, 2 = adhesive Concise™.

Effect of the adhesive type

The effect of the adhesive type on the ultimate shear strength was tested between the two nonfatigued groups with different brackets. Group A_0 was compared with C_0 and B_0 with D_0 (see Table 5, page 60). Two comparisons were made, that is for each bracket. The results of the t-tests are presented in Table 9. Figure 28 shows the results graphically and the statistically significant differences are marked with an asterisk.

Bracket type	**1**	**2**
p	0.000	0.000
Result	Null hypothesis rejected (significant)	Null hypothesis rejected (significant)

Table 9. t-test results of the effect of the adhesive type using different brackets. α=0.05. 1 = bracket Discovery®, 2 = bracket Ultra-Minitrim®.

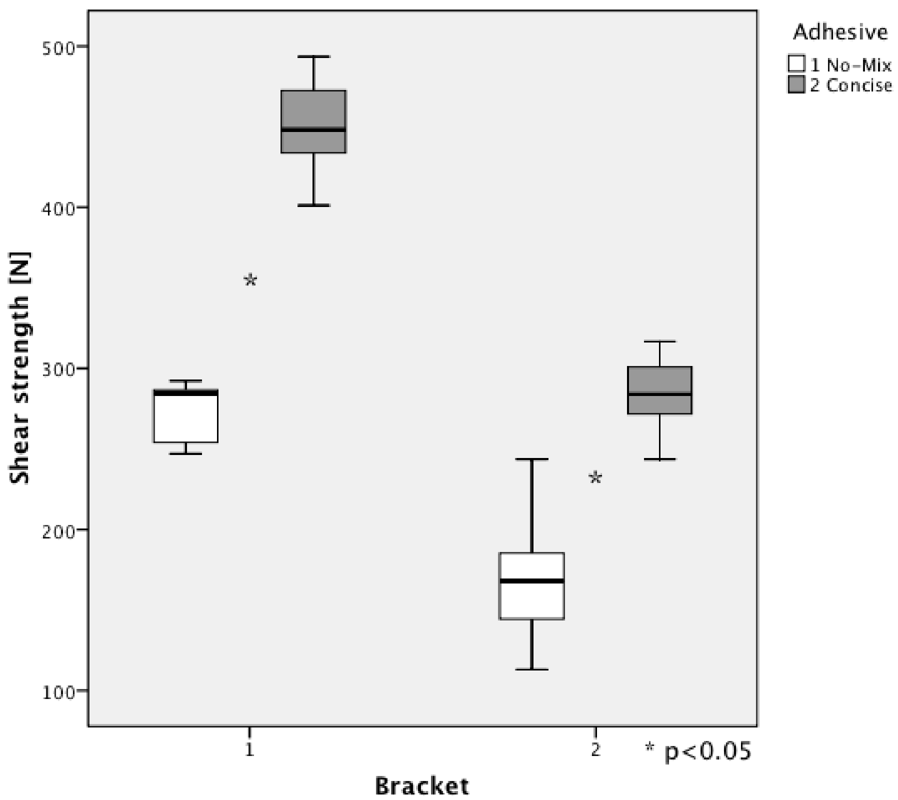

Figure 28. The ultimate shear strength of the two adhesives for each bracket. The asterisk between two groups indicates a statistically significant difference. 1 = bracket Discovery®, 2 = bracket Ultra-Minitrim®.

The adhesive Concise™ showed with both brackets statistically significant higher bond strength. With the bracket Discovery®, Concise™ showed 66% and with the bracket Ultra-Minitrim® 67 % higher shear strength respectively (see Table 5, page 60 and Figure 28, page 67). Although the ANOVA showed that the factors bracket and adhesive depend on each other, i.e. their influence is not always the same, the result show that the adhesive type has nearly the same influence when used with any of the brackets.

Fatigue limit

The shear fatigue limit and the standard deviation was calculated according to a statistical method proposed previously (Dixon and Mood, 1948; Draughn, 1979) solving Equations 1 and 2 respectively (page 53). The results are presented in Table 10.

Material Combination	A	B	C	D
Fatigue limit [N]	165	104	266	190
Standard deviation [N]	4	4	12	9

Table 10. The values of the fatigue limit and its standard deviation. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

Fatigue ratio

The shear fatigue ratio is the ratio of the shear fatigue limit and the ultimate shear strength of the nonfatigued specimens. The according data are presented in Table 11. The fatigue ratio was almost constant for material combinations A, B and C and was slightly increased for material combination D.

Material Combination	A	B	C	D
Fatigue limit [N]	165	104	266	190
Ultimate shear strength [N]	272	170	450	283
Fatigue ratio	0.61	0.61	0.59	0.67

Table 11. The fatigue ratio. Groups A, B and C showed an almost constant fatigue ratio. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

6.2 Fractography

6.2.1 Distribution of the fracture mode

The values of the fractography data are presented in Table 12 (page 70) as proportions of the bracket surface. It was distinguished whether the adhesive remained entirely on the disc (adhesive fracture between bracket and adhesive), on the bracket (adhesive fracture between adhesive and disc) or the adhesive fractured in the middle leaving portions on the bracket and the disc (cohesive fracture). The values are presented for all tested specimens including the ones that failed during the staircase method.

In order to gain a better overview of the complex distribution of the fracture modes, the mean values are also presented as columns in Figure 29.

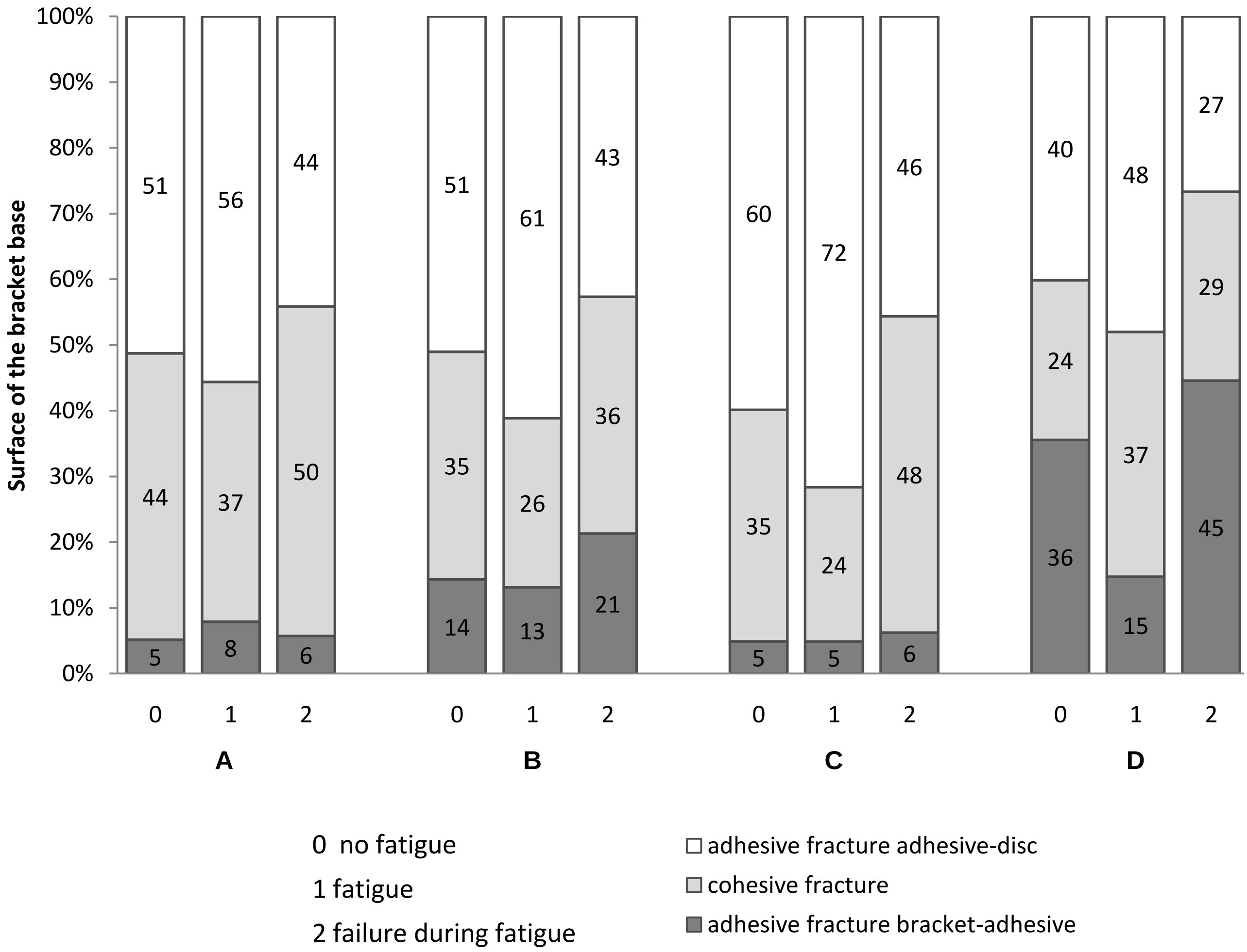

Figure 29. The distribution of the three different fracture modes for all the specimens. The numbers represent the mean values of the fractographic data. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

Material Combination	Fatigue		adhesive fracture [bracket-adhesive] [%]	cohesive fracture [%]	adhesive fracture [adhesive-disc] [%]
A	0	Mean	5	44	51
		Std. Deviation	2	14	15
	1	Mean	8	37	56
		Std. Deviation	9	20	26
	2	Mean	6	50	44
		Std. Deviation	1	20	20
B	0	Mean	14	35	51
		Std. Deviation	9	11	15
	1	Mean	13	26	61
		Std. Deviation	9	9	15
	2	Mean	21	36	43
		Std. Deviation	6	6	10
C	0	Mean	5	35	60
		Std. Deviation	2	15	16
	1	Mean	5	24	72
		Std. Deviation	1	12	12
	2	Mean	6	48	46
		Std. Deviation	2	22	24
D	0	Mean	36	24	40
		Std. Deviation	19	11	15
	1	Mean	15	37	48
		Std. Deviation	9	8	13
	2	Mean	45	29	27
		Std. Deviation	7	5	7
Total	0	Mean	15	34	51
		Std. Deviation	16	14	16
	1	Mean	11	31	58
		Std. Deviation	9	14	19
	2	Mean	19	41	40
		Std. Deviation	16	17	18

Table 12. The values of the fractographic data as percentage of the bracket surface. The fatigue group 2 stands for the specimens that failed during the staircase method. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™. 0 = no fatigue, 1 = fatigue, 2 = failure during fatigue.

In order to simplify the statistical analysis, the three fracture mode groups were combined in two. The favourable fracture mode was defined as the sum of the adhesive fracture between bracket and adhesive, and the cohesive fracture. The second group was defined as the most favourable mode, i.e. the cohesive fracture mode. This was done in accordance to the clinical situation, where a cohesive fracture is most desirable (less risk for enamel detachment, less adhesive to remove for the orthodontist) followed by the adhesive fracture between bracket and adhesive (less risk for enamel detachment). This way the outcome could distinguish, which factors led to a more desired fracture mode. The adhesive fracture between adhesive and disc incorporates the risk for enamel detachment and is not favourable. It can be evaluated by reverting the results for the favourable fracture.

6.2.2 Statistical analysis

Effect of fatigue

Favourable fracture mode

For the favourable fracture mode, the test of between-subjects effects in the 3-way-ANOVA (Table 13) revealed two significant main effects (bracket, fatigue) and one first order interaction (bracket * adhesive). This means that the bracket type had a significant effect on the percentage of the favourable fracture mode and that its influence depended on the adhesive type. The fatigue had also a significant effect, which was independent of

Dependent variable: favourable fracture [bracket-adhesive + cohesive] [%]

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	12202.992[a]	11	1109.363	4.242	.000
Intercept	278771.702	1	278771.702	1065.869	.000
Bracket	2627.804	1	2627.804	10.047	.002
Adhesive	146.943	1	146.943	.562	.455
Fatigue	5951.504	2	2975.752	11.378	.000
Bracket * Adhesive	3365.785	1	3365.785	12.869	.001
Bracket * Fatigue	6.295	2	3.147	.012	.988
Adhesive * Fatigue	317.784	2	158.892	.608	.547
Bracket * Adhesive * Fatigue	165.563	2	82.782	.317	.729
Error	27985.208	107	261.544		
Total	336243.432	119			
Corrected Total	40188.200	118			

a. R Squared = .304 (Adjusted R Squared = .232)

Table 13: ANOVA .Tests of between-subjects effects for the favourable fracture mode in the fractographic analysis.

other factors. This is an important finding, because it means, that fatigue plays the same role in all conditions and the influence doesn't get altered from the materials used. The adhesive had no significant main effect, but the incidence of a significant interaction with the bracket means that there could be some combined effect.

The ANOVA was repeated for the same subgroups as before, which were defined by eliminating the possible values for the factors bracket and adhesive, i.e. for one material combination at a time. The influencing factor "fatigue" had three possible values and therefore a one-way ANOVA was performed (Table 14). The analysis showed that fatigue had a significant influence on the distribution of the favourable fracture mode for material combinations B, C and D.

Material Combination	A	B	C	D
p	0.510	0.037	0.026	0.01
Result	Null hypothesis confirmed (non significant)	Null hypothesis rejected (significant)	Null hypothesis rejected (significant)	Null hypothesis rejected (significant)

Table 14. One-way ANOVA for evaluating the influence of fatigue on the favourable fracture percentage. α=0.05. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

In the post hoc analysis the Student-Newman-Keuls test was used after each ANOVA to reveal the homogeneous subsets for the tested factor fatigue. Table 15 shows the groups which differed from each other significantly for each material combination separately. In material groups B, C and D the fatigued specimens (1) showed a significantly different favourable fracture mode distribution than the ones that failed during fatigue (2). In group D there was also a difference between the specimens that failed during fatigue (2) and the nonfatigued ones (0).

Material Combination		A	B	C	D
Non homogeneous subsets of fatigue	0-1				
	1-2		*	*	*
	2-0				*

Table 15. Student-Newman-Keuls analysis. The asterisk marks the groups, which were found to differ significantly from each other. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™. 0 = no fatigue, 1 = fatigue, 2 = failure during fatigue.

In summary, the statistical analysis shows, that there is no significant difference of the percentage of favourable fracture mode (sum of adhesive fracture between bracket/adhesive and cohesive fracture) between the nonfatigued and the fatigued specimens in any material group. In material groups B, C and D there was a significantly greater area of favourable fracture mode on the specimens which failed during fatigue compared to the fatigued specimens which survived and were sheared after that. For material combination D there was also a significant difference between the nonfatigued and those that failed during fatigue.

The factor fatigue was found to play the same role for all conditions in the 3-way-ANOVA. This is graphically presented in Figure 30 as boxplots. The percentage is al-

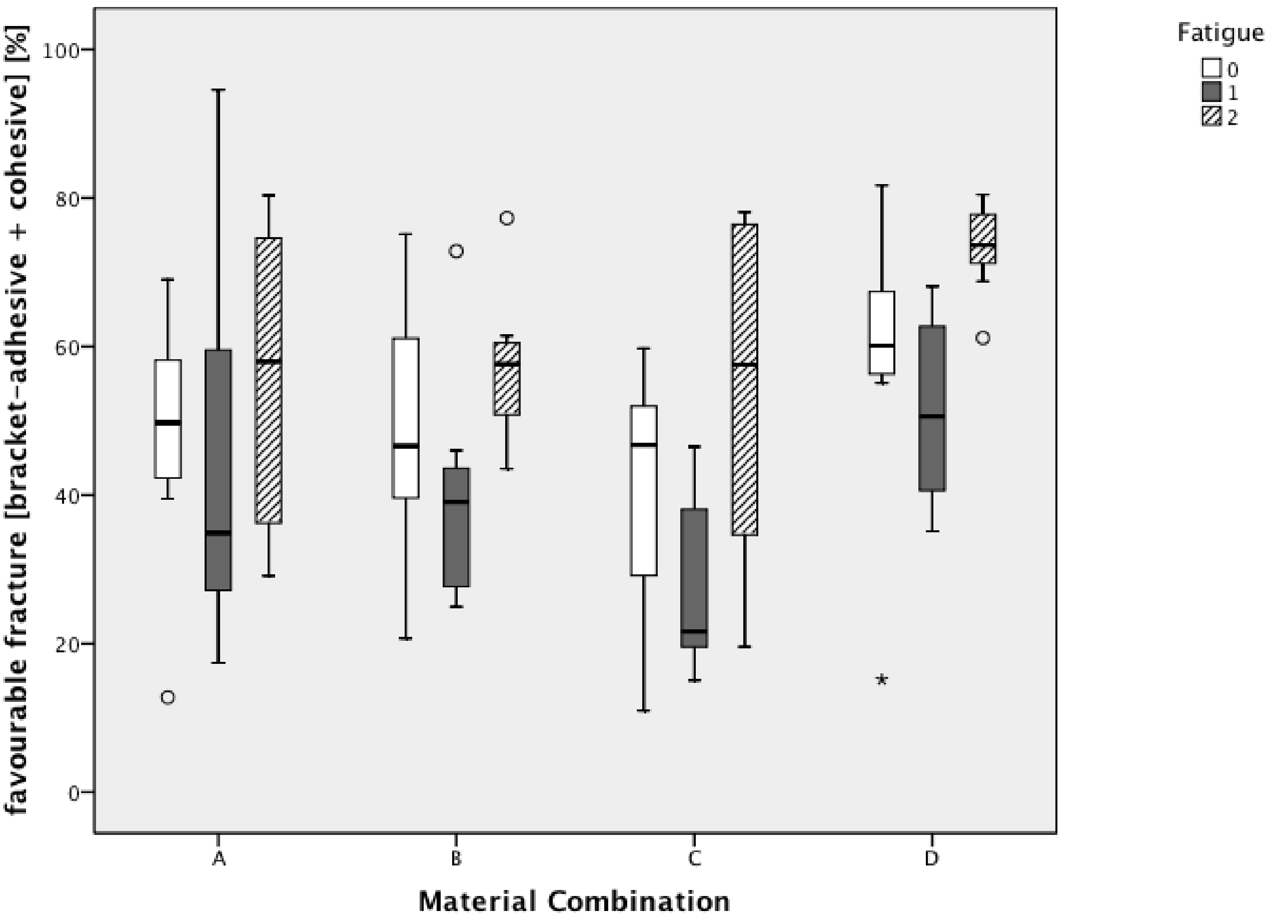

Figure 30. Boxplots of the percentage of the favourable fracture mode for each material combination. A statistically difference exists between groups B_1/B_2, C_1/C_2, D_1/D_2 and D_2/D_0. The “mild” outliers are presented by a circle and the “extreme” outliers by an asterisk. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™. 0 = no fatigue, 1 = fatigue, 2 = failure during fatigue.

ways lower for fatigue value 1 and higher for value 2. Although the fatigued specimens showed less and the failed-during-fatigue specimens a greater area of favourable fracture than the nonfatigued specimens, i.e. the fatigue mode had the same influence in all groups, the differences between the groups were not always significant. This can be due to the wide scatter of the results. The group D with the least scatter (the boxplots are smaller) showed the most significant differences.

Cohesive fracture mode

For the most favourable fracture mode, the cohesive fracture, the test of between-subjects effects in the 3-way ANOVA (Table 16) revealed two significant main effects (bracket, fatigue), one first order interaction (bracket * fatigue) and one second order interaction (bracket * adhesive * fatigue).

Dependent variable: cohesive fracture [%]

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	7763.073[a]	11	705.734	3.845	.000
Intercept	138034.739	1	138034.739	752.141	.000
Bracket	1957.577	1	1957.577	10.667	.001
Adhesive	672.779	1	672.779	3.666	.058
Fatigue	1635.590	2	817.795	4.456	.014
Bracket * Adhesive	230.276	1	230.276	1.255	.265
Bracket * Fatigue	1364.895	2	682.447	3.719	.027
Adhesive * Fatigue	395.905	2	197.953	1.079	.344
Bracket * Adhesive * Fatigue	1146.198	2	573.099	3.123	.048
Error	19636.909	107	183.523		
Total	173888.244	119			
Corrected Total	27399.982	118			

a. R Squared = .283 (Adjusted R Squared = .210)

Table 16. ANOVA .Tests of Between-Subjects Effects for the cohesive fracture mode in the fractographic analysis

This means that the factors bracket and fatigue had a significant effect on the percentage of cohesive fracture but this influence depended on each other and on the adhesive, since there were first and second order interactions. The influence of fatigue was not the same for all circumstances and had to be tested for all subgroups separately. The groups were defined as previously and a one-way ANOVA for each material group was performed.

Material Combination	A	B	C	D
p	0.32	0.052	0.025	0.007
Result	Null hypothesis confirmed (non significant)	Null hypothesis confirmed (non significant)	Null hypothesis rejected (significant)	Null hypothesis rejected (significant)

Table 17. One-way ANOVA for evaluating the influence of fatigue on the cohesive fracture percentage. α=0.05. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™.

The results (Table 17) show a significant influence of the factor fatigue on the cohesive fracture mode percentage only in groups C and D.
In the post hoc analysis the Student-Newman-Keuls test used after each one-way ANOVA revealed the groups of fatigue test mode which differed from each other significantly for each material combination separately (Table 18). In material groups C and D the fatigued specimens (1) showed a significantly different cohesive fracture mode distribution than the ones that failed during fatigue (2). In group D there was also a difference between the fatigued (1) and nonfatigued (0) specimens. The results are graphically presented in Figure 31 (page 76).

Material Combination		A	B	C	D
Non homogeneous subsets of fatigue	0-1				*
	1-2			*	*
	2-0				

Table 18. Student-Newman-Keuls analysis. The asterisk marks the groups which were found to differ significantly from each other. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™. 0 = no fatigue, 1 = fatigue, 2 = failure during fatigue.

In summary, the analysis showed that there was a statistically significant influence of the fatigue mode on the distribution of the cohesive fracture only in material groups C and D. In material group C the failed-during-fatigue specimens showed a significantly higher – and in group D lower – cohesive fracture distribution than the specimens which survived fatigue and were sheared after that. In group D the fatigued specimens showed a higher cohesive mode than the nonfatigued specimens.

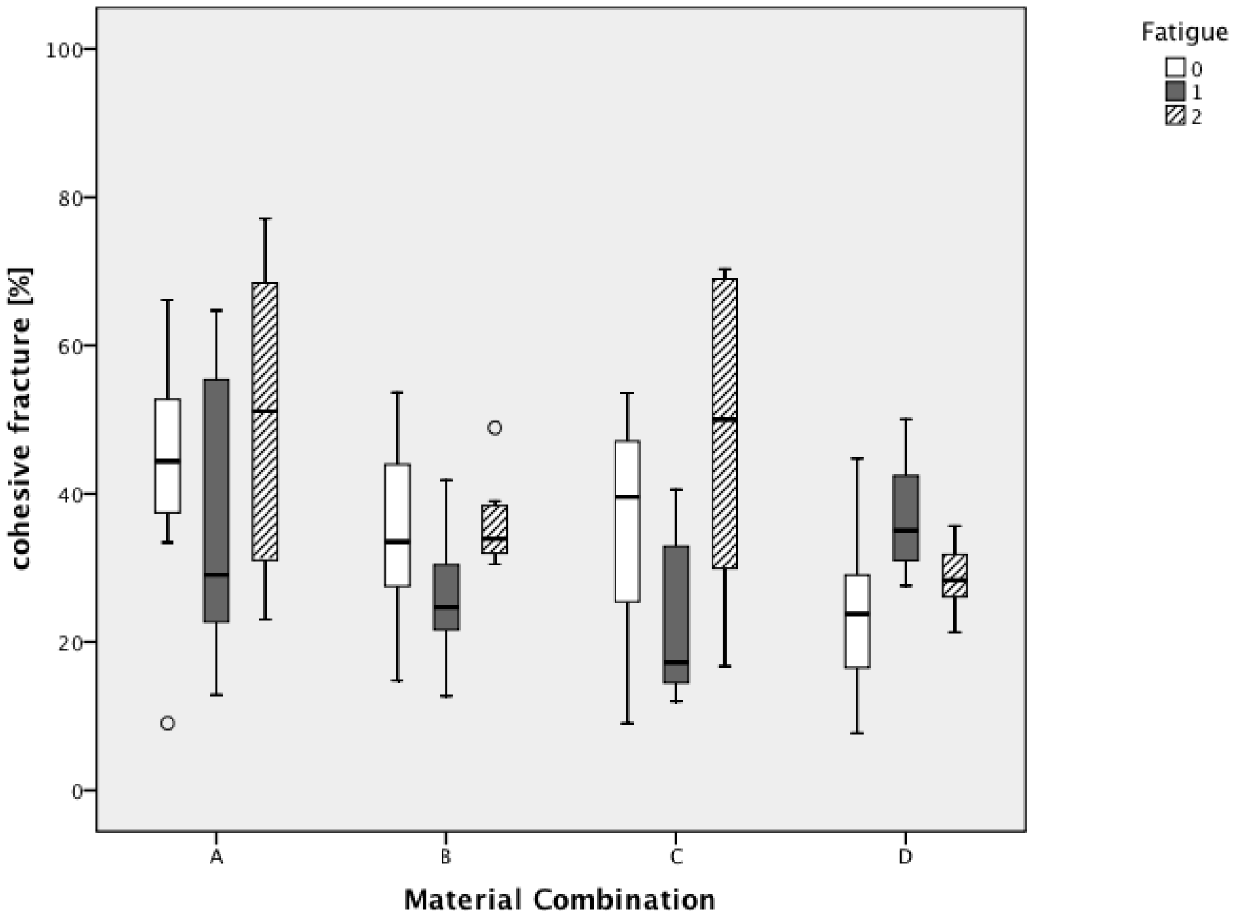

Figure 31. Boxplots of the percentage of the cohesive fracture mode in each material combination. Statistically significant differences were found between groups C_1/C_2, D_0/D_1 and D_1/D_2. The "mild" outliers are presented by a circle. A = bracket Discovery® / adhesive No-Mix, B = bracket Ultra-Minitrim® / adhesive No-Mix, C = bracket Discovery® / adhesive Concise™, D = bracket Ultra-Minitrim® / adhesive Concise™. 0 = no fatigue, 1 = fatigue, 2 = failure during fatigue.

Effect of the bracket type

The influence of the bracket type on the distribution of residual resin on the bases was examined separately for the nonfatigued specimens and depending on the adhesive used. Two groups were compared at a time and the t-test was used. The comparisons were made between groups A_0 / B_0 and C_0 / D_0.

Favourable fracture mode

The comparison of the specimens of one bracket type with the other one in the same adhesive group showed a significant difference only for adhesive 2. When using adhesive 2 (Concise™), the bracket 2 (Ultra-Minitrim®) showed a higher favourable fracture mode. When using adhesive 1 (No-Mix) no significant difference was found (Table 19). The results are graphically presented in Figure 30 (page 73).

Adhesive type	**1**	**2**
p	0.966	0.002
Result	Null hypothesis confirmed (nonsignificant)	Null hypothesis rejected (significant)

Table 19. t-test results about the difference of the area of favourable fracture mode between bracket 1 and 2 when using different adhesives. Significance was found only when using adhesive 2. α =0.05. 1 = adhesive No-Mix, 2 = adhesive Concise™.

Cohesive fracture mode

The bracket 2 (Ultra-Minitrim®) showed a significant smaller area of cohesive fracture mode when using adhesive 2 (Concise™). When using adhesive 1 (No-Mix) no significant difference was found between the two brackets (Table 20). The results are graphically presented in Figure 31 (page 76).

Adhesive type	**1**	**2**
p	0.087	0.031
Result	Null hypothesis confirmed (nonsignificant)	Null hypothesis rejected (significant)

Table 20. t-test results about the difference of the area of cohesive fracture mode between bracket 1 and 2 when using different adhesives. Significance was found only when using adhesive 2. α =0.05. 1 = adhesive No-Mix, 2 = adhesive Concise™.

Effect of the adhesive

The influence of the adhesive on the fracture mode was tested depending on the bracket used. The t-test was used. The comparisons were made between groups A_0 / C_0 and B_0 / $D_{0.}$

Favourable fracture mode

When comparing the favourable fracture area between specimens bonded with different adhesives, no significant difference was found for either bracket types (Table 21). The results are graphically presented in Figure 30 (page 73).

Bracket type	**1**	**2**
p	0.155	0.068
Result	Null hypothesis confirmed (nonsignificant)	Null hypothesis confirmed (nonsignificant)

Table 21. t-test results about the difference of the area of favourable fracture mode between adhesive 1 and 2 when using different brackets. No significant differences were found. α =0.05. 1 = bracket Discovery®, 2 = bracket Ultra-Minitrim®.

Cohesive fracture mode

The comparison of the specimens bonded with the two different adhesives showed a significant difference only when using bracket 2 (Ultra-Minitrim®, Table 22). Adhesive 2 showed lower cohesive fracture area than adhesive 1. The results are graphically presented in Figure 31 (page 76).

Bracket type	**1**	**2**
p	0.149	0.018
Result	Null hypothesis confirmed (nonsignificant)	Null hypothesis rejected (significant)

α =0.05

Table 22. t-test results about the difference of the area of cohesive fracture mode between adhesive 1 and 2 when using different brackets. Significance was found only when using bracket 2. 1 = bracket Discovery®, 2 = bracket Ultra-Minitrim®.

6.2.3 Visual examination

Material group A

In material combination group A (laser-structured bracket base Discovery® and two-component resin No-Mix) there was no statistically significant influence of the fatigue mode on the distribution of favourable and most favourable fracture mode. In the following figures the rough surface represents the impression of the disc and stands for the adhesive fracture between adhesive and disc, i.e. the least desirable one. The smoother formations in the middle represent the cohesive fracture mode, i.e. the most desirable one. The area, where the bracket base is exposed represents the adhesive fracture between bracket and adhesive and was only observed on areas where the base was smooth without any retention. This was mainly found on the border of the bracket at the side where the force came from. The sum of the area of adhesive fracture between bracket / adhesive and of the cohesive fracture area was defined as favourable fracture mode area (Figures 32, 33, 34 and 35)

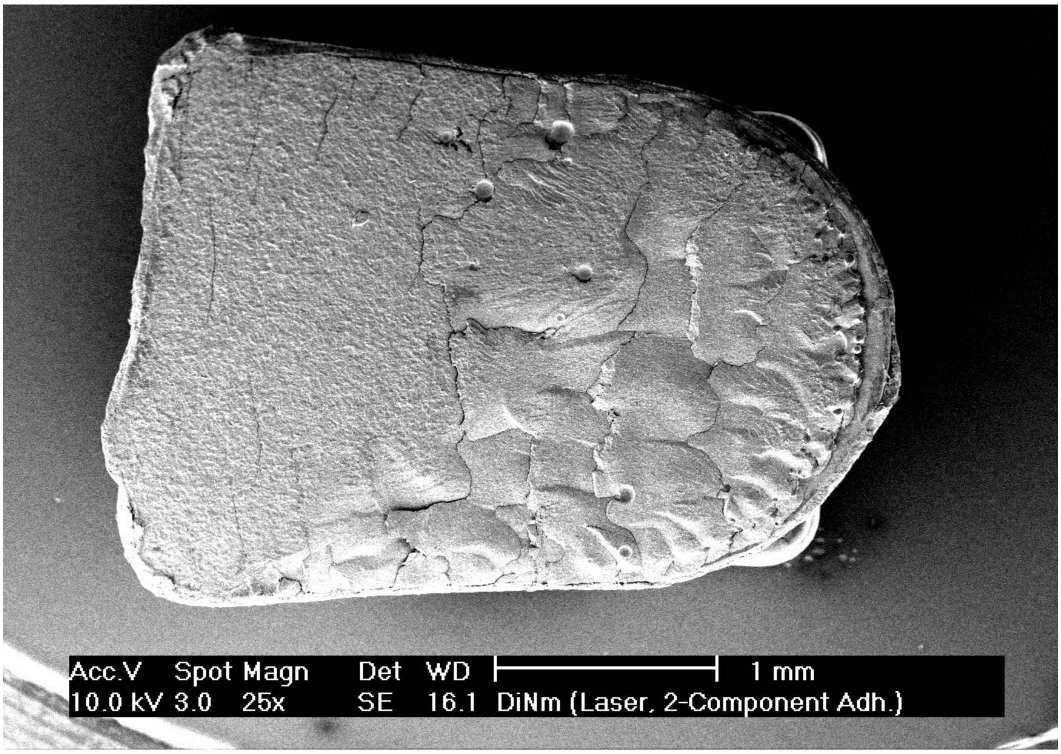

Figure 32. SEM-image. Example of a specimen in group A_0 (bracket Discovery® / adhesive No-Mix) after shear testing without fatigue.

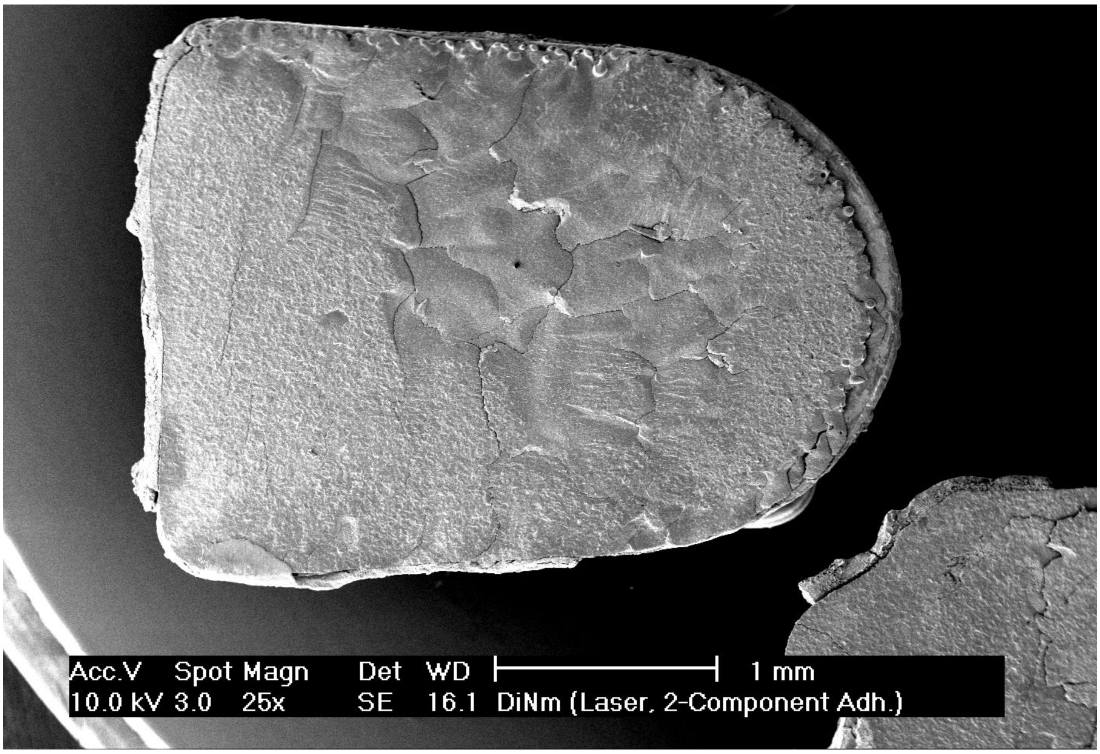

Figure 33. SEM-image. Example of a specimen in group A_1 (bracket Discovery® / adhesive No-Mix) that survived cyclic load and was sheared after that.

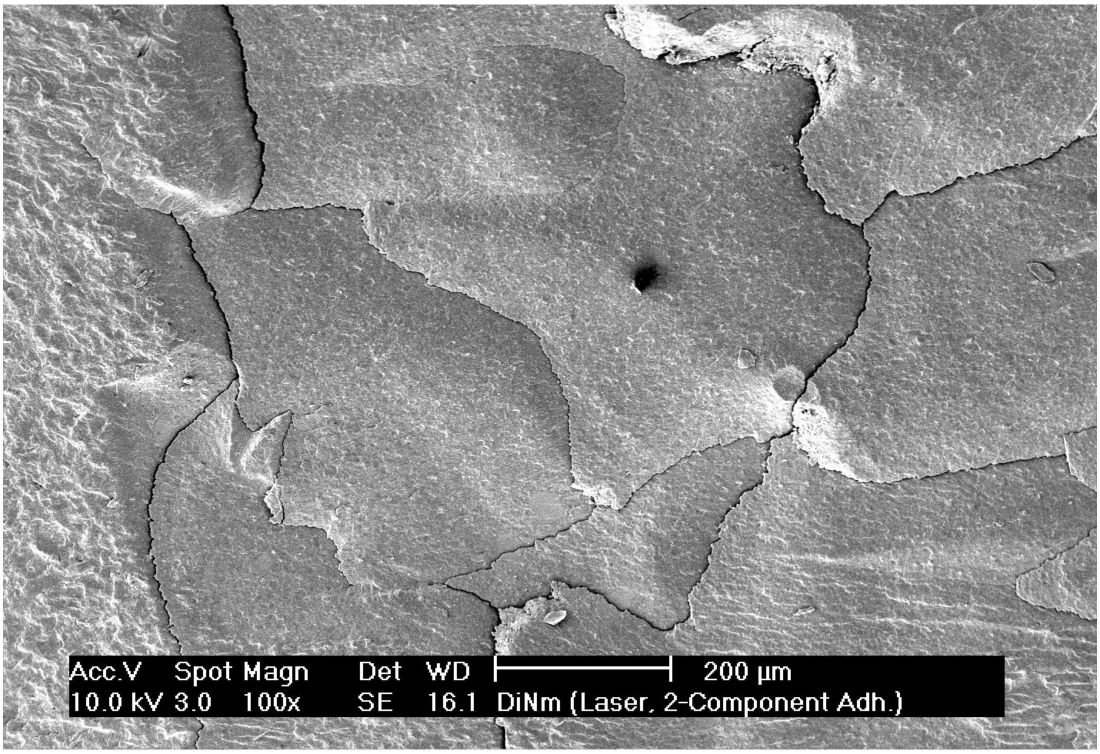

Figure 34. SEM-image. Magnification of the specimen in Figure 33 showing typical crack formations.

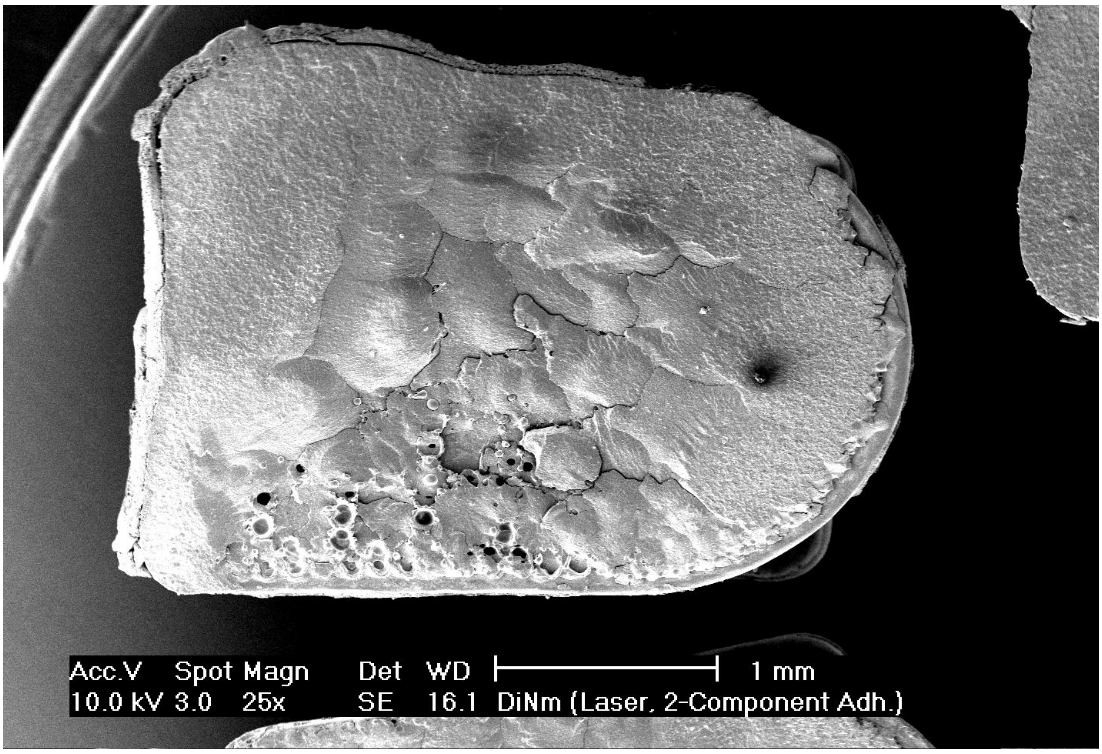

Figure 35. SEM-image. Example of a specimen in group A_2 (bracket Discovery® / adhesive No-Mix) which failed during fatigue.

Material group B

For material combination group B (foil-mesh bracket base Ultra-Minitrim® and two-component resin No-Mix), fatigue influenced only the favourable fracture mode area between the specimens which survived fatigue of 1,000 cycles and were sheared after that and the ones that failed during fatigue at a lower number than 1,000 cycles. The specimens that failed during fatigue showed a greater area of favourable fracture mode and thus a smaller area of adhesive fracture between disc and adhesive. The difference is mainly due to the different adhesive fracture area between bracket and adhesive. Fatigue showed no influence on the most favourable fracture mode, the cohesive fracture.

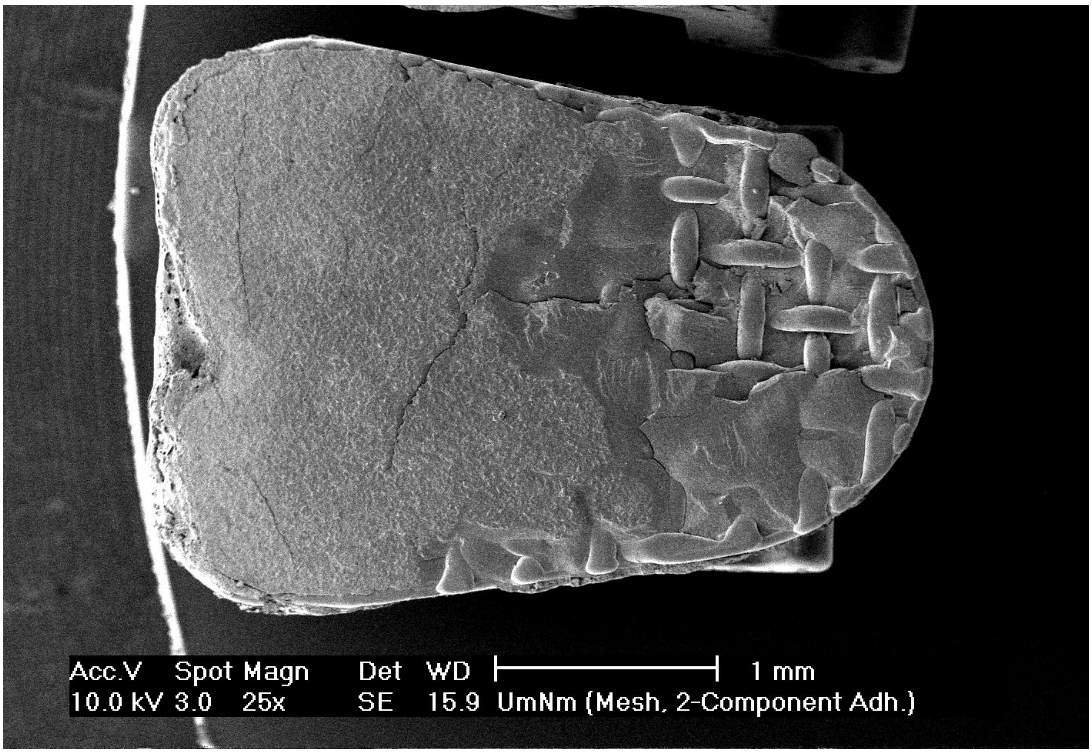

Figure 36. SEM-image. A nonfatigued sheared specimen of group B_0 (bracket Ultra-Minitrim® / adhesive No-Mix). The polished area in the middle represents the cohesive fracture. The area on the right, where parts of the mesh are uncovered, represents the adhesive fracture mode between bracket and adhesive. On the left, the entire adhesive remained on the bracket and the fracture occurred between adhesive and disc.

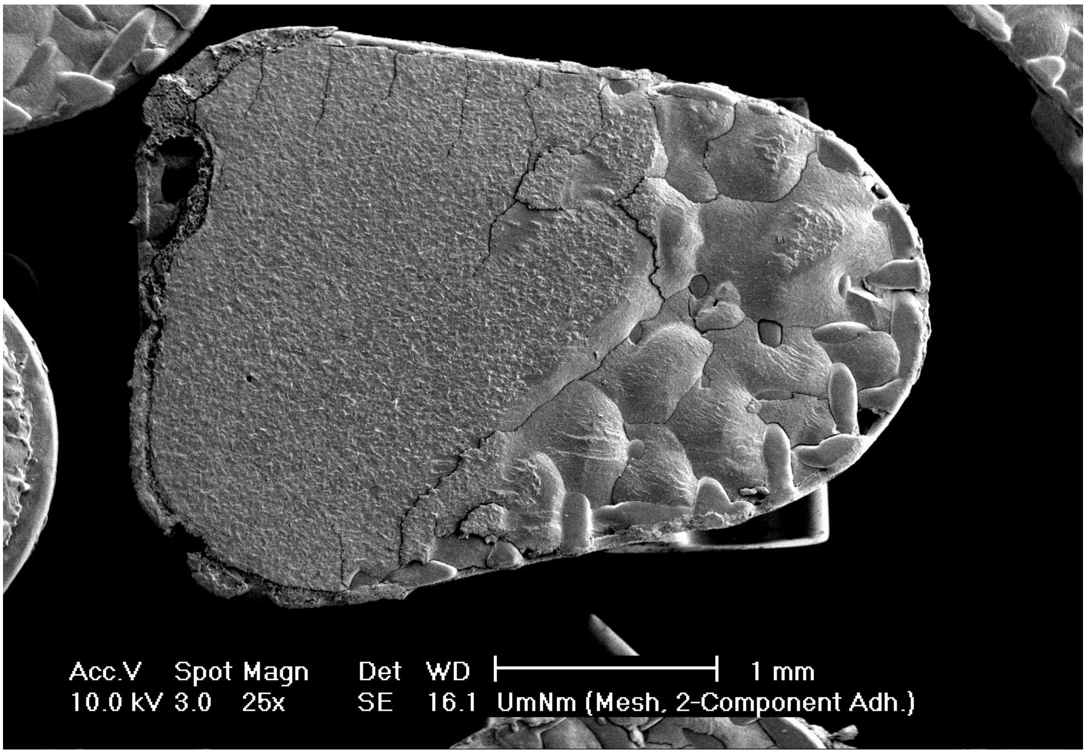

Figure 37. SEM-image. A sheared specimen after fatigue for 1,000 cycles (Group B_1, bracket Ultra-Minitrim® / adhesive No-Mix).

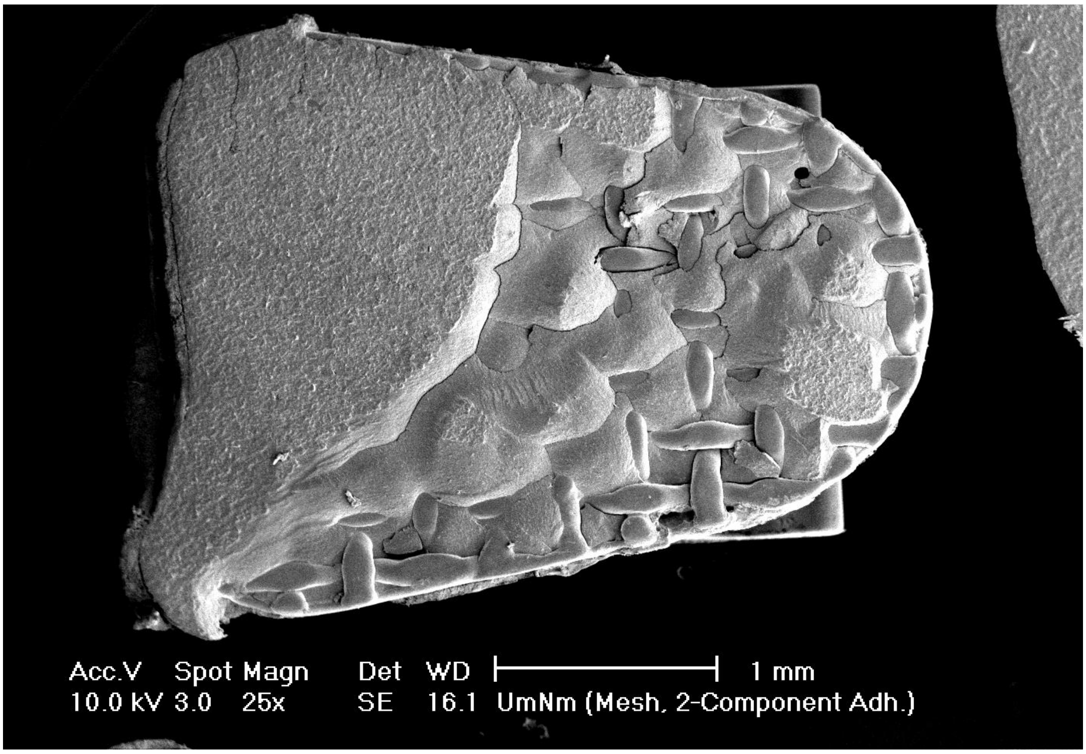

Figure 38. SEM-image. A specimen of group B_2 (bracket Ultra-Minitrim® / adhesive No-Mix), which failed during fatigue. The fracture between disc and adhesive (left side) occupies less area than on the specimen in Figure 37 and the area of favourable fracture mode is larger. The area of cohesive fracture (the broken resign in the middle) is nearly the same as on the specimen above: the difference is mainly due to the greater fracture area between bracket and adhesive (the area of the uncovered bracket mesh base) compared to Figure 37.

Material group C

The specimens of material combination group C (laser-structured bracket base Discovery® and four-component resin Concise™) which failed during fatigue showed a significant larger area of both favourable (cohesive + adhesive bracket/adhesive) and most favourable (cohesive) fracture mode than the ones that were fatigued and sheared after that. Consequently the area of adhesive fracture between disc and adhesive was smaller, i.e. less intact resin was left on the bracket. The difference was mainly due to the increase of the area of cohesive fracture mode.

The polished surface areas that were found were made by rubbing of the bottom and top of the crack. The specimens which survived fatigue showed no or little polished areas (Figure 40). The specimens that failed showed more and greater polished areas which involved clamshell markings (Figure 41).

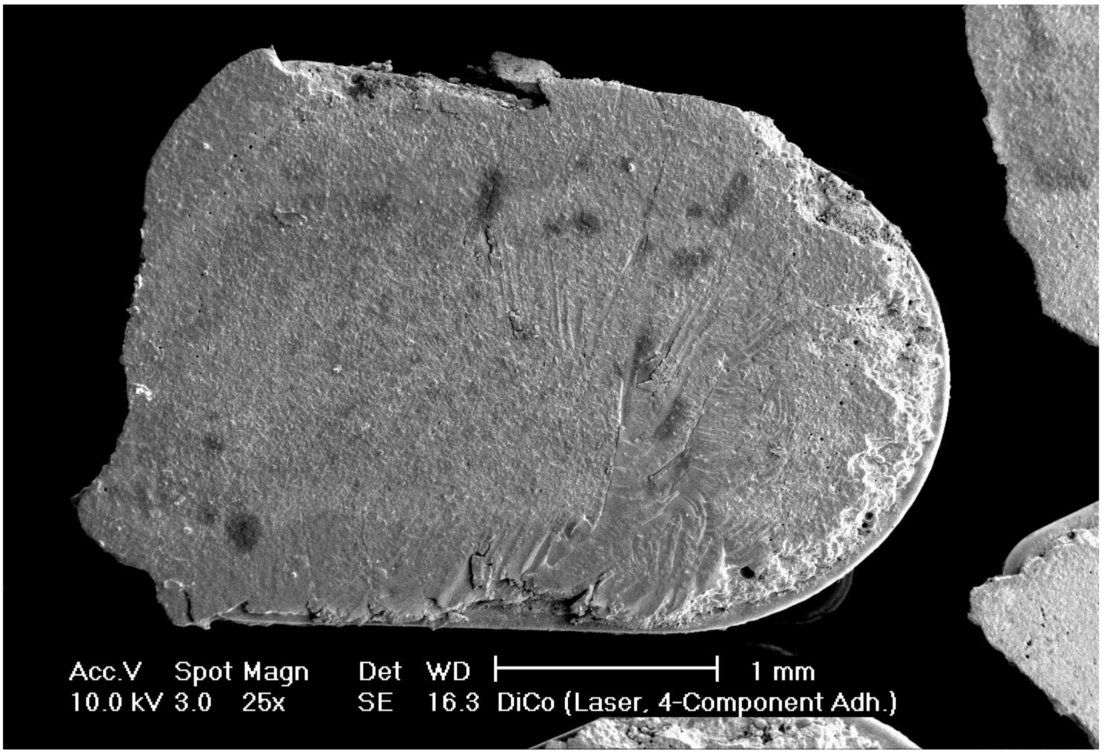

Figure 39. SEM-image. A nonfatigued sheared specimen of group C_0 (bracket Discovery® / adhesive Concise™).

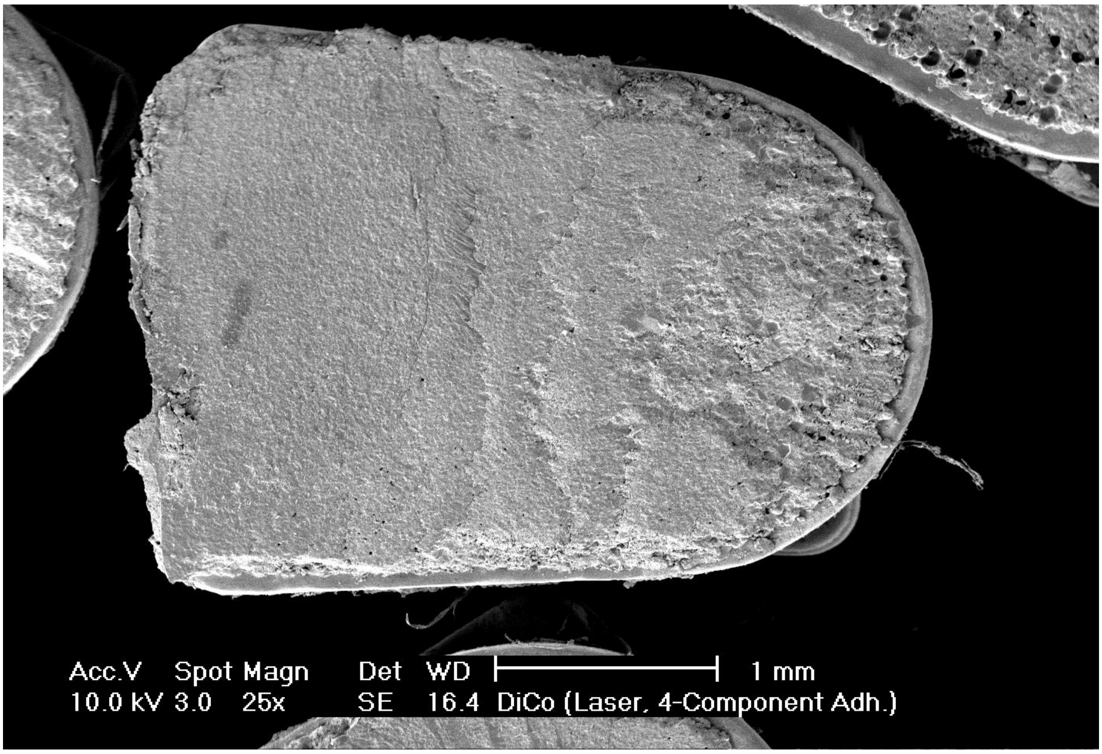

Figure 40. SEM-image. A sheared specimen of group C_1 (bracket Discovery® / adhesive Concise™) after fatigue for 1,000 cycles. There were no or little polished areas.

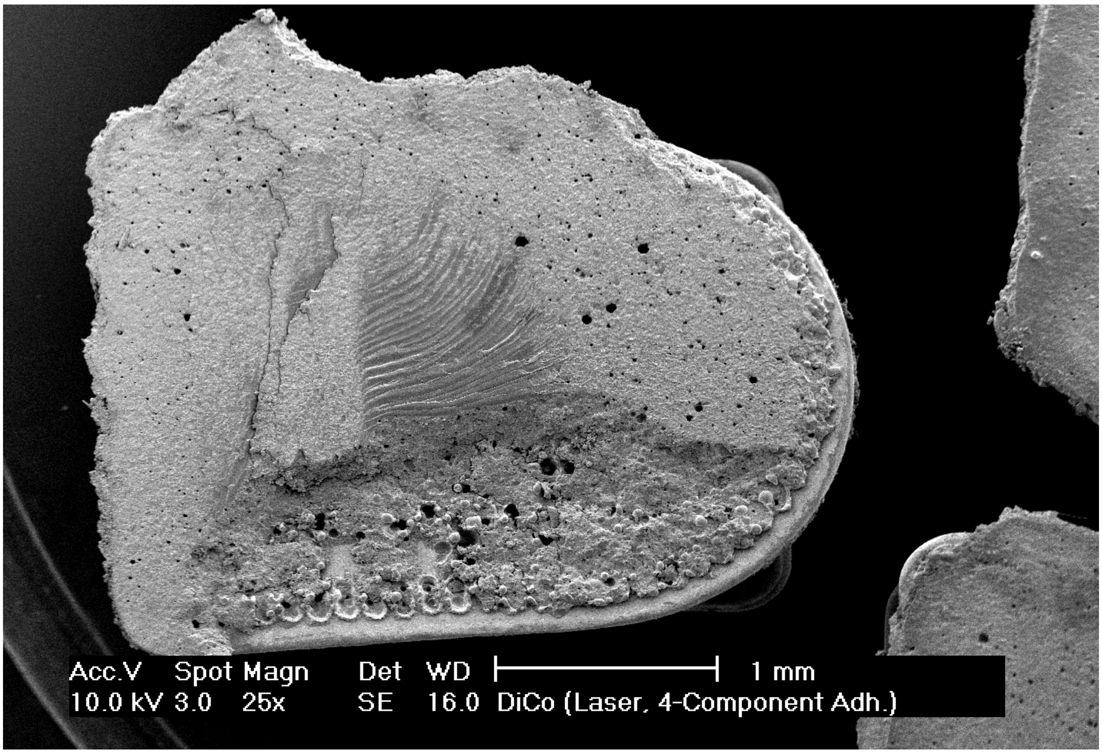

Figure 41. SEM-image. A specimen of group C_2 (bracket Discovery® / adhesive Concise™), which failed during fatigue. The cohesive fracture area was larger than in group C_1. The polished areas found were more and larger than in group C_1. On this bracket base, the polished area is in the middle and has lead to an overload and a sudden fracture of the resin, which appears granular in the rest of the cohesive area in the lower part of the picture.

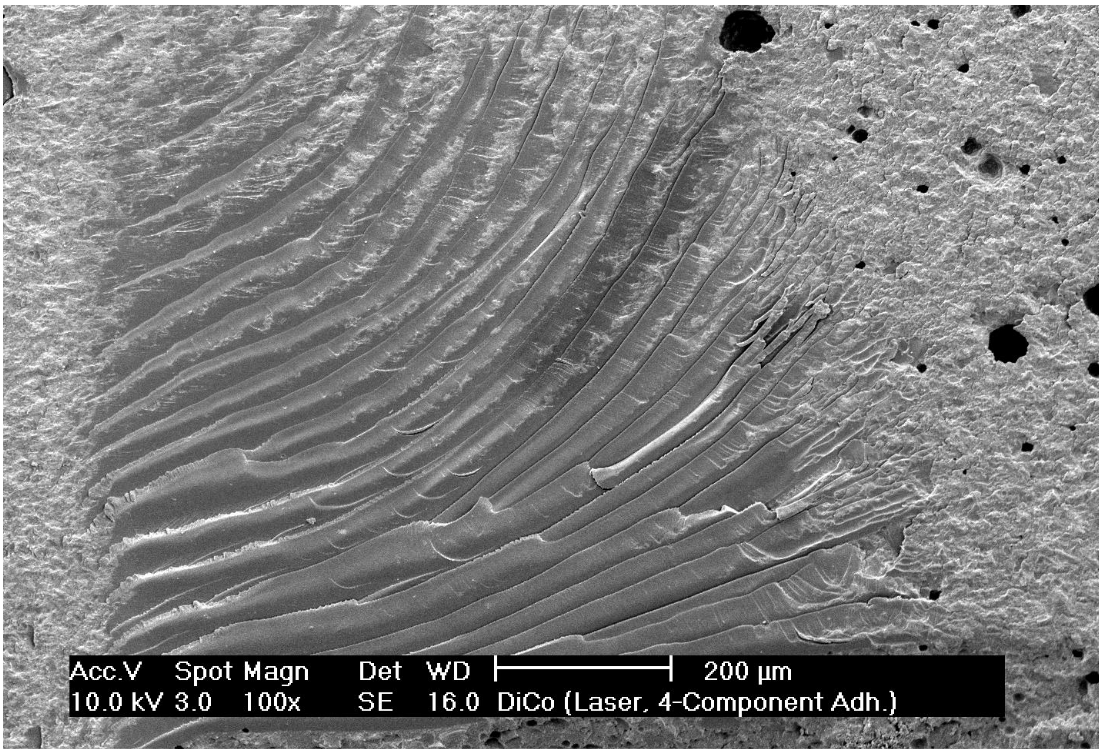

Figure 42. SEM-image. 100x magnification of the specimen in Figure 41 which shows typical clamshell markings.

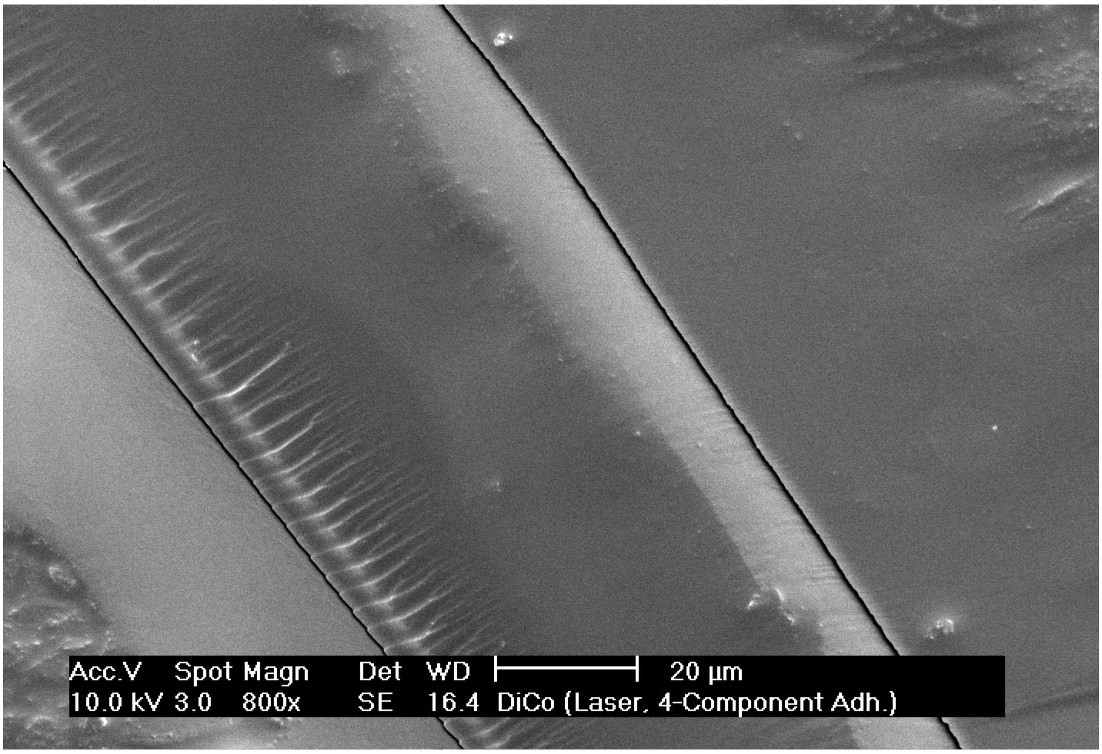

Figure 43. SEM-image. An 800x magnification of another specimen that failed during fatigue (group C_2, bracket Discovery® / adhesive Concise™). The picture shows a lot of epsilon discontinuous crack growth formations in the clamshell markings.

Material group D

In material combination group D (foil mesh bracket base Ultra-Minitrim® and four-component adhesive Concise™) the specimens that failed during fatigue showed a significantly larger area of favourable fracture than the fatigued ones that survived. The area of favourable fracture of the failed specimens was also greater than on the nonfatigued specimens. Fatigued specimens showed a significant increase in cohesive fracture compared to the nonfatigued ones and the failed-during-fatigue ones.

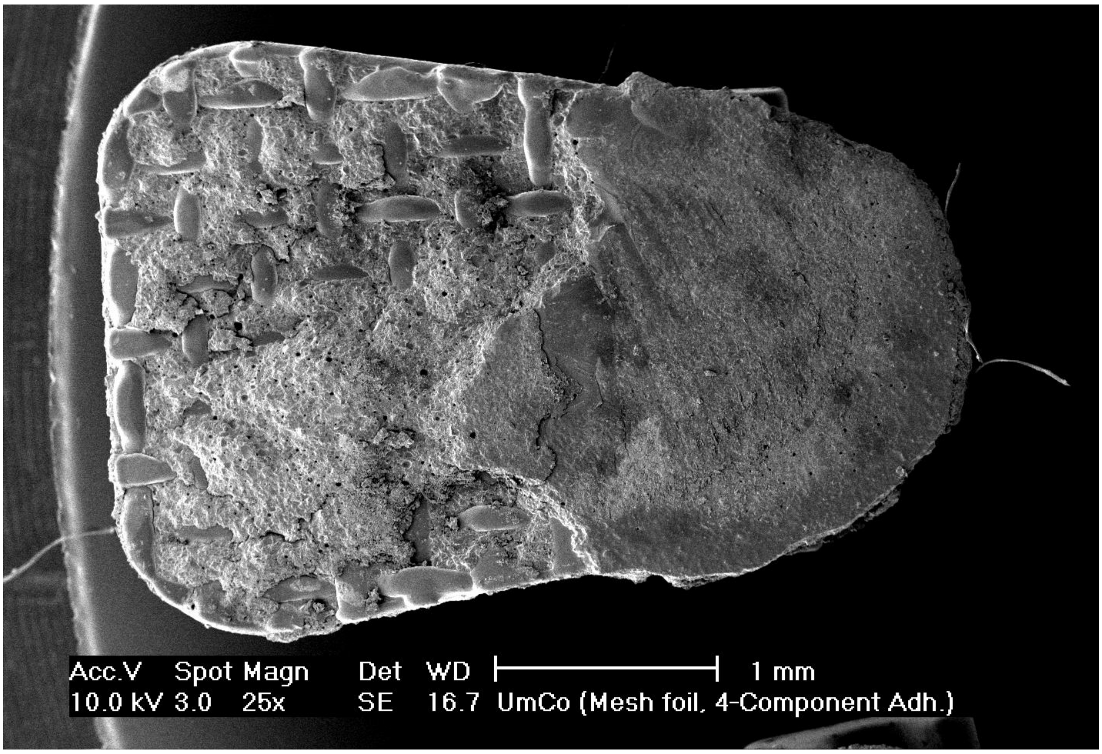

Figure 44. SEM-image. A nonfatigued sheared specimen of group D_0 (bracket Ultra-Minitrim® / adhesive Concise™).

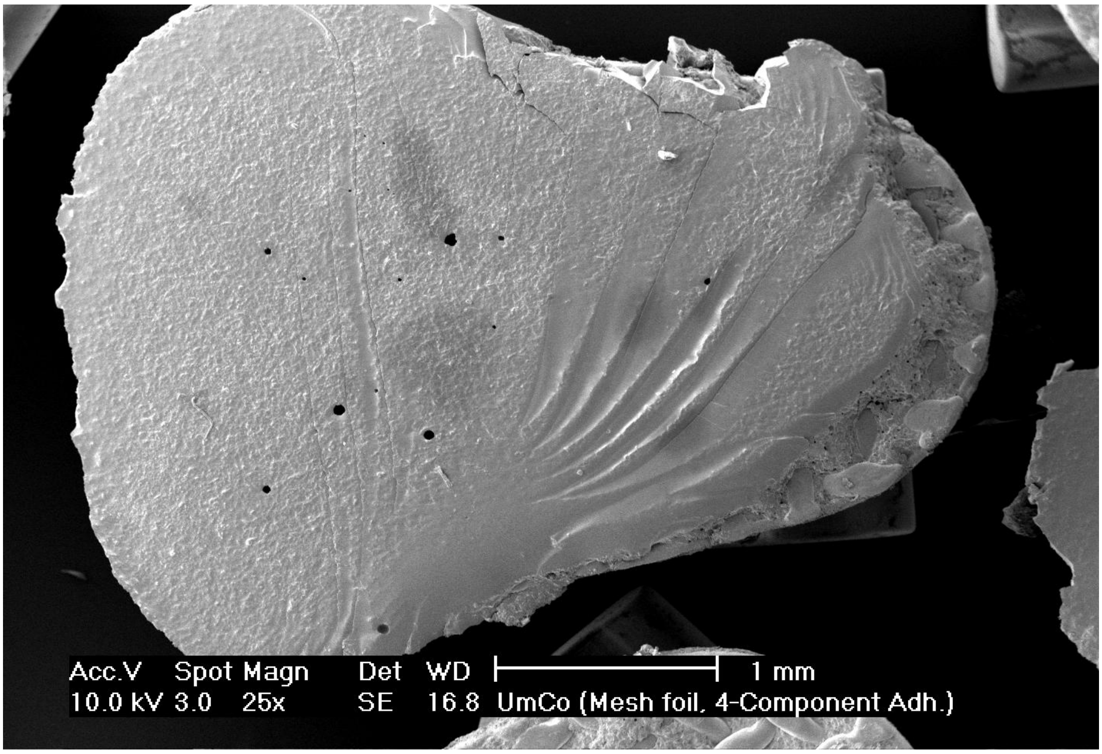

Figure 45. SEM-image. A sheared specimen of group D_1 (bracket Ultra-Minitrim® / adhesive Concise™) after 1,000 cycles of fatigue. The area of cohesive fracture (the polished part and the clamshell markings of the resin surface) is greater than on the nonfatigued specimen (Figure 44) and the failed during fatigue one (Figure 46).

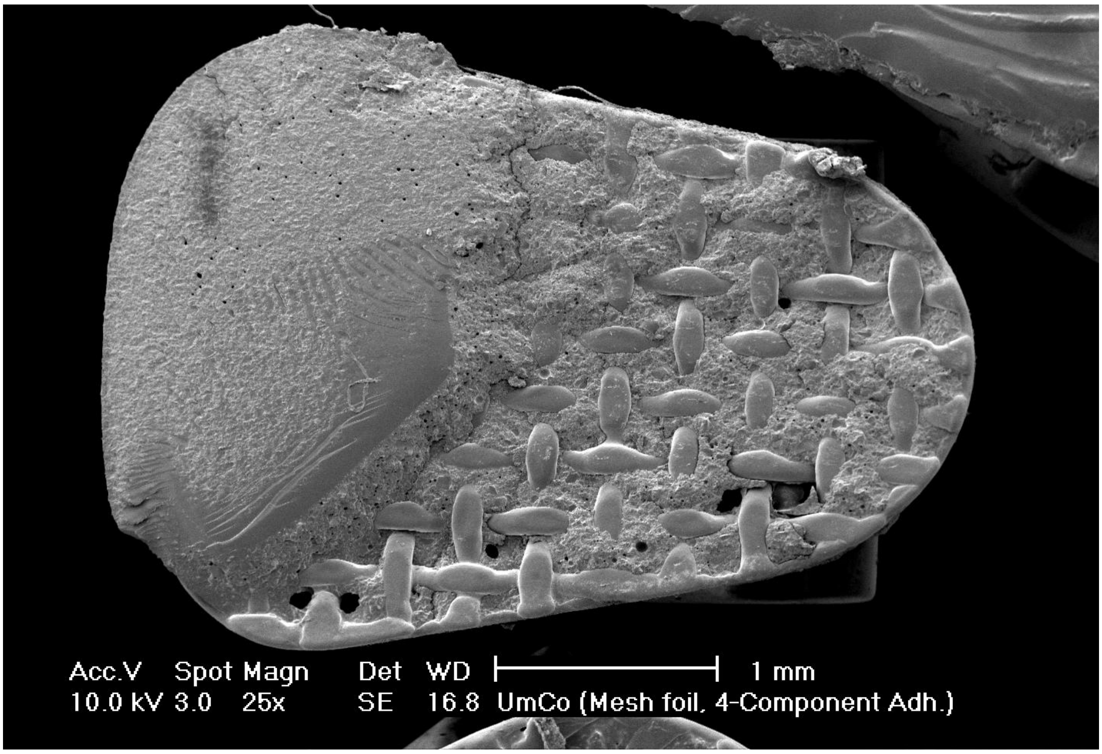

Figure 46. SEM-image. A specimen of group D_2 (bracket Ultra-Minitrim® / adhesive Concise™) that failed during fatigue. The polished part (cohesive fracture mode) of the area is smaller than on the fatigued specimen, which survived (Figure 45). The sum of cohesive fracture and fracture between bracket/adhesive, i.e. the area where parts or the entire adhesive is missing, is greater than in groups D_0 (Figure 44) and D_1 (Figure 45).

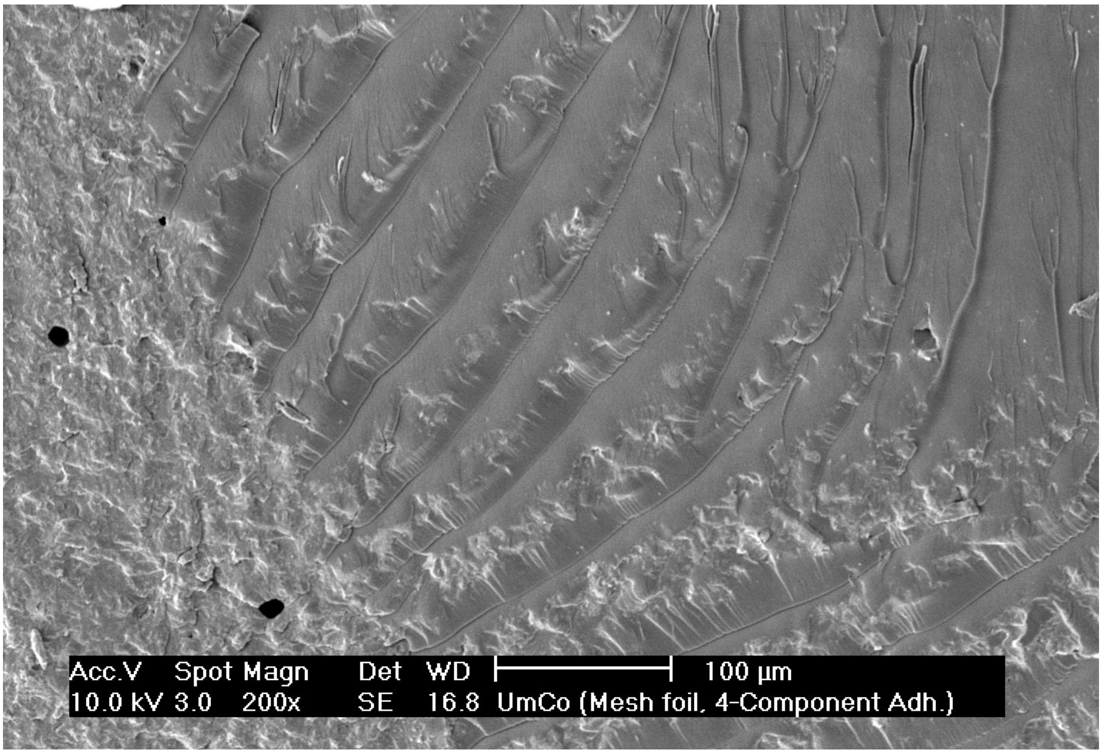

Figure 47. SEM-image. 200x magnification of the specimen in Figure 46 (fatigued and failed). The picture shows the border of the polished area, where the smooth surface of the clamshell markings (a result of the rubbing of the bottom and top of the crack) gets granular, due to the rapid failure of the material. Some fine epsilon discontinuous crack growth formations are visible.

7 Discussion

7.1 Discussion of the material selection

The selection of the materials used was mainly driven from contemporary orthodontic practice. In order to understand fatigue better, different materials and combinations of materials were used. The majority of the clinicians today use composite resins to bond attachments to teeth, though the use of glass ionomer cements and compomers have been popularised in orthodontics. In this study only composite resins were used. This allowed a better comparability and clinical usage of the results.

The initial intention to use one chemically and one light-curing adhesive had to be withdrawn, because the light-curing adhesive failed to adequately polymerise since light could not penetrate enough between the metal surfaces of the plates and the brackets.

In order to compare different adhesives, one two-component (No-Mix) and one four-component (Concise™) chemically curing resin was chosen, both of them containing Bis-GMA and TEGDMA. No-Mix contains relative less Bis-GMA and more TEGDMA (8.6 % / 42.35 %) than Concise™ (30 % / 25.25 %). The percentage in weight of the filler particles is similar for both adhesives (37-38.75 %).

The majority of orthodontic brackets used nowadays are made of stainless steel. They provide versatility, since they can be easily constructed and modified, good resistance to masticatory forces, easy removal by peeling them off and inexpensive manufacturing. However, resins do not chemically adhere to stainless steel. Retention mechanisms are necessary. The most common base retentions are mechanical. For this study, a bracket with the most common mechanical retention, the foil-mesh base, was chosen (Ultra-Minitrim®). In order to compare different retentions, a stainless steel bracket with laser structured base was selected (Discovery®). After a close comparison of the two bracket bases under the microscope, it is obvious that the laser structured bracket base has more undercuts and a greater effective surface area than the mesh base (see Figures 8, 9, 10 and 11 on pages 45 and 46). As expected, the strength of such retention has been reported to be higher than the mesh and more adhesive remains on the bracket after debonding (Sorel et al., 2002).

The brackets were bonded to silanised stainless steel surfaces. This was done in order to have a standardised surface and therefore exclude any effects on the bonding quality due to variation of the substrate quality. For example, when using teeth for bond strength experiments, the quality of the bonding can be affected by a more or less convex surface or by the varying microscopic quality of the tooth surface. The bond strengths found in this study (166 – 449 N) were much greater compared to specimens bonded to teeth, a fact that can be explained by the stronger bonding a silanised surface provides. The silanisation was necessary, because the steel discs had not any mechanical retention and the adhesives show no chemical affinity to metals. The higher magnitude of the force levels provided more exact adjustment of the testing machine and a smaller possibility for methodology errors. However, an extrapolation to the clinical situation should be made wisely.

7.2 Discussion of the methodology

The aim of this study was to investigate the sole influence of fatigue on the bracket-adhesive complex. The influence of other factors, such as test mode, aging, time, temperature, gap width or loading rate was not the objective. For these factors, the same values were used for all tests. Yet, since different materials were used in order to better describe fatigue induce, the influence of the two adhesives and two bracket types on the results was also investigated.

The test mode was shear, a common, easily controlled and reproducible research protocol in strength testing. Furthermore it resembles quite well the clinical condition, where masticatory forces are more likely to apply a shear load on the bracket. It is known that different testing modes, such as tension, shear, torsion, flexure or compression produce different results, which are not comparable with each other (Baran et al., 1998; Beatty and Pidaparti, 1993; Katona, 1997; Katona and Long, 2006; Suresh, 1998). Therefore, the results of this study should only be carefully compared to other research outcomes and should not be used to extrapolate effects in other test environments. Even when comparing only shear strength tests with each other, the configuration of the experiment affects the validity of the results: the applied force generates moments, which depend on the distance of the force application vector from the centre of resistance. For this reason, the force application vector in this study was kept parallel and as close to the bracket base as possible.

The multifaceted intraoral environmental milieu cannot be simulated with the currently available *in vitro* research methodologies. One common approach is to soak the specimens in water, which is known to affect the filler-matrix interface. It leaches out filler elements, induces flier and filler-matrix debonding and reduces the strength of the matrix. Most investigators found reduced strength of dental resins after soaking (Baran et

al., 1998; Braem et al., 1994b). Almost all strength research protocols include aging. Storage in water for 24 h is normally sufficient to discriminate between those materials that cannot and those that can withstand a wet environment (DIN Deutsches Institut für Normung e.V., 2002). In this study, the specimens were soaked in distilled water for three days. This time interval was chosen for consistency with another fatigue study (Aquilino et al., 1991). Furthermore, by letting time pass by, the adhesive was allowed to polymerise thoroughly and could reach higher bond strength, as described in the literature (Bishara et al., 1999b; Bishara et al., 2002; Braem et al., 1987; Chamda and Stein, 1996; Evans et al., 2002; Liu et al., 2004; Sharma-Sayal et al., 2003; Wendl and Droschl, 2004). Yet, the widely used protocol of thermocycling, which aims into simulating the thermal changes in the oral cavity, was not used in this study, in consistency with another fatigue study (Aquilino et al., 1991). All studies showed a decrease in bond strength of dental adhesive materials after thermocycling (Bishara et al., 2003; Bishara et al., 2007; Jassem et al., 1981).
The gap width between bracket base and metal disc was not controlled with the performed protocol. By using the same force for pressing the brackets on the metal disc, it can be assumed, that the gap width was kept constant among the same bracket specimens. Furthermore, the brackets used were manufactured with the intention to bond them on lower incisors. These teeth have the least convex surface and the corresponding brackets are nearly flat. Therefore, it can be assumed, that the gap width was almost uniform throughout the whole bonding surface and between the four material groups.
The crosshead speed during the fatigue testing in this study was adjusted at 5 mm/min and the mean frequency was 0.13 Hz, i.e. 8.06 cycles per minute. This allowed the fatigue testing to be completed in a reasonable amount of time (2-4 hours per specimen), The testing of the shear strength was performed at 1 mm/min, according to a recently published standard, where crosshead speeds of 0.75 mm/min ± 0.30 were recommended (DIN Deutsches Institut für Normung e.V., 2002). Loading velocity during testing may have an influence on bond strength. The data in the literature are contradicting. Some authors found decreased bond strength at higher loading rates (Bishara et al., 2005; Eliades et al., 2004), but these findings were not confirmed in other studies (Hara et al., 2001; Lindemuth and Hagge, 2000; Yamaguchi et al., 2006). Most studies were conducted at speeds of 0.1 – 10 mm/min. These values, including the ones used in this study, do not match clinical conditions. The chewing velocity is found to be 81 – 100 mm/sec (4860 – 6000 mm/min) and the chewing frequency 1.03 – 1.2 Hz (Buschang et al., 2000). For the fracture mode (cohesive vs. adhesive) the results are also contradict-

ing. It seems that there is no general rule and that each test protocol leads to different results.

In most strength testing studies, the debonding force was divided by the surface area of the bracket base to extrapolate the mean bond strength stress. The influencing factor “area of bracket base surface” is eliminated by this way. Nevertheless, the effective surface area of the bracket base in contact with the adhesive is far from the value used in this calculation. The presence of recesses, grooves, slots or spheres not only increases the effective surface area in contact to the bonding agent, but also improves the mechanical retention with the polymerised adhesive layer. The differences in overall morphology and in interfacial characteristics of the bracket-adhesive complex lead to variation in the load distribution pattern. Furthermore, the thickness of the adhesive layer is significantly affected by the design of the bracket base, meaning that smooth bracket bases lead to thinner adhesive film layers and a more homogeneous load application than rough bracket bases. This effect may depend upon the rheological properties of the adhesive and the size of the pores or grooves formed in the bracket base (Eliades et al., 1991). These considerations may validate the argument that clinicians should not be concerned with the expression of bond strength values in terms of stress, mean stress or stress distribution because this may be irrelevant to the actual force at which the system fails *in vivo*. Therefore the load measured in this study is given as a force [N]. In order to provide some comparability to other studies, the strength data are also given as a stress [MPa].

Many studies investigating fatigue use an experimental design of a ball mill (Millett et al., 2001). The mechanical action of ceramic spheres generates slow crack propagation in the bonding agent, which eventually leads to bond failure. This experimental design does not allow constant fatigue conditions, since forces of varying magnitude and direction operate in the ball mill.

In this investigation the detailed stages of fatigue response were not detected, because it is difficult to develop a method, sensitive enough to investigate these phenomena in the bracket-adhesive-complex. Therefore the total-life approach was used confining the description of flaw to the site of terminal failure, thus taking into consideration both the initiation and the propagation of fatigue cracks. However the initiation of dominant cracks can take up to 90 % of the cycles of the total fatigue life; the total-life-approach focuses mainly on the initiation of fatigue cracks (Suresh, 1998).

The fatigue method used in this study was the staircase or up-and-down method, which characterises the total fatigue life for a predefined number of cycles. This experimental protocol has been frequently used for testing dental materials (Aghadazeh Mohandesi et al., 2007; Aquilino et al., 1991; Braem et al., 1994b; Braem et al., 1995; Brandao et al., 2005; Frankenberger et al., 1999; Lohbauer et al., 2003; Lohbauer et al., 2005; McCabe et al., 1990; Saunders, 1990; Scherrer et al., 2003; Wiskott et al., 1999) and has been proposed as a fatigue testing standard (Brantley and Eliades, 2001).

Fatigue testing of dental materials reveals two types of behaviour. In type 1 behaviour – the classic fatigue behaviour – there is a clear relationship between fatigue life and fatigue stress, i.e. the fatigue life decreases with increasing applied fatigue stress. For type 2 behaviour no relationship exists between fatigue life and fatigue stress. The failure occurs at a level of stress below the ultimate strength of the material, but the values of fatigue life appear to be distributed randomly when several specimens of the same material are tested. For type 1 materials with data following a power law, there is a fatigue limit for each value of testing cycles. If the relationship follows a hyperbolic law, then there is one stress – one fatigue limit – below which no fracture will occur, even at high cycle fatigue. Type 2 materials are characterised by one fatigue limit, which is independent of fatigue life (survival time) and is the same for all values of testing cycles. For the dental materials, it appears that brittle materials such as dental plaster and heavily filled composites are likely to exhibit type 2 behaviour, whereas less brittle materials, such as more lightly filled composites, are more likely to exhibit type 1 behaviour (McCabe et al., 1990). For type 1 behaviour, testing should be carried out over a range of stresses and number of cycles in order for the relationship between the two properties to be characterised. If staircase testing is implemented in order to determine a fatigue limit, the testing should be carried out at a number of preselected fatigue life values (number of fatigue cycles). For each value of fatigue life about 15 specimens would be necessary. This requires the use of more specimens compared to the continuous method but gives data in which fatigue stress and fatigue life are better correlated. The measurement of a single fatigue limit value at one selected number of test cycles may not be a satisfactory way of evaluating type 1 materials. Such a method would only be valid in comparing two materials with similar fatigue behaviour, i.e. if the better performing material at high stresses also performs better at low stresses. If the fatigue behaviours are too different, the curves of two materials would intersect and cross. This would mean that at high stresses one material performs better than the other and at low stresses the position is reversed. If the character of a material is established as type 2, staircase testing can be adopted in any case for future evaluation (McCabe et al., 1990).

Composites are concerned to be brittle materials. The adhesives used in this study are therefore likely to show type 1 fatigue behaviour, but this is not evident. The tests were performed only at one fatigue life value, i.e. 1,000 cycles and the fatigue limit for this number of cycles was calculated. From the received results, the fatigue behaviour at another value of fatigue life cannot be extrapolated. This means that the better performing material combination would not necessarily perform better at a higher or lower number of fatigue cycles. Another concern with the staircase method is that its use virtually implies a fatigue limit. The assumption of a fatigue limit, i.e. a stress level below which no specimens will fail, is nonconservative and not useful for statistically based lifetime prediction efforts. Additionally, the predetermined cycle limit is seldom rationalised and the choice of a "low" limit will preclude observation of changes in fatigue mechanism. Furthermore, this method has been originally developed for the analysis of so-called sensitivity experiments and assumes that data are normally distributed, while the strength data for brittle materials typically fit the Weibull distribution (Dixon and Mood, 1948).

The design of this investigation was not sufficient to describe entirely the fatigue behaviour of the tested specimens. Nevertheless, the comparison of the fatigue behaviour of the tested materials was not the main objective of this study. The staircase method provided only a well-defined and reproducible protocol to induce fatigue to the specimens, so as to receive an experimental group to be compared to the nonfatigued control group. Since it is impossible to know before testing which type of fatigue behaviour a material will exhibit, it would be probably safer to carry out continuous fatigue cycling to failure for all materials in the first instance. If the character of a material is established as type 2, staircase testing can be adopted for future routine evaluations. If the fatigue behaviour is determined as type 1, the testing should be carried out at a number of preselected fatigue life values (McCabe et al., 1990). The comparison of two materials by this way should reveal more information about the parallelism of their fatigue behaviour, i.e. if the stronger material at a low number of fatigue cycles is also the better one at a high number of cycles. If one material performs better than the other at a low number of cycles and at high number the position is reversed, the curves of two materials would intersect and cross. Since *in vivo* fatigue failure occurs at different and unpredictable number of cycles, complete understanding of the fatigue behaviour of a material at any given number of cycles would be useful.

The staircase method assures that the fatigue testing is conducted at a load near the fatigue limit. The fatigue limit is the mean load, under which no failure occurs. Since this is a mean value, half of the specimens tested at this critical load are expected to fail and half to survive. The survived specimens are therefore loaded with a force just below the critical force, at which they are expected to fail. This way, the peak stress levels are of sufficiently high value for fatigue crack initiation and the specimens are loaded with the highest force possible without causing failure. The fatigue limit value depends on the number of fatigue cycles. The staircase testing assures that the specimens undergo a sufficient fatigue loading for the selected number of cycles. Therefore the method can be practiced for any number of cycles, assuring both that the highest possible fatigue force level and a sufficient number of cycles is used. If the loading pattern contains minimum and maximum peak values with large enough variation or fluctuation, all necessary factors for fatigue cracks initiation are fulfilled (NDT Resource Center, 2001). As a result, a standardisation of fatigue testing is possible.

However, using the staircase method to create an experimental group of fatigued specimens automatically utilises a predefined selection: Only survived specimens can be chosen for later strength testing. Due to the nature of the method, several specimens may be tested at the same load and can both fail or survive. Even if a test at the force level of the fatigue limit is conducted, half of the specimens are expected to fail and half to survive. Therefore it can be assumed, that the group made out of the survived ones, contains the best quality specimens. If fatigue testing is performed at a noncritical load, i.e. at a much smaller force level than the fatigue limit, assuming that all specimens survive, no specimen selection is made. All the fatigued specimens can be used for later strength testing. However, the peak stress levels are much smaller and probably not of sufficient high value for fatigue crack initiation.

Experimental protocols should be wisely planed, because testing at a critical load near the fatigue limit or at a much smaller noncritical load can lead to different results about the influence of fatigue. Experimental fatigue designs should take into account the mean chewing force, which was found to vary between 38 N and 160 N (Proffit and Fields, 1983; Proffit et al., 1983).

Fatigue cycling in dental materials has been reported for 100 to 1,000,000 cycles (Aghadazeh Mohandesi et al., 2007; Aquilino et al., 1991; Braem et al., 1994b; Draughn, 1979; McCabe et al., 1990; Moseley et al., 1995; Saunders, 1987; Williamson et al., 1993; Zardiackas et al., 1988). The number of 1,000 cycles chosen for this investigation was based on the relative slow cycling frequency of the Zwick testing machine at the shear loads used, which allowed each specimen to be tested within a reasonable amount of time (2-4 hours/specimen). Considering that as a result of chewing and swallowing the number of occlusal contacts per day is approximately 1,800 (Carranza F. A., 1984), the limit of 1,000 cycles represents < 1 day of *in vivo* usage.

Most researchers utilising fatigue testing compared the ultimate strength of the material with the fatigue limit and calculated the fatigue ratio (Aghadazeh Mohandesi et al., 2007; Drummond and Savers, 1993; Soderquist et al., 2006). The fatigue ratio may be a useful parameter to better understand fatigue behaviour. In addition, in the present study the ultimate shear strength of the nonfatigued specimens was compared to the shear strength of the fatigued specimens. This was done in accordance to other fatigue studies (Aquilino et al., 1991; Moseley et al., 1995) and is based on the assumption that failure will not only occur during low stress cyclic fatigue, but will also lead to a reduced strength against a sudden incident.

A scanning electron microscopic investigation of the bracket-base surfaces of the unused as well as the fatigued and nonfatigued adhesive-covered sheared specimens was performed. This was done to seek correlation of the fatigue behaviour with the morphological and structural features of the bare or adhesive-covered bases. The surface area was divided according to the fracture mode and measured.

The adhesive portions remaining on the bracket are usually described in the literature according to the Adhesive Remnant Index (ARI). The index simplifies the study of the fractured surfaces, containing only four possible scores depending on the amount of adhesive remaining on one surface. There is no distinguishing between different fracture modes, i.e. cohesive or adhesive fracture. Furthermore comparison to other studies is often not possible, because many researchers modify the ARI criteria. For these reasons the index was not used in this study. Instead, each fracture mode area was measured separately.

The large amount of fractographic data had to be reduced, in order to achieve a useful statistical analysis. The area of the adhesive fracture between bracket and adhesive is widely considered to be good, because it eliminates the risk of enamel detachment. However, the cohesive fracture mode is considered even more desired, because it combines the benefits of a low enamel detachment risk and easy removal of residual adhesive by the orthodontist. Furthermore a cohesive bond provides the strongest bonding (Matasa, 1989). The area of fracture between bracket and adhesive was added to the cohesive area and was defined as “favourable”. The cohesive fracture was evaluated separately, being the “most favourable”. The least desired fracture between adhesive and disc was not directly evaluated, but its influence is exactly the opposite from the “favourable” fracture mode.

7.3 Discussion of the results with reference to the findings in the literature

7.3.1 Shear strength

The comparison between nonfatigued and fatigued specimens in this study showed an influence, which depended on the material combinations tested. In material combination A (bracket Discovery® / adhesive No-Mix) the shear strength was increased by 8 % and in D (bracket Ultra-Minitrim® / adhesive Concise™) decreased by 10 % after fatigue. For material combinations B (bracket Ultra-Minitrim® / adhesive No-Mix) and C (bracket Discovery® / adhesive Concise™) no statistically significant differences were found. Therefore the common assumption that fatigue decreases the shear strength of the bracket-adhesive complex cannot be supported. In one case even increased shear strength of the fatigued specimens was found. A possible explanation for this is that the experimental group was put together from the survived specimens after staircase fatigue testing, a fact which probably selected the best quality specimens. A reason for no decrease of shear strength after fatigue in groups A, B and C can be that the applied stress was not sufficient to initiate and propagate crack growth during the predefined 1,000 fatigue cycles and therefore the behaviour of the fatigued specimens was not affected negatively. The fatigue behaviour strongly depended on the materials used.

The findings are partly consistent with the literature, where no uniform average effect of fatigue is reported. In most fatigue studies about dental composites, restorative materials have been used, which show no decrease of their strength after cyclic loading. In a study on prosthodontic adhesives, a decrease of the tensile bond strength of a 4-Meta-adhesive after cycling loading for 1,000 cycles according to the staircase method was found but no significant effect on Bis-GMA-systems. The more cross-linked nature of the Bis-GMA resins as well as the amount and composition of filler particles may have resulted in the greater resistance to tensile fatigue than for the unfilled methylmethacrylates used (Aquilino et al., 1991). Cyclic fatigue at low stress was reported not to reduce the shear bond strength of a resin-porcelain system. The specimens underwent aging for one week in 37°C distilled water and a fatigue load of 27,500 cycles at 26.6 N (2.3 MPa), which was only 13 % of the mean bond strength. The authors suggested implementing longer aging, more fatigue cycles and higher stresses in future investigations (Williamson et al., 1993). A four-point bending evaluation of dentin-composite interfaces revealed no influence of short-term thermo-cycling, NaOCl exposure, or 100,000 fatigue cycles at subcritical loads corresponding to stresses of the order of 40 % of the bending strength and frequency of 5 Hz (Staninec et al., 2008).

The only fatigue study about the bracket-adhesive complex comparing fatigued and non-fatigued specimens showed an influence of fatigue depending on the magnitude of the cyclic loading. Cyclic fatigue of brackets bonded to human teeth with composite resin or glass ionomer cement after aging at 37°C water for 24 hours at 0.5 Hz for 5,000 cycles showed no influence of low fatigue stress (5-10 N). However, fatigue stress at higher magnitudes (10-15 N) decreased the bond strength of the bracket complex. The reduction of shear bond strength after fatigue was 28-50 % for the composite and 6-49 % for the glass ionomer cement. The fatigue loads were less than 7 % (composite resin) and 20 % (glass ionomer) of the mean bond strength of the nonfatigued specimens, thus relative low (Moseley et al., 1995). These findings are partly contradicting to the findings of this study, where no uniform influence of fatigue was found, even at much higher force levels.

Some other studies reported a strength decrease after fatigue (Aghadazeh Mohandesi et al., 2007; Drummond and Savers, 1993; Soderquist et al., 2006). However, only the fatigue limit was compared to the ultimate strength of the specimens. The strength of the fatigued (and survived) specimens was not tested and therefore no comparison to the nonfatigued specimens was carried out.

The fatigue ratio was about 60 % for material groups A, B & C and 67 % for material group D. The adhesives used were both Bis-GMA systems. They have been reported to show a nearly constant (linear) fatigue ratio of 57-69 % in compressive and tensile fatigue testing (Aquilino et al., 1991; Draughn, 1979) in contradiction to unfilled methylmethacrylates, which showed a fatigue ratio of 38 % (Aquilino et al., 1991). Compressive fatigue tests of five dental composites (Bis-GMA, Bis -EMA, TEGDMA, UDMA) at 10 Hz for 100,000 cycles according to the staircase method after aging for two weeks in 37° distilled water resulted in a compressive fatigue ratio of 58-67 % (Aghadazeh Mohandesi et al., 2007). Other researchers found a less linear relationship between compressive fatigue limit and compressive strength varying between 0.52 and 0.70. The resin with the higher filler content of 64.2 % showed the highest compressive strength but also the lowest fatigue ratio (Brandao et al., 2005). Although the experimental design of this investigation differs substantially from previous ones, as it incorporates adhesives and brackets, the fatigue ratio surprisingly matched the results of fatigue studies of dental restorative materials. The findings are only partly consistent to another fatigue study about the bracket-adhesive system, where different brackets bonded to bovine teeth with the same light-cured composite resin were subjected to tensile fatigue at 2 mm/min crosshead speed at 1,000 cycles. The fatigue ratio varied between 68 and 92 % depending on the bracket base design and the only stainless steel bracket used showed a ratio of 86 % (Soderquist et al., 2006).

Knowledge of the fatigue ratio is useful as a predictor for the fatigue behaviour of the materials. Calculating the fatigue limit from the ultimate bond strength can be useful, because fatigue studies are more complex than a simple strength test. However, the benefit from this information is not always obvious, because new materials have to be tested both for ultimate and fatigue strengths in order to find out the fatigue ratio.

The laser structured base showed higher resistance against shear forces than the mesh base. The laser beam used to evaporate the metal during manufacturing leaves hole-shape retentions in the base. The effective surface area of the laser structured base is greater. More undercuts are present. This explains the higher ultimate bond strength of the bracket Discovery®, which was ca. 59 % higher than that of the mesh base bracket Ultra-Minitrim® and similar for both resins used. These findings agree with previously published data: under tensile testing the bracket Discovery® showed nearly twice as high strength than a foil mesh bracket using the adhesive No-Mix (Sorel et al., 2002). Under fatigue testing, the laser structured bracket base showed a fatigue ratio of ca. 0.60, which was again similar for both resins. The foil mesh bracket showed a fatigue ratio of ca. 0.60 with No-Mix and 0.67 with Concise™, indicating a better fatigue resistance at shear loading when used with the later adhesive.

Under shear strength testing, the four-component adhesive Concise™ showed about 66 % higher shear strength than the two-component No-Mix which was similar for both brackets. Under fatigue testing, No-Mix showed a similar fatigue ratio for both brackets of about 0.60. Concise™ showed a fatigue ratio of about 0.60 with the bracket Discovery® and 0.67 with Ultra-Minitrim®, indicating a better fatigue behaviour when used with the later bracket.

When tested with No-Mix, the bracket Discovery® failed either at a very low (relative quickly) or a relative high cycle number. The bracket Ultra-Minitrim® failed generally at a relative low cycle number. The bracket Discovery® was capable of surviving more cycles than Ultra-Minitrim®, although it tended to show also more quick failures. When tested with Concise™, no clear pattern was found for either bracket.

The results of this study clearly show that the influence of fatigue depended on the material combinations used and was not uniform under all conditions. The influence of the bracket design and the adhesive type on the shear bond strength of the nonfatigued specimens was consistent.

7.3.2 Fractography

The area of favourable fracture mode was not significantly different between fatigued and nonfatigued brackets. The area of the most favourable fracture mode, the cohesive fracture, was significantly larger in material group D on the fatigued specimens compared to the nonfatigued ones but no other differences were found. The data show that the adhesive amount entirely remaining on the bracket base after shear failure was the same regardless of whether the specimens underwent fatigue cycling. However there was a tendency for more adhesive to entirely remain on the bracket after fatigue, but no statistical significance was found. In material group D (Ultra-Minitrim® / Concise™) the adhesive which was entirely debonded from the bracket was less (and the total adhesive remaining on the bracket was more) after fatigue, indicating a stronger bond between bracket and adhesive after fatigue. There seems to be a negative correlation between the adhesion of bracket / resin and the shear strength after fatigue in group D, meaning that the smaller the shear strength after fatigue was, the more adhesive remained on the bracket. A possible explanation is that fatigue influenced negatively the adhesion between disc and resin, which is a smoother interface than the one between bracket base and resin. Crack initiation and propagation must have taken place more along the resin-disc interface. A reason for no differences in fractography between the fatigued and non-fatigued specimens in groups A, B and C can be that the applied stress was not sufficient to initiate and propagate crack growth during the predefined 1,000 fatigue cycles, i.e. the adhesive structure was not altered during fatigue. Therefore, the behaviour of the fatigued and nonfatigued specimens was the same. This assumption is supported by the fact that shear strength was not different between fatigued and nonfatigued specimens in groups A, B and C.

When examining the fatigued specimens that failed without completing 1,000 cycles in comparison to the ones that survived them and were sheared after that, a significant larger area of favourable fracture was found in groups B, C and D. In group D the area was also significantly larger compared to the nonfatigued specimens. The cohesive fracture area was smaller in group D and larger in group C. The data show that the amount of adhesive entirely remaining on the bracket was less for the failed-during-fatigue specimens in groups B (Ultra-Minitrim® / No-Mix), C (Discovery® / Concise™) and D (Ultra-Minitrim® / Concise™), although the total amount of adhesive remaining on the bracket did not differ in groups B and C. In group D, greater bracket surface area was completely uncovered, indicating a reduced bonding between adhesive and bracket for the failed-during-fatigue specimens. In group C the cohesive fracture was greater, but the overall adhesive remaining on the bracket was not different.

The brackets that failed during fatigue were loaded with a force which exceeded the fatigue limit in most cases. It is obvious that a crack initiation and propagation was induced, which leaded to failure before 1,000 cycles were completed. This crack growth led to more adhesive remaining on the disc, i.e. a more favourable fracture, indicating that the propagation took place more along the bracket-resin interface compared to the specimens that survived fatigue and failed at a sudden impact. This indicates that a specimen failing because of fatigue is likely to show a more favourable fracture than a specimen failing at a sudden impact after being fatigued.

The findings are partly consistent with another fatigue study, where the ARI-scores of brackets detached from bovine teeth were not different after fatigue (Soderquist et al., 2006). Furthermore, another study supports the assumption that the fracture surface morphology is visibly affected by the number of cycles until failure (Baran et al., 1998).

The subjective visual inspection of the bracket bases revealed that the area with no adhesive on the bracket (fracture between bracket and resin) was highly determined by the area with no retentions on the bracket base.

Polished surface areas were found mostly in combination with the adhesive Concise™. They are made by rubbing of the bottom and top of the crack. The specimens which survived fatigue showed no or little polished areas. The specimens that failed showed more and greater polished areas. Apparently, fatigue at higher stresses caused failure and rubbing of the crack surfaces, while the sudden impact on the survived fatigued specimens produced a more granular fracture surface.

Clamshell markings were also found on the polished areas, mostly with the adhesive Concise™, although the fatigue testing was not interrupted.

The influence of the bracket base on the residual adhesive of the nonfatigued specimens was not uniform. The laser structured base showed a higher retention to resin compared to the mesh base when using the composite Concise™. There was also a higher trend for the entire adhesive to remain on the bracket. The surface area of the laser structured base, which was entirely uncovered after debonding, was very small. Yet these differences were not found when using the composite No-Mix. The results partly agree with another investigation, where the bracket Discovery® showed higher proportions of the adhesive remaining on its base compared to a mesh base using the adhesive No-Mix (Sorel et al., 2002). This may be due to the different foil mesh bracket used. It is concluded, that the distribution of the residual adhesive on the bracket base depends on both the bracket and adhesive used.

The influence of the adhesive type was not uniform. The tested adhesives showed no difference in distribution of the favourable fracture area with either nonfatigued bracket-adhesive complex. This means that the area of the entire adhesive remaining on the bracket (which is the counterpart of the favourable fracture area) was statistically not different when using the two different adhesives. Both adhesives showed no differences in distribution of the most favourable (cohesive) fracture area when tested with the bracket Discovery®. The only difference found was a smaller cohesive fracture area for Concise™ compared to No-Mix when tested with the bracket Ultra-Minitrim®. This difference was mainly due to the larger area of uncovered bracket base, since the favourable fracture (i.e. the sum of cohesive fracture and the area of uncovered bracket base) was the same. When Concise™ failed with the mesh bases, the adhesive tended to uncover larger areas of the base than when No-Mix failed with the mesh bases, although the retention of entire adhesive portions to the bases was not affected. These findings indicate a more “all-or-nothing” fracture mode for the combination Concise™ / Ultra-Minitrim®, which is scientifically described as “alternating crack path”, i.e. a crack that jumps from one interface to the other. It is concluded, that the distribution of the residual adhesive on the bracket base depends on both the bracket and adhesive used.

7.4 Conclusions

Fatigue of the bracket-adhesive complex for 1,000 cycles showed a variable influence on shear strength and on the distribution of the residual resin on the bracket base. Its influence depended on the material combinations tested. The distribution of the residual resin on the nonfatigued specimens was also material-dependant. Only the shear strength of the nonfatigued specimens showed similar behaviour for all combinations tested. The staircase method can provide an easily utilised, reproducible experimental protocol for the standardisation of fatigue studies, if the influence of a load near the fatigue limit should be evaluated.

8 Abstract

A commonly encountered problem in orthodontics is the bond failure of brackets during treatment. The forces applied in the oral environment are more likely to be of a cyclical nature, well below the ultimate shear strengths reported in *in vitro* studies. Isolated or initial powerful impacts are seldom. The failure over time is much likely to be the result of fatigue. Powerful impacts may occur and lead to failure, particularly if the system has undergone fatigue loading.

The purpose of this study was to evaluate the influence of cyclic shear fatigue on the bracket-adhesive complex.

Brackets with laser structured bases (Discovery®, Dentaurum) and with foil mesh bases (Ultra–Minitrim®, Dentaurum) were bonded on silanised stainless steel flat plates with a two-component (No-Mix Bonding System, Dentaurum) and a four-component (Concise™, 3M Unitek) chemically-curing adhesive. The specimens were aged in distilled water at 37°C for 3 days. One group of the specimens was used as control to determine the ultimate shear bond strength without any fatigue procedure. The brackets of the second group underwent fatigue testing with a testing machine Zwick 1445 (Zwick GmbH & Co) according to the staircase method for 1,000 cycles. The survived fatigued specimens of the second group were subjected to shear strength testing. Comparisons between the values taken for fatigued and nonfatigued specimens were made to extrapolate the effect of fatigue on the shear bond strength. The shear fatigue limit and the fatigue ratio were calculated. The bracket bases were examined and photographed under 25x magnification with a scanning electron microscope. The distribution of the remaining adhesive on the bracket bases was analysed numerically and visually.

Shear fatigue of the bracket-adhesive complex for 1,000 cycles showed a variable influence on the shear strength of the bracket-adhesive complex, which was strongly dependent on the material combinations tested. Fatigued specimens showed an increase in shear strength of 8% in material group A (bracket Discovery® / adhesive No-Mix) and a decrease of 10 % in material group D (bracket Ultra-Minitrim® / adhesive Concise™). In material groups B (bracket Ultra-Minitrim® / adhesive No-Mix) and C (bracket Discovery® / adhesive Concise™) no statistically significant differences between the fatigued and nonfatigued specimens were found. The fatigue ratio was about 60 % for material groups A, B & C and 67 % for material group D, indicating a better fatigue behaviour of the combination Ultra-Minitrim® / Concise™. Among the nonfatigued specimens, the laser-structured bracket Discovery® showed about 59 % higher shear strength than the

foil-mesh bracket Ultra-Minitrim®. The four-component adhesive Concise™ showed about 66 % higher shear strength than the two-component adhesive No-Mix.

Concerning the distribution of residual resin on the bracket bases, fatigue was found to have different influence depending on the fracture mode tested. Fatigue played the same role on the area of favourable fracture mode (sum of fracture between bracket / resin and cohesive fracture) independently of the material combinations tested, but statistical significant differences were not always present. The area of favourable fracture mode was not significantly different between the nonfatigued and fatigued specimens. Yet when examining the fatigued specimens that failed without completing 1,000 cycles in comparison to the ones that survived and were sheared after that, a significant larger area of favourable fracture was found in groups B, C and D. In group D the area was also significantly larger compared to the nonfatigued specimens. The most favourable fracture mode, the cohesive fracture, was also fatigue-dependant, but fatigue had a different influence depending on the material groups. In groups A and B no significant influence of fatigue was found. Fatigue showed a significant increase in cohesive fracture in group D and the failed-during-fatigue specimens showed a smaller area than the survived ones. In material group C the failed-during-fatigue specimens showed a significantly higher cohesive fracture that the survived ones – in contrast to group D. Concerning the nonfatigued specimens, the influence of the bracket type on the distribution of residual resin was the same for both fracture modes and depended on the material combinations. Bracket 2 (Ultra-Minitrim®) showed a higher favourable and most favourable fracture mode than bracket 1 (Discovery®) when tested with adhesive 2 (Concise™) and no differences with adhesive 1 (No-Mix). The influence of the adhesive type was not the same for the two fracture modes and depended on the material combinations. Adhesive 1 and 2 showed no difference in the distribution of the favourable fracture area with either bracket. The most favourable fracture area was lower for adhesive 2 when tested with bracket 2 and no differences were found with bracket 1.

In general, fatigue had a variable influence on shear strength and on the distribution of the residual resin on the bracket base. Its influence depended on the material combinations tested. The distribution of the residual resin on the nonfatigued specimens was also material-dependant. Only the shear strength of the nonfatigued specimens showed similar behaviour for all combinations tested. The staircase method can provide an easily utilised, reproducible experimental protocol for the standardisation of fatigue studies, if the influence of a load near the fatigue limit is to be evaluated.

9 Literature

3M ESPE. Rocatec Bonding - Scientific Product Profile. 3M ESPE, 2001

Aghadazeh Mohandesi J, Rafiee MA, Barzegaran V, Shafiei F. Compressive fatigue behavior of dental restorative composites. Dent Mater J 2007; 26: 827-837

Albert WAJ. Über Treibseile am Harz. Archive für Mineralogie, Geognosie, Bergbau und Hüttenkunde 1838; 215-234

Aquilino SA, az-Arnold AM, Piotrowski TJ. Tensile fatigue limits of prosthodontic adhesives. J Dent Res 1991; 70: 208-210

Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984; 85: 333-340

Asmussen E. Factors affecting the quantity of remaining double bonds in restorative resin polymers. Scand J Dent Res 1982; 90: 490-496

Asmussen E, Jorgensen KD. Fatigue strength of some resinous materials. Scand J Dent Res 1982; 90: 76-79

Asmussen E, Peutzfeldt A. Influence of UEDMA BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. Dent Mater 1998; 14: 51-56

Baran GR, McCool JI, Paul D, Boberick K, Wunder S. Weibull models of fracture strengths and fatigue behavior of dental resins in flexure and shear. J Biomed Mater Res 1998; 43: 226-233

Baran GR, Boberick K, McCool J. Fatigue of restorative materials. Crit Rev Oral Biol Med 2001; 12: 350-360

Beatty MW, Pidaparti RM. Elastic and fracture properties of dental direct filling materials. Biomaterials 1993; 14: 999-1002

Bishara SE, Gordan VV, Vonwald L, Jakobsen JR. Shear bond strength of composite, glass ionomer, and acidic primer adhesive systems. Am J Orthod Dentofacial Orthop 1999a; 115: 24-28

Bishara SE, Vonwald L, Olsen ME, Laffoon JF. Effect of time on the shear bond strength of glass ionomer and composite orthodontic adhesives. Am J Orthod Dentofacial Orthop 1999b; 116: 616-620

Bishara SE, Laffoon JF, Vonwald L, Warren J. Effect of time on the shear bond strength of cyanoacrylate and composite orthodontic adhesives. Am J Orthod Dentofacial Orthop 2002; 121: 297-300

Bishara SE, Ajlouni R, Laffoon JF. Effect of thermocycling on the shear bond strength of a cyanoacrylate orthodontic adhesive. Am J Orthod Dentofacial Orthop 2003; 123: 21-24

Bishara SE, Soliman MM, Oonsombat C, Laffoon JF, Ajlouni R. The effect of variation in mesh-base design on the shear bond strength of orthodontic brackets. Angle Orthod 2004; 74: 400-404

Bishara SE, Soliman M, Laffoon J, Warren JJ. Effect of changing a test parameter on the shear bond strength of orthodontic brackets. Angle Orthod 2005; 75: 832-835

Bishara SE, Ostby AW, Laffoon JF, Warren J. Shear bond strength comparison of two adhesive systems following thermocycling. A new self-etch primer and a resin-modified glass ionomer. Angle Orthod 2007; 77: 337-341

Bliss C. The calculation of the dose-mortality curve. Annals of Applied Biology 1935; 22: 134-167

Braem M, Lambrechts P, Vanherle G, Davidson CL. Stiffness increase during the setting of dental composite resins. J Dent Res 1987; 66: 1713-1716

Braem M, Finger W, Van Doren VE, Lambrechts P, Vanherle G. Mechanical properties and filler fraction of dental composites. Dent Mater 1989; 5: 346-348

Braem M, Lambrechts P, Vanherle G. Clinical relevance of laboratory fatigue studies. J Dent 1994a; 22: 97-102

Braem MJ, Davidson CL, Lambrechts P, Vanherle G. In vitro flexural fatigue limits of dental composites. J Biomed Mater Res 1994b; 28: 1397-1402

Braem MJ, Lambrechts P, Gladys S, Vanherle G. In vitro fatigue behavior of restorative composites and glass ionomers. Dent Mater 1995; 11: 137-141

Brandao L, Adabo GL, Vaz LG, Saad JR. Compressive strength and compressive fatigue limit of conventional and high viscosity posterior resin composites. Braz Oral Res 2005; 19: 272-277

Brantley WA, Eliades T. Orthodontic materials - Scientific and clinical aspects. New York: Thieme, 2001

Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955; 34: 849-853

Buschang PH, Hayasaki H, Throckmorton GS. Quantification of human chewing-cycle kinematics. Arch Oral Biol 2000; 45: 461-474

Cacciafesta V, Bosch C, Melsen B. Clinical comparison between a resin-reinforced self-cured glass ionomer cement and a composite resin for direct bonding of orthodontic brackets. Part 1: Wetting with water. Clin Orthod Res 1998; 1: 29-36

Cantwell WJ, Roulinmoloney AC, Kaiser T. Fractography of Unfilled and Particulate-Filled Epoxy-Resins. Journal of Materials Science 1988; 23: 1615-1631

Carranza F. A. In: Glickmun's Clinical Periodontology. Philadelphia: W.B. Saunders Company, 1984: 73-81

Caspersen I. Residual acrylic adhesive after removal of plastic orthodontic brackets: a scanning electron microscopic study. Am J Orthod 1977; 71: 637-650

Chamda RA, Stein E. Time-related bond strengths of light-cured and chemically cured bonding systems: an in vitro study. Am J Orthod Dentofacial Orthop 1996; 110: 378-382

Ching E, Cook PA, Bubb NL, Wood DJ. The effect of early static loading on the in vitro shear/peel bond strength of a 'no-mix' orthodontic adhesive. Eur J Orthod 2000; 22: 555-559

Dickinson PT, Powers JM. Evaluation of fourteen direct-bonding orthodontic bases. Am J Orthod 1980; 78: 630-639

Diedrich P. Die Bracketentfernung und anschließende Schmelzpolitur — eine rasterelektronenmikroskopische Studie. Journal of Orofacial Orthopedics / Fortschritte der Kieferorthopädie 1980; 41: 491-502

Diedrich P. Enamel alterations from bracket bonding and debonding: a study with the scanning electron microscope. Am J Orthod 1981; 79: 500-522

Dieter GE. In: GE Dieter, editor Mechanical Metallurgy. New York: McGraw-Hill Book Co., 1961: 446-449

DIN Deutsches Institut für Normung e.V. ISO/TS 11405 Dental materials – Testing of adhesion to tooth structure. 2002

Dixon WJ, Mood AM. A method for obtaining and analyzing sensitivity data. J Amer Statist Assoc 1948; 43: 109-126

Draughn RA. Compressive fatigue limits of composite restorative materials. J Dent Res 1979; 58: 1093-1096

Draughn RA. Effects of temperature on mechanical properties of composite dental restorative materials. J Biomed Mater Res 1981; 15: 489-495

Draughn RA. Fatigue and fracture mechanics of composite resins. In: G VanHerle and DC Smith, editors. Posterior composite resin dental restorative materials. St. Paul (MN): 3M, 1988: 299-307

Drummond JL. Cyclic fatigue of composite restorative materials. J Oral Rehabil 1989; 16: 509-20

Drummond JL, Savers EE. In vitro aging of a heat/pressure-cured composite. Dent Mater 1993; 9: 214-216

Dyer KP, Isaac DH. Fatigue behaviour of continuous glass fibre reinforced composites. Composites Part B-Engineering 1998; 29: 725-733

Eliades T, Viazis AD, Eliades G. Bonding of ceramic brackets to enamel: morphologic and structural considerations. Am J Orthod Dentofacial Orthop 1991; 99: 369-375

Eliades T, Viazis AD, Lekka M. Failure mode analysis of ceramic brackets bonded to enamel. Am J Orthod Dentofacial Orthop 1993; 104: 21-26

Eliades T, Brantley WA. The inappropriateness of conventional orthodontic bond strength assessment protocols. Eur J Orthod 2000; 22: 13-23

Eliades T, Katsavrias E, Zinelis S, Eliades G. Effect of loading rate on bond strength. J Orofac Orthop 2004; 65: 336-342

Eliades T, Bourauel C. Intraoral aging of orthodontic materials: the picture we miss and its clinical relevance. Am J Orthod Dentofacial Orthop 2005; 127: 403-412

Evans LJ, Peters C, Flickinger C, Taloumis L, Dunn W. A comparison of shear bond strengths of orthodontic brackets using various light sources, light guides, and cure times. Am J Orthod Dentofacial Orthop 2002; 121: 510-515

Faust JB, Grego GN, Fan PL, Powers JM. Penetration coefficient, tensile strength, and bond strength of thirteen direct bonding orthodontic cements. Am J Orthod 1978; 73: 512-525

Fenske C, Sadat-Khonsari R, Bauss O, Seedorf H, Kirsch I, Kahl-Nieke B, et al. In vitro study on the fatigue limit of single-lap joints. J Orofac Orthop 2003; 64: 275-283

Ferracane JL, Berge HX, Condon JR. In vitro aging of dental composites in water — effect of degree of conversion, filler volume, and filler/matrix coupling. J Biomed Mater Res 1998; 42: 465-72

Fowler CS, Swartz ML, Moore BK, Rhodes BF. Influence of selected variables on adhesion testing. Dent Mater 1992; 8: 265-269

Fox NA, McCabe JF, Buckley JG. A critique of bond strength testing in orthodontics. Journal of Orthodontics 1994; 21: 33-43

Frankenberger R, Kramer N, Petschelt A. Fatigue behaviour of different dentin adhesives. Clin Oral Investig 1999; 3: 11-17

Garson GD, 2008: Syllabus for PA 765: Quantitative Research in Public Administration. http://www2.chass.ncsu.edu/garson/PA765/anova.htm (Access date: 30th March 2008)

Gladys S, Van Meerbeek B, Braem M, Lambrechts P, Vanherle G. Comparative physico-mechanical characterization of new hybrid restorative materials with conventional glass-ionomer and resin composite restorative materials. J Dent Res 1997; 76: 883-94

Gladys S, Braem M, Van Meerbeek B, Lambrechts P, Vanherle G. Immediate versus one-month wet storage fatigue of restorative materials. Biomaterials 1998; 19: 541-544

Hara AT, Pimenta LA, Rodrigues AL, Jr. Influence of cross-head speed on resin-dentin shear bond strength. Dent Mater 2001; 17: 165-169

Haydar B, Sarikaya S, Cehreli ZC. Comparison of shear bond strength of three bonding agents with metal and ceramic brackets. Angle Orthod 1999; 69: 457-462

Horst JJ, Spoormaker JL. Fatigue fracture mechanisms and fractography of short-glassfibre-reinforced polyamide 6. Journal of Materials Science 1997; 32: 3641-3651

Ireland AJ, Sherriff M. Use of an adhesive resin for bonding orthodontic brackets. Eur J Orthod 1994; 16: 27-34

Ireland AJ, Sherriff M. The effect of timing of archwire placement on in vivo bond failure. Br J Orthod 1997; 24: 243-245

Jahnig A, Henkel S. Glasionomer-Zemente als kieferorthopadische Bracketkleber. Eine In-vitro-Studie mit vier Glasionomer-Zementen (GIZ) und zwei konventionellen kieferorthopadischen Bracketklebern als Vergleichgruppe. Fortschr Kieferorthop 1990; 51: 204-207

James SP, Jasty M, Davies J, Piehler H, Harris WH. A fractographic investigation of PMMA bone cement focusing on the relationship between porosity reduction and increased fatigue life. J Biomed Mater Res 1992; 26: 651-662

Jassem HA, Retief DH, Jamison HC. Tensile and shear strengths of bonded and rebonded orthodontic attachments. Am J Orthod 1981; 79: 661-668

Kao EC, Eliades T, Rezvan E, Johnston WM. Torsional bond strength and failure pattern of ceramic brackets bonded to composite resin laminate veneers. Eur J Orthod 1995; 17: 533-540

Katona TR. A comparison of the stresses developed in tension, shear peel, and torsion strength testing of direct bonded orthodontic brackets. Am J Orthod Dentofacial Orthop 1997; 112: 244-251

Katona TR, Long RW. Effect of loading mode on bond strength of orthodontic brackets bonded with 2 systems. Am J Orthod Dentofacial Orthop 2006; 129: 60-64

Keizer S, Ten Cate JM, Arends J. Direct bonding of orthodontic brackets. Am J Orthod 1976; 69: 318-327

Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthet Dent 2002; 87: 642-649

Lindemuth JS, Hagge MS. Effect of universal testing machine crosshead speed on the shear bond strength and bonding failure mode of composite resin to enamel and dentin. Mil Med 2000; 165: 742-746

Liu JK, Chuang SF, Chang CY, Pan YJ. Comparison of initial shear bond strengths of plastic and metal brackets. Eur J Orthod 2004; 26: 531-534

Lohbauer U, von der Horst T, Frankenberger R, Kramer N, Petschelt A. Flexural fatigue behavior of resin composite dental restoratives. Dent Mater 2003; 19: 435-440

Lohbauer U, Rahiotis C, Kramer N, Petschelt A, Eliades G. The effect of different light-curing units on fatigue behavior and degree of conversion of a resin composite. Dent Mater 2005; 21: 608-615

Lopez JI. Retentive shear strengths of various bonding attachment bases. Am J Orthod 1980; 77: 669-678

MacColl GA, Rossouw PE, Titley KC, Yamin C. The relationship between bond strength and orthodontic bracket base surface area with conventional and microetched foil-mesh bases. Am J Orthod Dentofacial Orthop 1998; 113: 276-281

Maijer R, Smith DC. Variables influencing the bond strength of metal orthodontic bracket bases. Am J Orthod 1981; 79: 20-34

Matasa CG. Adhesion and its ten commandments. Am J Orthod Dentofacial Orthop 1989; 95: 355-356

McCabe JF, Carrick TE, Chadwick RG, Walls AW. Alternative approaches to evaluating the fatigue characteristics of materials. Dent Mater 1990; 6: 24-28

Millett D, McCabe JF, Gordon PH. The role of sandblasting on the retention of metallic brackets applied with glass ionomer cement. Br J Orthod 1993; 20: 117-122

Millett DT, Letters S, Roger E, Cummings A, Love J. Bonded molar tubes — an in vitro evaluation. Angle Orthod 2001; 71: 380-385

Montes GG, Draughn RA. Slow crack propagation in composite restorative materials. J Biomed Mater Res 1987; 21: 629-642

Moseley HC, Horrocks EN, Pearson GJ, Davies EH. Effects of cyclic stressing on attachment bond strength using glass ionomer cement and composite resin. Br J Orthod 1995; 22: 23-27

Murray SD, Hobson RS. Comparison of in vivo and in vitro shear bond strength. Am J Orthod Dentofacial Orthop 2003; 123: 2-9

NDT Resource Center, 2001: Introduction to Materials and Processes. http://www.ndt-ed.org/EducationResources/CommunityCollege/Materials/cc_mat_index.htm (Access date: 30th March 2008)

Newman GV. Epoxy adhesives for orthodontic attachments: progress report. Am J Orthod 1965; 51: 901-912

Oilo G. Biodegradation of dental composites/glass-ionomer cements. Adv Dent Res 1992; 6: 50-54

Oliver RG. The effect of different methods of bracket removal on the amount of residual adhesive. Am J Orthod Dentofacial Orthop 1988; 93: 196-200

Proffit WR, Fields HW. Occlusal forces in normal- and long-face children. J Dent Res 1983; 62: 571-574

Proffit WR, Fields HW, Nixon WL. Occlusal forces in normal- and long-face adults. J Dent Res 1983; 62: 566-570

Reid CN, Fisher J, Jacobsen PH. Fatigue and wear of dental materials. J Dent 1990; 18: 209-215

Reifsnider K. Fatigue Behavior of Composite-Materials. International Journal of Fracture 1980; 16: 563-583

Retief DH. Failure at the dental adhesive-etched enamel interface. J Oral Rehabil 1974; 1: 265-284

Reynolds IR, von Fraunhofer JA. Direct bonding of orthodontic attachments to teeth: the relation of adhesive bond strength to gauze mesh size. Br J Orthod 1976; 3: 91-95

Ruse ND, Shew R, Feduik D. In vitro fatigue testing of a dental bonding system on enamel. J Biomed Mater Res 1995; 29: 411-415

Saunders WP. The effect of fatigue impact forces upon the retention of various designs of resin-retained bridgework. Dent Mater 1987; 3: 85-89

Saunders WP. Effect of fatigue upon the interfacial bond strength of repaired composite resins. J Dent 1990; 18: 158-162

Scherrer SS, Wiskott AH, Coto-Hunziker V, Belser UC. Monotonic flexure and fatigue strength of composites for provisional and definitive restorations. J Prosthet Dent 2003; 89: 579-588

Sernetz F, Binder F. Improvement of bond strength of orthodontic titanium brackets and tubes by laser structuring. Proceedings of the 5th International Conference on Joining Ceramics, Glass and Metal; 1997 May 12-14; Jena, Germany. DVS Berichte 1997; 184: 82-85

Sharma-Sayal SK, Rossouw PE, Kulkarni GV, Titley KC. The influence of orthodontic bracket base design on shear bond strength. Am J Orthod Dentofacial Orthop 2003; 124: 74-82

Shawn MK, 1997: Fatigue
http://www.sv.vt.edu/classes/MSE2094_NoteBook/97ClassProj/anal/kelly/fatigue.html
(Access date: 30th March 2008)

Silverstone LM. The acid etch technique: in vitro studies with special reference to the enamel surface and the enamel-resin interface. In: LM Silverstone and IL Dogan, editors. Proceedings of an international symposium on the acid etch technique. St. Paul, Minnesota: North Central Publishing Company Ltd., 1975: 13-39

Soderquist SA, Drummond JL, Evans CA. Bond strength evaluation of ceramic and stainless steel bracket bases subjected to cyclic tensile loading. Am J Orthod Dentofacial Orthop 2006; 129: 175-175

Sorel O, el Alam R, Chagneau F, Cathelineau G. [Changes in the enamel after in vitro debonding of brackets bonded with a modified glass ionomer cement]. Orthod Fr 2000; 71: 155-163

Sorel O, El Alam R, Chagneau F, Cathelineau G. Comparison of bond strength between simple foil mesh and laser-structured base retention brackets. Am J Orthod Dentofacial Orthop 2002; 122: 260-266

Spanoudakis J, Young RJ. Crack-Propagation in a Glass Particle-Filled Epoxy-Resin. 2 Effect of Particle Matrix Adhesion. Journal of Materials Science 1984; 19: 487-496

Staninec M, Nguyen H, Kim P, Marshall GW, Rithchie RO, Marshall SJ. Four-point bending evaluation of dentin-composite interfaces with various stresses. Med Oral Patol Oral Cir Bucal 2008; 13: E81-E84

Suresh S. Fatigue of Materials. New York: Cambridge University Press, 1998

Tukey JW. Box-and-Whisker Plots. In: Explanatory Data Analysis. Reading, MA: Addison-Wesley, 1977: 39-43

van Noort R, Noroozi S, Howard IC, Cardew G. A critique of bond strength measurements. J Dent 1989; 17: 61-67

Wendl B, Droschl H. A comparative in vitro study of the strength of directly bonded brackets using different curing techniques. Eur J Orthod 2004; 26: 535-544

White LW. Glass ionomer cement. J Clin Orthod 1986; 20: 387-391

Willems G, Carels CE, Verbeke G. In vitro peel/shear bond strength evaluation of orthodontic bracket base design. J Dent 1997; 25: 271-278

Williamson RT, Mitchell RJ, Breeding LC. The effect of fatigue on the shear bond strength of resin bonded to porcelain. J Prosthodont 1993; 2: 115-119

Wilson AD, Kent BE. A new translucent cement for dentistry. The glass ionomer cement. Br Dent J 1972; 132: 133-135

Wilson AD, McLean JW. Glass-Ionomer Cement. Chicago: Quintessence Publishing Company Inc., 1988

Wiskott HW, Belser UC, Scherrer SS. The effect of film thickness and surface texture on the resistance of cemented extracoronal restorations to lateral fatigue loading. Int J Prosthodont 1999; 12: 255-262

Wöhler A. Versuche über die Festigkeit der Eisenbahnwagenachsen. Zeitschrift für Bauwesen 1860; 10:

Yamaguchi K, Miyazaki M, Takamizawa T, Tsubota K, Rikuta A. Influence of crosshead speed on micro-tensile bond strength of two-step adhesive systems. Dent Mater 2006; 22: 420-425

Zardiackas LD, Caldwell DJ, Caughman WF, Lentz DL, Comer RW. Tensile fatigue of resin cements to etched metal and enamel. Dent Mater 1988; 4: 163-168

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