UNIVERSITY of the
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The influence of mixing ratio on the fatigue behaviour of fibre reinforced polymers

Martin Ernst Stuhlinger

A thesis submitted in fulfillment of the requirements for the degree of Magister Scientiae in the Faculty of Dentistry, University of the Western Cape.

Supervisor: Prof Greta Geerts

DECLARATION

I, Martin Stuhlinger, declare that

'The influence of mixing ratio on the fatigue behaviour of fibre reinforced polymers'

is my own work and that it has not been submitted before for any degree or examination in any other university.

Signed: ..... ……..

Martin Ernst Stuhlinger Date

SUMMARY

Statement of the problem: Fibre reinforcement of polymethyl methacrylate (PMMA) denture base material is known to improve the strength, as well as the fatigue behavior, of the material. The powder liquid (P/L) ratio of PMMA is often changed to modify the handling properties of the material. Little is known about the effect of this deviation from manufacturer's guidelines on the fatigue behaviour of the fibre reinforced product.

Purpose: This study compared the flexural strength (FS) of PMMA reinforced with glass fibre using different P/L ratios, before and after cyclic loading.

Methods and materials: Three groups, with 50 glass fibre reinforced (everStick non-impregnated fibers) heat-cured PMMA resin (Vertex Rapid Simplified) specimens each, were prepared using a custom-made template (dimensions 10x9x50mm). Each group had a different P/L ratio: the control group (100%) had the manufacturer's recommended ratio; the 90% and 80% groups had reduced P/L ratios (by weight).Twenty five specimens from each group were subjected to a 3-point bending compression test using a universal testing machine. The remaining 25 specimens from each group were subjected to cyclic loading (10^4 cycles) before compression testing. The (FS) was calculated using the highest force (Fmax) before specimen failure. Flexural strength was calculated using the equation: $FS= 3WL/2bd^2$.

Within each group, median FS values before and after cyclic loading were compared by means of a non-parametric analysis of variance. The Aligned Ranks Transform method was used for the analysis. Statistical significance was set at p=0.05.

Results: The Fmax (N) of the control (100%), 90% and 80% groups fatigued and un-fatigued were 100%: 1665 (fat), 1465 (unfat); 90%: 1679 (fat), 1548 (unfat) and 80%: 1585 (fat), 1467 (unfit) respectively. There was no significant interaction between Mix ratio and Fatigue state, and the 80% mix had a significantly higher mean than either the 90% or 100% mix (with differences of about 0.3 units for both). The Fatigued state had a higher mean than the Un- fatigued state by about 6.0 units. Using FS (MPa) it was found that the fatigued 80% mix specimens had the highest value. The FS MPa of the control (100%), 90% and 80% groups fatigued and un-fatigued were 64.3, 60.6; 66.9, 65.6 and

70.2, 69.3 respectively. The fact that fatiguing strengthened the specimens merits further research.

When observing the broken specimens it was found that there was a complete debonding of the fibres and the PMMA.

Conclusion and clinical relevance:

a) Fibre: The benefit of using glass fibre bundles to reinforce prostheses fabricated using heat cured PMMA is questionable due to problems with bonding between the fibre bundles and the heat cured PMMA resin.

b) Fatiguing: An average person chews 10^7 times during a 3 year period. A limited period of average masticatory forces should not have a detrimental effect on prostheses made from heat cured PMMA resin.

c) Mix ratio: Within the normal parameters of laboratory techniques the mix ratio of PMMA resin had no significance on the fracture resistance of the prostheses.

Due to the high cost of the fibres used for the reinforcement and the limited success and insignificant results achieved in this study, this researcher cannot recommend using Stickbond or Stick fibers for the reinforcement of dentures made with heat cured PMMA resin.

KEYWORDS

Powder : Liquid ratio
Polymethyl methacrylate resin
Fibre reinforcement
Cyclic loading
Fatigue behaviour
Flexural strength

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DEDICATION

I dedicate this study to my loving mother

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ABBREVIATIONS

CSIR	-	council for scientific and industrial research
FS	-	flexural strength
FSu	-	ultimate flexural strength
FSp_I	-	flexural strength at proportional limit
FMax	-	maximum load before fracture
Fbreak	-	force registered at fracture
MPa	-	mega pascal
PMMA	-	polymethyl methacrylate
P/L	-	powder to liquid
RD	-	removable dentures
SEM	-	scanning electron microscopy
N	-	newton
Hz	-	herz
HDLPE	-	highly drawn linear polyethylene
thicklr	-	thickness, length and height

CHAPTER ONE

Literature review

1.1 Introduction

One of the most widely used materials in prosthetic dentistry is polymethyl methacrylate (PMMA) resin. It is the material of choice for manufacturing bases for removable dentures (RD). The main problem of denture base resin is its low impact strength and low fatigue resistance (Gutteridge, 1988). Denture base resins sometimes crack or break following prolonged chewing or if accidentally dropped or mechanically violated.

When dentures are subjected to masticatory forces, these forces may be high or low, sustained or intermittent, and this may take place over a prolonged time. This can lead to failure of the denture. In instances where high forces are anticipated or where the strength of the denture base is compromised, it may be advantageous to enhance the strength of the denture base.

Different approaches exist to increase the strength of denture base polymers, for example the adding of cross-linking agents to the mixture, the incorporation of rubber or fibres, or the use of metal wires or mesh. Due to bulk and colour, the use of metal as reinforcement material is limited.

The introduction of fibres as reinforcing agents addressed several problems associated with the use of metal as reinforcement. The advantages of using fibres include: chemical bond between fibre and polymer matrix, a neutral colour, flexibility, ease of adaptation to different shapes before polymerization and ease of repair. The most popular fibres in dentistry are polyethylene- and glass fibres. Both have been demonstrated to improve the physical properties of materials used for RDs (Hamza *et al.,* 2004).

PMMA resins used for dentures are usually available as a powder polymer and a liquid monomer. These are mixed in a certain ratio and then cured using different polymerization protocols depending on the type of resin used. Only a few studies (Syme *et al.*, 2001,

Geerts & du Rand, 2009) have been performed on the effects of changing powder to liquid (P/L) ratios of PMMA resins on the properties of these resins.

Clinicians and laboratory staff do not always follow manufacturers' recommended P/L ratios. The P/L ratio of PMMA is often changed to modify the handling properties of the material in order to achieve a certain consistency or to influence working time. Little is known about the effect of this deviation from manufacturer's guidelines on the fatigue behaviour of the fibre reinforced product.

The normal use of a denture involves repeated episodes of stress and relaxation, also called cyclic loading. Applying a cyclic load to a material leads to fatigue. The issue of fatigue in fibre reinforced PMMA resin has not been well examined or documented.

Fatigue refers to the fact that after cyclic loading a material will undergo failure at a lower applied stress than if it were not subjected to cyclic loading. The name "*fatigue"* is derived from the fact that a material seems to tire under this type of repetitive loading. The alternating stress application for fatigue testing should be below the proportional limit and should not exceed the proportional limit at any time during the test (Craig, 2002). Two ways are commonly used to discuss fatigue: *endurance loading* and *service lifetime* (Budynas, 1999; Askeland and Pradeep, 2003).

Endurance limit is the maximum applied stress that a material can withstand and still have an unlimited number of cycles to failure (Hibbeler, 2003; Dowling, 1998; Barber, 2001).

Service lifetime describes a way of predicting the number of cycles to failure a material can be expected to undergo prior to failure when it is loaded with a specific force (Bathias, 1999).

The rapid pace of advancement in dental materials' science sees some products come and go in a relatively short space of time. This fast turn-over may be as a result of quality control, research and/or design shortcomings. It is a challenge for the dental clinician to keep abreast of technological advancements and to become familiar with new materials and methods, some of which actually show no advantages over existing technology (Eliades, 2006).

1.2 Polymethyl methacrylate

PMMA is a popular base material for RDs. It is easy to work with, can be modified or repaired easily, is cheap and comes in different colours matching the colours of the oral tissues. However, the material is at risk from both impact and fatigue failure. Flexure fatigue and impact force are essentially the two forces that lead to denture failures (Jagger *et al.,* 1999). Denture base resins may crack or break if being accidentally dropped or after prolonged chewing stresses (Seo *et al.,* 2006).

According to Johnson and Matthews (1949) a person bites, on average, 500 000 times a year. The majority of denture fractures occur by the end of 3 years in service (Franklin *et al.,* 2005). Goguta (2012) found similar results with standard dentures breaking within 3 to 4 years of delivery. However, in his study, dentures that were reinforced with woven e-glass fibre outlasted the 5 year test period.

The following clinical factors were identified as enhancing the risk for mechanical failure (Farmer, 1983): improperly contoured mandibular occlusal plane, high frenum attachments, occlusal scheme, occlusal forces, denture foundation and denture base thickness. The thickness of a denture base, for example, may be compromised following the application of a long-term soft liner. Occlusal disharmonies, overload, incorrect handling and fatigue are some of the other common occurrences reported by Bertassoni *et al.* (2008). A pronounced incisal notch in an upper denture has proved to be an area where fracture and crack propagation can start. Twenty nine percent of all denture fractures were found to be upper mid-line fractures (Cheng *et al.,* 2010). Masticatory forces may be particularly concentrated and high in some areas as in the case where 1 or 2 teeth are replaced with acrylic pontics, or with parafunctional habits, flabby ridges, ill-fitting dentures and dentures opposing natural teeth (Dogan *et al.,* 2006).

When high forces are anticipated or if the strength of the denture base is compromised, it may be advantageous to enhance the strength of the denture base. An acrylic resin that can withstand high static and dynamic loading should prove to be less prone to clinical failure (Diaz-Arnold *et al.,* 2008). Popular methods for improving the strength of denture bases are the incorporation of cross-linking agents, metal wire or mesh, and, more recently, different types of fibres presented in uni- or multidirectional configurations.

1.3 Fibre reinforcement of polymethyl methacrylate resin

Before the introduction of fibres, metal wire and mesh were the most common methods of reinforcing (Carroll and von Frauenhofer, 1984; Ruffino, 1985; Vallittu and Lassila 1992a). High-strength metal increases the flexural strength and impact strength slightly but its application is limited because of colour and bulk. A reason for the limited strength-enhancing property of metal is the fact that there is no chemical bond to resins (Vallittu, 1993, 1996). Silanating and roughening of the metal does help somewhat with this problem (Vallittu, 1993).

Since the early 70's, the use of fibres to reinforce dental materials has been researched and marketed (Galan and Lynch, 1989). Several types of fibres are used for re-inforcement, including polyethylene fibres, aramid fibres, carbon fibres or glass fibres (Fajardo *et al.*, 2011). Already in 1982, Skirvin *et al.* reported that chopped carbon fibres increased the fatigue resistance of denture resins by between 16 and 83 %.

Compared to metal-reinforcements, fibres have several advantages: most have a neutral colour; they are flexible and easily adapted to different shapes before polymerization; they make repairs easy; and, last but not least, manufacturers claim chemical bond between fibre and polymer matrix (Vallittu, 1999).

For the improvement of the physical properties of fibre-resin composites, several parameters should be considered and possibly manipulated. Selection of matrix, selection of fibre type, fibre thickness, content of fibre by volume or by weight, distribution of the fibre, dimension, impregnation with resin, selection and use of different silane agents and techniques and conditions of construction should be carefully considered (Freilich *et al.*, 1998). The most popular fibres in dentistry are polyethylene and glass fibres. Both have been demonstrated to improve the physical properties of materials used for RDs, particularly the auto-, heat- and light-cure PMMA materials (Narva *et al.*, 2005a; Du Randt, 2008, thesis).

Manley (1980) found that the resistance to *fatigue failure* of heat-polymerized PMMA reinforced with carbon fibre was higher than a similar un-reinforced conventional denture base polymer. Skirvin *et al.* (1982) concurred and found that reinforcement of three different denture resins (cold and heat polymerized) with carbon fibres increased the *fatigue resistance* by up to 83 percent. Nohrstrom *et al.* (2000) showed that the effect of

glass fibre reinforcement of interim cold polymerizing acrylic resin fixed partial dentures became more evident with long span bridges. According to Hamza *et al.* (2004) the addition of fibres (glass fibres and polyethylene fibres) to provisional autopolymerising resin (polymethyl methacrylate, polyethyl methacrylate and bisacryl) increased both *fracture toughness* and *flexural strength*. They also found that the location of the fibre within the fixed partial dentures was important: the positioning of the reinforcement at the tension side increased the fracture resistance more than if it were placed at the compression side of the prosthesis. These results confirm those of Vallittu (1998) who did, however, find that even when the glass fibre reinforcements were positioned on the least favourable side of the prosthesis they still improved the strength and flexure resistance. Kanie *et al.* (2000) found that the impact strength of denture base polymer reinforced with woven glass fibres was significantly higher than unreinforced polymer. This was in agreement with the results of Uzun *et al.* (1999).

Narva *et al.* (2001) suggested that the correct positioning and the correct laboratory techniques were important to maximise the benefits of glass fibre reinforcement. Fibre reinforcements placed on the tensile side resulted in considerably higher flexural strength and flexural modulus values compared with the same quantity of fibres placed on the compression side (Narva *et al.*, 2005b). They also concluded that impregnated and pre-impregnated fibres reinforced denture base polymer more than non-impregnated fibres.

Fibres are available in unidirectional or multidirectional configurations. Woven fibres are thicker, and provide better flexural strength characteristics because of their multidirectional configuration (Vallittu, 1998).

Fibre strengtheners are available as chopped fibre pieces, longitudinal fibre bundles or as woven fibre mesh. Sometimes the longitudinal fibre bundles have the individual fibres running unidirectionally while other longitudinal bundles are woven or knitted and thus have individual fibres running in multidirectional configurations. Woven fibre bundles are thicker, and provide better strengthening characteristics because of their multidirectional configuration (Chow *et al.*, 1992; Karbhari, 2007). While unidirectional, longitudinal fibres give maximal reinforcement against one force or direction of load, their strengthening effect is much weaker against forces coming from other directions (Vallittu, 1998). In these cases, where the forces come from another direction, the fibres themselves don't break, the matrix surrounding the fibre bundle tears apart causing failure or fracture

(Vallittu, 1999). Thus, for optimal reinforcement the fibres must be placed at a 90-degree angle to the anticipated fracture line (Vallittu, 1999). Fibres provide strength only when they are stretched and thus engage with the force applied. When compressed or squashed, fibres give hardly any resistance to the force applied (Vallittu, 1999). Unidirectional fibres have special mechanical properties. These can be likened to the wood fibres running along the grain of a straight, tall tree. Unidirectional fibres have proved stronger in that one direction than multidirectional types of fibres although without the same flexural strength (Freilich *et al.,* 1997; Kostoulas, 2008). Minami *et al.* (2005) found no significant improvement by using woven fibre mesh to increase the load to fracture values of flexural specimens after thermo-cycling. In another study, Kanie *et al.* (2006) found that woven glass fibre mesh reinforcement of composites did afford considerable improvement even in compressive force situations which longitudinal fibres do not. The strength of the reinforced structure is also dependant on the volume of the fibres embedded in the PMMA matrix and the degree of adhesion between the fibre and the polymer. The higher the fibre content, and the better the adhesion, the better the strengthening characteristics will be (Vallittu *et al.,*1994).

Besides the fibre direction, the strength of the reinforced structure is also dependent on the volume of the fibres embedded in the PMMA matrix. The higher the fibre content, the better the strengthening characteristics are (Vallittu, 1994; Marei, 1999; Taner *et al.,* 1999).

1.4 Adhesion of fibres to resin

Over the last 30 years not only the dental field, but also the aeronautical, civil engineering and automotive industries have used fibre reinforcement. Fibres, mostly made of polyethylene, carbon/graphite or glass have produced equipment with improved mechanical properties. However, good bonding between the acrylic resin and the reinforcement is crucial for a significant stiffening effect. This bond is called adhesion and is defined as: ‘the molecular attraction exerted between the surfaces of bodies of dissimilar materials in contact’ (Von Fraunhofer, 2012). The adhesive bond will fail if the adhesive separates from the substrate or if there is internal breakdown of the adhesive itself (Von Fraunhofer, 2012).

Adhesion is influenced by contact angle and surface tension of adhesive and substrate: The larger the contact area between the materials, the better the adhesion. Contact area will be

increased if one or other of the materials has the ability to ‘wet’ the surface interface. This ‘wetting’ is the ability of a liquid to form an interface with a solid surface (Von Fraunhofer, 2012).

Mechanisms of adhesion can be listed as follows:

a. Chemical adhesion. If the adhesive and the substrate form a compound at their interface or union, the ionic or covalent bonds result in a strong bond between the materials (Von Fraunhofer, 2012).

b. Dispersive adhesion. In dispersive adhesion the surfaces of two materials are held together by Van Der Waal’s forces. These are the attractive forces between two molecules. The effectiveness of adhesion due to dispersive bonding is limited (Von Fraunhofer, 2012).

c. Diffusive adhesion. Some materials may merge or intermingle at the bonding interface by diffusion, typically when the molecules of both materials are mobile and/or soluble in each other. This is the action that takes place when a resilient denture liner is processed onto a denture base or when a denture base is repaired (Von Fraunhofer, 2012).

d. Mechanical adhesion. This occurs when uncured adhesives are fluid and they can flow over the substrate, filling the voids, rugosity and pores of the surface and attach or ‘bond’ to that surface by mechanical interlocking. Micromechanical adhesion probably contributes significantly to bonding achieved with resin-based adhesives (Von Fraunhofer, 2012).

It follows that when two materials are bonded there is often a modified molecular structure at the bonding interface. This is called the ‘adhesion zone’ (Von Fraunhofer 2012).

The better the degree of adhesion between the fibre and the polymer, the better the strengthening characteristics: any inability to adequately impregnate fibres with polymer and monomer mixtures of high viscosity, such as PMMA, represents a significant disadvantage to the use of fibres as reinforcement for dentures (Vallittu, 1999; Bertassoni *et al.,* 2008). To overcome this difficulty, the fibres can be impregnated with a more viscous resin mixture that has similar characteristics to those used in the restorative resin of choice. Such pre-impregnation will allow for good bonding with the less viscous PMMA

and thus improve the adhesion and overall strength of the reinforced product. Silanation of fibres (Ozdemir, 2003) and urethane oligomers (Kanie *et al.*, 2004) have also both proved successful as a method to promote better adhesion of fibres to resin. These findings agree with Vallittu (1997) who earlier found considerable value in pre-impregnating fibres with silane and polymerizing the fibres and silane together. However, Kanie *et al.* (2000) found in his experiments that the silane did not make any difference and that bonding between glass fibre and polymer matrix depended on mechanical retention by polymerization shrinkage and roughness. Dogan *et al.* (2006) analyzed specimens reinforced by glass-fibres by means of scanning electron microscopy and found the same to be true for glass fibre reinforcement specimens in their study. In both these cases the polymer matrix might have been too viscous for adequate mechanical adhesion to have taken place.

PMMA is most often available in powder and liquid form. A specific mixing ratio is recommended by the manufacturers, but practitioners often deviate from this in an effort to change handling properties or because of not using the correct measuring tools. The effect of changing this P/L mixing ratio on the adhesion of the PMMA to the pre-impregnated fibre-bundles is not well-known.

1.5 Powder-liquid ratios of PMMA resin

Clinicians and laboratory staff do not always follow manufacturers' recommended P/L ratios when mixing PMMA. The ratio is sometimes changed in order to achieve a certain consistency or to influence working time. Little is known about the effect of this deviation from manufacturers' guidelines on the strength of the PMMA material, particularly when it is reinforced with fibres.

Only a few studies have been performed on the effects of changing P/L ratios of PMMA resins. Jerolimov *et al.* (1989) did various tests with heat-cured PMMA resin and found that changing the P/L ratio made no difference to the impact resistance and dimensional accuracy of the acrylic resin. They did, however, recommend that the heat polymerization cycle should include a temperature above boiling water as this significantly reduced the free residual monomer. On the other hand, Williams *et al.* (2001) found that changing P/L ratio of four auto-polymerizing PMMA resins may have deleterious effects on the properties of the polymerised material: A lower P/L ratio resulted in significantly lower surface hardness and higher flexibility.

A higher liquid content in the mixture increases polymerisation shrinkage (Vallittu, 1994). For fibre reinforced resin, polymerisation shrinkage might cause slits between the fibre and the polymer matrix, reducing adhesion between the two components and ultimately the strengthening effect (Vallittu, 1999). During thermal, moisture and mechanical processes, these slits may grow inherently, further reducing adhesion between polymer and fibre (Vallittu, 1999). Geerts and Du Randt (2009) found that when using an autopolymerizing resin the flexural strength of a glass fibre reinforced PMMA resin was significantly higher when using the manufacturers recommended mixing instructions. When no reinforcing was used, the mixing ratio did not influence the flexural strength.

1.6 Flexural strength

Flexural strength (FS) is defined as the resistance of a material to being broken by bending stresses (Diaz-Arnold *et al.*, 2008). The ultimate flexural strength (FSu) is sometimes called catastrophic failure (Diaz-Arnold *et al.*, 2008). Also of significance is the flexural strength at the proportional limit (FSpl) that reflects the resistance to plastic deformation (Takahashi *et al.*, 1998). Once plastic deformation has occurred the functional ability of the prosthesis is compromised, although it has maybe not fractured completely.

Different laboratory tests such as the 3-point flexure test (ISO1567) exist to quantify static flexural strength. This test is sometimes also called the 3-point bend test.

The fracture force is recorded in Newton (N). The FSu and the FSpl of each specimen is then calculated in megapascal (MPa) using the formula (ISO 1567):

$$FS = 3FMaxL/2bd^2$$

Where FS is the flexural strength, FMax the maximum load before fracture (for FSu) or at the proportional limit (for FSpl), L the distance between the supports (mm), b the width of the specimen (mm), and d the height of the specimen (mm).

Mechanical properties of denture acrylic resins are important for the clinical success of multiple types of prostheses. Acrylic resins must be strong and resilient to withstand repeated impact. The ultimate flexure strength of an acrylic reflects its potential to resist catastrophic failure under a repeated flexural load. An acrylic resin capable of sustaining high flexure in combination with high resistance to cyclic loading may be less prone to clinical failure (Diaz-Arnold *et al.*, 2008). Flexure strength of acrylic polymers can be

manipulated by the addition of strengtheners and changing of mix-ratios of the ingredients and of the ingredient formulae (Dogan *et al.*, 2006, Hargreaves 1983).

1.7 Bite Force

To simulate clinical mastication forces for *in vitro* tests, information on the range of occlusal loading during function should be known. Bite force measurements are difficult and the results depend on a number of factors, such as gender, age, craniofacial morphology, occlusal factors, the presence of pain and temporomandibular disorders (Koc *et al.*, 2010). Kiliaridis *et al.* (1993) showed that the maximum bite force in the molar region increased with an increase in age from 7 to 24 years. They reported that maximal occlusal forces in the molar region can be as high as 900N in young adults. The clenching force in a Thai study of 30 individuals ranged from a maximum of 815 N to a minimum of 125 N (Supputmongkol *et al.*, 2008). In a periodontal study of 194 patients the occlusal forces were considered as 'high' above 500N for men and 370N for women (Takeuchi *et al.*, 2010). A Brazilian study found that the bite force of the very old and very young was lower than the median age group (Palinkas *et al.*, 2010). Men have significantly higher bite forces than women. A world-wide survey, including the mentioned Brazilian subjects, proved that, on average, the bite force of a man is 30% higher than that of a woman (Palinkas *et al.*, 2010).

However, normal chewing forces are considerably lower than the 900N mentioned previously. Jain *et al.*, (2013) reported normal chewing forces from 480 to 640 N. The biting forces of a person with removable dentures could be as low as 100-150N (Lassila *et al.*, 1985).

For in vitro simulation, Krejci and Lutz (1990) suggested and used 500-600N (for natural dentition). Researchers at the University of Hong Kong concluded that a force of 230N in the posterior region was a fair average while conducting strain analysis studies on maxillary dentures (Cheng *et al.*, 2010).

Svenson and Trulsson (2009) found that, as higher bite forces are needed to split a morsel of food, the duration as well as the intensity and rate of the biting force will increase. Mechanoreceptors in the periodontium are used to adapt the bite force rate to the hardness of the substance being chewed. However, when the patients were anaesthetised, they lost the ability to adapt the bite force to the type or hardness of the food (Swensson and

Trulsson, 2009). One could speculate that the fact that one loses a lot of the proprioceptive feeling when wearing a partial or full denture, could lead to unnaturally high forces being used to accomplish relatively small biting tasks. In a study comparing maximum bite force values in fully dentate mouths, fixed partial denture mouths, removable partial denture mouths and full denture mouths the opposite was clearly evident. With the natural dentition registering a bite force of 100%, the other forces were 80%, 35%, and 11% respectively (Miyaura *et al.*, 2000).

While information exists on the range of bite forces, static testing does not simulate the dynamic nature of chewing. Flexural strength data alone does not provide enough relevant information for long-term clinical performance, because correlations between monotonic flexure strength and resistance to *fatigue* are weak (Scherrer *et al.*, 2003). Dental resins typically fail as a result of many loading cycles or an accumulation of damage from stress and water. In terms of *in vivo* loading, the masticatory cycle consists of a combination of vertical and lateral forces, putting the ceramic under a variety of off-axis loading forces (Wood *et al.*, 2006).

In a small, but significant effort to better simulate the clinical environment, research protocols on dental materials may include cyclic loading.

1.8 Cyclic loading and fatigue behaviour

The normal use of a denture involves repeated episodes of stress and relaxation. It has been estimated that the average dental prosthesis must withstand more than 10^7 load cycles during an average 3 year functional lifespan (Hargreaves, 1983). Such a repetitive load is called a cyclic load. Applying a cyclic load to a material may lead to fatigue. Few studies were found that use cyclic loading tests to characterize material response to repeated stress (Hargreaves, 1983; Diaz-Arnold *et al.*, 2008).

The name “fatigue” is derived from the fact that materials seem to “tire” under repetitive loading. Fatigue refers to the fact that, after or during cyclic loading, a material will undergo failure at a lower applied stress than it normally would if it were not subjected to cyclic loading (Kelly, 1969; Hargreaves, 1983,). Fatigue often leads to failure of materials because it promotes crack propagation. Surface conditions (roughness and sharp angles) promote fatigue failure (Cheng *et al.*, 2010) as do surface anatomy like deep frenal notches (Vallittu *et al.*,1996). The fatigue process comprises an initial period of nucleation,

followed by crack propagation (Hargreaves, 1983). Fracture mechanics can thus be used to describe fatigue failure.

Fatigue failure is common in ill-fitting dentures, single upper dentures against natural lower teeth and in all dentures with soft liners (Kelly, 1969). Gonda *et al.* (2007) found the interphase between the overdenture and the coping it was resting on to be particularly prone to fatigue. Fatigue failure does not require strong biting forces; a relatively small stress caused by the masticatory system over a sustained period of time can eventually lead to the formation of a small crack, which propagates through the denture, eventually resulting in a fracture (Farmer, 1983). A study of fracture surface characteristics in removable acrylic dentures supports the fatigue failure mechanism as a main causative factor in denture fractures (Vallittu, 1996).

Cyclic loading can be incorporated in the testing method to simulate the clinical environment. Vallittu (2006) describes 'fatigue strength' of a material as the highest stress that a material can withstand for 10^7 loading cycles. Testing specimens at such a high number of cycles poses a challenge in the laboratory milieu. The number of cycles per second must be kept low enough to prevent heat generation in the specimen. Thus, at 2 Hz, 57.8 days are required to fatigue one specimen for 10^7 times. In a review article, Naumann *et al.* (2009) found that a protocol using 10^4 cycles at 50N and 5Hz satisfactorily simulates a year of function in dental materials.

Fatigue tests (cyclic loading) are considered more pertinent than monotonic (3-point bending) tests as to their predictive value (Scherrer *et al.*, 2003). However, stresses generated during chewing, have large ranges of direction and intensity that cannot exactly be simulated by in vitro fatiguing equipment.

Cyclic loading can be done in a dry or wet environment. Because PMMA is subject to water sorption, this may influence its strength. Even a short period of 24 hours in water proved to weaken the flexural strength of a resin: water taken up by the process of diffusion into the acrylic resin acts as a plasticizer, compromising the mechanical strength of the material (Takahashi *et al.*, 1998). Takahashi argued that plasticizers facilitate the movement of polymeric chains under load and thus lower the mechanical properties of the polymer. Their experiments were carried out on resin relined with different reline materials though. It is interesting to note that Reis *et al.* (2006) found the opposite with both the

ultimate flexural strength and the flexural strength at the proportional limit of tested denture base reline acrylic resin. A marked increase was recorded. He postulated that the free residual monomer in the resin reline material acted as a better plasticizer than the water that replaced it in the resin after water storage. This explained the increase in flexural strength of the two materials that exhibited this behaviour. Reis *et al.* (2006) also found that cyclic loading weakened the FSu and the FSpl of all materials tested in their study.

Fatigue behaviour in fibre reinforced heat-polymerized dental PMMA resin has not been well examined and documented (Reis *et al.,* 2006). This was confirmed by exhaustive searches using the Western Cape University E-Library and Pubmed database.

Therefore, it was decided to investigate the fatigue behaviour of glass fibre reinforced heat-polymerized PMMA resin used for dentures.

1.9 Aim, objectives and null-hypotheses

1.9.1 The aim of this study

This study compared the flexural strength (FS) of PMMA reinforced with glass fibre using different P/L ratios, with and without cyclic loading. The results of this research study could assist in a recommendation for the appropriate P/L ratio to be used in order to achieve maximum benefit from the glass fibre reinforcement of PMMA resin bases.

1.9.2 The objectives of this study

1. To establish the FS of fibre reinforced PMMA resin mixed according to 3 different P/L ratios without cyclic loading.

2. To establish the FS of fibre reinforced PMMA resin mixed according to 3 different P/L ratios after cyclic loading.

3. To establish the influence of fatiguing by comparing the FS for the same ratio groups with and without cyclic loading.

1.9.3 Null hypotheses

1. There will be no significant difference in FS among the 3 different P/L ratio groups.

2. There will be no significant difference in FS within each P/L ratio group with and without cyclic loading.

3. There will be no significant difference in FS among the 3 groups after cyclic loading.

CHAPTER TWO

Methods and materials

2.1 Introduction

The research proposal was approved and registered by the University of the Western Cape's Senate Research Committee.

The research design and methodology will be described in the following order: 1. the originally proposed methodology, 2. piloting of the originally proposed methodology, 3. the final methodology.

2.2 Proposed methodology

2.2.1 Study design

This is an *in vitro*, controlled, comparative study.

This research project was designed comprising 3 groups of 50 specimens each. Each group had a different L/P ratio. The group with the manufacturer's recommended L/P ratio acted as the control. Half of the specimens per group were subjected to cyclic loading, the other half not.

The study design is shown in Table 2.1. The arrows in the table indicate the direction of comparisons that were considered relevant for answering the research questions.

	Recommended L/P 2.3g/1ml	10% Lower L/P 2.07g/1ml	20% Lower L/P 1.84g/1ml
Monotonic test	25	25	25
Cyclic loading	25	25	25

Table 2.1: Study design

2.2.2 Materials

As denture base PMMA, Vertex Rapid Simplified (Vertex Dental, Zeist, the Netherlands) was used. Manufacturers' instructions for the use of materials were always followed, except when the P/L ratio of the PMMA was deliberately changed for the purpose of the research.

The manufacturer's specifications and instructions for the handling of the PMMA are shown in Addendum A and B. The recommended ratio for mixing the powder and liquid is as follows: 1ml of monomer liquid / 2.3g polymer powder. This ratio was used to prepare the specimens for the control group (100%). The other 2 groups had a 90% (1ml / 2.07g) and a 80% (1ml / 1.84g) P/L ratio (Table 2.1).

For the fibre reinforcement, pre-impregnated glass fibres (Pre-impregnated everStick C+B fibre by Stick Tech, Turku, Finland) were proposed by the manufacturer.

2.2.3 Manufacturing of the mould

A custom-made steel mould (Figure 2.1) was designed and manufactured to make the specimens. The dimensions and shape of the mould were governed by the following criteria:

- The thickness of a denture base;
- ISO 13003:2003 - Fibre-reinforced plastics: Determination of fatigue properties under cyclic loading conditions;
- The length of the fibres chosen for the study (50mm);
- Ease of removal of the polymerized specimens from the mould without damaging them;
- The shape of the specimen to allow a 3-point bending test and cyclic loading;
- Piloting: A few specimens 10mm wide, 4mm deep and 46mm long were prepared and sent to the Council for Scientific and Industrial Research (CSIR), who were engaged to do the fatigue testing. CSIR found that the fibres did not lie reliably on the tension side of the specimens (Addendum C.1). Due to the random positioning of the fibres the CSIR scientist also found it difficult to calibrate the tests and during telephonic conversation it was decided to change the dimensions of the specimens. Therefore the dimensions of the cavity were changed to: 10mm wide at

the top tapering to 9mm at the base of a depth of 10mm.

After machining and finishing, this would give a specimen of 9mm wide, 10mm deep and 46mm long.

The final mould consisted of the following components:

- Lid no.1 (Figure 2.1). This lid was used for proof-packing the first layer of PMMA. Five raised platforms of 3mm would fit perfectly over each cavity of the base to extrude excess PMMA to exactly 3mm from the surface of the base, providing a flat surface for the positioning of the fibre.
- Lid no.2. This lid had a flat inner surface and was used to close the mould once the fibre was positioned and the cavity in the base was completely filled with the second layer of PMMA.
- The base (Figure 2.2). The base had 5 cavities (10mm deep, 46mm long and 10mm wide tapering to 9mm) to receive the first layer of PMMA. Each cavity had 2 openings in its base. Plugs were machined to fit these openings. These plugs were used to aid in the removal of the specimens, once polymerized. The cavities were slightly diverging towards the surface to ease removal of the polymerized specimens. On each long-end of the cavity, slots (3mm deep, 1 mm long) were machined to standardize the 3-dimensional positioning and stabilization of the fibres.

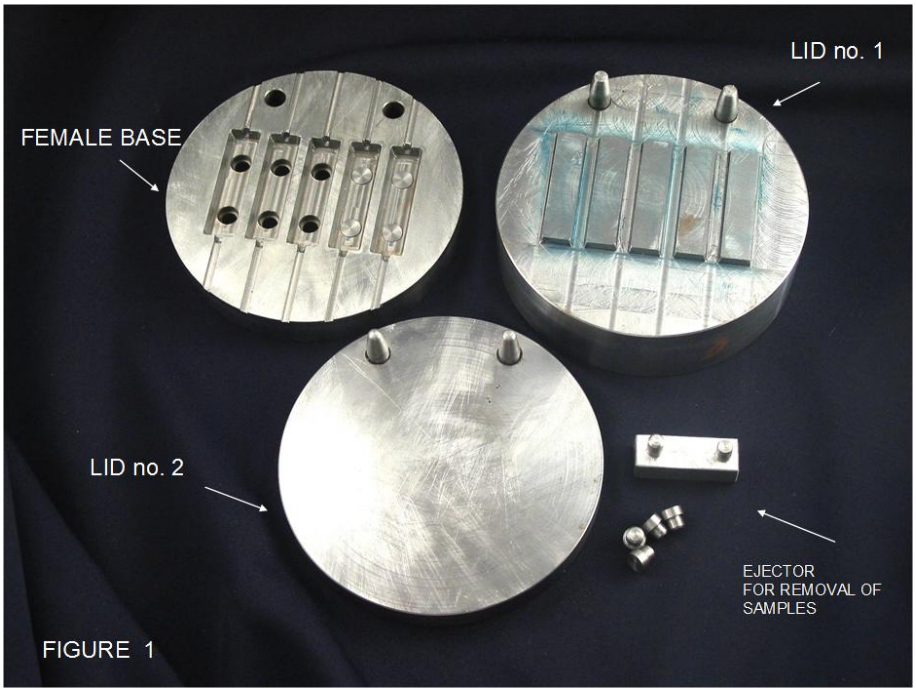

Figure 2.1: Custom made steel template.

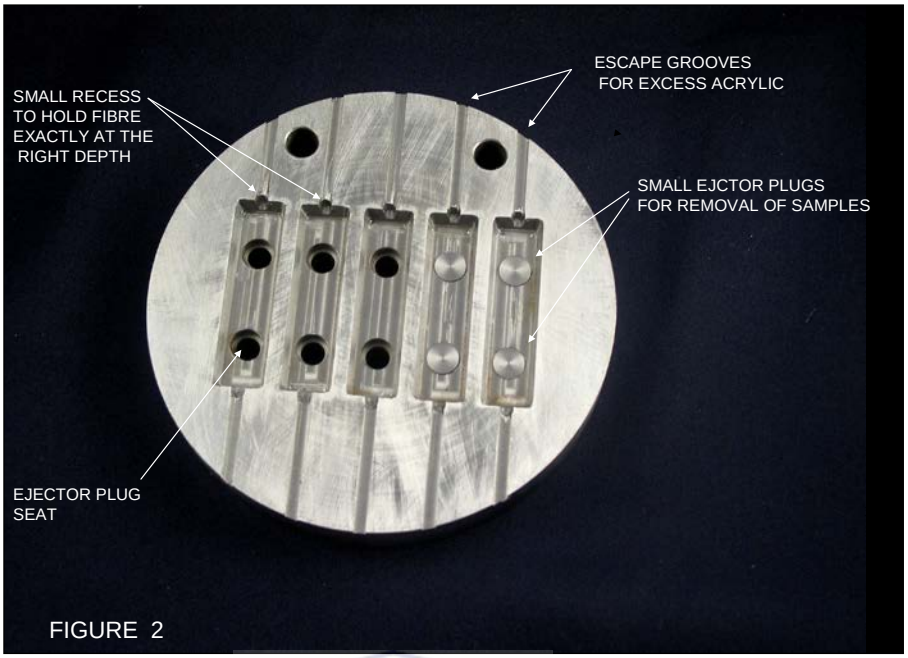

Figure 2.2: Close view of the base of custom made steel mould

2.2.4 Piloting of the manufacturing of the specimens

Manufacturing of the specimens was extensively piloted using metal wires and resin impregnated super-floss as substitutes for the fibres, to practice and refine the correct positioning of the fibre and to familiarize the researcher with the sequence of the manufacturing process.

The research proposal was sent to the manufacturers of the fibres in Finland to ask for possible sponsoring of the fibres. The fibres suggested in the research proposal were 50mm pre-impregnated ever-Stick fibres (Stick Tech). The thicker ever-Stick C+B was suggested by the manufacturer. Consequently, 150 fibres for the complete project were donated by the manufacturers.

After the arrival of the glass fibres from Finland, piloting was again performed with some of these fibres. It was noticed that the fibres were not all of equal length, some being longer, some shorter than the 50mm as they were marketed. This complicated the standardization of the position of the shorter fibres that did not reach the slots at the long-ends of the cavities in the base. For these fibres, an additional method of stabilization of the fibre was designed in the form of a staple-like thin metal wire (Figure 2.3). As the

staples are at the distal ends of the specimens it was accepted that they would have no effect on the flexural strength.

Figure 2.3: "Stapling" the shorter fibres into position.

The longer fibres were cut to exactly 50mm with surgical scissors (Figure 2.4).

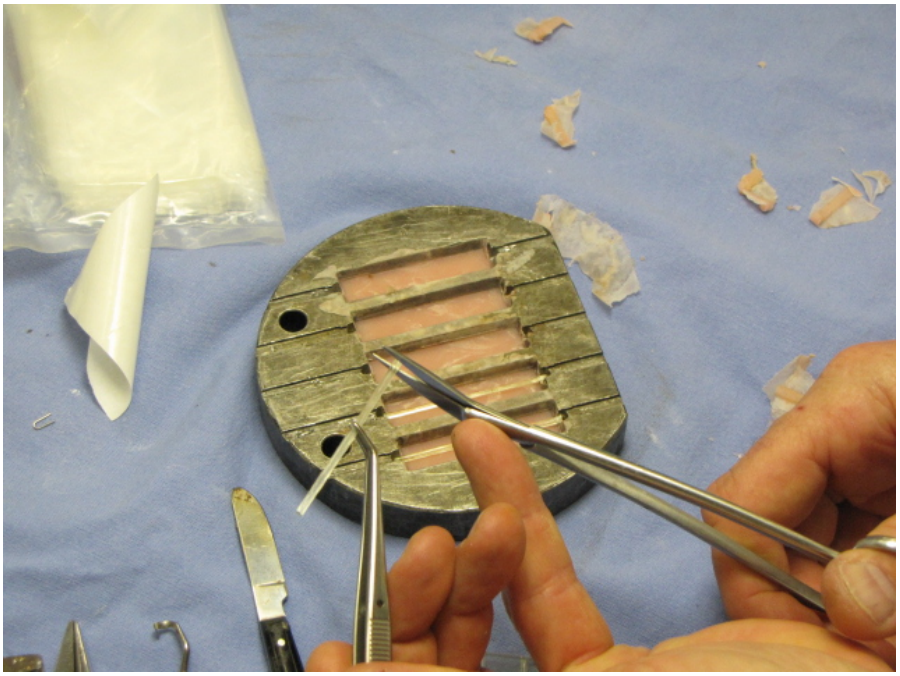

Figure 2.4: Cutting longer fibres to the correct length.

Piloting of the methodology taught the researcher that sets of 5 fibres of the correct length and additional stabilization for the shorter fibres needed to be ready prior to mixing of the PMMA.

2.2.5 Manufacturing of the specimens

Specimens were manufactured in batches of 5 - to fill the 5 cavities in the base - using the same PMMA mixture. Following the ratios as shown in Table 2.1, the polymer powder was weighed using an analytical laboratory balance (Denver Instrument Company, Gottingen, Germany) with an accuracy of 0.0001g. The polymer was weighed in a glass beaker using the taring option on the scale, which subtracted the glass beaker's weight to get the correct weight of the polymer.

The liquid monomer was titrated using a pipette (Finnpipette Digital 1-5 ml, Labsystems, Finland). Manufacturer's instructions demand that the PMMA is mixed and left to reach the dough stage before packing inside the mould. When the PMMA was covered to reach dough stage, the pre-impregnated fibres were polymerized for 2 minutes (Megalight Mini, Radeburg, Germany). The stiff polymerized fibres were placed with their ends positioned in the stops.

The mould was now filled with the 'dough stage' PMMA and lid no.2 was used to close the mould (note: at this stage, the need for lid no.1 was not realized yet. This will be explained in the following paragraph).

Because of the resilience of the resin in the dough stage, the fibres were consistently pushed from their correct position when the mould was closed and pressure was applied (Figure 2.5). A problem such as this has not been published in the literature of similar studies (Dogan *et al.*, 2006; Bertassoni *et al.*, 2008; Fajardo *et al.*, 2011).

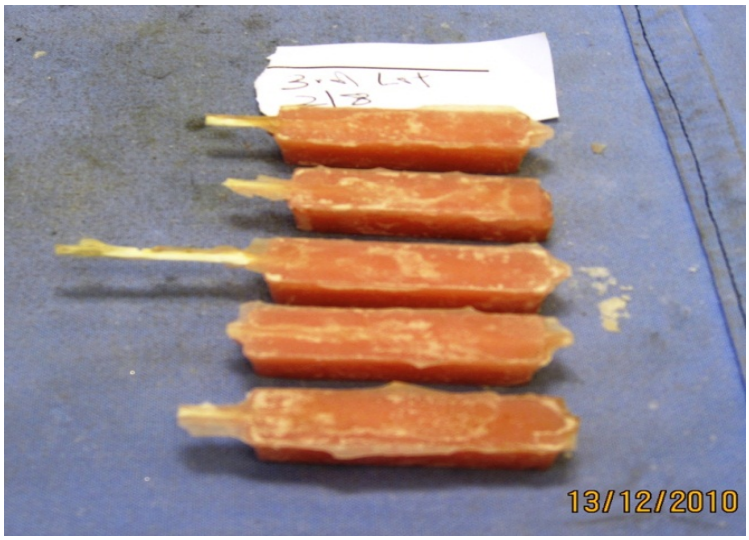

Figure 2.5: Fibres moving during the manufacturing process

The solution would be to manufacture the specimens in 2 stages. The cavities were to be filled with a first layer of PMMA-dough up to the level of the stops on which the ends of the fibre bundles were to be positioned and proof-packed. This proof-packing required an additional cover to be made. The original protocol described a single stage procedure with a flat cover. This additional cover (Lid no.1) was made with platforms that protruded into the cavities of the base up to the level of the stops which were set at 3mm deep (Figure 2.6).

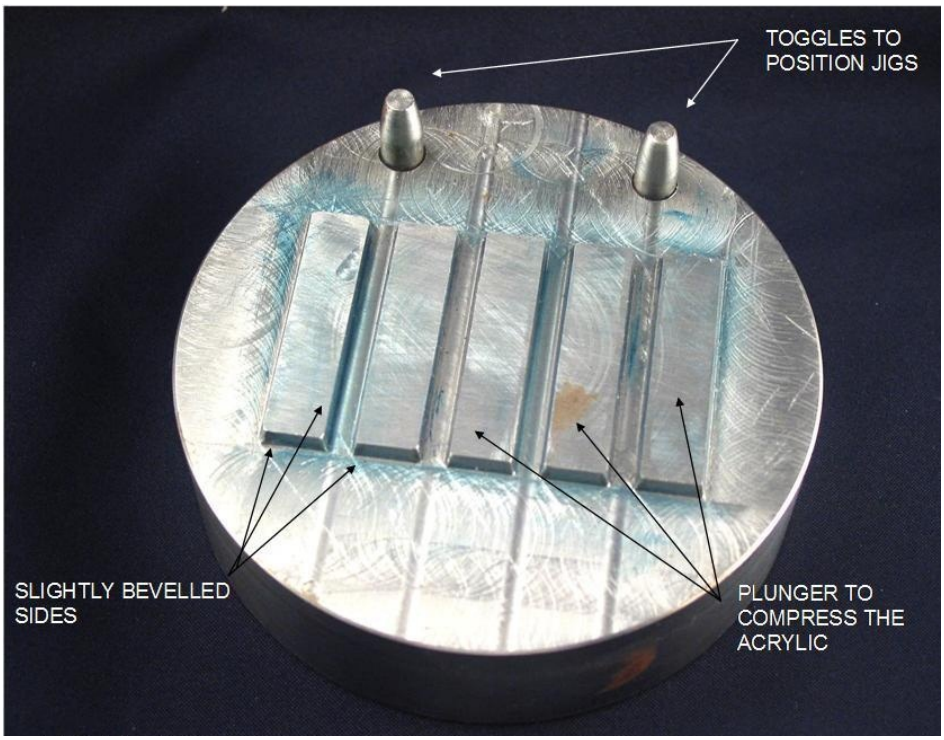

Figure 2.6: Lid no 1 with raised platforms for initial PMMA packing

The base of the mould, with the plugs in position, was filled with the dough and proof-packed with lid no.1. A thin polythene sheet was used as a separating medium to prevent the lid from sticking to the dough. Upon removing of lid 1, each of the 5 cavities had a flat PMMA surface 3mm below the surface of the base and level with the base of the slots at each end of the counter, ready to receive the fibres (Figure 2.7).

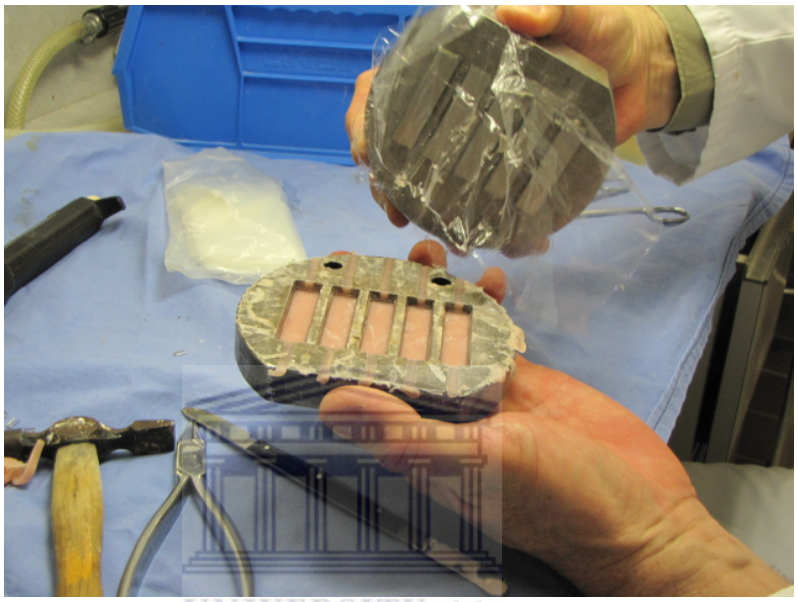

Figure 2.7: First a layer of PMMA was positioned right up to the lower level of the slot on which the fibres lay

The stiff polymerized fibres were positioned on this PMMA surface with their ends positioned in the slots or "stapled" in position in case of the shorter fibres.

The next step was another modification of the original protocol. After the positioning of the fibres on the flat surface produced by lid no.1, a second mixture using the same P/L ratio was prepared and flowed over the fibres to overfill the cavities (Figure 2.8). This second layer was left to "dough" in situ for the required 15 minutes.

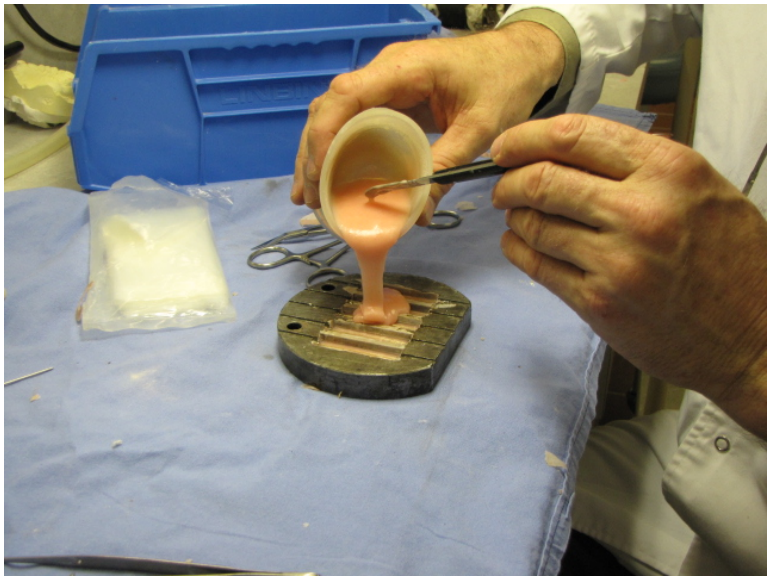

Figure 2.8: The second mixture is placed over the fibres to completely fill the cavities.

The mould was then closed with lid no.2 and compressed in a laboratory press (CH Wilhelm Wasserman, Feinwerk Hamburg) (Figure 2.9). To avoid the risk of displacing the fibres, the press was closed very slowly. Each closure took 22 minutes.

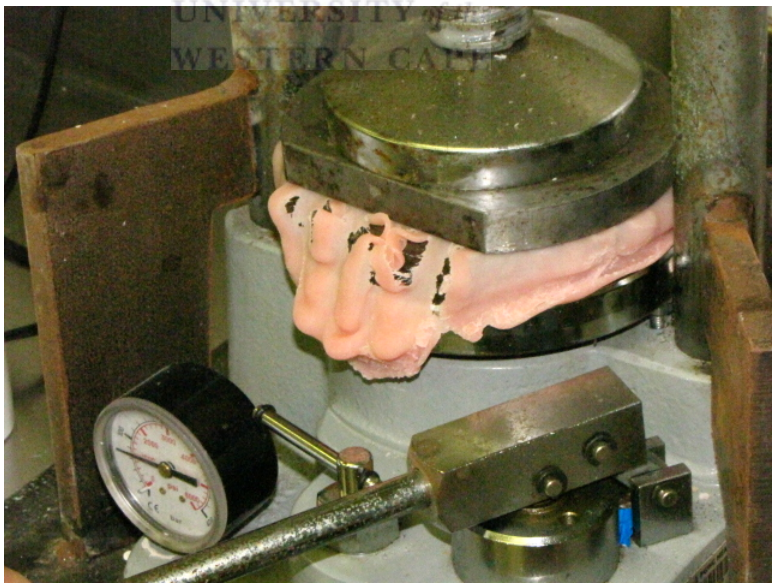

Figure 2.9: Compression to 200bars with laboratory press

Once the pressure reached 200 bars (3000 psi), and stayed static for 5 minutes, the press was opened and the mould transferred to a portable press. The mould and press were placed in a bath of boiling water. After the initial drop in temperature, the boiling of the

water resumed. Every batch was polymerized for 20 minutes at 100^0C. Thereafter the mould was allowed to bench cool to room temperature before opening. The specimens were removed using the ejector plugs provided. As the cavities were slightly tapered, the specimens could be teased out with the punch provided by the tool maker.

The specimens were inspected and checked for voids, cracks or bubbles. After inspection, the specimens were machined and lightly sanded (60 grit sandpaper) to make them rectangular to a width of 9mm. They were labeled and stored in a fridge until the specimens for all groups were made.

After each use, the mould was carefully cleaned. After removing the gross PMMA overflow and residue the mould was wiped with pure monomer to wash away any remaining remnants of PMMA.

2.2.6 Cyclic loading

To establish the cyclic load intensity, a group of test specimens were taken to the CSIR by this researcher. Together with the head of the laboratory he proceeded. Ten specimens in each of the three P/L groups were tested using a 3-point flexure test (ISO 1567: Specifications for denture base polymers). These were fractured to establish the fatigue load to be used in the cycling. The specimens were positioned on the supports of the 3-point bending apparatus with a 35 millimetres span. The specimens were placed with the fibre on the tension side of the load. A load was applied on the centre of the specimen perpendicular to the specimens' long axis. The crosshead speed was 6 mm/min using a loading cell of 5kN. Other researchers have used lower cross head speeds (2mm/min) arguing that a higher speed may produce a higher impact or momentum force (Tacir *et al* 2006).This downward force was continued until specimen failure (Figure 2.11). The proportional limit for each specimen was manually read from its respective load/deflexion graph.

Sinusoidal loads between the mean proportional limit (high load) and 10% of the mean fracture force (low load) were used for the cyclic loading to simulate the fatiguing process (Diaz-Arnold 2008).

The fatigue test was carried out in load control using a sinusoidal wave form at a frequency of 5HZ. A r-ratio of 0.1 was used in order to determine the stress range. ($r= \sigma_{min}/ \sigma_{max}$) The maximum stress was determined from the static test results – 60 to 100MPa. Thus, the maximum load was determined from the mean fracture stress of the static tests.σ

= $3PL/2.bd^2$, where σ = stress – Mpa, L = distance between supports – mm, P = load applied – newton, b = width – mm, d = depth – mm.

A cyclic load was applied for 10^4 cycles at 5 Hz. To establish the possible rise in temperature of the specimens during the cycling, a gauge was attached to the specimen and temperature fluctuations were monitored (Figure 2.10).

Figure 2.10: Monitoring temperature rises in specimens during cycling

The rise in temperature was less than one eighth of a degree Celsius. This was considered to be insignificant. This concurred with what Naumann *et al.* had found in 2009. The testing went forward using 10^4 cycles and 5 Hz for all fatiguing.

2.2.7 The testing procedure

All the specimens of the three groups were stored in water at 37^o C for 7 days prior to the start of the testing procedure.

All the specimens were machined and ground by hand to remove the slight taper and rectangular specimens were so acquired. One half (25 specimens) of each ratio group was randomly selected and subjected to a three point bending test using a Zwick Universal Testing Machine (Model 1446, Zwick, Ulm, Germany). Test data were captured by a

computer using the software TestXpert.

The specimens were positioned on the supports of the 3-point bending apparatus with a 35 millimetres span. The specimens were placed with the fibre on the tension side of the load. A load was applied on the centre of the specimen perpendicular to the specimens' long axis. The crosshead speed was 6 mm/min using a loading cell of 5kN. This downward force was continued until specimen failure (Figure 2.11).

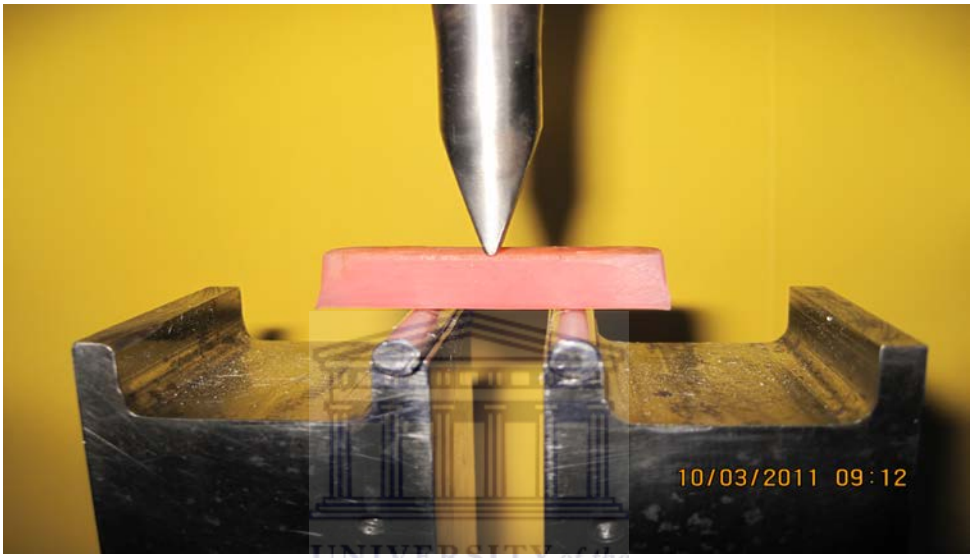

Figure 2.11: Breaking of the specimens with a 3-point-bending test

This maximum strength of the specimen before failure was recorded as Fmax in Newton. The flexural strength (FS) in MPa, was calculated using the equation (Kanie *et al.*, 2000, ISO 1567).

$$FS = \frac{3 F_{max} I}{2bd^2}$$

Where

Fmax = maximum load before fracture

I = distance between supports

b = width of specimen

d = height of specimen

The other half (25 specimens) of the three groups of specimens were subjected to fatigue testing using a cyclic loading machine (25 kN Instron Servo Hydraulic Testing Machine. Model 1342). The settings used in this machine were determined using a formula proposed

by Reis *et al.* (2006) based on discussions by Collins (1993). The testing was done at the CSIR laboratories in Pretoria, South Africa.
Sinusoidal loads (as discussed above in 2.2.6: Cyclic Loading) between the mean proportional limit (high load) and 10% of the mean fracture force (low load) were used. This cyclic load was applied for 10^4 cycles at 5 Hz.

Hereafter these 60 specimens were subjected to the same static 3-point bending test as described before.

Within each group, mean FS (flexural strength) values before and after cyclic loading were compared by means of a non-parametric analysis of variance (ANOVA). The changes in FS before and after cyclic loading were compared between groups to determine if one ratio was more fatigue resistant than others. The ratio most resistant to bending after cyclic loading was identified. A p-value of less than 0.05 was considered significant.

2.3 Piloting of the methodology

During the fracturing of the specimens, a "slit" or void was noted between the pre-impregnated fibre bundle and the heat-polymerized resin. The cross-section of the cavity left behind by the fibre bundle appeared to be larger than the dimension of the original fibre (Figure 2.12). The matrix surrounding the original fibre bundles also had disappeared, leaving tufts of dry fibres behind.

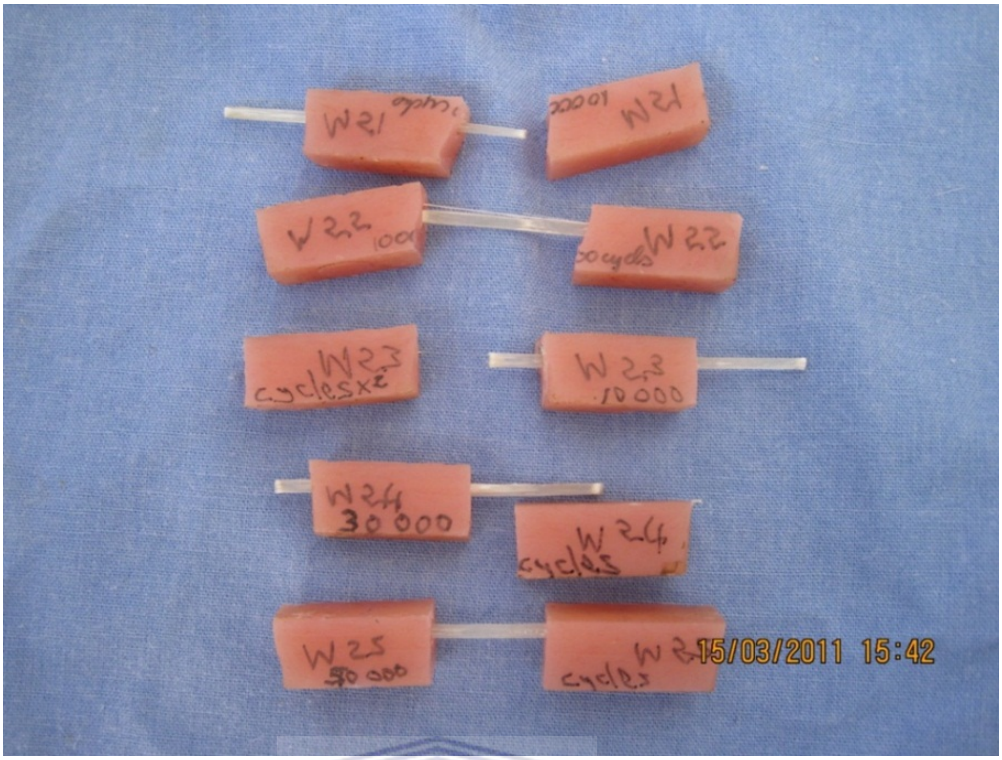

Figure 2.12: Delaminated fibre bundles after fracturing

Also, a void surrounding the fibre bundles was noted at the distal ends of the specimens (Figure 2.13).

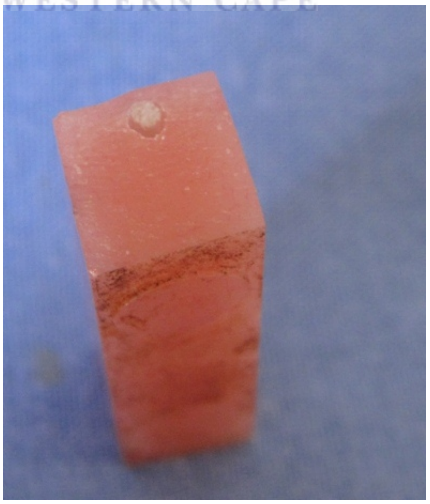

Figure 2.13: Void surrounding the fibre at the distal end of a specimen.

Extensive communication with the manufacturers in Finland and numerous further piloting exercises followed in order to eradicate any methodology flaws. A few of these letters are attached as addenda (Addenda C1-C4). Several proposed changes in the methodology were tested, such as:

1. Using a different heat-polymerizing PMMA material
2. Changing the polymerization cycle to a lower temperature over a longer period
3. Adding escape channels to the stainless steel mould
4. Repetition of the experiment with a new consignment of pre-impregnated fibres sent from Finland in a temperature controlled container.

However, the development of a slit between fibre bundle and PMMA matrix could not be avoided in the polymerized products.

The manufacturers eventually sent a third consignment of different fibres to repeat the complete experiment. This time, the fibres were of the non-impregnated type (Stick Fibres by Stickbond) (Figure 2.14).

Figure 2.14: Unidirectional, un-impregnated "Stick" fibre

These 'Stick Fibres' required 'wetting' with PMMA slurry that contained copious amounts of monomer, according to manufacturer's instructions. Otherwise, the methodology was exactly the same as described before. The whole experiment was repeated using this new fibre. The adapted methodology, incorporating all the small changes making the process possible was used throughout. This protocol was then accepted as the final methodology.

CHAPTER THREE

Results

3.1 Results for the piloting with the pre-impregnated fibres.

The first groups of specimens reinforced with the pre-impregnated fibres were all used to pilot and refine the dimensions of the specimens and fatiguing protocol (support span, load and number of cycles) done by the CSIR. Since the results in terms of FS of this piloting can only be regarded as preliminary, they are not included in the results section. A discussion of the piloting process will be presented in the next chapter.

However, the nature of the macroscopic fracturing pattern during this piloting phase deserves special attention: All fatigued and un-fatigued specimens from each P/L ratio group displayed an adhesive bond failure between fibre and PMMA (Figure 3.1).

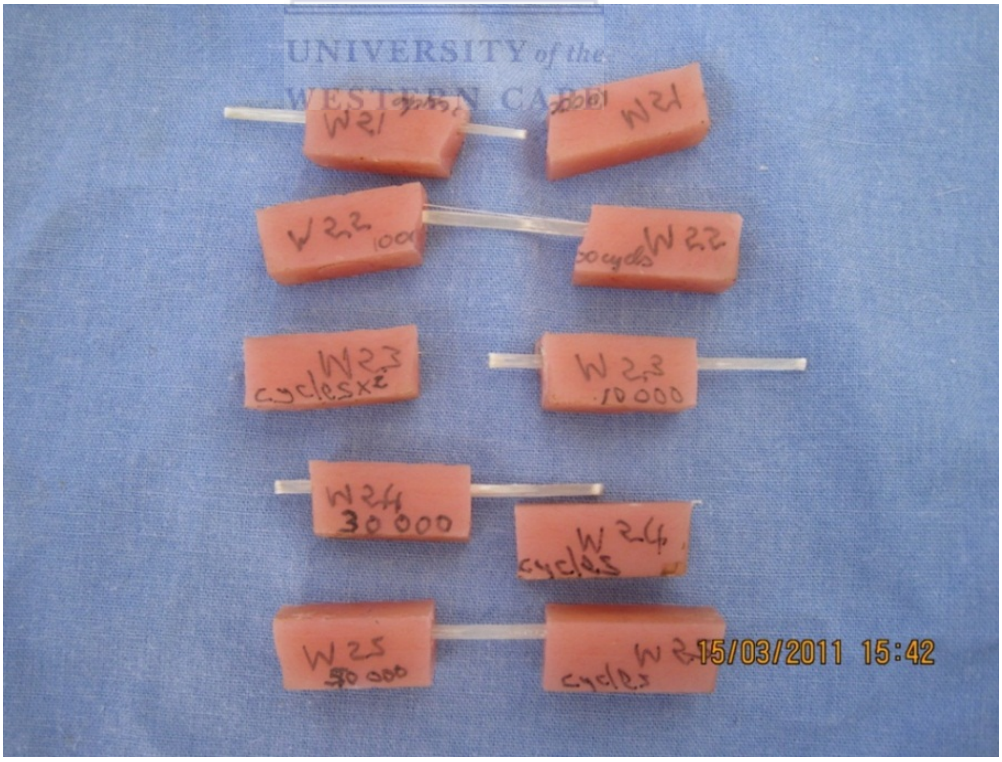

Figure 3.1: Adhesive bond failure between fibre and resin (fatigued specimens).

3.2 Results for the un-impregnated fibres.

3.2.1 Macroscopic fracture patterns

Similar to the specimens with the pre-impregnated fibres used during piloting, all the fractured specimens from the 3 ratio groups, fatigued and non-fatigued, displayed an adhesive bond failure between fibre and PMMA (Figure 3.2).

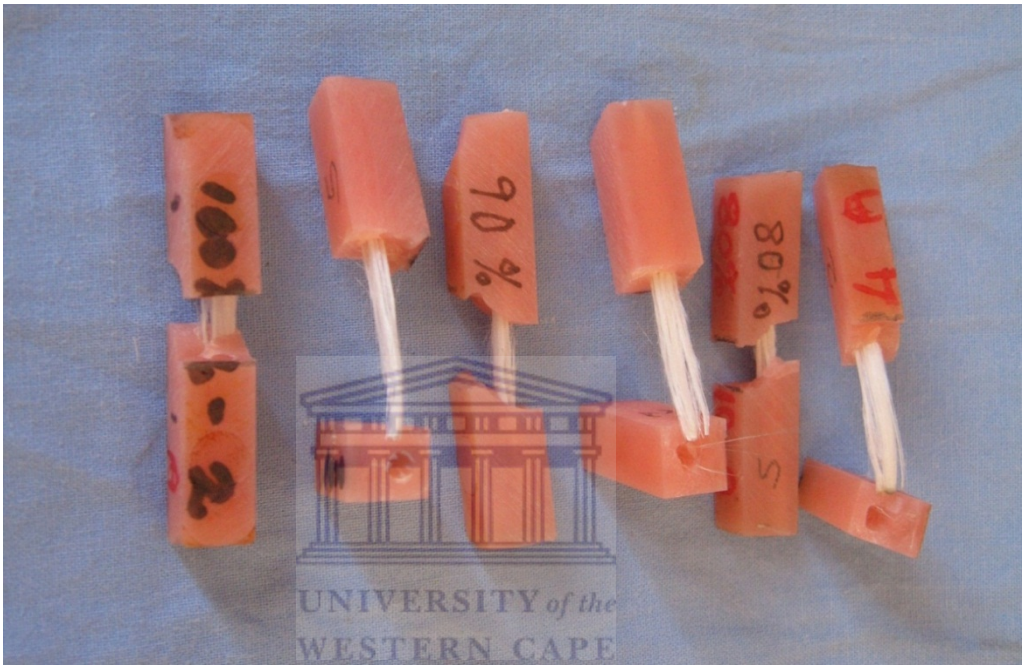

Figure 3.2: Specimens of the 80%, 90% and 100% P/L ratio groups demonstrating adhesive bond failure and tufting of fibres.

3.2.2 Raw data for the groups that were not subjected to cyclic loading (unfatigued).

Table 3.1 shows the raw data for the group of specimens that were mixed according to the recommended mixing ratio (100%) and not subjected to fatigue loading.

Mixing ratio 100% - unfatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
19	1673.49	1673.49	2.20	8.99	10.30	65.80
21	1534.49	1534.49	1.98	9.74	10.18	57.01
22	1592.93	1592.93	2.03	9.73	10.16	59.47
23	1436.61	1436.61	1.99	8.31	10.04	64.31
24	1619.50	1619.50	2.11	9.54	10.08	62.65

Mixing ratio 100% - unfatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
25	1121.07	1121.07	1.32	9.64	10.25	41.51
26	1684.33	1684.33	2.19	9.57	10.33	61.85
27	1408.70	1408.70	1.94	9.38	9.90	57.46
28	1723.32	1723.32	2.24	9.71	10.14	64.73
29	1327.44	1327.44	1.58	9.16	10.03	54.02
30	1682.12	1682.12	2.33	9.18	10.00	68.71
31	781.54	781.54	0.98	9.17	10.11	31.27
32	1320.11	1320.11	1.74	9.33	10.05	52.53
33	1593.58	1593.58	2.03	9.79	10.28	57.76
34	1285.00	1285.00	1.91	9.09	10.10	51.97
35	1617.16	1562.90	2.30	9.37	10.18	62.45
36	1382.65	1382.65	1.62	9.56	9.97	54.56
37	787.58	787.58	0.97	9.35	10.04	31.34
38	1758.36	1758.36	2.31	9.81	10.09	66.02
39	1606.99	1606.99	1.82	9.69	10.29	58.73
40	1848.77	1848.77	2.46	9.33	10.22	71.14
Average	**1465.99**	**1463.40**	**1.91**	**9.40**	**10.13**	**56.92**

Table 3.1: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilonFMax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the recommended ratio and not fatigued.

Table 3.2 shows the raw data for the group of specimens that were mixed according to the 90% mixing ratio and not subjected to fatigue loading.

Mixing ratio 90% - unfatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
41	1943.44	1943.44	2.68	9.33	10.22	74.79
42	1866.22	1866.22	2.29	9.81	10.15	69.25
43	1067.05	1067.05	1.43	9.02	10.15	43.06
45	1561.92	1561.92	1.91	9.64	10.29	57.38
46	1824.16	1824.16	2.41	9.08	10.19	72.55
47	1707.84	1707.84	2.38	9.20	10.06	68.79

Mixing ratio 90% - unfatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
48	1516.33	1516.33	1.87	9.30	10.17	59.12
49	1659.80	1659.80	2.24	9.41	9.93	67.08
50	1724.19	1724.19	2.04	9.80	10.15	64.04
51	1095.13	1095.13	1.23	9.84	10.25	39.72
52	1806.79	1806.79	2.24	9.54	10.00	71.02
53	1896.20	1896.20	2.58	8.91	10.22	76.41
54	1287.31	1287.31	1.60	9.57	9.94	51.05
55	1005.18	1005.18	1.16	9.54	10.08	38.89
56	1531.97	1531.97	1.96	9.28	10.11	60.57
57	1419.94	1419.94	1.87	8.90	10.01	59.71
58	1096.48	1066.30	1.27	9.59	10.10	42.03
59	1951.48	1881.09	2.56	9.54	10.01	76.56
60	1577.49	1577.49	1.93	9.40	10.16	60.97
61	1586.77	1586.77	2.17	9.35	10.07	62.76
62	1680.26	1680.26	2.13	9.67	10.16	63.12
63	1254.00	1254.00	1.57	9.10	10.16	50.06
Average	**1548.18**	**1543.61**	**1.98**	**9.40**	**10.12**	**60.41**

Table 3.2: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilonFmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the 90% ratio and not fatigued.

Table 3.3 shows the raw data for the group of specimens that were mixed according to the 80% mixing ratio and not subjected to fatigue loading.

Mixing ratio 80% - unfatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
66	1265.51	1265.51	1.72	8.71	10.00	54.49
67	1452.60	1452.60	2.17	8.64	10.04	62.55
68	1541.68	1541.68	2.27	8.64	10.02	66.65
69	1392.08	1392.08	2.30	8.30	9.86	64.69
70	1624.86	1624.86	2.52	8.65	9.91	71.73
71	778.75	778.75	1.32	8.64	10.13	32.94
72	1420.69	1420.69	2.29	8.11	9.92	66.76

Mixing ratio 80% - unfatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
73	1490.17	1490.17	2.20	8.25	9.99	67.87
74	1811.33	1811.33	2.66	8.96	10.04	75.21
75	1634.94	1634.94	2.40	9.08	9.98	67.79
76	1615.81	1615.81	2.40	8.84	9.84	70.79
77	1799.73	1799.73	2.80	8.63	9.85	80.60
79	1448.32	1448.32	2.28	7.77	10.04	69.34
80	1373.91	1373.91	2.17	7.88	9.98	65.65
82	1418.79	1418.79	2.32	8.18	9.93	65.96
83	639.58	639.58	0.99	8.70	10.08	27.13
84	1509.04	1509.04	2.32	8.32	10.17	65.76
85	1528.21	1528.21	2.54	7.59	10.19	72.72
86	1576.64	1576.64	2.61	8.25	10.19	69.02
87	1519.43	1519.43	2.62	8.10	9.86	72.36
88	1634.67	1634.67	2.82	8.20	10.11	73.14
89	1592.10	1592.10	2.61	8.05	9.96	74.76
90	1520.17	1520.17	2.79	7.62	10.19	72.05
91	1636.92	1636.92	2.65	8.28	9.93	75.18
Average	**1467.75**	**1467.75**	**2.32**	**8.35**	**10.01**	**66.05**

Table 3.3: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilonFmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the 80% ratio and not fatigued.

3.2.3 Raw data for the groups that were subjected to cyclic loading (fatigued)

Table 3.4 shows the raw data for the group of specimens that were mixed according to the 100% mixing ratio and subjected to fatigue loading.

Mixing ratio 100% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
137	1828.54	1828.54	2.80	9.13	10.13	73.19
138	1517.57	1488.29	2.22	9.67	9.99	58.97
139	1719.16	1719.16	2.11	10.01	10.19	62.02
140	1554.48	1554.48	2.07	9.88	9.89	60.32

Mixing ratio 100% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
141	1731.03	1731.03	2.48	9.30	10.06	68.97
142	1682.69	1682.69	2.20	9.48	9.90	67.91
144	1689.92	1689.92	2.18	9.73	10.13	63.47
145	1623.25	1623.25	2.45	8.85	9.92	69.90
146	1621.74	1621.74	2.29	9.24	9.90	67.15
147	1337.31	1337.31	1.80	9.35	10.15	52.06
148	1984.88	1984.88	2.35	10.25	10.58	64.87
149	1873.21	1873.21	2.48	10.56	10.20	63.94
150	1691.77	1691.77	2.30	10.14	10.14	60.85
151	1478.32	1478.32	2.12	9.30	9.93	60.45
152	1835.74	1835.74	2.61	9.42	10.10	71.64
153	1588.33	1588.33	2.29	9.83	9.96	61.08
154	1505.51	1505.51	2.14	9.10	10.06	61.30
155	1618.73	1618.73	2.11	9.40	10.04	64.06
156	1759.94	1759.94	2.47	9.08	10.24	69.32
Average	**1665.38**	**1663.83**	**2.29**	**9.56**	**10.08**	**64.29**

Table 3.4: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilon Fmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the recommended ratio and subjected to cyclic loading.

Table 3.5 shows the raw data for the group of specimens that were mixed according to the 90% mixing ratio and subjected to fatigue loading.

Mixing ratio 90% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
114	1964.43	1964.43	2.71	9.38	9.85	80.95
115	1477.65	1477.65	2.12	9.27	10.02	59.54
116	1745.08	1745.08	2.33	9.48	10.00	69.03
117	1505.16	1505.16	2.07	9.27	10.00	60.89
118	1506.36	1506.36	1.90	8.93	9.87	64.93
119	1821.48	1821.48	2.55	9.59	9.84	73.56
120	1739.08	1739.08	2.41	9.40	9.97	69.80

Mixing ratio 90% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
121	1507.21	1507.21	2.09	9.22	9.93	62.17
122	1774.24	1774.24	2.34	9.12	10.03	72.52
123	1462.76	1462.76	1.81	9.10	10.20	57.94
124	1853.15	1853.15	2.42	9.62	10.15	70.12
125	1521.98	1521.98	2.07	9.16	9.99	62.43
129	1697.85	1697.85	2.12	9.53	10.03	66.41
130	1722.18	1722.18	2.31	9.59	10.17	65.11
131	1599.90	1599.90	2.21	9.54	9.98	63.14
132	1646.11	1646.10	2.26	9.56	10.17	62.43
133	1751.78	1751.78	2.43	9.73	9.98	67.79
134	1859.48	1859.48	2.49	9.39	10.00	74.26
135	1740.03	1740.03	2.30	9.72	9.90	68.49
136	1700.34	1700.34	2.28	9.49	9.96	67.73
Average	**1679.81**	**1679.81**	**2.26**	**9.40**	**10.00**	**66.96**

Table 3.5: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilon Fmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the 90% ratio and subjected to cyclic loading.

Table 3.6 shows the raw data for the group of specimens that were mixed according to the 80% mixing ratio and subjected to fatigue loading.

Mixing ratio 80% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
92	1447.52	1447.52	2.52	7.35	10.02	73.56
93	1601.98	1601.98	2.77	7.84	9.81	79.62
94	1220.89	1220.89	1.83	8.53	10.03	53.35
95	1356.86	1356.86	2.11	8.35	9.86	62.68
96	1530.53	1492.32	2.49	7.95	9.91	73.51
97	1858.05	1858.05	3.04	8.67	10.07	79.25
98	1454.38	1454.38	2.58	8.21	9.81	69.03
99	1971.04	1971.04	2.95	9.27	10.06	78.79
100	1454.14	1454.14	2.18	9.27	10.06	58.12
101	1754.26	1754.26	2.60	8.67	10.09	74.53

Mixing ratio 80% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
102	1783.76	1783.76	2.48	9.12	10.12	71.62
103	1824.90	1824.90	2.54	9.37	9.98	73.33
104	1522.18	1522.18	2.83	7.63	10.01	74.66
105	1277.98	1277.98	1.94	8.17	10.07	57.85
106	1408.02	1408.02	2.12	8.36	10.05	62.53
107	1422.44	1422.44	2.11	8.75	9.89	62.33
108	1662.53	1662.53	2.55	8.65	10.06	71.22
109	1647.65	1647.65	2.97	8.04	9.82	79.69
110	1486.74	1486.74	1.99	9.53	9.96	58.97
111	1787.61	1787.61	3.07	8.85	9.81	78.71
112	1808.88	1808.88	2.72	9.50	9.84	73.74
113	1602.76	1602.76	2.70	7.59	10.10	77.63
Average	**1585.69**	**1583.95**	**2.50**	**8.53**	**9.97**	**70.21**

Table 3.6: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilon Fmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the 80% ratio and subjected to cyclic loading.

3.2.4 Descriptive statistics

The following analysis presents the descriptive statistics for the different mixing ratio values (80%, 90%, 100%) and fatigue level (1=fatigued, 0=not fatigued): the highest force registered before failure of the specimens (Fmax in N), the force registered at fracture (Fbreak in N), the width and height of the specimens (mm), deflexion (epsilon in mm), and the calculated flexural strength (FS in MPa).

Table 3.7 gives a summary of statistics for each mix Ratio-Fatigue combination.

Mix	Fatigued	# Obs	Variable	Mean	Median	Std Dev	Minimum	Maximum
80%	0	24	Fmax	1467.75	1519.80	265.77	639.58	1811.33
			Fbreak	1467.75	1519.80	265.77	639.58	1811.33
			Epsilon	2.32	2.36	0.44	0.99	2.82
			Width	8.35	8.29	0.41	7.59	9.08
			Height	10.01	10.00	0.11	9.84	10.19
			FS	66.05	68.44	12.28	27.13	80.60
	1	22	Fmax	1585.69	1566.26	202.05	1220.89	1971.04

Mix	Fatigued	# Obs	Variable	Mean	Median	Std Dev	Minimum	Maximum
			Fbreak	1583.95	1562.08	202.71	1220.89	1971.04
			Epsilon	2.50	2.55	0.37	1.83	3.07
			Width	8.53	8.59	0.64	7.35	9.53
			Height	9.97	10.02	0.11	9.81	10.12
			FS	70.21	73.42	8.27	53.35	79.69
90%	0	22	Fmax	1548.18	1582.13	299.29	1005.18	1951.48
			Fbreak	1543.61	1582.13	297.36	1005.18	1943.44
			Epsilon	1.98	2.00	0.45	1.16	2.68
			Width	9.40	9.41	0.28	8.90	9.84
			Height	10.12	10.15	0.10	9.93	10.29
			FS	60.41	61.86	11.83	38.89	76.56
	1	20	Fmax	1679.81	1711.26	145.50	1462.76	1964.43
			Fbreak	1679.81	1711.26	145.50	1462.76	1964.43
			Epsilon	2.26	2.29	0.22	1.81	2.71
			Width	9.40	9.44	0.22	8.93	9.73
			Height	10.00	10.00	0.10	9.84	10.20
			FS	66.96	67.07	5.66	57.94	80.95
100%	0	21	Fmax	1465.99	1592.93	289.05	781.54	1848.77
			Fbreak	1463.40	1562.90	287.88	781.54	1848.77
			Epsilon	1.91	1.99	0.42	0.97	2.46
			Width	9.40	9.38	0.35	8.31	9.81
			Height	10.13	10.11	0.12	9.90	10.33
			FS	56.92	58.73	10.78	31.27	71.14
	1	19	Fmax	1665.38	1682.69	155.06	1337.31	1984.88
			Fbreak	1663.83	1682.69	156.75	1337.31	1984.88
			Epsilon	2.29	2.29	0.23	1.80	2.80
			Width	9.56	9.42	0.45	8.85	10.56
			Height	10.08	10.06	0.17	9.89	10.58
			FS	64.29	63.94	5.16	52.06	73.19

Table 3.7: Summary of the statistics for each Mix ratio-Fatigue combination. 0= not fatigued; 1= fatigued; FS= flexural strength

3.2.5 Analytical statistics

3.2.5.1 Fmax

Table 3.8. Shows a summary of the descriptive statistics for Fmax for all the groups.

groups	n	min	max	mean	median	std dev.
100% (0)	21	781.54	1848.77	1465.99	1592.93	289.05
100% (1)	19	1337.31	1984.88	1663.83	1682.69	155.06
90% (0)	22	1005.18	1951.48	1548.18	1582.13	299.29
90% (1)	22	1462.76	1964.43	1679.81	1711.26	145.50
80% (0)	24	639.58	1811.33	1467.75	1519.80	265.77
80% (1)	22	1220.89	1971.04	1585.69	1566.26	202.05

Table 3.8: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (STD dev) of the highest load measured in newton (Fmax). (0= no fatiguing; 1= fatiguing)

Initially a comparison was drawn between the mean Fmax and means Fmax values for the different ratio and fatigue groups.

For the same mixing ratio, the mean, as well as the median Fmax after fatiguing (1) is always higher than the mean and the median Fmax of the groups that were not fatigued (0).

For both fatigued and unfatigued groups, the Fmax increases from 80% group to 90% group. The mean difference in Fmax between the mixratio of 80 and the mixratio of 90, over both fatigued and not fatigued, is only very marginally significant (P=0.087 for both groups) ($p<0.05$) When comparing the median values for mixratio 90%-100% though, the trend of Fmax values for the fatigued specimens is essentially in the opposite direction.

A graphical representation of the distribution of Fmax defined by factors of fatigue and ratio is given by a boxplot (Figure 3.3).

The ends of the box are approximate quartiles and the heavy line in the middle is the median. The table of means shows similar trends, as do the (analysis of variance) significance tests which are essentially comparisons of means. The (main effect), Fatigue is statistically significant, P=0.001.

Further examination of the dimensions of the specimens was done to possibly explain this trend fully.

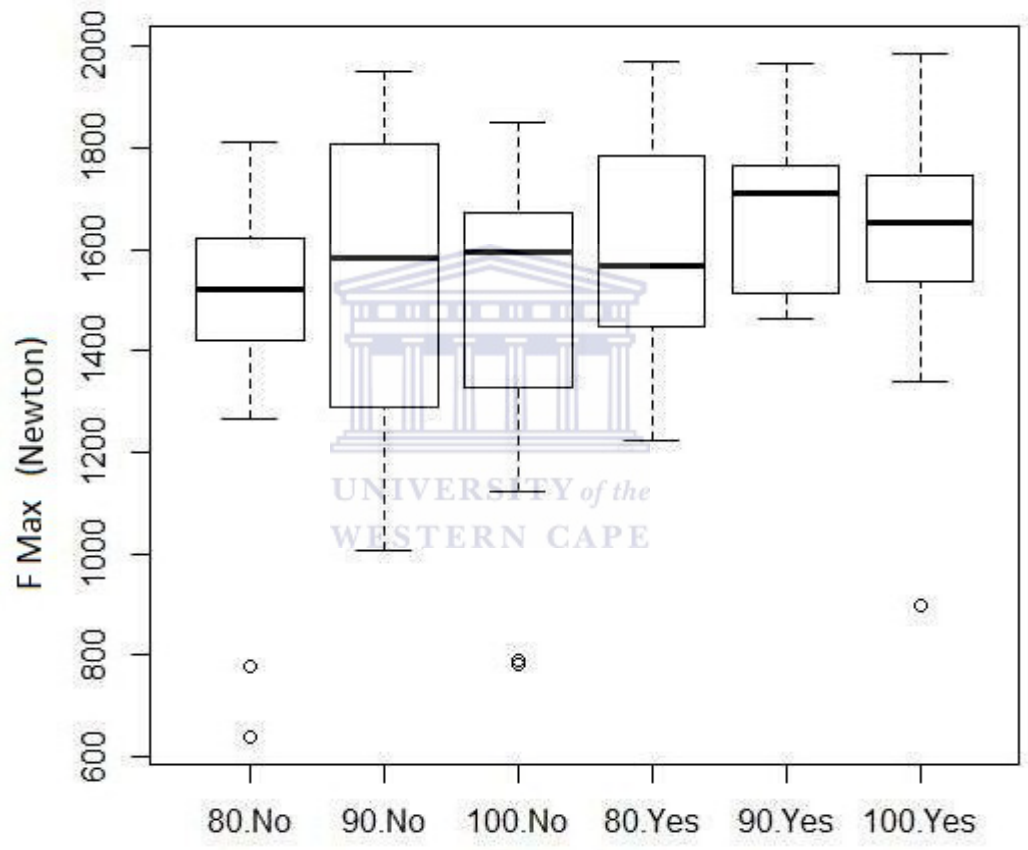

Figure 3.3: Box and whiskers plot of the median Fmax for the 3 mixing ratios without cyclic loading (no) and with cyclic loading (yes).

3.2.5.2 Dimensions of the specimens

After removal from the mould, the trapezoid specimens were machined and thereafter finished off by hand into square shaped blocks. There could, therefore, be small fluctuations in width and height of the specimens. Consequently each specimen was carefully measured in width and height.

3.2.5.2.1 Width

Table 3.9 shows a summary of the descriptive statistics for width for all the groups.

groups	n	min	max	mean	median	std dev
100% (0)	21	8.31	9.81	9.40	9.38	0.35
100% (1)	19	8.85	10.56	9.56	9.42	0.45
90% (0)	22	8.90	9.84	9.40	9.41	0.28
90% (1)	22	8.97	9.73	9.40	9.44	0.22
80% (0)	24	7.59	9.08	8.35	8.29	0.41
80% (1)	22	7.35	9.53	8.53	8.59	0.64

Table 3.9: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (std dev) of the width measured in mm. (0= no fatiguing; 1= fatiguing)

The mean width of the different groups was compared using a 2-way ANOVA test of fixed effects (Table 3.10). The numerator degrees of freedom and the denominator degrees of freedom are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
mix	2	122	85.02	< .0001
fatigued	1	122	2.44	0.12
mix*fatigued	2	122	0.58	0.56

Table 3.10: ANOVA test of fixed effects for the widths. Numerator (Num),Denominator (Den), Degrees of Freedom (DF).

Table 3.11 shows an abbreviated version of the Least Squares Means test. * Pairwise comparisons show the mean width for 80 is significantly lower than the mean width for 90 or 100 ($p<0.0001$).

mix	Estimate	Standard Error
80*	8.44	0.062
90	9.4	0.065
100	9.48	0.066
Fatigue	Estimate	Standard Error
0 (No)	9.05	0.051
1 (Yes)	9.17	0.054

* = statistically significant

Table 3.11: Statistical evidence of width differences using a least squares means test.

A box and whisker plot was used to demonstrate the different values and their effects on the distribution of the width of specimens (Figure 3.4).

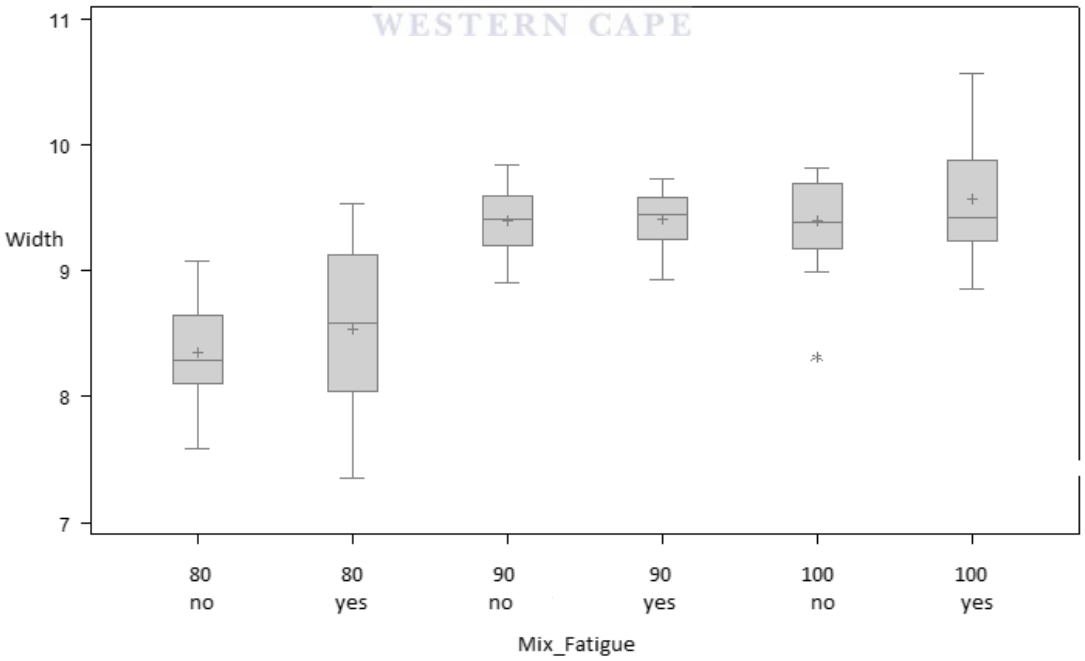

Figure 3.4: Boxplot of width for groups defined by the factors, fatigue and mixing ratio. The ends of the box are approximate quartiles and the line in the middle is the median. The + sign in the box represents the mean.

When considering width, there is a significant difference between that for 80% and each of 90% and 100% groups.

3.2.5.2.2 Height

Table 3.12 shows a summary of the descriptive statistics for height for all the groups.

groups	n	min	max	mean	median	st dev
100% (0)	21	9.90	10.33	10.13	10.11	0.12
100% (1)	19	9.89	10.58	10.08	10.06	0.17
90% (0)	22	9.93	10.29	10.12	10.15	0.10
90% (1)	22	9.84	10.20	10.00	10.00	0.10
80% (0)	24	9.84	10.19	10.01	10.00	0.11
80% (1)	22	9.81	10.12	9.97	10.02	0.11

Table 3.12: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (sd) of the height measured in mm. (0= no fatiguing; 1= fatiguing)

Similarly Table 3.13 shows statistical evidence of height differences using ANOVA test of fixed effects . The numerator degrees of freedom and the denominator degrees of freedom are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
mix	2	122	10.05	< .0001
fatigued	1	122	10.18	0.0018
mix*fatigued	2	122	1.39	0.2533

Table 3.13: ANOVA test of fixed effects. Numerator(Num),Denominator(Den), Degrees of Freedom(DF).

Table 3.14 shows an abbreviated version of the Least Squares Means test for the height of the specimens. * Pairwise comparisons show the mean for 80 is significantly lower than the mean for 90 or100 ($p<0.0001$)

mix	Estimate	Standard Error
80*	9.99	0.017
90	10.06	0.018
100	10.11	0.019

Fatigue	Estimate	Standard Error
0 (No)	10.08	0.01
1 (Yes)	10.02	0.02

* = statistically significant

Table 3.14: Statistical evidence of height differences using a least squares means test.

A box and whisker plot was used to demonstrate the different values and their effects on the distribution of the height of specimens. Figure 3.5 illustrates this clearly.

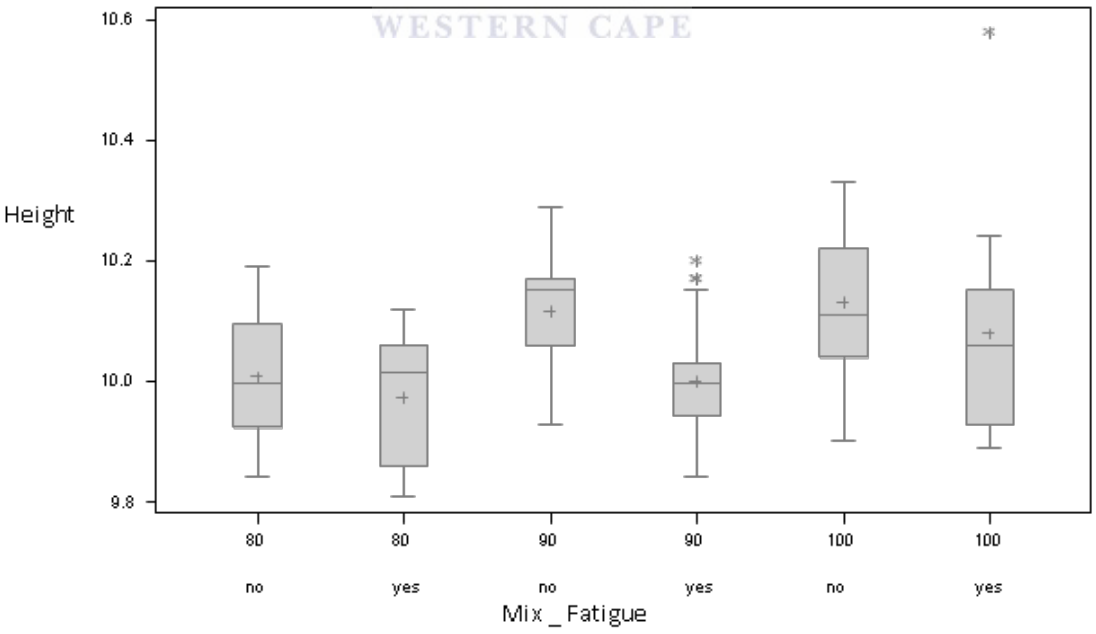

Figure 3.5: Boxplot of height for groups defined by the factors, fatigue and mixing ratio. The ends of the box are approximate quartiles and the line in the middle is the median. The + sign in the box represents the mean.

For height there is one outlier in the data (a value of 10.58 for the group with 100%, with fatiguing). With, or without the outlier analysis indicates significant differences in height.

As further confirmation of the influence of height and width on the properties of our specimens Figure 3.6 shows the distribution of thicklr (height and width and length) in the various mixing ratios and fatigue subgroups. Every dot represents one observation.

It is noted that the 80% 'yes' and 'no' values do not even overlap. This confirms the finding that the 80% group varies considerably from the 90% and the 100% groups in width and height.

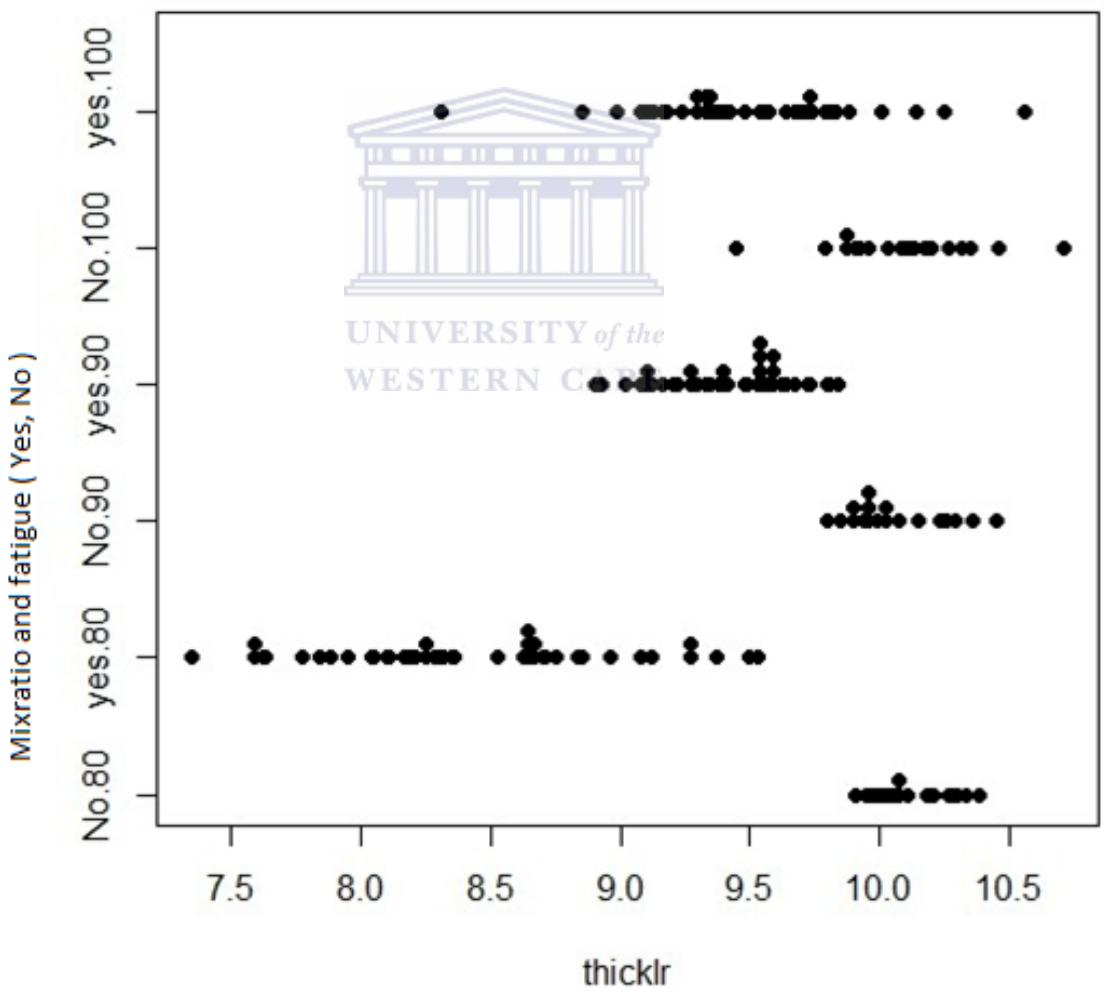

Figure 3.6: The distribution of thicklr (height and width and length) in the various mixratio and fatigue subgroups. Every dot represents one observation.

3.2.5.3 Deflexion

A measure of deflexion is given by the epsilon values before failure of the different specimen groups.

Table 3.15 shows a summary of the descriptive statistics for deflexion (epsilon) for all the groups.

groups	n	min	max	mean	median	sd
100% (0)	21	0.97	2.46	1.91	1.99	0.42
100% (1)	19	1.80	2.80	2.29	2.29	0.23
90% (0)	22	1.16	2.68	1.98	2.00	0.45
90% (1)	22	1.81	2.71	2.26	2.29	0.22
80% (0)	24	0.99	2.82	2.32	2.36	0.44
80% (1)	22	1.83	3.07	2.50	2.55	0.37

Table 3.15: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (sd) of the deflexion measured in mm. (0= no fatiguing; 1= fatiguing)

The results and plot of the residuals indicates skewness and non-normality. For this reason a nonparametric approach was taken rather that the more commonly used Kruskal-Wallis or Friedman tests. As the data was multifactorial a method known as the Aligned Ranks Transform (ART) was used for the analysis (Mansouri, 1999). The ART analysis tool was used to align and rank the data. ANOVA analysis was then done. The software used for statistical analysis was SAS v9 (SAS Institute Inc., Cary, NC, USA). The ART analysis was done in SAS using a user constructed macro.

Table 3.16 shows the results of the ART for the variable Epsilon. Numerator(Num), Denominator(Den), Degrees of Freedom(DF). The Num DF and the Den DF are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
mix	2	122	12.46	< .0001
fatigued	1	122	15.39	0.0001
mix*fatigued	2	122	0.56	0.5725

Table 3.16: Statistics of a nonparametric ART analysis of the data for the variable Epsilon.

Table 3.17: Shows a summary of the statistics of least squares means for the variable Epsilon. * Pairwise comparisons show the mean width for 80 is significantly lower than the mean width for 90 or 100 ($p<0.0001$). ** Mean with Fatigue significantly higher than without fatigue ($p=0.0001$)

mix	Estimate	Standard Error
80*	2.42	0.055
90	2.12	0.057
100	2.1	0.059
Fatigue	Estimate	Standard Error
0 (No)**	2.07	0.046
1 (Yes)	2.35	0.048

Table 3.17: Summary of the statistics of least squares means for the variable Epsilon.

Figure 3.7 shows boxplots of deflexion (using the epsilon values for strain development) against the mixing ratios and fatigue subgroups. The trends indicated by this graph are that median deflexion decreases from 80% to 90% groups and remains stable thereafter. A two way analysis of variance with response variable deflex and factors Mixratio and Fatigue confirms that there is a statistically significant change from 80% to 90%, ($p<0.001$), and that the change from 90% to 100% groups is not significant. Main effect Fatigue is significant, $p<0.001$.

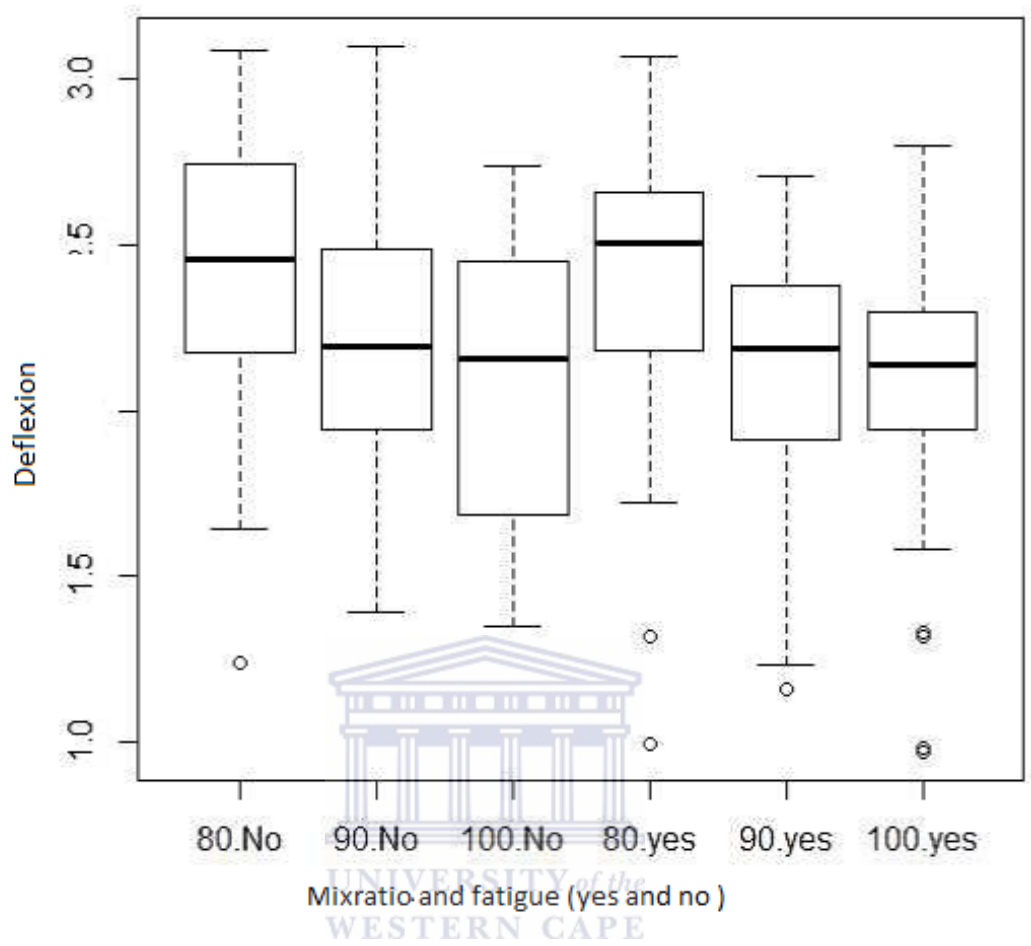

Figure 3.7: Boxplots of deflexion (epsilon Fmax) for the mixratio x fatigue subgroups.

3.2.5.4 Flexural strength

Table 3.18 shows a summary of the descriptive statistics for flexural strength (FS) for all the groups.

groups	n	min	max	mean	median	sd
100% (0)	21	31.27	71.14	56.92	58.73	10.78
100% (1)	19	52.06	73.19	64.29	63.94	5.16
90% (0)	22	38.89	76.56	60.41	61.86	11.83
90% (1)	22	57.94	80.95	66.96	67.07	5.66

groups	n	min	max	mean	median	sd
80% (0)	24	27.13	80.60	66.05	68.44	12.28
80% (1)	22	53.35	79.69	70.21	73.42	8.27

Table 3.18: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (sd) of the flexural strength measured in MPa. (0= no fatiguing; 1= fatiguing)

The variation in width, height and deflexion confirms that the standardized variable of FS would be appropriate to be used for analysis. Flexural strength was calculated using the equation: $FS= 3FMaxL/2bd^2$

Initially a standard two-way analysis of variance was done. However examination of the residuals from the model indicates that they are not normally distributed. For this reason a nonparametric approach was taken. The ART was again used. These analyses demonstrate that there is no significant interaction between Mix and Fatigue state, that the 80 mix has a significantly higher mean than either the 90% or 100% groups (with differences of about 4.4 and 7.5 units respectively), and that the Fatigued state has a higher mean than the Not Fatigued state by about 6.0 units.

Table 3.19 shows the results of a nonparametric ART analysisfor the variable FS. Numerator (Num), Denominator (Den), Degrees of Freedom (DF). The Num DF and the Den DF are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
Mix	2	122	11.82	< .0001
Fatigued	1	122	10.53	0.0015
mix*fatigued	2	122	0.26	0.7707

Table 3.19 Results of the ART for the variable FS.

Table 3.20: Shows a summary of the statistics of a nonparametric ART analysis of the data for least squares means for FS. * Pairwise comparisons based on ART analysis show the mean FS for 80% is significantly lower than the mean FS for 90% ($p<0.0001$) or 100% ($p=0.0026$)

** Mean FS with Fatigue significantly higher than without ($p=0.0015$)

mix	Estimate	Standard Error
80*	68.13	1.42
90	63.68	1.48
100	60.6	1.52

Fatigue	Estimate	Standard Error
0 (No)**	61.12	1.18
1 (Yes)	67.15	1.23

Table 3.20: Results of the ART analysis of the data for least squares means (FS)

In Table 3.21 both the fatigued and unfatigued groups display an increasing flexural strength. The 80% group in both fatigued and unfatigued specimens have the highest FS.

Unfatigued					
Mixing ratio	**F Max in N**	**Deflexion**	**Width**	**Thickness**	**Flexural strength**
100%	1557.85	2.04333	9.40444	10.13	60.6221
90%	1674.81	2.16823	9.39588	10.11	65.5972
80%	1536.71	2.43	8.32045	10	69.3206
Fatigued					
Mixing ratio	**F Max in N**	**Deflexion**	**Width**	**Thickness**	**Flexural strength**
100%	1665.38	2.2879	9.56421	10.08	64.2886
90%	1679.81	2.261	9.4045	10	66.9615
80%	1585.69	2.504	8.53045	9.9741	70.2145

Table 3.21: Flexural strength (MPa) defined by mixratio and fatigue status and sample dimensions.

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CHAPTER 4

Discussion

4.1 Introduction

The aim of this research study was to investigate the influence of changing P/L ratios on the fatigue behavior of a fibre reinforced PMMA used for denture bases.

The FS of 3 different P/L ratios of fibre-reinforced PMMA was compared. Half of the specimens in each P/L group were subjected to fatigue loading before the 3-point bending test was done.

The median and mean FS values before and after cyclic loading were calculated and compared by means of a non-parametric analysis of variance (ART). A p-value of less than 0.05 was considered significant.

The original protocol of this study accepted certain outcomes i.e.: a certain degree of adhesion between fibre and matrix. This proved to be wrong. The results from this study were unexpected and therefore it is difficult to answer the original research question.

Besides the unexpected adhesion problem, the researcher was faced with 2 further major challenges. The extent of these challenges could not be anticipated during the development of the protocol. The first was the difficulty of the manufacturing of the specimens using a custom-made template. This could not be deduced from reading literature on similar research projects. The second one was the infrastructure and expertise necessary to do cyclic loading.

Therefore, this discussion will start with a presentation of the piloting process prior to the discussion of the results.

4.2 Piloting process

4.2.1 The mould

The mould was designed to fit the length of the donated ever Stick fibres and to mimic the thickness of a denture base as closely as possible (Jerolimov *et al.,* 1989, Kanie *et al.,* 2000, Bertassoni *et al.,* 2008). An attempt was made to position the fibres closer to one side of the specimens to make full use of the strength supplied by inner support on the tension side of a material (Narva *et al.,* 2005b). It seemed almost impossible to position the fibres on the tension side of the these thin (4mm) specimens. The doughy consistency of the heat cured PMMA made this impossible. Researchers in previous studies pre-wet their fibres to obtain a better bond between fibre and PMMA (Vallittu 1999, Tacir 2006).

A few specimens were manufactured and sent to the CSIR in Pretoria for pilot testing (See 4.2.4: Cyclic loading). CSIR established that the fibres did not lie reliably on the tension side of the specimens and made results unreliable. The height of the specimens was to be increased by 6 mm to a total of 10 mm of height to facilitate correct positioning of the fibre bundle and calibration of the testing equipment. The depth of the cavities in the mould was modified accordingly.

4.2.2 The fibres

The research proposal was sent to the manufacturers of the fibres in Finland for possible sponsoring of the fibres. They kindly agreed. The fibres suggested by the Stick Company in Finland were 50mm pre-impregnated ever Stick fibres (Stick Tech). Consequently, 150 fibres for the complete project were donated by the manufacturers.

Due to the high cost of the fibres, the manufacturing of the specimens was extensively piloted using metal wires and resin impregnated superfloss as substitutes for the fibre. Both the wire and PMMA impregnated superfloss behaved differently from the fibres that were subsequently used. The fibres were much more difficult to work with.

The fibres were not all of equal length, some being longer, some shorter than the 50mm as stated in the marketing brochure. This complicated the accurate manipulation of the fibers and therefore complicated the manufacturing of the specimens. The custom-made mould

was already fabricated and the ends did not reach the slots. For these shorter fibres, an additional method of stabilization of the fibre was designed in the form of a staple-like thin metal wire (Figure 2.3 Methodology). The longer fibres were cut with surgical scissors to be exactly 50mm long (Figure 2.4: Methodology). The small light curing oven used for special tray manufacture was initially tried for the polymerization that the manufacturers required. Finally the pre-impregnated fibres were light polymerized for 2 minutes using a curing light (Megalight Mini, Radeburg, Germany).

4.2.3 Manufacturing of the specimens

In order to adhere to the protocol and limit unnecessary variations in specimen design and compilation manufacturer's instructions had to be followed carefully. These instructions demanded that the PMMA is mixed and left to reach the dough stage before packing inside the mould. The dough consistently pushed the fibers from their correct position when the mould was closed and pressure was applied (Figure 2.5: Methodology). A problem such as this one has not been mentioned in the literature of similar studies (Dogan et al., 2007; Bertassoni *et al.*, 2008; Fajardo *et al.*, 2011). At first it was attempted to close the mould lid extremely slowly (22 minutes per closure) so as not to dislodge the fibres. This did not work.

The solution was finally to manufacture the specimens in 2 stages. The cavities were filled with a first layer of PMMA-dough up to the level of the slots where the ends of the fibre bundles were to be positioned. The original protocol described a single stage procedure with a flat cover. This proof-packing required an additional mould cover to be made. This additional cover was made with platforms that protruded into the cavities of the base up to the level of the stops (Figure 2.1: Methodology).

A second mixture using the same P/L ratio was mixed and immediately poured over the fibres to overfill the cavities (Figure 2.8: Methodology). This second layer was left to "dough" in situ for the required 15 minutes before slowly closing the mould with the flat cover and applying pressure.

The mould was then compressed in a laboratory press (Figure 2.9: Methodology). As before, to avoid the risk of displacing the fibres, the press was closed very slowly. Every batch was polymerized for 20 minutes at 100^0C. Thereafter the mould was allowed to bench cool to room temperature before opening. The specimens were inspected and

checked for voids, cracks, bubbles and for foreign objects incorporated in the PMMA.

4.2.4 Cyclic loading

Fatiguing of specimens is done by a process of cyclic loading where a repeated force is used to simulate use of the material in clinical conditions.

Fatigue, and the testing thereof by means of cyclic loading, is a specialized field in science. Due to the nature of dentistry, the specimens for testing are small and often fragile. This requires machines sensitive enough to produce, and reliably analyze relatively small forces.

Outside the Western Cape region, the CSIR in Pretoria had the necessary equipment and scientists able to operate the equipment. Also, the CSIR has a project to assist academic institutions in performing research projects and agreed to help with this study at a reasonable fee.

Vallittu (2006) describes 'fatigue strength' of a material as the highest stress that a material can withstand for 10^7 times. Testing specimens at such a high number of cycles poses a challenge in the laboratory milieu. The number of cycles per second must be kept low enough to prevent heat generation in the specimen. Thus, at 2 Hz, 57.8 days are required to fatigue one specimen for 10^7 times. However, in a review article, Naumann *et al.* (2009) found that a protocol using 10^4 cycles at 50 N and 5 Hz satisfactory simulated a year of function in dental materials. Cyclic load was thus applied for 10^4 cycles at 5 Hz. Each specimen was fatigued 10,000 cycles.

4.2.5 Flexural testing

Random specimens were selected from all 3 P/L ratio groups, both fatigued and non-fatigued. These pilot specimens were subjected to a 3 point bending test by the CSIR. All the specimens tested in all three P/L ratio groups displayed an adhesive bond failure between fibre and PMMA. Macroscopically it was noticed that a void surrounded the fibre bundle. This was an unexpected finding as the literature essentially stated the opposite (Bertassoni *et al.*, 2008). These results were in direct conflict with other studies and research papers read by the author except for a study done by Ladizesky *et al.* (1993) where they found that delamination may occur during some processing stages. However, the tests were conducted with highly drawn linear polyethylene (HDLPE) fibre, and not glassfibres as in my study.

The CSIR compared the mean FS of this pilot sample of specimens and there was no significant difference in different P/L ratio groups. The association with the failure pattern (Figure 3.1: Results), together with the lack of difference in FS results, were suggestive of the fact that the different mixtures of PMMA did not differ significantly in strength due to any interaction with the fibres. Of course as the fibres were lying in a void in the acrylic resin this was not surprising.

These unexpected preliminary results prompted an investigation into potential reasons for the adhesive failures encountered during piloting, as the aim of this study was to determine the influence of P/L ratio on the strength of the fibre re-inforcement of heat cured PMMA. This implied an efficient bond between fibre and matrix as pre-condition. At this stage it was suspected that the nature of the failure of the specimens was related to some step in the manufacturing process of the specimens.

These preliminary findings were communicated to the company who had read the research proposal prior to the study and then supplied the fibres.

CD's with images explaining every step of the process, my protocol and proposed methodology as well as a number of specimens of each P/L ratio were sent to the manufacturers in Finland.

Following suggestions from scientists from the manufacturing company, several issues were explored:

1. *The 2-stage method: The same PMMA but at different dough stages on each side of the fibre bundle were packed into the same cavity. Refer to communication with Pasi Alander - 6/2/2011 & 6/5/2011 (Addendum C3 and addendum C5).*

   Following this comment, specimens were made using the 2 stage technique, without fibre reinforcement. All the specimens were fractured using the 3 point bending test. There was no difference in the FS. No voids or air bubbles were noticed at the fracture interface or on the outside of the specimens where the 2 layers joined. This was suggestive that the PMMA at different dough stages was not the reason for the void formation along the fibre.

2. *The amount of monomer. Refer to communication with Pasi Alander - 5/13/2011 (Addendum C4).*

   The 3 groups with the different P/L ratios, including the group with the recommended ratio, had the same adhesive failure pattern.

3. *Partial polymerization of the matrix of the fibre bundle due to heat fluctuation during transport. Refer to communication with Pasi Alander - 6/5/2011 (Addendum C5).*

   A new batch of fibres in a special cooler box with controlled temperature was sent from Finland. New specimens were manufactured. A random selection of these specimens was subjected to the 3-point breaking test. Again there was a 100% adhesive bond failure between fibre and PMMA.

4. *Compatibility of the PMMA and the fibre. Refer to communication with Pasi Alander* - 7/12/2011*(Addendum C6).*

   Three different heat-polymerizing PMMA were used to manufacture the specimens. Again there was a 100% adhesive bond failure between fibre and the 3 PMMA's.This lead to the assumption that the presence of a fibre was instrumental in the formation of the void.

5. *Polymerization cycle.*

   The polymerization cycle was modified as follows:

   a. Heat-polymerization by means of carefully controlling the temperature at 98°C, just below the boiling temperature of the water in the water bath. The rationale behind this is: should any gas develop during polymerization, that this would be limited to a minimum.

   b. Heat-polymerization at a lower temperature, but instead of the recommended 20 minutes, a conventional polymerization time of 6 hours was chosen. The rationale behind this was to establish whether the metal mould was maybe interfering with the heat transfer to the specimens and thus slowing down the proper polymerization.

   Changing the polymerization cycle did not influence failure pattern between fibre and the PMMA.

This piloting exercise consumed another batch of fibres. When the results of the piloting were communicated with the manufacturer, the manufacturer admitted that it was not known if the fibres used for this project were suitable to be used for heat-polymerizing PMMA. *Refer to communication with Pasi Alander 8/1/2011(Addendum C7)*

The manufacturer agreed to send different, non-preimpregnated fibres with a proven history of cohesive bonding between fibre and both cold- and heat-polymerizing PMMA. The handling of these fibres is different and more difficult compared to the impregnated fibres. *Refer to communication with Pasi Alander 8/31/2011(Addendum C2)*

The complete experiment was repeated using the batch of un-impregnated fibres. Regardless of the eventual outcome these specimens were to be accepted as the final specimens for testing.

4.3 Discussion of the results

The Stick Fibre is a unidirectional glass fibre bundle and should be used where high strength is needed for instance in full dentures or in composite bridge frames. (Figure 2.14: Methodology)

According to instructions 'wetting' of the fibres with a slurry of sloppy PMMA is very important. This is not easy as the fibres separate when they are wetted and are then difficult to handle and position correctly. However, this was overcome and this type of fibre became the one used for the final methodology.

The fatigued and unfatigued groups each had a cohort of specimens of 100%, 90% and 80% P/L ratio and were all re-inforced with the un-impregnated fibres.

Fatiguing was done at the CSIR Laboratories, while the 3-point breaking tests were done at the Dental Faculty of the University of the Western Cape.

Analysis of results was done using only specimens that were intact (no bubbles, cracks, voids) and that did not display extraordinary readings (machine malfunction, computer glitches etc). This is the reason why the groups have slightly different numbers of specimens. Some specimens were also lost due to operator error while working the universal testing machine.

4.3.1 Macroscopic fracture patterns.

On examination it was found that the specimens with the un-impregnated fibres failed adhesively, the same failure pattern encountered as for the pre-impregnated fibres during the first piloting process. Every specimen fractured with the fibre debonding from the PMMA. Again it was found that the fibres lay in a void inside the heat cured PMMA. These voids appeared larger than the diameter of the fibre bundle.

As the aim of this study was to assess the influence of the fibre on the strength of heat cured PMMA with and without fatiguing, this observation would inevitably complicate answering the hypotheses.

However, an attempt was made to carefully examine the specimens and at least see whether certain trends could be observed among the different specimen groups.

4.3.2 Strength

Prior to testing, the trapezoidal cross-section of the specimens as they emerged from the mould, was machined and finished into a rectangular shape. This was done at the CSIR. A certain variation in width and height was noticed. The influence of this variation was examined and found to be a confounder (Figure 3.6: Results). The distribution of height width and length in the various mixratio/ fatigue subgroups was measured and plotted.
Notice that the 80% fatigued and unfatigued values do not even overlap.

Figure 3.7 (Results) shows boxplots of deflexion for the different ratio and fatigue subgroups. Within the fatigued = Yes and fatigued = No groups the trends, with the ratio are similar: mean deflex drops quite sharply from 80% to 90% groups and then does not change much from 90% to 100%. This is clear, i.e. the thinner mix bends more. Williams *et al.* (2001) also found that changing P/L ratio of four auto-polymerizing PMMA resins may have deleterious effects on the properties of the polymerised material: A lower P/L ratio resulted in significantly lower surface hardness and higher flexibility.

A boxplot (Figure 3.3: Results) of the subgroups using F max and ratio and fatiguing in the different subgroups shows a different trend. With specimen = No (unfatigued) the Fmax increases with the increased ratio, while with the specimen = Yes (fatigued) the trend is essentially not as clear. This is where the different confounders play a role.

To standardize these results a formula was used to find a covariate measurement for thicklr which could lead to an accurate calculation of actual strength of the specimens.

Flexural strength was used to standardize the measurements in all the three subgroups of groups 1 (Fatigued) and 0 (un-fatigued).

The results of the two groups (Table 3.21: Results) clearly demonstrate that the unexpected reversed trend illustrated in Figure 3.3 has now been corrected. In both fatigued and unfatigued specimens there is now a slight rise in strength from the 100% to the 80% mixture.

The fact that this study showed that the fibres do not actually adhere to the PMMA makes this result surprising. One would expect the specimens with the higher P/L ratio to be stronger. It can be postuated, however, that the more viscous 80% mix did in effect impregnate the fibres ever so slightly more than the stiffer 90% and 100% mixes of PMMA. This would explain the higher FS of the 80% mix in both the unfatigued and fatigued specimen groups.

The increase in FS after cyclic loading, however, is an interesting trend to explore further in future studies. Could it be that cyclic loading results in an initial pseudo tempering of the acrylic?

4.3.3 Comparison of results with other studies

There are very few studies and research projects that concentrate on the fiber strengthening of heat-cured denture PMMA. Possibly this is because of the difficulty of using these fibres in PMMA that is, per definition, very thin and rarely exceeds 3.5 mm in thickness. In a study that compared heat-cure and microwave-cured PMMA fibre reinforced specimens Tacir *et al* (2006) found that strengthening with fibers lowered the flexural strength of the specimens but increased the flexural resistance. This compares favourably with this study. In this research study the manufacturer's instructions for all materials and fibres used was followed to the letter. The results are as published. In his 1999 study, Vallittu (1999) however placed great emphasis on the impregnating of the fibre bundles with monomer prior to use. It is possible that this change in the methodology allowed him to record the results he achieved.

4.4 Limitations and further research

In vitro studies have several limitations. The specimens are usually symmetric, unlike the variation and curvatures found in natural dentures. This is purposely done to control geometric variables and allow consistent loading on a flat surface in the same location for each specimen. The loading should also be consistent with other studies.

Clinical performance versus lab testing is a problem that has dogged researchers for a long time.

Clinical performance is classically defined in terms of safety and effectiveness. Dr. Gunnar Ryge, while in the employ of the United States Public Health Service (USPHS), came up with the most famous of the rating scales. This was considerably extended and the Modified USPHS Scale for Clinical Performance and Acceptability (Bayne, 2007) can now assess almost any dental procedure and material. Ryge isolated five variables or factors that he logically felt may describe many influences on clinical outcome. They include operator factors, design factors, material factors, intra-oral location factors and patient factors (Bayne, 2007).

As an adjunct to this, exists the method known as ‘practice based research’ where the research is actually carried out in a real surgery environment of the dental practice. Of course here the clinicians’ different treatment decisions and their variations in assessment of clinical quality are a huge hurdle and a factor to be considered (Mjor, 2007).

It could be speculated that the relative difficulty and longer time it takes to fabricate the heat-polymerized specimens actually leads researchers to shun this group of materials in favour of the quicker and easier groups presented by the light- and auto-polymerizing resins.

The greatest limitation of this study is that the researchers could not achieve bond between the pre-impregnated C+B Stick fibres or the un-impregnated Stick fibres and the heat-cured PMMA. Up to this moment this result has not been explained either by this study or by the suppliers of the fibres in Finland. Not a single one of the ratio subgroups of either the un-fatigued or fatigued subgroups was significantly re-inforced by the addition of the fibres. This was clearly as a result of the debonding that took place between the heat-cured

PMMA and the Stickbond C+B and the Stick fibres. Despite numerous different approaches and techniques no method was found so far to successfully use Stick or Stickbond fibres with heat cure acrylic.

It could be argued that 'debonding' may be a misnomer as there may not have been a bond to start with. Jagger *et al.* (2003) found the same in their study with treated PMMA fibres where impact strength, modulus of rupture, modulus of elasticity, transverse strength and F Max were all negatively affected by the addition of fibres.

Once it was established that the bonding of the fibres was a problem, it could have been a good idea to use specimens with no fibres included as an additional control. This would have established with certainty whether the fibres bonded or not.

A further limitation of this study could be the 10,000 cycle load cap in the fatiguing process. In a previous study (Diaz-Arnold *et al.*, 2008) it was found that the 10,000 fatigue cycles had little or no effect on 5 different materials that were compared. The number and frequency of the cycles was based on previously reported literature, piloting and test time constrains. The testing time of every specimen at 10,000 cycles was 33 minutes (over 41 hours for all specimens). With the high demand for testing equipment, there is a tendency to limit cycling frequency.

Within the two groups, 0 (un-fatigued) and 1 (fatigued), the wetter mix (80%) gave the highest FS, but the differences in the three subgroups were not significant. Therefore, the practitioner can change the P/L ratio to improve the handling for certain applications, without detrimental effect. Geerts and Du Rand (2009) also found no difference in FS for different ratios of un-reinforced chemically-cured PMMA. Since the PMMA used in my research did not bond to the fibres, the specimens could be regarded as 'un-reinforced".

All three the P/L ratios showed an increase in the FS after the fatiguing of the specimens from the 10,000 to the 20,000 cycle mark (Figure 4.1).This result was unexpected as the thought is that the cyclic loading (fatiguing) of the specimens would weaken them.

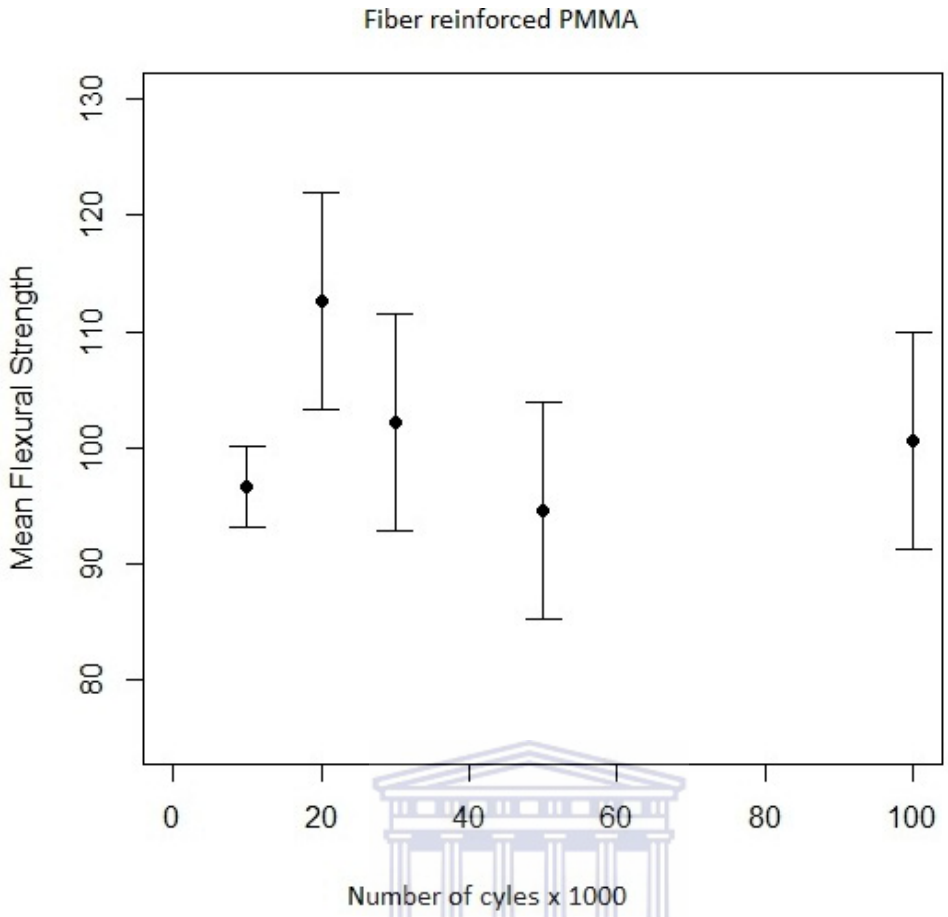

Figure 4.1: Plot of mean flexural strength against number of fatigue cycles showing an initial increase of strength with higher cycling

The interesting phenomenon found by the CSIR that the specimens actually got stronger after cyclic loading (fatiguing) cannot be explained satisfactorily. One possible explanation could be that gentle cyclic loading actually anneals and aligns the PMMA chains in a similar fashion that tempering strengthens metals.

The fact that this result was achieved with heat-cured PMMA could also have a bearing on this result.

Further tests with a possible more vigorous loading cycle could be undertaken, to investigate whether this trend is short-lived and just takes place at a relatively low number of cycles.

Although the fibres in all subgroups were macroscopically fully debonded, Table 3.20 (Results) shows clearly the fact that: the wetter the mix, the stronger the specimen. This could possibly mean that even though the fibres do not successfully bond to the heat-cured

PMMA, the wetter mixes of the PMMA do have a slightly better adhesion between fibre and heat-cure acrylic. By examining fracture patterns, Geerts and Du Rand (2008) also found that adhesion between the wetter mixture of cold-cure PMMA and the fibre bundle was more efficient. In the case of chemically-cured acrylic, it did not result in a higher FS value though.

Tacir *et al* (2006) suggested that even pre-impregnated fibres should be soaked in monomer for 10 minutes to allow for better bonding with the acrylic resin.

The recommended ratio proposed for the PMMA used in this study proved to be the weakest mix of the three used. It could be possible that within the parameters of functional strength the manufacturers actually suggested the use of a mix that incorporated more powder in the fluid leading to increased consumption of the product.
Due to the fact that the specimens are manufactured and finished by hand, a certain variation in thickness and width was found. However, when variations were discovered, these variations were compensated for in the analysis and interpretation of the data.

4.5 Conclusions and clinical relevance

After exhaustive testing and using different PMMA materials and glass fibre bundles it was found that ever Stick and Stickbond glass fibre bundles do not bond to heat cured PMMA when using the recommended protocol and methods used in this study.

No reason for this could be established and exhaustive correspondence with the manufacturers of both the fibres and the PMMA has shed no further light on this problem.
In this study it was concluded that the fibre re-inforcing of heat-cured denture bases with this type of fibre is ineffective.

In vitro fatiguing results must always be interpreted with care. The assumption that the material with the highest FS after fatiguing would be the best or most appropriate material for the job at hand is not necessarily correct. Decisions on the selection for the most appropriate material should always be made within the broader clinical context.

The results of this study showed that, either debonding of the fibres and the heat-cured PMMA used in the study took place, or no bonding ever took place between the fibres and

PMMA.

Due to the cost of the fibres used for re-inforcement, it is imperative that the system should work as proposed. This cannot be achieved by using fibres in a heat-cure PMMA during flasking and processing of dentures. Possibly it would be better to use the method described in the Stick Company instruction CD and use a cold cure acrylic to insert the fibres after the denture in heat-cure acrylic has been manufactured.

Finally, it may be concluded that with regards to the PMMA:

1. There is no significant interaction between Mix and Fatigue state.
2. The 80 % mix has a significantly higher mean FS than either the 90% or 100 % mix (with differences of about 4.4 and 7.5 units respectively).
3. The Fatigued state has a higher FS mean than the Not Fatigued state (by about 6.0 units).

4.6 Recommendations

The debonding of the fibres and the heat-cured PMMA or non- bonding between the fibres and PMMA was totally unexpected.
An additional study to examine other similar fibres from different manufacturers may help to identify a product that works optimally or to expose a flaw in the suggested use of these strengtheners.

FS (MPa) across all three P/L ratios increased after the fatiguing of the specimens from the 10,000 to the 20,000 cycle mark (Figure 4.1).This result was contrary to the widely held belief that the cyclic loading (fatiguing) of such materials would weaken them.
Research into this phenomenon could possibly lead us to be able to predict the behavior of acrylics used in dentistry more accurately.

The interesting phenomenon found by the CSIR that the specimens actually got stronger after initial cyclic loading (fatiguing) cannot be explained satisfactorily. One possible explanation could be that gentle cyclic loading actually anneals and aligns the PMMA chains in a similar fashion that tempering strengthens metals.
Research into this phenomenon could possibly lead us to be able to predict the behavior of acrylics used in dentistry more accurately.

The fact that this result was achieved with heat-cured PMMA and was not seen by Geerts and Du Randt (2009) in their research with self-cure acrylics could also have a bearing on this result.

Further tests with a possible more vigorous loading cycle could be undertaken, to investigate whether this trend is short-lived and just takes place at a relatively low number of cycles. Other different heat-cure PMMA materials could also be tested for comparison.

The recommended ratio proposed for the PMMA used in this study proved to be the weakest mix of the three used. Possible comparison with other dental acrylics would establish whether this was a single, product-specific finding or a definite characteristic of PMMA used for denture construction.

This researcher concluded that the fibre re-inforcing of heat-cured denture bases with this type of glass- fibre is ineffective.

No reason for this could be established and exhaustive correspondence with the manufacturers of the fibres and the PMMA used has shed no further light on this problem.

Correspondence with other manufacturers of glass fibre re-inforcing may shed light on this finding.

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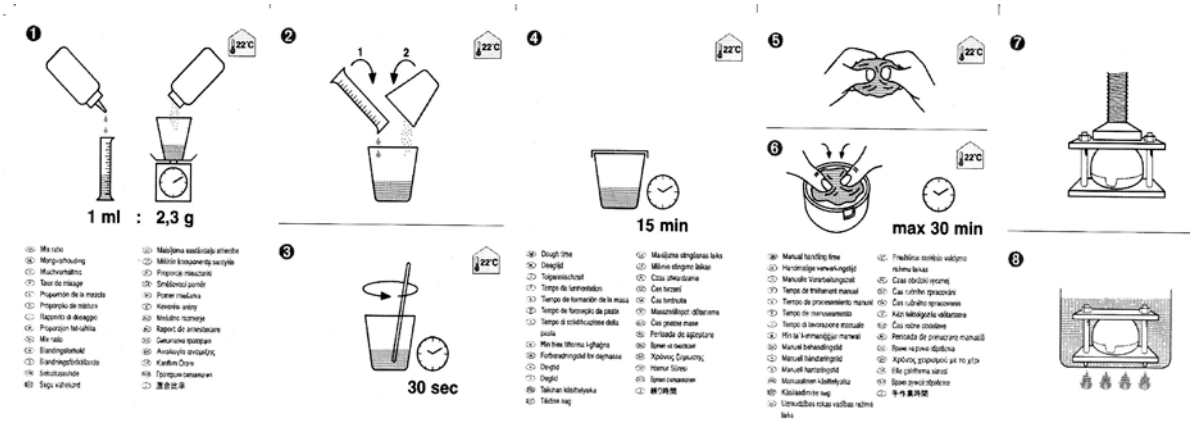
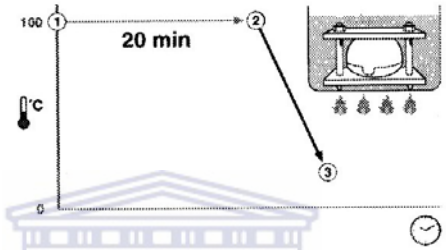
ADDENDA

Addendum A: Technical specifications for Vertex Rapid Simplified.

Dough time	15 minutes
Working time	30 minutes
Curing time	20 minutes at 100°C
Mixing ratio by volume / parts by weight	1 ml / 0.95 g liquid (monomer) 2.3 g powder (polymer)
Impact-resistance	11.3 kJ/m2
Flexural strength	85.2 MPa
Flexural modulus	2367 MPa
Water sorption	22.5 µg/mm3
Solubility	0.11 µg/mm3

Addendum B: Mixing instructions for Vertex Rapid Simplified Acrylic Resin:





Addendum C: Different letters of communication.

Addendum C.1

From: Pasi Alander <pasi.alander@sticktech.com>
To: Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date: 6/2/2011 12:12 AM
Subject: VS: comments about your samples
Attachments: 077.JPG

Dear Martin,
Sorry for late answer. I have been in a vacation. I had entrance examination in this week and before that I prepared for it.
I watched carefully your samples and CD with Prof Vallittu. We couldn't figure out any clear reason for this kind gap phenomenon. After that I spend more time with this dilemma and now come my guess. Reason is related to the two-step technique you use for filling the molds. There are two different unpolymerized acrylics in the mould. Those are in the different polymerization stage when thinking polymerization and time. The line between different "stage" acrylics is seen in some samples or pictures, like 077. I have got air bubbles in the denture repairs when mixing acrylics which are made in the little bit different time.
Please let me know if you and Prof Geerts agree this comment. I can't figure any other reason for the gaps. Even you but the rubber band in to the acrylic, there should not be any visible gap between the acrylic and rubber band after the polymerization. this is mystery for me.
Best regards
Pasi

Pasi Alander
Product Manager

Stick Tech Ltd
P.O.Box 114
FI-20521 Turku
FINLAND

Phone : +358 2 4808 2500
Mobile: +358 40 9000 754

E-mail: pasi.alander@sticktech.com<mailto:pasi.alander@sticktech.com>
www.sticktech.com<http://www.sticktech.com/>
www.puuttuvahammas.fi<http://www.puuttuvahammas.fi/>

Lähettäjä: Martin Stuhlinger [mailto:mstuhlinger@uwc.ac.za]
Lähetetty: 27. toukokuuta 2011 10:21
Vastaanottaja: Pasi Alander
Aihe: Re: comments about your samples

Addendum C.2

From: Pasi Alander <pasi.alander@sticktech.com>
To: Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date: 6/5/2011 10:45 PM
Subject: VS: VS: comments about your samples

Dear Martin
some comments from me too. Now when you mentioned that fibres really dry, like matrix have gone, I started to think if the acrylic is dissolving the matrix away. This can happen if fibres are too long time inside unpolymerized acryl. But what makes empty space around them. I don't know.
I was also wondering if the two stages of the acrylic will do the porosities in to the acrylic. This porous might be as a one big empty area around the fibre. Does the two stage acrylic technique affect to the acrylic strength values, I don't know. This can be tested by fabricating some control samples with two different method. In a first group the mould is filled once with acrylic and in a other group with two step technique.
Also the reason can be that the heat can polymerize the fibres during the transportation. everStickC&B fibres should be totally flexible when using those. There should be also thin oxygen inhibition layer around the fibre bundle after light polymerization. Don't use vacuum or place fibres inside the silicone while polymerizing those with light. We can send new fibres for you with the data clocker. It will tract the temperature of the parcel from here to you.
Your test sample size is so big that you should put more fibres in to the test samples. Now the fibre amount by volume is less than 2 %. This is not enough for getting proper reinforcement effect. You will need 2-3 bundles at least in one test sample to find out differences between the control group without fibres and reinforced group. Is that possible? Samples can also be smaller if possible. As I probably told you earlier, by adding the stick fibres also to this study it will be more informative. But maybe too much work with this little time.
We can send the fibres directly for you by TNT, it needs your address and phone number.
Best regards
Pasi
Pasi Alander
Product Manager

Stick Tech Ltd
P.O.Box 114
FI-20521 Turku
FINLAND
Phone : +358 2 4808 2500
Mobile: +358 40 9000 754
E-mail: pasi.alander@sticktech.com<mailto:pasi.alander@sticktech.com>
www.sticktech.com<http://www.sticktech.com/>
www.puuttuvahammas.fi<http://www.puuttuvahammas.fi/>

Lähettäjä: Martin Stuhlinger [mailto:mstuhlinger@uwc.ac.za]
Lähetetty: 3. kesäkuuta 2011 14:52
Vastaanottaja: Pasi Alander
Aihe: Re: VS: comments about your samples

Addendum C.3

From:	Pasi Alander <pasi.alander@sticktech.com>
To:	Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date:	8/1/2011 2:46 PM
Subject:	VS: my newest results.
Attachments:	5 1018 Stick product family updated 2011_04 low res.pdf

Dear Martin
I just came back from summer vacation. I do not have clear answer to this problem. Reason can be that everStick fibres are not working well with heat cured acrylic.
The acrylics we have used with everStickC&B fibres are mostly self-cured acrylics, like Palapress from Heraeus Kulzer. We do have that much experience with heat cured acrylic products. Test with everStickC&B and heat cured acrylics have not been done, because the other our fibre, named Stick, is fully tested. These tests showed that it will work well with both types of acrylics (self and heat cured). That makes us believe the same with everStickC&B. You have proved that we were wrong. everStickC&B fibre can be used with self-cured acrylic, but is maybe not suitable for heat cured acrylics. It is very valuable information for us.
We do still have reinforcing product for heat cures acrylic. it 's name is Stick. See the different fibre products dental laboratories in the attachment.
Best Regards
Pasi

Addendum C.4

From: Pasi Alander <pasi.alander@sticktech.com>
To: Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date: 8/31/2011 12:33 PM
Subject: VS: VS: VS: my newest results.

Dear Martin
Nice to hear that can continue your study.
I want highlight one more time that Stick fibre need wetting with slurry acrylic mixture before placement. I hope this is not problem for the research question/topic. Use metal instrument for manipulating fibres during the wetting to ensure the proper wetting.
Both everStickC&B and Stick have 4000 single fibres in one bundle. But Stick fibre needs more hand skills than everStickC&B. After wetting all 4000 single fibres are loose from each other, because wetting acrylic will dissolve totally the porous PMMA matrix of Stick fibre. Fibre bundle will swell also some amount. The final diameter will be more than 1.5mm. This makes the handling little bit tricky. Use two tweezers (both ends) to lift the fibres in to the right position. I just want to inform you beforehand these things, which might affect for sample fabrication.
I will start my on study at October. I will be totally away from work more than one year.
Please contact to the Eija Säilynoja if more information is needed.
You will get the e-mail when we sent the fibres to you.
Best Regards
Pasi

Pasi Alander
Product Manager

Stick Tech Ltd
P.O.Box 114
FI-20521 Turku
FINLAND

Phone : +358 2 4808 2500
Mobile: +358 40 9000 754