AN IN VITRO INVESTIGATION OF THE FLEXURAL STRENGTH AND MICROSTRUCTURE OF “STICK GLASS FIBER” AND “WIRE MESH” REINFORCED HEAT CURED DENTURE BASE ACRYLIC

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SEPTEMBER 2008

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This study represents original work by the author and has not been submitted in any form to another University. Where use was made of the work of others, it has been duly acknowledged in the text.

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DEDICATION

This study is dedicated to noble intuitive minds, which are essentially, illuminated to scientific phenomena.

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"If I have seen further than others, it is by standing upon
the shoulders of Giants"

(Isaac Newton, 1642–1727)

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ABSTRACT

Globally in the field of Dental Technology, polymethyl methacrylate (PMMA) resin continues to be the popular material for the fabrication of denture bases in removable prosthodontics. However, the mechanical strength of the denture base is a concern due to fractures occurring intra-orally or when accidentally dropped.

The objective of this in vitro investigation was therefore to evaluate and compare the flexural strength and microstructure of stick® glass fibre and wire mesh reinforced PMMA resin after thermocycling. The selection of the materials used in this study was based primarily on their popularity and availability in South Africa. These materials were selected to ensure that the results of this study would have further implicational value in the commercial dental industry when published.

This investigation was conducted by means of fabricating a total of 90 PMMA resin specimens and divided in three groups consisting of 30 specimens each. Sample groups 1 and 2 were reinforced with stick® glass fibres and wire mesh respectively. The un-reinforced sample group was the control. All 90 specimens were thermocycled in water at temperatures between 5˚C and 55˚C for 2100 cycles. The flexural strength of each specimen was tested using a universal testing machine and the microstructure of the fractured surfaces was then analysed using scanning electron microscopes (SEM).

SPSS version 15.0 was used for data analysis. A p-value of <0.05 was considered as statistically significant. Data were analysed using parametric and non-parametric statistical methods. Statistically significant differences in flexural strength existed between the three sample groups ($p<0.001$) with the stick® glass fibre and wire mesh sample groups being significantly superior to the control.

Furthermore there was a significant association between fracture modes and sample groups.

Microscopic analysis revealed the presence of voids. Statistically, in terms of microstructure (% of voids present), a significant difference existed between all sample groups. With regards to surface texture of the compression and tension sides of the test specimens, significant differences existed between the three sample groups. Furthermore microscopic analysis revealed partial impregnation and distribution of the fibres to the PMMA resin matrix and un-bonding between the wire mesh and PMMA resin matrix. Statistically, the Mann-Whitney test was conducted to compare flexural strength between sample groups with and without voids. The flexural strength was higher in sample groups with voids than those without.

This is an important finding from the clinical perspective because, in some structures of dentures, toughness is a desired property. Nevertheless in order to find the long-term data especially on clinical behaviour of these new fibre reinforcement systems, more studies should be conducted.

TABLE OF CONTENTS

CHAPTER ONE: THE PROBLEM AND ITS SETTING

PAGE

PAGE

CHAPTER TWO: REVIEW OF THE RELATED LITERATURE

CHAPTER THREE: METHODOLOGY

CHAPTER FOUR: RESULTS

CHAPTER FIVE: DISCUSSION

PAGE

CHAPTER SIX: CONCLUSION AND APPLICATION OF FINDINGS

LIST OF TABLES

LIST OF FIGURES

LIST OF GRAPHS

LIST OF PLATES

APPENDICES

CHAPTER ONE

The grand aim of all science is to cover the greatest number of empirical facts by logical deduction from the smallest number of hypotheses or axioms

Albert Einstein (1879-1955)

1. THE PROBLEM AND ITS SETTING

1.1 INTRODUCTION

Denture base fracture, particularly poly (methyl methacrylate) (PMMA) resin denture base material in removable dentures, is an unresolved problem in the dental industry (Jagger, Allen and Harrison, 1999). PMMA resin is the most commonly used material in the fabrication of denture bases (El-Sheikh and Al-Zahrani, 2006; Kim and Watts, 2004; Rantala, Lastumaki, Peltomaki and Vallittu, 2003). The material however, is not ideal in every respect and it is the combination of some properties rather than one single desirable property that accounts for its popularity (Winkler and Apellbaum, 1984). The forthcoming chapter will discuss the properties which have led to the popularity of PMMA resin denture base material.

Fracture of PMMA resin denture bases in clinical use is time consuming and costly to both patient and dental professionals. Most PMMA resin denture base fractures result from two different types of forces, namely, flexural fatigue and impact (Lonĉar, Vojvodić, Matejiĉek and Jerolimov, 2006; El-Sheikh and Al-Zahrani, 2006). Flexural fatigue occurs after repeated flexing of the PMMA resin during clinical use. The flexural fatigue is a mode of fracture whereby the denture base fails after being repeatedly subjected to mastication loads. According to El-Sheikh and Al-Zahrani (2006) impact failure of dentures (which is delimitated in this study) usually occur out of the mouth as a result of sudden blow or accidentally dropping on hard surface during cleaning, coughing or sneezing.

Flexural strength of PMMA resin is therefore crucial for the success of a denture base during clinical use. PMMA resin denture base material can be reinforced with

either fibres or wire mesh to increase its flexural strength (Lonĉare, *et al*. 2006; Narva, Lassila and Vallittu, 2005a; Nohrström, Vallittu and Yli-Urpo, 2000; Jagger, *et al*. 1999). Hence this study investigates the flexural strength and microstructure of heat cured PMMA resin reinforced with stick® glass fibres or wire mesh. All the specimens will be exposed to thermal changes simulating conditions of the oral environment.

Research in the past decade has concentrated on the flexural strength of reinforced PMMA resin denture bases. However, in terms of flexural strength and microstructure there is no systemic data available in the literature to assess the actual service performance intra-orally. In addition thermal stress is crucial to provide systemic data on intra-oral performance of reinforced PMMA resin (Nohrström, Vallittu and Urpo-Yli, 2000). Furthermore, testing the performance of dental materials and specifically PMMA resin has been done, mostly, under ideal environment and equipment without reflecting performance in actual application.

The recent introduction of the stick® glass fibre reinforcements to the South African Dental industry further supports the need for more research in reinforced PMMA resin (Visser, personal communication. 23 May 2006). Currently in the South African Dental industry the use of wire mesh as a mode of reinforcing dentures is still common. Nevertheless, in this era of metal free restorations, there is need to understand more on the contemporary application of Fibre Technology in dentistry (Van Rensburg, 2006). The results in this study may provide valuable technical information to the dental laboratory industry, both in training and practice. More importantly the results may further assist dental manufacturing companies and dentists when planning treatment for their patients.

Although many investigators question the reliability of an in vitro approach, this method was nonetheless used for this study. In addition, the in vitro approach may provide additional data into the effects of thermal stresses on the flexural strength

and microstructure of reinforced PMMA resin. The results will further serve to have application for the future *in vivo* studies.

1.2 STATEMENT OF THE PROBLEM

The purpose of this in vitro investigation is to determine the flexural strength and microstructure of stick® glass fibre and wire mesh reinforced heat cured PMMA resin.

1.3 THE OBJECTIVES

The following are the three objectives to be investigated in this study:

1.3.1 Objective One

To evaluate and compare the flexural strength of stick® glass fibre and wire mesh reinforced heat cured PMMA resin in order to identify parameters for comparison with the control.

1.3.2 Objective Two

To determine the microstructure of stick® glass fibre and wire mesh reinforced heat cured PMMA resin in order to identify parameters for comparison with the control and further ascertain the distribution, impregnation and bonding of the reinforcements to the PMMA resin matrix.

1.3.3 Objective Three

To determine whether a relationship exists between the flexural strength and microstructure of the stick® glass fibre and wire mesh reinforced heat cured PMMA resin.

1.4 THE HYPOTHESES

The following are the three hypotheses to be investigated in this study:

1.4.1 Hypothesis One

It is hypothesised that the stick® glass fibre and wire mesh reinforced PMMA resin will produce different levels of flexural strengths to the control.

1.4.2 Hypothesis Two

It is hypothesised that the stick® glass fibre and wire mesh reinforced PMMA resin will have differences in microstructure to the control, and thereby set parameters for comparison.

1.4.3 Hypothesis Three

It is hypothesised that a relationship exists between flexural strength and components of microstructure of stick® glass fibre and wire mesh reinforced heat cured PMMA resin.

1.5 THE ASSUMPTIONS

1. The polymer and monomer utilised in this study were assumed to be materials meeting the International Standard Organization (ISO, 1567:1999) criteria.
2. It was assumed that the stick® glass fibres and wire mesh were flawless.
3. It was assumed that the thermocycling machine used in this study
   - is of a high precision engineering;
   - will maintain regulated temperature, dwell time and cycles.

4. It was assumed that thermocycling temperatures will influence the flexural strength and microstructure of the PMMA resin denture base material.
5. It was assumed that the differences in coefficient of thermal expansions of the stick® glass fibre, gilded wire mesh and PMMA resin will create stresses between the interfaces.

1.6 THE DELIMITATIONS

This study will not examine the influence of the effects on:

- Polymerisation cycles
- Thermocycling stresses
- Water absorption levels
- Level of residual monomer
- Grinding and polishing stresses
- Chemical composition of PMMA resin, stick® glass fibre and gilded wire mesh.
- Type of thermocycling medium.
- The mechanical properties of the materials in relation to their chemical composition.
- The release of residual monomer into water from stick® glass fibre/PMMA resin composite

Although the findings of this study may have applications in Dental industry, the study will not evaluate the:

- Effects of different types of glass fibres on flexural strengths of PMMA resin.
- Effects of different types of metal strengtheners on flexural strength of PMMA resin.

- Flexural strengths of different types of acrylic resins used as denture base materials.

The study is aimed at testing only the flexural strengths of the samples and not any other types of strengths such as impact.

Due to the time and cost constraints of the study, the investigation was limited to 2100 thermocycling cycles. This is equivalent to three months clinical service of a PMMA resin denture base.

Pre-treatment methods of wire mesh to improve adhesion, such as silanization of metal, sandblasting of metal wire with Al_2O_3 and metal adhesive resins, will not be applied in this study.

1.7 DEFINITION OF TERMS AND CLARIFICATION OF CONCEPTS

1.7.1 Denture Base

Denture base is that part of a denture (whether metal or resinous material) that rests on the foundation tissues of the oral cavity and to which the artificial teeth are attached (McGivney and Castleberry, 1995:5).

1.7.2 Prosthodontics

This denotes the branch of dental art and science that deals specifically with the replacement of missing dental and oral structures (McGivney and Castleberry, 1995:4).

1.7.3 Prosthesis

Prosthesis is an engineered device replacing or restoring tissue or organs but not normally exposed to body fluids such as blood etc. The plural is prostheses (Walker, 1999:923).

1.7.4 Polymer

A material that is made up of repeating units, or mers, most polymers are based on carbon (-C-C-C-C-) backbone in polymer chain, although Silicone (-O-Si-O-Si-O-) backbone is important in many polymers (O'Brien, 2002:11).

1.7.5 Glass Transition Temperature (Tg)

This is the temperature at which a sharp increase in thermal expansion coefficient occurs, indicating increased molecular mobility (Anusavice, 2003:21). The glass transition temperature of PMMA denture base acrylic is a thermal range in which the polymerised resin passes from soft, rubbery state to a rigid, glass state. The glass transition temperature of PMMA is approximately 105˚C (Anusavice, 2003:740).

1.7.6 Linear Coefficient of Expansion

This is the relative linear change in length per unit of initial length during heating of a solid per ˚K within a specified temperature range (Anusavice, 2003:21).

1.7.7 Isotropic

A material is isotropic when it exhibits uniform properties when measured along axes in all directions (Smith, A., Datta, Smith, G., Campbell, Bentley and McKenzie, 1997:349).

1.7.8 Rheometric Properties

This is the flow behaviour of solid polymers which involves a combination of elastic and plastic deformation (Anusavice, 2003:150).

1.7.9 Coefficient of Variation

This is the ratio of the standard deviation to the absolute value of the mean, expressed as a percentage (Kuzma and Bohnenblust, 2005:62).

1.7.10 Flexural Strength

Flexural strength, transverse strength, or modulus of rupture, as this property is variously called, is essentially a strength test of a bar supported at each end, under a static load. The mathematical formula for computing the flexural strength is as follows:

$$\sigma = 3pl/2bd^2$$

Where σ is the flexural strength, l is the distance between the supports, b is the width of the specimen, d is the depth or thickness of the specimen, and p is the maximum load at the point of fracture (Anusavice, 2003:89; Phillips, 1991).

1.7.11 Anisotropic

Describes a medium in which physical properties, such as the velocity of electromagnetic radiation transmission, electrical or heat conductivities, or compressibility have different values when measured along axes in different directions (Smith, *et al*. 1997:40).

1.7.12 Compression, Tensile and Shear Stresses

A material is subjected to compressive stress when the material is squeezed together or compressed. Tensile stress occurs when a material is pulled apart. Shear stress occurs when a material is forced to slide by another portion. These types of stresses are considered for evaluation of the mechanical properties of various materials (Craig, Powers and Wataha, 2000).

1.7.13 Modulus of Elasticity

This is the measure of the stiffness of a material (Craig, *et al*. 2000:22).

CHAPTER TWO

Literature is strewn with the wreckage of men who have minded beyond reason the opinions of others

Virginia Woolf (1882-1941)

2. REVIEW OF RELATED LITERATURE

2.1 INTRODUCTION

The clinical performance and success of removable dentures with PMMA resin denture base is dependent upon the mechanical and physical properties of the material. The resistance to biodegradation of PMMA resin when exposed to physiological conditions of the oral environment further influences the life span of the prosthesis. The primary concern however is the denture base's poor strength characteristics, which are the low flexural fatigue resistance and impact strength (Lonĉare, *et al*. 2006; El-Sheikh and Al-Zahrani, 2006).

In dentistry, denture bases are commonly fabricated using acrylic resins or metal alloys. A denture base is that part of a denture (Figure 2.1), which rests on the soft tissues of the oral cavity and does not include the artificial teeth (McCabe and Angus, 1998:96).

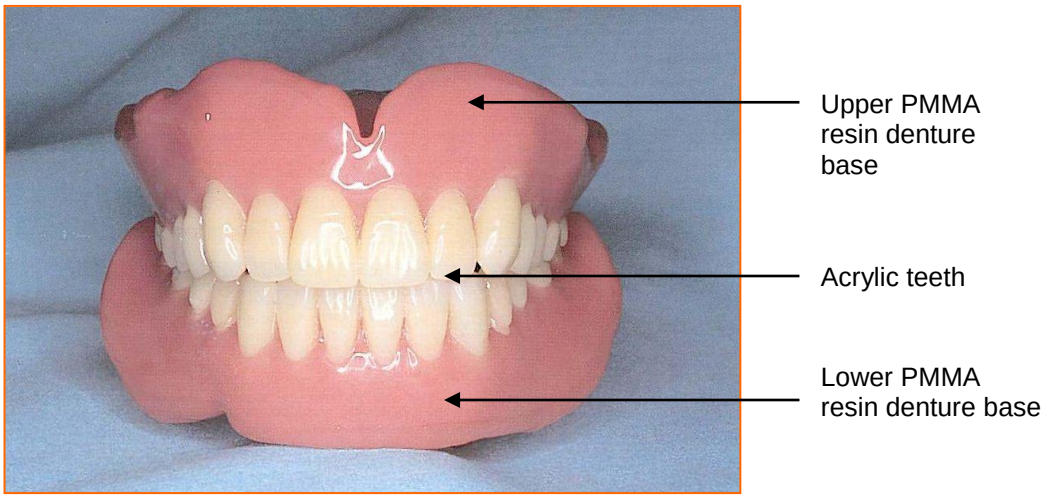


Figure 2.1: Complete upper and lower denture (Adapted from Hayakawa, 2001:222)

According to Dale and Ascheim (1993:200) denture bases in removable prosthodontic work function as a:

- retaining mechanism to attach artificial teeth and to attach retainers to abutment teeth
- labial flange to restore lost bone contours
- connecting device to hold together all elements in the prosthesis
- support for the lip form and position (especially the labial and buccal flanges).

The ensuing review of related literature provides the historical background of denture bases and the global use of the different denture base materials in dentistry. In addition, specific emphasis is given on the advantages and disadvantages of the different materials used as denture bases for several decades.

2.1.1 Historical Background

Historically, a variety of materials have been used to replace human lost teeth. Animal teeth and pieces of bone were among the earliest of these primitive

materials (History of dentures Online, 2006-08-03). In the 1800s, denture bases were fabricated using the art of hand carving ivory or wood, (Wulfes, 2004; O'Brien, 1997; Dale and Aschheim, 1993). According to Van Noort (1994), some denture bases were retained in the patient's mouth by use of mechanical devices such as springs or by tying them to the remaining teeth with silk threads (Figure 2.2). However, retention of hand-carved and spring retained denture bases gravely diminished as more teeth were lost from the patient's mouth. Consequently, this necessitated for the development of superior denture base materials which could have a better retention in the oral cavity. In the mid 19th century to 1930s other materials such as, Nitrocellulose, Phenol formaldehyde, Vinyl plastics, Porcelain, Polycarbonates, Polyamides and Vulcanite were used for denture bases (Manappalill, 2003:99).

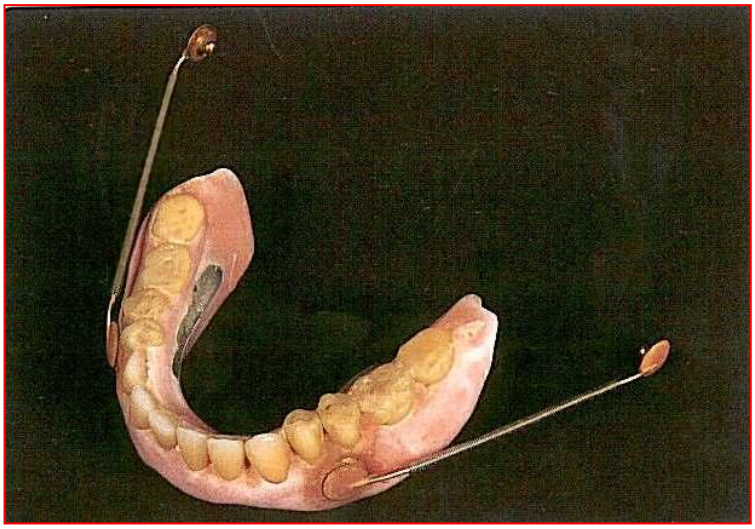


Figure 2.2: Lower denture with springs (Adapted from Wulfes, 2004:22)

The use of Nitrocellulose products as a denture base was limited due to its tendency to warp during function and leaching of the plasticizer contained in the material into the oral environment (Van Noort, 1994). Furthermore the leached plasticizer caused blistering of the oral tissues, staining and loss of colour of the denture base within a few months of clinical function. The use of Phenol

formaldehyde (bakelite) as a denture base material diminished due to technical difficulties during fabrication and also rapid loss of colour of the denture base during clinical use (Manappalill, 2003:99).

Vinyl resins as denture base materials were found to have a low resistance to fracture and failure of dentures were common possibly due to flexural fatigue (Van Noort, 1994). The potential for brittle fracture was the greatest weakness of dental porcelain as a denture base material (Manappallil, 2003; Valega and Reese, 1990). Polycarbonates had remarkably good impact strength but they were difficult to mould as denture base materials (Combe, 1992:158). As reported by Cahn, Haasen and Kramer (1992), Poly-amid (Nylon) was not a successful denture base material due to its tendency to absorb water.

Vulcanite, a highly cross-linked rubber with a dark brown natural colour, was the popular denture base material before the late 1940s (Van Noort, 1994). The difficulty in pigmentation and the intake of saliva by the vulcanite denture base however produced unfavourable dental prostheses. In addition, the inert material used for pigmentation decreased the mechanical strength of the rubber, consequently limiting its use in the dental industry. The mechanical, physical and biological inadequacies of all these materials therefore motivated for the development of acrylic resins.

In the mid 19th century metal alloys such as gold, aluminium-manganese, stainless steel and silver-tin were also used in the fabrication of denture bases (Wulfes, 2004:17). However, associated high costs in terms of using expensive equipment during fabrication, amongst other limitations that will subsequently be discussed, restricted the use of metals in removable denture work (Zarb and Hickey, 1980). This further necessitated the development of acrylic resins.

Acrylic resins were first used in dentistry in the 1940s (Gladwin and Bagby, 2000:128). In 1946, it was estimated that more than 95% of all removable dentures in United States of America were constructed using PMMA resin (Craig, *et al*. 1992; Winkler and Apellbaum, 1984). Jagger *et al*. (2002) further reported the widespread use of heat cured PMMA resin denture base material in the fabrication of denture bases. PMMA resin, however, has limitations particularly in terms of flexural and impact strength. Anusavice (2003:722) later confirmed that most number of denture bases were fabricated using PMMA resins. Although new polymer based materials such as polystyrene and light-activated urethane dimethacrylate have been developed and introduced in the global dental market, PMMA resin however continues to remain the preferred denture base material in the fabrication of removable complete and partial dentures (Meng and Latta, 2005). The literature continues by giving an overview of the ideal properties required for a denture base material.

2.2 OVERVIEW OF THE IDEAL PROPERTIES OF DENTURE BASES

Several authors (Mannappalill, 2003; Phillips and Moore, 1994; Combe, 1992; Sowter, 1986; McCabe, 1985) have conveniently listed certain ideal properties required for a denture base material to be successful in the oral environment. These properties include:

- Sufficient flexural strength to resist fracture.
- High value of elastic limit to ensure that stresses encountered during biting and mastication do not cause permanent deformation.
- An appearance that matches the natural oral tissues.
- Radiopacity so that the material can be detected using normal diagnostic radiographic techniques.

- Glass transition temperature (T_g) that is high enough to prevent softening and distortion during use.
- Dimensional stability so that the shape of the denture base does not change during function.
- Low specific gravity and high modulus of elasticity in order for the dentures to be as light as possible.
- High value of thermal conductivity in order to enable the denture wearer to maintain a healthy oral mucosa and retain a normal reaction to hot and cold stimuli.
- Adequate fatigue life, high impact strength and abrasion resistance.

Furthermore, according to Manappallil (2003) an ideal denture base material should also be chemically inert, insoluble in oral fluids and should not absorb water or saliva since this may alter the mechanical properties of the material. More importantly, the unmixed or uncured states of the denture base material should not be harmful to the user involved in its handling during fabrication. The denture base should be relatively inexpensive and easily fabricated without the use of very costly processing equipment.

Since the 18th century, denture bases have been fabricated using different materials. The different materials used were due to technological advancement and, more importantly, an attempt to achieve an ideal material to be used universally for denture bases. Currently in the field of dental industry, denture bases are commonly fabricated using acrylic resins or metal alloys (Kanie, Arikawa, Fujii and Ban 2004). The review of the related literature will therefore continue by comparing the performance of denture bases fabricated using acrylic resins or metal alloys. Subsequently a discussion on the popularity of PMMA resin as a denture base material ensues.

2.2.1 Resin versus Metal Denture Bases

Metal has isotropic strength that accommodates for the fabrication of thin plates for denture bases, consequently allowing for sufficient flexural strength, high value of elastic limit, adequate fatigue life and high impact strength (Kanie, *et al*. 2004). The use of metal denture bases however is limited due to high cost, allergic reactions and the use of very expensive equipment during fabrication (Zarb and Hickey, 1980).

Metal alloys used in fabrication of denture bases for removal dentures are in long-term contact with the teeth and oral tissues (Craig, *et al*. 2000:231). Moreover, all dental cast alloys release metal ions intra-orally thereby having the potential to interact with the oral tissues (Schmalz and Garhammer, 2002). The biological interactions of cast metal alloys with the oral tissues occur by different mechanisms and these include bacterial adherence, toxic effects and allergic reactions. Although, bacterial adhesion may be eliminated by intense oral hygiene measures, toxic effects and allergy, however, may lead to clinically adverse local reactions such as chronic urticaria, acute urticaria, anaphylactoid exanthema and other mucosal symptoms (Canay, Hersek, Çulha and Bilgiç, 1998). From a corrosion perspective, Canay *et al*. (1998) further argue that titanium plates, although being a metallurgical advanced material, corroded intra-orally when used by patients for six months. This corrosion is a health hazard and may be attributed to either the casting or processing conditions of the metal itself or the effects of the oral environment.

Conversely, acrylic resins have superior aesthetic properties and do not cause the allergic reactions often associated with metals (Zarb and Hickey, 1980). In addition, acrylic resins are free from toxicity, easily repaired, have the ability to reproduce accurately and capable of retaining indefinitely the details and dimensions of a pattern (Winkler and Apellbaum, 1984). Furthermore acrylic resin PMMA resin absorbs less water or oral fluids than any other resin and therefore it

is favoured as a denture base material (Phillips and Moore, 1994:107). At present, PMMA resin is universally preferred for denture base fabrication because of its working characteristics (ease and speed of processing and finishing), accurate fit, stability in the oral environment, and use with inexpensive equipment (Manappallil, 2003:100; John, Gangadhar and Shah, 2001). The popularity of PMMA resin denture base material has therefore motivated its use in the study at hand.

The weak mechanical properties of acrylic resins such as flexural strength, hardness, tensile strength, modulus of elasticity and impact strength have contributed to PMMA resin dentures cracking and breaking, especially during repeated chewing stresses or if accidentally dropped (McCabe and Walls, 1998; Vallittu, 1983). This recurring disadvantage of acrylic resin denture bases breaking during clinical service or when accidentally dropped is a major clinical concern to the dental industry (Darbar, Huggett and Harrison, 1994).

The proceeding discussion will therefore address the common causes and types of fractures experienced in heat cured PMMA resin which is the material being investigated in this study. Moreover, the methods adopted to reinforce PMMA denture bases in order to increase their flexural strength are specified.

2.3 COMMON CAUSES AND TYPES OF PMMA RESIN DENTURE BASE FRACTURES

Flexural fatigue stress exerted by repeated masticatory forces is one of the primary causes of PMMA resin denture base fractures (Chitchumnong, Brooks and Stafford, 2003; Jagger, *et al*. 1999; Darbar, *et al*. 1994; Lamb, 1993). According to Franklin, Wood and Bubb (2005) majority of PMMA resin dentures fracture at the end of three years in service or gradually during function. Alternately, fractures do occur more rapidly when the denture is dropped on a hard surface. El-Sheikh and Al-Zshrani (2006) further confirmed that dentures fracture when used intra-orally

for more than 1 year but less than 3 years. In addition, the most common type of denture fractures occurring is the breakdown of the PMMA resin denture base.

Several authors (Vallittu, *et al*. 1993; Yli-Urpo, *et al.* 1985), as cited by Narva Vallittu, Helenius and Yli-Urpo (2001) have reported that denture fractures commonly occur in the PMMA resin denture base and are less often associated to damage of the artificial teeth. Most denture fractures that occur in the mouth during function are primarily produced by the various functional stresses such as compressive, tensile and shear, consequently causing resin fatigue of the denture base material (Allen, Bayne, Cronin, Dovan, Kois and Summit, 2004; John, *et al*. Shah, 2001).

Uneven re-sorption of the residual ridges may further contribute to maxillary PMMA resin denture base fracturing during clinical service (John, *et al.* 2001; Blakeslee, Renner and Shiu, 1980). Under such conditions the maxillary denture base flexes across the hard palate, consequently producing a fracture down the midline of the palate. McCabe and Walls (1988) however, have reported that fracture of a denture base *in situ* often occurs by fatigue mechanism whereby relatively small flexural stresses over a period of time may eventually lead to the formation of small cracks propagating through the denture base.

Fractured denture portions can pose a significant health hazard if inhaled by a patient (O'Brien, 1997:89). Hashmi, Walter, Smith and Latis (2004) reported on a 34 year old patient accidentally swallowing a partial denture that lodged in the upper oesophagus, at the level of sternal notch. Multiple attempts to remove it with various instruments were unsuccessful. Consequently oesophagotomy was performed through lateral pharyngeal approach to retrieve the partial denture. Although the partial denture swallowed was not a result of fracture, this analogy to the fractured pieces being swallowed is a concern.

Other contributing factors in PMMA resin denture base fractures include stress intensification, deep incisal notching at the labial frena (especially when in conjunction with middle diastema), sharp changes at the contours of the denture base, deep scratches, heavy anterior occlusal contact, high forces due to strong mandibular elevator musculature and induced processing stresses (John, *et al*. 2001; Zarb, Bolender and Carlsson, 1997).

Lately, Ming and Latta (2005) have argued that causes of denture base fractures are more often related to design errors rather than problems with the resin itself. Such denture fractures can occur in excessively thin areas or weakened flanges around frenal notches. Midline fractures of PMMA resin dentures have proven to be incredibly troublesome and dental specialists often have to selectively recommend increasing the bulk of material in regions subjected to deformation and fractures. The locations include the palatal incisal junction, the posterior palatal midline and the mandibular incisal area adjacent to the lingual and labial attachments.

Increasing acrylic resin bulk, however, produces denture bases that are too thick, consequently causing the patient to gag or dislodge the denture due to reduced tongue space. More importantly, excessive denture base thickness in a maxillary denture can interfere with the coronoid process of the Temporomandibular joint during functional and para-functional movement of the mandible (Ming and Latta, 2005; Zarb and Hickey, 1980). PMMA resin denture bases should therefore not be excessively thick as it significantly impacts on the denture functioning successfully intra-orally.

In an attempt to avoid excessively thick PMMA resin denture bases to minimise fractures, PMMA resins can be chemically modified to produce various materials with significant differences in mechanical properties. One such chemical formulation is the addition of high levels of cross-linking resins and heat activated

initiators (Sledgehammer®, Maxipak® and ProBase Hot®), which help to maximize the physical and mechanical properties of the PMMA resin. Another formulation is the addition of rubber compounds to the PMMA resin (Lucitone 199® and Fricke Hi-Impact®), which improve shock resistance and strength (Ming and Latta, 2005; Craig, *et al*. 2000). The chemical modification of acrylic resin through the incorporation of rubber in the form of butadiene styrene has been successful in terms of improving the impact strength (Rodford and Braden, 1992). However, the incorporation of rubber has not been entirely successful in that it has detrimental effects on the modulus of elasticity and hence the rigidity of the denture base is compromised (Jagger, *et al*. 2002).

In comparison to the conventional PMMA resins, these modified PMMA resins materials are more expensive. "The affordability of denture-service to the patient is very important because a large number of denture wearers are indigent, elderly or retired and have very limited funds" (Denturism Online, 2007-09-20). In an attempt to address the affordability and mechanical strength of PMMA resin based dentures, reinforcing materials such as stainless steel mesh, glass fibres, carbon fibres, sapphire whiskers, aramid fibres, nylon or ultra-high-modulus polyethylene fibres (UHMPE) can be incorporated in the conventional PMMA resin to improve the mechanical strength (John, *et al*. 2001).

However, denture wearers are often advised to replace their dentures after five years of clinical service. Ultimately, a denture fracturing prior to this time is an inconvenience to the patient. Therefore there is need for further research on PMMA resin denture base material in order to increase the life span of the dentures. Since PMMA resin reinforcing materials have been used for several decades to increase mechanical strength, with some reinforcing materials losing popularity, the review of the literature will continue by comparing PMMA resin reinforced with different types of fibres or wire mesh. Additionally, the effectiveness of different fibres and wire mesh (metal) reinforcements on the flexural strength of

PMMA resin then follows and includes the factors that contribute to the popularity of stick® glass fibres.

2.4 PMMA RESIN REINFORCED WITH FIBRES OR WIRE MESH

Metal plate or wire mesh has been commonly used as a reinforcing material for PMMA resin denture bases in the dental laboratory industry (Teraoka, 2001; Vallittu, 1996; Braden, 1988; Ruffino, 1985; Carrol and Fraunhofer, 1984). Metals, however do not chemically bond to resins. Consequently, the PMMA resin around the metal shrinks away leaving the material with voids that weakens the structure by creating new points of stress concentration (Kim and Watts, 2004a).

More importantly, metal strengtheners have a minor effect on the strength of denture base acrylics because they do not impregnate well with the PMMA resin matrix, consequently weakening the resin rather than strengthening it (Daryll, Allan and Klaus, 2000). Metal rods or wire meshes are also difficult to handle and cut during fabrication and produce an unaesthetically black appearance beneath the resin. In this new era of metal free restoration, fibre reinforcement composites (FRC's) were therefore introduced to the dental industry as they have the ability to exhibit better mechanical and aesthetical characteristics (Sedda, Borracchini, Monticelli, Goracci and Ferrari, 2006; Narva, *et al*. 2001; Gutteridge, 1988; Ladizesky, *et al*. 1994).

According to Larson, Dixon, Aquilino, and Clancy (1991), carbon/graphite and aramid fibres increase the strength of PMMA resin based polymers. Aramid fibres however were difficult to handle and carbon/graphite fibres imparted a black colour to the acrylic resin. This therefore restricted their use as reinforcements. Although nylon (polyamide) fibres provided good shock resistance, their use diminished due to water absorption negatively affecting the mechanical properties of PMMA resin

as a denture base material (John, *et al*. 2001). The incorporation of polyethylene in the PMMA resin denture base provided excellent strengthening properties; however the technical difficulties associated with the need of additional processing procedures limited their use in the Dental Technology industry (Daryll, *et al*. 2000).

The inadequacies of the previously used fibre reinforcement materials motivated for the use of glass fibres in the Dental Technology industry. Commercially, there are different glass fibres produced, which include E-glass, S-glass, R-glass, V-glass and Cemfil. The stick® glass fibres, which are Fibre Reinforced Composites (FRC'S) used in this study, are modified unidirectional E-glass® fibres that contain a high alumina, low alkali and borosilicate content that produces superior flexural strength properties to PMMA resin (John, *et al*. 2001).

Translucency of glass fibres provides for aesthetically pleasing dentures. More importantly, they have an excellent adhesion to the PMMA resin matrix, and consequently an outstanding ability to increase mechanical properties (Vallittu, 1999). Adhesion to the PMMA resin matrix is further enhanced by the unique Interpenetrating Polymer Network structure (IPN structure) within stick® glass fibres (Sticktech Online, 2006-08-23; Stickbond Online, 2006-08-25).

2.5 FIBRE REINFORCED COMPOSITES (FRC'S)

Composites are fabricated when two or more materials are used together to give a product with combination of properties (Vallittu, 1998). Fibre reinforced composites provide improved properties on strength, fatigue resistance and elastic modulus. Initially, FRC's had gained widespread acceptance in a diverse range of structural engineering applications (Askeland and Phulé, 2003; Vallittu, 1998). The diverse range extends from the inexpensive, easily processed small components to the

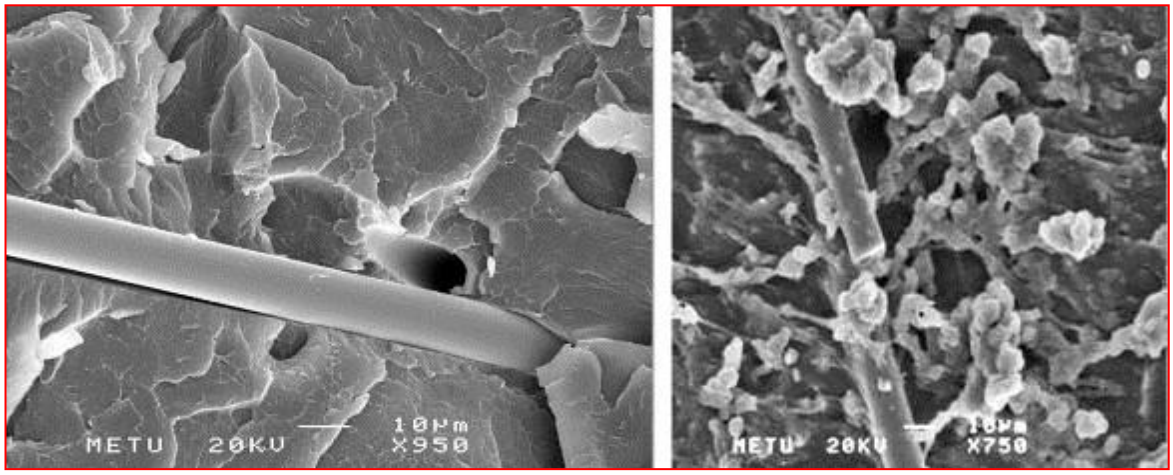
high cost FRC's for aerospace and other demanding applications that require ultimate performance.

The global use of FRC's in the engineering world significantly influenced its introduction and use in the Dental industry, particularly in the past decade. Fibre reinforced composites such as stick® glass fibres produce PMMA resin denture base of increased stiffness and strength. The PMMA resin polymer matrix protects the fibres and fixes their geometrical arrangement by holding them at predetermined positions that will provide optimum reinforcement (Vallittu, 1998). The interface between the two components vitally transfers flexural loads from PMMA resin matrix to the stick® glass fibres, thereby enhancing the clinical longevity of the dentures. Freilich, Duncan, Meiers and Goldberg (1998) reported that fibres, particularly glass fibres have shown improvement in the mechanical properties of denture base polymers. This effectively improves the overall fatigue and aesthetic properties of PMMA resin denture bases.

A brief overview of microstructure analysis of fibre reinforced PMMA resin ensues, followed by a discussion on the use of glass fibres with multiphase PMMA resin.

2.6 MICROSTRUCTURE OF FIBRE REINFORCED PMMA RESIN

According to Askeland and Phulé (2003:30) microstructure is "the structure of a material at a length-scale of ~10 to 1000 nm". Microstructure typically includes such features as average grain size, distribution, orientation and other features related to defects in materials. Figure 2.3 displays microstructure of glass fibres in PMMA resin matrix, (a) unmodified and (b) modified by addition of ethylenediamine (EDA) to improve adhesion.



a b

Figure 2.3 Glass fibres in PMMA resin structure (Adapted from Çökeliler, *et al*. 2007).

Scanning Electron Microscopy (SEM) micrographs of PMMA resin reinforced with continuous E-glass fibres and stainless steel wire showed fatigue striations on the surface of the wire (Vallittu, 1996). The fractographs of the fractured surfaces of the PMMA resin revealed a “mirror smooth zone” adjoining a “misty zone” (Ma Gang and Xia, 2004). Although good impregnation of the fibres was revealed by SEM evaluation of various FRC’s, voids within their matrices were also present (Chai, Dlaw, Takashi, Hisama and Shimizu, 2003). Most of the voids were located at or proximal to the fibre-matrix junction.

2.7 THE USE OF GLASS FIBRES WITH MULTIPHASE ACRYLIC RESIN

Denture bases are typically fabricated from polymer (powder) and monomer (liquid) to form a multi-phase acrylic resin by polymerisation. The stick® glass fibres can be used in two ways to reinforce a multi-phase denture base acrylic. These include using the glass fibre in the entire denture base, termed total fibre

reinforcement (TFR) or partially placing the fibres accurately at the weak site of the PMMA resin denture base, termed partial fibre reinforcement (PFR) (Narva, *et al*. 2001; Vallittu, 1998). A clinical survey of the performance of glass fibres in repairing PMMA resin removable dentures concluded that "PFR" and "TFR" can prevent recurrent fractures in PMMA resin dentures (Narva, *et al*. 2001). This study will use "TFR" to reinforce the specimens.

Several factors such as orientation and quantity of fibres, impregnation of fibres with the matrix and fibre adhesion to the polymer matrix have an impact on the mechanical strength of PMMA resin (Narva, *et al*. 2001; Vallittu, 1998). The knowledge gained from the proceeding discussion on each factor is crucial as it lends itself when interpreting the scanned images of the PMMA resin denture base reinforced with stick® glass fibres.

2.7.1 Orientation and Quantity of Fibres

Unidirectional stick® glass fibres are fibre rovings or yarns consisting of 1,000 to 200,000 single glass fibres (Vallittu, 1998). Unidirectional fibres give an anisotropic mechanical property to the polymer and are suitable for applications in which the direction of the highest stress is known. The stick® glass fibres used in this study are unidirectional glass fibres and differ to that of bi-directional glass fibres, commonly known as glass fibre weaves. The latter is used to reinforce acrylic resin denture base in all directions when the clinician or dental technician finds it difficult to predict the direction of the highest stress in the prosthesis.

Glass fibres can also be woven in such way that the fibres are oriented in two or three directions, consequently contributing to the so-called orthotropic mechanical properties. Nohrstrom *et al*. (2000) confirmed that the positioning of reinforcements influences the strength of the dentures considerably. Vallitu (1995 and 1994) demonstrated that an increase in the quantity of the fibres in the PMMA

resin matrix enhances the flexural and impact strength of PMMA resin. The length and degree of glass fibre concentration in the PMMA resin significantly increases the transverse strength, elastic modulus and impact strength (Karacaer, Polat, Tezvergil, Lassilla and Vallittu, 2003).

2.7.2 Impregnation of Fibres with Polymer Matrix

It has been documented (Vallittu, 1995; 1994; 1992) that the "compression moulding" technique produces poor fibre impregnation with the PMMA resin dough during fabrication. Consequently, the denture will have an uneven impregnation of the fibres within the polymer matrix. The problem of inadequate impregnation is further compounded by the difficulty of using reinforcing fibres with multiphase acrylic resins, such as powder liquid resins (Tacir, Kama, Zortuk and Eskimez, 2006). To curb this problem of poor impregnation of the glass fibre with the PMMA resin, the "dough moulding" technique is used in the study at hand.

Aside from the technique used, glass fibres also have a tendency to produce poorly impregnated regions in the FRC that results in voids, cracks or un-bonding of the glass fibre to the matrix (Miettinen and Vallittu, 1997). Consequently, this increases the risk of water absorption in the PMMA resin thereby reducing its mechanical properties (Chai, *et al*. 2004).

More importantly, any voids, cracks or un-bonded interfaces are oxygen reserves that inhibit radical polymerisation of the PMMA resin inside the FRC. From a biological perspective, the poorly impregnated regions will attract oral microbes into the voids of the PMMA resin denture base. Inevitably this will negatively impact on the aesthetics of the denture (Lastumäki, Kama, Zortuk and Eskimez, 2003). To minimise the problem, pre-impregnated fibres are advocated as they allow the matrix to maximally wet the surface of the fibres. The stick® glass fibres are pre - impregnated with highly porous PMMA resin to facilitate the maximal wetting to the

polymer matrix. Intra-orally, this will assist in reducing stress concentration under functional load (Chai *et al*., 2004; Vallitu, 2000) as cited by Bouillaguet, Schütt, Alander, Schwaller, Buerki, Michler, Cattani-Lorente, Vallittu and Krejci (2006).

2.7.3 Adhesion of Fibres to PMMA Matrix

Surface treatment of PMMA resin denture base material with plasma (although delimited in this study) increases the surface energy as it modifies the chemistry of the surface. Consequently, the flexural strength of the polymer increases due to the adequate adhesion of the fibres (Çökeliler, Erkut, Zemek, Biederman and Mutlu , 1996). In addition, Silane coupling agents have also been used to improve the adhesion between PMMA resin and glass fibres (Drown *et al*., 1992; Rosen, 1978; Clark and Plueddemann, 1963; Björksten and Yager 1952) as cited by Vallittu (1998).

According to Daryll *et al*. (2000) the surface treatment of fibres with butadiene styrene latex emulsion is an important factor in the success of glass fibre reinforcement. This was later confirmed by Ellakwa, Shortall and Marquis (2002) when improved flexural strength of the PMMA resin was achieved after wetting with a bonding agent. The unique Interpenetrating Polymer Network structure (IPN structure) of the stick® glass fibres is therefore expected to produce better adhesion to the PMMA resin.

Since thermocycling is a crucial procedure in this study, the review of the literature will further continue by giving an overview of thermocycling and its effects on dental restorations. Subsequently, the criteria for the selection of the appropriate regimens for temperatures, cycles and dwell time are discussed.

2.8 AN OVERVIEW OF THERMOCYCLING PROCEDURES

Intra-orally, PMMA resin dentures are usually under thermal variations and masticatory loading conditions. Exposure of the prosthesis to such thermal changes and cyclic loading may cause fatigue fractures during long-term clinical use (Rantalla, Lastumaki, Peltomaki and Vallittu, 2003). Mechanical degradation of polymer matrix and fillers has also been reported (Øysaed and Ruyter, 1988) as cited by Meriç and Ruyter (2006).

According to Palmer, Barco and Billy, 1992) thermocycling is a common "*in vitro*" test for dental materials in establishing suitability for *in vivo* use. Thermocycling simulates thermal changes during eating and drinking of cold and hot food/beverages. This significantly impacts on the mechanical properties of polymer materials as noted by Oshida *et al*. (1995) and cited by Lonĉar *et al*. (2006). Thermocycling temperatures and cycles used for in vitro studies should meet the basic principal requirements and ideally simulate in a manner similar to that of the oral environment (Darvel and Gale, 1998).

According to Tanaka, Kamada, Matsumura and Atsuta (1995) cooled water at 4˚C to 5˚C and warm water at 50˚C to 60˚C are commonly used as test solutions. Darvel and Gale (1998) have widely investigated the median temperatures and dwell times (time taken to soak the specimens) for thermocycling dental restorations, and have reported that the acceptable median temperatures are 5.0˚ C (median-low) and 55˚C (median-high). A median dwell time of 30 seconds was recommended. The frequency of cycles in vivo remains undetermined and requires formal estimation. In the absence of this information, it was therefore proposed that cycles might occur between 20 and 50 times in a day. This effectively calculates to about ±10 000 cycles for one service year (Gale and Darvel, 1999). Since the specimens in this study will be tested for flexural strength,

the literature review ensues by defining flexural strength and discussing its relevance to denture base fracture. In the final analysis, an emphasis on the rationale for using stick® glass fibres in reinforcing PMMA resin denture bases and its performance intra-orally is given.

2.9 TESTING FLEXURAL STRENGTH OF PMMA RESIN

"Flexural strength, transverse strength, or modulus of rupture, as this property is variously called, is essentially a strength test of a bar supported at each end, under a static load" (Anusavice, 2003:89). This test is a collective measurement of tensile, compressive and shear stresses simultaneously. The flexural test measures the force required to bend a beam under a three-point loading. The parameters for flexural test are the speed of the load, support span and the maximum deflection for the test specimen, which are defined differently by ASTM (American Standards of Testing Materials) and ISO (International Standards Organization), (Flexural Strength Testing, Online, 2008-04-11). The ISO Flexural strength test (3-point bending) is used in this study as it is considered relevant to the masticatory loading characteristics of a denture base in a clinical situation (Jagger, *et al*. 2002). Transverse tests are further considered to be appropriate for polymeric dental materials since compression and tension stresses are correlated to specific fractures that occur during flexural load.

At present both stick® glass fibre and wire mesh are used to reinforce removable dentures. Although stick® glass fibre has gained increased popularity in the global market place, its use in South Africa is limited (Visser, personal communication. 23 May 2006). An investigation of the flexural strength and microstructure of PMMA resin reinforced with both types of materials may therefore provide

significant data to the South African dental industry, which may impact on the selection of optimum denture base reinforcement for removable dentures.

CHAPTER THREE: METHODOLOGY

"Most of the fundamental ideas of science are essentially simple, and may, as a rule, be expressed in a language comprehensible to everyone."

(Albert Einstein, 1879-1955)

3. MATERIALS AND METHODS

3.1 MATERIALS

DENTURE BASE MATERIALS

The selection of the materials used was based primarily on their popularity and availability in Durban (South Africa). These materials were selected to ensure that the results of this study would have further implicational value in the commercial dental industry when published.

Excel® PMMA polymer and monomer (Excel®, DB AV WHG3001 and Excel® DB WHG3015, Wright Health Group Ltd, Dundee, Scotland), gilded wire mesh (Art. No.222-1100, Renfert, Germany) and stick® glass fibres (PMMA resin pre-impregnated unidirectional E-glass fibres, StickTech Ltd, Finland) were the selected materials used to produce the specimens (Plate 3.1).

Plate 3.1: PMMA resin armamentarium

3.2 METHODOLOGY

3.2.1 Sample Size

Thirty specimens for each sample group were used for the purpose of valid statistical analysis [1].

3.2.2 Preparation of Samples

With the use of stainless steel dies (Plate 3.2), manufactured by the Department of Mechanical Engineering (Steve Biko Campus, Durban University of Technology), specimens were accurately replicated.

Plate 3.2: Stainless Steel Dies

The purpose of the stainless steel dies were to:

1. Ensure that quadratic PMMA resin specimens with smooth surfaces were equally replicated in length, width and height (65mm × 10mm × 3mm). This is in compliance to the International Standards Organization (ISO Standard, 1999:1567) for fabricating specimens for testing flexural strength of denture base polymers.

[1] The central theorem states that sample sizes should be at least twenty five, however the larger the sample sizes the better the approximation of the mean and standard deviation (Kuzma and Bohnenblust, 2005:110).

2. Provide a consistent mould during the “flasking and packing”[2] procedures (Plate 3.3).

Plate 3.3: Dental Stone Moulds

Prior to packing the PMMA resin dough into the mould, cold mould seal (Wright Health Group Ltd, Dundee, Scotland) was applied to prevent:

a. Water being incorporated into the PMMA resin dough from the dental stone during processing as this negatively impacts on the polymerisation rate and colour of the resin (Combe, 1992).
b. Dissolved polymer and free monomer from soaking into the dental stone mould surface (Combe, 1992).

Gilded wire mesh supplied in preformed measurements of 10 x 10cm and stick® glass fibres supplied in lengths of 10cm were sectioned, using scissors, to sizes of 64 x 8mm and 64mm respectively. This was done to simulate the length and breadth of the stainless steel dies. Ninety PMMA resin specimens were fabricated using Excel® (Wright Health Group Ltd in Dundee Scotland), which is a heat-

[2] Flasking and Packing is a process of converting a wax pattern to resin. This is done by using the wax pattern to make a mold into which denture base resin is inserted and cured (Sowter, 1986)

polymerised PMMA resin denture base material. All specimens were flasked and packed as follows:

Polymer/monomer in the ratio of 23g/10ml was thoroughly mixed with a spatula for one minute in a mixing vessel. Once the mixture reached the dough stage, it was kneaded and placed into the mould. With slow pressure, the flasks were then clamped in a bench press to ensure even flow of the material within the mould. Curing of the flasks in a water bath then followed. After the curing cycle, the flasks were bench cooled for ten minutes prior to immersing in cold water (curing and quenching was done in accordance to the manufacturer's instructions). All specimens were trimmed and polished to produce 3 sample groups (Table 3.1).

Table 3.1: Excel® PMMA Resin Sample Groups

Sample Groups	No. Of Specimens
1. Stick® glass fibre reinforced PMMA resin (PMMA pre-impregnated unidirectional E-glass fibres, StickTech td, Finland)	30
2. Wire mesh reinforced PMMA resin (Renfert, Germany)	30
3. Un-reinforced PMMA resin (Control)	30
TOTAL	**90**

It must be noted that although all sample groups were fabricated using the standardised method of flasking and packing, the following differences existed in the sample groups reinforced with stick® glass fibres and gilded wire mesh respectively:

1. Prior to packing specimens with stick® glass fibres, an analytical balance was used to weigh the fibres. This ensured that each specimen contained the same quantity of fibres. The average weight of fibres used was 0.16g.
2. Stick® glass fibres were pre-impregnated with a thin mixture of monomer and polymer prior to packing to facilitate the bonding of the fibres with the PMMA resin matrix (StickBond Online, 2007-10-30).
3. During packing of the PMMA dough in the mould the stick® glass fibres and gilded wire mesh were positioned on the tension side of the dough specimen (Plate 3.4). The positioning of the reinforcements was controlled by marking the stainless steel dies in the middle with a coloured marker cross-sectionally. The coloured mark was duplicated in the dental stone moulds in order to guide in positioning of the reinforcements and control variability in the location. The positioning was to maximise strength and to ensure that the test specimens do not fracture at a lower force (Narva, *et al*. 2004).

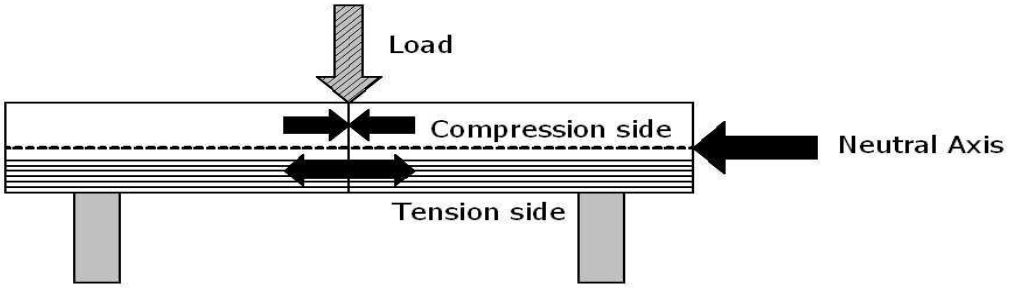


Figure 3.1: Schematic diagram highlighting the compression, neutral and tension sides of the test specimen (Adapted from Narva, *et al*. 2004).

3.3 INCLUSION CRITERIA

The following criteria were used to ensure that all specimens produced were the same thereby improving the reliability of this investigation:

- The stick® glass fibre reinforcements were completely and uniformly covered by the PMMA resin denture base material.
- The gilded wire mesh reinforcements were completely and uniformly covered by the PMMA resin denture base material.
- Only those specimens conforming to the correct dimensions and containing the desired position of the reinforcements were accepted in the study.
- All specimens had smooth, cleaned and highly polished surfaces.

3.4 CLASSIFICATION OF SPECIMENS WITHIN SAMPLE GROUPS

The specimens were numbered on both ends using a permanent marker such that the fractured pieces, after flexural strength testing could be matched. The specimens were numbered and grouped as reflected in Table 3.2.

Table 3.2: Numbering of specimens

Sample Groups	Numbers
Stick® glass fibre reinforced PMMA specimens	1 - 30
Wire mesh reinforced PMMA specimens	31 - 60
Un-reinforced PMMA specimens (Control)	61 - 90

3.5 THERMOCYCLING OF SPECIMENS

The PMMA resin specimens meeting the requirements of inclusion criteria were therefore completely immersed in water at room temperature for 21 days in order to reach saturation levels. PMMA resin denture base material may require a period of 17 days to become saturated with water. The absorbed water compensates for polymerisation shrinkage and relieves curing stresses (Anusavice, 2003).

On reviewing the related literature, specifically in terms of thermocycling restorations, the sample sizes, temperatures and cycles documented are reflected in Table 3.3. In view of Table 3.3, it was therefore decided that 2100 cycles and temperatures of 5˚C to 55˚C would be used for this study.

Table 3.3: Thermocycling Temperatures and Cycles

AUTHORS OF RELATED STUDIES	SAMPLE SIZE	THERMOCYCLING CYCLES	TEMPERATURE RANGES
1. Tirado *et al*. (2001)	25	2000	3˚C to 60˚C
2. Mudford *et al.* (1997)	46	500	5˚C to 55˚C
3. Kountouras *et al.* (1999)	10	1000	4˚C to 60˚C
4. Rossouw *et al*. (1999)	15	500	5˚C to 55˚C
5. Dubois *et al*. (1999)	24	3400	5˚C to 55˚C
6. Grobler *et al.* (2007)	15	500	5˚C to 55˚C

A custom made thermocycling machine (Oral and Dental Research Institute, University of the Western Cape, South Africa) consisting of two stainless steel chambers, a digital thermostat control and a small container attached to a mechanical arm was used (Appendix 1). However, not all specimens could be

thermocycled at the same time due to the limitation of the size of the container. Consequently, the specimens were randomly divided into 3 groups of 30 specimens each (to minimise bias) and thermocycled for 18 hours respectively.

In one hour the machine cycled 120 cycles and this effectively ensured that each specimen was exposed to 2160 cycles. Significantly, this was equivalent to a person wearing a PMMA resin denture for 3 months (Gale and Darvell, 1999). After thermocycling all the specimens were tested for flexural strength.

3.6 TESTING SPECIMENS FOR FLEXURAL STRENGTH

Flexural strength was performed on all specimens using a universal testing machine (Zwick® Model 1446, Ulm, Germany). The machine was set to provide a cross head speed of 5mm/min for testing flexural strength (ISO 1567, 1999). A span length of 50mm between the two supports was used and force was applied using a centrally located rod until fracture occurred (Roussouw, personal communication. 22 May 2007). The maximum force measured was recorded by the computer software attached to the flexural testing machine and this was used to calculate the ultimate flexural strength using the following formula:

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

Where:

- FS is the flexural strength,
- l is the distance between the supports,
- b is the width of the specimen,
- d is the depth or thickness of the specimen,
- p is the maximum load at the point of fracture.

(Anusavice, 2003:89)

Following the results of flexural strength, the coefficient of variation was considered as a useful measure of relative dispersion for comparing two or more data sets with different means (Brink, 1990). The coefficient of variation was therefore calculated using the following formula:

Coefficient of variation (%) = Standard Deviation **/** Mean **x** 100.

After flexural strength testing, visual analysis by a magnification microscope (Nikon FX-35A, Japan) was used to identify fracture modes of the specimens. It was anticipated that the information gained would have significant application to the findings in this research. The identified fracture modes were photographed (Canon, model G3, Japan). The type of fracture was determined in the way the specimen broke.

3.7 SEM OBSERVATION OF SAMPLES

On assumption that reinforced PMMA resin specimens will have differences in microstructure to the control, five specimens with the lowest and highest flexural strengths from each sample group were selected. This resulted in a total of 30 specimens available for analysis. These specimens were analysed objectively under scanning electron microscopes (Philips XL 30, Hitachi; Model S-570, Hitachi Ltd, Tokyo, Japan and LEO 1450, Japan) in an attempt to establish:

- Presence of air bubbles or voids of the fractured surfaces
- The impregnation and distribution of the stick® glass fibres to the PMMA resin matrix
- Bonding of the wire mesh reinforcements to the PMMA resin matrix
- Texture of the upper and lower sides of the specimens.

Note: During flexural strength testing, the upper and lower sides of the specimen are scientifically referred to as compression and tension sides respectively.

The 30 selected specimens were each clamped and sawed in 10 mm lengths in order to fit on the mounting stubs. The specimens were also numbered to assist in identifying them during SEM analyses. The specimens were mounted on aluminium stubs in groups of 5 and arranged systematically such that the compression and tension sides were facing opposite directions (Plate 3.4).

The PMMA resin specimens were not electrically conductive. The electrical charge conferred upon the specimens by the electron probe was not bled away to earth. The specimens were therefore automatically coated (Polaron, E-5100, Japan) with an alloy of gold/palladium for 5 minutes to facilitate improved analysis.

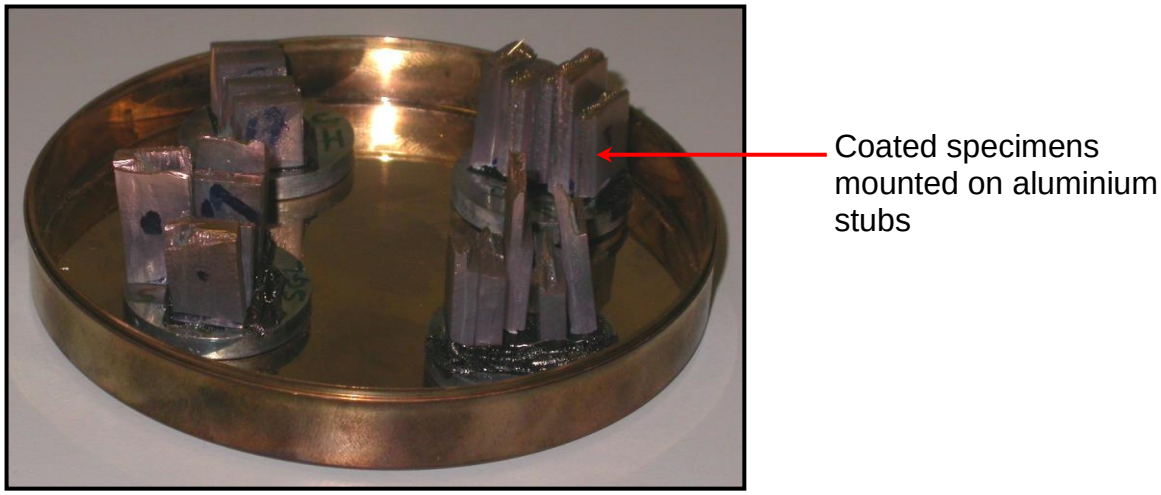


Plate 3.4: Coated specimens mounted on Aluminium stubs

Each specimen was SEM analysed with magnifications ranging between 50x and 100x respectively (Wesley-Smith, personal communication. 05 July 2007). Analysis of the specimens was performed by visually identifying the:

1) presence of bubbles or voids, which were recorded by a nominal scale.

2) impregnation and distribution of the stick® glass fibres in order to assess whether the fibres were distributed evenly across the entire specimen. A binary nominal scale of either fully or partially distributed was used.

3) bonding of the wire mesh to the PMMA resin matrix. This was performed by classifying the micrographs in terms of a gap existing between the wire and PMMA matrix. If a gap existed, it was classified as un-bonding (Chai, *et al*. 2007). The data for bonded and un-bonded wire to the matrix was also recorded using nominal scale.

(Esterhuizen, personal communication. 27 August 2007).

3.8 TEXTURE ANALYSIS OF MICROGRAPHS

The micrographs of the 30 selected specimens were visually and quantitatively analysed. For the latter, a macro image pro-plus computer software package (Howard College, University of Kwa-Zulu Natal, Durban, South Africa) was used. The macro routine software quantified the differences in surface texture (data) by classifying the compression and tension sides of the specimen as “high texture” and “low texture” respectively. It was therefore anticipated that differences in surface texture between the compression and tension sides would provide significant information related to microstructure. To minimise bias, areas with voids or unidentified morphologies were avoided.

3.9 STATISTICAL ANALYSIS

SPSS version 15.0 (SPSS Inc., Chicago, Illinois, USA) was used for data analysis. A p value of <0.05 was considered as statistically significant. The following statistical tests were applied to address the hypotheses:

Flexural strengths

Kruskal-Wallis test and Dunn's multiple comparison post hoc tests were used to compare the flexural strength of the three sample groups. Quantitative variables were checked for normality using the skewness statistic and histograms. Non-normal data were presented using median, quartiles and range. Box and whisker plots showed this data graphically by group.

Microstructure

One way Analysis of Variance (ANOVA), Bonferroni multiple comparison post hoc and Pearson's chi-square tests were used to assess the statistical significance of the microstructure between the sample groups. Components of the microstructure were measured categorically on a binary scale. Cross-tabulations were used to assess the percentage of samples from each sample group. Non-normal data were presented using median, quartiles and range. Box and whisker plots showed this data graphically by group.

Association between flexural strength and microstructure

The Mann- Whitney test was used to compare flexural strength between samples with and without voids. Mean flexural strengths were compared between the components of microstructure. Spearman's correlation analysis was used to assess relationships between flexural strength and texture.

CHAPTER FOUR

Scientists should not, under any circumstances, change their data or observations (Mouton, 2001).

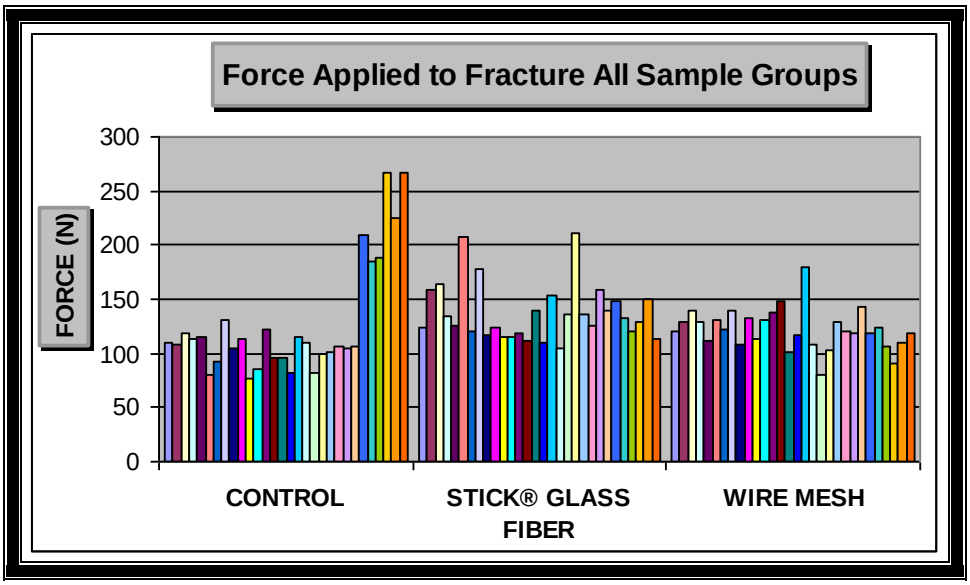
4. RESULTS

4.1 ULTIMATE FLEXURAL STRENGTH

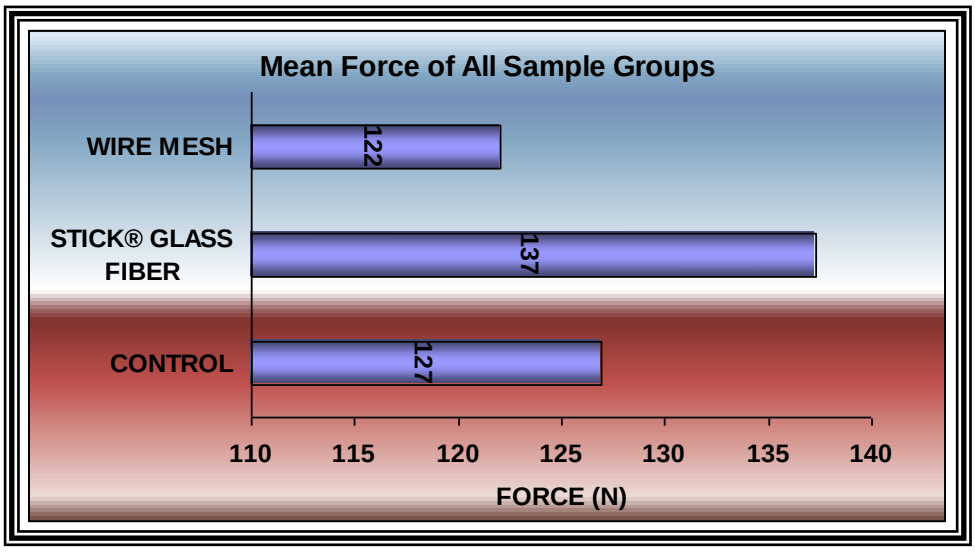
The results of the force applied (Newton) to fracture all three sample groups are summarised in Table 4.1 and illustrated in Graph 4.1 respectively.

Table 4.1: Summary of the Force (N) applied to fracture sample groups

Specimen Number	*Control (Un-reinforced)*	*Stick® Glass Fibre*	*Wire Mesh*
1	102	123	120
2	108	159	130
3	119	164	140
4	114	134	130
5	115	125	111
6	80	207	131
7	93	121	122
8	130	179	139
9	104	117	109
10	113	125	132
11	77	115	113
12	85	115	130
13	122	119	137
14	95	111	149
15	95	140	102
16	82	109	118
17	115	153	180
18	110	104	108
19	82	137	80
20	99	212	102
21	101	137	130
22	107	125	121
23	104	158	118
24	106	139	143
25	209	149	118
26	184	132	125
27	189	120	106
28	267	129	90
29	225	150	110
30	267	114	119
MEAN	**127**	**137**	**122**
SD	**53**	**27**	**19**



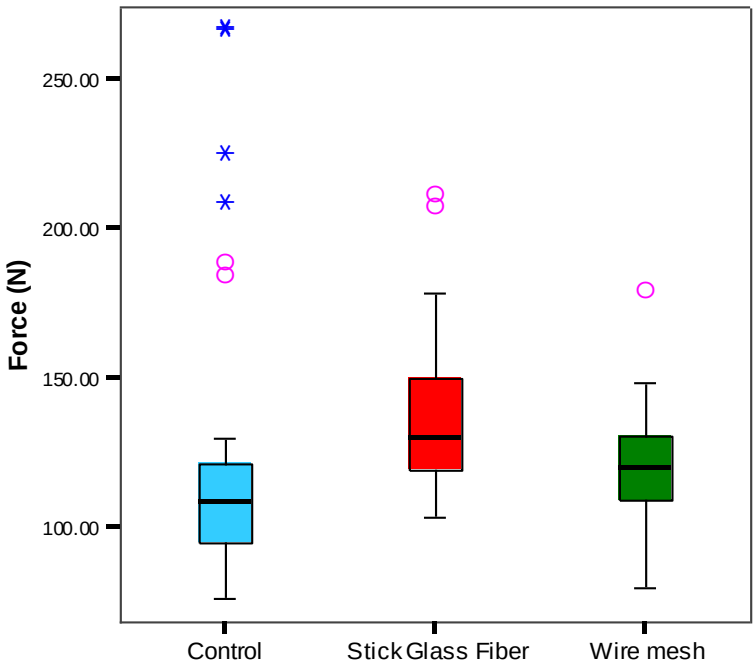
Graph 4.1: Summary of the force applied to fracture specimens



Graph 4.2: Mean Force applied to fracture specimens

Graph 4.2 illustrates the mean force applied to fracture all sample groups; however, Graph 4.3 further demonstrates a “box and whisker” plot of the median force, interquartile range, outliers and the extreme values in each of the three

sample groups. The box represents the interquartile range (top edge is the 75th percentile whilst the bottom edge is the 25th percentile). The horizontal line in the centre of the box is the median (50th percentile). The whiskers represent the range of values. The circles outside the box are the outliers (1.5 to 3 box lengths from the top edge of the box) and the asterisks are the extreme values (more than 3 box lengths from the top edge of the box). Table 4.2 further illustrates descriptive statistics of the median and range of force used to fracture the test specimens.



Graph 4.3: Box and whisker plot of the distribution of force (N) by sample group

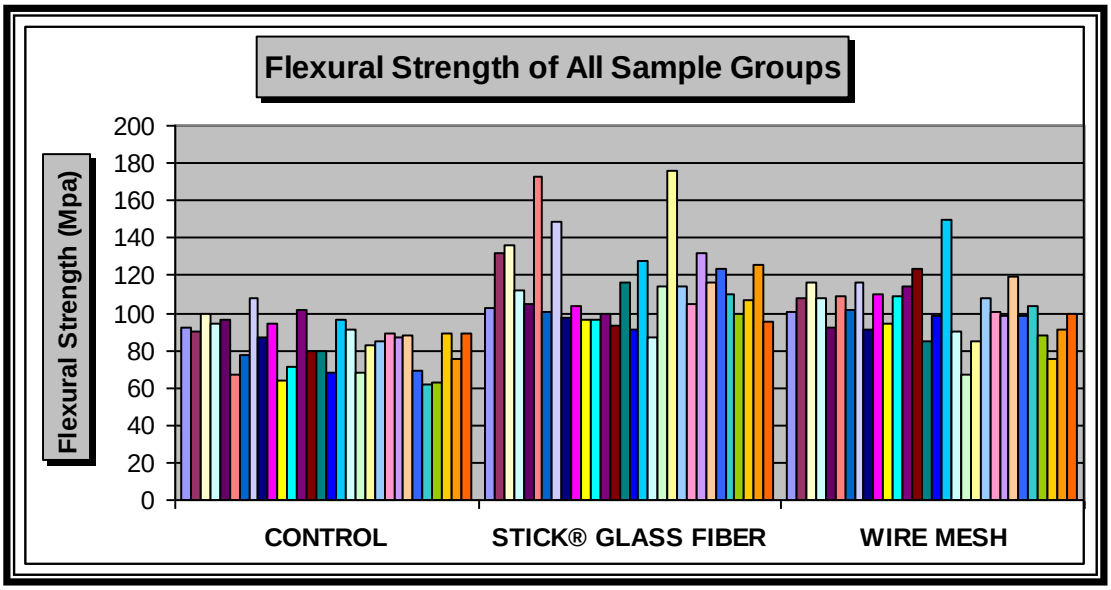
Table 4.2: Descriptive statistics for applied force by sample group

FORCE (N)	SAMPLE GROUPS		
	CONTROL	STICK® GLASS FIBRE	WIRE MESH
Median	109	131	121
Percentile 25	95	119	110
Percentile 75	122	150	131
Range	190	1081	100

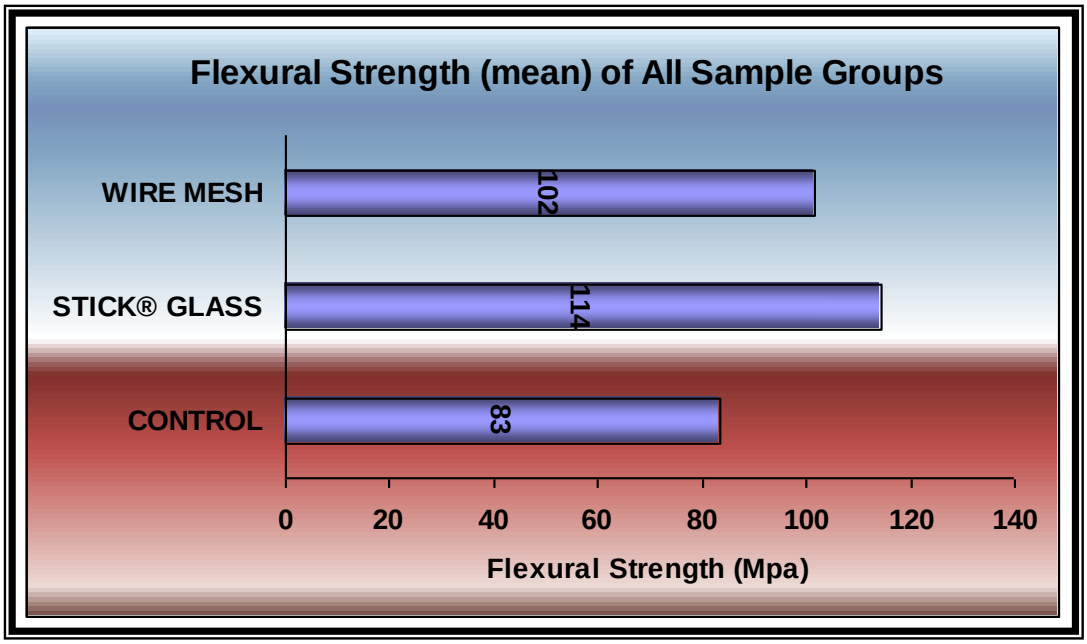
The results of the flexural strength of all three sample groups are summarised in Tables 4.3, 4.4 and 4.5 and Graphs 4.4 and 4.5, respectively.

Table 4.3: Summary of the Flexural Strength (MPa) of specimens

Specimen Number	Control (Un-reinforced)	Stick® Glass Fibre	Wire Mesh
1	92	103	100
2	90	132	108
3	99	136	117
4	95	112	108
5	96	104	92
6	67	173	109
7	77	101	101
8	108	149	116
9	87	97	91
10	94	109	110
11	64	96	94
12	71	96	109
13	101	100	115
14	80	93	124
15	79	1178	85
16	68	91	98
17	96	127	150
18	91	87	90
19	68	114	67
20	83	176	85
21	85	114	108
22	89	104	101
23	87	132	98
24	88	116	119
25	70	124	98
26	61	110	104
27	63	100	88
28	89	107	75
29	75	125.	91
30	89	95	99
MEAN	**83**	**114**	**102**
SD	**13**	**22**	**16**



Graph 4.4: Flexural Strength of all Sample Groups

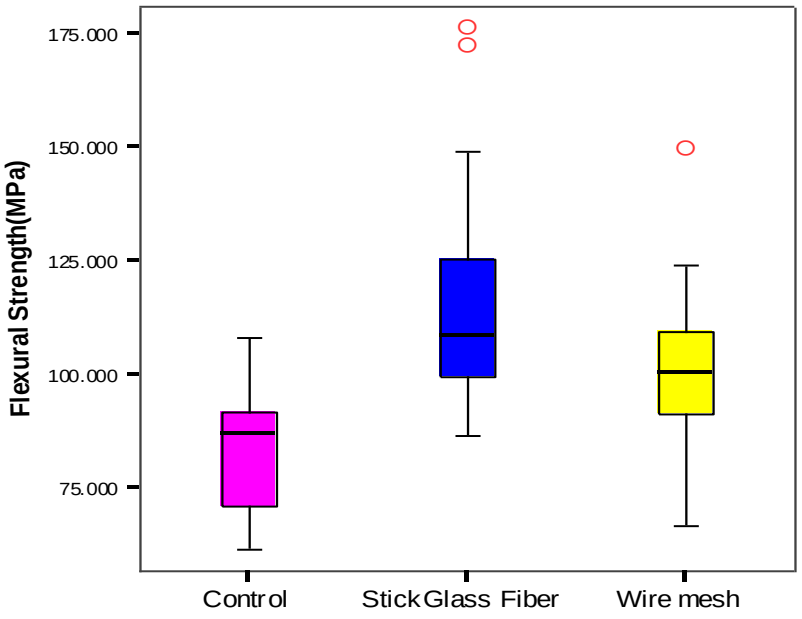


Graph 4.5: Mean Flexural Strength of all Sample Groups

Table 4.4: Descriptive statistics for flexural strength by sample group

FLEXURAL STRENGTH	SAMPLE GROUPS		
	CONTROL	STICK® GLASS FIBRE	WIRE MESH
Median	87	109	100
Percentile 25	71	100	91
Percentile 75	92	125	109
Range	47	90	83

Graph 4.6 is a "Box and Whisker" plot illustrating the median flexural strength, Interquartile range and the outliers in each of the three sample groups.



Graph 4.6: Box and whisker plot of the distribution of flexural strength by sample group

A summary of the Mean, Standard deviations (SD) and Coefficient of variations (CV) of the Flexural Strengths between the sample groups is illustrated in Table 4.4. Stick® glass fibre group with the highest coefficient of variation (19.3%).

Table 4.5: Mean, Standard deviations (SD) and Coefficient of variations (CV) of Flexural Strengths of ALL sample groups.

	CONTROL	STICK® GLASS FIBRE	WIRE MESH
MEAN	83	114	102
SD	13	22	16
CV	15.7%	19.3%	15.7%

4.2 VISUAL EXAMINATION OF PMMA FRACTURED SURFACES

The nature of the objective results as reflected in Tables 4.1 - 4.5 and in terms of validity and reliability warranted the subjective examination by visual and microscopic analysis of the fracturing modes of the PMMA resin specimens. This examination revealed 3 fracturing modes namely:

1. Complete straight fracture (**CSF**) - Specimen cleanly broke into two separate pieces (Plate 4.1).

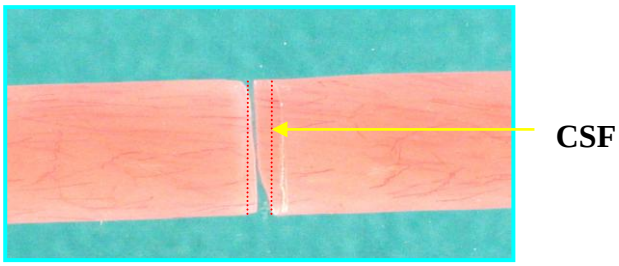


Plate 4.1: Complete Straight Fracture (**CSF**) of un-reinforced PMMA resin

2. Tension Fracture (**TF**) **–** Fracture contained within the specimen that produced tearing on the tension side (Plate 4.2).

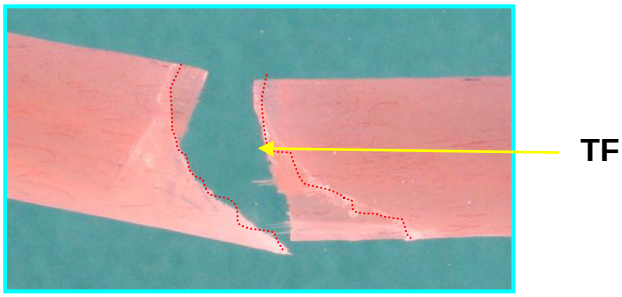


Plate 4.2: Tension Fracture (**TF**) Of Stick® glass fibre reinforced PMMA resin

3. Incomplete Fracture (**IF**) **-** specimen not cleanly broken into two separate pieces (Plate 4.3).

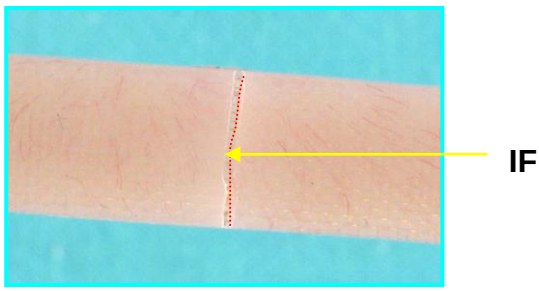


Plate 4.3: Incomplete Fracture (**IF**) of wire mesh reinforced PMMA resin

Figure 4.1 and Graph 4.7 further reflect the association between fracture mode and sample groups.

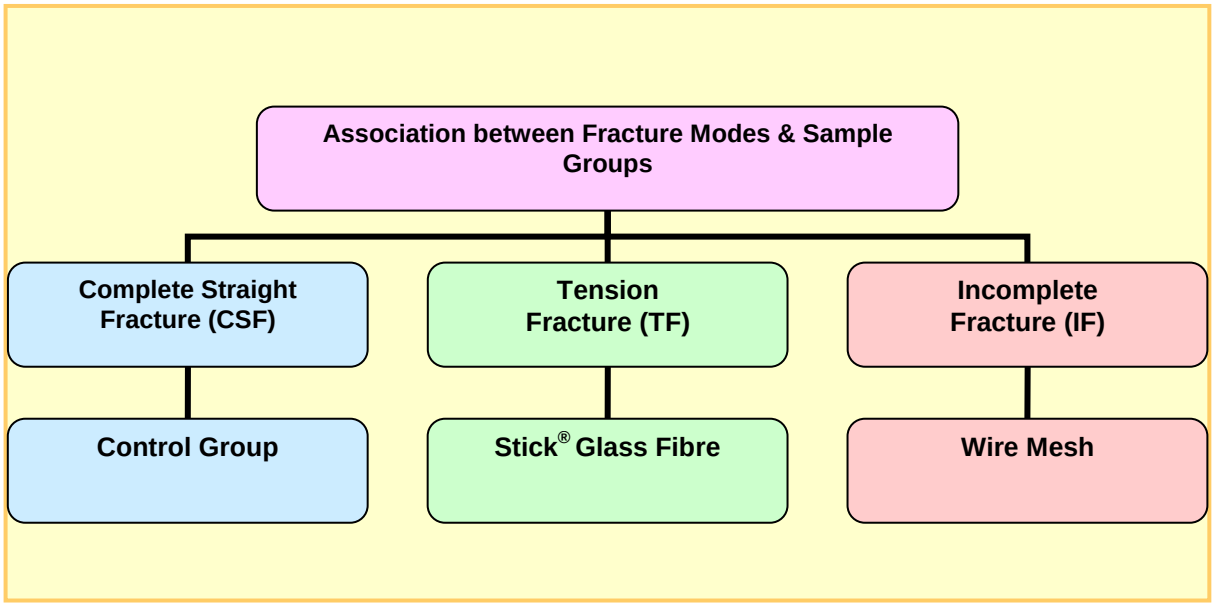
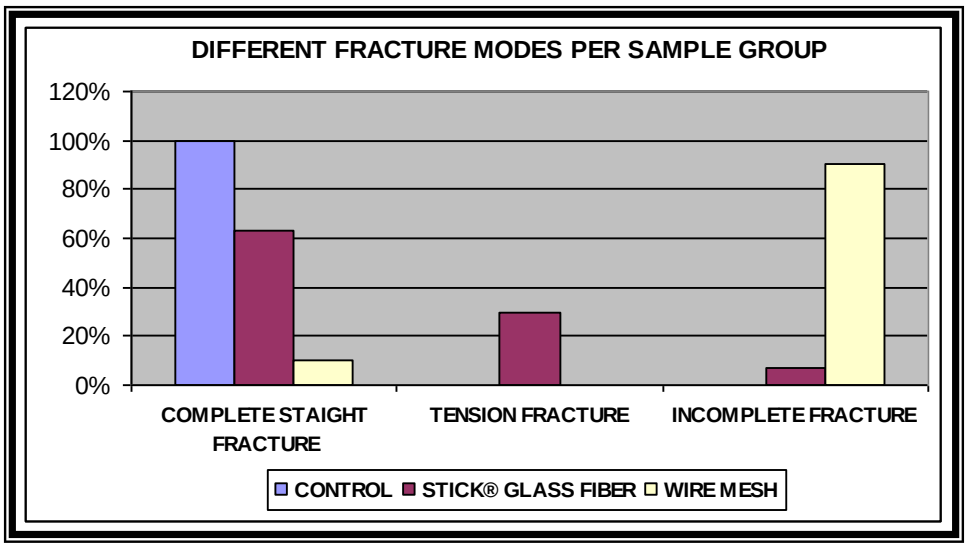


Figure 4.1: Association between Fracture Modes and Sample Groups

Graph 4.7 illustrates that 100% of the control group had Complete Straight Fracture (**CSF**). Tension Fracture (**TF**) was only prevalent in stick® glass fibre group (30%). Incomplete Fracture (**IF**), although common to both stick® glass fibre and wire mesh groups, was more prevalent in the wire mesh sample group (90%).

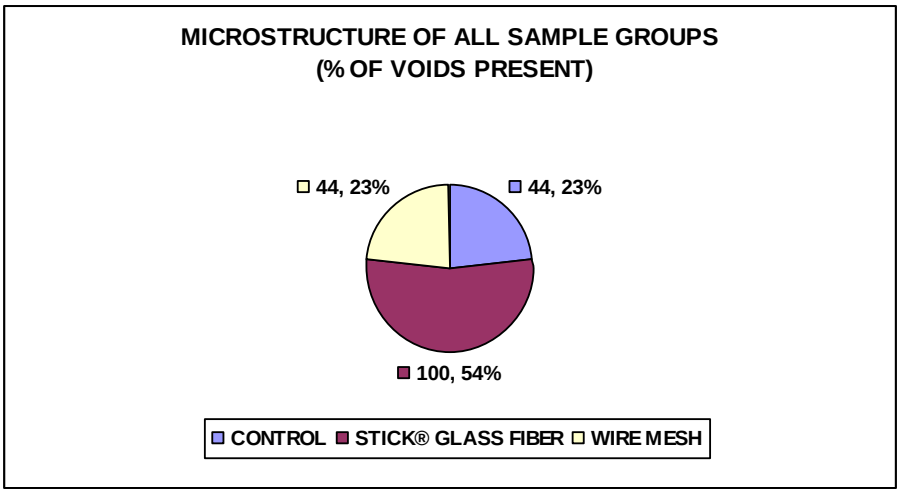


Graph 4.7: Different Fracture Modes per Sample Group

4.3 SEM ANALYSIS ON MICROSTRUCTURE OF PMMA FRACTURED SURFACES

4.3.1 Presence of Voids on the PMMA resin fractured surfaces

Microscopic analysis of the microstructure revealed that all the sample groups had voids as depicted in Graph 4.8. The stick® glass fibre group had 100% voids whilst the control and wire mesh groups had 44.23% each.



Graph 4.8: Microstructure (% of voids present) of all Sample Groups

4.3.2 Distribution and Impregnation of Stick® Glass fibres to PMMA Resin Matrix

Microscopic analysis of the transverse sections of the stick® glass fibres sample group revealed a high percentage of partial distribution and impregnation of the fibres to the PMMA resin matrix (Plate 4.4 and Graph 4.9).

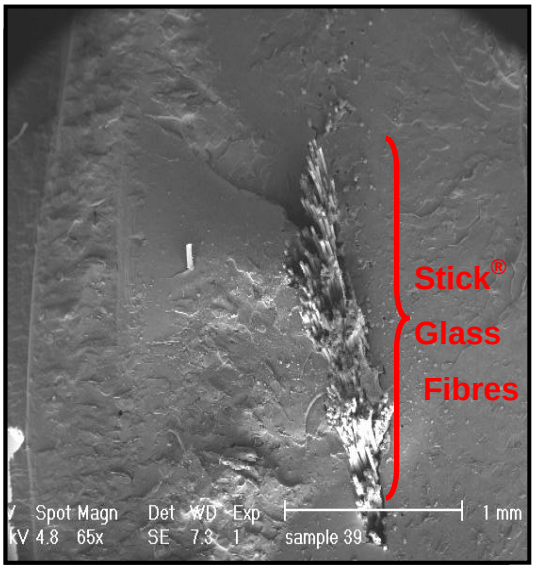
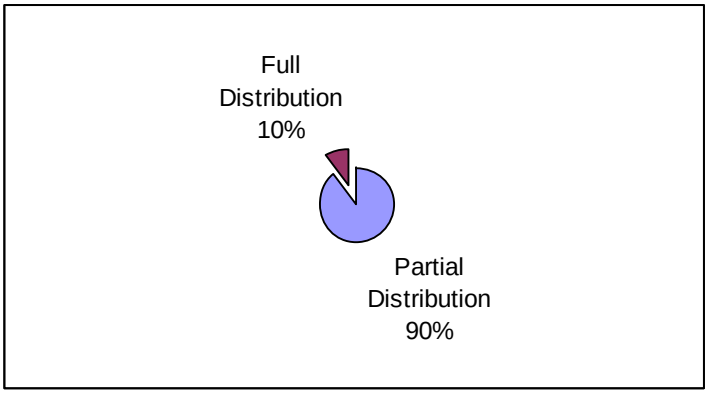


Plate 4.4: Partial distribution and impregnation of the Stick® Glass Fibres to the PMMA resin matrix
Scale bar: 1mm



Graph 4.9: % Distribution of Stick® Glass Fibres to the PMMA resin matrix

4.3.3 Bonding of Wire Mesh to the PMMA Resin Matrix

Microscopic analysis of the transverse sections of the Wire Mesh sample group revealed a high percentage of un-bonding between the wire mesh and PMMA resin matrix (Plate 4.5 and Graph 4.10.).

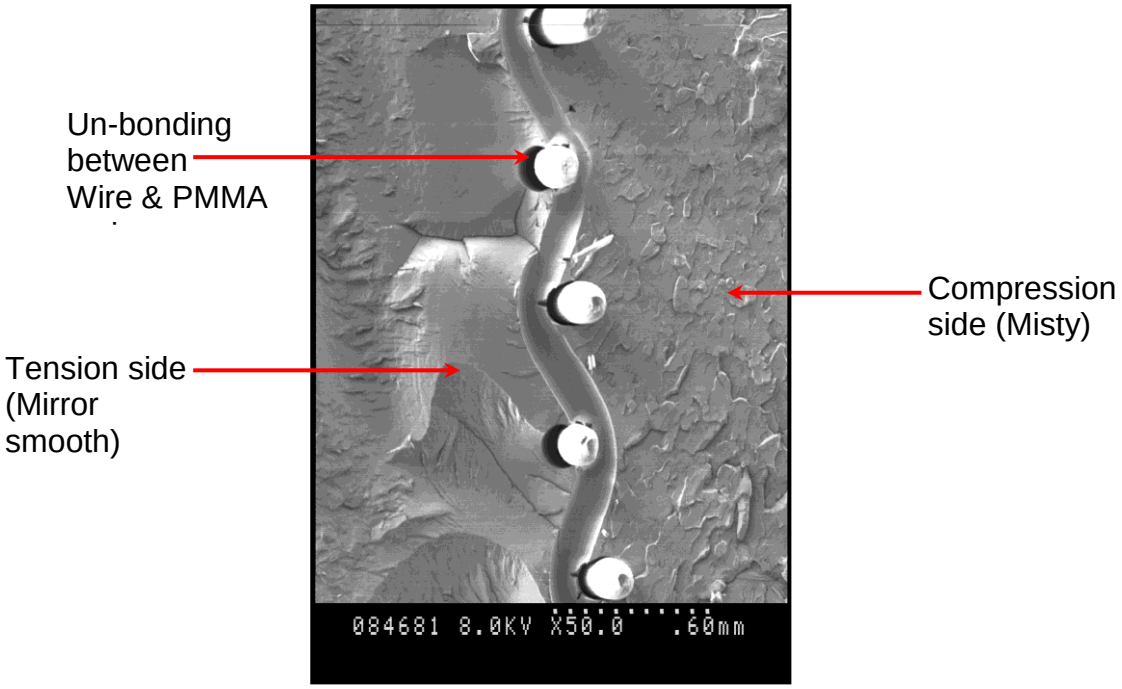
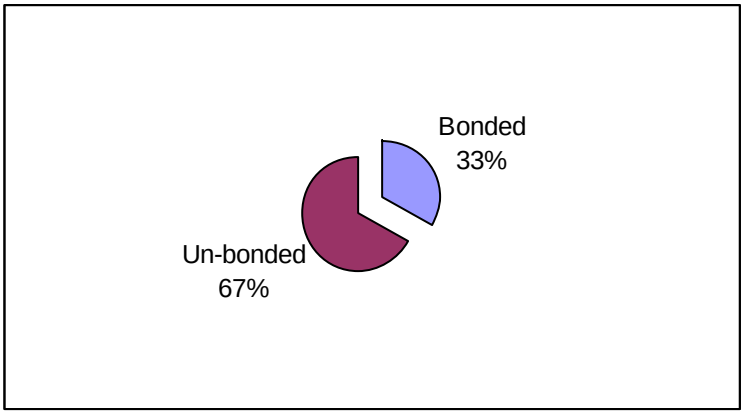


Plate 4.5: Un-bonding between wire mesh and PMMA resin matrix
Scale bar: 0.60mm



Graph 4.10: Percentage of bonded and un-bonded wire mesh to PMMA resin matrix

4.3.4 Surface Texture of Compression and Tension sides

Visual analysis of the PMMA resin fractured surfaces revealed two distinctive surface textures. The texture of the compression side was “misty” whilst the texture of the tension side was “mirror smooth” (Plate 4.4).

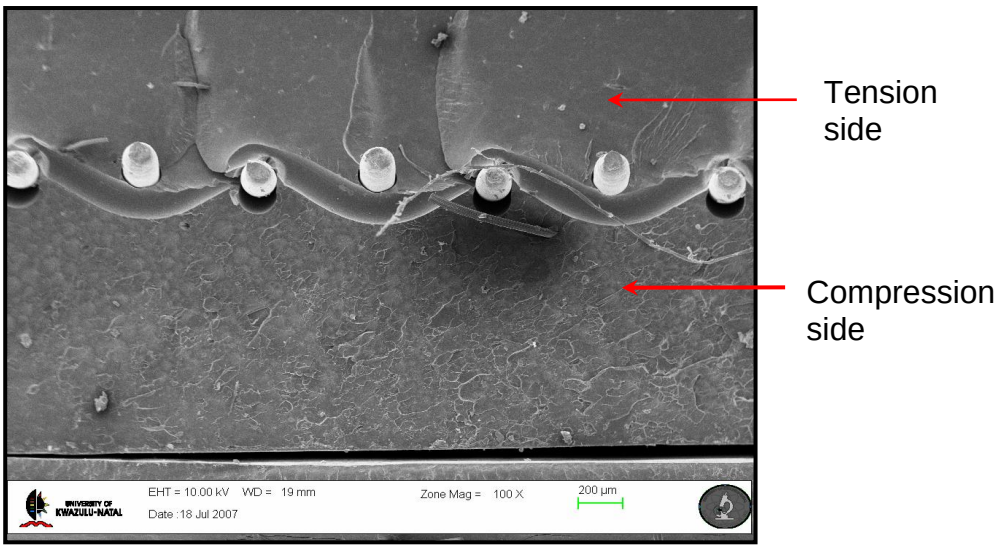


Plate 4.6: A specimen micrograph illustrating texture of the compression and tension sides

Quantitative analysis of surface textures of all sample groups revealed the following:

- Low texture values (approximately normally distributed) ranging within an average mean and standard deviation of 6.19 and 1.85, respectively (Table 4.6).
- High texture values (approximately normally distributed), ranging within an average mean and standard deviation of 5.99 and 2.34, respectively (Table 4.7).

Table 4.6: Low Texture Values of Sample Groups

DESCRIPTIVE STATISTICS	SAMPLE GROUPS			AVERAGE
	CONTROL	STICK® GLASS FIBRE	WIRE MESH	
MEAN	7.52	3.98	7.06	6.19
Std. Deviation	2.62	0.79	2.16	1.85
Std. Error of Mean	0.82	0.25	0.72	0.59

Table 4.7: High Texture Values of Sample Groups

DESCRIPTIVE STATISTICS	SAMPLE GROUPS			AVERAGE
	CONTROL	STICK® GLASS FIBRE	WIRE MESH	
MEAN	6.96	3.64	7.37	5.99
Std. Deviation	2.29	2.09	2.64	2.34
Std. Error of Mean	0.72	0.66	0.88	0.75

4.4 ASSOCIATION BETWEEN FLEXURAL STRENGTH & COMPONENTS OF MICROSTRUCTURE

Statistically, the Mann - Whitney test was performed to compare flexural strength between sample groups with and without voids. The flexural strength was higher in sample groups with voids than those without ($p = 0.035$, Appendix 8).

Although weak statistically the Spearman's correlation analysis revealed a significant negative correlation between flexural strength and low texture ($p = 0.002$, Appendix 8). A trend was identified that suggests increased flexural strength accompanied decreased low texture values. High texture values exhibited no correlation with flexural strength.

CHAPTER FIVE

Discovery consists in seeing what everyone else has seen and thinking what no one else has thought.
Albert Szent-Gyorgi (1937)

5. DISCUSSION

5.1 INTRODUCTION

The Intra-oral success of a poly (methyl methacrylate) resin denture base material significantly depends on the mechanical properties of the material and its resistance to biological degradation. An increase of PMMA resin denture bases fracturing during clinical service due to flexural fatigue, which is the continued flexing of the denture base during function, has therefore influenced the design of this in vitro study. The objective of the experiment was to investigate and establish the relationship between the flexural strength and microstructure of reinforced PMMA resin.

In general, the main findings of this study were that the flexural strengths of reinforced PMMA resin reflected a significant difference to the control after thermal exposure. The microstructure of PMMA resin, particularly stick® glass reinforced, was identified as a source of porosity when excess monomer was applied during the pre-impregnation process, consequently enhancing water absorption of the fibre reinforced composites.

5.2 ULTIMATE FLEXURAL STRENGTH

Statistical analyses showed the correlation between the flexural strength and stick® glass fibre and wire mesh reinforcements. This is manifest by the fact that the p-value, which allows one to make the decision to either accept or reject the null hypothesis, was less than 0.05 (Appendix 2).

Phillips and Moore (1994) reported that the average biting force in the posterior part of the mouth of a person with natural dentition is about 755N.

Anusavice (2003) further reported that according to the Guinness book of Records (1994) the highest biting force recorded is approximately 756N. However, Anusavice (2003) continued to argue that biting forces differ due to their dynamic nature during mastication. More importantly, the biting forces vary markedly from one area of the mouth to the other and from individual to another. Furthermore, the biting forces during mastication are absorbed by the food bolus, teeth, periodontal ligaments and underlying alveolar bone. The design of removable denture is therefore important to ensure that the denture base serves its intended functions effectively, safely and for a reasonable period. Conversely the average biting force of a person with complete and partial removable dentures is 111N (Craig, *et al*. 1992). These studies however failed to reveal the force used to calculate the flexural strength of PMMA resin based removable dentures, on which this study has focused.

During flexural strength testing the forces applied (which significantly influenced the calculation of flexural strength) to fracture the test specimens were not normally distributed (Section 4.1-Table 4.1, Graph 4.1, Appendix 1). The skewness statistic and standard error of skewness was 1.8 and 0.254 respectively (Appendix 2). Graph 4.3 clearly illustrates that the sample groups differed in median force applied to fracture the test specimens and there was a wide spread of data values, especially in the control group. Table 4.2 further depicts the difference in median force between the sample groups.

According to Brink (1990) non-parametric statistical methods are applied when the data is not normally distributed. In this study, the mean force applied to fracture the specimens was not a good indicator of the mid point of the data since it was pulled on one side by the outliers. The median force was therefore considered to be a more stable indicator of the midpoint. Hence non-parametric tests which are based on the median rather than mean were used for statistical analysis (Esterhuizen, 2007, Appendix 2).

In terms of force required to fracture the specimens, statistical analysis conducted using the Kruskal-Wallis test, reflected that a significant difference existed between the three sample groups (p= 0.002 - Appendix 2). Statistically, the Dunn's multiple range test for individual group comparisons further revealed that the control and the stick® glass fibre groups were significantly different from each other (p<0.01 - Appendix 2). The highest median force was applied in the stick® glass fibre sample group while the lowest was in the control sample group (Section 4.1 - Table 4.2). This is indicative that the stick® glass fibre reinforced PMMA resin was more resistant to fracture as opposed to the wire mesh reinforced PMMA. Unlike previous studies (Lonĉar, *et al*. 2006; Kim and Watts, 2004b; Jagger, *et al*. 2002; John, *et al*. 2001; Narva, *et al*. 2001), this study demonstrates that during clinical function PMMA resin denture base reinforced with stick® glass fibres require more force to fracture than conventional (un-reinforced) acrylic resin. The force applied to fracture the specimens was used to calculate the flexural strength as reflected in Table 4.3.

The flexural strength is a combination of compressive, tensile and shear strengths, all of which directly reflect the stiffness and resistance of a material to fracture (Anusavice, 2003; Jagger, *et al*. 2002; John, *et al*. 2001). The flexural strength of all sample groups as summarised in Section 4.1 (Tables 4.3 & 4.4, Graph 4.5) were also not normally distributed (Graph 4.4 - Appendix 2). The median flexural strength was highest in the stick® glass fibre group and lowest in the control group as reflected in the Table 4.5. The degree of skewness reflected was much more than twice its standard error between the control sample group and reinforced sample groups (Appendix 2). The skewness was associated with the processing errors or different rates of water absorption between the sample groups, which were delimited in this study. Consequently this validated the use of non-parametric tests where the median was used to establish whether significant differences existed between all the sample groups (Esterhuizen, personal communication by email. 27 August 2007).

In terms of flexural strength, Graph 4.6 (Section 4.1) suggests that a significant difference existed between each of the sample groups as there was a minimal overlap in distribution (Esterhuizen, personal communication by email. 27 August 2007). Statistical analysis confirmed that significant differences in flexural strength existed between the three sample groups ($p<0.001$, Appendix 2), with the stick® glass fibre reinforced group being the strongest. This is indicative of the superior material properties of the stick® glass fibre reinforcements.

According to (Kuzma and Bohnenblust, 2005; Brink, 1990) an important application of the mean and the standard deviation is the coefficient of variation (CV), which depicts the size of the standard deviation relative to its mean. In this study, the CV of the stick® glass fibre sample group was higher than both the wire mesh and control sample groups (Section 4.1-Table 4.5). This further confirmed that differences in the flexural strength between the sample groups existed.

This study concurs with previous investigators (Çökeliler, *et al*. 2007; Nohrström, *et al*. 2006; Tacir, *et al*. 2006; Karacaer, *et al*. 2005; Aydin, Yilmaz and Caglar, 2002; Vallittu, 1998; Ramos, Runyan and Christensen, 1996) who have revealed that significant differences exist between glass fibre and wire/metal reinforced PMMA resins. They have further observed that the flexural strength of glass fibre reinforced PMMA resin primarily depends on the impregnation of the glass fibres to the matrix and the degree of water absorption. However, these studies failed to simulate the in vivo function of the PMMA resin denture base material and the effects of thermal stresses on the flexural strength of reinforced PMMA resins on which this study has focused.

Although a durable adhesion between glass fibres and PMMA resin matrix is desired to ensure an efficient stress transfer between the phases of the material, little attention has been paid to the effect of thermal stresses on the fibre-resin matrix interfacial bond strength, on which this study has focused. In

comparison to PMMA resin possessing a high coefficient of thermal expansion (CTE) of 81 x 10^{-6} / ˚C (Anusavice, 2003), stick® glass fibres have a very low CTE of 5.0 x 10^{-6} / ˚C (Tezvergil, Lassila and Vallittu, 2003). These differences between thermal coefficients of the PMMA resin and stick® glass fibres could have contributed to the high interfacial strains during the thermocycling process. This study therefore corroborates the findings of Janda, Roulet, Latta and Ruttermann (2005), and Merice and Ruyter (2006) that resin based materials show a decrease in flexural strength after thermocycling. This is critical as failure of the interfacial adhesion between glass fibres and PMMA resin matrix is associated with thermal strains. Without adequate interfacial adhesion between the phases, the fibres act as voids in the PMMA resin matrix, thereby ultimately weakening the flexural strength of PMMA resin (Nohrström, *et al*. 2000).

Several authors (Lonĉar, *et al*. 2006; Chai, *et al*. 2004; Kim and Watts, 2004a; Ellakwa, Shortall and Marquis, 2002; Vallittu, 1998; Miettinen and Vallittu, 1997; Vallittu, 1992) have reported that voids, cracks, or un-bonded interfaces between PMMA resin matrix and glass fibres increase water absorption. These absorbed water molecules are able to penetrate polymer materials due to their weak secondary bonds and act as a plasticiser (Chai, *et al*. 2004; Rantala, *et al*. 2003; O'Brien, 2002).

Water absorption negatively impacts on the mechanical properties of reinforced PMMA resin (Narva, *et al*. 2004). Regardless of the stick® glass fibre sample group containing unidirectional E-glass® fibres to decrease water absorption and improve dimensional stability, voids were present within the PMMA matrix. It is therefore postulated that such voids contributed to the reduced flexural strength of the PMMA resin. This study is in agreement with Narva, *et al.* (2004) and Vallittu (1998) who reported that E-glass® and Silica-glass® reinforced FRC decreased their flexural strength mainly during the first 4 weeks of immersion in water.

Visual examination revealed that an association between sample group and their fracture mode existed (Section 4.1, Figure 4.1 and Graph 4.7). Pearson's chi-square statistical analysis further confirmed the association between the sample group and their fracture mode ($p<0.001$, Appendix 2). The control sample group (un-reinforced) demonstrated a smooth complete straight fracture (Section 4.2 - Plate 4.1). Conversely, the stick® glass fibre and wire mesh groups revealed different types of fractures as discussed in the ensuing section.

The IPN (Inter-polymer Penetrating Network) structure of stick® glass fibres bonding with PMMA resin matrix contributed to the tension fracture mode of the stick® glass fibre sample group (Section 4.2 - Plate 4.2). Consequently, the force applied produced stresses that were transferred to the fibres. This corroborates several studies (Narva, *et al*. 2005; Lassila and Vallittu, 2004; Nohrström, *et al*. 2000) that fibre reinforcements placed on the tension side of the test specimen under flexural loading produce considerably higher flexural strength as stresses are transferred from the polymer matrix to the fibres.

It is apparent that wire mesh reinforcements provide a mechanical reinforcement to PMMA resin. Consequently, this largely contributed to the incomplete fracture mode (Section 4.2-Plate 4.3). In agreement with Vallittu (1996), the significant difference between the "IF" and "CSF" modes is that "IF" resin pieces are held together by the wire mesh. From a clinical and biological perspective this is critical as it reduces the risk of the patient swallowing the fractured pieces (Hashmi, Walter, Smith and Latis, 2004; Frankling, *et al.* 2003; O'Brien, 1997). The differences in fracture modes between the control, stick® glass fibre and wire mesh sample groups that existed is therefore directly related to the significant differences in flexural strength.

Statistically, this study produced significant differences in flexural strength between the reinforced sample groups and control sample group. The hypothesis for objective one, that the stick® glass fibre and wire mesh

reinforced PMMA resin denture base acrylic will produce different levels of flexural strengths to the control, and will thereby set parameters for comparison, was therefore accepted.

5.3 MICROSTRUCTURE OF PMMA FRACTURED SURFACES

The microstructure of PMMA resin fractured surfaces was scrutinized in order to employ a quantitative measurement tool when analysing the presence of voids, surface texture , distribution and impregnation of the stick® glass fibres, and bonding of wire mesh to the PMMA resin matrix. However, only presence of voids and surface texture could be quantified. A qualitative tool was therefore employed that analysed the impregnation and bonding of the reinforcements to the PMMA resin matrix. A discussion on the reasons for accepting the validity of these findings follows.

5.3.1 The Presence of Voids on the PMMA resin fractured surfaces

SEM analysis of transverse surfaces of PMMA resin revealed the presence of voids in all the sample groups. Statistically, in terms of microstructure (% of voids), a significant difference existed between all sample groups ($p = 0.013$, Appendix 3) with the stick® glass fibre sample group exhibiting voids in all specimens, particularly at or near the fibre–resin matrix interface. Although the control and wire mesh sample groups shared the same numbers of voids, the number of voids present was significantly lower as compared to stick® glass fibre sample group (Section 4.3-Graph 4.8).

The presence of voids in the stick® glass fibre sample group was associated with the polymerization shrinkage due to the excess monomer applied during pre-impregnation procedures. This is in agreement with several authors (Vojdani and Khaledi, 2006; Chai, *et al*. 2004; Lassila, 2003; Narva, *et al*. 2001; Vallittu, 1992; Ruffino, 1985) who have reported that regardless of the good

impregnation of glass fibres to the PMMA resin matrix, voids were present. Such voids were located at or proximal to the fibre-matrix interface.

The correct polymer/monomer ratio fundamentally influences the physical properties of a PMMA resin denture base material (Anusavice, 2003). The polymer/monomer pre-impregnation process used for stick® glass fibres, which is partly user controlled, has no documented mixing ratio. It is therefore postulated that this may have contributed in excess monomer being applied during the wetting of the stick® glass fibres. This study further concurs with Lassila and Vallittu (2004) that excess residual monomer and high void volume content significantly decreases the flexural properties of FRC's. It is therefore impractical to expect precise control from even a skilled dental technician when polymer/monomer pre-impregnation procedures are employed unless quantified ratios are used.

5.3.2 Distribution and Impregnation of Stick® Glass Fibres to the PMMA resin matrix

After exposure of all test specimens to thermocycling and flexural strength testing, SEM analysis was conducted on transverse section of the fractured surfaces in order to analyse the interface between the reinforcements and the PMMA resin matrix. It has been documented that adhesion of stick® glass fibres to the PMMA resin matrix is further enhanced by the unique Interpenetrating Polymer Network structure (IPN structure) within stick® glass fibre (Sticktech Online, 2006-08-23 and Stickbond Online, 2006-08-25). Although this method of pre-impregnation was applied in this study, partial distribution and impregnation of the fibres to the PMMA matrix resulted as illustrated in Plate 4.4. Graph 4.9 further reflects that 90% of the stick® glass fibre reinforced specimens were partially distributed whilst 10% had full distribution. The ensuing discussion will therefore highlight on the possible technical causes that may have contributed to the partial distribution and impregnation of the stick® glass fibres.

The common practice of section and adding stick® glass fibres to the PMMA resin dough during fabrication is by hand. However, this influences the lateral spreading of fibres in the PMMA resin, which is largely controlled by the user. In corroboration with Vallittu (1999), difficulty is experienced during handling of glass fibres, specifically during sectioning to the required dimensions, and accurately incorporating the glass fibres in the desired region of the PMMA resin. These technical difficulties contributed to the poor impregnation and distribution of stick® glass fibres in the PMMA resin in this study.

Furthermore, according to Vallittu *et al*. (1995) poor wetting of glass fibres and polymerization shrinkage of the PMMA resin also contributes to the layer of resin on the surface of the fibres being destroyed. This is further compounded by the bench press method of mixing the dough and the pre-impregnated stick® glass fibres as an in-homogenous distribution of fibres throughout the PMMA resin results. Ultimately this decreases the bond between the fibres and the polymer.

Not withstanding the above, this study concurs with Lastumäki *et al*. (2003), that fibres must be well impregnated and evenly distributed within the PMMA resin matrix as this enhances the mechanical strength of the prosthesis. Moreover, stick® glass fibres or wire mesh that is not well integrated within the matrix enhances crack propagation and is characteristic of the fibres having poorly bonded inter-facially. Consequently, stress concentration increases as fibres may be plucked out of the polymer matrix thereby weakening the PMMA resin denture base material. From a biological perspective, the poorly impregnated regions attract oral microbes into the voids of the PMMA resin denture base. Inevitably this negatively impacts on the aesthetics and hygiene of the denture base.

5.3.3 Bonding of Wire Mesh to PMMA Resin Matrix

In reviewing the literature with regards to wire/metal reinforcements, this study further corroborates with Kim and Watts (2004b), Teraoka (2001), Vallittu

(1996), Ruffino (1985) and Carrol and Fraunhofer (1984), that metals or wire mesh do not chemically bond to PMMA resin and produce an un-aesthetic appearance beneath the resin. Consequently, the PMMA resin material around the wire mesh withdraws, leaving the material with voids that may weaken the structure by creating new points of stress concentration (Section 4.3 - Plate 4.5). Graph 4.10 further confirms that 67% of wire mesh was un-bonded to the PMMA resin matrix whilst 33% was bonded.

In order to improve adhesion between PMMA resin and metal reinforcements several methods such as silanization of the metal, sandblasting of metal wire with aluminum oxide (Al_2O_3) and metal adhesive resins have been recommended (Vojdani and Khaledi, 2006; Kim and Watts, 2004a). However, these methods of improving adhesion between wire and PMMA resin are delimitated in this study.

Statistically this study produced significant differences in components of microstructure (% voids and texture). Therefore, the hypothesis for objective two that the stick® glass fibre and wire mesh reinforced PMMA resin denture base material will have differences in microstructure to the control, and thereby set parameters for comparison was accepted.

5.3.4 Surface Texture of the Compression and Tension Sides

The upper and lower portions of the test specimens in this study during flexural testing were in compression and tension respectively (Figure 5.1). Consequently, the stresses on the upper surface were compressive whereas those on the lower surface were tensile. Shear stress was also produced near the supported ends of the specimen, but did not play a significant role in the fracture process (Anusavice, 2003). The differences in surface texture of the compression and tension sides of the test specimens were therefore associated with compressive and tensile stresses respectively (Section 4.3, Plate 4.6).

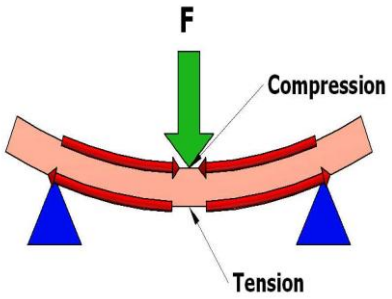


Figure 5.1: Diagrammatic presentation of a 3-point (flexural) bending test

The PMMA resin test specimens experienced irreversible strain (plastic) behaviour due to the polymer chains sliding over one another and becoming relocated within the material. Consequently, the material becomes permanently deformed (Anusavice, 2003). This phenomenon influences the type of stress produced, especially when a force is exerted, due to the texture of the material.

As the force applied to fracture all the sample groups was significantly different, this directly impacted on the magnitude of the stress applied on the PMMA resin specimens. Consequently, this produced differences in surface textures of the compression and tension sides. In corroboration with Gang Ma and Xia (2004) and Vallittu (1996) the fractured surfaces of the PMMA resin appeared “misty” on the compression side adjoining a “mirror smooth“ surface on the tension side.

The stick® glass fibre group revealed the lowest mean value of “low texture” values. There was evidence of a significant difference between the stick® glass fibre group and other two groups with regard to “low texture” values (p=0.001- Appendix 3). With reference to “high texture” values the stick® glass fibre group further revealed low mean values (Section 4.3, Table 4.7). Statistical analysis (ANOVA) of the mean of “high texture” values indicated that a significant difference existed between the three sample groups (p= 0.003 – Appendix 3). Bonferroni multiple comparison post hoc tests for “high texture”

values between the three groups further revealed that the stick® glass fibre group was significantly lower than the control (p= 0.012 – Appendix 3) and the wire mesh group (p= 0.006), but the control group and wire mesh group were not significantly different from each other (p= 1.00).

It is therefore postulated that the significant differences in texture between the groups is associated with the differences in material composition of the sample groups. By virtue of this characteristic and in comparison to the wire mesh and control sample groups, stick® glass fibres absorbed more compressive and tension stresses present within the PMMA resin.

5.4 ASSOCIATION BETWEEN FLEXURAL STRENGTH AND MICROSTRUCTURE

Past studies have investigated either the flexural strength of glass fibre reinforced PMMA resin (Tacir, *et al*. 2006; Lonĉare, *et al*. 2006 Kim and Watts, 2004b; Jagger, *et al*. 2002; John, *et al*. 2001; Narva, *et al*. 2001; Vallittu, 1999), effects of polymerization cycle on the flexural strength of PMMA resin (Sedda , Borracchini, Monticelli, Goracci and Ferrari, 2006;), PMMA resin denture base fractures (Darbar, *et al*. 1994), fatigue resistance of glass fibre reinforced PMMA resin (Narva, *et al*. 2004; Rantala, *et al*. 2003) or water absorption (Chai, Dlaw, Takashi, Hisama and Shimizu, 2004; Takashi, Chai and Kawaguchi,1999; Dogan, Bek, Çevik and Usanmaz, 1995,). However, there appears to be minimal documentation on the association between microstructure and flexural strength of PMMA resin after thermal exposure simulating the oral environment.

Qualitative analysis of impregnation and bonding of stick® glass fibres and wire mesh to PMMA resin matrix reinforces the notion that increased thermal changes and exposure time affects not only the microstructure of PMMA resin but significantly influences the propensity towards water absorption.

Statistically, the Mann-Whitney test was used to compare flexural strength between sample groups with and without voids. The flexural strength was higher in sample groups with voids than those without (Section 4.4 - p = 0.035, Appendix 4). However, this does not imply that voids increase the flexural strength of PMMA resin as they were produced as a result of the technical procedures involved in the fabrication of FRC's (p = 0.002, Appendix 3). The ensuing discussion is intended to clarify on the negative correlation coefficient revealed in this study between flexural strength and microstructure (texture) values.

According to Brink (1990) the correlation coefficient (r) is an index that measures the strength or magnitude and direction of a linear relationship between two variables in terms of summarising how perfect the relationship is. The possible values for a correlation range from -1.00 to +1.00. If two related variables are perfectly correlated, the correlation coefficient is expressed as 1.00. When variables are totally unrelated the correlation coefficient is equal to zero. When there is a perfect inverse relationship between two variables the correlation coefficient is -1. Therefore numbers between (+1 and 0) and (-1 and 0) indicate intermediate degrees of relatedness (Brink, 1990). In this study the variables being correlated were flexural strength and microstructure in terms of texture.

Statistically the Spearman's Correlation analysis revealed no association between "high texture" values and flexural strength (r= -0.294 & p= 0.122, Appendix 4). The "high texture" was observed on the compression side of the test specimens. Although weak, a significant negative correlation existed between flexural strength and "low texture values" (r = -0.542 & p= 0.002, Appendix 4). Thus there was a trend that suggested that the higher the flexural strength the lower the "low texture values". It is therefore further postulated that the stick® glass fibres and wire mesh, which were positioned on the tension side of the test specimen, contributed to the significant correlation between flexural strength and "low texture" values. This concurs with Tacir *et al*. (2006)

and John *et al*. (2001) that the high modulus of elasticity (ratio of stress to strain) of glass fibres allows tensile stresses to be absorbed thereby producing “low texture” values.

Statistically this study produced significant differences between flexural strength and components of microstructure (% voids and low texture values). Therefore the hypothesis of objective three that a relationship exists between flexural strength and components of microstructure of stick® glass fibre and wire mesh reinforced PMMA resin denture base material is accepted.

CHAPTER SIX

I have been impressed with the urgency of doing. Knowing is not enough, we must apply. Being willing is not enough, we must do.

(Leonardo da Vinci, 1452–1519).

6. CONCLUSION AND APPLICATION OF FINDINGS

6.1 ULTIMATE FLEXURAL STRENGTH

The statistical difference in flexural strength values of PMMA resin between the reinforced groups and control group conclusively indicated that:

- Reinforcing PMMA resin denture base material with stick® glass fibres significantly improves the flexural strength, consequently increasing the life span of the prosthesis during clinical use.
- Stick® glass fibre reinforcement offers a metal free, strong and economical alternative for strengthening PMMA resin denture base material for most composite and PMMA resin laboratory processed units. From a Dental Technology perspective, the inclusion of fibre technology in restorations increases the range of clinical services with maximum benefits, specifically to dental specialists.

In addition, reinforcing PMMA resin denture base material with gilded wire mesh, although improving the mechanical properties, is aesthetically not appealing to the patient as it imparts a dark colour to the PMMA resin. In addressing this, stick® glass fibre reinforcements were introduced to the South African Dental industry. Such fibre technology therefore supports the need for further research in providing systemic data on intra-oral performance of reinforced denture base resins.

6.2 MICROSTRUCTURE OF THE REINFORCED PMMA RESIN

Through SEM analysis, this study was able to observe the microstructure of the reinforced PMMA resin material in determining its influence on the flexural strength.

- The findings of this study confirm that differences in microstructure exist between the stick® glass fibres reinforced, wire mesh reinforced and un-reinforced PMMA resin denture base material. The combined effects of pre-impregnation process and the microstructure at the resin-fibre interface positively enhance the flexural strength of PMMA resin denture base material.
- This study has explicitly underlined the need for dental manufacturers of stick® glass fibres to formulate a mixing ratio of polymer/monomer used during pre-impregnation of fibres.
- To improve adhesion between PMMA resin and wire mesh reinforcement pre-treatment methods such as silanization of the wire mesh, sandblasting of wire mesh with aluminium oxide (Al_2O_3) and application of adhesive resins is recommended. Herein lies an important area for future research.

6.3 ASSOCIATION BETWEEN FLEXURAL STRENGTH & MICROSTRUCTURE

The findings of this study confirm that an association between flexural strength and microstructure of the stick® glass fibres reinforced PMMA resin exists.

Although no relationship exists between texture of the compression side and flexural strength, a relationship did exist between flexural strength and texture of the tension side. This was as a result of the stick® glass fibres reinforcement absorbing applied stresses. It is therefore concluded that if denture reinforcements

are positioned on the tension side of a denture base they resist masticatory forces. Consequently fracture resistance of the PMMA resin denture base increases. This enhances the life span of the denture intra-orally.

6.4 APPLICATION OF FINDINGS

Contemporary rehabilitation of edentulous patients encompasses an ever-widening spectrum of clinical and laboratory responsibilities. Thus, excellent patient care requires that clinicians and technicians be familiar with modern methods of assessing the patient needs, advances in technology and progressive methods of fabricating prostheses. Moreover, the clinician and technician need to evaluate the appropriateness of a range of prostheses or procedures in light of established therapeutic practice.

Where fracture strength is questionable, particularly in patients with heavy occlusal load, the PMMA resin denture base material must be reinforced with stick® glass fibres. Such fibre technology provides better clinical service in terms of longevity of the appliance intra-orally.

From a clinical perspective, some structures contained within the denture depend on the toughness of the PMMA resin material used. For example, in over-dentures the regions of precision attachments are often thin. However, the application of fibres in that region may diminish the risk of perforation of the denture base. The margins of laboratory fabricated crowns also critically depend on the toughness of the material as this eliminates marginal defects during clinical procedures. Therefore, in order to find the long term data especially on clinical behaviour of these new fibre reinforcement systems, further research is required.

It is therefore proposed that, in order for dental manufacturers to remain strategically viable on the competitive dental market, the pre-impregnation properties of the present stick® glass fibres be improved. This new and/or improved reinforcing material should be:

(i) chemically compatible with PMMA resin,

(ii) readily pre-impregnated, and

(iii) biocompatible.

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APPENDIX 1: THE THERMOCYCLING MACHINE

The chambers of the thermocycling machine (Plate 1) were filled with tap water. The temperatures of the chambers were maintained at 55˚C and 5˚C respectively by the digital thermostat. The specimens were then exposed to these chambers cyclically, i.e. the specimens were completely immersed and moved automatically from the cold to hot chamber and vice versa. As a precaution water was often added to overcome any loss due to evaporation and spilling.

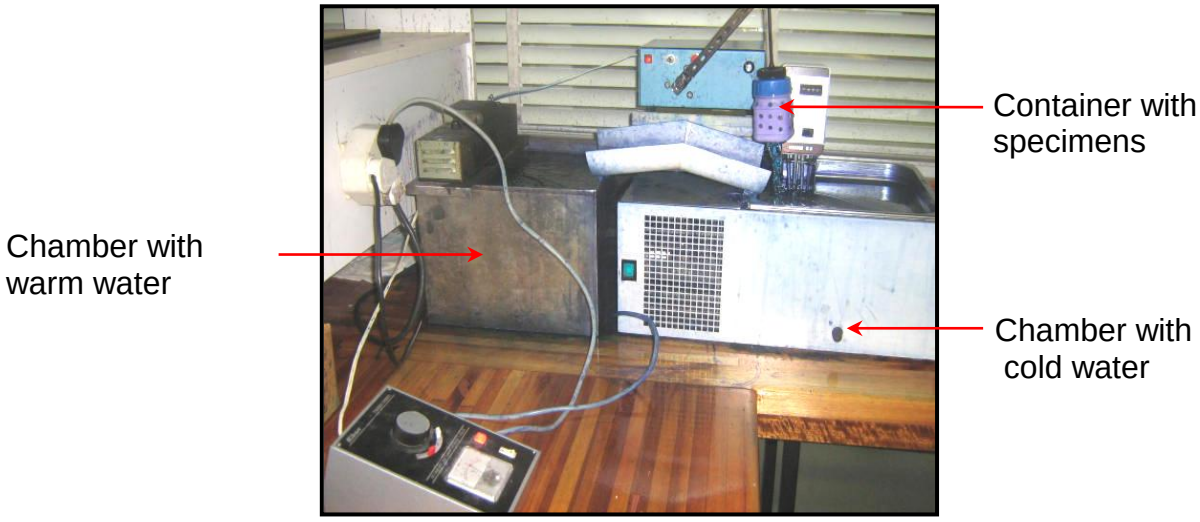

Plate 1: Thermocycling Machine

APPENDIX 2: STATISTICAL RESULTS OF FLEXURAL STRENGTH

Kruskal-wallis Test to compare force between the sample groups

	Group	N	Mean Rank
Force (N)	control	30	33.93
	stick® glass fiber	30	57.85
	wire mesh	30	44.72
	Total	90	

	Force (N)
Chi-Square	12.612
df	2
p value	0.002

P < 0.05

Dunn's Multiple Comparison Post hoc Test to compare force between the sample groups

Dependent Variable	(I) Group	(J) Group	Mean rank difference	p value
Force (N)	control	stick® glass fiber	-23.917	<0.01
	control	wire mesh	-10.783	>0.05
	stick® glass fiber	wire mesh	13.133	>0.05

P < 0.05

Kruskal-wallis Test to compare flexural strength (Mpa) between the three groups

	Group	N	Mean Rank
Flexural Strength	control	30	22.60
	stick glass fiber	30	63.62
	wire mesh	30	50.28
	Total	90	

	Flexural Strength
Chi-Square	38.484
df	2
p value	<0.001

P < 0.05

Dunn's multiple comparison post hoc Test for flexural strength (Mpa)

Dependent Variable	(I) Group	(J) Group	Mean rank difference	p value
Flexural strength	control	stick glass fiber	-41.017	<0.001
	control	wire mesh	-27.683	<0.001
	stick glass fiber	wire mesh	13.333	>0.05

P < 0.05

Standard error of skewness of Force and Flexural strength

		Force (N)	Flex Strength
N	Valid	90	90
	Missing	0	0
Mean		128.7546	99.84674
Median		119.9400	98.21250
Std. Deviation		36.07643	21.363134
Skewness		1.811	1.011
Std. Error of Skewness		.254	.254
Minimum		76.67	61.460
Maximum		266.83	176.308
Percentiles	25	108.7100	88.23125
	50	119.9400	98.21250
	75	137.7875	109.47292

Skewness is more than twice standard error of skewness

Cross tabulation between group and fracture mode

			Fracture mode			Total
			broke into two separate pieces	Fracture originating from tension side	fracture incomplete	
Group	control	Count	30	0	0	30
		% within Group	100.0%	.0%	.0%	100.0%
	stick glass fibre	Count	19	9	2	30
		% within Group	63.3%	30.0%	6.7%	100.0%
	wire mesh	Count	3	0	27	30
		% within Group	10.0%	.0%	90.0%	100.0%
Total		Count	52	9	29	90
		% within Group	57.8%	10.0%	32.2%	100.0%

Pearson's chi square 86.10, p<0.001

P < 0.05

APPENDIX 3:STATISTICAL RESULTS OF MICROSTRUCTURE

Cross tabulation between group and presence of voids

			Voids		Total
			yes	no	1
Group	control	Count	4	5	9
		% within Group	44.4%	55.6%	100.0%
	stick® glass fiber	Count	10	0	10
		% within Group	100.0%	.0%	100.0%
	wire mesh	Count	4	5	9
		% within Group	44.4%	55.6%	100.0%
Total		Count	18	10	28
		% within Group	64.3%	35.7%	100.0%

Pearson's chi square 8.642, p=0.013

P < 0.05

ANOVA comparison of mean low texture values between sample groups

	Sum of Squares	df	Mean Square	F	p value
Between Groups	73.365	2	36.682	9.106	0.001
Within Groups	104.740	26	4.028		
Total	178.104	28			

P < 0.05

Bonferroni multiple comparison post hoc tests for low texture

Dependent Variable: Low texture

Bonferroni

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	p value	95% Confidence Interval	
control	stick glass fiber	3.54140(*)	.89760	0.002	1.2445	5.8383
	wire mesh	.46037	.92220	1.000	-1.8995	2.8202
stick glass fiber	control	-3.54140(*)	.89760	0.002	-5.8383	-1.2445
	wire mesh	-3.08103(*)	.92220	0.008	-5.4409	-.7212
wire mesh	control	-.46037	.92220	1.000	-2.8202	1.8995
	stick glass fiber	3.08103(*)	.92220	0.008	.7212	5.4409

* The mean difference is significant at the .05 level.

Descriptive statistics for high texture values by Sample group

Group	Mean	N	Std. Deviation	Std. Error of Mean
control	6.96340	10	2.292647	0.724999
stick® glass fiber	3.64390	10	2.089977	0.660909
wire mesh	7.37144	9	2.635782	0.878594
Total	5.94538	29	2.827363	0.525028

ANOVA comparison of mean high texture values between Sample groups

	Sum of Squares	df	Mean Square	F	p value
Between Groups	81.635	2	40.817	7.463	0.003
Within Groups	142.197	26	5.469		
Total	223.832	28			

P > 0.05

Bonferroni multiple comparison post hoc tests for high texture values

Dependent Variable: High texture

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	p value	95% Confidence Interval	
control	stick glass fiber	3.319500(*)	1.045859	0.012	.64321	5.99579
	wire mesh	-.408044	1.074518	1.000	-3.15768	2.34159
stick glass fiber	control	-3.319500(*)	1.045859	0.012	-5.99579	-.64321
	wire mesh	-3.727544(*)	1.074518	0.006	-6.47718	-.97791
wire mesh	control	.408044	1.074518	1.000	-2.34159	3.15768
	stick glass fiber	3.727544(*)	1.074518	0.006	.97791	6.47718

* The mean difference is significant at the .05 level.

Paired t-test to compare mean high and low texture values

	Mean	N	Std. Deviation	Std. Error Mean
Low texture	6.1597	29	2.52208	0.46834
High texture	5.94538	29	2.827363	0.525028

	Paired Differences					t	df	p value
	Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference				
Low texture - High texture	0.214276	2.749807	.510626	-.831695	1.260246	0.420	28	0.678