OPTIMISATION OF TITANIUM-HYDROXYAPATITE COMPOSITES USING BIOACTIVE GLASS FOR FABRICATION OF FUNCTIONALLY GRADED DENTAL POSTS

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OPTIMISATION OF TITANIUM-HYDROXYAPATITE COMPOSITES USING BIOACTIVE GLASS FOR FABRICATION OF FUNCTIONALLY GRADED DENTAL POSTS

ABSTRACT

Optimisation of the chemical, physical and mechanical properties of Titanium-hydroxyapatite (Ti-HA) composites using various ratios of bioactive glass to fabricate a functionally graded dental posts (FGDPs). Bioactive glass (BG) (containing SiO_2, NaO, CaO, B_2O_3, P_2O_5, CaF_2, MgO, TiO_2) was prepared and added in three different ratios (5%, 10%, and 15%) to five different Ti-HA composites. Section 1; mixtures were prepared, milled, pressed, sintered and visually checked. In section 2; the optimum ratio of bioactive glass was selected according to section 1 results and was then added to the Ti-HA composites and divided into two groups, each containing five layers. Samples in Group 1 were sintered with an air atmosphere furnace, while samples in Group 2 were sintered with a vacuum furnace and then all groups were tested with XRD and EDX. Density, micro-hardness, modulus of elasticity and compression strength was also measured. Section 3; based on the results in part 2, functionally graded dental posts (FGDPs) were then fabricated. Section 4; FGDPs and other post types were restored in endodontically treated teeth with 10 teeth in each group. Fracture resistance and failure modes were evaluated using a universal testing machine and stereomicroscope and data were analysed using one-way ANOVA and Chi-square tests. Section 5; stress distributions were also evaluated using teeth models restored with the FGDPs, titanium, fibre, and stainless steel posts and analysed using finite element analysis. Ti-HA composites that were incorporated with a 10% ratio of BG were stable and crack free when compared to other groups. In the second section, samples sintered in Group 1 (air atmosphere furnace) showed a significant difference (p= 0.000) as compared to Group 2 (vacuum furnace), in terms of compression strength, micro-hardness values density and modulus of elasticity. In terms of XRD analysis, Group 1 also showed that major phases belonged to Ti and HA, and a minimum amount of Ti was oxidised to Ti_2O. Whereas, in Group 2; the main phases belonged to Ti and HA, and the minor phases showed that HA had decomposed into TCP and TTCP. EDX also showed that there was no contamination recorded in both groups. In terms of fracture resistance, FGDPS and titanium groups showed a higher fracture resistance compared to the stainless steel group (p= 0.000 and p= 0.032, respectively). In terms of failure modes, FGDPs group also showed higher restorable failures than other groups, followed by the fibre group. In terms of stress distribution, regardless of load direction, maximum stress was concentrated at the core-crown interface in the coronal third of the crown, and was the highest in fibre and FGDPS models, resulting in restorable failures. 10% BG was the most suitable ratio to be added to Ti- HA composites compared to other ratios. Sintering Ti-HA-BG composites using air atmosphere furnace resulted in better chemical, physical and mechanical properties compared to those sintered in vacuumed furnace. It was found that teeth restored with FGDPs and titanium posts showed a higher fracture resistance than those restored with stainless steel posts. Failure modes tended to be more restorable with teeth restored with FGDPs than those restored with other types of dental post. Stress distribution was uniformly distributed in FGDPs and fibre models as compared to titanium and stainless steel models.

Keyword: titanium; hydroxyapatite; bioactive glass; sintering process; composites.

OPTIMISATION OF TITANIUM-HYDROXYAPATITE COMPOSITES USING BIOACTIVE GLASS FOR FABRICATION OF FUNCTIONALLY GRADED DENTAL POSTS

ABSTRAK

Untuk mengoptimumkan ciri-ciri berbagai nisbah komposit Titanium-Hydroxiapatite dengan menggunakan pelbagai nisbah bahan kaca bioaktif dalam menentukan pembentukan tiang pergigian berfungsi gred. Kaca bioaktif (mengandungi; SiO_2, NaO, CaO, B_2O_3, P_2O_5, CaF_2, MgO, TiO_2) yang telah disediakan dan dicampur dalam kadar 3 nisbah berlainan (5%, 10% dan 15%) kepada 5 lapisan komposit Ti-HA yang berlainan, Campuran disediakan, digiling, ditekan, disinter dan diperiksa secara visual. Nisbah optima kaca bioaktif kemudian ditambah kepada komposit Ti-HA dan dibahagikan kepada 2 kumpulan dimana setiap satu mengandungi 5 lapisan. Sampel pada kumpulan 1 telah disinter dengan ketuhar udara admosfera sementara kumpulan 2 disinter dengan ketuhar vakum dan kedua-dua kumpulan diuji dengan XRD, EDX, ketumpatan, kekerasan mikro anjalan modulus dan mampatan. Kemudian tiang pergigian berfungsi gred (FGDPs) telah direka bentuk. Tiang (FGDPs) dan jenis tiang lain telah digunakan untuk rawatan endodontik. Setiap kumpulan mempunyai 10 batang gigi. Rintangan patah dan mod kegagalan telah dinilai dengan mengunakan mesin ujian Universal dan stereomikroskop dimana data dianalisis mengunakan One-way ANOVA dan ujian Chi-square. Pengagihan tekanan juga dinilai dengan mengunakan model gigi yang telah direstorasikan dengan tiang pergigian berfungsi gred, titanium, fiber, keluli nir karat mengunakan analisis finite element. Komposit Ti-HA yang dicampurkan dengan 10% nisbah kaca bioakftif didapati stabil dan tiada rekahan berbanding dengan kumpulan lain. Pada bahagian yang kedua, sampel yang telah disinter dalam kumpulan 1 (ketuhar udara admosfera) menunjukkan perbezaan yang signifikan (p=0.000) berbanding kumpulan 2 (ketuhar vakum) dari segi mampatan, padat ujian mikro kekerasan dan keanjalan modulus. Hasil dari ujian XRD, kumpulan 1 menunjukkan fasa major didapati

oleh Ti dan HA dan jumlah minima titanium telah dioksida kepada Ti_2O. Walau pun dalam kumpulan 2 fasa pertama didapati daripada Ti dan HA. HA juga telah dikomposkan kepada TCP dan TTCP. Ujian EDX juga menunjukkan tiada kontaminasi direkodkan pada kedua -dua kumpulan. Pada ringtangan fraktur FGDPs dan kumpulan titanium menunjukan rintangan fraktur yang lebih tinggi berbanding dengan kumpulan besi nirkarat (p=0.000 dan p=0.032 masing-masing). Dalam mod kegagalan, kumpulan FGDPs telah menunjukkan kegagalan yang masih boleh direstorasikan lebih tinggi dari kumpulan lain dan diikuti dengan tiang fiber. Bagi taburan tekanan walau dari mana arah beban, tekanan maksima padat pada teras-korona di sepertiga korona dan yang tertinggi dalam model fiber dan FGDPs juga menunjukan kegagalan yang masih boleh direstorasikan. 10% kaca bioaktif (BG) adalah nisbah yang paling sesuai untuk dicampurkan pada komposit Ti-HA dari nisbah yang lain. Komposit Ti-HA-BG yang disinter menggunakan ketuhar admosfera berbanding dengan yang disinter dalam vakum. Gigi yang direstorasikan dengan tiang FGDPs dan tiang titanium menunjukkan rintangan patah yang lebih tinggi berbanding dari yang direstorasikan dengan tiang keluli nirkarat. Mod kegagalan yang masih boleh direstorasikan adalah dengan FGDPs daripada gigi yang direstorasikan dengan tiang jenis lain. Taburan tekanan adalah lebih seragam pada model FGDPs dan fiber berbanding dengan titanium dan keluli nirkarat.

Kata kunci: Titanium, Hydroxyapatite, kaca bioaktif, proses pensinteran, komposit.

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LIST OF SYMBOLS AND ABBREVIATIONS

BG	Bioactive glass.
EDX	Energy dispersive spectroscopy.
FGDPS	Functionally graded dental posts.
FEA	Finite element analysis.
HA	Hydroxyapatite.
ISO	International Organization of Standardisation.
TCP	Tricalcium phosphate.
TTCP	Tetracalcium phosphate.
Ti	Titanium.
TiO	Titanium oxide.
VHN	Vickers hardness number.
XRD	X-ray diffraction.

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CHAPTER 1: INTRODUCTION

1.1 Background of the study

Root canal (endodontic) treatment is an important discipline that is widely studied in dentistry. Teeth may undergo loss of structure and integrity due to various factors. Some of these factors include dental caries, trauma, and even root canal procedures. All these factors may weaken a tooth and subjecting it to fracture during loading (Habekost *et al.,* 2007; Krishan *et al.,* 2014).

One of the aims of root canal treatment is to protect and maintain the remaining structures of treated tooth, followed by an appropriate coronal restoration to avoid tooth loss (Bachicha *et al.,* 1998). The teeth position, whether they are anteriorly or posteriorly located, and the amount of tooth structure lost will both determine the type of restoration to be placed on the endodontically treated tooth (Morgano *et al.,* 1994; Baratieri *et al.,* 2000; Chen *et al.,* 2007; Ng *et al.,* 2010).

A dental post should only be placed to restore endodontically treated teeth when there is excessive destruction of tooth structures to provide retention for the coronal restorations. The dental post can either be casted or prefabricated from several materials, such as fibre, titanium, stainless steel and chromium cobalt (Schwartz & Robbins, 2004; Hussain *et al.,* 2007; Garg & Garg, 2010; Aurélio *et al.,* 2016; Mahmoudi *et al.,* 2017).

Several post types, such as stainless steel, titanium and zirconium posts have showed high strength characteristics, fracture resistance and toughness. However, they have some disadvantages, such as being difficult to retrieve, producing non-restorable failures and loss of retention (debonding from the root and/or the core) (Hu *et al.,* 2003; Schwartz & Robbins, 2004; Aurélio *et al.,* 2016). Hence, fibre posts with a modulus of elasticity close to that of dentine were introduced. Teeth restored with fibre posts exhibit minimal non-restorable failures but are still susceptible to fractures when forces are applied over those teeth (Cormier *et al.,* 2001; Akkayan & Gülmez, 2002; Goracci & Ferrari, 2011; Uthappa *et al.,* 2015; Habibzadeh *et al.,* 2017).

All commercial posts are made from one type of material, which makes them difficult to distribute stresses when forces are applied and increases the possibility of non-restorable failures. Due to the challenges faced with the previously mentioned posts, in addition to post types, functionally graded post may be a suitable alternative (Madfa, 2011a, 2011b).

In 2011, three functionally graded dental posts were fabricated based on Ti-HA-ZrO_2-Al_2O_3 materials by Abu Kasim *et al.* (2011). This dental post matches the high stiffness at the coronal region and its stiffness is gradually reduced apically. The high stiffness in the coronal region eradicates the stress from the core and the gradual reduction of stiffness would uniformly dissipate the stress from the post to the dentine. Abu Kasim *et al.* (2011) also investigated the stress distribution of a prototype functionally graded dental post (FGSP) and compared them to posts fabricated from a homogeneous material. They demonstrated that stress distribution at the post-dentine interface of FGSPs was better than that of homogenous posts.

It is well known that hydroxyapatite has a similar chemical and crystallographic structure to the bone mineral, and is biocompatible with hard human tissues (Healy & Ducheyne, 1992; Rivera, 2011; Al-Sanabani *et al.,* 2013). However, it has poor mechanical properties (Jansen, 1991). On the other hand, mechanical properties of titanium are good, but it has poor biocompatibility compared to hydroxyapatite (Kleebe *et al.,* 1997; Brunette *et al.,* 2012; Attar *et al.,* 2014).

During sintering of Ti/HA at high temperature, dehydration and decomposition of HA is prevalent (Ning & Zhou, 2004), which can reduce its bioactivity and mechanical properties. To decrease the sintering temperature and maintain excellent properties of initial components, low melting point additives could be used as a sintering aid to realise sintering densification. That material should be non-toxic, have good biocompatibility and provide sufficient mechanical strength to the sintered material.

Previous studies have indicated that the addition of bioactive glass to hydroxyapatite not only increased the mechanical properties of hydroxyapatite but also improved its biological properties (Santos *et al.,* 1995; Ning & Zhou, 2004; Prasad *et al.,* 2017).

In a previous study by Moskalenko and Smirnov (1998) showed that a 50%Ti/40%HA/10%BG composite sintered at 1200 ^{o}C had a good *in vitro* bioactivity. That means a bioactive glass could be a good candidate for sintering hydroxyapatite and titanium and overcome the problem of sintering temperature. However, that study was performed on Ti-HA-BG composites, not on functionally graded materials, in which the used composites remained separated and not fused to form functionally graded materials with many layers merged together.

Until today, there have been no studies on the fabrication of Ti-HA functionally graded dental posts with incorporation of bioactive glass materials.

In this study, different ratios of Ti-HA composites incorporation with various ratios of bioactive glass were produced. Their physical, chemical and mechanical properties were studied to assess their ability to be used together to fabricate functionally graded materials. Subsequently, Ti-HA-BG functionally graded dental posts were fabricated, mechanically tested and compared with different types of commercial used dental posts.

Moreover, finite element analysis was performed to investigate stress distributions and was compared to different types of dental posts.

1.2 Aim of the study

This study aims to evaluate the possibility of improving the chemical, physical and mechanical properties of novel functionally graded Ti-HA dental posts (FGDPs) by incorporating bioactive glass (BG) compounds with low melting temperatures.

1.3 Objectives of the study

1- To optimise different titanium-hydroxyapatite composites by using various ratios of bioactive glass.

2- To characterize and optimise titanium-hydroxyapatite-bioactive glass composites to fabricate functionally graded dental posts.

3- To investigate the fracture resistance and failure mode of endodontically treated teeth restored with functionally graded dental posts and compare them with stainless steel, titanium, and fibre posts.

4- To perform finite element analysis and compare the stress distribution within teeth that were restored by functionally graded dental posts with those restored by other post types.

1.4 Organization of the thesis

Chapter 2 reviews the importance of endodontically treated teeth and the restoration procedures that should be used. This chapter also reviews the indications, types, classifications and considerations of dental posts. Finite element analysis and materials that are widely used in composites and functionally graded techniques will be reviewed in this chapter too.

Chapter 3 focuses on materials and methods in which different ratios of bioactive glass in addition to five different ratios of titanium-hydroxyapatite mixtures will be prepared and optimised. Sintering of Ti-HA-BG composite in air atmosphere and vacuum furnace is also described in this chapter. Fabrication and machining of functionally graded dental posts and evaluation of fracture resistance and failure mode of teeth restored with FGDPs and commercial posts are explained. Finite element analysis of four models is also explained in this chapter.

Chapter 4 shows the results of each experiment. The chemical (XRD and EDX), physical (density) and mechanical (micro-hardness), modulus of elasticity and compression tests that are performed on various ratios of Ti-HA-BG composites were analysed. Fracture resistance and failure mode of teeth restored with FGDPs and commercial posts were also evaluated. Finite element analysis of four models that mimic teeth restored with four different dental posts were analysed by using Solid Works software.

Chapter 5 discusses the importance of incorporating BG with Ti- HA composite in order to minimise the HA decomposition and Ti oxidations. Moreover, the reasons for sintering the Ti-HA-BG composites and functionally graded Ti-HA-BG multilayered composites in air atmosphere and vacuum furnaces are discussed. This chapter also discusses the mechanism of functionally graded dental posts on fracture resistance, failure mode and stress distribution of endodontically treated teeth.

Chapter 6 includes the benefits and conclusions of this study. The study limitations and recommendations for further studies are also listed in this chapter.

CHAPTER 2: LITERATURE REVIEW

2.1 Tooth structure and properties

2.1.1 Introduction

The tooth consists of four layers, which are enamel, dentine, pulp and cementum. Dentine and pulp are the inner layers, whereas enamel and cementum are the outer layers (Figure 2.1). The study on structural, mechanical and physical properties of the tooth is a very important matter which helps in fabrication of suitable restorative materials that can properly replace damaged parts of the tooth structure. Hardness and elasticity of tooth structure are the most important amongst these properties (Braden, 1976; Waters, 1980; Heymann *et al.*, 2014; Mount *et al.,* 2016). Therefore, understanding the tooth structure and its properties is essential in order to propose proper restoration procedures for the tooth.

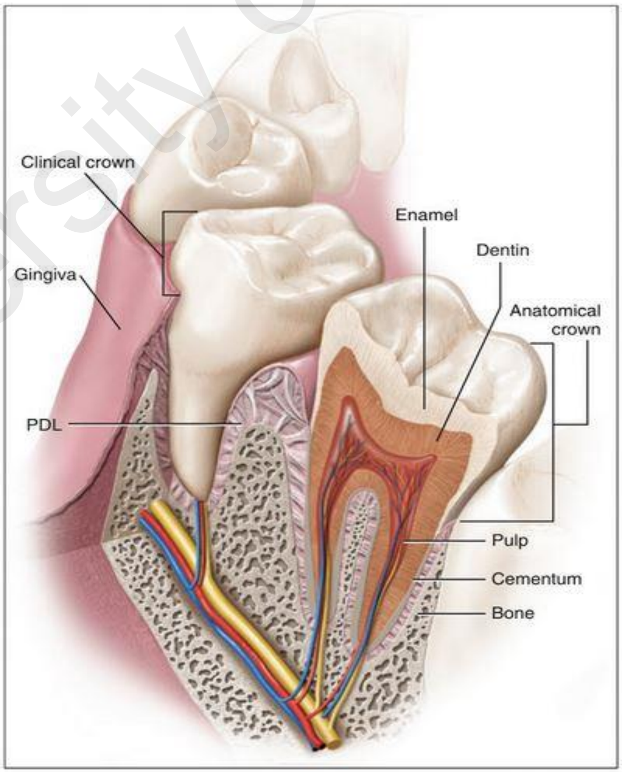


Figure 2.1: Diagram of tooth and its supporting structure (Adopted from Antonio Nanci (2017)

2.1.2 Enamel

Enamel is the outer layer of tooth that is considered the hardest and most highly mineralised tissue in the human body. It has the ability to withstand forces that are applied to the tooth during mastication. Enamel is more condensed in cusps and the incisal regions. Due to that its translucent white or whitish blue colour is more obvious in these regions. Whereas in the other regions of the crown, enamel tends to be more yellowish due to the underlying dentine (Heymann *et al.*, 2014; Mount *et al.*, 2016).

Enamel contains 95% inorganic materials, 4% water and 1% organic materials. It is mostly composed of hydroxyapatite and when damaged (mostly by wear or dental caries) it cannot be regenerated or replaced. Subsequently, it should be restored by a restoration procedure that could compensate and maintain its properties to properly withstand the mastication forces (Simmelink & Piesco, 1987; Nanci, 2017).

Mechanically, the outer surface of tooth has the hardest enamel, whereas the dento-enamel junction has the lowest. However, the inner enamel surface has higher stress distribution abilities compared to that in the outer surface (Meredith *et al.*, 1996; He & Swain, 2009).

2.1.3 Dentine

Dentine is an avascular layer that forms the bulk of tooth which surrounds the pulp in crown and root, and is produced by odontoblasts. It supports the enamel and helps to withstand the forces of mastication. It is yellowish white in colour and is 60% inorganic material, 30% organic material and 10% water. The inorganic materials are composed of apatite and the organic substances are fibrillar protein collagen (Kinney *et al.*, 1999; Heymann *et al.*, 2014; Mount *et al.*, 2016; Nanci, 2017).

Dentine is an elastic layer which is more flexible than enamel. It can also withstand more tensile strain than enamel. The hardness and elasticity of the dentine are different according to the location. Peritubular dentine has more elasticity and less hardness compared to that in the intertubular dentine. Superficial dentine is harder than the deeper dentine (Pashley *et al.,* 1985; Kinney *et al.*, 1996). Dentine is sensitive and has the ability to repair itself and protect the pulp when damaged by bacteria or other factors by stimulating odontoblasts to produce secondary dentine (Farges *et al.,* 2009; Farges *et al.*, 2013; Gaudin *et al.*, 2015).

2.1.4 Dental pulp

Dental pulp is a connective tissue which contains veins, arteries, nerves and lymphatic systems. Dental pulp is larger in children and young people than that in older people due to it decreasing in size with age. It is considered as the vital layer of the tooth, enclosed by dentine. In addition, it is related functionally with dentine in which it produces and nourishes dentine, containing nerves that make dentine sensitive to stimuli, such as thermal, chemical or electrical (Gaudin *et al.*, 2015; Mount *et al.*, 2016; Nanci, 2017). When dental pulp is inflamed or defected by trauma, bacteria due to dental carries or by a restoration preparation process, odontoblasts that are found in dentine can provide protection to the pulp from external risks by producing secondary dentine around the damaged pulp area (Farges *et al.*, 2013; Gaudin *et al.*, 2015).

2.1.5 Cementum

Cementum is a vascular bone like tissue which surrounds the root. It is produced by cementoblasts that are formed by mesenchymal cells of the dental follicle connective tissue. It attaches tooth roots to the collagen fibres of periodontal ligaments at which these fibres connect the tooth roots to the neighbouring alveolar bone tissues (Mount *et al.*, 2016).

Cementum is a harder tissue than the bone so it can withstand resorption which may occur due to friction during orthodontic and physiological teeth movements (Torabinejad *et al.,* 2014; Mount *et al.,* 2016). It is light yellow in colour and it consists of 45%-50% of inorganic materials that are mainly hydroxyapatite, 50%-55% organic materials (mostly composed of collagen and protein saccharides) and water (Heymann *et al.,* 2014; Nanci, 2017).

2.2 Root canal treatment

2.2.1 Differences between root canal treated teeth and vital teeth

Many studies have reported that the mechanical properties of dentine in vital teeth are different from that in root canal treated teeth. Dentine in root canal treated teeth is more brittle due to moisture loss as compared to that in vital teeth (Carter *et al.,* 1983; Goldman *et al.,* 1984). Furthermore, the condition of the alveolar bone that is anchoring the teeth in addition to the number of intact tooth surfaces also affects the tooth strength (Carter *et al.,* 1983; Goldman *et al.,* 1984). Root canal treated teeth are always subjected to structure loss and exhibit a reduction in modulus of elasticity, which affects the tooth strength and makes it more susceptible to fracture under loading. In contrast, vital teeth are usually more resistant to tooth fracture (Balooch *et al.,* 1998; Tang *et al.,* 2010).

In contrast, a study carried out by Huang *et al.* (1992) on the mechanical and physical properties of dentine in vital teeth and root canal treatment teeth at different hydration levels, reported that dentine properties were not affected by root canal treatment or dehydration. In addition, a study by Papa *et al.* (1994) reported that the difference between endodontically treated teeth and vital teeth in terms of loss of moisture is not significant and what actually affects the strength of the teeth is the amount of remaining tooth structures.

Another study also reported that edodontically treated teeth are weaker and more susceptible to fracture than vital teeth due to loss of tooth structures during endodontic and restorative processes (Tang *et al.,* 2010).

2.2.2 The effect of access preparation on root canal treated teeth

Dental caries, access preparation, and root canal treatment are factors that could affect teeth structural integrity; hence, it will reduce the mechanical properties and increase the susceptibility of the tooth to fracture (Kishen, 2006; Tang *et al.,* 2010). Pantvisai and Messer (1995) showed that the deflection of tooth cusp during loading was increased by access preparation that will increase the possibility of cusp fracture and/or micro-leakage. Endodontic access, root canal irrigation, instrumentation and obturation processes could subject the tooth to a fracture risk and reduce its ability to resist fracture (Whitworth *et al.,* 2002; Krishan *et al.,* 2014).

2.2.3 Restoration of root canal treated teeth and post indications

Tooth location is one of the main factors that determine which final restoration should be used after the root canal treatment. Anteriorly, the post must be used when teeth are badly damaged and has lost most of its surfaces to retain the core that will support the restoration (Cheung, 2005; Aurélio *et al.,* 2016). A post does not strengthen the tooth and increase its fracture resistance. In fact, a post subjects the tooth to more structure loss during post space preparation, which makes it weaker than before (Sorensen & Martinoff, 1985; Hou *et al.,* 2013; Aurélio *et al.,* 2016).

Wahab (2004) stated that a post has to be placed when a crown is to be used to restore a compromised tooth structure, or when the tooth is going to be an abutment to provide retention for a bridge restoration. Anterior teeth that have more than two intact surfaces are more preferable to avoid using a dental post. Instead, the use of bonded restorations will be more suitable so as to prevent extra stress being applied to the tooth (Butz *et al.*, 2001; Schwartz & Robbins, 2004; Cheung, 2005; Aurélio *et al.*, 2016).

In addition, in cases where a more aesthetic restoration is needed, veneers can be used. Teeth veneered with direct composites displayed no improvement in their resistance to root fracture when restored with a dental post as reported by Baratieri *et al.* (2000). Great precaution should be taken during insertion of dental post in lower incisors, as these teeth contain a very thin root surface, and more pressure would subject them to fracture (Cheung, 2005).

In premolars, dental posts are always recommended to be used, since premolars have less tooth surface area compared to molars. Caution is also needed to avoid perforations during instrumentation because premolars' anatomical features are not simple. It has a taper root, lingually inclined crown, and some have invagination (a developmental malformation of tooth) in proximal roots (Cheung, 2005; Baba & Goodacre, 2011).

In molars, since the concept of the post is providing retention to the core in teeth with fewer crown structures, it is advised to be inserted in the palatal canal in upper teeth and in the distal canal in lower molars because they are larger and straighter compared to other molar canals (Wahab, 2004; Baba & Goodacre, 2011; Rubina *et al.*, 2013).

2.3 Dental post

2.3.1 Post systems

The dental post is defined as a prosthesis which is inserted in the root canal, following root canal procedures to provide retention to the core which will maintain the crown (Garg & Garg, 2010). Many materials are available and involved in the fabrication of a dental post which show noticeable features and advantages from one to another (Anusavice *et al.*, 2012; Hou *et al.*, 2013).

Dental post materials can be divided into materials with high strength, such as stainless steel, titanium and ceramic and low strength material such as glass fiber (Al-Wahadni *et al.,* 2008; Anusavice *et al.,* 2012). Meanwhile, other materials are considered as materials with modulus of elasticity close to that in dentine. These materials are well known and widely used because they exhibit flexibility when forces are applied over them. Furthermore, it was reported that these materials are suitable to be used when aesthetic is required, especially at anterior teeth (Bateman *et al.,* 2003; Hou *et al.,* 2013). They are made from fibre which are introduced to the dental post fabrication in various types, such as glass, quartz and carbon fibre (Bateman *et al.,* 2003; Özkurt *et al.,* 2010; Anusavice *et al.,* 2012; Hou *et al.,* 2013). The combination of restoring a root canal treated tooth with a dental post, core and crown are shown in Figure 2.2.

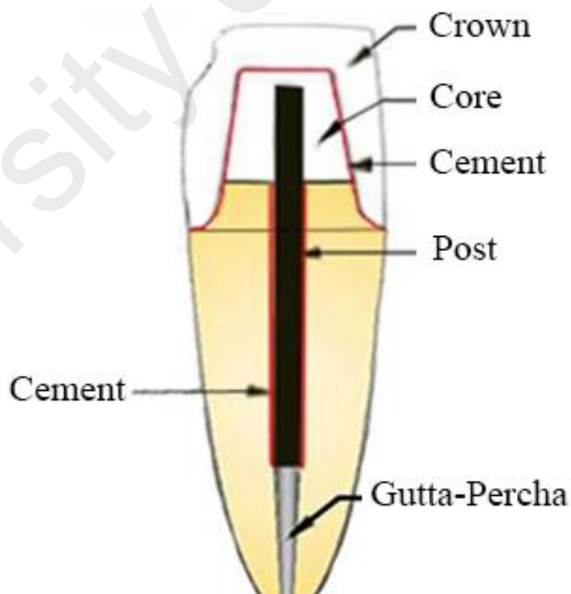


Figure 2.2: Diagram of tooth restored with a post, core and crown

2.3.2 Classifications of the dental post

These classifications depend on multiple characteristics, such as fabrication methods, types of material, surface design, and retention. According to the fabrication methods of dental post can be classified as cast and prefabricated (readymade); while according to design, it can be classified as parallel-sided or tapered. In terms of retention, it can be classified as active (threaded) and passive (serrated and smooth) posts (Schwartz & Robbins, 2004). There are many dental posts introduced with different materials, designs and brands, as listed in Table 2.1 (Schwartz & Robbins, 2004; Cheung, 2005; Terry & Swift, 2010).

Table 2.1: Common dental posts that are widely used in dentistry

Post name	Materials	Manufacturer	Passive VS active	Parallel VS tapered
Peerless post	Glass fiber	Sybron Endo	Passive	Tapered
Parapost Taper Lux	Glass fiber	Coltene/Whaledent	Passive	Tapered
Ancorex	Titanium	E. C. Moore	Active	Tapered
Surtex	Titanium, stainless steel, brass.	Dentatus USA	Active	Tapered
Cosmopost	Zirconium	Ivoclar Vivadent	Passive	Parallel
Parapost fiber lux	Glass fiber	Coltene/Whaledent	Passive	Parallel
FibreKleer	Glass fibre	Pentron	Passive	parallel
Parapost fiber white	Glass fiber	Coltene/Whaledent	Passive	Parallel
Parapost XH	Titanium	Coltene/Whaledent	Passive	Parallel
Parapost XP	Titanium, stainless steel	Coltene/Whaledent	Passive	Parallel
Parapost XT	Titanium	Coltene/Whaledent	Active	Parallel
Golden screw post	Brass	E. C. Moore	Active	Parallel
Flexi-Flange	Titanium, stainless steel	Essential Dental Systems	Active	parallel
Radix-Anchor	Nickel-Titanium	Dentsply/Maillefer	Active or Passive.	Parallel
Parapost Fiber Lux	Glass fiber	Coltene/Whaledent	Passive	Parallel
Cytco-K Nickel Titanium Post	Titanium	Dentsply/Maillefer	Active or Passive.	Parallel Tapered

Snowlight	Zirconia AR glass fiber	Danville	Passive	Parallel-tapered
Snowpost	Zirconia RA glass fiber	Danville	Passive	Parallel-tapered
Carbopost	Carbon fiber	Danville	Passive	Parallel-tapered
AZtec Post System	Titanium	Dentatus USA	Active	Parallel-tapered

2.3.2.1 Cast post and core

For decades cast posts have been considered as one of the techniques to provide retention for the coronal restoration in root canal treated teeth. In long-term use, it showed high success rates and low failure rates, making it the post of choice for many dentists (Cheung, 2005; Theodosopoulou & Chochlidakis, 2009; Özkurt *et al.*, 2010).

There are various studies which evaluated the success and failure rates of teeth restored with cast posts for a long observation period. Some of them observed these teeth in a period of three to four years (Aquilino & Caplan, 2002; Theodosopoulou & Chochlidakis, 2009; Özkurt *et al.*, 2010), while others observed the teeth for a longer period (10 to 25 years) (Valderhaug *et al.*, 1997; Ferrari *et al.*, 2000). These studies have shown good survival rates and a low failure rate ranging between 0%, 10% and 11% failures (Valderhaug *et al.*, 1997; Ferrari *et al.*, 2000; Aquilino & Caplan, 2002; Theodosopoulou & Chochlidakis, 2009; Özkurt *et al.*, 2010).

Valderhaug *et al.* (1997) restored 106 teeth with 106 cast posts and retained with crowns and bridges. For a very long observation period of 25 years, failures started to occur after 10 years of restoration, and the rate of failures in 25 years was 20%. Ellner *et al.* (2003) reported 0% failure in teeth restored with cast posts over a 10 year period. One of the main materials used in the fabrication of cast posts was gold alloys (type 3 or 5). They can tolerate the forces during loading due to their high compressive strength, and were advised to be used in misaligned or badly destructed teeth (Cheung, 2005; Theodosopoulou & Chochlidakis, 2009).

A study carried out by Balkenhol *et al.* (2007), 802 cast posts and cores in 565 patients were evaluated with regards to the type, location of the restored teeth and type of cement materials used. In addition, cast and core fabrication methods, whether direct or indirect, post and core age, alloy and failure reasons were also considered too. Survival rates were analysed by Kaplan-Meyer analysis. The results indicated that 7.3 years was the estimated survival period of the cast post and core, and the failures rate was 11.2%. Most of these failures were related to retention demands of post and core. Post and core cast in gold alloy showed less failure rates than those casted with other materials. Moreover, type of final restoration also affects the survival rate of the post and core. Fabricating the post and core by indirect methods also improved their survival rates more than by using direct fabrication method.

One of the advantages of the cast post is that it is easy to be retrieved when retreatment was needed (Schwartz & Robbins, 2004). On the other hand, many disadvantages have been reported. For example, it is time consuming (as a post is usually fabricated by a dental laborator), esthetic demands (shows a dark line in the gingival margin area), more expensive than other post types and non-restorable failures are more likely to occur with cast posts. Moreover, during the post space preparation process, tooth structures to be removed were more than that in teeth with prefabricated posts (Mitsui *et al.*, 2004; Cheung, 2005; Terry & Swift, 2010).

2.3.2.2 Prefabricated post

The prefabricated post is a readymade post that comes with different diameters to fit in the root canal. It is an alternative for cast posts are not as time-consuming since the prefabricated posts can be easily placed in the root canal in one visit. Prefabricated posts are fabricated from many materials and are of various designs. There are many advantages and disadvantages of prefabricated posts where some of them have high compressive strength while others exhibit better flexibility and esthetic features (Bateman *et al.,* 2003; Schwartz & Robbins, 2004; Cheung, 2005; Terry & Swift, 2010).

In general, prefabricated posts have shown a good success rate. Torbjörner *et al.* (1995) restored 788 root canal treated teeth with dental posts in 638 patients, in which 456 cast posts, and 332 Para-Post posts were used. After four to five years of posts placement, failure rates were evaluated and the results showed that teeth restored with prefabricated para-post showed only 8% failure, whereas teeth restored with casted posts showed 15% failure.

(a) ***Metal posts***

Metals have been widely used in dentistry for many decades for dental post fabrication. They exhibit good mechanical properties such as toughness and stiffness. Most popular metal materials used to fabricate dental posts include stainless steel, titanium, chromium and cobalt. Stainless steel and titanium are usually used in prefabricated dental post (Schwartz & Robbins, 2004; Terry & Swift, 2010).

Stainless steel and titanium have advantages and disadvantages. Stainless steel is tough and radio-opaque in X-rays. However, its disadvantage is that it does not resist corrosion. Hence, titanium was introduced as it does resist corrosion. However, it has low fracture resistance as it is not as tough as stainless steel. Titanium posts are also difficult to retrieve, and it cannot be distinguished from gutta-percha and sealer with x-rays (Schwartz & Robbins, 2004).

Mechanical properties of metal posts are superior to fibre posts and many studies have investigated their ability to resist fractures in root canal treated teeth. They discovered that stainless steel and titanium posts had higher fracture resistance than fibre posts (Amarnath *et al.*, 2015; Abdulmunem *et al.*, 2016).

It was also reported that metal posts have higher fracture resistance and better survival rate than zirconia posts (Heydecke *et al.,* 2001). A study which included 60 endodontically treated lower premolars that were restored with stainless steel or fibre posts after being covered with core materials, found that the stainless steel posts had higher fracture resistance than fibre posts (Amarnath *et al.,* 2015).

(b) ***Ceramic and zirconia posts***

Due to esthetic problems that result from using metal dental posts in root canal treated teeth, especially when used in anterior teeth with an all ceramic crown restoration, new materials with similar tooth colour have been proposed. Zirconia materials can be a good alternative to the metal post as it has excellent esthetic properties (Özkurt *et al.,* 2010; Madfa *et al.,* 2014). Their first use in dental posts was reported by Meyenberg *et al.* (1995). They showed that zirconia has good flexural strength that ranges from 900 MPa-1200 MPa.

Additionally, zirconia has good physical, chemical and mechanical properties (zirconia exhibits high strength and toughness features). Its modulus of elasticity is close to that of stainless steel alloy. Nowadays, zirconia is widely used because of these properties (Piconi & Maccauro, 1999; Vichi *et al.*, 2000; Pilathadka *et al.,* 2007; Özkurt *et al.,* 2010; Raut *et al.,* 2011).

Unfortunately, zirconia posts also display many disadvantages, such requiring extra removal of the root canal structures as they cannot be used with a small diameter due to their brittleness and thus will weaken the tooth. They are also weaker than metal posts (Asmussen *et al.,* 1999; Schwartz & Robbins, 2004; Madfa *et al.,* 2014). Zirconia has a very high modulus of elasticity of 150 GPa, dissipates high stress to the attached dentinal tissue when the tooth is under loading, and thus increases the possibilities of root fractures (Bateman *et al.,* 2003; Hu *et al.,* 2003; Novais *et al.,* 2009).

Zirconia also alters core retention when composite materials are used since it cannot be etched to receive core materials. This problem occurred due to the need to increase the mechanical properties of the zirconium by minimising the glass materials and increasing the crystalline content; hence, the produced zirconia becomes more resistant to acid etching. It is worth mentioning that zirconia is also not easily retrieved (Butz *et al.,* 2001; Hedlund *et al.,* 2003; Schwartz & Robbins, 2004; Raut *et al.,* 2011).

In another study carried out by Abduljabbar *et al.* (2012), the lower first premolars were restored with zirconia, cast post and core, and fibre posts before being restored with ceramic crowns. All teeth were subjected to load under a Universal Testing Machine. The results indicated that in terms of strength, zirconia posts displayed higher fracture strength than fibre posts and cast post and core. However, in terms of failure, 90% of the failures were described as non-restorable.

(c) ***Fibre posts***

Since the previously mentioned dental posts were made of materials with a high modulus of elasticity and high strength, with non-restorable fractures, a material with flexibility and modulus of elasticity close to that in dentine (such as fibre materials) is highly sought after to be incorporated in dental post fabrication. The modulus of elasticity of fibre post is 20 GPa, which is close to that of dentine (18 GPa), whereas the modulus of elasticity of metal cast post and core is 200 GPa and in zirconia posts, the value is 150 GPa (Novais *et al.,* 2009). There are many types of fibre posts, including carbon, quartz, silica and fibre posts which exhibit a radiolucent appearance (Plotino *et al.,* 2007; Kim *et al.,* 2009; Goracci & Ferrari, 2011).

Fibre posts were first used clinically in France and were made of carbon fibre materials, and there are publications related to the study on the evaluation and properties of fibre posts in 1990 (Bateman *et al.,* 2003; Goracci & Ferrari, 2011). The fibre posts have good stiffness, electrical conductivity, low toxicity and good tensile strength. These properties make fibre posts perfectly usable in dentistry (Soares *et al.,* 2008). Because of their modulus of elasticity, reparable fractures can occur more in teeth restored with fibre posts than those restored with other types of commercial dental posts (GU & Kern, 2006). Unlike zirconia and metal posts, fibre posts can be easily retrieved when root canal retreatment is required (Pitel & Hicks, 2003; Bitter *et al.,* 2006).

Many studies have under taken to investigate fibre posts behaviour when used to restore root canal treated tooth. These studies indicated that fibre posts have performed very well in laboratory and clinical studies (Ferrari *et al.,* 2000; Novais *et al.,* 2009; Goracci & Ferrari, 2011; Abdulmunem *et al.,* 2016).

A clinical study conducted by Uthappa *et al.* (2015) where patients with 40 root canal treated teeth were selected and underwent posts placement procedures. Twenty teeth were restored with a metal post while the other 20 teeth were restored with fibre posts. Results showed that failures were less likely to occur with teeth restored with fibre posts.

The ability of fibre posts to resist root fractures in root canal treated upper central incisors with cervical cavities was investigated by Abduljawad *et al.* (2016). Results showed that fibre posts improved the fracture resistance of the restored teeth. Restorable failures occur more in root canal treated teeth which have been restored with fibre posts cemented with resin cement compared to those cemented with zinc phosphate cement. Moreover, restorable failures occurred more commonly in teeth restored with fibre posts compared to teeth restored with titanium or stainless steel (Abdulmunem *et al.,* 2016).

Teeth restored with fibre posts were also compared with cast Ni-Cr posts and zirconia post and cores. Those restored with fibre posts showed better resistance to fracture than those restored with zirconia posts (Habibzadeh *et al.,* 2017).

(d) ***Functionally Graded Dental Post***

All commercial posts are structured from a homogenous material which makes it difficult for them to distribute forces during loading, and more susceptible to produce un-repairable failures. To overcome this problem, a functionally graded dental post was produced. Abu Kasim *et al.* (2011) fabricated three functionally graded dental posts (FGPs) based on Ti-HA-ZrO_2-Al_2O_3 materials. These dental posts provided high stiffness at the coronal region that gradually decreased apically.

The benefits of this gradual change in functionally graded dental posts (FGDPs) are to have a different and graded stiffness from on part of FGDPs to another in order to dissipate stress regularly (evenly) to the dentine (Abu Kasim *et al.,* 2011). Abu Kasim *et al.* (2011) also investigated the stress distribution of prototype functionally graded dental posts (FGSPs) and compared them to posts fabricated from a homogeneous material. They demonstrated that the stress distribution at the post-dentine interface of FGSPs was better than that of homogenous posts.

2.3.3 Post considerations

2.3.3.1 Post retention and resistance

Post retention is an important feature that may affect their life-time in root canal treated teeth and which may eventually threaten the success of endodontic treatment. Dental post retention can be influenced or improved by the diameter, length and dental post design, and type of luting cement. Furthermore, cementation methods, post space preparation and root location also affect the posts retention (Adanir & Belli, 2008; McLaren *et al.*, 2009; Rasimick *et al.*, 2010).

Teeth restored with threaded posts (active posts) have shown improved retention compared to passive posts. Also, long thick posts provide more retention than short or thin posts. In addition, post design also plays a role in post retention in which parallel-sided posts exhibit better retention than taper posts (Grieznis *et al.*, 2006; McLaren *et al.*, 2009; Rasimick *et al.*, 2010).

Another important factor that affects the long-term success of root canal treated teeth restored with posts is the post resistance, which is the ability of the root and post to resist forces during mastication or under loading (Schwartz & Robbins, 2004). There are many factors that can influence the post resistance such as the amount of remaining tooth structure, anti-rotation features, post hardness, post length, post diameter and the existence of the ferrule (Grieznis *et al.*, 2006; McLaren *et al.*, 2009; Zicari *et al.*, 2012; Amarnath *et al.*, 2015).

2.3.3.2 Post length

Adanir and Belli (2008) showed that using a long post can achieve good retention of a dental post when used in root canal treated teeth. In agreement with that, a study by Sorensen and Martinoff (1984) investigated teeth that had undergone post placement with various post lengths. The authors reported that teeth restored with a post that have a longer or the same post length as the crown exhibited high fracture strength while those restored with shorter posts showed a lower fracture resistance.

A study by Amarnath *et al.* (2015), 60 teeth were collected and restored with stainless steel and fibre posts (with different post lengths; 4 mm, 5 mm and 10 mm) and were then subjected to a fracture resistance test. In both fibre and stainless steel posts groups, roots restored with a long post (10 mm) showed higher fracture resistance, followed by those restored with 5 mm post length. Meanwhile groups restored with the shortest post (4 mm) had the lowest fracture resistance value.

On another hand, another study conducted on roots restored with fibre posts with 1/3, ½ and 2/3 length of the post to the root length. Authors found that post lengths did not affect the dental posts resistance but it affected the types of failure, short dental posts resulting in with a non-restorable failure (Abdulrazzak *et al.,* 2014).

Caution must be taken during a long post space preparation in short roots as perforation may happen, therefore, providing a post with the same length as the crown is much preferred to a shorter post (Adanir & Belli, 2008; Amarnath *et al.,* 2015).

2.3.3.3 Post diameter

Post diameter that is one-third of the root diameter is preferred and mentioned by many studies. In addition, 1 mm of dentine ferrule should be provided and surrounded the dental post to prevent future root fractures. Posts that have 1.3 mm and 1.2 mm diameters have also been reported to be favour than those with a smaller diameter (Lloyd & Palik, 1993; Peroz *et al.,* 2005; Terry & Swift, 2010).

Since the removal of more root structure will affect the fracture resistance of the tooth and increase the possibility of non-restorable failures, it was proposed that it is better to restore the roots with a small post diameter to preserve the remaining root structures so as to decrease the failure (Grieznis *et al.,* 2006).

2.3.3.4 Post designs

The restoration survival rate is also affected by dental post design and shape, as they affect the retention of the post-root canal interface. Post shape can be paralleled, tapered, or with parallel-tapered end. Many studies have investigated the effectiveness of post design and the property of each shape in providing the best retention to the post in the root canal (Qualtrough *et al.,* 2003; Cheung, 2005; Peutzfeldt *et al.,* 2008; Upadhyaya *et al.,* 2016).

Each design has its own indications, in which a parallel-sided post is preferred to be placed in long roots with a good amount of intact structures to minimise the possibility of root fracture during space preparation. Whereas tapered posts need less root canal structure removal since it mimics the canal-shape and can be used in teeth with minimal structures (Caputo & Standlee, 1987; Schwartz & Robbins, 2004; Soares *et al.,* 2012).

Many studies have shown that parallel-sided posts provided superior retention combined with better success rate and more restorable failures compared to tapered posts (Caputo & Standlee, 1987; Torbjörner *et al.*, 1995; Qualtrough *et al.,* 2003; Peutzfeldt *et al.,* 2008). Moreover, parallel sided post has better shear, tensile and fracture resistance, distribute stresses better and demonstrate less stress during placement as compared to tapered post (Ross *et al.,* 1991; McAndrew & Jacobsen, 2002; Upadhyaya *et al.*, 2016).

However, a parallel-sided posts display some disadvantages as they may weaken the root apically due to the need to remove extra tooth structure. On the contrary, tapered posts initiate wedge effect during placement and subsequently increase the risk of root fracture (Caputo & Standlee, 1987; Asmussen *et al.,* 2005). The parallel tapered end post can solve these problems as it exhibits good retention coronally and mimics the shape of the apical third of the root canal; thus, avoiding the need to weaken the area by extra preparation and tooth removal (Fernandes *et al.,* 2003).

In terms of post surface configurations, posts can be classified into threaded (active), serrated or smooth (passive). Smooth and serrated posts produce less stress during insertion, which decreases the incidence of root fracture compared to threaded posts, which generate more stress (Cheung, 2005). Threaded posts are engaged in the root canal and thus show higher retention than smooth and serrated posts which depend on their cement materials to increase retention (Ross *et al.*, 1991; Cheung, 2005). All posts designs can be incorporated into a vent to reduce stress during post placement and cementation (Cheung, 2005).

2.3.3.5 Post space preparation

The post space preparation should not be more extensive apically to avoid perforation or influence the apical seal (Goodacre & Spolnik, 1995). In order to avoid root perforation during space preparation, the anatomy of the root and crown inclination to the root must be known before pursuing the preparation (Gutmann, 1992; Upadhyaya *et al.*, 2016).

There are a few ways to remove the gutta-percha and prepare the canal prior to post placement. The mechanical method is a well-known method which can be done by using gates-glidden drills and P-type reamers with a low-speed hand-piece to remove gutta-percha. Thermal methods can also be performed by using hot pluggers. In this method, 4 mm to 5 mm of gutta-percha used must remain intact to provide a suitable apical seal and avoid perforation. Subsequently, the post space can be prepared by using post drills that come with the manufactured post (Gordon, 1982; Goodacre & Spolnik, 1995; Zicari *et al.*, 2012, 2013).

2.3.3.6 Ferrule

Ferrule is an important factor that affects retention and resistance of the post which is located in the cervical third of the root (Mezzomo & Dalla, 2003; Pantaleón *et al.*, 2017; Kar *et al.*, 2017).

Several types of research has been performed to consider the ferrule effect on the posts resistance form and many have stated that a good ferrule length can provide maximum resistance (Abdulrazzak *et al.*, 2014; Kar *et al.*, 2017; Pantaleón *et al.*, 2017).

Preparing 1 mm of ferrule increases the ability of root canal treated teeth to resist the fracture, while the presence of 2 mm of ferrule gives much higher resistance to fractures (Yue & Xing, 2003; Aykent *et al.*, 2006). Ferrule shape is shown in Figure 2.3.

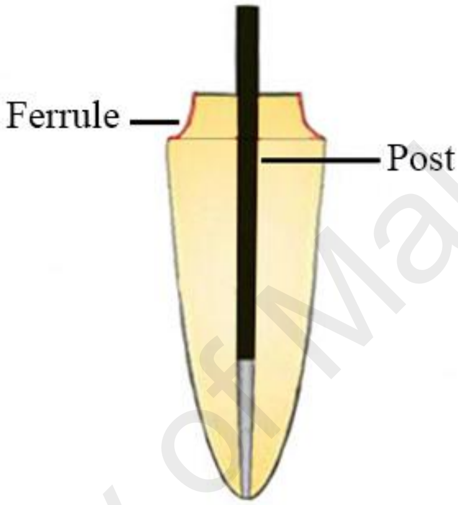


Figure 2.3: Diagram of tooth shows ferrule preparation

Increasing ferrule length can increase the fracture resistance of teeth restored with dental posts. A study by Abdulrazzak *et al.* (2014) concluded that roots with 4 mm ferrule exhibited higher fracture resistance than those without ferrule or with 2 mm ferrule.

Another study by Pantaleón *et al.* (2017) was conducted on 60 upper central incisors restored with a 2 mm complete ferrule, 2 mm, 3 mm, 4 mm and 6 mm incomplete ferrule (has one missing interproximal wall); all roots were restored with cast post and core. The study concluded that the 2 mm complete ferrule group showed a higher fracture resistance compared to the other groups. Roots prepared with 3 mm and 4 mm incomplete ferrule also showed good resistance to fracture.

Another group of researchers reported on a study involving 40 lower premolars restored with fibre posts and divided into four groups. Roots were prepared with 3 mm, 2 mm, 1 mm and 0 ferrule height and were then subjected to fracture resistance test (Kar *et al.,* 2017). It was discovered that roots prepared with 3 mm ferrule had higher fractures resistance, followed by roots with 2 mm, 1 mm and 0 mm (no ferrule) ferrules, respectively. Thus, a higher ferrule length can improve the fracture resistance.

In contrast, other studies had ignored the need to prepare the ferrule if 2 mm of coronal tooth structure was present (Osman, 2004). In another study, Al-Hazaimeh and Gutteridge (2001) reported that it was not necessary to prepare ferrule when a dental post was luted with resin cement in the root canal as this type of cement provides good retention and resistance for a dental post.

2.3.3.7 Luting cements

Most common types of cement that are used widely to cement dental posts in root canals are glass ionomer cement, zinc phosphate cement, resin-modified glass ionomer cement and composite resin cement (Pameijer, 2012).

Table 2.2 shows the different cement materials, brands, polymerization methods (Ladha & Verma, 2010; Pameijer, 2012; Stamatacos & Simon, 2013).

Table 2.2: Different dental cements that widely used in dentistry

Cement name	Cement type	polymerization method	Retention	Compressive strength/MPa	Modulus of elasticity /GPa
Elite 100® (GC)	Zinc phosphate cement.	Acid-based reaction cement.	-Provide low to moderate retention.	80–110	13
Durelon Maxicaps (3M™/ESPE)	Zinc Polycarboxylate cement.	Acid-based reaction cement	-Low retention.	55–90	4–5
Ketac™ Cem Easymix (3M™/ESPE)	Glass ionomer cement.	Acid-based reaction cement	-Moderate retention.	93–226	8–11
RelyX™ Unicem, (3M™/ESPE)	Self-adhesive resin cements.	Dual-cure cement.	-High retention.	52–224	1.2–10.7
G-CEM (GC)	Self-Adhesive resin Cement.	Dual-cure cement.	-High retention.	52–224	1.2–10.7
Maxcem Elite™ (Kerr Corporation)	Self-adhesive resin cements.	Dual-cured cement	-High retention.	52–224	1.2–10.7
RelyX™ Luting Plus Cement (3M™/ESPE)	Resin-modified glass Ionomer Cements	Self-cure cements.	-Moderate to high retention.	85–126	2.5–7.8
GC Fuji Plus™ (GC)	Resin-modified glass Ionomer Cements	Self-cure cements.	-Moderate to high retention.	85–126	2.5–7.8
Nexus RMGI (Kerr Corporation)	Resin-modified glass Ionomer Cements	Dual-cure resin cement.	-Moderate to high retention.	85–126	2.5–7.8
Panavia™ 21 (Kuraray)	Resin cement.	Self-cure resin cements.	-Moderate retention.	180–265	4.4–6.5
MultiLink® (Ivoclar Vivadent)	Resin cement.	Self-cure resin cements.	-Moderate retention.	180–265	4.4–6.5

C&B Metabond ® (Parkell)	Resin cement.	Self-cure resin cements.	-Moderate retention.	180–265	4.4–6.5
Choice™ 2 Light-Cured Veneer Cement (BISCO)	Resin cement.	Self-cure resin cements.	-Moderate retention.	180–265	4.4–6.5
RelyX™ ARC (3M™/ESPE)	Resin cement.	Dual-cure resin cement.	-Moderate retention.	180–265	4.4–6.5
Panavia™ F 2.0 (Kuraray)	Resin cement.	Dual-cure resin cement.	-Moderate retention.	180–265	4.4–6.5
Bistite II DC (J. Morita)	Resin cement.	Dual-cure resin cement.	-Moderate retention.	180–265	4.4–6.5
Duo-link™	Resin cement.	Dual-cure resin cement.	-Moderate retention.	180–265	4.4–6.5
Variolink ® II (Ivoclar Vivadent)	Resin cement. (Luting composite cement).	Dual-cure resin cement.	-Moderate retention.	180–265	4.4–6.5
Calibra® (Dentsply Caulk)	Resin cement.	Dual-cure resin cement.	-Moderate retention.	180–265	4.4–6.5
RelyX™ Veneer (3M™/ESPE)	Resin cement.	Light-cure resin cements	-Moderate retention.	180–265	4.4–6.5
Variolink ® Veneer (Ivoclar Vivadent)	Resin cement.	Light-cure resin cements	-Moderate retention.	180–265	4.4–6.5

Zinc phosphate cement is one of the most commonly used cements in dentistry. It is very popular due to its long-term success, simplicity in use and offers a higher retention compared to other cement types. Zinc mechanically adheres to dental posts and root surfaces (Radke *et al.*, 1988; Consani *et al.*, 2003; Mezzomo *et al.*, 2006; Lad *et al.*, 2014; Yu *et al.*, 2014).

Zinc phosphate cement is also reported to have good mechanical properties in which it exhibits good tensile, stiffness and compressive strength, making it capable of resisting fracture resistance more than other cement types (Garg & Garg, 2010; Anusavice *et al.,* 2012).

Three types of dental posts were divided into two groups, in which posts in Group 1 were cemented with zinc phosphate cement when posts were inserted in root canals, while posts in Group 2 were cemented with a dual-cure resin cement. Posts cemented with zinc phosphate cement exhibited a higher fracture resistance, whereas teeth cemented with resin cement exhibited higher restorable failure rates (Abdulmunem *et al.*, 2016)

Besides zinc phosphate cement, resin cement is also a good choice in dental posts cementation (Schwartz & Robbins, 2004). It provides good retention, short-term reinforcement to the teeth and exhibits less leakage than other cement types (Mezzomo & Dalla 2003; Reid *et al.,* 2003). Hence, resin cement has become the material of choice by many clinicians. Luting dental posts with dual or self-cure resin is more favourable than other resin cement types (Ferrari *et al.*, 2001; Peroz *et al.*, 2005). In the case when a cyclic load is applied, resin cement performs better in terms of fracture resistance than other cement types (Junge *et al.,* 1998).

When cementing a post with resin cement, extra preparation in root canal must be done to remove all remaining sealing and gutta-percha materials to provide good retention to the canal surfaces. That may lead to removing of unneeded internal root canal structures which are considered a disadvantage of resin cement (Schwartz & Robbins, 2004; Cheung, 2005).

2.3.3.8 Post cementation

Cementation methods of dental posts in the root canal are different and depend on cement materials that will be used in the process. It is recommended to apply zinc phosphate or glass ionomer cement to root canals by using lentulo spiral and paper point. Then cement can be applied over the post surfaces prior to insertion at root canals (Nathanson, 1993). Lentelo spiral is not preferred for applying resin cement because it increases the setting time of this cement. It is advisable to use an applicap with elongation tip to provide good cementation without voids (Schwartz & Robbins, 2004). Moreover, a disposable syringe with a needle can also be used to cement a cast post in root canal, where it shows good features and results (Gaikwad & Badgujar, 2015).

2.4 Fracture resistance and failure mode of dental posts

2.4.1 Failure mode

Failure mode is a major factor related to fracture resistance. Factors that may affect failure are the presence of ferrule, post types, post cement material, and type of core material (Schwartz & Robbins, 2004). Teeth prepared with ferrule have been shown to fail more favourably compared with those prepared without a ferrule (Al-Hazaimeh & Gutteridge, 2001; Abdulrazzak *et al.,* 2014).

Failure mode can be categorized as suggested by Fokkinga *et al.* (2003) and Silva *et al.* (2011), and as explained in Figure 2.4. Failure in tooth 1 is either complete or partial post-core-crown complex debonding (restorable failure), whereas failure in Tooth 2 is oblique fracture above bone level (the horizontal line in the figure represents the bone level) (restorable failure). Tooth 3 has an oblique fracture which extends below the bone level (non-restorable failure), whereas tooth 4 has a vertical fracture which extends below the bone level (non-restorable failure), and Tooth 5 has cracks below the bone level (non-restorable failure).

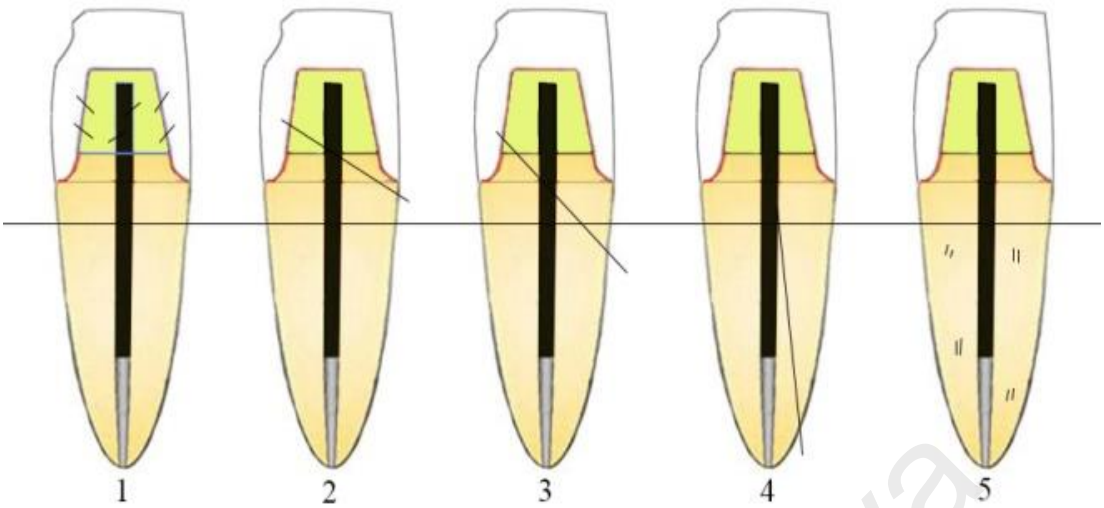


Figure 2.4: Types of failure that could occur in teeth restored with post. 1 and 2 are restorable failures. 3, 4 and 5 are non-restorable failures

2.4.2 Fracture resistance

Fracture resistance means the ability of both post and tooth to withstand lateral and rotational forces. Many studies have been done to evaluate the fracture resistance and failure mode of endodontically treated teeth with various post types (Table 2.3).

Table 2.3: Comparative studies of fracture resistance and failure mode of teeth restored with various types of post

Authors	Groups	Results	No. of teeth	Conclusion
Abdulmunem *et al.* (2016)	Two groups each has three subgroups restored with titanium, fiber and stainless steel posts. Posts in G 1 were luted with zinc phosphate whereas posts in G 2 luted with composite resin cements.	Posts cemented with zinc phosphate cement (G 1) showed higher fracture resistances. However, luting of dental posts with composite resin provided more restorable failures in endodontically-treated teeth.	60 maxillary central incisors.	Teeth restored with Fiber posts exhibited desirable fracture resistances with more restorable failure modes, compared with those restored by titanium or stainless steel posts.

Soundar *et al.* (2014)	Cast post and core (CPC), Zirconia post (ZP), milled zirconia post (MZ), pressable ceramic post (PCP), prefabricated zirconia post (CP). Post diameters were 1.4 mm and 1.7 mm in all groups.	In groups with 1.4 mm diameters; CPC group showed highest fracture resistance compared to other groups. Whereas, in groups with 1.7 mm diameters; CP group showed the heights fracture resistance.	48 maxiilary central incisors.	Specimens restored with cast posts and cores showed root fractures, whereas post fractures where noticed in specimens restored with ceramic posts.
Barcellos *et al.* (2013)	Control group (sound teeth), Fiber post (FP), fiber post cemented with resin composite (FPC), ,cast post and core (CPC),	FPC and control groups showed higher fracture resistance compared to other groups.	70 upper canines.	Unfavorable failures were noticed in CPC group, whereas, favorable failure were occurred in FP, and FPC groups.
Makade *et al.* (2011)	Cast post and core group, stainless steel post and composite core group, glass fiber post and composite core group, control group (without post placement).	Stainless steel group showed highest fracture resistance compared to other groups. On the other hand, glass fiber group showed the most favorable failures.	50 maxillary central incisors	Teeth restored with glass fiber post usually tend to be failed with favorable failures.
McLaren *et al.* (2009)	Stainless steel post, quartz fiber post, glass fiber posts, and control group (with no post placement).	Stainless steel post showed higher fracture resistance compared to quartz and glass fiber reinforced posts. On the other hand, Fiber post groups have failed with no root fractures unlike stainless steel group who failed with 25% of root fractures.	70 single rooted premolars.	Best support may offered by using stainless steel post with resin composite core than using fiber reinforced post with it.
Al-Wahadni *et al.* (2008)	Glass fiber posts, carbon fiber posts, and titanium posts groups.	Titanium group showed higher fracture resistance compared to other groups.	30 anterior teeth.	Fractures were mostly catastrophic in all groups.

Qing *et al.* (2007)	Nickel-chromium cast post and core (control group), glass fiber and zircon post (test group) with composite resin cores.	Cast post and core group showed higher fracture resistance compared to glass fiber and zircon group.	24 teeth.	All teeth exhibited oblique root fractures and cracks.
Sadeghi (2006)	Cast post and core, zirconium and quartz fiber posts groups.	Teeth restored with casted post and core exhibited a higher fracture resistance compared with other groups.	36 teeth	Quartz fiber posts group showed more favorable failures than other groups.
Goto *et al.* (2005)	Cast gold post and core (CPC), fiber reinforced resin post with composite core (FPC), titanium post with composite core (TPC).	FPC showed higher resistance to failure followed by TPC and CPC respectively	15 maxillary central incisors	FPC provided better retention and adhesion between crowns and endodontically treated teeth compared to CPC.
Y. H. Hu *et al.* (2003)	Serrated parallel-sided cast posts and cores (SPCPC), serrated parallel-sided prefabricated posts (SPP), carbon fiber reinforced posts (FP), and ceramic posts groups (CP).	There was no significance difference in fracture resistance between the groups.	40 maxillary incisors	CP group showed more unfavorable fractures compared with other groups.
Akkayan and Gülmez (2002)	Titanium posts, quartz fiber posts, glass fiber and zirconium posts groups.	Quartz fiber post group showed higher fracture resistance compared to other groups.	40 maxillary canines.	Quarts and glass fiber groups showed restorable failures, whereas zirconium and titanium groups showed non-restorable failures.
Heydecke *et al.* (2001)	Titanium post, zirconium post, roots filled with hybrid composite and controlled groups (only the access openings were filled).	The controlled group showed the highest fracture resistance followed by zirconium, titanium posts and roots filled with hybrid composite.	64 maxillary canines.	The controlled group showed less unfavorable failure mode compared to other groups.

2.5 Finite element analysis

2.5.1 Introduction

Finite element analysis (FEA) is a computerized method used to predict what will happen when the product is used. It can be used to solve different equations and can be applied to many combinations of fluids, solids and gases, such as static or dynamic, elastic or plastic conditions. The geometry model of the structure is build up and divided into elements. In these elements, equations will be formed according to the relation between the load and displacement. Then the equations will be gathered to form global FEA equations in which they will be solved by using a computer. The geometrical parameters of the structures, materials properties and load direction could be easily manipulated. It can be used to analyse elastic deformation, vibration, bucking behaviour, stress and deflection due to loading or any subjected displacements. In FEA software, the structure is subdivided into many blocks or elements (Manual, 1997; Geng *et al.*, 2008).

2.5.2 Application of finite element analysis in dentistry

The finite element method has been widely used in dentistry by many researchers in their studies. In a study by Thresher and Saito (1973) homogenous and non-homogenous tooth models were used to analyse stress distributions by using finite element analysis (FEA). The study observed that the enamel has tolerated most of the load and then transferred it to the surrounding structures. Moreover, high stress was noticed in the root.

A 3D finite element model was built by Darendeliler *et al.* (1992) to analyse the stress distribution of upper central incisor. The tooth model was composed of enamel and dentine only and meant to be elastic, isotropic and homogenous. They concluded that the stresses were highly condensed in the cervical line and incisal margin of the tooth model.

Joshi *et al.* (2001) used a 3D finite element to investigate the stress distribution of teeth restored with titanium, carbon fibre and stainless steel dental posts. They showed that a tooth model with a stainless steel post has a higher stress distribution compared to other post materials.

Al-Omiri *et al.* (2011) made 3D finite element teeth models in which one tooth was restored with a post and a crown whereas the other was restored with a crown only. Stress distribution was evaluated and the results showed that the tooth model with a post and a crown has higher stress compared to that restored with a crown only.

Abdulmunem *et al.* (2016) evaluated two groups of teeth in which each had three subgroups restored with fibre, stainless steel and titanium dental posts. Different types of post cemented with zinc phosphate cement were labelled as Group 1 while those cemented with composite resin cement were labelled as Group 2. A 3D finite element method was performed to analyse and compare the stress distribution that would have occurred due to different types of dental post.

Results showed that the stress distributions were significantly affected by the type of dental posts. Teeth restored with fibre posts exhibited high-stress absorption at the core area whereas teeth restored with titanium and stainless steel posts exhibited stresses that were more concentrated at the apical third of the posts. A tensile stress concentration was also noticed in the dentine-core interface of teeth restored with stainless steel posts.

2.6 Hydroxyapatite

2.6.1 Introduction

Hydroxyapatite (HA) is a biocompatible, non-restorable, osteoconductive material and the main composition of the bone and tooth minerals. It was widely used in research related to periodontal defects (Nandi *et al.,* 2009; Sopyan & Naqshbandi, 2014). The chemical composition of hydroxyapatite is very similar to that in teeth and bone, making it the biomaterial of choice in many dental and medical applications (Dorozhkin & Epple, 2002; Comín *et al.,* 2017).

Hydroxyapatite is considered as the main inorganic component of enamel, dentine, cementum and bone. Because of its amazing bioactive properties, it is widely used in orthopaedics and in dentistry (Roeder *et al.,* 2008; Mendelson *et al.,* 2010).

2.6.2 Hydroxyapatite properties

Hydroxyapatite has excellent biocompatibility and osteoconductive properties, making it attachable to the bone and accelerates bone formation process (Martz *et al.,* 1997; Sopyan *et al.,* 2007; Pattanayak *et al.,* 2011). On the other hand, it has poor mechanical properties, making it brittle and unable to withstand high forces during load applications (Choi *et al.,* 1998; Veljović *et al.,* 2011; Sopyan *et al.,* 2013).

2.6.3 Hydroxyapatite applications

Hydroxyapatite has been used in many dental applications and involved in many root canal treatments, including pulp capping, repairing periapical defects and perforations at bifurcation areas (Chohayeb *et al.,* 1991; C. Liu *et al.,* 1997). It was used as a coating material with dental implants and as a filler in the reinforcement process of composite resins (Munting *et al.,* 1990; Chang *et al.,* 1997; Arcís *et al.,* 2002). Moreover, it is also used to treat bone defects (Yukna *et al.*, 1985), and intrabony periodontal pockets (Meffert *et al.*, 1985).

Recently, hydroxyapatite has been modified to be very close to bone and teeth in terms of mineral composition. This modification improves uses of hydroxyapatite in bone grafts, with dental implant and materials (such as; calcium hydroxide liner and calcium phosphate cement). A new synthesised hydroxyapatite based sealer (calcium phosphate cement) was compared to other types of calcium phosphate sealers by Kim *et al.* (2004). Results indicated that the biocompatibility of all groups was acceptable.

The addition of materials to hydroxyapatite overcome its poor mechanical properties and widen its applications in biomedical field has been investigated. Multiple materials have been used with hydroxyapatite to reinforce it (Miao *et al.*, 2001; Silva *et al.*, 2001), including 20%-30% of Fe-Cr to form HA matrix composites and increase its fracture resistance and strength (Suchanek and Yoshimura, 1998). Another study added silica coated titanium to increase the mechanical properties of Ti-HA composites (Wakily *et al.*, 2015).

In another study by Ramires *et al.* (2001), a composite was made from HA and TiO_2 and produced by a sol-gel process. The study reported that the biocompatibility and cell activity of the composite were encouraging. In a study done by Volceanov *et al.* (2006) the mechanical properties of hydroxyapatite matrix reinforced by zirconia were evaluated. The study reported that the mechanical characteristics of the produced HA composites were enhanced.

Hydroxyapatite is stable in body fluids that make it useable as coating materials (Hench & Wilson, 1993; Mistry *et al.*, 2011). The use of hydroxyapatite as a coating material was introduced in the 1960s. The hydroxyapatite-coated implant was first clinically used in 1985 (Furlong & Osborn, 1991). Many studies have shown that using implants coated with hydroxyapatite improved the clinical success rate of (Groot *et al.*, 1994; Geesink, 2002; Mistry *et al.*, 2011).

2.7 Titanium

2.7.1 Introduction

Titanium is a widely used material in the world. It was first noticed in rutile structure and also reported to be found in stars, interstellar dust and the earth's surface. It is considered the fourth most widespread metals, surpassed by aluminium, iron and magnesium. It is also more prevalent than other common materials, such as chromium, nickel, cooper, tungsten. Titanium is characterised by its high strength and ability to resist corrosion, making titanium suitable to be used in many industrial applications (Welsch *et al.,* 1993; Guo *et al.,* 2006; Veiga *et al.*, 2012; Attar *et al.*, 2014).

2.7.2 Titanium properties

Titanium has a high mechanical strength and its corrosion resistance abilities make it the favourable metal material to be used in biomedical applications (Elias *et al.*, 2008; Veiga *et al.,* 2012; Attar *et al.*, 2014).

In terms of physical properties, titanium has the ability to resist corrosion due to the presence of a thin (about 4 nm) oxide layer on its surface which is spontaneously formed by titanium (Welsch *et al.,* 1993; Elias *et al.*, 2008). Titanium has good resistance to corrosion and low ion formation abilities in watery conditions; thus titanium is considered as a biocompatible material (Brunette *et al.*, 2012).

2.7.3 Titanium applications

Production of titanium is beneficial in many fields, including aerospace, medical, frames, engine components, chemical and petrochemical industries. The highlights of titanium and its temperature and strength properties were widely recognised in the 1940s and 1950s (Welsch *et al.*, 1993; Veiga *et al.*, 2012).

Titanium, stainless steel and cobalt alloys are the most used metals in biomaterial fields. Titanium has shown promising uses in medical applications and has been successfully used in implant devices to replace damaged hard tissue, in which replacement include the use of titanium in synthesis of artificial body joints at knee and hip areas (Welsch *et al.*, 1993; Elias *et al.*, 2008; Brunette *et al.*, 2012). In addition, it is used as screws to fix fractured bone and in cardiac and cardiovascular applications, such as artificial hearts, valves and pacemakers (Elias *et al.*, 2008; Brunette *et al.*, 2012).

In dentistry, titanium is widely used and in many applications, including dental implants, crowns and bridges, overdentures and dental posts (Elias *et al.*, 2008). There are special made titanium alloys (titanium mixed with 6% aluminium and 4% vanadium (Ti-6Al-4V)) and commercially pure titanium (four grades of unalloyed commercial pure titanium (CPTi) specified for dental implant applications (Brunette *et al.*, 2012; Elias *et al.*, 2008). These titanium materials exhibit low shear strength and poor wear resistance when applied in orthopaedic applications. Ti-6Al-4V is also considered as the main titanium alloy for biomedical implants in addition to dental implants (Elias *et al.*, 2008).

The difference between Ti-6Al-4V and CPTi implants, when in contact with bone tissue, has been widely studied. CPTi implants have shown better bone connection compared to Ti-6Al-4V (Johansson *et al.*, 1998). However, Ti-6Al-4V implant was reported to show strong attachment to the bone of goats and rabbits when coated with hydroxyapatite, especially during the first two weeks of implantation which has been reported to be four times stronger compared to that of bone implanted with uncoated titanium implant (Oonishi *et al.*, 1989).

In general, the surface roughness and titanium implant composition affect the bone-implant interface connection and bone formation. Titanium implants with a rough surface is favourable for biomechanical retention while titanium implants coated with calcium phosphate enhance the bone-implant interface connection and simulate fast bone formation (Guéhennec *et al.,* 2007; Mistry *et al.,* 2011).

2.8 Bioactive glass materials

2.8.1 Introduction

Bioactive glass materials are silicate based materials which can strongly attach to hard and soft tissues. It has high biocompatibility and shows osteoconductive abilities which make it able to form a hydroxyapatite layer when connected to body fluid or bone tissues. The possibility of its use as an implant in body tissue was studied many decades ago. It was first proposed in 1969, when the failures of implanting metals and polymers in body occurred due to the formation of fibrous tissue. A material which can provide ossteoconductivity with low failures rate was introduced as an alternative choice to these materials. The proposed bioactive glass (nowadays known as 45S5 and Bioglass® which consist of Na_2O-CaO-SiO_2-P_2O_5) showed strong attachment to the bone which was not easily broken (Hench, 2006; Kaur *et al.,* 2014; Prasad *et al.,* 2017).

In the production of bioactive glass materials, the sol-gel method gives better properties as compared to that of a conventional method in which the properties produced from sol-gel technique exhibit lower sintering temperature, better purity, more homogeneous and improves control in bioactivity behaviour as compared to that of the conventional materials (Abbasi *et al.,* 2015).

2.8.2 Bioactive glass properties

Bioactive glass materials have a low melting temperature and excellent biocompatibility with body tissues, making it one of the important materials to be used in tissue engineering field. Its strong chemical bonds to body tissue depend on CaO ratio in its composition. High CaO ratio improves the bonds which occur due to the formation of hydroxyapatite layers by bioactive glass when it touches body tissue or simulated body fluid (SBF) which have similar concentration to the plasma of the human's blood (Laudisio & Branda, 2001; Mistry *et al.*, 2011; Kaur *et al.*, 2014; Prasad *et al.*, 2017).

Moreover, it was reported that bioactive glass is non-toxic and does not cause inflammation of body tissues (Greenspan *et al.*, 1994). On the other hand, it has low mechanical properties, making it difficult to be used in load-bearing applications (Bachar *et al.*, 2012). However, this problem can be solved by adding materials such as nitrogen and fluorine to the bioactive glass to improve its mechanical properties. Addition of these materials would enhance mechanical properties as they reduce the melting and transition temperature of glasses, and increase the hardness and modulus of elasticity of bioactive glass (Hanifi *et al.*, 2011).

In addition, Prasad *et al.* (2017) produced different composites with various 45S5 (bioactive glass) and hydroxyapatite ratios (95% Ti-5% HA, 90% Ti-10% HA, 85% Ti-15% HA, 80% Ti-20% HA). The composites were sintered at 1000 ^{o}C -1050 ^{o}C and then immersed in simulated body fluids to investigate the ability of hydroxyapatite to reinforce bioactive glass and improvement in its bioactivity and mechanical properties. Results reported that the bioactivity and mechanical characteristics were improved in composites with higher HA ratios providing higher modulus of elasticity, density, shear and compressive strength, whereas Poisson's ratio remained unchanged.

2.8.3 Bioactive glass applications

Bioactive glass has been used in many medical and dental applications, including scaffold, bone graft, coating materials and to treat tooth hypersensitivity (Boccaccini *et al.,* 2010; Brovarone *et al.,* 2012; Kaur *et al.,* 2014). In 1986 it is first clinically used clinically as a middle ear prosthesis to treat hearing problems (Abbasi *et al.,* 2015).

There are many bioactive glass products that have special applications and benefits. Endosseous ridge maintenance implant which is a Bioglass® implant can repair dental root defects and can also be used as an abutment to retain dentures (Abbasi *et al.,* 2015). Perioglas® is used as a bone graft in damaged jaw bone and to treat bone defects due to periodontal lesions, promote tissue regeneration, demineralise dental surfaces and is successfully used as disinfectant in root canal during instrumentation process (Vollenweider *et al.*, 2007; AboElsaad *et al.*, 2009; Yadav *et al.*, 2011).

Biogran® and BonAlive® are mainly used as artificial bone graft materials (Turunen *et al.,* 2004; Jones, 2015). NovaMin® is used in corporation with toothpaste to treat dental hypersensitivity and provides rapid remineralisation to the dental surfaces (Earl *et al.,* 2011; Mehta *et al.*, 2014).

Furthermore, bioactive glasses also show antibacterial ability. S5sP4 (a bioactive glass product) is able to kill various bacteria that cause dental caries and periodontitis, such as *Streptococcus mutans* and *Actinobacillus mutans* (Zhang *et al.,* 2010). This ability is enhanced by adding antibacterial elements (silver, zinc, and copper) to bioactive glass materials (Aina *et al.,* 2007).

Bioactive glass can also be used as a coating material in dental applications. In a study by Mistry *et al.* (2011), two groups of titanium dental implants were coated with hydroxyapatite and bioactive glass in 31 patients to evaluate the effectiveness of both coating materials on osseointegration in bone-implant interface. In the first group, implants were coated with hydroxyapatite by using microplasma spray method, whereas in the second group implants were coated with bioactive glass material by using vitreous enamelling methods. Results showed that both coating materials were biocompatible. Bioactive glass group showed excellent osseointegration rate similar to that of hydroxyapatite. The study also concluded that bioactive glass was the material of choice to be used as alternative coating materials of the dental implant.

2.9 Functionally graded materials

2.9.1 Introduction

Functionally graded materials (FGM) are advanced composites composed of two or more materials graded in many layers to gradually change properties. This can provide new graded (layered) composites with the best characteristics of the used materials (Lannutti, 1994; Shukla *et al.,* 2007; Mahamood & Akinlabi, 2017). It was developed to overcome the problem that happens due to the use of composites in applications that require high temperature at which the used materials have various expansion and thermal characteristics (Wang, 1983; Niino *et al.*, 1987; Gupta & Talha, 2015).

In 1984, a group of researchers in Japan did a study on this subject (functionally graded materials). A hypersonic space plane needed different thermal layers in which the inner surfaces should tolerate 1000 K and the outer ones should tolerate 2000 K with <10 mm in thickness. They had produced the materials and introduced the term ‘functionally graded materials’ to the research field (Narayan *et al.,* 2006; Gupta & Talha, 2015).

In composites, the presence of sharp interfaces can cause failures. This problem can be addressed by functionally graded materials at which these interfaces were replaced by graded interfaces producing a proper transition from one material to another (Groves & Wadley, 1997; Alla *et al.*, 2011). The uniqueness of functionally graded materials is that they can be made to perform according to the required and specified uses (Shanmugavel *et al.*, 2012).

Functionally graded materials are also found to be in humans and animals, i.e. in teeth and bones (Knoppers *et al.*, 2005; Liu *et al.*, 2017). Tooth crown is the best example of the functionally graded materials at which it contains enamel as an outer layer to resist high wear and dentine from the inside to support the brittle enamel. Graded chemical and structural composition and characteristics of dental layers, and the presence of their perfect structural arrangement, distribution and dimensions, especially between enamel and dentine produce functionally graded features across the tooth (Marshall *et al.*, 1997; Tesch *et al.*, 2001; Freeman & Lemen, 2008; Liu *et al.*, 2017).

Furthermore, layers of the tooth are connected by graded surfaces such as dento-enamel junction (DEJ) and cemento-dentinal junction (CDJ), the attachment of tooth to the alveolar bone by periodontal ligaments are also considered as graded interfaces (Marshall *et al.*, 2001; Imbeni *et al.*, 2005; Wopenka *et al.*, 2008; Chan *et al.*, 2011; Lu & Thomopoulos, 2013; Liu *et al.*, 2017).

Fabrication of functionally graded materials depends on which type of FGM is to be formed. There are two major FGM groups; thin (thin surface coating) and bulk FGM (materials need more intensive work). Thin FGMs are fabricated by plasma spraying, chemical or physical vapour deposition, electrodeposition and electrophoretic etc. whereas bulk FGMs are fabricated by powder metallurgy method, solid free form and centrifuge (Knoppers *et al.*, 2005; Ivosevic *et al.*, 2006; Mahamood & Akinlabi, 2017).

2.9.2 Applications

Functionally graded materials have many uses in fields, such as medical, aerospace, aircraft, sensors, tools manufacturing, drilling, energy and defense (Mahamood *et al.*, 2012; Rangasreenivasulu & Reddy, 2017). Its ability to tolerate high temperatures makes it the proper material to be applied in space plane and rocket engine structures (Marin, 2005). Furthermore, due to its ability to prevent crack propagation, FGM can also be used in military as materials that resist penetrations, including bullet-proof vests and armour plates (Lu *et al.*, 2011).

Additional applications of FGM are in the optoelectronics field as refractive index materials and in audio-video discs magnetic applications. Moreover, it is also used in automobile engine structures, cutting tool insert coatings, sensors, fire retardant doors and turbine blades and many more applications which are increasing as FGM is improving day by day (Xing *et al.*, 1998; Kawasaki & Watanabe, 2002; Malinina *et al.*, 2005; Woodward & Kashtalyan, 2012).

In medical applications, it is widely used as it provides characteristics similar to bone and teeth, and thus can be considered as natural functionally graded materials. It is used in orthopaedics, replacing defected bones, dental implants and recently it is also used to fabricate dental posts (Watari *et al.*, 2004; Arifin *et al.*, 2014; Mahamood & Akinlabi, 2017).

In 2011, three FGM dental posts were produced by Abu Kasim *et al.* (2011) and their stress distribution was evaluated and showed better strain and stress distribution as compared to other types of dental posts. Abu Kasim has also reported that a new generation of dental posts can be made by using FGM and it could improve root canal treatment.

Madfa (2011b) investigated the fracture resistance and failure mode of these FGS posts and compared them to commercial posts restored in root canals of bovine extracted incisors. He concluded that there was no significant difference in terms of fracture resistances among FGSP, titanium and cast posts. They also reported that teeth restored with titanium and cast posts resulted in a higher number of irreparable fracture whilst the FGS posts and root treated only, had a higher number of reparable fractures. Functionally graded materials methods were shown to be useful in designing new types of dental posts.

Watari *et al.* (1997) have combined hydroxyapatite and titanium to produce Ti-HAP graded dental implants. These dental implants (with different Ti-HAP ratios) were pressed by using Cold Isostatic Press and sintered in an Argon gas environment at 1300 ^{o}C. Subsequently, implants were implanted in femors of rats for 1 week, 2 weeks and 4 weeks. Some of the specimens were stained painted with toluidine blue while others were unpainted. They were evaluated by using EPMA mapping technique in order to check the bone formation in the implant-bone interface. Bone was formed around the implants and no inflammation was observed, resulting in good biocompatibility of the produced FGM implant.

A multilayered crown was produced by Rahbar and Soboyejo (2011) which is inspired by the graded interfaces of dentino-enamel junction (DEJ). Stress distribution was computationally evaluated and the results showed that the stress concentrations were the highest at the crown outer layer and reduced at the interface layers, which represent DEJ of the bioinspired functionally graded layers. These layers also improved the crack size.

Ti-HA combinations are widely used as composites and in functionally graded materials in which HA provides biocompatibility and osseoconductive ability and Ti provides good mechanical strength to the FGM (Edwards *et al.,* 1997; Nanci *et al.,* 1998; Rivera, 2011; Brunette *et al.,* 2012; Veiga *et al.,* 2012; Arifin *et al.,* 2014). Sintering these materials are challenging since Ti needs to be sintered in a vacuumed furnace or an inert atmosphere to prevent oxidation process, whereas HA needs air atmosphere furnace to avoid decomposition (Raemdonck *et al.,* 1984; Weng *et al.,* 1994).

To overcome this problem Ti-HA FGM should be sintered at a lower temperature while maintaining their good properties. This can be achieved by adding low melting materials to Ti-HA FGM. Using bioactive glass as a sintering aid in Ti-HA FGM could be a good candidate to sinter these FGM at low temperature, as it maintains and improves mechanical and biocompatible properties of HA (Santos *et al.*, 1995; Kleebe *et al.*, 1997; Ning & Zhou, 2004). Good bioactivity characteristics were reported when Ti 50%-HA 40%-BG 10% composites were made and sintered at 1200 ^{o}C by Moskalenko and Smirnov (1998).

2.10 Chapter summary

This chapter reviewed tooth structures, composition and the difference between the vital and endodontically treated teeth. It also reviewed the types of restoration that can be used to restore endodontically treated teeth. In this chapter, a thorough highlight of dental post indications, classifications, types and considerations were also reviewed. Comparisons between various cement types and post designs and types were made in this chapter as well. Studies that involved the comparison between fracture resistance and failure mode in endodontically treated teeth with different types of post systems were listed and described in this chapter. Introduction of finite element analysis and its uses in dentistry were reviewed too.

This chapter also reviewed the composition and applications of hydroxyapatite, titanium and bioactive glass materials in various fields. It also reviewed the importance of the functionally graded technique, overview and applications in the medical, dental, and engineering fields.

CHAPTER 3: MATERIALS AND METHODS

3.1 Introduction

This chapter describes the preparation and incorporation of different ratios of bioactive glass at different ratios of Ti-HA mixture by using the processing method. Preparation and fabrication five layers of Ti-HA-BG composites and sintering them in two different sintering conditions are also described. Subsequently, functionally graded posts were prepared by milling, pressing and sintering the five different Ti-HA-BG mixtures in air atmosphere furnace.

FGDPS and different post types were restored in extracted teeth in order to evaluate fracture resistance and failure mode by using universal testing machine and a stereomicroscope. Four models were restored with one of four post materials; FGDPS, fibre, stainless steel and titanium posts and stress distribution was analysed. Flow chart of the samples preparations and test methods is shown in Figure 3.1.

3.2 Optimisation of different Ti-HA composites using various ratios of bioactive glass

3.2.1 Bioactive glass powder preparation

Eight different materials; silicon dioxide (SiO_2), sodium oxide (NaO), calcium oxide (CaO), phosphorus pentoxide (P_2O_5), boron trioxide (B_2O_3), titanium dioxide (TiO_2), calcium fluoride (CaF_2), and magnesium oxide (MgO), were selected to form bioactive glass. These materials with different percentage (Table 3.1) of each one (Ning & Zhou, 2004) were weighted (OHAUS, Pioneer™, NJ, USA) and then milled together by using a planetary ball milling machine (XQM, Changzhou, China) at 200 rpm for 5 hours to produce bioactive glass as shown in Figure 3.2.

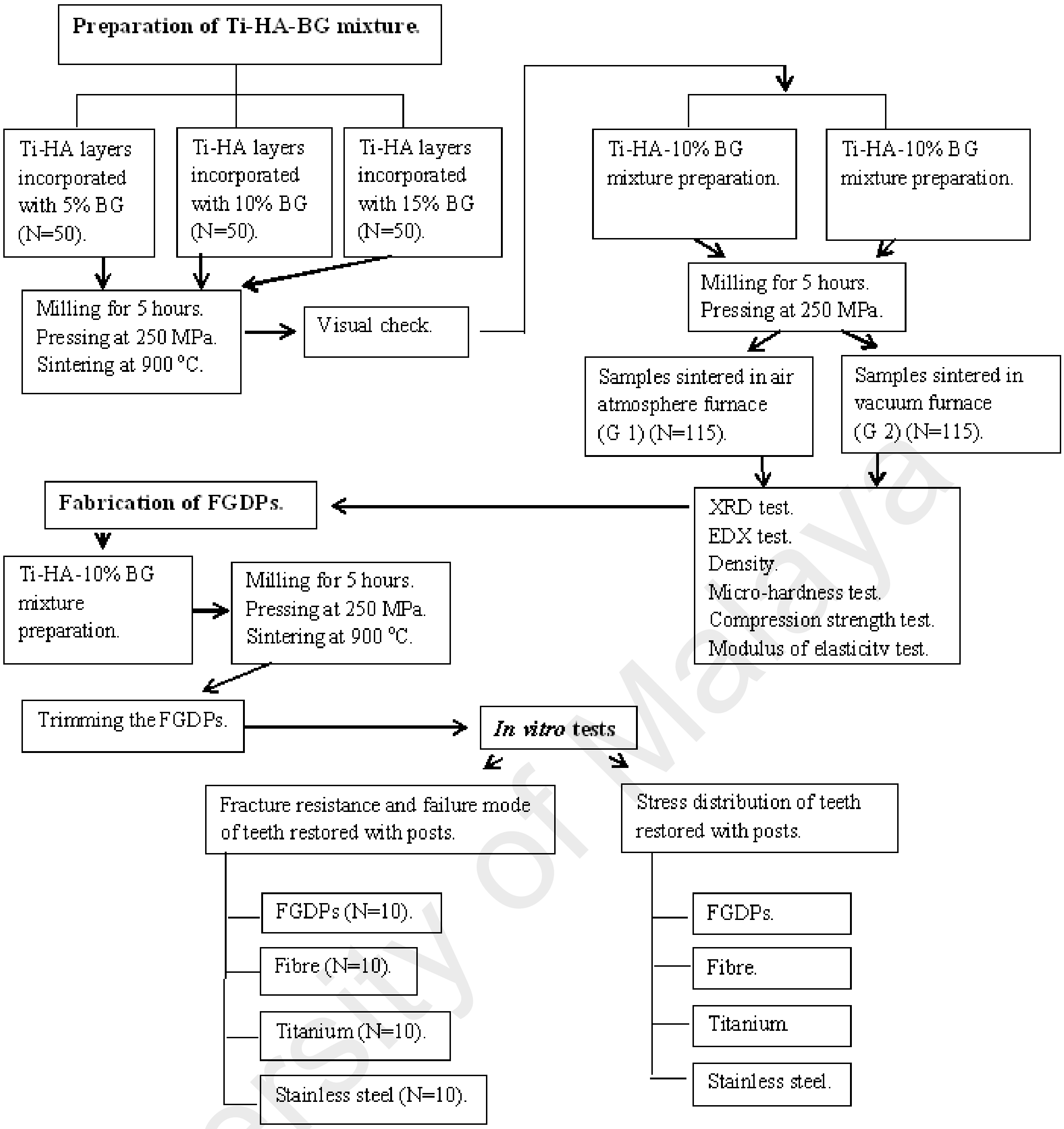


Figure 3.1: Flow chart of samples preparation and test methods

Table 3.1: Percentages and materials that form bioactive glass

Material	Percentage	Material	Percentage
SiO_2	48%	Na_2O	10%
CaO	13%	MgO	4.87%
P_2O_5	9.2%	B_2O_3	9.7%
CaF_2	0.98%	TiO_2	4.25%

Figure 3.2: Bioactive glass powder

3.2.2 Titanium-hydroxyapatite-bioactive glass powder preparation

Titanium with particle size of ~ 45 µm, and hydroxyapatite of ~ 5 µm particle size were purchased and prepared (Sigma-Aldrich, Darmstadt, Germany).

Ti-HA and BG powders were dried in an incubator at 110 ^{o}C for 12 hours to avoid particles from sticking to each other and reduce the possibility of agglomerates formation of the mixtures when they were sent for milling and mixing (Madfa, 2011a).

Ti-HA powders were then poured into special bowls and then mixed and milled together by using a planetary ball milling machine (Figure 3.3) for five hours to form five layers of Ti-HA mixtures with five different ratios. Three different percentages of bioactive glass were added to these five Ti-HA mixtures (Table 3.2) before mixing and milling the mixture together.

Figure 3.3: Powders placed in the jars of the milling machine for mixing procedure

3.2.3 Samples grouping

In this part of the study, 150 samples were divided into three main groups and five layers at which each layer has 10 samples, as shown in Table 3.2. In Group 1; 5% BG was added to different Ti-HA mixtures in all five layers, while in Group 2; 10% BG was added to Ti-HA mixtures and in Group 3; 15% BG was added to Ti-HA mixtures in all layers.

Table 3.2: Samples distribution in the groups and layers

Layers	Group 1	n	Group 2	n	Group 3	n
Layer 1	Ti 95% BG 5%.	10	Ti 90% BG 10%.	10	Ti 85% BG 15%.	10
Layer 2	Ti-74% HA 21% BG 5%.	10	Ti 70% HA 20% BG 10%.	10	Ti 67% HA 18% BG 15%.	10
Layer 3	Ti 47.5% HA 47% BG 5%.	10	Ti 45% HA 45% BG 10%.	10	Ti 42.5% HA 42.5% BG 15%.	10
Layer 4	Ti 21% HA 74% BG 5%.	10	Ti 20% HA 70% BG 10%.	10	Ti 18% HA 67% BG 15%	10
Layer 5	HA 95% BG 5%.	10	HA 90% BG 10%.	10	HA 85% BG 15%.	10

3.2.4 Samples preparation

Moulds were made by using light body silicone impression materials (Aquasil; Dentsply, Konstanz, Germany) by taking an impression of a plastic tube with 10 mm in diameter and 30 mm in length (Figure 3.4). The powders were poured into these moulds, each layer in different moulds separately (Figure 3.5A). The excess of the moulds was sectioned by using sharp blades and then all moulds were sealed with the same impression materials to facilitate samples pressing (Figure 3.5B).

Figure 3.4: Taking impression of the plastic tube using silicone putty

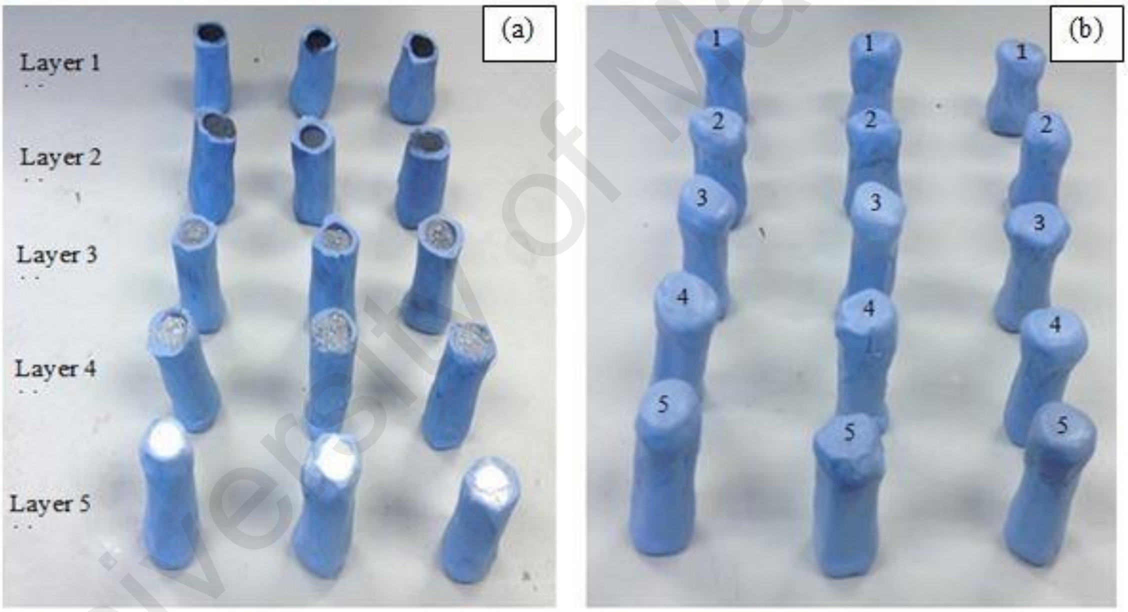


Figure 3.5: (a) Filling the moulds with the Ti-HA-BG powders. (b) Sealing the moulds

3.2.5 Samples pressing

Samples were sent to be pressed by using a cold isostatic press. Samples were placed in gloves and tightly wrapped to prevent machine oil from entering the samples. The samples were then placed in a special container and inserted in the cold isostatic machine. Figure 3.6 shows samples placed in a container in order to be inserted in a hole filled with oil. Subsequently, the machine was tightly closed for samples pressing at 250 MPa for 20 minutes. Samples after pressing process are shown in Figure 3.7.

Figure 3.6: Putting the samples in the cold isostatic press machine

Figure 3.7: Samples after being pressed

3.2.6 Samples sintering

Moulds were then removed and samples were sintered by using a non-vacuumed furnace at a heating rate of 5 ^{o}C/min (XINYOO-1700, Henan, China, Figure 3.8) at 900 ^{o}C. The samples were heated from room temperature to 500 ^{o}C for 240 minutes. The temperature was gradually increased to 900 ^{o}C in 240 minutes and kept at the same value for 240 minutes, as shown in Figure 3.9.

Samples were then cooled overnight for 8 hours and were removed from the furnace. Samples were then polished and grounded flat and smooth by using polishing and grinding machine (Metaserv® 2000 grinder, Coventry, England) and mechanically and chemically tested.

Figure 3.8: XINYOO-1700 air atmosphere furnace

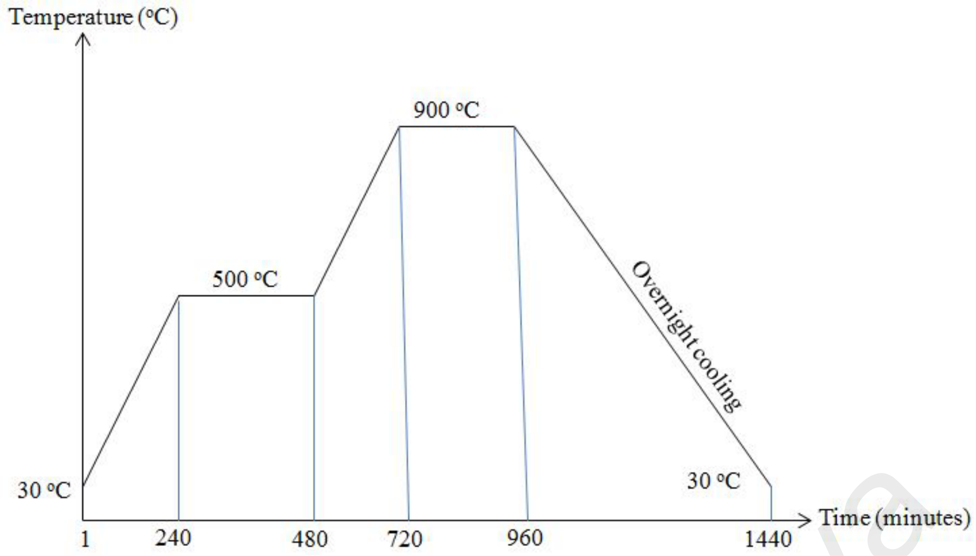


Figure 3.9: Sintering cycle of the samples

3.3 Characterization and optimisation of the Ti-HA-BG composites to fabricate functionally graded dental posts

3.3.1 Ti-HA-BG powders preparation

For the preparation of Ti-HA-BG mixtures, commercially pure titanium (45 μm), hydroxyapatite (~5 μm), bioactive glass materials, were purchased from Sigma-Aldrich, Germany. Bioactive glass was prepared by mixing and milling the eight materials at different ratios as previously mentioned in the 'bioactive glass powder preparation' Section 3.2.1.

3.3.1.1 Drying the Ti-HA-BG powders

Prior to milling and mixing of the powders, Ti, HA, and bioactive glass were dried at 110 ^{o}C in an incubator overnight to prevent the powder mixtures from sticking together during the milling and mixing process.

3.3.1.2 Mixing and milling the Ti-HA-BG mixtures

The Ti-HA-BG was prepared by milling and mixing the mixtures containing five different weight ratios of HA, Ti, and BG as following:

Layer 1: Ti 90%-BG 10%
Layer 2: Ti 70%-HA 20%-BG 10%
Layer 3: Ti 45%-HA 45%-BG 10%
Layer 4: Ti 20%-HA 70% -BG 10%
Layer 5: HA 90%-BG 10%

The mixing and milling procedure was done by using a planetary ball milling machine (XQM, Changzhou, China) at a speed rate of 200 rpm with zirconia balls. Five different mixtures were placed in the bowls of the milling machine and then the bowls were closed tightly. The different mixtures were then milled for 5 hours to get a homogenous particle shape and a uniform mixture composition (Figure 3.10).

Figure 3.10: Different mixtures of the five layers

3.3.1.3 Ti-HA-BG mixtures pressing

The obtained mixtures were then poured into the prepared silicone moulds and sealed with the same silicone material as previously mentioned in this chapter (Section 3.2.4) layer by layer separately. Each mould has a different layer mixture and was labelled as layer 1, layer 2, layer 3, layer 4 and layer 5. The moulds were then placed in sample holders and inserted in the cold isostatic machine and tightly closed. The samples were then pressed by using the cold isostatic press at 250 MPa for 20 minutes. The samples, after being pressed in cold isostatic press, are shown in Figure 3.11.

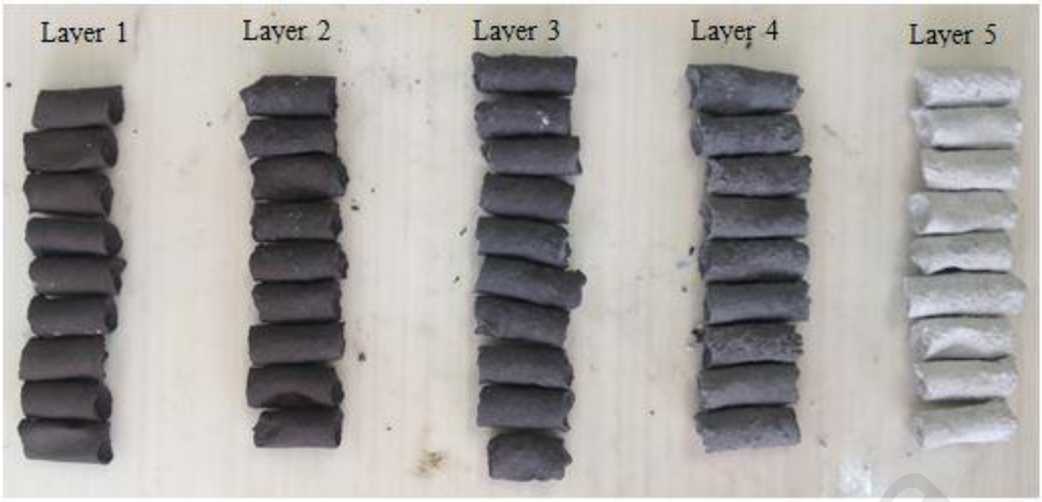


Figure 3.11: Samples after being pressed in cold isostatic press

3.3.2 Sintering the samples in air atmosphere and vacuum furnaces

In this part of the study, 230 samples were divided into two main groups (each of 115 samples) and five layers (subgroups) in each group as shown in Table 3.3. 130 samples were disc-shaped samples to be subjected to XRD, EDX, density and micro-hardness tests while 100 samples were made cylindrical in shape for compression strength and modulus of elasticity tests.

Table 3.3: Samples distribution in the groups and layers

Layers	Group 1 (air atmosphere furnace)	n*	Group 2 (vacuum furnace)	n*
Layer 1	Ti 90% BG 10%.	23	Ti 90% BG 10%.	23
Layer 2	Ti 70% HA 20% BG 10%.	23	Ti 70% HA 20% BG 10%.	23
Layer 3	Ti 45% HA 45% BG 10%.	23	Ti 45% HA 45% BG 10%.	23
Layer 4	Ti 20% HA 70% BG 10%.	23	Ti 20% HA 70% BG 10%.	23
Layer 5	HA 90% BG 10%.	23	HA 90% BG 10%.	23

* n. = number of samples.

In Group 1, samples were sintered with air atmosphere furnace at 900 ^{o}C with a heating rate of 5 ^{o}C/min (Figure 3.7). While in Group 2, samples were sintered in vacuum furnace at 900 ^{o}C/min (Nisshin Giken, Tokyo, Japan, Figure 3.12). The pressure used to remove the air from the vacuum furnace was 10^{-6} Torr. In both furnaces, the temperature was gradually raised from room temperature to 500 ^{o}C within 240 minutes and then the temperature was kept at 500 ^{o}C for 240 minutes. Subsequently, the temperature was raised again to 900 ^{o}C within 240 minutes, after which, it was maintained at 900 ^{o}C for 240 minutes. The temperature was then gradually decreased from 900 ^{o}C by setting the furnaces to the overnight cooling mode for 8 hours (Figure 3.9).

Figure 3.12: Sintering the samples in a vacuum furnace

Samples were then removed from the air atmosphere (Figure 3.13) and vacuum (Figure 3.14) furnaces and sent for polishing by using a polishing and grinding machine for chemical, physical and mechanical testing processes. Samples were divided into disc-shaped samples (for chemical, physical and micro-hardness tests purposes and cylindrical-shaped samples for compression test.

Figure 3.13: Samples after being sintered in air atmosphere furnace

Figure 3.14: Samples after being sintered in vacuum furnace

3.3.3 Evaluation of the chemical properties of the samples

3.3.3.1 X-ray diffraction (XRD) analysis

After sintering, 30 samples (disc-shaped) of both groups (each of 15) and five layers (each of 3) with 10 mm in diameter and 5 mm in thickness were prepared and distributed as indicated in Table 3.3. Samples were then subjected to chemical testing and evaluation in which they were sent for x-ray diffraction (PANalytical EMPYREAN, Lelyweg Almelo, Netherlands). All data were analysed by using X'Pert HighScore software (X'Pert Pro, Panalytical, Almelo, Netherlands, Figure 3.15). XRD was used to analyse the chemical compositions of each layer and to determine whether the Ti was oxidised or the HA decomposed.

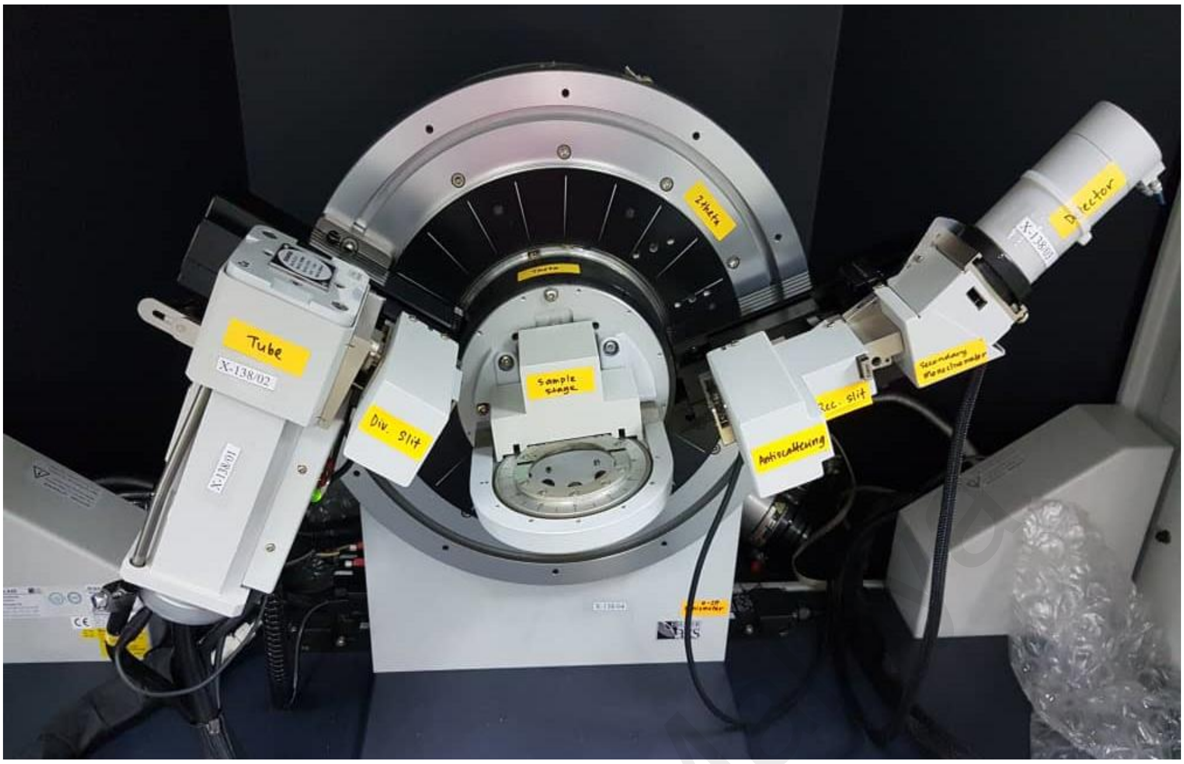


Figure 3.15: XRD machine

3.3.3.2 Element analysis

Thirty samples were then subjected to energy dispersive x-ray spectroscopy (EDX) (Inca software, Oxford Instruments, Bucks, UK) attached to scanning electronic microscope (SU8220, Hitachi, Tokyo, Japan) for element analysis purposes. It was used to check the concentration of the elements and to check the contamination of each layer.

3.3.4 Measuring the density of the samples

The density of 100 disc-shaped samples with 10 mm in diameter and 5 mm in thickness (prepared and distributed as described in Table 3.3) was measured by using a densitometer (Sartorius AX224, Goettingen, Germany) and the values were calculated by using the Archimedes' principle (Equation 1):

$$\rho = W_a \times \rho w / W_a - W_w, \qquad (1)$$

At which ρ is the density, W_a, is the weight of the samples in the air, while W_w is the weight of the samples in water, and ρ_w is the water density, which is 0.99 g/cm^3.

3.3.5 Evaluation of mechanical properties of samples

3.3.5.1 Evaluation of micro-hardness

The same 100 disc-shaped samples that were used to be physically (density) evaluated were then subjected to Vickers hardness tester (HMV- Shimadzu Kyoto, Japan, Figure 3.16) to test the mechanical properties of the samples. All samples were mounted in silicone moulds with epoxy resin and then ground and smoothed by using a grinding and polishing machine (Metaserv® 2000 grinder, Coventry, England) with 600 and 800 grit abrasive paper (Buehler, silicon carbide, , Illinois, USA). Each sample was subjected to three indentations with a load of 4.90 N for 15 seconds. Micro-hardness was automatically evaluated by measuring the pyramids diagonals that were made by the indenter on the layer surfaces.

Figure 3.16: Vickers micro-hardness tester

3.3.5.2 Evaluation of the compression strength and modulus of elasticity

Compression strength and modulus of elasticity of the 100 cylindrical-shaped samples with 10 mm in diameter and 12 mm in length (prepared and distributed as mentioned in Table 3.3) were evaluated. Samples were compressed at 0.5 mm/min by using the universal testing machine (HMV, Shimadzu. Japan, Figure 3.17).

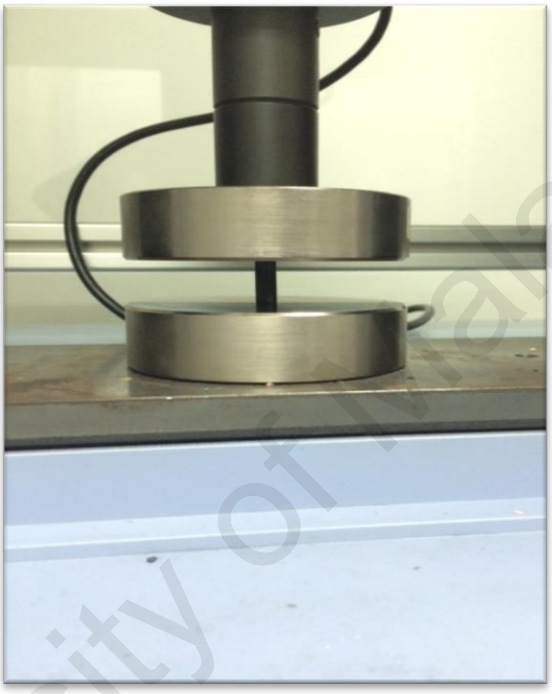


Figure 3.17: Sample subjected under universal testing machine for compression test

3.3.6 Fabrication of the functionally graded dental post

3.3.6.1 Samples preparation

Moulds were made by using silicone impression materials (Aquasil; Dentsply), Ti-HA-BG powders were poured into these moulds layer by layer starting from layer 1 to layer 5 and then sealed with the same materials used for impression (Figure 3.18). Thickness of the layers was standardized by standardizing the mixture's weight (500 mg) for each layer.

Figure 3.18: Moulds being sealed with impression material

The samples were then pressed by using Cold Isostatic Press with 250 MPa. Subsequently, the moulds were removed and the samples were sintered by using atmosphere furnace at 900 ^{o}C. These FG composites were then polished and ground by using a polishing and grinding machine (Figure 3.19).

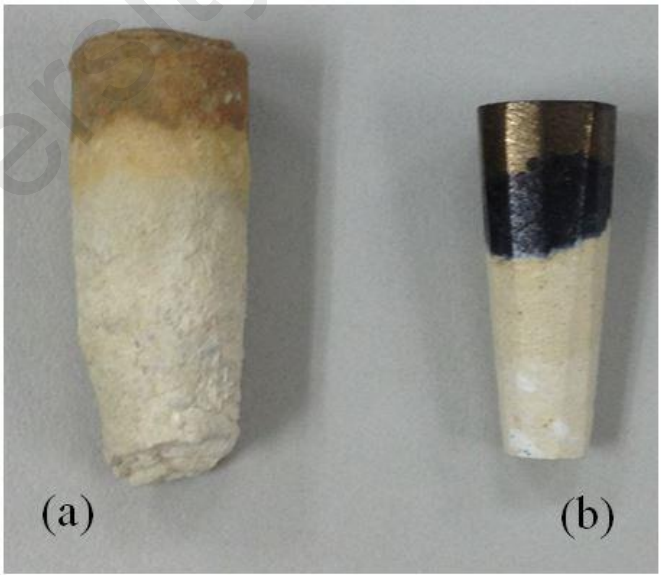


Figure 3.19: (a) Unpolished FG composite. (b) Polished FG composites

3.3.6.2 Machining and shaping of FG multilayered composites

Samples were sent for machining and shaping to be a functionally graded dental post (FGDP) by using a Centreless grinder (Palmary PC-12S-NC. Taichung, Taiwan, Figure 3.20). A Centreless grinder is a machine based on the use of the abrasive cutting technique to grind the samples to the desired dimension. Samples were placed one by one on a V-shape sample holder in the Centreless grinder attached to two wheels from left and right sides.

One wheel is stationary and the other one is a moving wheel under the water cooling system to avoid any damage caused by the heat to the samples during the abrasive cutting process. Samples were then sloped into the wheels and ground by the wheels with a speed of 1200 round per minute (R.P.M) to get the desired FGDPS with the required diameter of 1.5 mm for all samples (Figure 3.21).

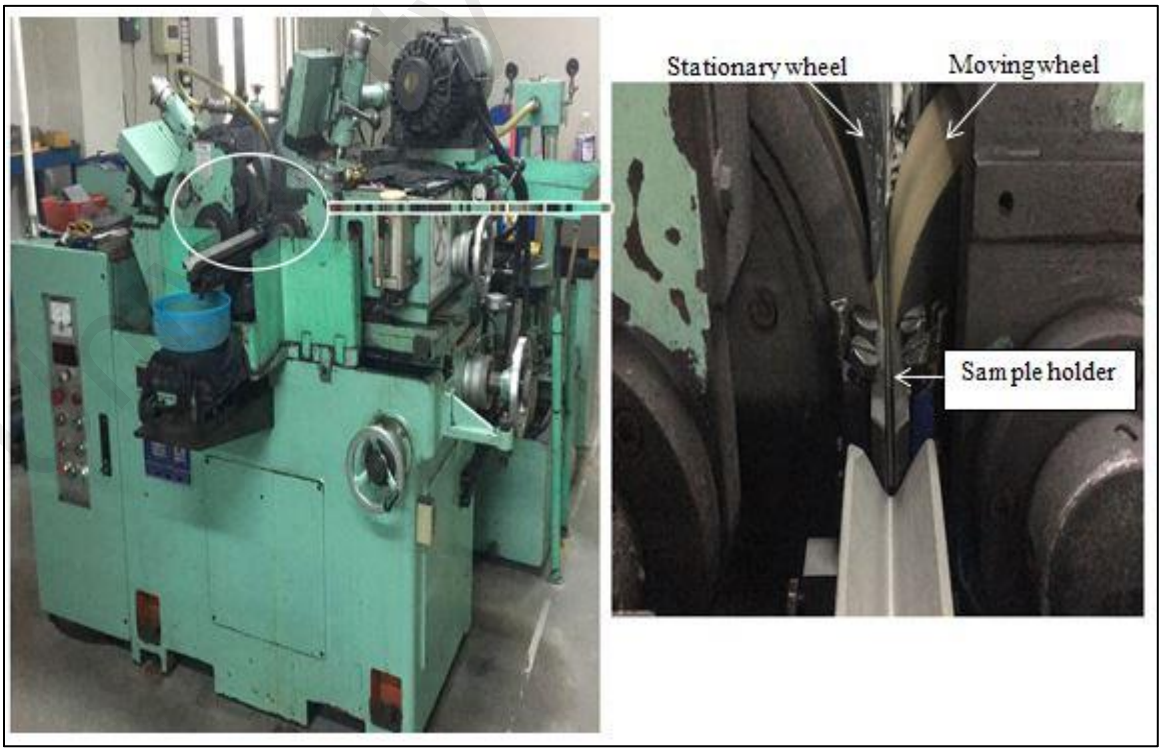


Figure 3.20: Palmary PC-12S-NC centreless grinder

Figure 3.21: Functionally graded dental post after machining and shaping

3.4 Fracture resistance and failure mode evaluation of the endodontically treated teeth restored with FGDPs and other post types.

3.4.1 Selection and decoronation of the teeth

A total of 40 extracted human maxillary central incisors without caries, cracks or previous endodontic treatments were selected (Figure 3.22). Teeth were then disinfected for one week in 5% chloramine T trihydrate solution according to ISO/TS 11405 (2003). Ultrasonic scaler (Peizon Master 400, Nyon, Switzerland) was used to remove all debris and calculus from the teeth. Teeth were then examined under a stereomicroscope (Olympus, U-CMAD3, Tokyo, Japan) to check for any existing cracks on the teeth.

Figure 3.22: Selection of the teeth

Dimensions of all teeth were standardised to be 7 to 8 mm at the buccolingual surface and 5 to 6 mm at the mesiodistal surface, where the measurements were done by using a digital caliper (Mitutoyo, Kanagawa, Japan) along the cemento-enamel junction (CEJ). Also the root morphology, canal abnormalities, and canal number were checked with periapical radiographs.

Distilled water was used to store all the teeth at 4 ^{o}C in a refrigerator prior to further procedures and the water was weekly changed. All teeth were measured at 16 mm from the apex to the cemento-enamel junction (Figure 3.23a), then the rest of the crowns were sectioned horizontally to the root long axis (Figure 3.23b) by using the diamond disc (Edenta, Diamantscheibe, Switzerland) attached to straight hand-piece.

After that, a polishing and grinding machine (Metaserv 2000, Coventry, England) with 600 grit abrasive paper (Buehler, silicon carbide, , Illinois, USA) was used to smoothen all the sectioned aspects of the roots.

Figure 3.23: (a) Sound tooth measured with digital caliper. (b) Sectioned root with 16 mm length

3.4.2 Root canal treatment procedures

Pulp tissues were removed by using barbed broach (Dentsply Maillefer, Ballaigues, Switzerland) and then canals length was measured before starting the instrumentation by placing size 10 K-file (Dentsply/Maillefer, Ballaigues, Switzerland) in the canals to the apical foramen. Then 1 mm was reduced from the file length to get the working length. Instrumentation started with size 30 K-file followed by 35 K-file, 40 K-file and 45 K-file as a master file, the working length for all of these files is 14 mm.

Size 50 K-file was used to enlarge the canals with 14 mm in length followed by 55 K-file with 13 mm and 60 K-file with 12 mm in length. After each instrumentation step, 10 K-file was used to check the canals patency and canals were irrigated with 3.0 ml of 1% sodium hypochlorite solution (Clorox (M), Kuala Lumpur, Malaysia) to remove the debris from the root canals. Then all root canals were irrigated with 17% ethylene diamine tetra aetic acid (EDTA) (SmearClear, CA, USA), followed by distilled water to flush away EDTA traces from the root canals.

Papers points (Dentsply/Asia, Hong Kong, China) were used to dry all the root canal and size 45 master gutta-percha (Dentsply/Asia, Hong Kong, China) were then inserted into root canals to determine the working length. The used sealer for root canals obturation was resin-based sealer (AH-plus, Dentsply DeTrey, Konstanz, Germany, Figure 3.24). The sealer's two pastes were mixed 1:1 ratio by using a metal spatula on a glass slab based on manufacturer's guidelines. Paper points were used to insert the sealer into the root canals and master gutta-percha was also emerged in the sealer and then placed in the root canals.

Figure 3.24: AH Plus sealer

Obturations were carried out by using lateral condensation technique and a space for accessory gutta-percha was provided by using a finger spreader (Dentsply, Ballaigues, Switzerland). All excess of the gutta-percha was removed by using a heated condenser at the root canal orifices to be restored with temporary cement (IRM Caulk, Dentsply, Philadelphia, USA). All roots were then stored in the incubator (Memmert GmbH, Schwabach, Germany) for 24 hours at 37 ^{o}C with 100% humidity to achieve the complete set of the sealer.

3.4.3 Samples grouping

After the obturations were carried out for all the 40 root canals; they were divided into four groups with 10 of each.

Group 1: Functionally graded dental posts (FGDPs) with 14 mm length and 1.50 mm diameter were used for this group.

Group 2: In this group, titanium posts (Para Post XP, Coltene/Whaledent, Ohio, USA) with 14 mm in length and 1.5 mm in diameter were used to restore all roots.

Group 3: Roots in this group were restored with fibre posts (Para Post Fibre Lux, Coltene/Whaledent, Ohio, USA) with 14 mm in length and 1.5 mm in diameter.

Group 4: All root in this group were restored with stainless steel posts (Para Post XP, Coltene/Whaledent, Ohio, USA) with 14 mm in length and 1.5 mm in diameter.

3.4.4 Post space preparation

Size 2 and size 3 gates-glidden drills (Dentsply Maillefer, Ballaigues, Switzerland) attached to a low-speed hand-piece were used to remove the gutta-percha from the root canals.

The working length of the gates-glidden drills was 9 mm while 5 mm of gutta-percha was kept intact at the apex of the roots to inhibit an adequate apical seal. After that, special drills (Figure 3.25) with a working length of 9 mm that matched each post were used to prepare the post space.

Figure 3.25: Post drill

3.4.5 Post placement and cementation

Sodium hypochlorite of 1% was used to flush all canals followed by distilled water. The canals were then dried by using papers prior to post cementation and posts were placed in the canals to make sure that the acquired post length of 9 mm was achieved. Zinc phosphate cement (Elite Cement 100, GC Corporation, Tokyo, Japan) (Figure 3.26) was used to cement all the posts in the canals according to the manufacturer's guidelines.

A size 30 K-file was used to insert the cement into the canals and then the posts were coated with the cement and inserted into the canals. Cement was left to be set by applying a finger pressure for 5 minutes to all posts in each canal.

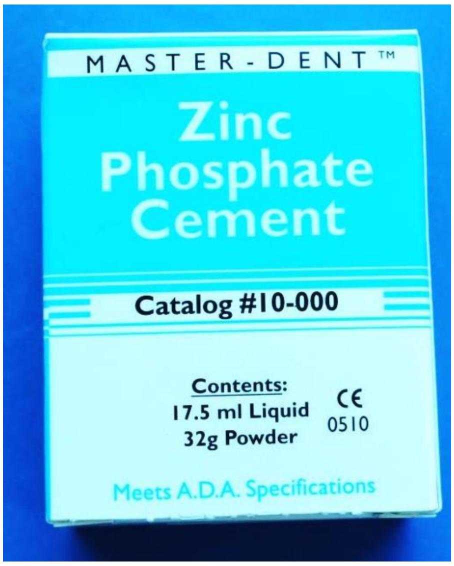


Figure 3.26: Zinc phosphate cement (Elite Cement 100)

3.4.6 Tooth mounting and silicone impression materials

Free gingival depths were simulated by marking 3 mm below the cemento-enamel junction. Roots were then coated with light body silicone materials (Aquasil Ultra XLV, Dentsply, Caulk, Konstanz, Germany) inserted in the applying gun (Figure 3.27) with 0.15 mm-0.40 mm in thickness from the marked area to the roots apexes in order to simulate the periodontal ligament.

All roots were then vertically stabilised in the silicone moulds (23 mm in length, 23 mm in width and 25 mm in depth) and then all moulds filled with epoxy resin (PACE Technologies, Quick mount 2 Epoxy Resin, Tucson, USA) to the marked areas of the roots. Epoxy resins were left to be completely set for 6 hours.

Figure 3.27: Aquasil ultra XLV kit and the applying gun

3.4.7 Core material and build up

Composite resin (Filtek Z 350, 3M/ESPE, Utah, USA, Figure 3.28) was used to build up the cores over the posts and roots. Phosphoric acid gel of 37% (Scotchbond, 3M/ESPE, Minnesota, USA) was used to etch all the root sectioned areas for 15 seconds according to the manufacturer's guidelines and then was rinsed with water and dried by using air syringe spray. The etched areas were brushed with a bonding agent (Adper Single Bond 2, 3M/ESPE, Minnesota, USA) and light cured for 15 seconds with a light cure unit spectrum 800 (Dentsply/Caulk, Philadelphia, USA).

Figure 3.28: Composite resin (Filtek Z 350 3M/ESPE)

The coronal surfaces of the roots were then covered with cellulose crowns (Frasacao Gmbh, Tettnang, Germany) and filled with composite resin layer by layer, each of 2 mm in depth. Each layer cured for 40 seconds with light cure unit and the built-up cores were 5 mm in height. Once completed, cellulose crowns were removed and cores were ensured to be completely set by light, curing them for 40 seconds, and polished with white stone.

3.4.8 Crown preparation

A chamfer diamond bur with a guiding pin (NTI, Diamond Bur, Kahla, Germany) attached to a high-speed hand-piece, were attached to a paralleling device (Custom Made at the Engineering Department, University of Malaya, Figure 3. 29) was used to provide a standard preparation of the cores.

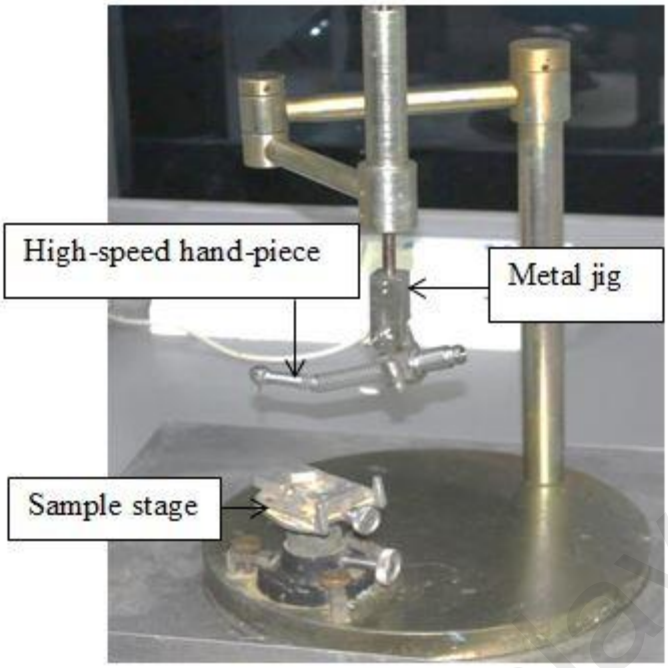


Figure 3.29: High speed hand-piece fixed to the paralleling device

A 2 mm of ferrules were prepared around the cores margins below the CEJ (Figure 3.30) and they were determined by the guiding pin that attached to the chamfer bur. The epoxy resin blocks were then placed in the metal jig with a 45^{o} to the horizontal plane to prepare the bevel flat cut by using the diamond discs fixed to a straight hand-piece which is attached to a surveyor (AF 30 Milling and Surveying Machine, Wallisellen, Switzerland).

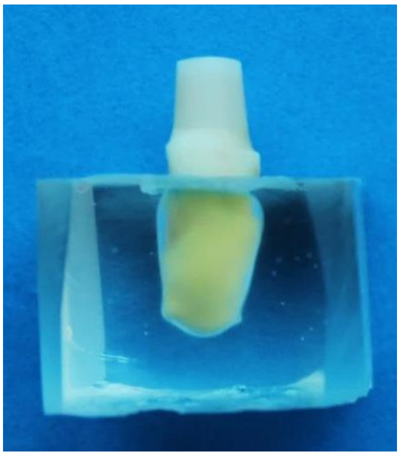


Figure 3.30: Specimen with prepared core mounted with epoxy resin block

3.4.9 Fabrication of the metal coping

All samples were sent to the lab in order to fabricate the metal crown. Wax patterns (Degussa, Hanau, Germany) were applied over the cores and carved to resemble the shape of the cores with 1 mm in thickness in all surfaces, except for the incisal has 2 mm thickness. All wax patterns were then removed from the cores and sprued, invested, melt-down in an oven for 45 minutes at 980 ^{o}C and then casted with Ni-Cr alloy. The inner surfaces of the metal copings were checked for air bubbles and were sandblasted (Figure 3.31).

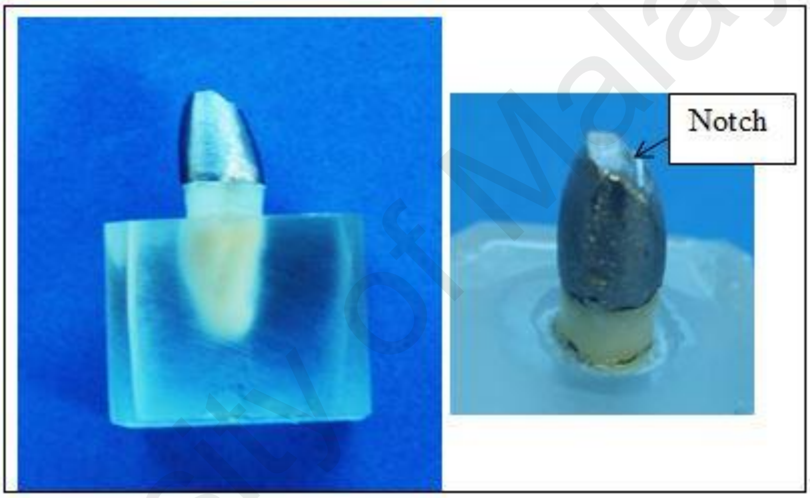


Figure 3.31: Specimen with Ni-Cr coping

A notch was made at the palatal surface each 2 mm below the incisal edge of metal coping (Figure 3.30) to receive the universal testing machine's (Shimadzu, Autograph AG-X, Kyoto, Japan) crosshead pin by using a diamond bur (NTI, diamond bur, Kahla, Germany) attached to a straight hand-piece.

3.4.10 Metal copings cementation

All fabricated metal copings were cemented with zinc phosphate cement (Elite Cement 100, GC Corporation, Tokyo, Japan). The zinc phosphate cement was prepared on a glass slab and mixed for 20 seconds as recommended by the manufacturer's guidelines. The mixed cement was applied to the inner surfaces of the metal copings and then placed over the cores and subjected to a finger pressure for 5 minutes. Cement excesses were removed by using a dental explorer and all samples were placed in a container filled with distilled water and were then stored in an incubator at 37 ^{o}C in 100% humidity for 24 hours.

3.4.11 Thermocycling procedure

All samples were thermocycled group by group in a thermocycling machine (Customised in University of Malaya). Samples were placed in a wire mesh attached to a samples holder and subjected to 500 cycles in a cold bath at 5 ^{o}C and a hot bath at 55 ^{o}C each of 20 seconds (ISO/ 2003). The transferring time between the cold and hot baths was 2 seconds. A thermometer was used to check the temperatures of both cold and hot baths. After thermocycling, all samples were removed to be dried at room temperature for 24 hours.

3.4.12 Evaluation and determination of fracture resistance and failure mode

Each sample was aligned in a special jig fixed to a Shimadzu universal testing machine in order and loaded at a 45^{o} angle plane to the long axis of the sample and at 135^{o} angle plane to the loading crosshead pin, as shown in Figure 3.32 (adopted from Abdulmunem *et al.* (2015)). The crosshead pin with a speed of 0.5 mm/min was applied to the prepared notch of the metal copings until the failure occurred and the fracture values were evaluated in Newton.

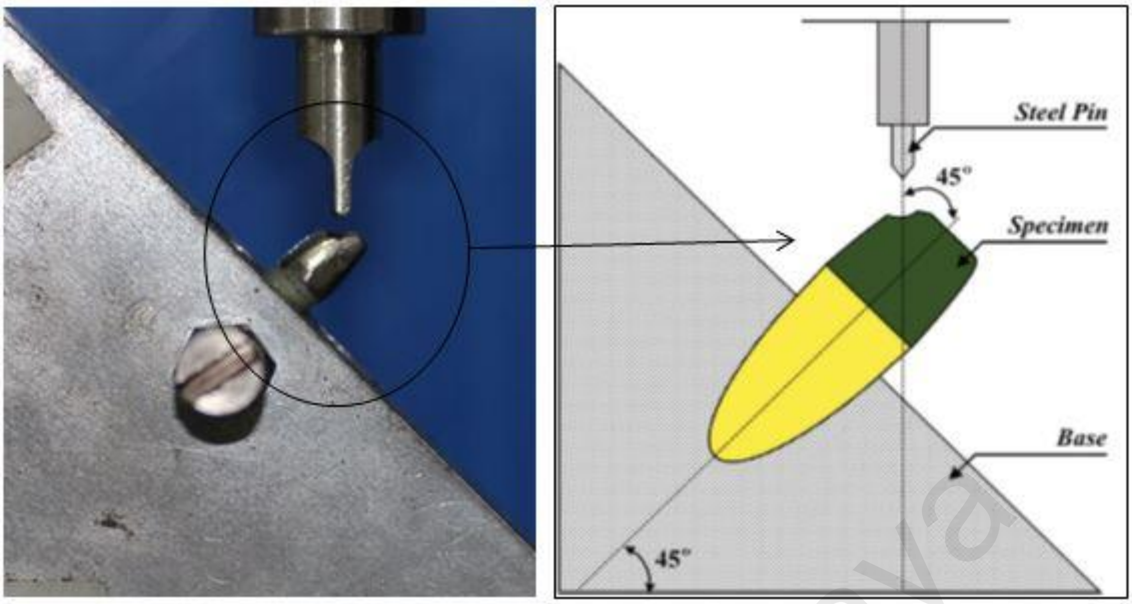


Figure 3.32: Specimen subjected under Shimadzu Universal Testing Machine

Types of the failure mode (restorable and non-restorable) of each sample were investigated visually and then samples were sent for checking under the stereomicroscope (Figure 3.33). The failures were recorded as follows (Chapter 2, Figure 2.4):

- **Restorable:**

Post-core debonding or post-core-tooth complex fractures or cracks of the teeth above the epoxy resin level were considered as restorable failures.

- **Non-restorable:**

Post-core-tooth complex fractures or cracks below the epoxy resin level were considered as non-restorable failures.

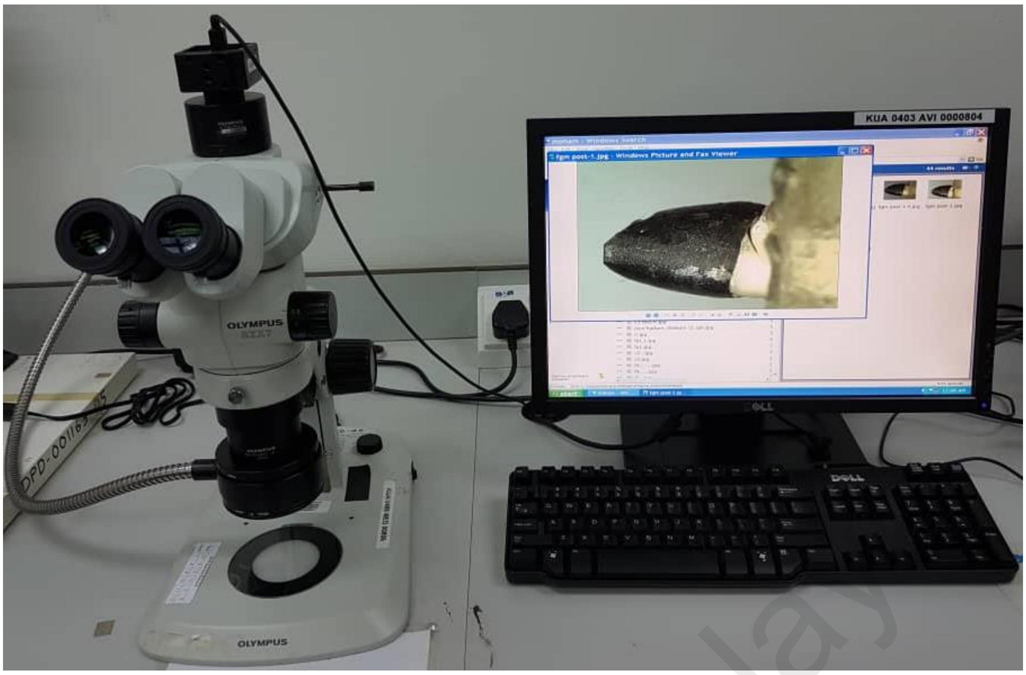


Figure 3.33: Investigation of the failure mode using stereomicroscope

3.5 Finite element analysis

Stress distribution was evaluated using teeth models restored with the functionally graded dental post, titanium, fibre and stainless steel posts by using finite element analysis.

3.5.1 Model geometry

Four models which resemble human maxillary central incisors based on dimensions given in the literature (Zarone *et al.*, 2006; Santos *et al.*, 2009; Abu Kasim *et al.*, 2011) were made by using Solid Works software (Dassault Systèmes SolidWorks Corporation, Massachusetts, USA). Each model has different post types where the first model, established with the functionally graded post (which contains five layers, as shown in Figure 3.34), the second model, established with fibre post, the third model with stainless steel post and the fourth model was with titanium post.

The periodontal ligament (PDL), Cortical cancellous bone, gutta-percha, composite core, dental posts, dentine, zinc phosphate cement, and metal coping were obtained in all the four models (Figure 3.35) with dimensions listed in Table 3.4.

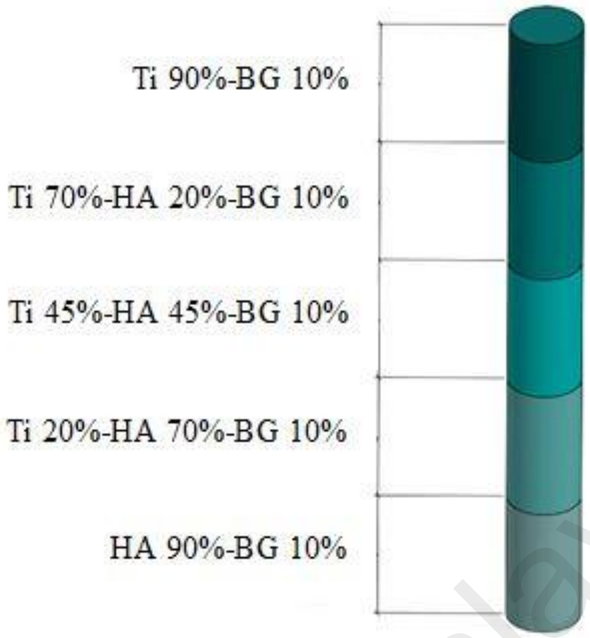


Figure 3.34: The composition of the five layers of the functionally graded dental post

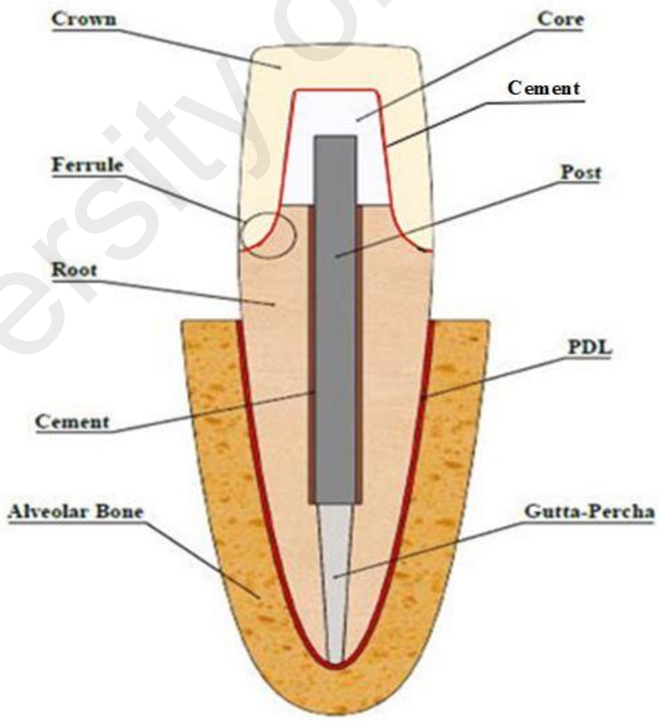


Figure 3.35: Schematic illustration of the geometry used in FEA

Table 3.4: Dimensions of geometric models

Parts		Dimension (mm)
Crown	Height	9
	Thickness at the incisal third	2
	Thickness at the cervical third	1.5
Core	Height	5
	Diameter	4
	Thickness over the post	2
Root	Height	16
	Width	8
	Ferrule height	2
Gutta-percha	Height	5
	Diameter	1.5
Post	Height	14
	Diameter	1.5
Bone	Thickness	0.18
PDL	Thickness	2

3.5.2 Generation of the mesh

The geometry of the finite element model was subdivided into small elements. SolidWorks/Solid Mesh used to provide a free mesh technique in the current study. Because of the complicated geometry of the FE models, tetrahedral elements were used to separate the parts and the convergence was achieved. To analyse the stress, four nodes, first order, linear tetrahedral solid elements were used (C3D4). A tetrahedral mesh of 56129 (Figure 3.36) was used and the error recorded was below 0.1% for two mesh sizes of 56120 and 63806.

Figure 3.36: Tetrahedral mesh of the 3D FE model

The modulus of elasticity and Poisson's ratios of the restorative materials, tooth, and periodontium components were recorded in each component of the four different models and were listed in Table 3.5. All these recorded components were assumed to be homogeneous, isotropic and linearly elastic.

Table 3.5: Modulus of elasticity and Poisson's ratio

Material	Elastic modulus [Mpa]	Poisson's ratio	References
Dentine	18600	0.30	(Ferrari *et al.*, 2000; Dejak *et al.*, 2012)
Crown (Ni-Cr)	209200	0.24	(Toparli, 2003; Diana *et al.*, 2016)
Core (composite resin)	16600	0.24	(Joshi *et al.*, 2001)
Bioactive glass	76740	0.261	(Srivastava *et al.*, 2012b)
Titanium post	116000	0.33	(Toparli, 2003)
Stainless steel post	207000	0.30	(Barjau *et al.*, 2006)
Fibre post	9500	0.34	(Abdulmunem *et al.*, 2016)
Gutta-percha	0.69	0.45	(Holmes *et al.*, 1996)
Zinc phosphate cement	22400	0.35	(Holmes *et al.*, 1996)
PDL	68.9	0.45	(Holmes *et al.*, 1996; Ruse, 2008)
Enamel	84100	0.30	(Ausiello *et al.*, 2011)
Cortical cancellous	13700	0.30	(Holmes *et al.*, 1996)

Loads were applied to the four models in three directions with 100 N as recommended by a previous study (Abu Kasim *et al.*, 2011). These three loads were applied in order to simulate the masticatory force, external traumatic force and the bruxism (Joshi *et al.*, 2001; Genovese *et al.*, 2005). The loads are vertical, oblique and horizontal.

3.5.3 The boundary condition

All nodes of the created four models were fixed at the end part of the maxillary central incisors models. No gap was assumed to occur in the components of the teeth restored with different post types. The nodes and elements number for each part of the models are listed in Table 3.6.

Table 3.6: Number of nodes and elements for each part of the models

Models	No. of nodes	No. of elements
Cement	27565	17715
Bone	61801	36739
Core	28131	18917
Crown	27867	18331
Dentine	29890	19902
Gutta-percha	17586	11730
PDL	24317	12059
Post	27351	17944
Post composite /for one part	26574	18032

3.5.4 Finite element analysis

The stress distributions were calculated by using SolidWorks/surface modeling Version premium 2015, Dassault Systèmes, USA. Von Mises was evaluated within the post, tooth and at the post-core-tooth interfaces areas.

3.6 Data analysis

All data were statistically analysed by using Statistical Package for Social Sciences (SPSS) Version 12.0 (SPSS Inc, Chicago, USA).

All values were analysed by using the One and Two-Way ANOVA, and pair-wise comparisons were done by using Tukey test. Failure modes were analysed by using Chi-square test.

3.7 Chapter summary

This chapter explained the materials and methods that used in this research. It explained the various ratios of bioactive glass materials to different 5 ratios of titanium-hydroxyapatite composites were prepared. Sintering Ti-HA-BG composites in different sintering conditions (air atmosphere and vacuum furnaces) were also listed in this chapter. Ti-HA-BG composites were characterised using XRD, EDX. Physical and chemical test including density, micro-hardness, modulus of elasticity and compression test were explained.

This chapter also focused on fabrication and machining of functionally graded dental posts based on Ti-HA-BG materials. Moreover, restoring the teeth with functionally graded dental, fibre, stainless steel and titanium posts were highlighted. Fracture resistance and failure mode of these teeth were evaluated by using a universal testing machine. Finite element analysis of four models restored with functionally graded dental post, fibre, stainless steel and titanium posts was also explained in this chapter.

CHAPTER 4: RESULTS

4.1 Introductions

There are four sections in this chapter. In Section 4.2 samples were visually assessed for cracks and fractures. Tests in Sections 4.3 were analysed and evaluated by using XRD, EDX and SPSS statistical analysis software. Data in Section 4.4 were evaluated using SPSS statistical analysis software. In Section 4.5, finite element analysis software was used to analyse stress distributions of the four generated models.

4.2 Optimisation of different Ti-HA composites using various ratios of bioactive glass

In Group 1; BG ratio in this group was 5%. Samples in all layers (Figure 4.1a) were crack free and intact after sintering process, except for layer 3 (Figure 4.1b) which showed long crack from top to bottom. As a result it is impossible to merge them in order to produce a functionally graded dental post.

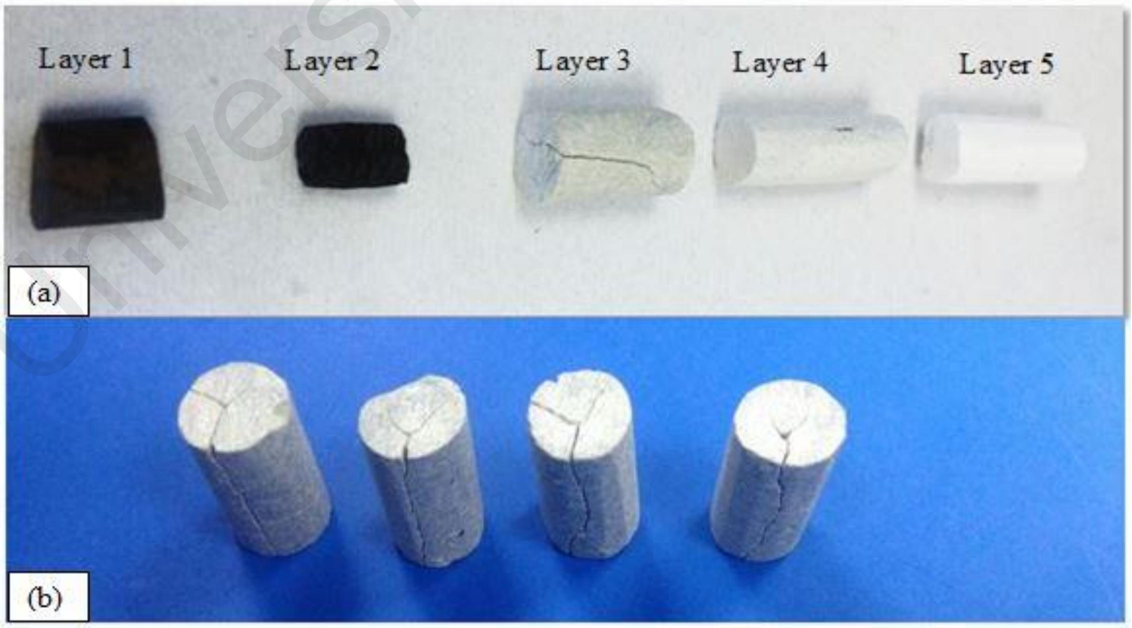


Figure 4.1: (a) Titanium-hydroxyapatite layers with 5% bioactive glass. (b) Fracture line in layer 3

In Group 2; BG ratio in this group was **10%.** Samples in all layers were crack free and intact. Therefore it is possible to merge the layers for further use and to produce a functionally graded dental post (Figure 4.2). XRD analysis (as explained in section 4.3.1) showed that these layers were intact because the amount of Ti oxidation was not significant and HA decomposition was not detected which means the Ti and HA were intact in this group, resulting in crack free layers.

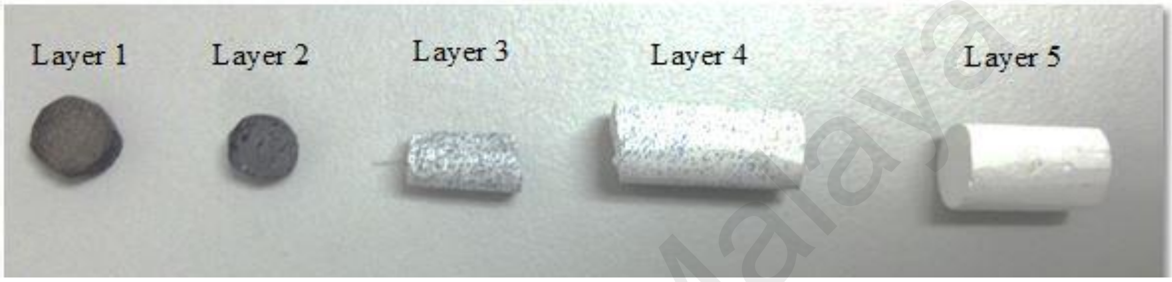


Figure 4.2: Titanium-hydroxyapatite layers with 10% bioactive glass

Group 3; BG ratio in this group was 15%. Layer 3, layer 4 and layer 5 (Figure 4.3) showed no cracks and fractures, while layer 1 and layer 2 were totally fractured, spoiled and not useful to be used in further applications and research (Figure 4.3 and Figure 4.4). These fractures were mostly due to oxidation of Ti and decomposition of HA.

Figure 4.3: Titanium-hydroxyapatite layers with 15% bioactive glass

Figure 4.4: Layer 1 and layer 2 with 15% bioactive glass

4.3 Characterization and optimisation of the Ti-HA-BG composites to fabricate functionally graded dental posts

4.3.1 XRD analysis

In this section of the study, layers (samples) were divided into 2 groups. Layers sintered in air atmosphere furnace were categorized as Group 1 (G 1), while layers sintered in vacuum furnace were categorized as Group 2 (G 2). The x-ray diffraction (XRD) patterns for Group 1 are shown in Figure 4.5. XRD pattern of layer 1 showed that the majority of titanium has retained stability and matched JCPDS card no. 44-1294. While some amounts of titanium oxide in the form of Ti_2O (JCPDS card no. 01-073-1116) and TiO (JCPDS card no. 65-2900) were also recorded. In layer 2, layer 3 and layer 4, Ti phase was gradually decreased while HA (JCPDS card no. 009-0432) phase increased. However, there were no interactions between Ti and HA compounds as Ti_5P_3 and $CaTiO_3$ were not detected in all layers. In layer 5, the crystalline phase mostly belonged to HA.

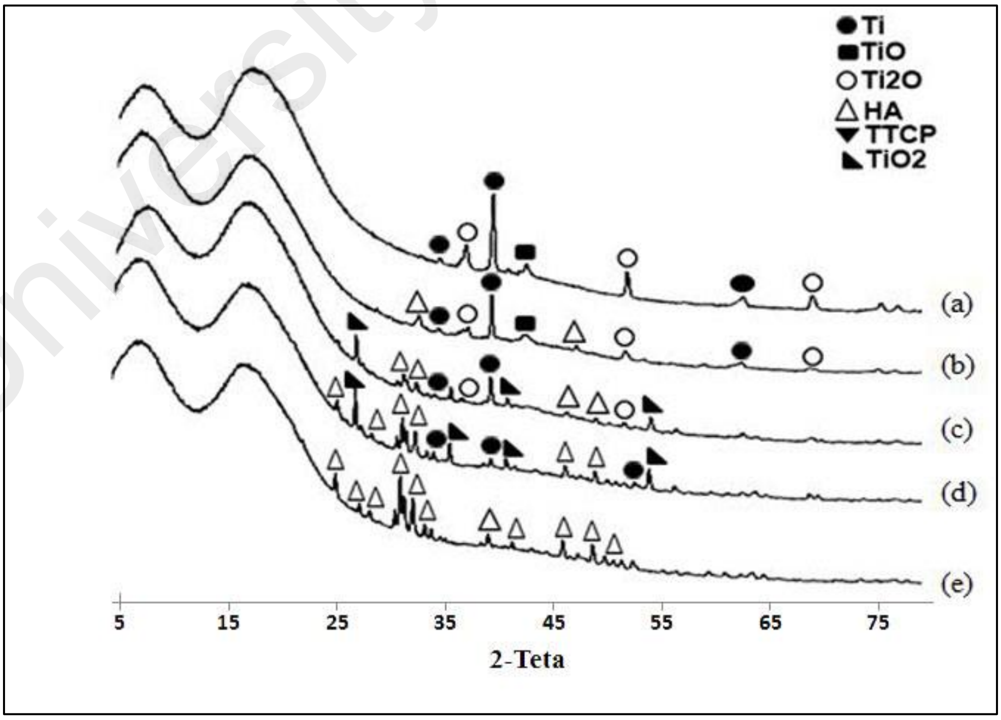


Figure 4.5: XRD of 5 layers sintered with air atmosphere furnace (G 1). (a) Layer 1. (b) Layer 2. (c) Layer 3. (d) Layer 4. (e) Layer 5

In Group 2 (G 2), XRD patterns are shown in Figure 4.6. Samples in this group were sintered in a vacuum furnace. Layer 1 showed that the major phases belong to Ti and also there was a presence of Ti_2O and TiO. In layer 2, layer 3 and layer 4; Ti phases was gradually decreased while HA phases were increased. Also, Ti_2O, TiO_2, TTCP (JCPDS card no. 00-025-1137), and TCP (JCPDS card no. 00-009-0348) were present in these layers.

In layer 5 most of the major peaks belong to HA. However, HA has also decomposed to TTCP and TCP which were obvious in the minor phases of this layer.

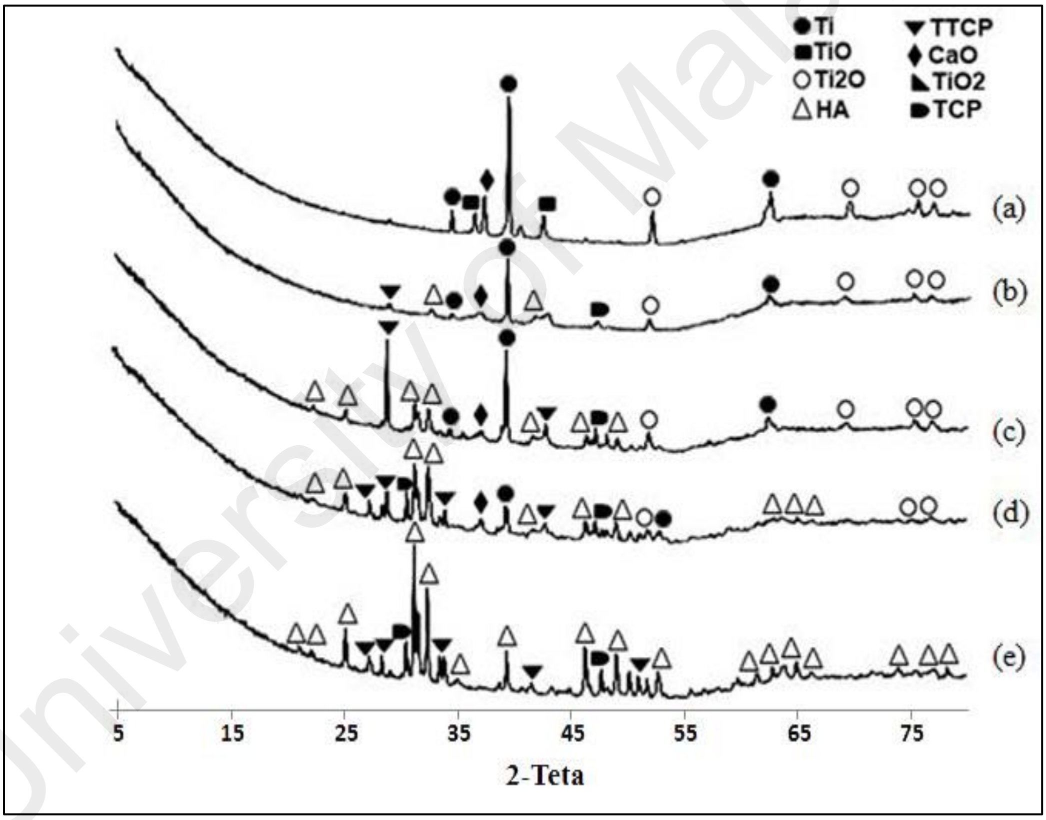


Figure 4.6: XRD of 5 layers sintered with vacuum furnace (G 2). (a) Layer 1. (b) Layer 2. (c) Layer 3. (d) Layer 4. (e) Layer 5

4.3.2 EDX analysis

Table 4.1 shows the gradual change of the material weight ratio from layer 1 to layer 5 in Group 1 (G 1). The chemical compositions of the layers were analysed by using

EDX (Figure 4.7). Ti occurred with a high percentage in layer 1 and gradually decreased until layer 4 and disappeared in layer 5.

P in combination with Ca and O which may indicate HA, was not found in layer 1, and occurred in layer 2, with low percentages and increased gradually until layer 5 which contains HA and BG only. BG was indicated by recording its main elements which are Si, Mg, Ca, Na and C.

Table 4.1: Elements in the five layers sintered in air atmosphere furnace (G 1)

Element	Weight Ratio (%)				
	Layer 1	**Layer 2**	**Layer 3**	**Layer 4**	**Layer 5**
Na	-	-	-	0.53	-
Mg	-	0.44	-	0.87	0.50
C	7.36	14.39	10.33	8.08	6.67
O	41.66	26.36	42.42	45.48	43.14
Si	1.02	1.54	1.01	1.09	1.05
P	-	3.34	4.86	11.00	15.90
Ca	11.20	10.77	26.94	25.39	32.74
Ti	38.77	43.16	14.44	8.42	-
Total	100.00	100.00	100.00	100.00	100.00

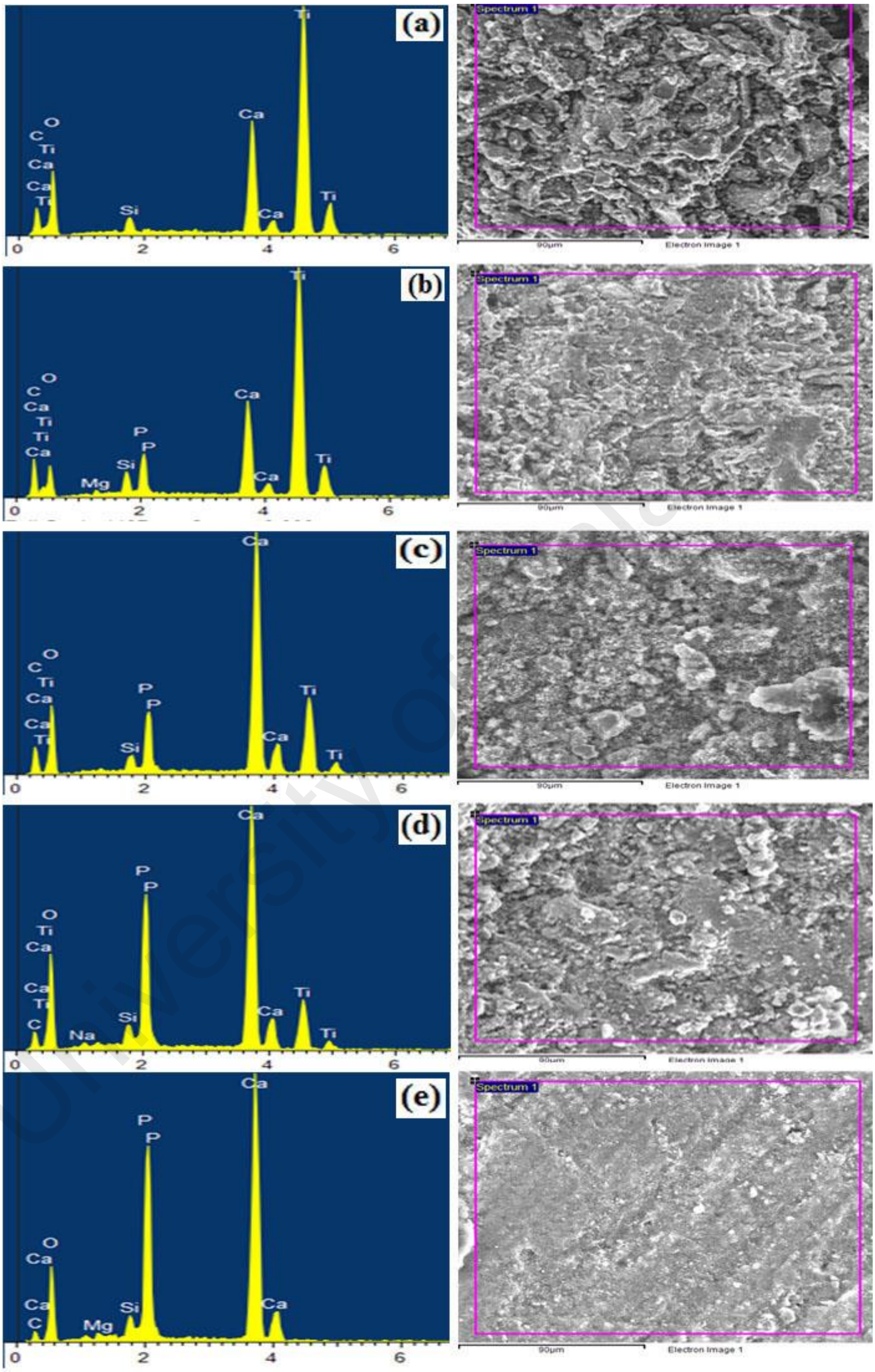


Figure 4.7: EDX analysis of layers sintered in air atmosphere furnace (G 1). (a) Layer 1. (b) Layer 2. (c) Layer 3. (d) Layer 4. (e) Layer 5

In layers sintered with vacuum furnace (G 2) (Figure 4.8), layer 1 showed the highest percentage of Ti, at which it decreased gradually layer by layer until totally disappearing in layer 5. HA was indicated by recording its main elements (CA, O, P). HA disappeared in layer 1 as no HA was used in that layer and occurred in layer 2 at which it started to increase until it reached the highest ratio in layer 5. BG was also recorded by checking the presence of Si, Mg, Ca, Na and C. Table 4.2 shows the material weight ratio of the layers.

Table 4.2: Materials weight ratios in the five layers sintered in vacuum furnace (G 2)

Element	**Weight Ratio (%)**				
	Layer 1	**Layer 2**	**Layer 3**	**Layer 4**	**Layer 5**
Na	-	-	-	0.42	0.68
O	38.04	31.60	40.04	41.74	45.10
C	7.48	9.68	17.91	6.20	5.69
Mg	-	0.45	0.52	-	0.46
Si	0.92	3.14	2.31	1.81	2.46
P	-	5.08	8.05	11.54	15.41
Ca	23.54	17.15	20.70	30.80	30.21
Ti	30.02	32.92	10.48	7.48	-
Total	100.00	100.00	100.00	100.00	100.00

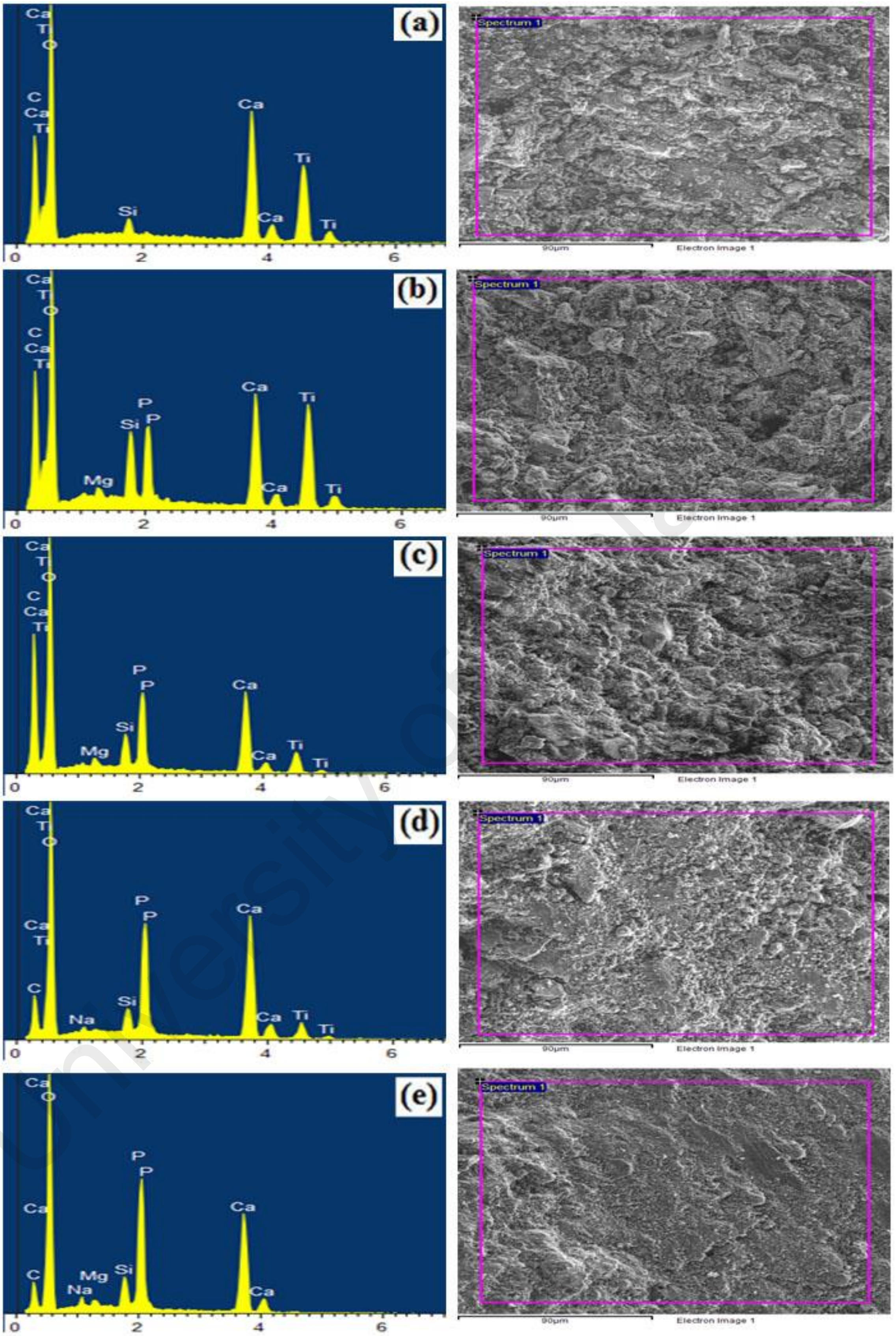


Figure 4.8: EDX analysis of layers sintered in vacuum furnace (G 2). (a) Layer 1. (b) Layer 2. (c) Layer 3. (d) Layer 4. (e) Layer 5

4.3.3 Density

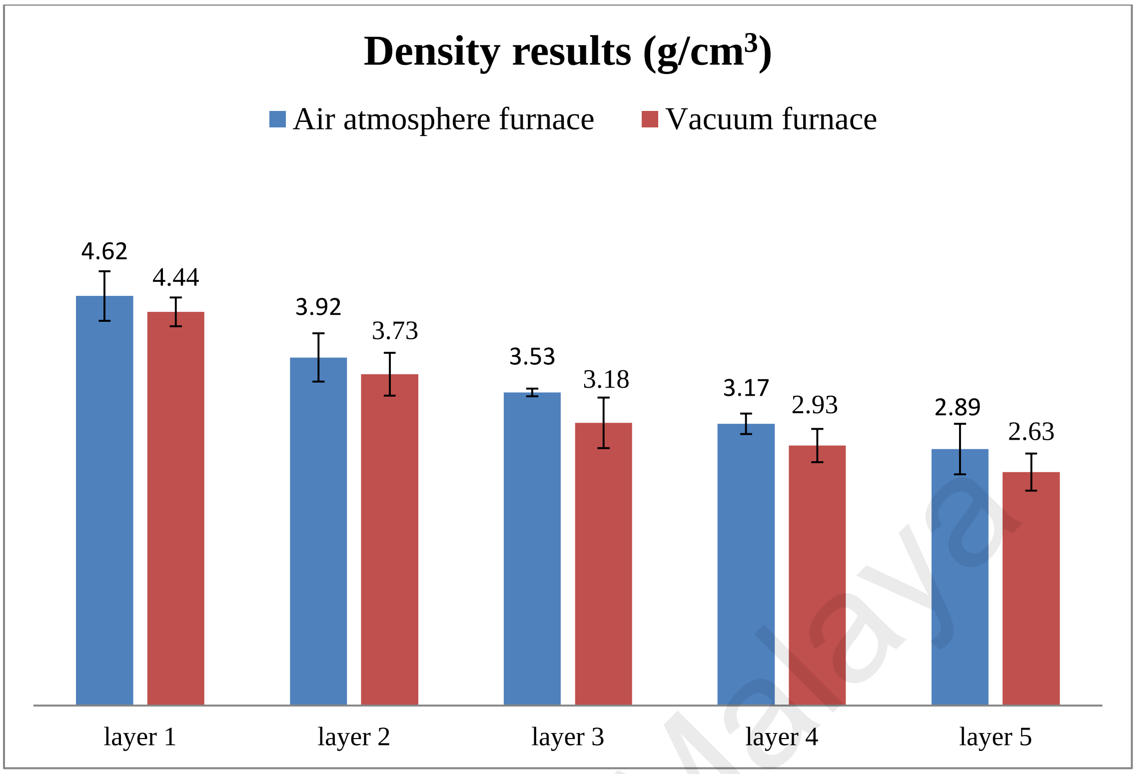
4.3.3.1 Descriptive statistics and pair-wise comparisons

Pair-wise comparisons between air atmosphere (G 1) and vacuum furnaces (G 2) were verified by using Two-way ANOVA. Layers in air atmosphere furnace groups showed a significantly higher density than those in vacuum furnace group (*p*= 0.000), as shown in Table 4.3.

In both groups, layers with high Ti ratio (layer 1 and layer 2) showed higher density than those with low Ti ratios (layer 3, 4 and 5). Layer 1 showed the highest density followed by layer 2, layer 3, layer 4 and layer 5 respectively. (Means and standard deviation are shown in Figure 4.9 and Appendix A).

Table 4.3: Pair-wise comparison of density between air atmosphere (G 1) and vacuum furnaces (G 2) groups

Furnace Type	**Furnace Type**	**Mean Difference (I-J)**	**Std. Error**	***P* value**	**95% Confidence Interval for Difference(a)**	
					Lower Bound	Upper Bound
Air atmosphere F.	Vacuum F.	0.244	0.044	0.000	0.155	0.332
Vacuum F.	Air atmosphere F.	-0.244	0.044	0.000	-0.332	-0.155



* Values represent the means, and the bar lines represent the standard deviations

Figure 4.9: Density means and standard deviation of the layers sintered in air atmosphere (G 1) and vacuum furnaces (G 2)

4.3.3.2 Multiple comparisons using One-way ANOVA test

One-way ANOVA was used to compare the layers in each main group by using Tukey test, as shown in Table 4.3. In air atmosphere furnace (G 1) group, there was a significant difference between all layers (Table 4.4). While in vacuum furnace (G 2) group, there was a significant difference in density between all layers except between layer 3 and layer 4 (p=0.094), as shown in Table 4.5.

Table 4.4: Multiple comparison of the density between the layers within air atmosphere furnace group (G 1)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	0.695	0.411	0.979	0.000
	Layer 3	1.088	0.804	1.372	0.000
	Layer 4	1.442	1.158	1.726	0.000
	Layer 5	1.728	1.444	2.012	0.000
Layer 2	Layer 3	0.392	0.108	0.677	0.003
	Layer 4	0.746	0.462	1.031	0.000
	Layer 5	1.032	0.748	1.316	0.000
Layer 3	Layer 4	0.354	0.069	0.638	0.008
	Layer 5	0.639	0.355	0.923	0.000
Layer 4	Layer 5	0.285	0.001	0.569	0.048

Table 4.5: Multiple comparison of the density between the layers within vacuum furnace group (G 2)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	0.703	0.422	0.984	0.000
	Layer 3	1.252	0.971	1.533	0.000
	Layer 4	1.506	1.225	1.787	0.000
	Layer 5	1.808	1.527	2.089	0.000
Layer 2	Layer 3	0.549	0.268	0.830	0.003
	Layer 4	0.803	0.522	1.084	0.000
	Layer 5	1.105	0.824	1.386	0.000
Layer 3	Layer 4	0.254	-0.027	0.535	0.094
	Layer 5	0.555	0.274	0.836	0.000
Layer 4	Layer 5	0.301	0.020	0.582	0.030

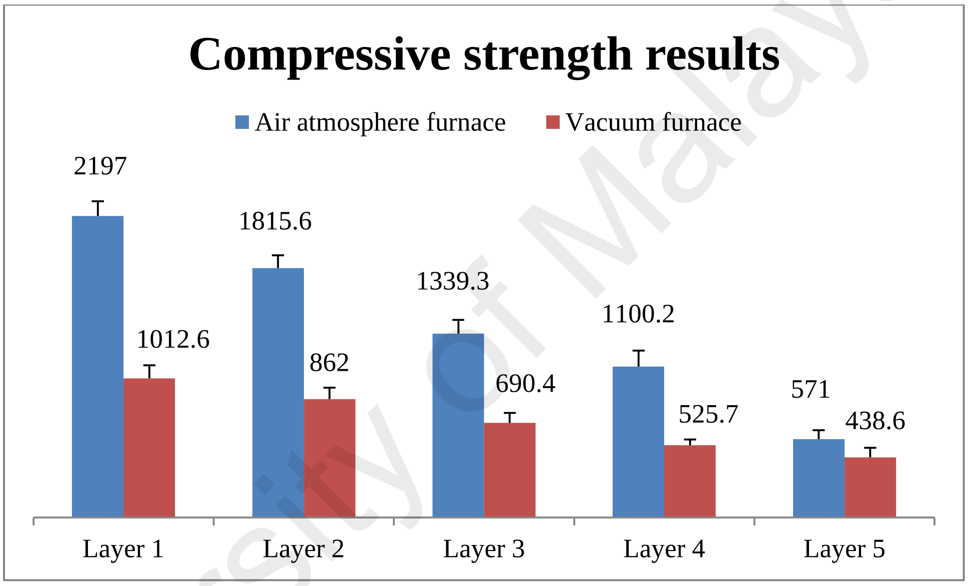
4.3.4 Compression test results

4.3.4.1 Descriptive statistics and pair-wise comparisons

Two-way ANOVA was used to check the pair-wise comparisons between air atmosphere (G 1) and vacuum furnaces (G 2) groups. The results showed that samples sintered in air atmosphere furnace (G 1) have higher compression strength than those sintered in a vacuum furnace (G 2) (p= 0.000), as listed in Table 4.6. Means and standard deviations of each layer in each furnace (main) group are illustrated in Figure 4.10 and Appendix B. Layers with high Ti (layer 1 and layer 2) ratios showed higher compression strength compared to those with low Ti ratios (layer 3, layer 4 and layer 5).

Table 4.6: Pair-wise comparison of compression strength between air atmosphere (G 1) and vacuum furnaces (G 2) groups

Furnace Type	Furnace Type	Mean Difference (I-J)	Std. Error	*P* value	95% Confidence Interval for Difference	
					Lower Bound	Upper Bound
Air atmosphere F.	Vacuum F.	698.760	17.389	0.000	664.215	733.305
Vacuum F.	Air atmosphere F.	-698.760	17.389	0.000	-733.305	-664.215



* Values represent the means, and the bar lines represent the standard deviations

Figure 4.10: Means and standard deviation for compression test of groups sintered in air atmosphere (G 1) and vacuum furnaces (G 2)

4.3.4.2 Multiple comparisons using One-way ANOVA test

One-way ANOVA was also performed to check the multiple comparisons between the layers (subgroups). There was a significant difference between layers in groups G 1 (p=0.000, Table 4.7) and G 2 (p= 0.000, Table 4.8). In G1, layer 1 showed higher compression strength compared to layer 2, 3, 4 and 5 (p= 0.000). Layer 2 showed significant difference with layer 3, layer 4 and layer 5 (p= 0.000), while layer 3 showed higher compression strength compared to layer 4 and layer 5 (p= 0.000). In G2, there was a significant difference between all layers except layer 4 and layer 5 (p=0.082).

Table 4.7: Multiple comparison of the compression strength between the layers within air atmosphere furnace group (G 1)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	381.40	256.65	506.14	0.000
	Layer 3	857.70	732.95	982.44	0.000
	Layer 4	1096.80	972.05	1221.54	0.000
	Layer 5	1626.00	1501.25	1750.74	0.000
Layer 2	Layer 3	476.30	351.55	601.04	0.000
	Layer 4	715.40	590.65	840.14	0.000
	Layer 5	1244.60	1119.85	1369.34	0.000
Layer 3	Layer 4	239.10	114.35	363.84	0.000
	Layer 5	768.30	643.55	893.04	0.000
Layer 4	Layer 5	529.20	404.45	653.94	0.000

Table 4.8: Multiple comparison of the compression strength between the layers within vacuum furnace group (G 2)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	150.60	56.524	244.675	0.000
	Layer 3	322.20	228.124	416.275	0.000
	Layer 4	486.90	392.824	580.975	0.000
	Layer 5	574.00	479.924	668.075	0.000
Layer 2	Layer 3	171.60	0.108	0.677	0.000
	Layer 4	336.30	0.462	1.031	0.000
	Layer 5	423.40	0.748	1.316	0.000
Layer 3	Layer 4	164.70	0.069	0.638	0.000
	Layer 5	251.80	0.355	0.923	0.000
Layer 4	Layer 5	87.10	0.001	0.569	0.082

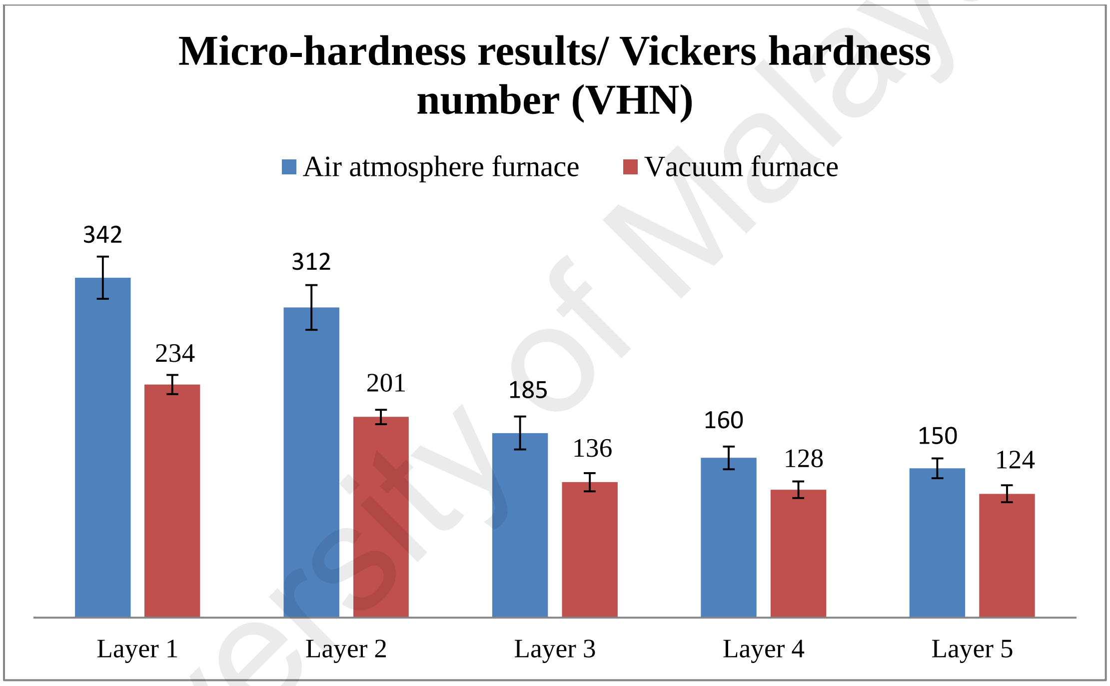
4.3.5 Micro-hardness test results

4.3.5.1 Descriptive statistics and pair-wise comparisons

Two-way ANOVA was performed to check the significant difference between the groups. Layers sintered in air atmosphere furnace (G 1) showed higher micro-hardness than those sintered in a vacuum furnace (G 2) (p= 0.000) (Table 4.9). Means and standard deviation illustrated in Figure 4.11 and Appendix C.

Table 4.9: Pair-wise comparison of micro-hardness between air atmosphere (G 1) and vacuum furnaces (G 2) groups

Furnace Type	Furnace Type	Mean Difference (I-J)	Std. Error	*P* value	95% Confidence Interval for Difference(a)	
					Lower Bound	Upper Bound
Air atmosphere F.	Vacuum F.	65.040	2.705	0.000	59.666	70.414
Vacuum F.	Air atmosphere F.	-65.040	2.705	0.000	-70.414	-59.666



* Values represent the means, and the bar lines represent the standard deviations

Figure 4.11: Means and standard deviation for micro-hardness of groups sintered in air atmosphere (G 1) and vacuum furnace (G 2)

4.3.5.2 Multiple comparisons using One-way ANOVA test

One-way ANOVA was also applied to check the multiple comparisons between the layers in air atmosphere (G 1) and vacuum furnaces (G 2) groups. In air atmosphere furnace group (G 1) (Table 4.10), layer 1 showed significant higher micro-hardness compared to layer 2 (p= 0.003), 3, 4 and 5 (p= 0.000). While layer 2 showed higher micro-hardness as compared to layer 3, layer 4 and layer 5 (p= 0.000). Layer 3 was significant with layer 4 (*p*=0.016) and 5 (*p*= 0.000), and there was no significant difference between layer 4 and layer 5 (*p*=0.645).

Whereas, in vacuum furnace group (G 2) (Table 4.11), layer 1 showed a significant difference with layer 2, layer 3, layer 4 and layer 5 (*p*= 0.000). Layer 2 showed significant difference with layer 1, layer 3, layer 4 and layer 5 (*p*= 0.000). Layer 3 was significantly different as compared to layer 5 (*p*= 0.032), while there was no significance between layer 4 and layer 5 (*p*=0.839).

Table 4.10: Multiple comparison of the micro-hardness between the layers within air atmosphere furnace group (G 1)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	29.90	8.229	51.57	0.003
	Layer 3	156.30	134.62	177.97	0.000
	Layer 4	181.30	159.62	2.2.97	0.000
	Layer 5	191.80	170.12	213.47	0.000
Layer 2	Layer 3	126.40	104.72	148.07	0.000
	Layer 4	151.40	129.72	173.07	0.000
	Layer 5	161.90	140.22	183.57	0.000
Layer 3	Layer 4	25.00	3.329	46.67	0.016
	Layer 5	35.50	13.829	57.170	0.000
Layer 4	Layer 5	10.50	-11.170	32.170	0.645

Table 4.11: Multiple comparison of the micro-hardness between the layers within vacuum furnace group (G 2)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval Lower bound /N	95% Confidence Interval Upper bound/N	*P* value
Layer 1	Layer 2	32.50	21.49	43.50	0.000
	Layer 3	98.20	87.19	109.20	0.000
	Layer 4	105.90	94.89	116.90	0.000
	Layer 5	109.90	98.89	120.90	0.000
Layer 2	Layer 3	65.70	54.69	76.70	0.000
	Layer 4	73.40	62.39	84.40	0.000
	Layer 5	77.40	66.39	88.40	0.000
Layer 3	Layer 4	7.70	-3.30	18.70	0.289
	Layer 5	11.70	13.829	22.70	0.032
Layer 4	Layer 5	4.00	-11.170	15.00	0.839

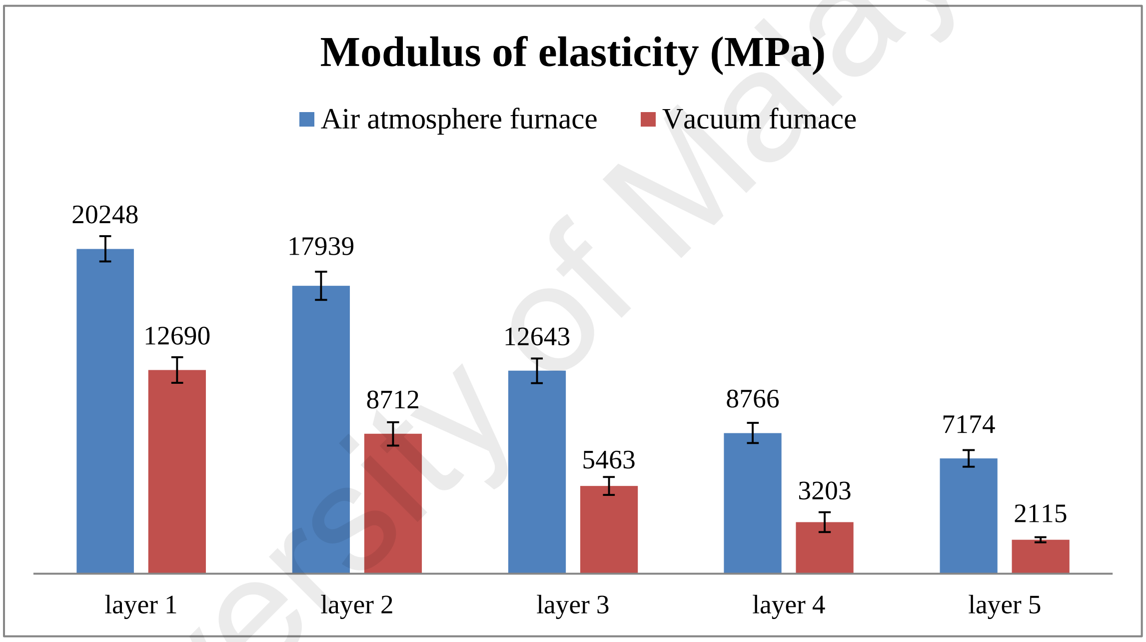
4.3.6 Modulus of elasticity

4.3.6.1 Descriptive statistics and pair-wise comparisons

Two-way ANOVA was performed and pair-wise comparison indicated that layers in air atmosphere furnace group (G 1) have a significantly higher modulus of elasticity than those in a vacuum furnace (G 2) (Table 4.12). Modulus of elasticity's means and standard deviation are illustrated in Figure 4.12 and Appendix D. In both Group 1 and Group 2, modulus of elasticity values were higher in layers with high Ti ratios compared to those with no or low Ti ratios. Layer 1 showed the highest modulus of elasticity followed by layer 2, layer 3, layer 4 and layer 5, respectively.

Table 4.12: Pair-wise comparison of modulus of elasticity between air atmosphere (G 1) and vacuum furnaces (G 2) groups

Furnace Type	Furnace Type	Mean Difference (I-J)	Std. Error	*P* value	95% Confidence Interval for Difference (a)	
					Lower Bound	Upper Bound
Air atmosphere F.	Vacuum F.	6925.200	133.899	0.000	6659.186	7191.214
Vacuum F.	Air atmosphere F.	-6925.200	133.899	0.000	-7191.214	-6659.186



* Values represent the means, and the bar lines represent the standard deviations

Figure 4.12: Modulus of elasticity means and standard deviations of the layers sintered in air atmosphere (G 1) and vacuum furnace (G 2)

4.3.6.2 Multiple comparisons using One-way ANOVA test

One-way ANOVA was also performed to check the multiple comparisons between the layers in each group by using Tukey test. In the air atmosphere furnace group (G 1), there was a significant difference between all layers. In the vacuum furnace group (G 2), there was also a significant difference between all layers. Multiple comparisons of the modulus of elasticity of layers in the air atmosphere furnace group are listed in Table 4.13, while layers in vacuum furnace group are listed in Table 4.14.

Table 4.13: Multiple comparison of the modulus of elasticity between the layers within air atmosphere furnace group (G 1)

Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	2309.40	1383.50	3235.29	0.000
	Layer 3	7604.60	6678.70	8530.49	0.000
	Layer 4	11481.50	10555.60	12407.39	0.000
	Layer 5	13073.80	12147.90	13999.69	0.000
Layer 2	Layer 3	5295.20	4369.30	6221.09	0.000
	Layer 4	9172.10	8246.20	10097.99	0.000
	Layer 5	10764.40	9838.50	11690.29	0.000
Layer 3	Layer 4	3876.90	2951.00	4802.79	0.000
	Layer 5	5469.20	4543.30	6395.09	0.000
Layer 4	Layer 5	1592.30	666.40	2518.19	0.000

Table 4.14: Multiple comparison of modulus of elasticity between the layers within vacuum furnace group (G 2)

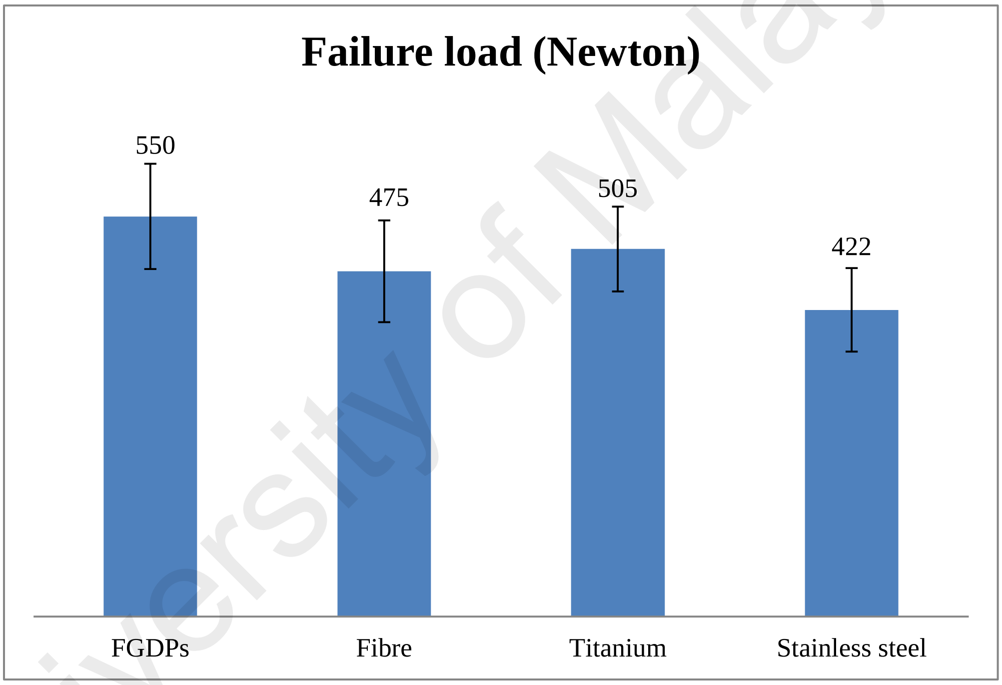
Number of layer		Mean difference (I-J)/N	95% Confidence Interval		*P* value
			Lower bound /N	Upper bound/N	
Layer 1	Layer 2	3939.70	3171.41	4707.98	0.000
	Layer 3	7188.10	6419.81	7956.38	0.000
	Layer 4	9448.10	8679.81	10216.38	0.000
	Layer 5	10536.40	9768.11	11304.68	0.000
Layer 2	Layer 3	3248	2480.11	4016.68	0.000
	Layer 4	5508.40	4740.11	6276.68	0.000
	Layer 5	6596.70	5828.41	7364.98	0.000
Layer 3	Layer 4	2260.00	1491.71	3028.28	0.289
	Layer 5	3348.30	2580,01	4116.58	0.032
Layer 4	Layer 5	1088.30	320.01	1856.58	0.002

4.4 Evaluating the fracture resistance and failure mode of the endodontically treated teeth restored with FGDPs and other post types

4.4.1 Evaluating the fracture resistance

4.4.1.1 Descriptive statistics

Mean and standard deviation of the dental post groups are illustrated in Figure 4.13. The highest mean value was recorded for FGDPs groups (mean= 550) followed by titanium group (mean= 505), fibre group (Mean= 475) and the lowest value was for stainless steel group (mean= 422).



* Values represent the means, and the bar lines represent the standard deviations

Figure 4.13: Means and standard deviations of fracture resistance the dental post groups

4.4.1.2 Multiple comparisons using ANOVA test

One-way ANOVA was used to determine the significant difference between the dental post groups by performing post-hoc (Tukey test) for multiple-comparisons (Table 4.15).

There was a significant difference between the dental posts groups; teeth in the FGDPs group showed a significantly higher fracture resistance than those in the stainless steel dental post group (*p*= 0.000). the titanium group also showed a higher fracture resistance than the stainless steel group (*p*= 0.032). There was no significant difference between the FGDPs, titanium and fibre groups.

Table 4.15: Multiple-comparisons of the fracture resistance between the dental post groups

Type of post (I)	**Type of post (J)**	**Mean Difference (I-J)**	**Std. Error**	***P* value**	**95% Confidence Interval**	
					Lower Bound	Upper Bound
FGDPs	Titanium	44.70300	29.0237	0.425	-33.4633	122.8693
	Fibre	75.40300	29.0237	0.062	-2.7633	153.5693
	Stainless steel	128.40300	29.0237	0.000	50.2367	206.5693
Fibre	Titanium	-30.70000	29.0232	0.717	-108.8663	47.4663
	Stainless steel	53.00000	29.0232	0.278	-25.1663	131.1663
Titanium	Stainless steel	83.70000	29.0232	0.032	5.5337	161.8663

4.4.2 Evaluating the failure mode

4.4.2.1 Chi-square test

Chi-square test was performed to investigate the effect of dental post types on the failure mode.

FGDPs group showed higher restorable failures (10 restorable failures) than other groups. After FGDPs, the fibre group was recorded with seven restorable failures and three non-restorable failures only. The titanium group showed five restorable failures and five non-restorable failures. The worst failure modes value was recorded for stainless steel group where teeth in this group failed with four restorable failure and six non-restorable failures (Table 4.16).

Table 4.16: Number and percentages of failure modes of the dental post groups

Type of dental post	n	Failure mode	
		Restorable	Non-restorable
FGDPs	10	10 (100%)	0 (0%)
Fibre	10	7 (70%)	3 (30%)
Titanium	10	5 (50%)	5 (50%)
Stainless steel	10	4 (40%)	6 (60%)

4.4.2.2 Evaluating the failure mode using the stereomicroscope

The types of failure modes for all teeth in each dental post groups were checked by using stereomicroscope, as shown in Figure 4.14.

Figure 4.14a shows the restorable failure pattern of the tooth in the FGDPs group, where the fracture line is 2 mm below the CEJ and above the epoxy resin block line. While Figure 4.14b shows the restorable failure that occurred in the fibre group. The fracture also started obliquely and ended with a horizontal line of 2 mm below CEJ and above the epoxy resin block line.

Non-restorable failures are illustrated in Figure 4.14c for the titanium group, the fractures and cracks extended below the epoxy resin block line to the middle third of the root. Figure 4.14d shows the non-restorable failure mode of the stainless steel group and the fracture line started obliquely and extended below the epoxy resin block line.

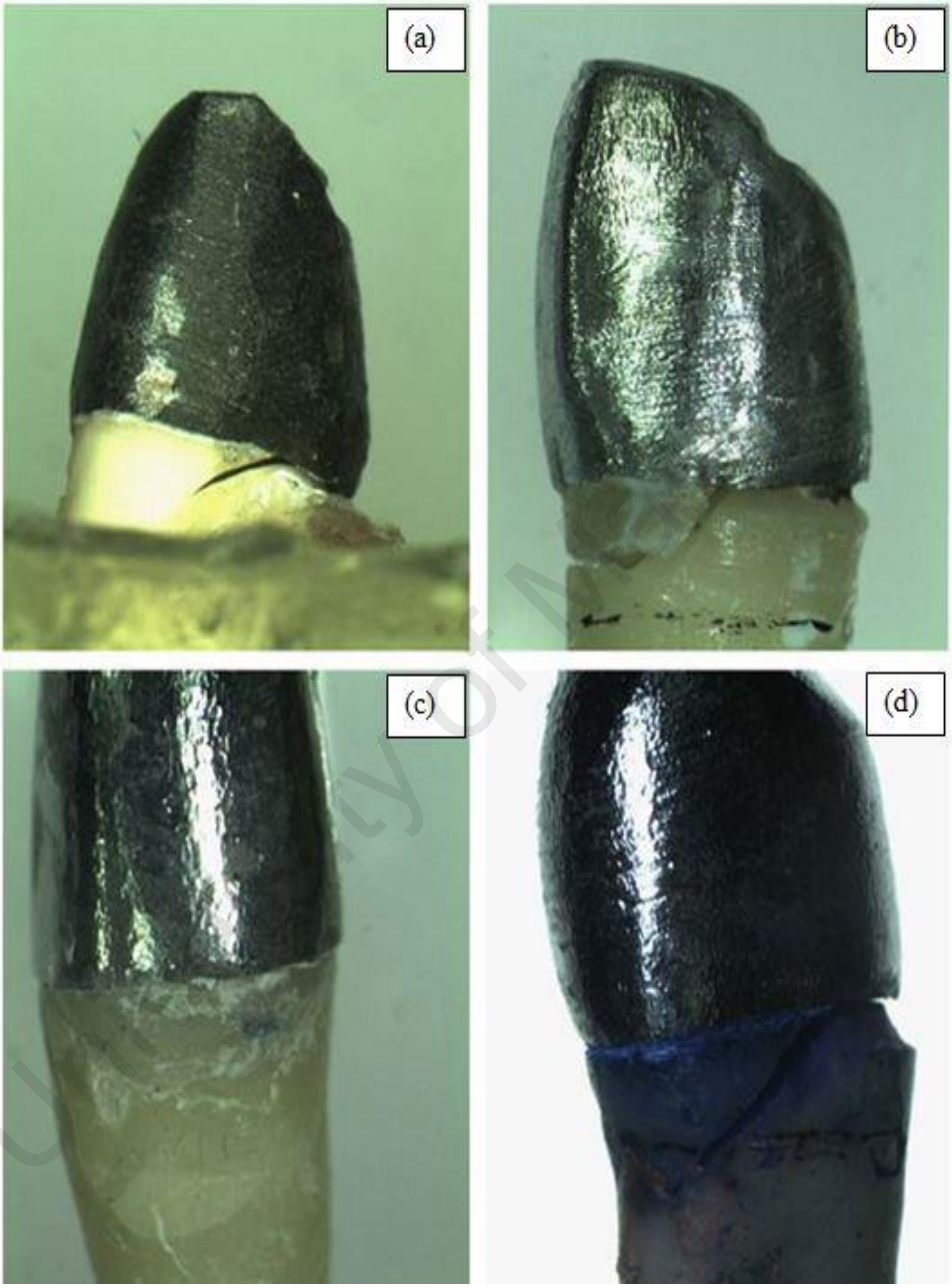


Figure 4.14: (a) Restorable failure of the FGDPs group; (b) Restorable failure of the fibre group; (c) Non-restorable failure of the titanium group; (d) Non-restorable failure of the stainless steel group

4.5 Finite element analysis

Stress distributions under three different load directions were evaluated for different tooth models with FGDP, fibre, titanium or stainless steel dental posts using finite element analysis.

4.5.1 Stress distribution under vertical load

In terms of vertical load direction (Figure 4.15), Von Mises showed that the maximum stress was higher in fibre post model compared to other models. Most of the stresses amongst all models were concentrated in the core-crown interface in the coronal third of the crown except in the fibre model which showed maximum stresses in the coronal and cervical third of the crowns, as well as at the cervical third of the root.

Stainless steel and titanium models, in addition to the core-crown interface, also showed maximum stresses at the apical third of the post-root interfaces, which may result in non-restorable failures. In terms of stress distribution along the post in the stainless steel and titanium models, there was relatively little stress at the coronal and middle third of the posts and the stress was maximal at the apical third of the posts.

In the FGDP model, the stress was distributed at the root cervical third much less than that in the other models and a little stress was distributed at the apical third of the root. Worthy to mention, a minimum stress was noticed in the apical third along the FGDP, while no stress was noticed at the coronal and middle third of the post.

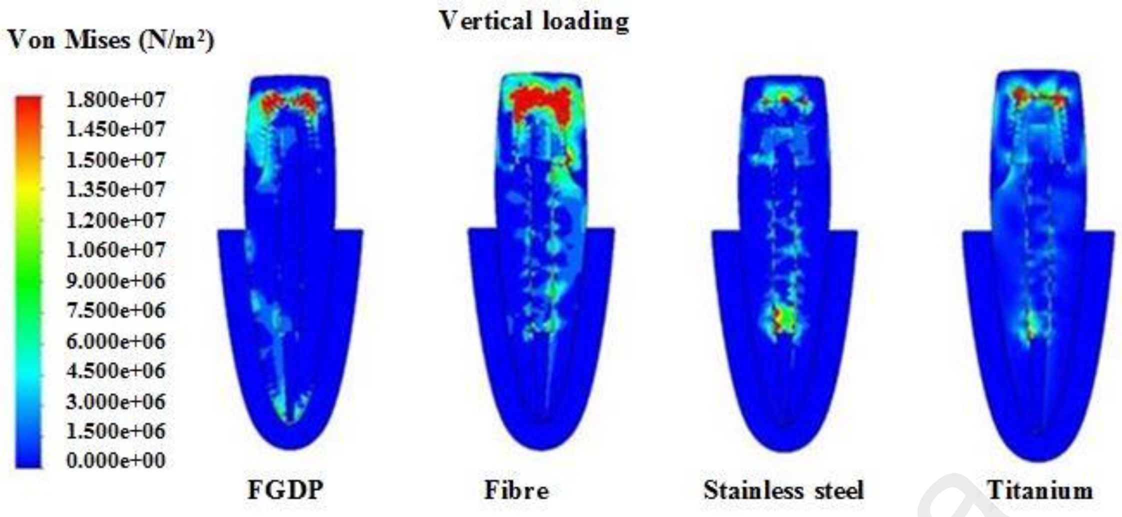


Figure 4.15: Stress distribution under vertical loading for different tooth models

4.5.2 Stress distribution under oblique load

Under oblique load direction (Figure 4.16), the fibre model showed a higher maximum stress compared to other models at the core-crown interfaces at the coronal third of the crowns. Less stress was distributed to the cervical and middle third of the root, followed by the titanium model, which showed higher stress concentrations than FGDP and stainless steel models.

In the titanium model, maximum stress was concentrated at the core-crown interfaces at the coronal third of the crown and less stress was distributed to the cervical and middle third of the root. Along the titanium post, less stress was noticed at the coronal third of the post and increased at the apical third of the post.

Maximum stress distribution was also shown in stainless steel model at the core-crown interfaces and disappeared at the middle and cervical third of the crown. Maximum stress along the stainless steel post disappeared at the coronal and middle third of the post and occurred at the apical third of the post.

FGDP showed high-stress concentrations at the core-crown interface and it showed a uniform stress distribution to the root structure and a very little stress distribution occurred along the post.

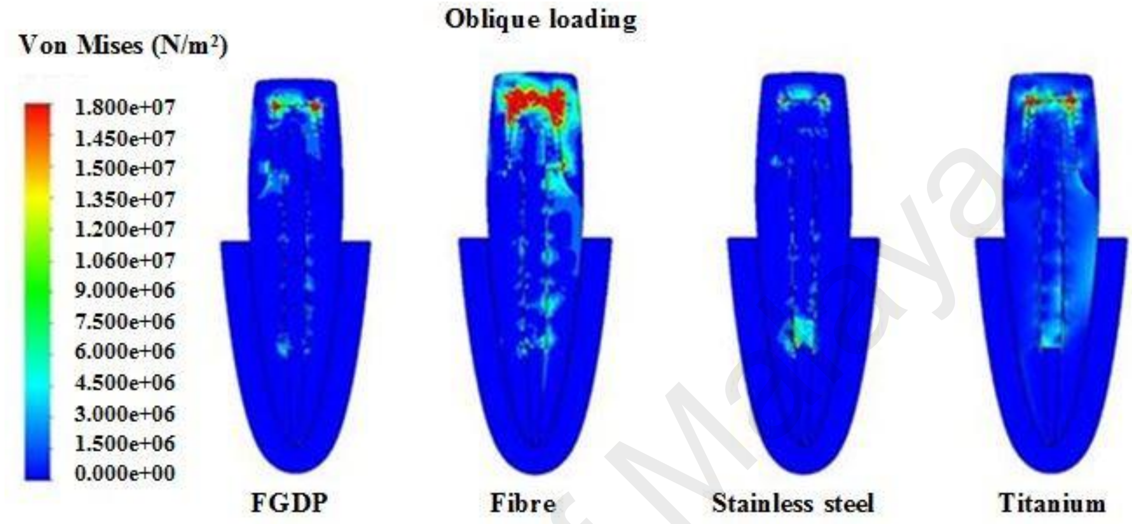


Figure 4.16: Stress distribution under oblique loading for different tooth models

4.5.3 Stress distribution under horizontal load

In the horizontal loading (Figure 4.17), maximum stress was higher in titanium model followed by FGDP model, while stainless steel and fibre models showed fewer stress concentrations.

In the fibre model, maximum stress concentrations occurred at the core-crown interface at the coronal third of the crown and a little stress occurred at the crown-root interface at the cervical third of the root. No stress occurred along the post surfaces of the fibre model.

Titanium also showed less stress distribution in the middle and cervical third of the crown, and the cervical third of the root. Maximum stress occurred at the core-crown interface at the coronal third of the crown and less stress distribution was occurred at the core-crown interface at the middle third of the crown and at the crown-root interface at the cervical third of the root. Along the post, less stress occurred in the middle of the post and the stress decreased apically.

Maximum stress in the stainless steel model was concentrated at the coronal third of the core-crown interface and decreased at the crown-root interface at the cervical third of the root. Along the stainless steel post, a little stress was noticed at the coronal and middle third of the post and disappeared at the apical part of the post.

In the FGDP model, maximum stress occurred at the coronal third of the core-crown interface and at the core-root-crown interfaces at the cervical of the crown. A very little stress was noticed at the apical third of the root. Along the FGDP surfaces, only a little stress was noticed at the junction of the coronal and middle third of the post and no stress was noticed at the apical third of the post.

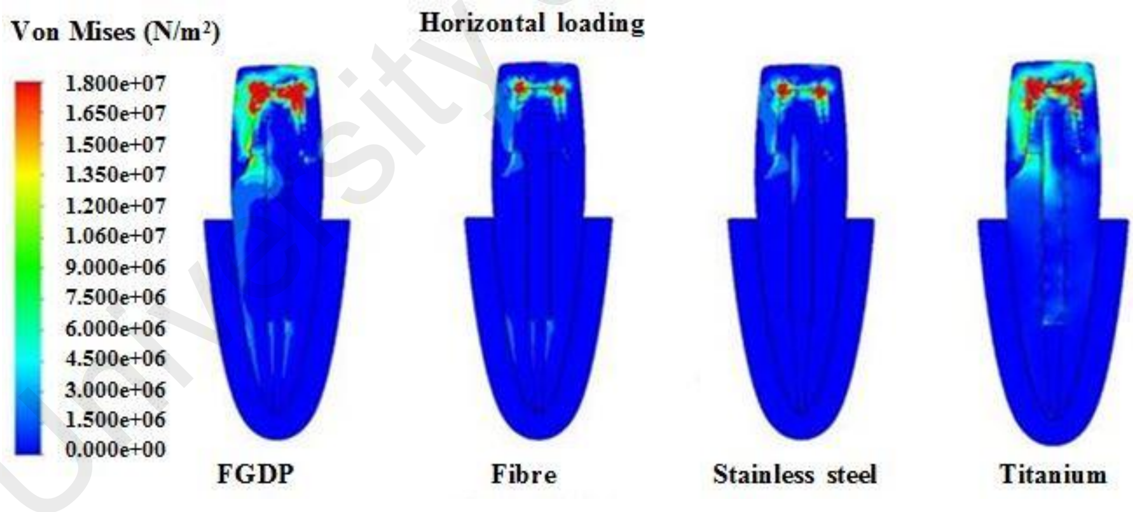


Figure 4.17: Stress distribution under horizontal loading for different tooth models

In the horizontal load direction, no stress was concentrated at the middle and apical third of the roots in all models, in contrast to the vertical and oblique load directions which showed some stress concentrated at these areas.

Maximum principle stress distributions along the post surfaces and at post-root interfaces under vertical, oblique and horizontal load directions for FGDP, titanium, fibre and stainless steel models are more explained and illustrated in Figure 4.18 and Figure 4.19.

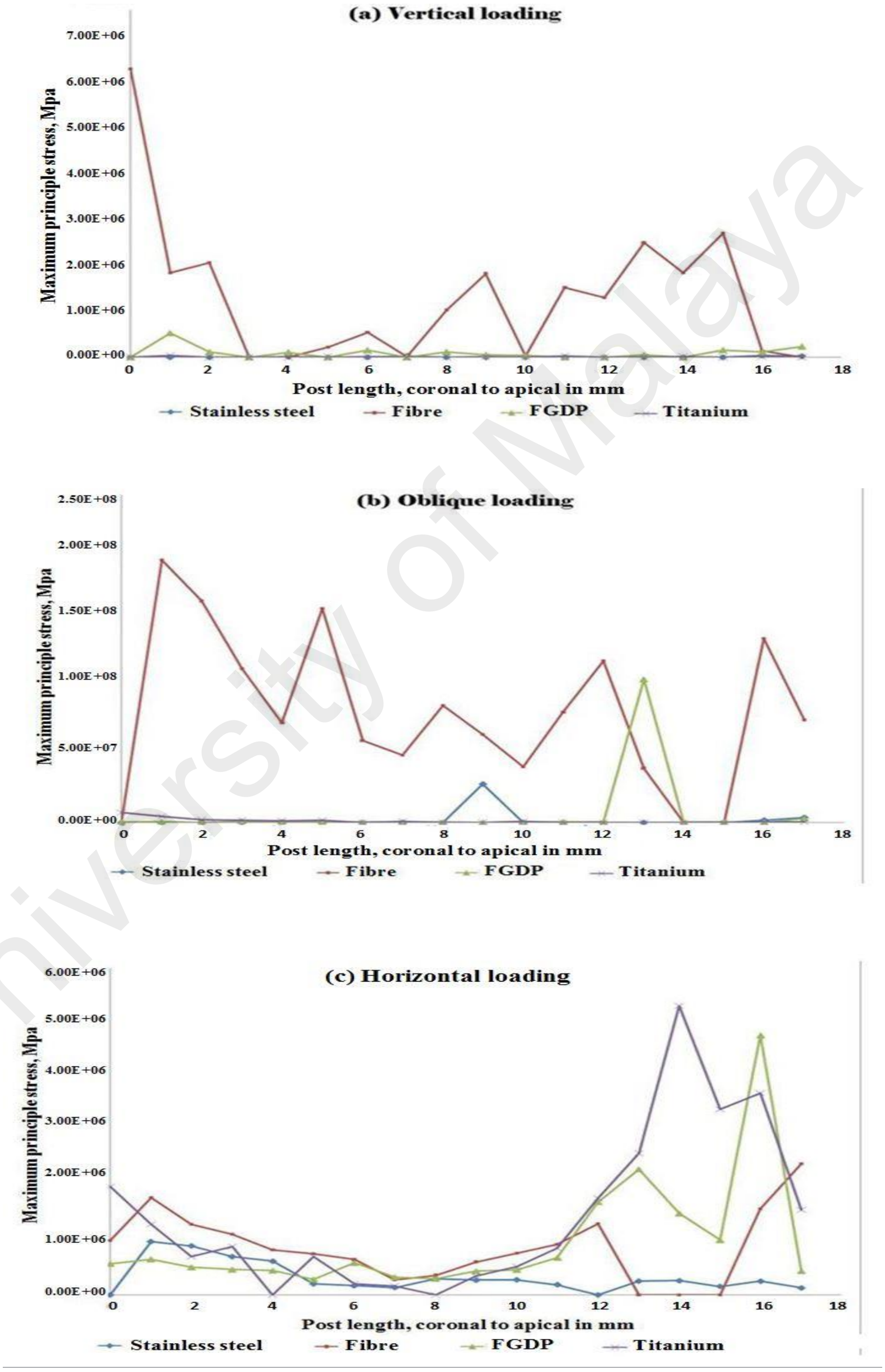


Figure 4.18: Maximum stress distributions along the posts when loaded in vertical (a), oblique (b) and horizontal directions (c)

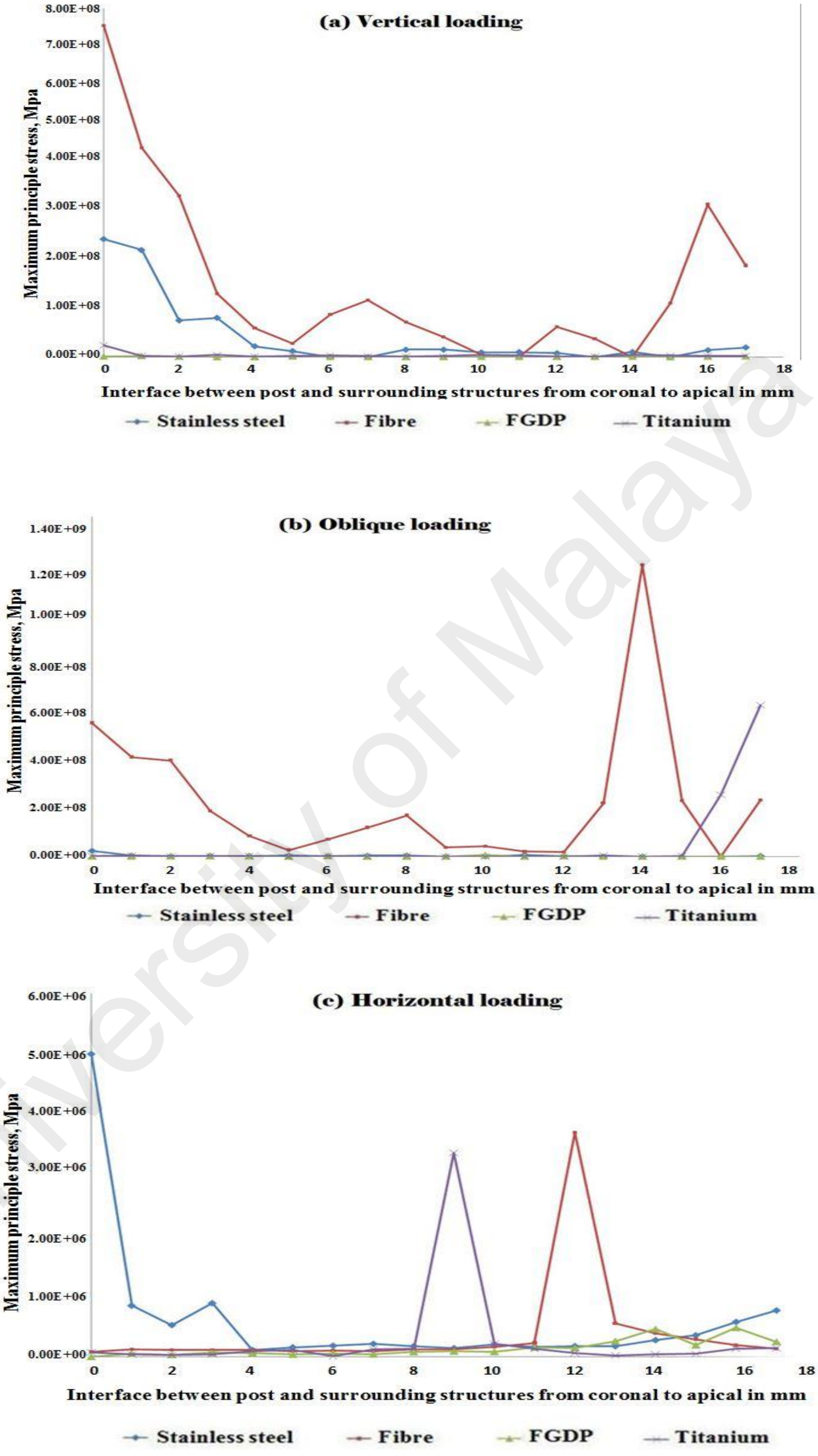


Figure 4.19: Maximum stress distributions at the post-root interface when loaded in vertical (a), oblique (b) and horizontal directions (c)

4.6 Chapter summary

Experiments and tests used in this study were evaluated and thoroughly analysed in this chapter. Ti-HA-BG composites (layers) were chemically characterised by using XRD and EDX. Physical (Density) and mechanical (micro-hardness (Vickers), modulus of elasticity and compression) tests that were done on various ratios of Ti-HA-BG composites were also analysed.

Fracture resistance and failure mode of teeth restored with FGDPs, fibre, stainless steel and titanium posts were also evaluated. Stress distribution was also analysed by using finite element analysis. Four models restored with functionally graded dental post, fibre, stainless steel and titanium posts were analysed in this chapter by using Solid Works software.

CHAPTER 5: DISCUSSION

5.1 Introduction

This chapter has five sections. Section 5.2 discusses how Ti-HA composites were optimised by using BG. The optimum way to sinter Ti-HA-BG composites using air atmosphere and vacuum furnaces is described in section 5.3. The reasons for using processing and layering technique in fabrication of functionally graded dental posts are also discussed in this section. The methods for storing the teeth, instrumentation, obturation, restoring the teeth with FGDPs and other commercial post types are later explained in section 5.4. In this chapter, the results of the current study regarding fracture resistance and failure mode are discussed too. The stress distributions in the four FEA generated models restored with FGDPs, fibre, titanium and stainless steel posts in the vertical, horizontal and oblique directions are explained in section 5.5.

5.2 Optimisation of different Ti-HA composites using various ratios of bioactive glass

There are many methods to fabricate the composites and functionally graded multi-layered (FGM) composites, such as coating, lamination and powder processing (Kieback *et al.*, 2003). In this study, the powder processing method was used to fabricate both the composites and functionally graded composites due to ease of use, speed and cost compared to other methods. Moreover, it does not require any complicated equipment to fabricate the composites. It requires only the powders that will be used to fabricate the desired composites, and the ordinary equipment that is usually available in laboratories for mixing, pressing and sintering processes (Kieback *et al.*, 2003; Madfa, 2011a).

In this study, Ti-HA-BG mixtures were dried in an incubator for 24 hours at 110 ^{o}C to avoid the moistening of the mixtures (Shahrjerdi *et al.*, 2011; Oshkour *et al.*, 2014).

The mixtures were then mixed and milled using a ball milling machine for 5 hours to achieve a uniform particle size and to minimise the powders aggregations (Madfa, 2011a; Shahrjerdi *et al.,* 2011; Oshkour *et al.,* 2014; Halim, 2017).

The mixtures were then poured into silicone moulds to achieve the desired composites shape and FG composites, and to facilitate pressing the samples by using a cold isostatic press (CIP). The hydraulic press was used in the pilot study and showed that many fractures and cracks occurred in the interfaces between the layers due to the insufficient pressing of the samples. Hence, an alternative pressing method, such as cold isostatic press was used in this study.

Samples were pressed by using CIP at 250 MPa as it was reported that pressing the samples at >180 MPa will reduce the porosity and improve the mechanical and physical properties of the samples (Madfa, 2011a; Shahrjerdi *et al.*, 2011; Oshkour *et al.*, 2014). Ti-HA composites can be successfully pressed and fabricated by using the cold isostatic press as discussed and has been used in many studies (Shahrjerdi *et al.,* 2011; Bonfim *et al.,* 2014; Halim, 2017). Unlike the uniaxial hydraulic press, CIP applies the pressure to the samples from all directions and thus provides the maximum density to these pressed samples (Atkinson & Davies, 2000; Arifin *et al.,* 2014).

In this study, to fabricate the functionally graded dental post which was composed of different layers, five separated layers (composites) based on different ratio of titanium-hydroxyapatite-bioactive glass materials were fabricated and characterized.

Titanium has good mechanical properties and HA has good biocompatibility. Using Ti-HA materials together, provided a composite with a combination of good mechanical and biocompatibility properties that can be used in biomedical applications (Arifin *et al.,* 2014; Arifin *et al.,* 2015; Miranda *et al.,* 2016). However, Ti requires to be sintered at high temperature in a vacuum or an argon atmosphere furnace, while HA requires to be sintered in air atmosphere furnaces. Merging them in one composite and sintering the composite at high temperature in an environment furnace, will cause oxidation and decomposition of Ti-HA materials to TTCP, TCP, Ti_2O and TiO at which may cause cracks and fractures to the new produced composites (Weng *et al.,* 1994; A Nanci *et al.,* 1998; Guo *et al.,* 2006; Marcelo *et al.,* 2006; Arifin *et al.,* 2014; Goudarzi *et al.,* 2014).

To overcome this problem, Ti-HA is sintered at low temperature with low sintering temperature additives. Thus Ti-HA properties can be maintained and provide a good sintering process for the produced composites. Bioactive glass materials can aid in sintering the Ti-HA composites and Ti-HA functionally graded composite and also enhance their mechanical, chemical and bioactivity properties (Kleebe *et al.,* 1997; Moskalenko & Smirnov, 1998; Ning & Zhou, 2004; Prasad *et al.,* 2017).

Incorporation 10% of bioactive glass to various ratios of Ti-HA composites has been shown to enhance the bioactivity and maintains the properties of the used materials (Moskalenko & Smirnov, 1998; Ning *et al.,* 2000; Ning & Zhou, 2004).

In this study, different ratios of bioactive glass (5%, 10% and 15%) were incorporated with various ratios of Ti-HA composites and sintered at 900 ^{o}C in an air atmosphere furnace. The optimum ratio of BG that can produce Ti-HA-BG composites that were free from cracks and fractures with minimum Ti oxidation and HA decomposition were obtained.

Different Ti-HA ratios with a fixed 10% of BG showed that composites were free from cracks and fractures, while that of Ti-HA composites with 5% and 15% of BG showed many cracks and fractures to the sintered composites. Many studies (Moskalenko & Smirnov, 1998; Ning *et al.,* 2000; Ning & Zhou, 2004) have reported that by using 10% of BG will allow sintering the Ti-HA composites at low temperature, and thus minimise the oxidation and decomposition process of Ti and HA. Whereas, Ti-

HA layers that incorporated with 5% and 15 % BG were cracked and fractured due to Ti oxidation and HA decomposition. The results were confirmed by XRD and EDX as discussed in Section 5.3.1 of this chapter.

5.3 Characterization and optimisation of Ti-HA-BG composites to fabricate functionally graded dental posts

In this part of the study, 10% bioactive glass was added to five different ratios of Ti-HA mixtures. The mixtures were prepared using the powder processing method and sintered with the cold isostatic press as recommended and used by many previous studies (Shahrjerdi *et al.,* 2011; Arifin *et al.,* 2014; Bonfim *et al.*, 2014; Halim, 2017).

The green samples were then divided into two groups, Group 1 sintered in air atmosphere furnace while Group 2 sintered in the vacuum furnace. The purpose of this part of the study was to obtain the optimum sintering environment of Ti-HA-BG mixtures, which minimises Ti oxidation and HA decomposition.

Many studies were done on sintering HA and Ti in air atmosphere and in vacuum or in Argon atmosphere furnaces. Results suggested that HA should be sintered in an air furnace to minimise the decomposition at which sintering HA in non-air furnace will cause dihydroxylation of HA (losing of -OH from HA) and then decomposition to TCP and TTCP will also occur. On the other hand, Ti should be sintered in vacuum or in Argon atmosphere furnaces to reduce the oxidation process (Weng *et al.,* 1994; Marcelo *et al.,* 2006; Arifin *et al.,* 2014; Goudarzi *et al.,* 2014; Wakily *et al.,* 2015).

Sintering the metal-ceramic bio-composites with and without incorporation of BG materials at 1000, 1100 and 1200 ^{o}C was also performed. A few of these studies reported that HA and Ti were totally decomposed and oxidised to TTCP, TCP, $TiCaO_3$, TiO, Ti_2O and TiO_2 at which they have poor mechanical properties compared to Ti and

HA (Balbinotti et al., 2011; Arifin et al., 2015). Others reported a partial decomposition of HA and oxidation of Ti at which the main phases were associated with the decomposed compositions and the minor phases belonged to HA and the intact Ti (Ning & Zhou, 2004; Shahrjerdi *et al.,* 2011; Balbinotti *et al.,* 2011; Arifin *et al.,* 2015; Halim, 2017). That could be due to absence of materials that could aid in sintering process of Ti-HA composites (such as bioactive glass), which may help to minimize HA decomposition and Ti oxidation (Moskalenko and Smirnov, 1998).

5.3.1 Chemical analysis

In this study, the five layers sintered in an air atmosphere furnace (G 1) showed the expected results. The XRD and EDX analysis indicated that the major peaks and phases in this group for all layers belonged to the main materials used in the fabricated composites. In layer 1, peaks and major phases belonged to Ti. Titanium showed some oxidation to TiO and Ti_2O while TiO_2 was not detected in this layer. TiO was totally oxidised to TiO_2 in layer 3 and layer 4. Titanium was gradually decreased in ratio from layer 1 to layer 4 and completely disappeared in layer 5 since no Ti was added in this layer. These results were in agreement with previous studies, which have indicated that Ti started to oxidise to TiO and Ti_2O at 700 ^{o}C, 800 ^{o}C and 850 ^{o}C (Balbinotti *et al.,* 2011; Arifin *et al.,* 2015; Comín *et al.,* 2017).

HA was not added to layer 1, hence HA was not detected in this layer. HA, however, was observed in every other layer from layer 2 to layer 5. No HA decomposition was noticed in all the HA layers in this group and thus, HA was totally intact. The XRD analysis showed that there was no diffraction peaks related to BG because all BG was converted to HA, as was reported in the previous study (Ning & Zhou, 2004).

Ning *et al.* (2000) and Ning and Zhou (2004) have sintered the BG with Ti-HA composite at different temperatures. They reported that BG amorphous phase occurs at

600 ^{o}C, while at 800 ^{o}C all BG diffraction peaks were converted to HA. They concluded that BG could improve the bioactivity and properties of Ti-HA composites and also the sintering process of these composites. This was in agreement with the results of the current study at which BG has improved the sintering of Ti-HA composites by preventing the HA decomposition and the complete Ti oxidation when sintered in air atmosphere furnace.

It is noted that composites in this study were sintered at 900 ^{o}C to produce a stable composite below HA decomposition temperature and to avoid Ti oxidation. It was also reported that HA decomposition to TCP occurs at 1250 ^{o}C when sintered with Ti in air and at 1050 ^{o}C when sintered in vacuum furnace, while Ti oxidise to Ti_xP_y and CaTiO3 at 1200 ^{o}C (Ning & Zhou, 2004; Balbinotti *et al.*, 2011; Comín *et al.*, 2017). Therefore no HA decomposition was detected in the layers sintered in air atmosphere furnace in this study.

The main phases of the five layers sintered in vacuum belonged to Ti and the minor phases belonged to TiO, Ti_2O and TiO_2. In the layers that contained HA, decomposition to CaO, TCP and TTCP were noticed from layer 2 to layer 5. The observations are in agreement with many studies which reported HA decomposition when Ti-HA was sintered in vacuum or Argon atmosphere furnace (Balbinotti *et al.*, 2011; Arifin *et al.*, 2015; Comín *et al.*, 2017; Halim, 2017).

In this study, EDX analysis was used to confirm the XRD results and the gradual decrease in Ti among layer 1 to layer 4. The gradual increase in HA ratio from layer 2 to layer 5. EDX analysis also verified that no contamination occurred among all the composites since no new elements were identified in the fabricated composites.

EDX results in this study agreed with many studies involved in the fabrication of Ti-HA and Ti-HA-BG composites and FG composites (Ning & Zhou, 2004; Madfa, 2011a; Comín *et al.*, 2017; Halim, 2017).

5.3.2 Physical and mechanical properties

In this study, layers sintered in air atmosphere furnace showed a higher density than those sintered in the vacuum furnace. In general, layers with a high ratio of Ti showed a higher density than those with no or lower ratio of Ti. That was because the density of Ti (4.5 g/cm^3) is higher than that of HA (3.156 g/cm^3) and BG (2.707 g/cm^3) (Hedia & Mahmoud, 2004; Srivastava *et al.*, 2012a; Arifin *et al.*, 2015).

The densities of the Ti-HA-BG composites were changed and gradually decreased from layer 1 to layer 5. As mentioned in chapter 4 Section 4.3.3, the experimental densities in this study were not much lower than the theoretical densities. In general, densities can be affected by many factors such as a powder characteristics and pressing process (Madfa, 2011a). There is a big difference between the particle size and shape between Ti and HA at which Ti particles are much larger than HA particles. However, it was believed that the space between the large Ti particles can be filled by the smaller HA particles, and thus the spaces will be reduced and the density of the fabricated Ti-HA-BG composites will be increased (Madfa, 2011a).

Chemical compositions were the most important factor that affected the physical and mechanical properties of the fabricated Ti-HA-BG composites in this study. The decomposed and oxidised components were weaker than Ti and HA, and thus will reduce the density, modulus of elasticity, strength and hardness of the composites (Watari *et al.*, 1997; Madfa, 2011a; Prasad *et al.*, 2017).

Composites sintered in an air atmosphere furnace showed a higher density, modulus of elasticity, hardness and compressive strength than those sintered in the vacuum furnace. The reason was because composites sintered in air do not show HA decomposition for all the 5 layers (composites) while those sintered in vacuum showed some decomposition of HA to composite with low density, such as TCP (3.07 g/cm^3) and TTCP (3.06 g/cm^3) as compared to that of HA. The density and mechanical properties of the fabricated Ti-HA-BG will be affected, as reported by many studies (Kong *et al.,* 2002; Bodhak *et al.,* 2011; Wakily *et al.,* 2015; Comín *et al.,* 2017; Halim, 2017; Prasad *et al.,* 2017).

5.3.3 Functionally graded dental post

In this study, functionally graded composites were fabricated by powder processing methods as described previously in Section 3.2. Five different ratios of Ti-HA-BG layers (mixtures) were poured layer by layer starting from layer 1 to layer 5 into the produced silicone moulds. The process allowed the desired cylindrical FG composites shapes to be formed when poured in the current study silicone moulds, or in the elastic tube or geometrical die as recommended and used by previous studies (Madfa, 2011a; Wakily *et al.,* 2015; Halim, 2017). The silicone moulds were rounded and cylindrical in shape at which there were no sharp corners to avoid stress concentration and eliminate any failures at corners (Madfa, 2011a).

Pouring layer by layer into the silicone moulds allows gradual fabrication of graded layers. Their microstructures can also be controlled easily with non-expensive types of equipment (Madfa, 2011a). The occurrence of sharp interfaces in the composites produced stress and caused failures. Functionally graded technique (layering method) terminates these sharp interfaces areas, and thus reduced the stress between layers and avoided failures that could occur due to sharp edges. It was reported that a suitable transition of the fabricated materials could be achieved by using the functionally graded materials technique (Madfa, 2011a; Alla *et al.*, 2011).

It was also discovered that composites sintered in air atmosphere showed better chemical, physical and mechanical properties than those sintered in vacuum furnace. As a result, in this work FG composites were pressed by CIP and sintered in the air at 900 ^{o}C.

After sintering, the samples were sent for machining and shaping by using a centreless grinder to produce the functionally graded dental posts with the desired diameter (1.5 mm) and length (14 mm). Centreless grinding was considered the main method in the machining process due to its high accuracy and flexibility (Rascalha *et al.*, 2013). The FG dental posts were machined and shaped with 1.5 mm in diameter because it is the suitable one to be restored later in the root canals of the maxillary central incisor teeth as recommended by the manufacturers of many prefabricated dental post types and the previous study (Abdulmunem *et al.*, 2016).

5.4 Evaluating the fracture resistance and failure mode of the endodontically treated teeth restored with FGDPs and other post types

5.4.1 Disinfection and storing of the teeth

To avoid infection, teeth in this study were disinfected with Chloramines T trihydrate 0.5% as suggested by ISO/TS 11405: 2003 (ISO, 2003) for one week, which is considered as safe materials to store the teeth and which has no negative effect on the structures and strength of the dentine. Teeth were then stored in distilled water at 4 ^{o}C to minimize the dehydration and deterioration, at which the distilled water was weekly changed (Secilmis *et al.*, 2013; Freitas *et al.*, 2016).

5.4.2 Instrumentation and obturation

The instrumentation was carried out for all the root canals using the step-back technique as it minimizes the coronal and middle thirds dentinal interfaces and provides efficient and faster instrumentations (Torabinejad, 1994). After each instrumentation step, root canals were irrigated with 3 ml of 1% NaOCl to provide antimicrobial effects (Giardino *et al.,* 2009).

Root canals were then irrigated with 3 ml of 17% EDTA to remove the smear layer that was produced during the instrumentation in order to provide a good adhesion between the root canal structures and the cement materials (Wu *et al.,* 2009; McLaren *et al.,* 2009; Ulusoy & Görgül, 2013; Silveira *et al.,* 2017). EDTA and NaOCl traces were removed by irrigating all root canals with 10 ml of distilled water.

All root canals were obturated with AH Plus sealer by using lateral condensation method as it is the method of choice to be used in straight canals, especially when restored with dental posts (Clinton & Himel, 2001; Aykent *et al.,* 2006; Zapata *et al.,* 2009). To eliminate the possibility of leakage, all roots were placed in the incubator at 100% of humidity and 37 ^{o}C for 24 hours to ensure a complete set of the sealer (Bodrumlu, 2008).

5.4.3 Dental post space preparation and PDL simulation

To prepare post space, gutta-percha in all canals was removed by using Gate-Glidden drills attached to a low-speed hand-piece, in which they are fast and easy to be used to remove the gutta-percha from the root canals, as reported in the previous study (Giuliani *et al.,* 2008). Gutta-percha of 5 mm at the apical third of the root canal was left intact to provide a good apical sealing (Wahab, 2004; Bodrumlu, 2008; Gopi *et al.,* 2015; Madan *et al.,* 2016).

Special drills that match each dental post types (according to the manufacturers) were used to prepare 9 mm of the dental post space in the root canals, as recommended by a previous study (McLaren *et al.,* 2009). Then, posts were cemented in the root canals with zinc phosphate cement according to the manufacturer's guidelines. Zinc phosphate cement is considered as one of the most widely used cement in dentistry. It is reported to have a long-term success, good compressive strength, and the cement of choice to be used to cement different dental restorations (dental post and crowns) (Øilo & Espevik, 1978; Abdulmunem *et al.,* 2016; Schwendicke *et al.,* 2016; Penelas *et al.,* 2016; Rojpaibool & Leevailoj, 2017; Sriyudthsak *et al.*, 2018).

The biological width of the periodontal ligaments was simulated by painting the root surfaces with 0.15- 40 mm of light body of silicone impression materials. A length of 3 mm below the cemento-enamel junction CEJ were left without being covered with the silicone impression materials or epoxy resin materials to simulate the free gingival depth (Akkayan, 2004; Al-Wahadni *et al.,* 2008; Hitz *et al.,* 2010; Bassir *et al.,* 2013; Ilgenstein *et al.*, 2015).

5.4.4 Ferrule preparation, metal coping and thermocycling

A 2 mm ferrule was prepared around the cores margin in this study as they provided a higher fracture resistance of teeth restored with dental post than those without a ferrule preparation (Abdulrazzak *et al.,* 2014; Aggarwal *et al.,* 2014; Kar *et al.,* 2017; Valdivia *et al.,* 2018).

Ni-Cr metal copings were used in this study because they have good hardness and higher strength properties compared to ceramic crowns as reported and used in many previous studies (Campbell, 1989; Giovani *et al.,* 2009; Pasqualin *et al.,* 2012; Haralur *et al.*, 2017; Moris *et al.*, 2017; Verri *et al.*, 2017).

All teeth were then thermocycled according to ISO/TS 11405: 2003 (ISO, 2003) by using a thermocycling machine. That has been done to simulate the temperature and moisture of the oral environment and was used and recommended by several studies (Al-Khatieeb, 2018; Linsuwanont *et al.,* 2018; Mohajerfar *et al.,* 2018; Stepp *et al.,* 2018).

5.4.5 Loading

Teeth were positioned in a metal jig with 45^{o} angle and load was applied at 135^{o} angulation to the long axis of the roots. Teeth loaded at 130^{o} -135^{o} angulation simulates the inter-incisal angle in the anterior teeth area and mimic the Class 1 occlusion in the human mouth, as reported and used by many studies (Akkayan, 2004; Rosentritt *et al.,* 2015; Hazzaa *et al.,* 2015; Preis *et al.,* 2016; Beltagy, 2017).

A flat-ended cross-head pin with a speed of 0.5 mm/min was applied on the palatal surface of the metal coping on the notch that was prepared 2 mm below the incisal edge to avoid slipping of the pin during loading (Abdulrazzak, 2011; Rosentritt *et al.,* 2015; Zhang *et al.,* 2015).

In this study, to evaluate the maximum fracture resistance value in the endodontically treated teeth restored with different post types, an increased (static) compressive load was applied on the prepared notches of the metal copings. However, the intraoral situations were simulated more by cyclic load than the static one.

The masticatory forces that occur during mastication tends to be repetitive and the forces were applied on a large surface area which was simulated by cyclic load than the static load which can be applied on a specific area until failure occurs (Mohammadi *et al.,* 2009; Turk *et al.,* 2015; Kurthukoti *et al.,* 2015; Aurélio *et al.,* 2016).

However, teeth in human mouth should resist both cyclic and static load and there were many studies which investigated the fracture resistance of teeth restored with different dental restorations under static load (El-Damanhoury *et al.,* 2015; Dastjerdi *et al.,* 2015; Abduljawad *et al.,* 2016; Kubo *et al.,* 2018).

5.4.6 Results

5.4.6.1 Fracture resistance of the endodontically treated teeth restored with FGDPs and other post types

In the current study, teeth restored with FGDPs and titanium posts showed significantly higher fracture resistance than those restored with stainless steel posts. In addition, there was no significant difference between FGDPs (550 N), titanium (505 N) and fibre posts (475 N).

These results were in agreement with a study done by Madfa (2011b) in which he studied the fracture resistance of teeth restored with different types of FGDPs based on ZrO_2-Ti-HA, Al_2O_3-Ti-HA and Ti-HA-Ti formulations and other teeth restored with cast and titanium posts. The results showed that there was no significant difference between teeth restored with different types of FGDPs and those restored with titanium posts.

Furthermore, fabricating a dental post based on functionally graded concept (Stiffer at coronal part and less stiff at the apical part of the post) will provide a modulus of elasticity close to that of dentine and also helps to reduce the forces gradually to the apical third of the root; thus, that will help the tooth to resist the applied forces during function and increase their ability to resist the fracture (Matsuo *et al.,* 2001; Ramakrishna *et al.,* 2001; Abu Kasim *et al.,* 2011; Madfa, 2011a, 2011b; Senan & Madfa, 2017).

As previously mentioned in the results chapter (4.3.6 modulus of elasticity), modulus of elasticity of the first layer of the FGDP (which will be retained with the core materials) was 20248 MPa and the second layer was 17939 MPa at which these are almost close to that of dentine (18600 MPa) as reported by Ferrari *et al.* (2000). Moreover, the modulus of elasticity of the fourth and fifth layer was 8766 MPa and 7147 MPa, respectively. Hence, the stress was concentrated coronally and decreased apically and thus the ability of tooth to resist the fracture was increased as mentioned in section 5.5 (finite element analysis) and approved by many studies (Ramakrishna *et al.*, 2001; Fujihara *et al.*, 2004; Abu Kasim *et al.*, 2011; Madfa, 2011a).

In this study, results showed that teeth restored with titanium posts had a significantly higher fracture resistance than those restored with stainless steel posts. The results agreed with Abdulmunem *et al.* (2016) at which they concluded that teeth restored with a titanium post and cemented with zinc phosphate cement have higher fracture resistance than those restored with stainless steel posts.

It was also discovered in this work that there was no significant difference between teeth restored with glass fibre posts and those restored with stainless steel posts. The results contradicted with that reported by Sonkesriya *et al.* (2015). They have studied the fracture resistance of 40 maxillary central incisors restored with cast, stainless steel, glass fibre and carbon fibre posts. Their findings that teeth restored with stainless steel posts had a higher fracture resistance than those restored with both glass and carbon fibre posts.

The disagreement could be due to the posts in Sonkesriya *et al.* (2015) report were cemented with dual-cure resin whilst those of the current study were cemented with zinc phosphate. Endodontically treated teeth restored with dental posts cemented with zinc phosphate provide higher fracture resistance than those cemented with dual-cure resin (Abdulmunem *et al.*, 2016).

Posts in this study were cemented with zinc phosphate cement because it provided good retention, stiffness, high compressive strength (96-133 MPa), easy to be mixed, inexpensive and adhere mechanically to the post, crowns and root structures (Mezzomo *et al.,* 2006; Hill, 2007; Hill & Rubel, 2009; Anusavice *et al.,* 2012; Lad *et al.,* 2014). In addition, there was no extra post space preparation required when post was cemented with zinc phosphate. Whereas, resin cement requires more post space preparation to remove the smear layers and provide good retention with the post and tooth structures that will weaken the root structures (Schwartz & Robbins, 2004; Hill & Rubel, 2009).

It was also reported that when ferrule was prepared around the CEJ, there will be no significant difference between cementing the post with resin or zinc phosphate on the fracture resistance of the endodontically treated teeth (Mezzomo & Dalla 2003; Mezzomo *et al.,* 2006). Ferrules of 2 mm was prepared in this study, as reported in Section 3.4.8, and therefore the use of resin instead of zinc phosphate as cement in this study was not needed.

The results of this research are also in disagreament with those of Amarnath *et al.* (2015) study. They studied the fracture resistance of teeth restored with stainless steel posts and fibre posts with different posts length (4, 7 and 10 mm). Amongst all groups, teeth restored with stainless steel posts showed higher fracture resistance than those restored with fibre posts. That could also be due to the posts being cemented with paracore dual-cure resin unlike in the present study which used zinc phosphate as cement.

In this study also, teeth restored with titanium posts had a mean fracture resistance of 505 N, while those restored with fibre posts had 475 N. However, there was no significant difference between both groups. These results were in disagreement with that reported by Al-Wahadni *et al.* (2008) who studied the fracture resistance of teeth restored with titanium, glass fibre and carbon fibre posts. They reported that teeth restored with titanium posts have higher fracture resistance than those restored with fibre posts. That was because of titanium posts used in that study composed of Nickel titanium alloy and cemented with resin cement unlike the titanium posts that used in this study which composed of pure titanium and cemented with zinc phosphate cement.

5.4.6.2 Failure mode of the endodontically treated teeth restored with FGDPs and other post types

The current study showed that teeth restored with FGDPs (100% restorable failures) failed with more restorable failures as compared to those restored with fibre (7% restorable failures), titanium (50% restorable failure) and stainless steel (40% restorable failures) dental posts.

All failure modes in this study were investigated according to the cracks and fractures line position, at which post-core de-bonding, post-core tooth complex fractures and cracks above the epoxy resin block, were considered as restorable failures. Whereas, fractures and cracks below the epoxy resin block line were considered as non-restorable failures, as recommended by Fokkinga *et al.* (2005).

The results of this study agreed with the findings of Madfa (2011b). They reported that teeth restored with FGDPs (Based on ZrO_2-Ti-HA, Al_2O_3-Ti-HA and Ti-HA-Ti) tend to fail with restorable failure than those restored with cast and titanium posts. The titanium and cast post and core groups showed catastrophic failures where most of the teeth failed with non-restorable failures and only one tooth from each group failed with restorable failures. Whereas, teeth restored with all three types of FGDPs showed 80%-90% of restorable failures.

Dental posts with functionally graded multilayered composition experienced gradual changed in stiffness from the dental post coronal third to the apical third. Layers with high stiffness (at the coronal third) will eliminate the stress from the core and layers with lower stiffness will spread the stress to the neighbouring root structures in a regular way, causing a restorable failure (Ramakrishna *et al.,* 2001; Abu Kasim *et al.,* 2011).

Teeth restored with fibre posts, failed with 7% restorable failures. That was because the modulus of elasticity of the fibre post is closed to that of dentine. Dental posts with a low modulus of elasticity such as fibre posts tend to fail with more restorable failures. That was because, fibe post flexed during loading and distributed the load homogenously to teeth structures as reported and evaluated by many studies (Fokkinga *et al.,* 2005; Novais *et al.,* 2009; Goracci & Ferrari, 2011; Uthappa *et al.,* 2015; Abduljawad *et al.,* 2016; Habibzadeh *et al.*, 2017).

The rsults in the present study were also in agreement with those of Makade *et al.* (2011). They investigated the failure mode of teeth restored with cast (type III cast gold alloy) post and core, stainless steel or glass fibre posts. They reported that teeth restored with fibre posts demonstrated more restorable failures than those restored with stainless steel and cast post and cores. These results were also in agreement with another study, which reported that restorable failures were more likely to occur in teeth restored with fibre post than those restored with metal posts (Dastjerdi *et al.,* 2015).

Teeth restored with titanium or with stainless steel showed more non-restorable failures compared to those restored with FGDPs and fibre. That could be due to high modulus of elasticity of titanium and stainless steel which tends to result in more non-restorable failure (Novais *et al.,* 2009; Makade *et al.,* 2011; Abdulmunem *et al.,* 2015; Amarnath *et al.,* 2015).

5.5 Finite element analysis

In order to understand and analyse the mechanical behaviour of the teeth restored with dental restorations (such as dental posts and crowns) when forces are applied, fracture tests must be performed. However, these tests are limited and not able to predict the stress areas in both the restoration and tooth structure when failures occur during loading; thus, by using a non-destructive test, such as stress distribution where it can be applied and analysed using FEA is more recommended. Analysing the stress concentrations is important in order to predict the failure modes and position in the tooth-restoration complex when failure occurs (Jacobsen *et al.*, 2006; Soares *et al.*, 2007; Soares *et al.*, 2008; Adigüzel *et al.*, 2011; Upadhyaya *et al.*, 2016).

In this study, forces that occur during bruxism, external trauma and mastication in the human mouth were simulated by applying 100 N of a static load in vertical, oblique and horizontal directions as recommended in previous studies (Joshi *et al.*, 2001; Genovese *et al.*, 2005; Hu *et al.*, 2013).

Four models were generated using finite element software at which these models resembled the maxillary central incisors restored with FGDP, titanium, fibre and stainless steel posts retained with composite cores and metal copings in accordance to the parameters given in the literature.

Restoring endodontically treated teeth with high modulus of elasticity and homogenous dental posts, such as titanium and stainless steel posts will dissipate the stress to the root structures irregularly, causing non-restorable failures (Abu Kasim *et al.*, 2011; Adigüzel *et al.*, 2011; Abdulmunem *et al.*, 2016).

In the present study, the stainless steel and titanium models exhibited more maximum stress concentrations than fibre and FGDPs models at the apical third of post-root interfaces. However, there is a study which disagrees with the finding and has reported that posts with a high modulus of elasticity exhibits a uniform and minimal stress distribution to the surrounding root structures (Upadhyaya *et al.,* 2016).

On the other hand, restoring endodontic treated teeth with a low modulus of elasticity, such as fibre post provides better stress distribution from the post to the root structures as reported previously (Garhnayak *et al.*, 2011; Kumar & Rao, 2015). Fibre post tends to exhibit maximum stress amongst the long axis of the post and distribute minimum stress to the adjacent root structures (Kumar & Rao, 2015).

The results of the present study showed that the fibre model displayed better stress distribution than stainless steel and titanium models and the stress distributed to the root structures was minimal as compared to these models.

In order to avoid irregular stress distribution from the post to the tooth structures, the dental post should maintain different and gradual modulus of elasticity along the long axis. The coronal third of the post should contain a higher modulus of elasticity compared to the middle and apical thirds in order to provide better retention to the cores. The modulus of elasticity of dental post should be reduced gradually towards the apical third to minimise the stress and distribute it regularly to the root structures and minimise the possibility of non-restorable failures. This can be achieved by producing a dental post with functionally graded compositions (Ramakrishna *et al.,* 2001; Abu Kasim *et al.,* 2011; Madfa, 2011b; A. Madfa *et al.,* 2014). This was achieved and shown in this study at which layer 1 and layer 2 had a higher modulus of elasticity compared to layer 3, 4 and 5 as shown and discussed in Section 4.3.6 and Section 5.3.2.

The results of the present study showed that the FGDPs (FE) model exhibited better stress distribution as compared to titanium and stainless steel models. No maximum stress concentrations were shown at the apical third of the post-root interface. The stress that was distributed in these models to the root structures was minimal compared to the stainless steel and titanium models.

In general, maximum stress in all models (FGDPs, fibre, titanium and stainless steel models) was concentrated at the core-crown interfaces at the coronal third of the crown. That was in disagreement with a study reported that maximum stress was concentrated at the cervical third of the post and the crown and the coronal third of the crown. No maximum stress was seen at the core-crown interface at the coronal third of the crown, (Upadhyaya *et al.,* 2016). The disagreement because of they used nickel chromium cast post and core with tapered design while posts used in the present study were prefabricated parallel posts.

The highest stress concentrations were seen in the models that were loaded in a vertical direction compared with those loaded in oblique and horizontal directions. This finding disagree with another study which reported that models loaded at the horizontal direction exhibited a higher maximum stress than those loaded in a vertical direction (Al-Omiri *et al.,* 2011). This could be due to the different post diameters and designs used in that study as compared to that of the present models with the standard post with diameters of 1.5 mm and with parallel sided post design which provide better retention compared to tapered post (Torbjörner et al., 1995; Qualtrough et al., 2003; Peutzfeldt et al., 2008).

5.6 Chapter summary

This chapter discusses the reasons for adding BG to Ti- HA composites and how BG can improve Ti-HA composite by minimizing the HA decomposition and Ti oxidations. Moreover, the reasons for sintering the Ti-HA-BG composites and functionally graded Ti-HA-BG multilayered composites in different sintering conditions (air atmosphere and vacuum furnaces) are discussed and elaborated. The optimum sintering condition was also explained and discussed in this chapter.

This chapter also discusses the effectiveness of functionally graded dental posts and other commercial post types used to restore endodontically treated teeth with regard to their fracture resistance and failure mode. Stress distribution in endodontically treated teeth restored with functionally graded dental post, fibre, titanium and stainless steel post were also elaborated.

CHAPTER 6: CONCLUSION

6.1 Conclusion

Based on the limitations of this study, the following conclusions were as:

1-Adding 10% BG to Ti-HA composites could reduce the sintering temperature to 900 ^{o}C, minimizing the decomposition of HA and oxidation of Ti and thus maintain the good properties of Ti-HA composites.

2-Sintering the Ti-HA-BG composites and functionally graded material in air atmosphere furnace showed better chemical (the HA decomposition and Ti oxidation were minimized), higher physical (density), mechanical (micro-hardness, compression strength and modulus of elasticity) results compared to those sintered in vacuum furnace (due to decomposition of HA and oxidation of Ti).

3- Teeth restored with FGDPs showed higher fracture resistance and more restorable failures compared to those restored with fibre, titanium and stainless steel dental posts.

4- Stress was more regularly distributed in fibre and FGDPs models compared to titanium and stainless steel models.

6.2 Limitations of the study

1. Samples were pressed at 250 MPa only because this was the highest pressing point of the available CIP machine.

2. Teeth were load under static compressive load instead of cyclic load and thus fatigue was not simulated in this study.

3. Material powders with different particle size were used which may have affected the material particles distribution of the fabricated composite and thus their density may be affected too.

4. No *in vivo* studies were done and only *in vitro* studies were performed in this study.

6.3 Recommendations for further studies

1. Pressing the functionally graded material at more than 250 MPa is advised at which it may improve the FGM composite's mechanical and physical properties.

2. Evaluation of fracture resistance of teeth restored with dental posts under cyclic force.

3. Evaluation of mechanical performance of teeth restored with FGDPs *in vivo*.

4. Trimming the FGDPs below 1 mm to be inserted in posterior root canals at which the post in the present study trimmed to 1.5 mm and inserted in anterior central incisors root canal.

5. Check the possibility of sintering the FGM in hot press furnace.

6. Using Nano-particles powders to optimise and improve the performance of the FGDPs.

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LIST OF PUBLICATIONS AND PAPERS PRESENTED

Submitted paper

1. Mohamed Abdulmunem, Muralithran G. Kutty, Wan Haliza Abd Majid, Ali Dabbagh, Noor Hayaty Abu Kasim, Noor Azlin Yahya, Hadijah Abdullah. The effect of bioactive glass and sintering conditions on the properties of titanium-hydroxyapatite composites. Sains Malaysiana.

2. Mohamed Abdulmunem, Muralithran G. Kutty, Wan Haliza Abd Majid, Noor Azlin Yahya, Hadijah Abdullah. Mechanical properties of teeth restored with novel functionally graded posts based on titanium-hydroxyapatite-bioactive glass. International Journal of Materials Research.

Conference presentation

1. Mohamed abdulmunem, muralithran g. Kutty, wan haliza abd majid, ali dabbagh, noor hayaty abu kasim, noor azlin yahya, hadijah abdullah. Characterization and optimisation of titanium-hydroxyapatite composites using bioactive glass. International Conference on Oral Immunology and Oral Microbiology, 14th -15th August 2018.

Patent

1. Functionally graded dental post using bioactive glass, patent, PI 2016703069, 23/08/2016.