Investigating and Optimising Composite Veneered Zirconia Laminates

Submitted in partial fulfilment of the requirements for the Degree of Master of Philosophy (MPhil)

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In The Name of God, Most Gracious, Most Merciful

I would like to dedicate this thesis to my inspiring loving parents, wife and my Sheffield born son, who supported me every step of the way.

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Summary:

Ceramic is a widely used veneering material in dentistry due to its outstanding properties of biocompatibility and aesthetics. One of the most used dental restorations is the porcelain fused to metal (PFM) restoration, after which developed the current range of all-ceramic restorations. Different tough ceramic cores are used and one of the newest core materials used in all-ceramic restoration is zirconia.

Problem: Zirconia/ceramic restorations have excellent aesthetic properties but they also prone to damage due to the veneer ceramic chipping. Ceramic may be repaired by adding composite but this is a difficult technique and a sensitive procedure. A further drawback with zirconia/ceramic restorations is the exaggerated tooth wear that they may cause and possible discomfort caused by the ceramic veneer with strong bite patients.

Aim and objectives: Investigate and optimise zirconia/composite veneered dental prosthesis that combines the advantages of the two materials. Zirconia is a strong core to protect the underlying tooth, Composite an aesthetic, less abrasive material that is easily reparable.

Methods: Specimens shaped as discs and crowns (n=10) were fabricated of different materials and tested. The same samples were designed digitally and Finite Element Analysis (FEA) was carried out to compare with the mechanical testing.

Materials used:

- Zirconia: VITA In-Ceram YZ CUBES for CEREC.
- Composite: VITA VMLC and 3M ESPE Sinfony.
- Ceramic: VITABLOCS Mark II for CEREC and VITAVM9 veneering material.

Results and Conclusion: Based on testing conditions in-vitro, zirconia/composite laminates showed acceptable results when compared to single composite structures and zirconia/ceramic laminates. A good bond was observed between composite and zirconia that is observed even after sample fracture. Veneering zirconia seems to cause higher stress to the zirconia substrate in both Biaxial Flexural Strength calculations and FEA modelling. More tests need to be completed, taking into account the conditions in-vivo.

Table of Contents

List of tables:

List of Figures:

List of Equations:

1 Introduction:

Dental Technology is the science and art of designing and making oral appliances and restorations to restore function and improve aesthetic appearance. Such devices include crowns, bridges, dentures and maxillofacial appliances.

Dental devices are custom made from various materials depending on the requirements of the clinical application. They may be considered in several categories:

- Fixed prosthetics: permanently cemented restorations such as crowns, bridges, veneers, inlays and onlays.
- Removable prosthetics: removable complete or partial dentures.
- Orthodontic appliances: retainers, habit breaking appliances and space maintainers.
- Maxillofacial appliances: extra-oral appliances including: artificial (nose, ear and eye) and inter-oral appliances such as obturators, splints and surgical stents.
- Dental implants: Comprise fixture, abutment and restoration supported.

The use of replacement materials in dentistry started more than 6500 years ago using different materials such as beeswax (Bernardini et al., 2012). In modern dentistry metal alloys, composites and ceramics are the most significant materials for replacement of tooth tissue, the latter due to its aesthetic and physical properties being close to that of the natural tooth.

Ceramics, or dental porcelain have evolved since their inception in 1838, when dental porcelain that almost mimics natural teeth was produced by Elias Wildman (Southan, 1970). Crowns and bridges incorporating ceramic have become one of the most popular treatments to restore teeth. The Adult Dental Health survey in the UK in 1998 revealed that around 33% of elderly people had at least one crown and about 50% of the adults aged between 45 to 54 years old had one or more crowns (Smith and Howe, 2007).

Indications for using indirect restorations:

The most common oral disease treated with dental restorations is dental caries (Deligeorgi et al., 2001) but it is not the only indication for making indirect restorations. Other common reasons are:

- Badly broken teeth.
- Primary trauma.
- Tooth wear.
- Appearance: To change the shape, size or inclination of teeth.
- As part of another restoration.

Restoration materials should comprise certain critical characteristics such as (van Noort, 2007):

- Biocompatibility, with the surrounding oral tissues.
- Longevity and resistance to fracture.
- Appearance, to be aesthetically acceptable.

These restorations may be made from a range of materials using different processing routes. We can divide them in to the following categories (Shillingburg, 1997):

- Full metal crowns:
- Metal-Ceramic Crowns or Porcelain Fused to metal crowns (PFM).
- All-ceramic crowns.
- Resin-bonded crowns

Full metal crowns:

Full metal dental restorations were one of the first restorations used to restore teeth. These restorations are usually used for posterior teeth where aesthetics is not of concern. They allow less tooth preparation even in patients with a strong bite due to the strength of the material (Walmsley, 2007). They can be made out of different alloys with gold alloys being the most widely known. Gold alloys are favoured due to their biocompatibility and corrosion

resistance. These restorations are typically made using the lost wax technique. The principle of this technique is to design and shape the desired restoration in wax on a model of the prepared tooth. A mould is produced from the wax pattern and the alloy fills the mould. The primary disadvantage of such crowns and bridges is the lack of aesthetics (Smith and Howe, 2007).

Metal-Ceramic Crowns:

Also known with different names such as ceramco crown, porcelain veneer crown (PVC), porcelain fused to gold (PFG) and metal ceramic crown (Smith and Howe, 2007). Such restorations are made of two parts: the framework or substructure (metal) and veneer or overlay (ceramic) bonded to it. The metal framework is typically produced via a lost wax technique after producing a wax pattern manually, or using a CAD/CAM system to mill a 'plastic' pattern. Alternative production routes using CAD data are Direct Metal Laser Sintering (DMLS) and milling from a block (van Noort, 2012).

Typically a substructure is produced from metal alloys to support the ceramic from possible breakage under tensile and shear stress situation. Alloys are divided into three types depending on the percentage of noble metal contained such as gold, platinum, palladium, ruthenium and iridium (Anusavice and Phillips, 2003). The first type is high-noble alloys which contain a high percentage of noble metal elements (between 40% and 60%). The second type is semi-noble alloys which contain more silver and less percentage of gold or platinum, but still contain over 25% noble metals. The third type is base metal alloys which have high percentage of nickel or chromium (less than 25% of noble metal elements) (van Noort, 2007).

According to (Knosp et al., 2003), these 'Bonding' alloys differ to the alloys used for full metal crowns in that they must:

- Have a sufficient melting temperature to prevent sagging in the ceramic furnace when firing veneer.
- Produce an oxide layer to aid ceramic bonding.
- Be stiff to prevent the ceramic cracking.

- Have a coefficient of thermal expansion (CTE) that matches that of the veneering ceramic to eliminate any thermal stresses that may arise when cooling from high firing temperatures.

For veneering the metal framework, ceramic is used which is a powder mixture of oxide elements such as silica, alumina, potash and soda. Metal oxides are added to the mix before fusing it and quenching the molten mix in water, this procedure is called fritting. After that the mix is crushed to produce the ceramic powder (van Noort, 2007).

The veneering ceramics that are used for the different types of restoration all share some properties. There are different translucencies available, opaque, dentine and enamel (translucent). They may be produced via sintering, pressing or milling and most have similar mechanical properties.

The metal-ceramic restoration is one of the most versatile and reliable restorations because of their high successful rate (only 8% failure in 10 years) (Scurria et al., 1998), but yet, they are susceptible to failure (Ozcan, 2003). Some common PFM restoration failure causes are: CTE mismatch between substructure and veneer, inadequate built substructure and over thickened unsupported veneer (Diaz-Arnold et al., 1989). The other limitations relate to the limited aesthetic appearance of PFM restorations for anterior restorations in comparison to all-ceramic restorations (Bello and Jarvis, 1997).

All-Ceramic Crowns:

The new advances in dental materials and technology aim to conserve tooth structure by removing less tooth and often utilising advances in bonding of ceramic to teeth rather than relying on mechanical retention to achieve this. Adhesive bonding of materials has improved the reliability of all ceramic restorations. Early trials to make reliable all ceramic restorations started with techniques such as building ceramic on foil in 1903 by Dr. Land using feldspathic porcelain (Land, 1903). The same method was used in making veneers for Hollywood actors in the 1930's by Dr. Pincus (Pincus, 1938). Both resulted in crowns and veneers that were brittle with a high rate of fracture.

To overcome this weakness, feldspathic restorations are either bonded to the underlying tooth structure resulting in a resin bonded restoration, alternatively the feldspathic ceramic is supported by a tougher core material. Early attempts in making a stronger core material used alumina added to the feldspathic porcelain which resulted in the porcelain jacket crown (PJC) (van Noort, 2007). The PJC core is made by making a slurry and either adapting it to a die made of a refractory material before being fired or using a platinum foil to support the green state ceramic during firing.

Using a similar technique, Vita In-Ceram (VITA Zahnfabrik H. Rauter GmbH & Co.KG) can be produced as a substructure material for all-ceramic restorations. Three types of In-ceram were initially developed (Anusavice and Phillips, 2003):

- In-ceram Spinell for anterior single unit restorations.
- In-ceram Alumina for anterior and posterior restorations.
- In-ceram Zirconia for posterior bridges.

The materials are now processed using CAD/CAM rather than slip casting but the indications remains the same. Slip cast materials were fabricated by adapting a slurry ceramic on to a refractory die. This slurry is dried before an initial firing cycle is carried out. The result is a porous material that is infiltrated with glass to improve the mechanical and aesthetic properties (Deany, 1996).

An alternative route was to cast ceramic; in 1968 MacCulloch made denture teeth using glass ceramic for cookware (MacCulloch, 1968). The most significant result was the production of a castable ceramic system called Dicor in the 1980's, which was easy for technologists to adopt due to using the same wax up procedure used with conventional lost wax techniques (Shillingburg, 1997).

Hot-pressing technique is another method similar to lost wax technique but use a ceramic ingot which is heated before being pressed to form the substructure (Griggs, 2007). Common materials with this technique are those from Ivoclar (Ivoclar Vivadent AG) the IPS Empress (Leucite reinforced porcelain) and IPS Empress II (lithium-disilicate glass).

Newer materials were introduced later, mostly for all-ceramic frameworks, such as alumina and zirconia (van Noort, 2007). CAD/CAM has helped with the increasing popularity of zirconia based substructure materials by overcoming the difficulties of handling such materials manually. CAD/CAM's contribution in zirconia popularity as dental restorative material will be is discussed later.
On the other hand, veneer chipping off and framework fracture of all-ceramic fixed dentures are considered as major disadvantage unlike conventional ceramic-metal FPD's (Sailer et al., 2007).

1.1.1 General guidelines when making crowns:

Dental technologists manually fabricate most types of restorations, which is time demanding and skilled work. Increasingly dental restorations are being produced using computer-aided design and computer aided manufacturing (CAD/CAM), particularly for high strength materials.
There are two procedures when making dental restorations: clinical and laboratory procedures. The clinical procedure is similar for all types of crowns, differing in the amount of tooth reduction and design of margins for example. An impression of the prepared tooth is taken using an accurate impression material. The impression is then sent to the dental laboratory to design and make the restoration.

In the dental laboratory, materials and production methods differ from one type of crown and another.
The conventional method for making a PFM crown is to pour a class IV stone to produce a replica of the patient's teeth. Each prepared tooth (die) is separated to produce a sectional model making the die easier to remove and handle. The wax pattern of the substructure is produced and used to create a mould by pouring investment material around the pattern within a casting ring.
The wax is removed from the pattern and replaced with molten alloy. The sprues are removed using abrasive cut-off discs and the metal framework is prepared for ceramic application.
The ceramic is applied in stages (Shillingburg, 1997):

1. Opaque: used to block the metal, establish the bond and initiate the colour.
2. Dentine: used to build the body of the crown and provide the colour
3. Enamel: used to give translucency along the incisal edge and to the mesial and distal aspects of the crown.

Finally staining colours are added to the crown to mimic its natural adjacent teeth and glaze is added to give it the natural tooth shine and seal porosities.

Production of High Strength Ceramic Crowns differs by replacing the metal substructure with a ceramic core, which is usually veneered with a feldspathic porcelain in the same way done with PFM crowns. Those ceramic substrates are mostly made using CAD/CAM or by injection moulding technique.

For a less destructive tooth preparation and areas with low occlusal load and aesthetic demand like in the anterior, resin bonded crowns may be used. Reliability of such thin crowns rely mostly on resin bonding since no strong core is present (Blair *et al*., 2002).

2 Review of literature:

2.1 CAD/CAM:

Computer Aided Design/Computer Aided Manufacture (CAD/CAM) or Rapid Prototyping is a technique that has been used in industry since the 1950s, mainly for large simple shapes, unlike dental restorations but the same system could be used to create dental crowns (Rekow, 2006). CAD/CAM was first used in the dental field in the 1970's but with limitations, according to (Miyazaki et al., 2009):

- CAD/CAM camera resolution was limited and difficulties in identifying the prepared tooth margin as well as antagonist and proximate teeth were experienced.
- Milling restorations with sharp edges and thin margins was unreliable due to chipping of the ceramic material.
- The CAD/CAM milling units were too large and expensive for dental laboratories, compared to conventional production routes.
- Designing and producing restorations was a lengthy process. Early design software was rudimentary and not suitable for designing sophisticated tooth anatomy.

CAD/CAM in the dental field really started in the seventies and eighties when Duret, Moermann and Andersson were all developing CAD/CAM systems.

Duret was one of the early developers of CAD/CAM in dentistry making restorations using different systems with simple occlusal surfaces from as early as 1971 leading to the Sopha system. That system contributed later in the development of CAD/CAM in the field of dental technology (Duret and Preston, 1991).

Moermann developed the CEREC CAD/CAM system focusing on using CAD/CAM in the dental clinic. The prepared tooth was measured inside the patient's mouth using an optical camera. The data was then used in the CAD/CAM machine that designs and mills the ceramic restoration while the patient waits (Mormann et al., 1989).

The Procera system, developed by Andersson in early 1980's, tackled the production process differently by having one centralised production centre where electronic data could be sent (Andersson and Oden, 1993).

As a result CAD/CAM systems may be considered as chair-side, lab-based or milling centre. Where a milling centre is involved, the scanning and design procedure is carried out within the dental laboratory, and the data sent to an external centre for milling or sintering. Chair side systems tend to be limited to single unit restorations, whereas laboratory CAD/CAM systems allow full models to be scanned and long span restorations to be produced.

There are three main components common to all CAD/CAM:

- Data acquisition; using a optical or laser scanner,
- Restoration design; using CAD software
- Restoration production; using CAM software and a milling, sintering or 3d printing unit.

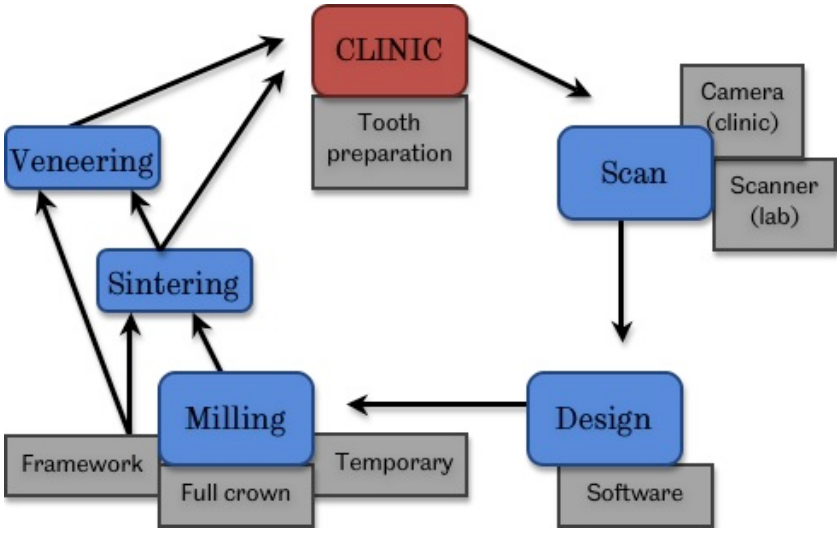


Figure 1: Steps when using CAD/CAM in fabricating restorations.

As shown in (Figure 1), the CAD/CAM system works in the following steps: scan, design and milling (fabrication).

Data acquisition in chair side systems involves the use of an Intra-oral camera whereas laboratory based systems use a scanner that scans either the model or the impression of the teeth.

Data is sent to specially designed software, where it may be manipulated and the restoration deigned. CAD software enables manipulation of restoration

design to build-up, smooth and cut the restoration design, mimicking the conventional method by hand.

Once complete the design is sent to the milling or sintering unit. In milling units a block or blank of material is fixed and reduced by a conventional milling process. Materials that are milled range from metals such as titanium, to pre-sintered zirconia and fully compacted glass ceramics. Alternatively a wax substitute may be milled to enable conventional casting methods to be used.

An alternative production route is to send the design to an external processing centre to be milled, or produced by additive manufacture. Unlike the subtractive manufacturing in conventional CAD/CAM systems, additive manufacturing uses different techniques to build up the desired shape instead of carving it out of a block. Examples of additive technique: selective electron beam melting, laser powder forming and inkjet printing (van Noort, 2012).

CAD/CAM can save time in the production of high strength materials such as alumina and zirconia that are difficult to produce using conventional techniques. Eventually these will increase the life of the restoration in the mouth (Strub *et al.*, 2006).

Problems with CAD/CAM that have been highlighted by Trost et al., 2006 are:

- The scanner or oral camera sometimes fails to detect the finish line of the prepared tooth that leads to a restoration with an open margin, which causes gingival irritation.
- The high cost of equipment and training for both dentists and technicians.

Dental CAD/CAM systems are starting to expand all around the world. Different systems are available from different companies. These systems may share the same principle but are different in the technique and materials.

CEREC system: Was one of the first dental CAD/CAM systems, produced by Sirona in 1985. Many updates and new models have been released to date (Figure 2). This system can fabricate restorations in just one hour (Miyazaki *et al.*, 2009). CEREC offers different materials to be used for crowns and bridges

such as feldspar ceramics, glass ceramics, high-performance polymers and composites for temporary crowns and bridges.

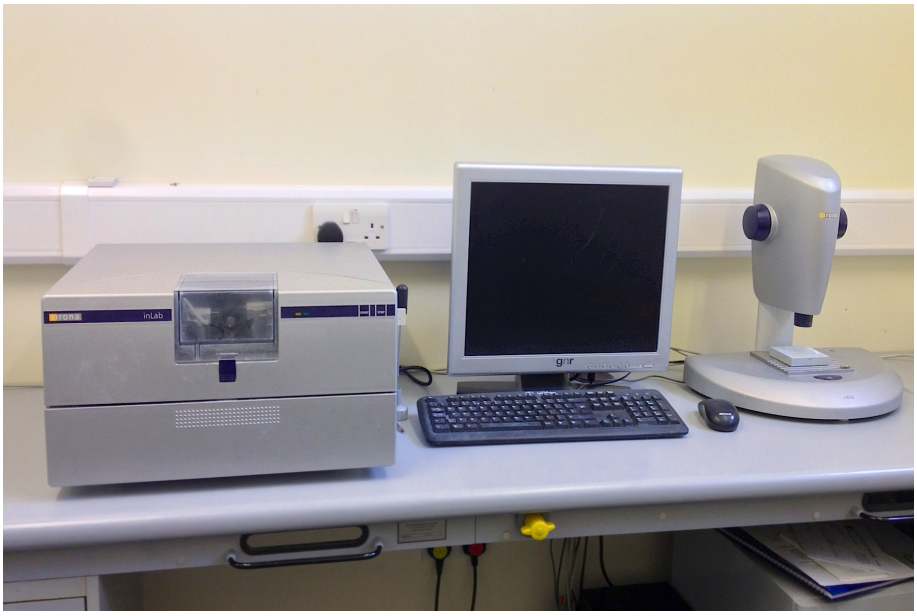


Figure 2: CEREC milling unit (left) and acquisition unit (right).

Procera system: In this system the scanner is purchased alone without the milling unit. It is suitable for laboratories that cannot afford to buy a whole CAD/CAM system. The die is scanned in the laboratory and designed using Procera software. The data is then sent to the lab electronically where the restoration framework is milled and sintered. Then completed framework is sent back to the laboratory by mail (Miyazaki *et al*., 2009) to be veneered and finished. Restorations in this system can be made from different kinds of materials such as zirconia used in making crowns, bridges and implants. Zirconia is known with its high flexural strength, 1200 MPa. Other material is alumina used for anterior crowns, bridges and veneers with a flexural strength of 700 MPa. Finally, titanium is used for making implant bridges (Nobel Biocare Services AG, 2012).

Cercon system: This system is produced by DENTSPLY Ceramco company. It is consisted of these three parts: Cercon Eye, Cercon Art and Cercon Brain. Cercon Eye scans prepared stone models using a three-camera system and a laser (DENTSPLY, 2012). The Cercon Art is software to design the restoration and after the design, the data is sent to the Cercon Brain for milling. Zirconium

oxide blocks are used for milling crowns and bridges. Dental labs can also use the Cercon Eye and Cercon Art and receive the zirconium oxide restoration by mail from DENTSPLY milling centre after sending the design without the need of purchasing the Cercon Brain (Beuer et al., 2008).

KaVo Everest system: Is produced by KaVo Dental GmbH and it consist of four parts: Everest scan pro, Everest engine, Everest therm and Everest elements. This in-lab 5 axes system is capable of milling metal and ceramic materials along with other materials such as (KaVo Dental GmbH, 2012):

- Cam select (nickel-free cobalt chrome).
- Everest ZS (pre-sintered zirconium oxide).
- Everest ZH (sintered pressed zirconium oxide).
- IPS. e.max. CAD (innovative lithium disilicate glass ceramic).
- VITABLOCKS mark2 (feldspar ceramic).
- Everest C-Temp (high-performance polymer plastic).
- Everest C-Cast (Polymethylmethacrylate) used to be invested.
- Everest T (Grade 2 pure surgical titanium).
- Everest G (Leucite-reinforced glass ceramic).

2.2 Zirconia

Zirconium oxide (ZrO_2), partially stabilized zirconia or simply zirconia, is considered one of strongest ceramic core materials with a BFS of about 1000MPa (Guess *et al.*, 2008) and a fracture strength peaking around 2000N (Ozcan *et al.*, 2011). It is also one of the most recently developed materials used in dentistry and other medical fields. It is widely used in industry in different applications, such as machinery parts, furnace elements, and aerospace industry (Lee and Rainforth, 1994). Due to the material's biocompatibility it is used widely in the medical field in making hip and joint replacements. Until 1997, about 300,000 hip joints made of zirconia in Europe and USA were implanted (Ghevalier et al., 1997).

Zirconia can be found from natural sources such as zircon and baddeleyite (Figure 3). Zirconium oxide ZrO_2 was first recognised in 1789 a German chemist called Martin Heinrich Klaproth (Vagkopoulou et al., 2009). As a biomaterial, zirconia was first presented in 1969 by Helmer and Driskell as a substitute for titanium and alumina in making femolar heads (Helmer and Driskell, 1969).

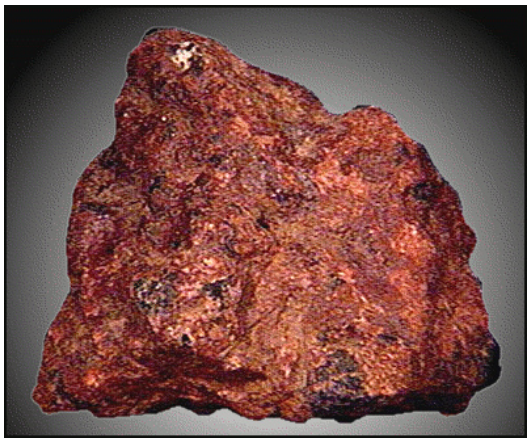


Figure 3: Baddeleyite. (Source: www.wpclipart.com)

Zirconia comes in a monoclinic phase at room temperature and can transfer to tetragonal then to cubic phases above 1170 and 2370˚C respectively. On cooling, phase transformation occurs from tetragonal to monoclinic causing expansion of about 3 to 4%. This increase in size prompts stresses and eventually causes zirconia to fracture (Piconi and Maccauro, 1999). Its

capabilities can be improved by small changes in its composition or subjecting it to heat treatment; an example is stabilizing of zirconia with different oxides. This is essential when producing a manufacturing material. An example is to stabilize at room temperature by incorporating yttrium oxide (Ozcan *et al*., 2011). Other stabilizers can be used such as Calcium and Magnesium.

There is concern regarding zirconia's biocompatibility and the possibility of forming microcracks caused by something called low temperature degradation LTD (Swab, 1991). This phenomenon reduces zirconia's strength and is increased in wet conditions elevating the tetragonal to monoclinic phase transformation causing microcracks (Piconi et al., 1998). A possible way to overcome such a problem is to ensure full coverage of zirconia substructure from oral fluids and environment (Koutayas *et al*., 2009).

Another phenomena involves reverse transformation from tetragonal to monoclinic phase called transformation toughening. An expansion is associated with the transformation acting in favour of closing cracks (Papanagiotou et al., 2006).

Sintering is an important step in processing zirconia; the zirconia is raised to a high temperature (about 1500°C) having the furnace raising temperature in a steady rate. The process results in pores being reduced between particles (Figure 4), increasing strength but results in shrinkage of the structure (Lee and Rainforth, 1994).

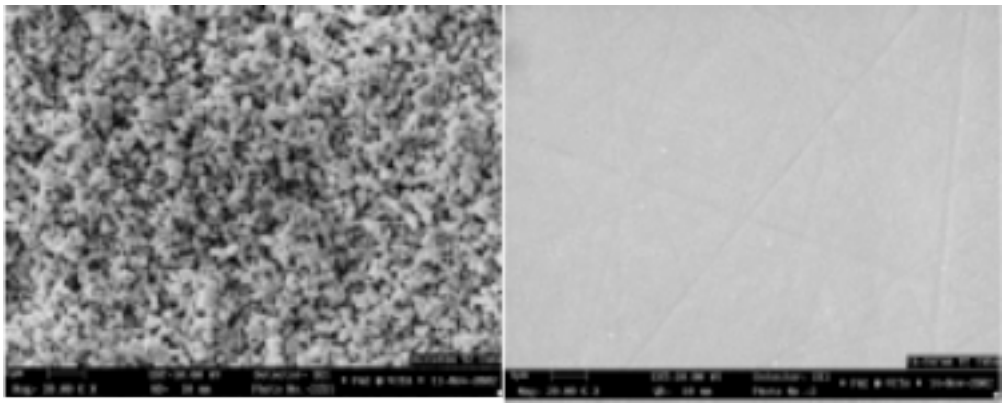


Figure 4: Microscopic image of pre-sintered zirconia (left) and and after sintering (right). (Source: VITA In-ceram YZ manual).

2.2.1 Zirconia in Dentistry:

Zirconia can be used to replace metal in the conventional PFM restorations. It has adequate mechanical properties of metal with its outstanding flexural strength but there are some drawbacks. It is considered to be a non-etchable ceramic surface that may effect restoration fit in the patients' mouth and therefore leakage (Ozcan *et al*., 2008).

Zirconia has become easy to fabricate since the introduction of CAD/CAM although other techniques for fabricating zirconia restorations (used with cerium-tetragonal polycrystal), are available using an additive technique called electrophoretic deposition (Koutayas *et al*., 2009).

As a result zirconia based frameworks and restorations are increasing in popularity such that demand is growing at approximately 12% annually (Chevalier, 2006). Zirconia material is presented in the form of pre-sintered pressed CAD/CAM blocks or discs (Figure 5), the blocks are produced in different sizes depending on the restoration kind and number of units milled.

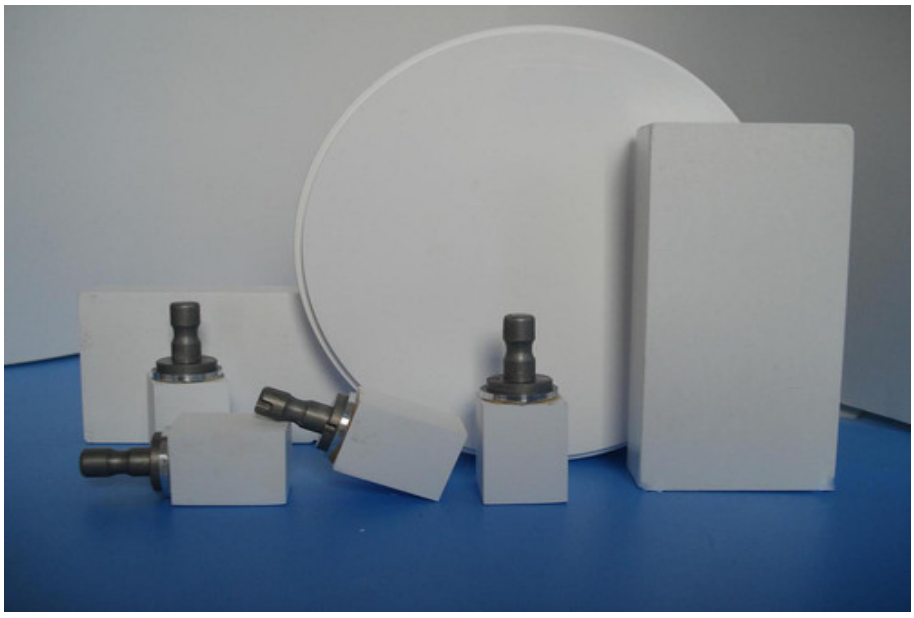


Figure 5: Different sizes of zirconia blocks and disc. (With permission from Bloomden Bioceramics Co.,Ltd).

Milled restorations always require some manual finishing including the framework being separated from the remaining block and the remaining rough surface can be finished using a diamond bur. The framework is heat treated (sintered) in a furnace to 1530°C for 2 hours before gradually cooling to room temperature. Sintering causes a strong bond between the particles

and closes the pores between them (Lee and Rainforth, 1994) which causes the framework to shrink for about 20%. This shrinkage is compensated for in the software and calculated when designing the restoration by milling an over sized framework (Figure 6). The sintered zirconia framework is extremely tough (compared to pre-sintered ones) with an opaque white colour.

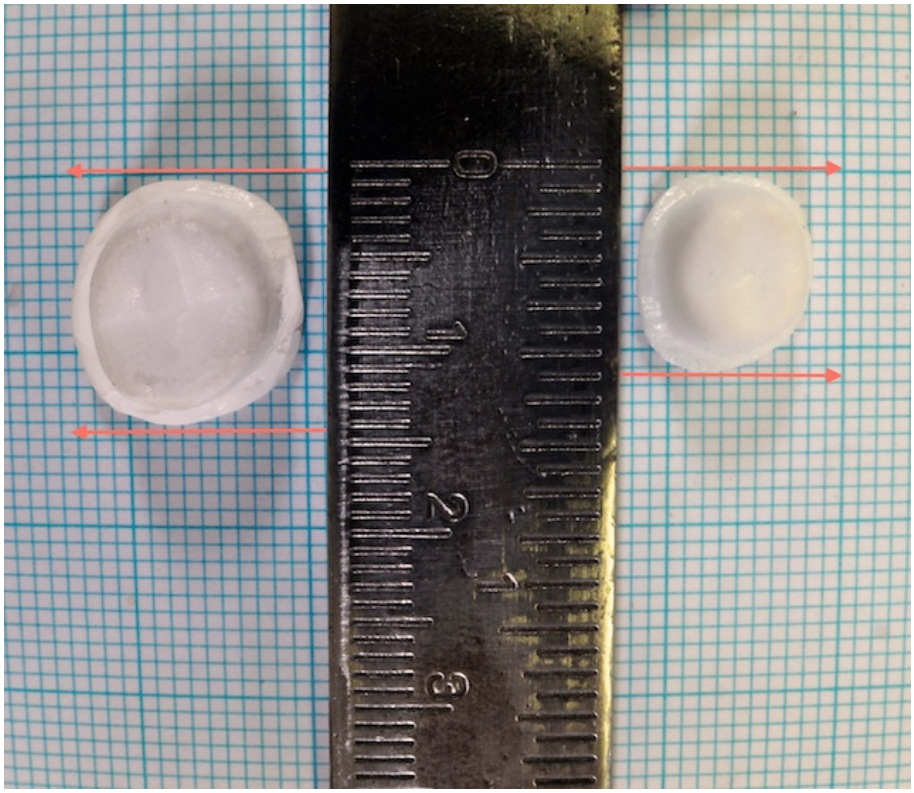


Figure 6: Pre-sintered coping (left) and sintered coping (right) with about 21% change in volume.

In a study of posterior restorations lasted 3 years, Suarez stated that zirconia ceramic can act as a dependable restorative material in clinic (Suarez et al., 2004). Zirconia frameworks are considered for both anterior and posterior application unlike the rest of all-ceramic materials, which are only recommended for use in anterior (Raigrodski, 2004).

Zirconia is usually veneered with feldspathic porcelain via sintering or pressing. The ceramic veneer provides greater aesthetic wear properties but is susceptible to chipping (Koutayas *et al*., 2009). Such veneer chipping and fracture is considered as a common drawback in zirconia dental FPD's (Sailer *et al*., 2006), (Manicone et al., 2007) and (Hammond, 2009). Another possible drawback is that such veneers may cause wear of the opposing teeth (Stawarczyk *et al*., 2011). Also replacing damaged restoration requires more

tooth reduction which in the long run may cause remaining tooth to break of or even be extracted (Sharif *et al*., 2010).

Techniques for ceramic repair using light cure composite on the ceramic after various chemical and, or mechanical surface treatments have been discussed by (Kim *et al*., 2005). Kim *et al* outlines that the repair is mostly done combining shot-blasting, hydrofluoric etching and silanes on the ceramic surface to establish a durable bond to repairing composite. Kim showed that most studies reviewed concerned feldspathic ceramic rather than alumina and zirconia ceramics, which are not adequately responding to such techniques, as stated by other studies they reviewed.
Table 1 shows types of zirconia used in fabricating dental prosthesis and their operating systems.

Table 1: Typical zirconia used in fabricating prostheses and their systems from companies' websites.

System	Material	Company
densir®	Hot Isostatic Pressed Yttrium Oxide Stabilized Zirconium Dioxide blocks milled at the company's milling centre and sent to the dental technologist to be veneered.	Cad.esthetics AB, Sweden
ceramill zi	Pre-sintered Y-TZP zirconium-oxide	Amann Girrbach AG, Austria
Cercon® Zirconia	Yttria-stabilized tetragonal zirconia which can be milled either inlab or at the company's milling centre.	DeguDent GmbH, Germany
DentaCAD	Hint-ELs ZrO2 HIP/ W/ G is the product name of the zirconium dioxide used with the DentaCAD system.	Hint-ELs® GmbH, Germay
Kavo-everest	Pre-sintered, yttrium-stabilised zirconium oxide (Everest ZS) and sintered, yttrium-stabilised pressed zirconium oxide (Everest ZH)	KaVo Dental GmbH, Germany
GC Aadva CAD/CAM	Aadva Zr Blocks (Yttria stabilized zirconia) are milled to produce abutments and copings at the company's milling canters.	GC Corporation, Japan
CEREC®	VITA In-Ceram YZ yttrium-stabilized Zirconia comes as a pre-sintered block and milled using in-lab milling machine.	VITA Zahnfabrik H. Rauter GmbH & Co, Germany

2.3 Composites:

Composites have been used in dentistry for more than forty years (Bowen, 1962) and in that time have become the most widely used material for direct restorations, mainly due to their aesthetic capability when compared to amalgams. They are defined as a mixture of two or more different parts to achieve the desired features (Ferracane, 1995). The material is versatile and may be used both as a direct restoration material and as a laboratory produced indirect restoration. Prior to the introduction of composites, clinicians were limited to using materials such as amalgam or acrylic resins, both of which do not fulfil the ideal requirements of a restorative material (Craig and Powers, 2002). Drawbacks such as marginal gap, discoloration and relative weakness of acrylic resins (Fusayama et al., 1971), whereas amalgam causes concern regarding the mercury in its composition and the destructive nature of the cavity preparation required (Hengchang et al., 1990) (Bates, 2006) .

Composites excel with their aesthetic life-like shades, relative ease of use and repair, good wear characteristics and minimal preparation requirements (Shahdad and Kennedy, 1998) and (Rathke *et al*., 2009).

Composites are made of three parts (Peutzfeldt, 1997):

- Organic matrix: a fluid monomer that is transformed to a rigid polymer by a radical addition reaction. These monomers are usually related to metacrylate monomers such as Bis-GMA and UDMA (bisphenol A diglycidylether methacrylate and urethane dimethacrylates) are examples of widely used monomers.
- Inorganic filler: which improves the composite's strength and hardness properties. The filler also impacts on the optical and thermal characteristics.
- Coupling agent: are silanes that bond filler to matrix in order to get a mechanically reliable composite.

An activation process is required to initiate the polymerisation of the composite. Composites can be chemically activated, mixing (powder/paste)

and liquid, or light cured using an external light source. Usually in the chemical activated system, the powder or paste contains the fillers whiles the liquid activates the reaction. The paste contains 1% of peroxide initiator and the liquid 0.5% of tertiary amine activator.

Ultraviolet light activated composite has an initiator, typically benzoin methyl ether. While in the visible-light cured composite, the initiator is a mixture of amine and dikitone (McCabe and Walls, 2008).

Light cure activation, developed in the 1970's (Mount and Hume, 1998), is commonly used mainly due to longer working time and on-demand hardening (Walmsley, 2007). Light curing can be affected by the distance between light source and composite surface, and the shade of the composite can affect the depth of cure. UV light curing systems had few drawbacks mainly the depth of cure, causing the production of the of visible light curing (VLC) system (Alvim *et al.*, 2007).

Curing units produce light with a wavelength of around 470nm, the point where camphoroquinone absorbs the light and initiates polymerization of the composite (Alvim *et al.*, 2007). Available as a desktop box or a hand held unit, both require cooling, a filter to eliminate harmful spectrum and a timer. In both types, halogen bulbs can be used. A newer light source is the light emitting diode (LED), which is considered convenient since no heat is generated, and light spectrum can be customised to specific range (McCabe and Walls, 2008).

The British Standards Institution has classified dental polymer-based restorative materials (BS EN ISO 4049-2009) into three classes as detailed in (Figure 7) below.

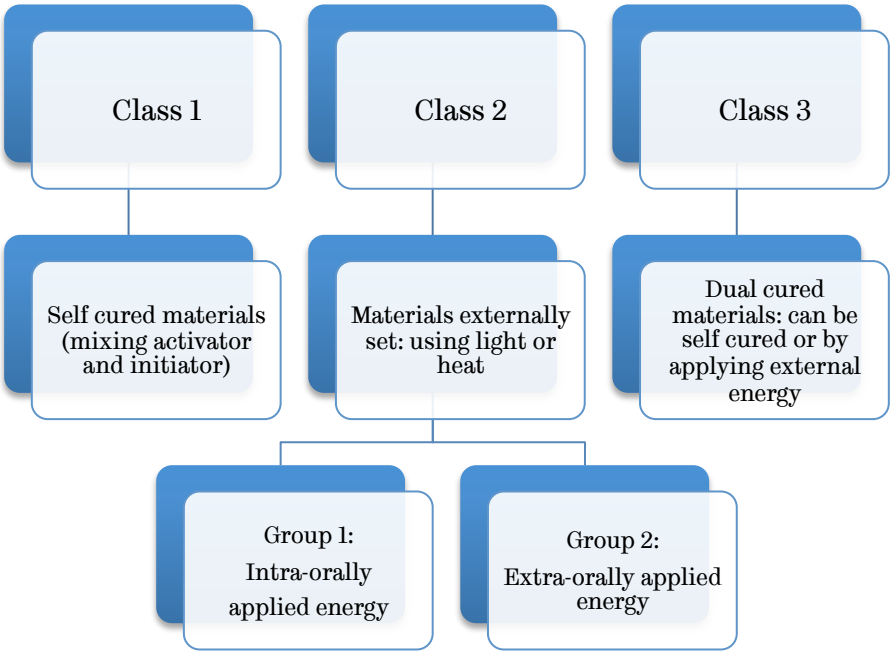


Figure 7: Classification of dental polymer-based restorative materials (BS EN ISO 4049:2009) according to British Standard Institution.

Researchers indicated different composite classifications depending on various criteria. One classification of composites depending on the fillers is: macro filler, micro filler and hybrid composite (Lutz and Phillips, 1983).

Another way of classifying composites is the one used by companies were they class composites into: flowable composites and packable composites. Choosing which one depends on the patient's case and the application feature (McCabe and Walls, 2008).

2.4 Bonding methods:

In restorative dentistry, bonding two materials together or to the remaining tooth tissue is essential to the success of restoring teeth with conservative preparations for composite to minimal preparation applications such as veneers or minimal preparation bridges that rely on micro-mechanical features (Smith and Howe, 2007).

Bonding to enamel, dentine, resin and other materials such as ceramic and metal has become of great interest to material developers, particularly with the advent of high strength ceramic materials.

According to (McCabe and Walls, 2008), bonding can be characterised to:

1. Micro-mechanical: an example using acid-etching technique.
2. Chemical adhesion: an example using coupling agents and cements.
3. Complex mechanism: including wetting, penetration and forming a layer bonding two surfaces.

Examples of such bonding in dental restorations:

Micro mechanical retenion in non-precious metals can be achieved by etching the metal fitting surface, or using a complex method of bonding by forming silica layer prior using silane coupling agent (McCabe and Walls, 2008). In ceramics, silanes shows better results bonding in ceramics than metals and enhances the composite bond to ceramic by 25% (Matinlinna et al., 2004).

An example of silane commonly used in dentistry is 3-methacryloyloxypropyltrimethoxysilane (Figure 8) (Aboushelib *et al*., 2008).

Another bonding enhancement in dentistry is something called primer, an example of a primer is the one used to cover wood before adding fixative (van Noort, 2007). In dentistry, primers are silanes that act as bond promoters between different materials, organic or inorganic. Besides dentistry, silanes have been used in other applications for around 40 years (Matinlinna et al., 2004). These primers are usually an alcohol-based solution with different component composition. Recent modifications to primers include the addition

of an etching component that allows the solutions to bond and etch the surface at the same time (McCabe and Walls, 2008).

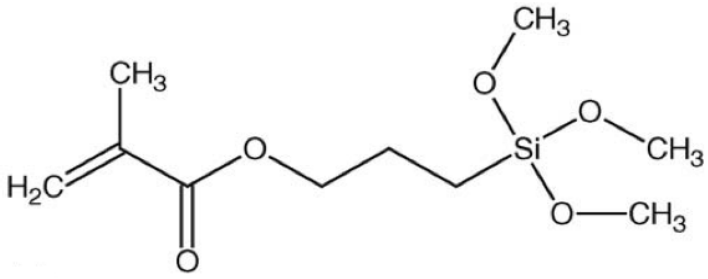


Figure 8: 3-methacryloyloxypropyltrimethoxy silane. Source: (Matinlinna *et al.*, 2006).

2.4.1 Bonding composite to zirconia:

There have been many researchers trying different techniques and methods to establish a reliable bond to oxide ceramics and in particular between these two materials. Using bonding resins seems essential for a more durable bonding between composite and zirconia, depending on various studies (Ural *et al.*, 2010).

There is some debate on certain methods that they may affect the properties of bonded material. For ceramics such as zirconia and alumina, shot-blasting them with Al_2O_3 is considered an effective way to bond to them but some suggest that this causes micro cracks in the surface resulting in weakness of the core material (Ural *et al.*, 2010). Conversely, roughening the zirconia surface can increase the bonding surface and promote the bond by interlocking with the other material (mechanical bonding). (Guazzato et al., 2005), (Vagkopoulou et al., 2009) and (Qeblawi et al., 2010) showed the effect of zirconia surface treatment on flexural strength and shear bond strength on a resin cement. The flexural strength increases when the zirconia surface was mechanically treated. Other workers show that roughening the surface increases the bonding area and thereby promote bonding (Ozcan *et al.*, 2008).

Metal primers were used with zirconia as bond promoters after shot-blasting the surface but the strong bond may not stand wet conditions (Ozcan *et al*., 2008).

Micro-mechanical retention cannot be created on zirconia surface using hydrofluoric acid due to the glass-free structure of zirconia making it a non-etchable material. Attempts have been made to change the zirconia surface characteristics by airborne particle abrasion prior silanisation, but this air-borne abrasion has been criticised for possibility of causing crack growth in zirconia (Ozcan *et al*., 2008). Effective bonding can be achieved using silane coupling agents which lowers the surface tension, wet and raise the surface energy (Matinlinna *et al*., 2006).

Metal primer was recommended as a cheap and easy to apply method to promote zirconia bonding after air-borne abrasion. They seem to exhibit a strong bond in dry environments but some debate its performance in wet conditions (Ozcan *et al*., 2008).

Table 2 in page 36 summarises the different bonding methods explored by other workers.

Simple in-lab bonding trials between composite (VITA VM LC) and zirconia (VITA In-Ceram YZ) to create a laminate disc was summarised table 3. The best method was observed when combining micromechanical bonding (by shot-blasting) and chemical bonding (universal primer) to bond composite to zirconia as stated in literature by (Kern *et al*., 2009), (Yun *et al*., 2010), (Yang *et al*., 2010) and (Attia *et al*., 2011).

A different method was to bond composite to zirconia using airborne particle silica coating system (3M Rocatec system)*. The system claims to form a silica layer by shot-blasting the surface using alumina particles coated with silica. Sintered zirconia discs were blasted in 40-psi pressure for about 5 seconds in about 5 cm distance and cleaned with oil-free air. After that 3M ESPE Si (silane) was applied on the blasted surface and left for 10 minutes prior building composite starting with opaque than dentine. This laminate disc de-

*Special thanks to Mr.Stephen H Nelson from 3M for providing Rocatec system.

bonded even before trying to test the specimens. The same procedure was done using different composite (3M ESPE Sinfony) where it showed a slightly better bond but not enough to last for testing its biaxial flexural strength. There were studies that stated silica coating did not improve bong to zirconia compared to alumina (Kern and Wegner, 1998).

Table 2: Bonding composite to zirconia trials done by others.

Bonding method	Description	Result	Done by
Applying directly	After shot-blasting	Primary bond which failed afterwards	Kern M. and Wegner SM. 1998
Adding silane	Espe-Sil, 3-methacryloyloxypropyl trimethoxysilane in isopropanol before applying composite	Primary bond which failed afterwards	Kern M. and Wegner SM. 1998
Tribochemical silica coat	After cleaning by shot-blasting with 100 μm Al_2O_3 at 2.5 bars (0.25 MPa). It is shot-blasted with special silica particle containing 110μm Al_2O_3 (Rocatec-Plus powder) and cleaned in isopropanol ultrasonically. The surface is silanated (Espe-Sil) prior to the application of composite.	Primary bond which reduced obviously	Kern M. and Wegner SM. 1998
Acylizing	Applying acryle (Kevloc oven method) before adding the composite	Primary bond which failed afterwards	Kern M. and Wegner SM. 1998
Panavia EX	A chemical cured phosphate monomer modified resin composite was applied directly to the shot-blasted ceramic surface.	Strong bond	Kern M. and Wegner SM. 1998
Paniva 21 EX	Chemical cured with MDP modified resin composite was applied directly to the shot-blasted ceramic surface.	Strong bond	Kern M. and Wegner SM. 1998
Dyract Cem	Chemical cured polyacid modified resin composite was used directly on the shot-blasted ceramic surface	Primary bond which reduced obviously	Kern M. and Wegner SM. 1998
No air abrasion and no primer	------	Debonded eventually after stored in water	M. Kern, A. Barloi and B. Yang 2009
No air abrasion with primers	- Metal/zirconia primer (MZP) - Alloy primer (AP) - Clearfield ceramic primer (CCP)	Enhanced primary bond, which eventually weakened after storage.	M. Kern, A. Barloi and B. Yang 2009
After air abrasion with groups AP and CCP	Air abrasion with 0.05 MPa or 0.25 MPa with groups contained: 10-methacryloyloxy-decyldihydrogenphosphate monomer in AP and CCP primers	Higher tensile bond strength than MZP primer group containing phosphoric acid acrylate	M. Kern, A. Barloi and B. Yang 2009

Table 3: Bonding VITA YZ disks to VITA LC trials

Bonding material	Description		Result
No material used	Applying Vita LC directly out of the syringe to the YZ disks with the assist of solfane paper and a flat piece of glass to flatten the composite then light cure it for 4 min using (Complex Lux S 8, Pressing Dental)		A very weak bond between composite and zerconia disk.
VM9 EFFECT BONDER	YZ disk bonding side is washed with Vita VM9 effect bonder (powder & liquid) and fired (table) and the Vita LC directly adapted to the EFFECT BONDER surface with the assist of solfane paper and a flat piece of glass to flatten the composite then light cure it for 4 min using (Complex Lux S 8, Pressing Dental)		Very poor primary bond
VM9 EFFECT BONDER with rough surface	Same as previous procedure but before firing EFFECT BONDER a thin layer of the EFFECT BODER powder is sprinkled on it to give rough surface after firing and to initiate mechanical bonding.		Stronger primary mechanical bond was initiated.
Shot-blasting & VM9 EFFECT BONDER	A fired layer of EFFECT BONDER on a YZ disk was shot-blasted with 50μm and LC was adapted on top of it and light cured it for 4 min using (Complex Lux S 8, Pressing Dental)		Very poor primary bond
Vita LC opaque	LC opaque (powder & liquid) were mixed according to manual and, with a brush, applied on bonding surface of the YZ disk and light curd for 6.00 minutes using (Complex Lux S 8, Pressing Dental) and composite is placed on top of the opaque as done before.		Very poor primary bond
Vita LC bonder	After applying LC bonder to YZ disc, it was kept under 60w lamp at a distance of about 10 cm for 20 minutes and composite was applied as done in previous trials.		A poor primary bond but better than the one with LC opaque
Roughing surface before sintering	Shot-blasting bonding surface (50μm alumina) with a fine mesh hold against it and sintered as done before.	Applying composite with the use of VITA modelling liquid and a plastic spatula.	Very poor primary bond
		Applying composite after firing a rough layer of effect bonder, using modelling liquid and plastic spatula.	Stronger primary mechanical bond was initiated.
Universal primer (Monobond plus)	Zirconia bonding surface was shot-blasted with 50μm alumina and a layer of primer was applied using a disposable brush and left for 10 minutes before building composite layer.		Strongest primary mechanical bond was initiated.

2.5 Biaxial Flexural Strength:

Mimicking the conditions that a restorative material is subjected to in the human mouth in order to examine restoration behaviour is difficult to achieve and as such there is no method used in vitro that can fulfil all of the necessary requirements (Anusavice et al., 2007).

The BFS is an evaluation that combines three measurements (tensile, compressive and shear stresses) at the same time. When testing ceramics such evaluation is preferred with these brittle materials (Anusavice and Phillips, 2003).

BFS testing is advantageous over the three-point test, which is not preferable due imperfection of results caused by defects in sample edges and therefore, shaping samples as discs can reduce disparity in results (Wagner and Chu, 1996). Such evaluation can be achieved by applying load using a ball shaped indenter on disc samples supported by a rounded metallic ring (Johnson *et al.*, 2000). The max strength (load at fracture N) recorded in the machine in used in an equation to work out the biaxial flexural strength BFS in MPa. Different equations were found for calculating the BFS of single layered discs, some include the Poisson's ratio of the material (Pagniano *et al.*, 2005) and some do not (Piddock *et al.*, 1986).

For the monolayer samples, BFS was calculated according to the following Equation A (Piddock *et al.*, 1986)

$$\sigma_{\max} = \frac{P}{h^2}[0.606\log_e(a/h) + 1.13]$$

Equation A

However, for bi-layered or multi-layered samples other methods for calculating BFS have been used by different researchers. These differ to the single layer equation by involving the elastic modulus and Poisson's ratio.
(Hsueh and Kelly, 2009) in a paper titled: "Simple solutions of multilayered discs subjected to biaxial moment loading" used Equation B for bi-layered samples:

$$\sigma_T = \frac{(6E_2M/(1-v_2))\left[E_1t_1^2/(1-v_1)+E_2t_2^2/(1-v_2)+2E_1t_1t_2/(1-v_1)\right]}{\left[E_1t_1^2/(1-v_1)+E_2t_2^2/(1-v_2)\right]^2+4E_1E_2t_1t_2(t_1^2+t_1t_2+t_2^2)/((1-v_1)(1-v_2))}$$

$$\sigma_B = \frac{(-6E_1M/(1-v_1))\left[E_1t_1^2/(1-v_1)+E_2t_2^2/(1-v_2)+2E_2t_1t_2/(1-v_2)\right]}{\left[E_1t_1^2/(1-v_1)+E_2t_2^2/(1-v_2)\right]^2+4E_1E_2t_1t_2(t_1^2+t_1t_2+t_2^2)/((1-v_1)(1-v_2))}$$

Equation B

With the value of M and v equals respectively:

$$M = \frac{-P}{8\pi}\left\{(1+v)\left[1+2\ln\left(\frac{a}{c}\right)\right]+(1-v)\left[1-\frac{c^2}{2a^2}\right]\frac{a^2}{R^2}\right\}$$

$$v = \frac{v_1t_1+v_2t_2}{t_1+t_2}$$

Where: E= elastic modulus, v= Poisson's ratio, P= fracture load, t_1= thickness of bottom surface, t_2= thickness of top surface, a=radius of ring support, c= radius of piston and R= radius of sample.

From Hsueh and Kelly equation B, an equation was derived to calculate the BFS for single layered disc such as that:

$$\sigma_T = -\sigma_B = \frac{6M}{(t_1+t_2)^2}$$

Equation C

Using equation C to calculate BFS for monolayers gave irregular results to the known BFS of tested materials. This gave the reason to look for different bi-layed BFS equations.

A further equation was used to calculate BFS for Bi-layered by Hsueh *et al*., 2006 in their paper titled: Analyses of multilayered dental ceramics subjected to biaxial flexure tests. In this paper they requested to replace the radius of the piston value with one third of sample thickness when using ball on ring method, which was not mentioned in the previous article and led to the assumption to piston on ring configurations. The BFS equations for bi-layered structures are:

$$\sigma_{r1} = \sigma_{\theta 1} = \frac{-PE_1(1+v)(z-z_n^*)}{8\pi(1-v_1^2)D^*} \times \left[1 + 2\ln\left(\frac{a}{c}\right) + \frac{1-v}{1+v}\left(1 - \frac{c^2}{2a^2}\right)\frac{a^2}{R^2}\right]$$

$$(0 \le z \le t_1)$$

$$\sigma_{r2} = \sigma_{\theta 2} = \frac{-PE_2(1+v)(z-z_n^*)}{8\pi(1-v_2^2)D^*} \times \left[1 + 2\ln\left(\frac{a}{c}\right) + \frac{1-v}{1+v}\left(1 - \frac{c^2}{2a^2}\right)\frac{a^2}{R^2}\right]$$

$$(t_1 \le z \le t_1 + t_2)$$

Equation D

Knowing that the neutral surface position and flexural rigidity, respectively, equals:

$$z_n^* = \frac{\frac{E_1 t_1^2}{2(1-v_1^2)} + \frac{E_2 t_2^2}{2(1-v_2^2)} + \frac{E_2 t_1 t_2}{1-v_2^2}}{\frac{E_1 t_1}{1-v_1^2} + \frac{E_2 t_2}{1-v_2^2}}$$

And

$$D^* = \frac{E_1 t_1^3}{3(1-v_1^2)} + \frac{E_2 t_2^3}{3(1-v_2^2)} + \frac{E_2 t_1 t_2 (t_1 + t_2)}{1-v_2^2} - \frac{\left[\frac{E_1 t_1^2}{2(1-v_1^2)} + \frac{E_2 t_2^2}{2(1-v_2^2)} + \frac{E_2 t_1 t_2}{1-v_2^2}\right]^2}{\frac{E_1 t_1}{1-v_1^2} + \frac{E_2 t_2}{1-v_2^2}}$$

No definitive method, so calculations using each equation to compare with real world testing would be ideal. This thesis will test both bi-layered BFS equations on different laminate disc samples.

2.6 Occlusal Fracture Resistance "Crunch the Crown" test

One way of testing dental restorations is to use a universal testing machine to evaluate their fracture resistance. Since the crown shape is irregular and variable, and the dimension and materials likewise, this may affect the results compared with other regular shaped specimens (Casson et al., 2001).

This testing method is under discussion between researchers as to whether it is a useful tool to test brittle ceramic's performance and how relevant it is to a crowns behaviour in the mouth (Isgro *et al*., 2011). Consideration must be given to the argument that a materials strength data does not necessarily reflect crown irregular structures behaviour (Kelly, 1995). Also the restoration strength may be affected because of variables such as the thickness of veneer and coping, cement and underlying abutment (Scherrer and de Rijk, 1992).

Several researchers have studied the forces of mastication inside the mouth and tried recording it. Such force varies largely depending on gender and age, but all agree that the molar region has a higher bite force than the incisal area (Tinschert et al., 2001).

Different configurations have been described by different researchers; such as using a ball indenter rather than vertical bar or changing the material that the crowns are mounted to. Examples are extracted teeth, resin, metals or dies made of gypsum.

A recommendation by Körber and Ludwig was that any restoration in molar area should be able to sustain an occlusal load of about 500N (Körber and Ludwig, 1983).

Therefore when evaluating crowns in-vitro, it was thought that a posterior metal-free restorations should withstand an occlusal force of at least 1000N, with the assumption that the mastication forces in the moist oral environment may weaken the restoration up to half its known fracture resistance force (Mehl et al., 2010).

Table 14 below summarises results of fracture resistance from different studies and materials.

Table 4: Results of crown fracture resistance done by others using different materials.

Author	Type of crown	Type of die	n=	Fracture force N	SD
(Probster, 1992)	In-ceram	Co/Cr-alloy	6	964.3	-
	IPS Empress layered	Co/Cr-alloy	6	750.6	-
	Metal-ceramic	Co/Cr-alloy	6	1494.1	-
(Neiva *et al.*, 1998)	IPS Empress	Resin die	10	2180	480
	In-Ceram	Resin die	10	2145.6	354
	Procera AllCeram	Resin die	10	1904	369
(Zahran *et al.*, 2008)	VITA Mark II	Resin die	10	1272	109
	In-Ceram YZ	Resin die	10	1459	492
(Al-Makramani et al., 2009)	Procera AllCeram	Non precious metal alloy	10	1954	211
	In-Ceram	Non precious metal alloy	10	2042	200
(Burke, 1999)	Leucite-reinforced ceramic	Natural tooth	10	880	284

2.7 Finite Element Analysis:

Finite Element Analysis FEA, work can be simply explained as: the ability to calculate the stress distribution in virtually complicated frameworks (Hsu *et al*., 2009). The use of computer software to design the proposed geometry can save time and materials and quickly explain the failure that occurs on test samples.

The FEA was firstly used to overcome manufacture drawbacks in the field on aviation more than 50 years ago and since than it was used in most industries to optimise their production (Geng et al., 2001).

In dentistry, FEA has gained popularity as it helps in understanding how teeth react with different restoration designs and materials in a non-damaging or time consuming way (Ausiello et al., 2002) (Thompson *et al*., 2011). It also contributes in overcoming the ethical problems of testing materials intra-orally and the cost of such experiments (Magne, 2007). Early attempts to analyse the stress in dental restorations was by using different techniques on actual physical models, which resulted in insufficient results (Yettram et al., 1976).

Using a computer, a mathematical model is designed then a virtual load is applied to analyse the stress created. One of the common software packages used in dental research is ANSYS (ANSYS Inc., PA, USA) (DeHoff and Anusavice, 2004).

3 Summary of the literature review:

The advances in dental materials have led to the all-ceramic restoration being considered a desirable method in restoring teeth replacing the conventional PFM restorations, with various materials and fabrication methods to choose from.

Zirconia based restorations are usually veneered with feldspathic porcelain benefiting from the outstanding aesthetic results and acceptable bond between veneer and core. Possible chipping of veneer and wear of the opposing teeth remain of concern. More tooth reduction to restore broken crowns can cause remaining tooth to break of or even be extracted.

Composites are widely used in dental clinics as filling material replacing parts of fractured teeth but rarely used as definitive full coverage crowns, especially in the posterior region, due to the lack of strength. Composite restorations have the benefit that fractures may be repaired intra-orally with the same material, avoiding the need of restoration removal or tooth preparation.

The introduction of universal primers shortened the steps for bonding composite to zirconia and saved the time as well. Most literature found was about bonding cement copings bonding and not much found about composite as a veneer of a metal-free restoration.

Till now, no single in-vitro test was found that might imitate conditions inside the human mouth, but other in-vitro tests can be carried out for such restorative materials. Another non-destructive method is Finite Element Analysis which can be used to analyse the stress on different materials with different geometry designs using computer software.

4 Aims and Objectives:

The aim of this research is to overcome some of the drawbacks in all-ceramic zirconia based and composite restorations by proposing a restoration made of zirconia core and veneered with in-lab light cured composite.

Such restoration benefits:

- The zirconia biocompatible aesthetic strong substructure.
- The aesthetic, easy to handle and intra-oral repairable composite.
- Better wear characteristics.

And the objectives are:

- Explore literature for possible methods to bond composite to zirconia and apply it in-vitro on discs specimens.
- Compare the strength of feldspathic ceramic, zirconia and composite materials solely and as laminates after bonding ceramic and composite to zirconia.
- Within the composite-zirconia laminates:
    a. Compare different zirconia surface modification in the bond to composite.
    b. Use different types of composite (flowable and packable) for bonding to zirconia.
- Create crown samples using the best method found to bond composite to zirconia in previous disc samples to bond the veneer to coping. Zirconia core will be milled out a pre-sintered block using CAD/CAM and manually build the outer veneer surface using a light cure paste composite. The crowns will be cemented to stone models and subjected to load to fracture with a ball indenter using a universal machine (n=10).
- Analyse the stress on previous different sample shapes and material. Virtual samples and Finite Element Analysis FEA will be carried out using computer software called ANSYS (ANSYS, USA), where the virtual samples are designed and stress within them is analysed.

5 Methods:

5.1 Biaxial Flexural Strength and Fracture Resistance of disc samples:

Groups of 10 specimens of single and double layered discs were fabricated to a dimension of 12mm diameter and 1mm thick disc.

Firstly, each of zirconia, composite and felspathic ceramic discs will be tested alone. Further disc samples were produced as laminates of zirconia veneered with feldpathic porcelain, and zirconia veneered with light cured composite. A summary of the materials used can be found in Table 5.

The first assessment was the Fracture Resistance (N) and the Biaxial Flexural Strength (MPa) of a disc samples (n=10) for each group, single and laminates ones.

The samples were tested using a Lloyd LRX universal testing machine (Figure 10) to record the maximum load that the disc could withstand before fracturing. This data was used to calculate the biaxial flexural strength BFS. The disc samples were fractured by applying load using the ball on ring technique at a cross-head speed of 1 mm/min (Figure 9). For bi-layer samples, the veneer faced the ball and zirconia layer rested on the ring.

The load (N), the specimens could withstand before fracturing, was recorded using the universal machine along with other measurements such as the sample deflection in mm. Using the recorded fracture load the BFS (MPa) was determined using equations for monolayer (Equation A) and bi-layered samples (Equation B and Equation C). In bi-layered samples, the veneering material is facing upwards when tested, as it is the surface subjected to outer affects in dental restoration functioning inside mouth.

Table 5: Main materials used in the Biaxial Flexural Strength and Fracture Resistance tests.

Type	Brand name	Description
Zirconia	VITA In-Ceram® YZ CUBES for CEREC®	zirconium dioxide (ZrO_2), yttrium oxide (Y_2O_3) 5%, hafnium oxide (HfO_2) < 3%, aluminium oxide (Al_2O_3) and silicon dioxide (SiO_2) <1% (weight percentage)
Composite	VITA VMLC	UDMA, TEGMA, Silica, primary paricle (40-50nm)
Composite	3M ESPE Sinfony	HEMA and 10% to 30% (octahydro-4,7-methano-1H-indenediyl) bis(methylene)diacrylate), strontium-aluminium borosilicate glass, silicon oxide, silane and photoinitiators.
Ceramic	VITABLOCS® Mark II for CEREC®	Mixture of feldspathic crystalline particles embedded in a glassy matrix
Ceramic	VITAVM9 veneering material	High-fusing, fine structure feldspathic ceramic for veneering zirconia substrates benifeting a CTE similar to YZ ceramic.
Primer	Monobond® Plus	Alcohol solution of: 3-methacryloxyprophyl-trimethoxysilane, phosphoric acid methacrylate and sulphide methacrylate.

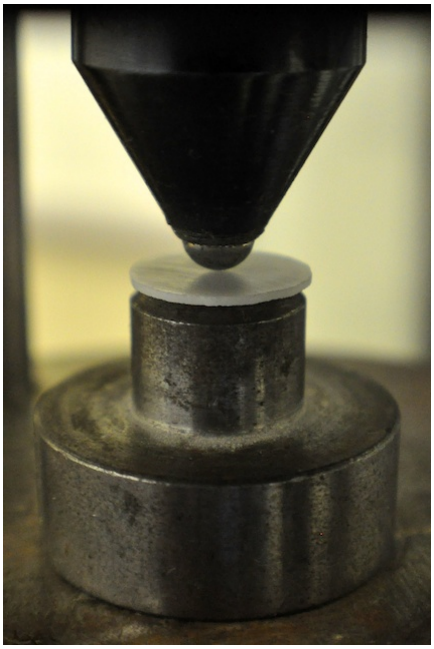


Figure 9: Ball shaped indenter repositioned on zirconia disc sample.

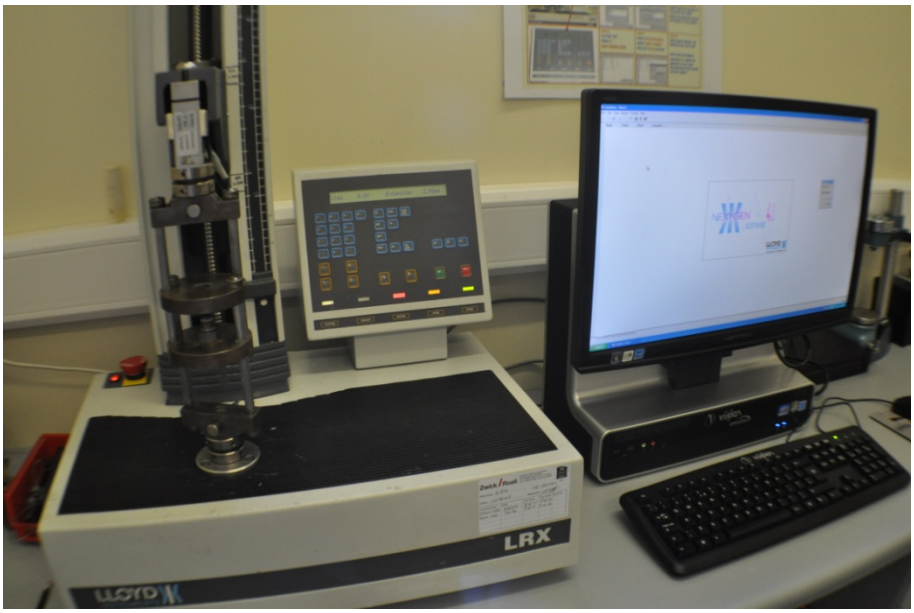


Figure 10: Lloyd LRX universal testing machine.

5.1.1 Fabrication of specimens:

All prepared samples were 1.0mm thick (excluding two groups in zirconia, 0.5mm and 0.5mm primed) and 12.0mm diameter round discs for all materials single and veneered ones. Beginning with single layered discs the fabrication process carried out in dental laboratory is mentioned in detail, followed by the veneered discs.

Mono-layered Disc Samples:

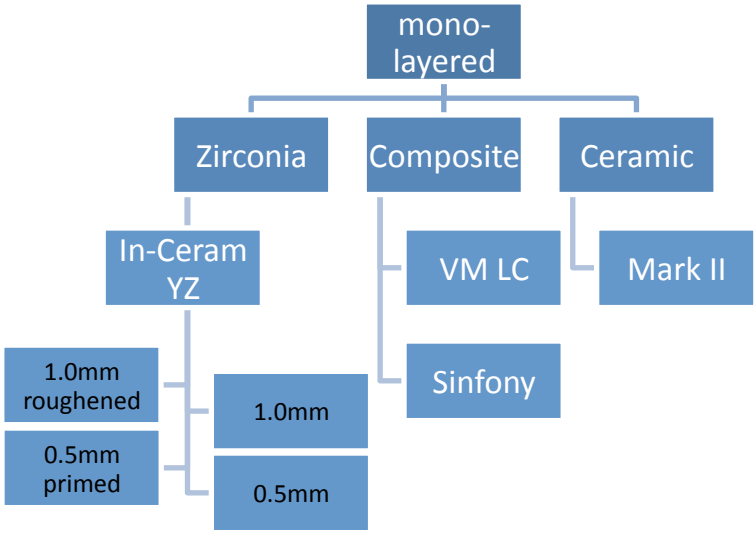


Figure 11: Mono-layered disc samples tested groups.

Zirconia, ceramic and composite, were evaluated individually as shown in (Figure 11).

Composite:

- VITA VM LC (LC):

10 discs of LC were prepared using a template made from a translucent plastic plate 1.0mm thick. The plate was drilled using a 12.0mm diameter-drilling bur and a vent created using a thin cutting disc to allow excess composite to escape. The composite (Base dentine, VITA VM LC) was placed in the drilled hole out of the syringe packing and covered with a polythene sheet (Isofolan Scheu) before gently pressing on top by hand using a flat piece of glass (Figure 12). Polymerisation was carried out using a halogen light-curing unit

(coltolux75, Coltène Whaledent Group, Switzerland) (Figure 13) and initially light cured for 1 minute and repeated again after being removed out of the plastic mould.

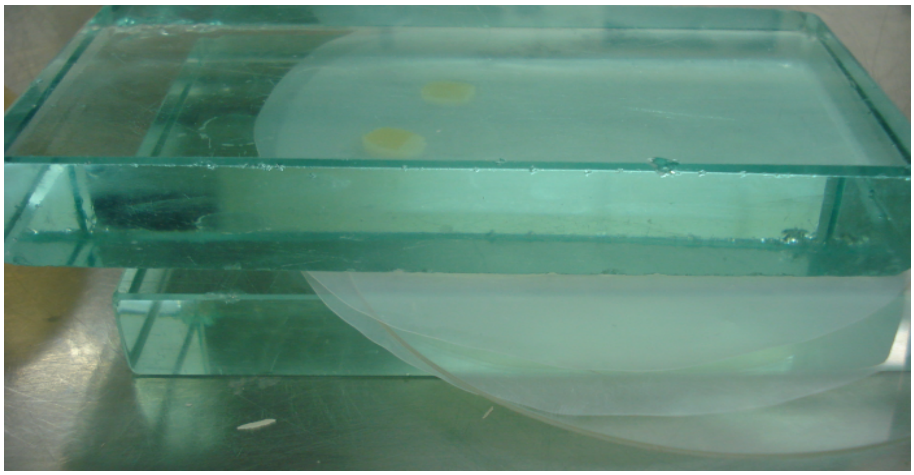


Figure 12: Preparing composite samples using glass to press them before light curing.

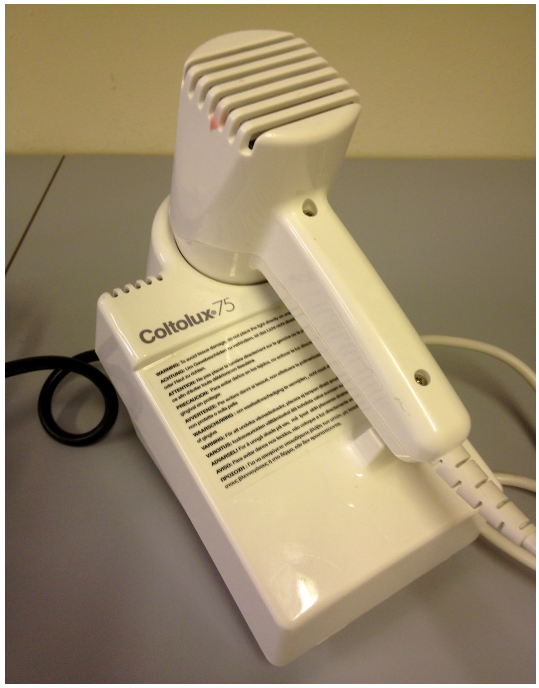


Figure 13: Halogen light curing unit.

- 3M Sinfony (Sin):

The flowable composite 3M ESPE Sinfony Indirect lab composite, was prepared using a silicone mould of a 1.0mm thick discs and 12.0mm in diameter (Figure 14). The composite is shaped in the moulds and the top surface is light cured for four minutes followed by light curing under vacuum for a further four minutes (program 4) using Visio™ Beta Vario Light Unit (Vacuum polymerization for light-cured composite, USA) (Figure 15). The same program was repeated for the other side and a final light curing for all samples was carried on for 1 minute and vacuumed and light cured for 14 minutes (program 1). Samples are finished using SiC paper (P400 grit) under water.

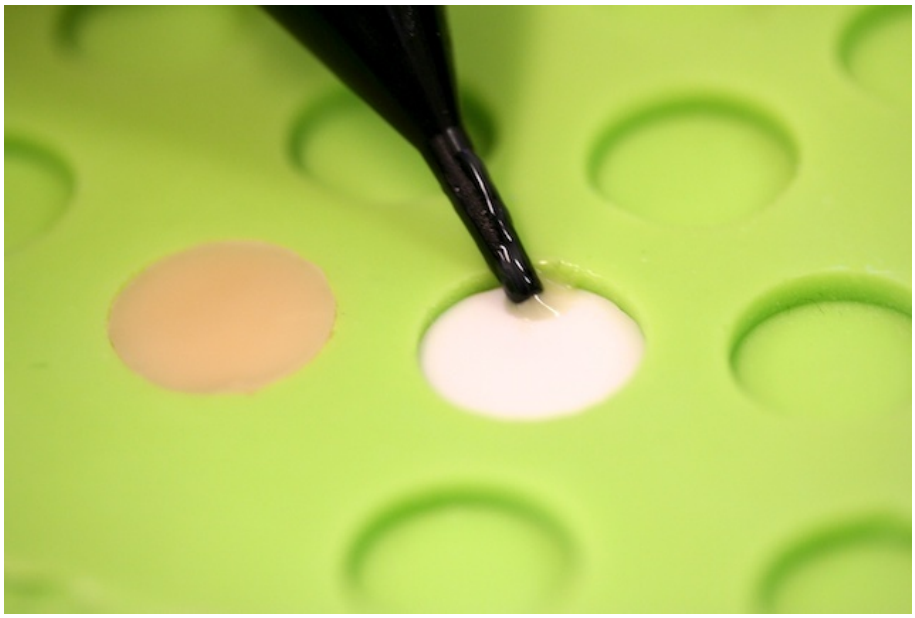


Figure 14: Filling composite on top zirconia disc in the silicon mould.

Figure 15: Visio™ Beta Vario Light Unit

Ceramic:

VITA mark II (Mk II) block for CEREC (12 × 14 × 18 mm) was drilled using a 12.0 mm diameter core drill to produce a 12.0 mm cylindrical block to be sliced afterwards acquiring disc specimens. A diamond blade 127mm × 0.4 mm (Buehler diamond wafering blade, USA) was used for cutting in a precision diamond saw (Leco VC-50) under water-cooling to produce the 12.0mm × 1.0mm discs (10 samples).

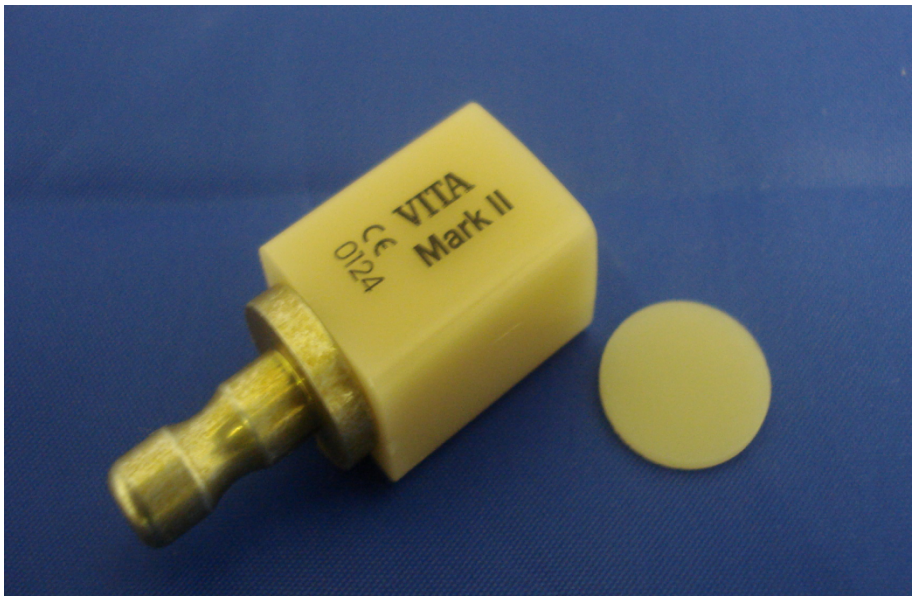


Figure 16: VITA Mark II cerec block and sample disc.

Zirconia:

All zirconia samples were produced from CEREC blocks (VITA In-ceram CAD/CAM blocks)(YZ) but with different thickness and surface finishing.

Group 1. Zirconia 1.0mm:

VITA In-Ceram YZ CEREC blocks were sliced using a 127mm × 0.4mm diamond blade (Buehler diamond wafering blade, USA) in a precision diamond saw (Leco VC-50) under water-cooling to a 1.2mm thick squared slice. Using a fine diamond bur at low speed, the square slices were rounded with the assistance of a metal template disc to a diameter of 15mm. The metal template was waxed, invested and casted. The zirconia discs were sintered in tube furnace at 1530°c with a heating rate of 10°c per minute for two hours before cooling down in the same rate to room temperature. The shrinkage of the samples caused by the sintering (approximately 20%) resulted in the final sample thickness of 1.0mm and diameter of 12mm.

This group of unmodified sintered zirconia was to be compared with other groups of monolayer ceramic and composites.

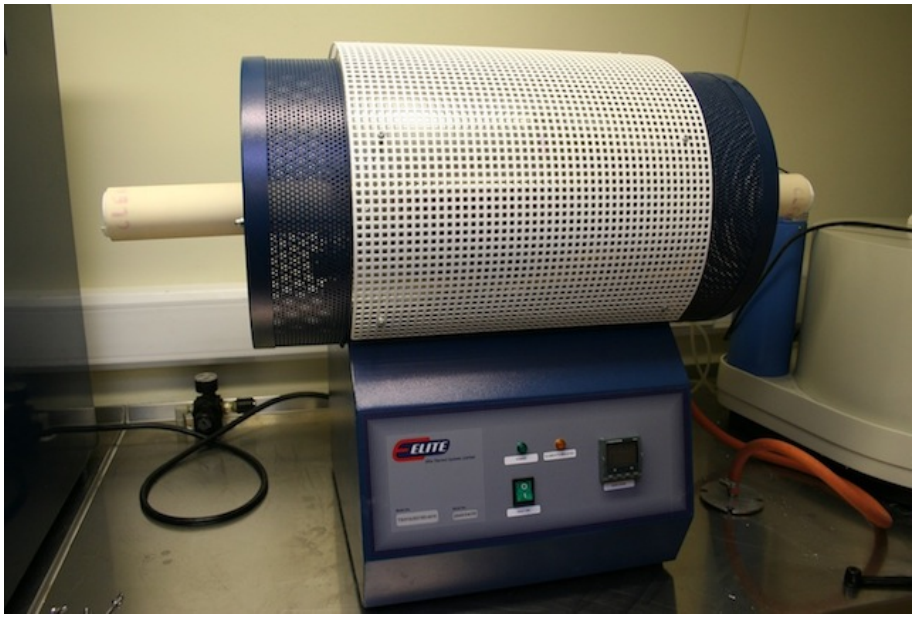


Figure 17: Tube furnace with closed ends used for sintering zirconia samples.

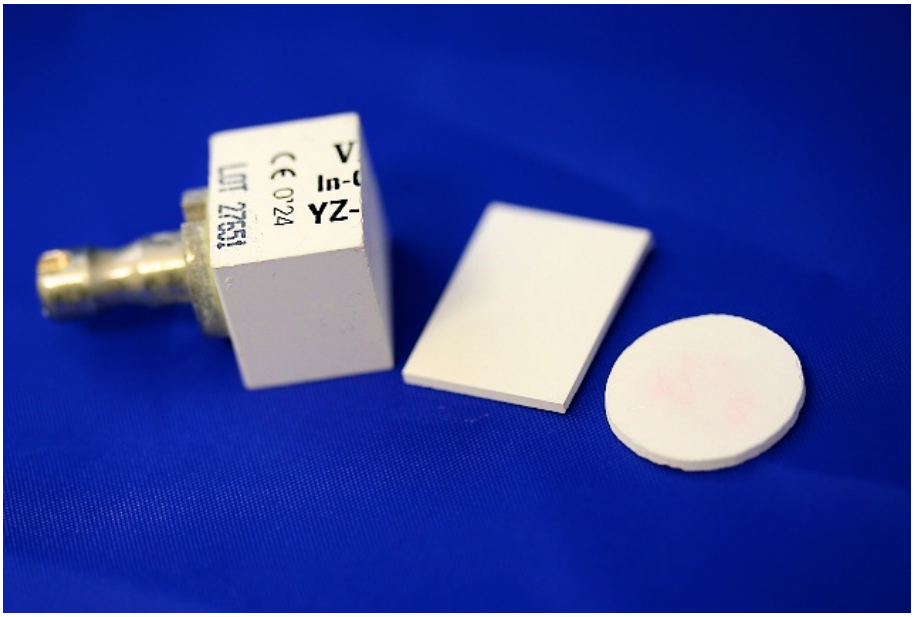


Figure 18: Zirconia sample stages from the CAD/CAM block.

Group 2: Zirconia 0.5mm were produced using the same method but with a pre-sintered thickness of 0.7mm to result after sintering in a 0.5mm thick 12mm diameter zirconia disc. At first, different trials were done to achieve the correct diameter and thickness after calculating the 20% shrinkage mentioned in manufacturer manual.

Using this group, a comparison will be made between these discs and the affect of adding another 0.5mm top layer of either ceramic or composite.

Figure 19: 10 samples of zirconia 0.5mm discs.

Two other zirconia samples were prepared with different surface treatments prior and after heat treatment.

Group 3: Pre-sintered roughened zirconia: These samples were produced with mechanical retention on the surface prior to sintering. The retention was achieved by shot-blasting the pre-sintered zirconia discs through a fine plastic mesh using 50μm Alumina. The dimensions after sintering were 1.0mm thick and 12 mm diameter disc. Retentive features are shaped as a square of 0.5mm length (Figure 19).

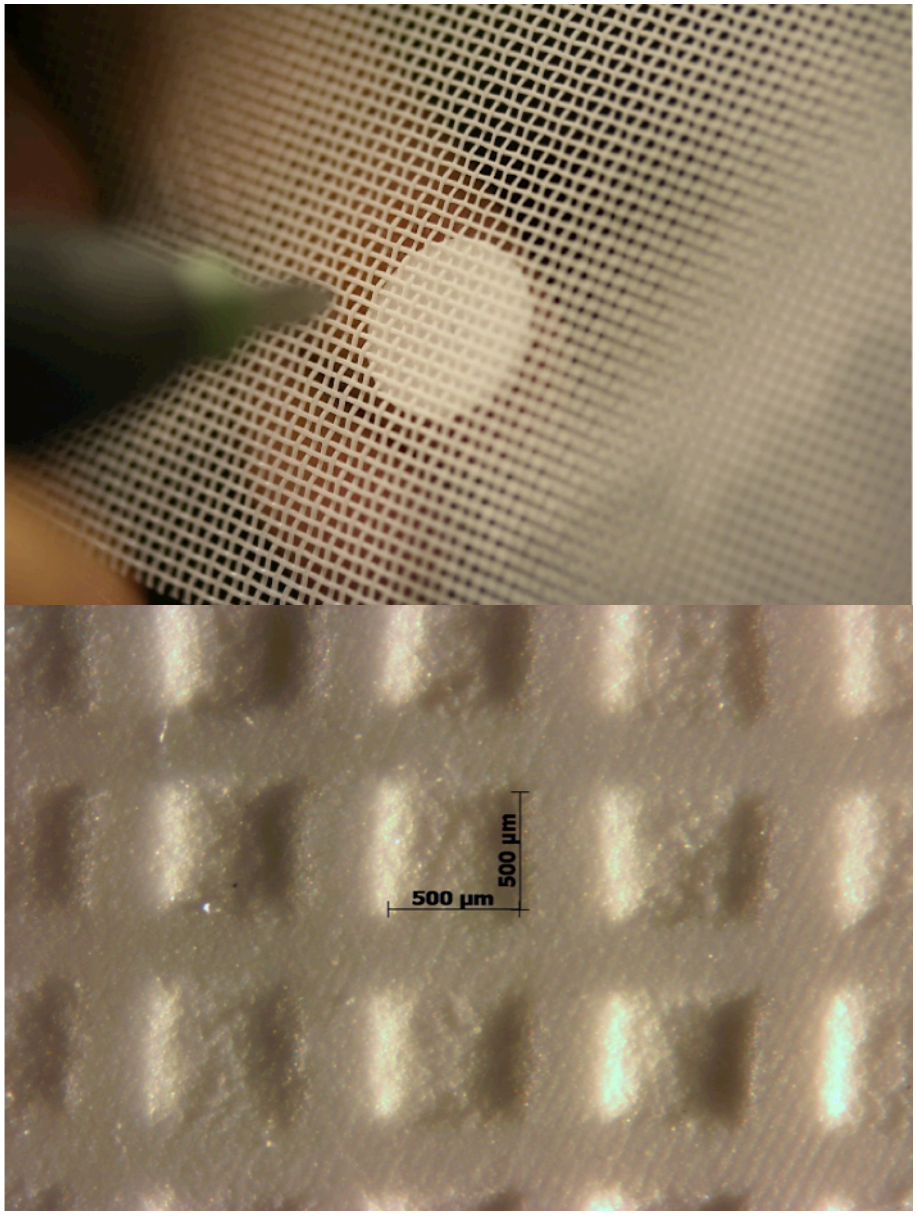


Figure 19: Creating the mechanical retention by shot-blasting the pre-sintered zirconia through the plastic mesh (top) and 2x-magnified surface of the pre-sintered roughened zirconia (bottom).

Group 4: Primed zirconia: Sintered 0.5 mm zirconia discs were lightly shot blasted for 5 seconds using 50μm Al_2O_3 particles under 35bar pressure and 10mm distance between nozzle and sample. The discs were rinsed with water and dried with oil free air. Finally a layer of primer (Monobond® plus) was applied on the bonding surface using a disposable brush and left to set for at 10 minutes.

Figure 20: Applying the primer on the 0.5mm zirconia disc.

5.1.2 Bi-layered Disc Samples:

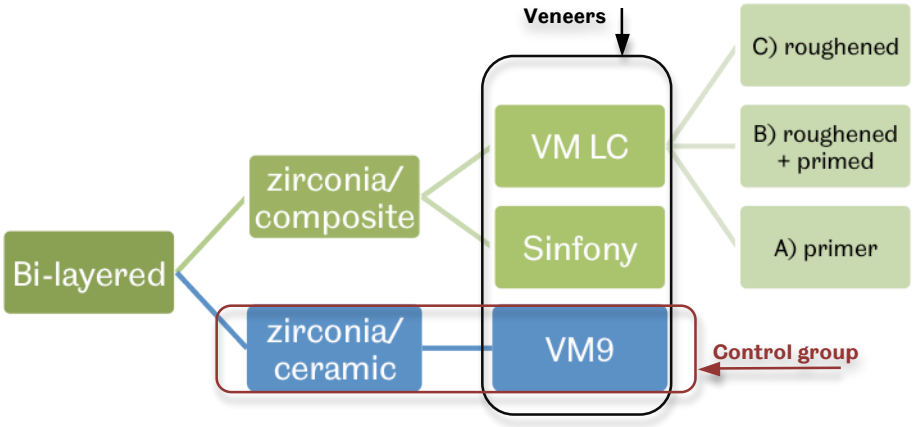


Figure 21: Bi-layered disc samples tested groups

Zirconia/Ceramic laminates:

The zirconia samples were veneered using a feldspathic porcelain, (VITA VM9) which has a thermal expansion coefficient that matches that of the zirconia. The sintered zirconia discs first had a layer of VITA VM9 effect bonder (powder & liquid) sintered to the surface before the veneering ceramic was built to full thickness. Base dentine was layered on with the disc in a silicon mould to aid shaping the dentine layer. After dentine firing at 910°C in a vacuum furnace (VITA VACUMAT 40 T), the veneer was finally polished using silicone carbide paper P600 grit to a final thickness of 1.0mm.

Figure 22: Ceramic (powder/liquid) used to veneer zirconia samples.

Zirconia/Composite laminates:

The composite was bonded to the zirconia using a universal primer Monobond® plus (Ivoclar Vivadent AG).

Three groups were fabricated using same composite to test the difference in bond strength as a result of differing surface finishes of zirconia.

Groups compared were:

a. Primer + mechanical retention (shot blasted).
b. Mechanical retention only (on pre-sintered zirconia).
c. Primer + mechanical retention (on pre-sintered zirconia and shot blasted after sintering).

A further group was produced using flowable composite (3M Sinfony).

VITA VM LC: This composite was used in fabricating the three zirconia/composite laminates a, b and c mentioned above.

A total of 10 samples for each of the three groups were prepared as follows:

For groups a and c, zirconia surface was slightly shot blasted after sintering with 50μm Alumina particles and rinsed with water before left to dry. Then a thin layer of primer was applied on the zirconia-bonding surface and left for 10 minutes to set up.

In group b, the zirconia base was prepared as done previously in: pre-sintered roughened zirconia in 5.1.1.3 Zirconia. For all three groups, the opaque layer is established after mixing the powder and liquid (1:1 ratio) and light cured for 1 minute. The composite dentine was shaped on top of the opaque layer and gently pressed with a flat piece of glass and light cured for 2 minutes. The final thickness of the laminate disc is finished to 1.0mm using a 400-grit silicon grinding paper (Buehler).

3M Sinfony: This composite is relatively flowable compared to the first composite so an idea of a better interlocking with shot-blasted zirconia surface is taken in mind. After shot blasting and cleaning zirconia discs, primer is brushed and left for 10 minutes to set. Composite opaque was applied on

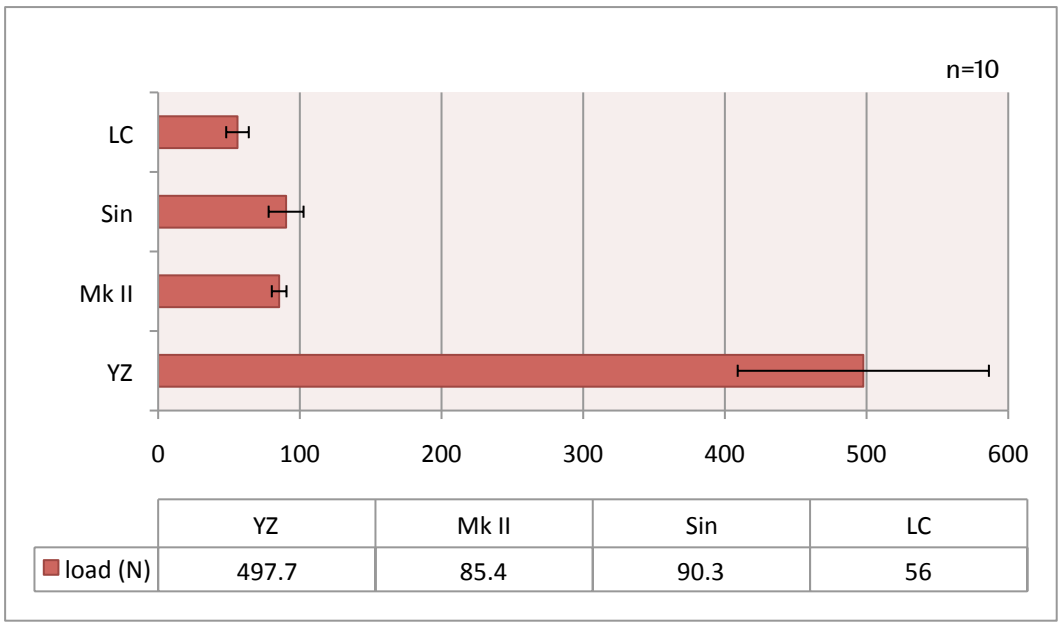
the zirconia surface and light cured using 3M visio beta vario for 7 minutes (program 2). Then samples are placed in the mould with the opaque surface facing outwards for the dentine to be applied and shaped on top of it. Program 1 is chosen for final polymerizing.

5.1.3 Results:

Monolayer groups:

Fracture resistance of single layer (1.0mm thick) groups is shown in (Figure 23) below, with a highest result recorded for zirconia core material.

A thinner zirconia 0.5mm showed fractured in an average load of about 128N.



	YZ	Mk II	Sin	LC
load (N)	497.7	85.4	90.3	56

Figure 23: Max strength (N) that single layered 1.0mm thick specimens withstands before fracturing.

Table 5 shows the BFS of all tested monolayer specimens with the mean and standard deviation for each group.

Zirconia demonstrated a high biaxial flexural strength of 1011.2 and 1005.6MPa in specimens of 1.0mm and 0.5mm thickness respectively with a standard deviation of 173.9 and 93.7 respectively.

Table 6: BFS results of all single layer groups (MPa) calculated using the first single layer Equation A.

No.	Ceramic	Composite		Zirconia			
	(1.0mm)	LC (1.0mm)	Sinfony (1.0mm)	(1.0mm)	P.RS (1.0mm)	(0.5mm)	Primed (0.5mm)
1	194.0	112.4	189.6	1085.9	804.1	1317.5	1115.7
2	156.9	106.1	162.9	905.1	790.2	969.9	1240.5
3	185.4	125.8	182.1	1190.0	880.1	908.6	960.8
4	174.7	116.8	180.8	1032.9	1047.3	995.0	1117.6
5	172.4	113.6	156.5	1013.0	938.1	1134.6	1404.7
6	187.8	116.0	169.9	578.4	1012.6	979.5	1404.1
7	174.4	97.3	183.0	1010.8	809.9	701.4	1030.8
8	182.9	103.9	199.3	1028.1	864.0	1266.7	1144.4
9	171.0	139.9	186.3	1098.2	909.0	857.1	805.4
10	185.1	105.0	178.1	1169.9	1011.9	925.9	1155.0
Mean	**178.5**	**113.7**	**178.9**	**1011.2**	**906.7**	**1005.6**	**1137.9**
STDEV	10.7	12.2	12.7	173.9	93.7	186.9	184.7

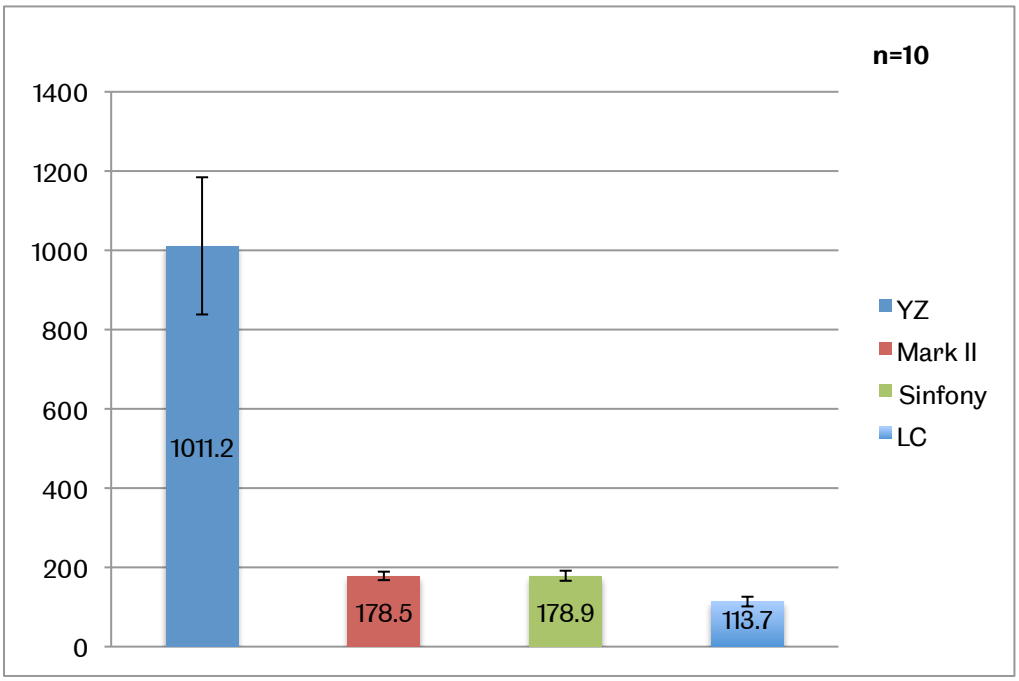


Figure 24: BFS (MPa) of monolayer groups of zirconia (YZ), ceramic (Mark II) and composite (Sinfony and LC).

The pre-sintered mechanical retention group showed lower BFS than first two groups at 906.7MPa, whereas the 0.5mm shot blasted and primed discs exhibited a BFS of 1137.9MPa.

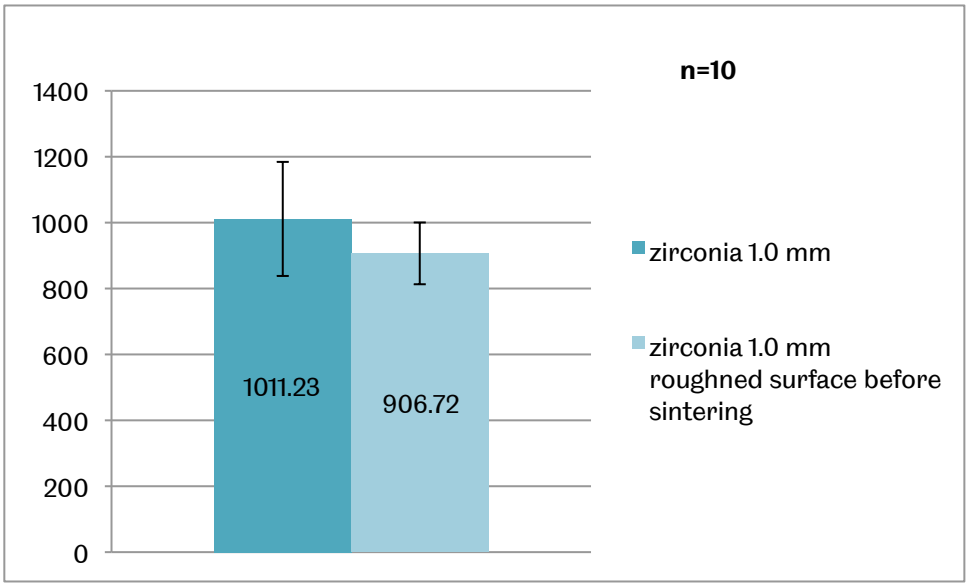


Figure 25: BFS of 1.0mm thick YZ discs of two different groups

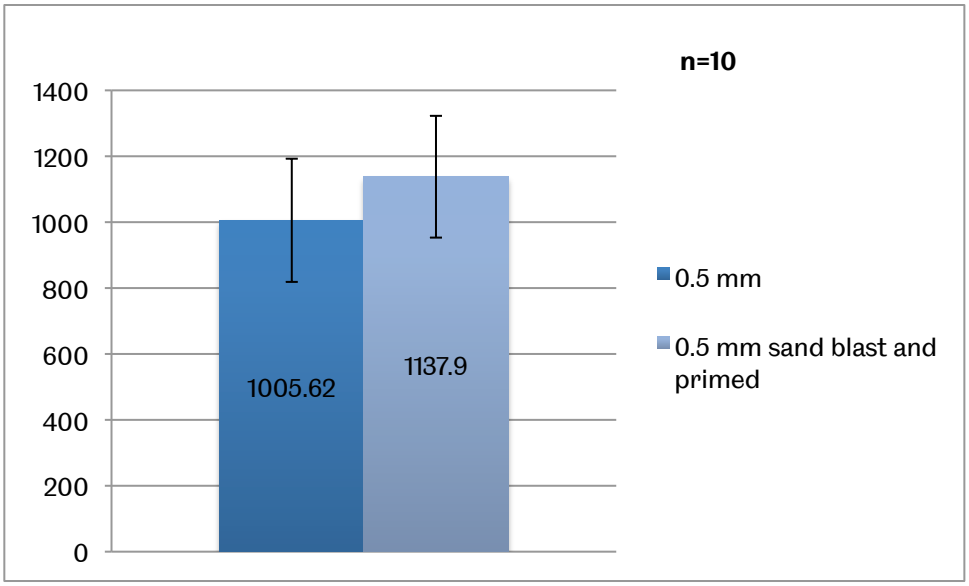


Figure 26: BFS of 0.5mm thick YZ discs of two different groups

Monolayer specimens of Vita Mark II were produced from such blocks showing an average BFS of 178.5MPa for the ten ceramic discs as shown in (Table 6) and (Figure 24).

Two indirect light cure composites were tested, VITA VMLC and 3M Sinfony. The first group showed an average BFS of 113.7MPa and 178.9MPa for the second composite group for a 1.mm thick specimens as shown in Table 6.

Bi-layered groups:

Table 7 shows the Fracture Resistance of all veneered (bi-layered) discs. Zirconia/ceramic group was used as a control group as this is used in dental practice. VITA VM9 (feldspahtic porcelain for veneering zirconia) was bonded to zirconia and these laminate discs fractured at an average load of about 377N.

Table 7: Fracture resistance of all veneered 1.0mm thick discs groups in (N).

No.	Ceramic+zirconia	Composite+zirconia			
Veneer	Control group	Sin	LC		
	VM9		Primer	Mechanical +Primer	Mechanical
1	424	269	238	134	175.8
2	442	282.5	210.9	234	158.3
3	407.8	289.6	229.5	243.8	148.8
4	380.6	110.5	240.3	272.7	175.6
5	367	205.5	299	232.7	210
6	384	227	249	190	114
7	297.6	241.5	257	193.8	131.7
8	344.6	243	200	169.5	209
9	395.5	199.5	251	152	148
10	332.9	187	253	243.6	141.5
Mean	**377.6**	**225.5**	**242.7**	**206.6**	**161**
STDEV	43.6	53	27	45	31

Three groups of a zirconia veneered with composite with different modifications were tested as a start. Slightly shot-blasted with Al_2O_3 and primed zirconia (chosen best method) for bonding composite group exhibit an average fracture resistance of 242.7N. The other two had the zirconia roughened prior to sintering and one of them combined all the pre-sintered roughening, shot blasting after sintering and primed. The mean load at fracture was around 161N and 206.6N respectively. With the pre-sintering roughened zirconia/composite samples, veneers were separated from zirconia in most samples almost completely.

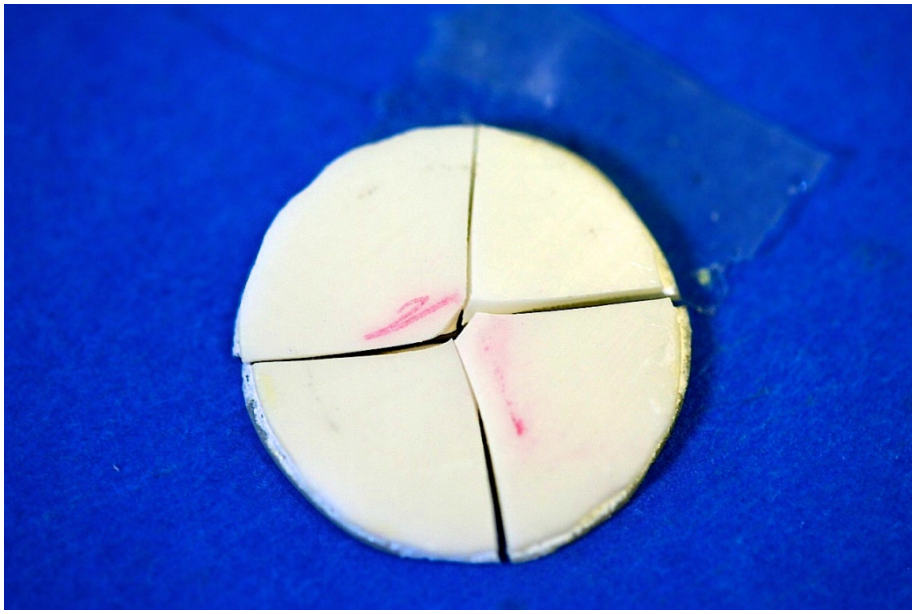


Figure 27: Zirconia/composite (YZ/LC) broken specimen. The bond was established by shot blasting and using universal primer, and as seen that both layers are still bonded after applying the occlusal load.

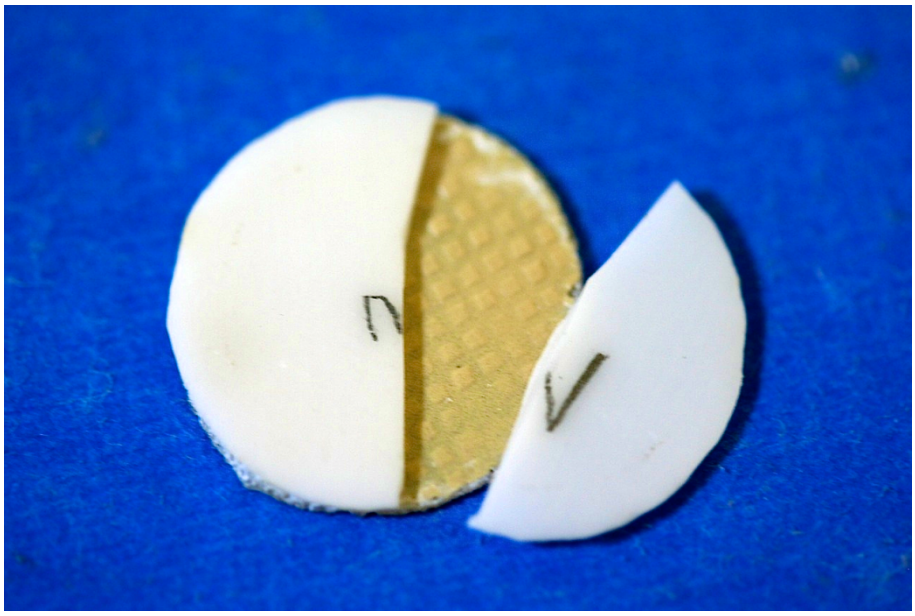


Figure 28: Zirconia/composite (YZ/LC) broken specimen. The bond was purely mechanically and was enhanced by the grooves created prior sintering zirconia, and as seen that layers almost separated completely after applying the occlusal load.

Group 2 was veneered with 3M Sinfony in-lab light cure composite using primer to promote bonding to zirconia disc. This group showed a fracture resistance of about 225.5N, as seen in Table 7.

The chart below reveals the average load fracturing specimens (1.0mm) of bi-layered groups and monolayer groups.

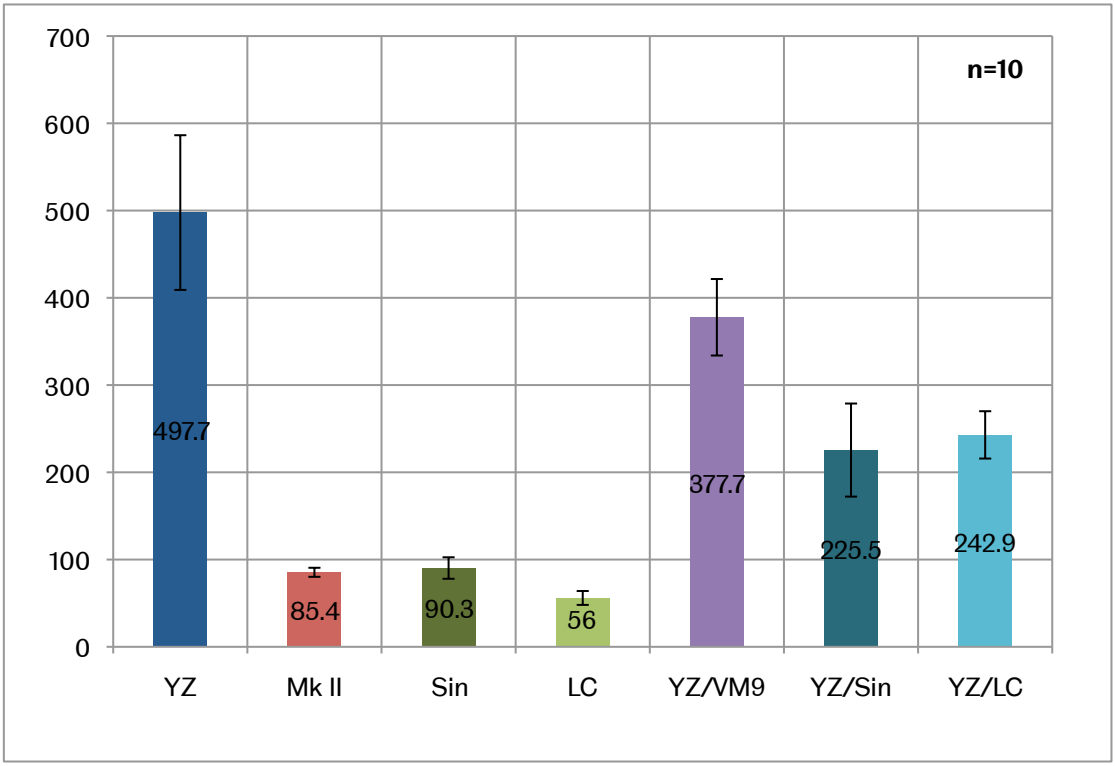


Figure 29: Load at fracture N for monolayer groups 1.0mm (YZ,Mk II, Sin and LC) and bi-layered groups (0.5mm each) of groups YZ/VM9, YZ/Sin and YZLC.

The depth of the sample deflection caused by the ball indenter in mm is shown in Table 8 and Table 9. The average deflection and standard deviation of ten specimens was calculated for each group. All samples in table 7 were the same thicknesses of 1.0mm for mono and bi-layered samples. The greatest deflection before fracture was demonstrated by the YZ/LC system. In table 8, the YZ/LC laminates bonded with primer also showed the highest deflection value.

Table 8: Average deflection (mm)

	YZ	*Mk II*	*Sin*	*LC*	*YZ/VM9*	*YZ/Sin*	*YZ/LC*
Mean mm	0.69	0.26	0.41	0.48	0.61	0.51	1.0
STD	0.12	0.09	0.07	0.05	0.18	0.04	0.04

Table 9: deflection in mm for YZ/LC with different surface treatment.

	YZ/LC		
	Primer	Mechanical+Primer	Mechanical
Mean mm	1.0	0.98	0.46
STD	0.04	0.05	0.11

Two different equations (equation B and D) were used in calculating the biaxial flexural strength for the bi-layered samples. Table below show the BFS for zirconia/ceramic (YZ/VM9) laminates and zirconia/composite (YZ/LC) laminates groups. Table 10 displays the fracture resistance and the BFS of each layer in both zirconia laminate systems (ceramic and composite veneered) using equation B.

Table 10: BFS results for bi-layered groups using equation B by (Hsueh and Kelly, 2009).

Sample	**Load to fracture N**	Bottom layer (core) MPa	Top layer (veneer) MPa
YZ/LC	**242**	791±88	45±5
YZ/VM9	**377**	548±63	263±30

Using equation D gave the BFS of each layer of both zirconias laminates system (ceramic and composite veneered) as shown in (Table 11).

Table 11: BFS results for bi-layered groups using equation B by (Hsueh *et al.*, 2006).

Sample	**Load to fracture N**	Bottom layer (core) MPa	Top layer (veneer) MPa
YZ/LC	**242**	1682±187	35±3
YZ/VM9	**377**	1128±130	326±37

5.1.4 Discussion:

All samples were produced as accurately as manually possible. Difficulties in producing zirconia discs was resolved using a cast metal disc as a guideline to the diameter required.

The results for the single layered discs, demonstrate the difference between the materials. Zirconia showed highest biaxial flexural strength among ceramic and composite.

Comparable results were found of zirconia, ceramic and composite performance after bonded together as laminate discs. In the bi-layered groups, zirconia is considered as the substructure of the disc (bottom layer) and the composite or ceramic is the veneer (top layer). Ceramic (feldspathic porcelain) is already used in dental laboratory to veneer zirconia-based all-ceramic restorations and some brands are commercially produced for this purpose. And because of that it has been tested along side with zirconia-composite specimens to be compared with.

A feldspathic industrially sintered and pressed ceramic blocks (VITA Mark II) will give the optimum performance compared to liquid/powder lab base ones.

After the initial bonding trials, the method chosen for the composite zirconia laminates was to use a universal primer after shot-blasting zirconia surface to enhance the micromechanical boning. This resulted in a good bond as demonstrated in (Figure 27), were the fractured discs pieces showed the composite still bonded to zirconia after failure. The technique is supported by (Kern *et al*., 2009), (Yun *et al*., 2010), (Yang *et al*., 2010) and (Attia *et al*., 2011). These samples were tested in dry conditions and were not exposed to thermal or moisture variables.

Some literature; (Kosmac *et al*., 1999), (Zhang *et al*., 2004) and (Phark *et al*., 2009) have criticised any mechanical roughening of a sintered zirconia surface by either shot-blasting of using rotary bur. They stated that such surface modification might lead to damaging ceramic surface and hence drop in its

fracture resistance. The manufactures do not recommend any surface modification of zirconia after sintering although if necessary a diamond bur in low speed and wet condition can be used (VITA manual).

(Guazzato et al., 2005), contradicts this showing that mechanical surface modification of zirconia may weaken it. This study found that higher strength was observed after shot blasting and grinding zirconia surface. Also supported by (Vagkopoulou *et al*., 2009) and (Qeblawi et al., 2010).

Two groups were prepared to test if there is any effect on fracture resistance and biaxial flexural strength of zirconia discs when shot-blasted with 50µm Alumina particles and primer added. Two groups of 0.5mm zirconia discs were tested. First group was treated, as composite will be bonded to. The sintered disc was shot-blasted using 50µm Al_2O_3 for a short time, rinsed with water and dried before applying the primer using a disposable brush and left for at least 10 minutes before testing them. And second was tested after sintering with no surface modification. The primed group showed a higher BFS of 1137MPa compared to 1005MPa, but with no significant difference according to one-way ANOVA test (see Appendix).

From the previous literature (Mirmohammadi *et al*., 2010), an idea was to try to create retentive surface on the pre-sintered zirconia. This study compared samples produced with and without mechanical retentive surfaces created before and after sintering on the fracture and strength of the laminate. The purpose of the roughened surface is to increase the bonding surface and to promote mechanical interlock between core and veneer materials. The first step was to compare the zirconia samples with mechanical retention created pre-sintering with non-modified zirconia to give an idea on the influence on the core material before comparing the laminates. The results showed that the modified zirconia (all 1.0mm thick) showed a lower BFS of 906MPa compared to 1011MPa, which is a 10% percentage decrease.

The effect of the surface modifications was compared in the zirconia/composite laminates with three different zirconia surface modifications:

- Primer after shot blasted with 50µm Al_2O_3.
- Mechanical retention only (on pre-sintered zirconia).
- Primer + mechanical retention (on pre-sintered zirconia and shot blasted after sintering).

The lowest result was recorded with samples not primed (mechanical retention on pre-sintered zirconia) that fractured at a load of 160N and BFS of as shown in Table 7.

Groups using primer showed an average fracture resistance of about 240N and about 200N when combined with mechanical retention. The weakest group (mechanical only) showed major delamination between composite and zirconia layer after applying load unlike the other two (with primer) were some samples showed minor delamination.

This roughening to the pre-sintered zirconia did improve the bonding. In both single layer and in the laminates, it weakened the zirconia disc. This is most probably due to the decrease in thickness and the modified shape, giving rise to crack initiation sites.

For veneering materials, two in-lab light cured composites were used and characterised to: Flowable composite (Sin) and packable composite (LC) and they showed average BFS of about 178MPa and 113MPa respectively. Ceramic was tested (VITA mark II) and showed a BFS around 170MPa and both ceramic and composite showed expected lower results than the zirconia with a BFS of approximately 1000MPa (Figure 24). These results were calculated using Equation A (Piddock *et al.*, 1986). All Biaxial Flexural Strength equations were discussed previously in page 38.

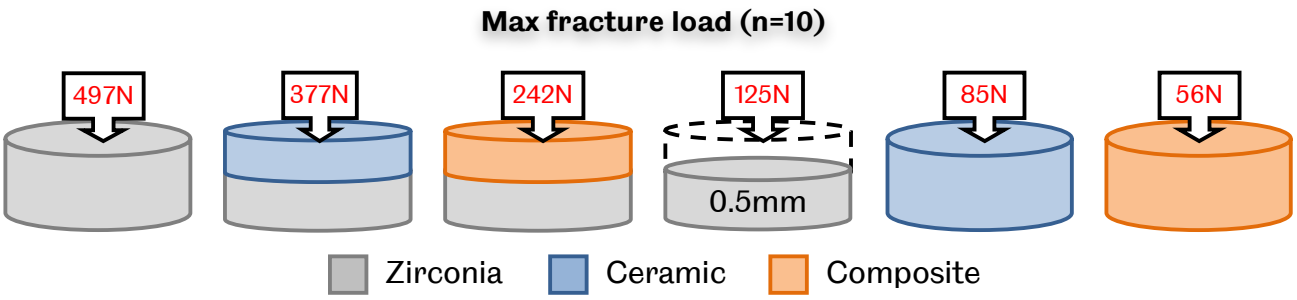


Figure 30: Illustration showing the max fracture load N between different specimens of different materials and thicknesses.

When comparing fracture resistance, the load at fracture was recorded and different groups can be compared without any computation. The results are shown in (Figure 30). Considering a 1mm thick disc, comparison between discs differing only in the material made of, the whole sample could be from a single material or laminates out of 0.5mm thick materials bonded to form 1mm thick disc.

As expected the 1mm zirconia showed a high fracture resistance (497N) to the ball indenter when supported by a ring, and the 1.0mm ceramic and composite showed significantly lower strengths of 85N and 56N, respectively. These results come in sequence with the zirconia, ceramic and composite different stiffness, which accordingly are 210, 65 and 4.5Gpa. Replacing the top 0.5mm zirconia with ceramic gives a 1.0mm thick laminate that fractures at an average load of around 370N, and when replaced with a much less stiff 0.5 thick composite the result was around 240N when using VITA VM LC and 225N with 3M Sinfony (elastic modulus of 3.1Gpa).

As for the BFS of bi-layered groups, the results when using Equation B (Hsueh and Kelly, 2009) demonstrated an anomaly since the fracture resistance of the zirconia/ceramic (377N) laminate was higher than the zirconia-composite (242N), which contradicted the BFS calculated using this equation (548MPa and 791MPa respectively).

However when using the single layer equation (Equation C), the one derived from (Hsueh and Kelly, 2009 *et al*, Equation B), for calculating BFS for single layer groups gave different results than the ones acquired from Equation A.

For instance, it showed a BFS in MPa for the 1mm thick samples of zirconia, ceramic and composite of 490, 79, 55, respectively, which are almost half of their known BFS. Such finding may give a reason to doubt the outcome of equations B and C.

Equation D was the other bi-layered BFS equation (Hsueh *et al*., 2006) used in this study. Zirconia/ceramic laminate showed some how reasonable result (1128MPa) with the bottom layer BFS equals the known zirconia strength. On the other hand the zirconia/composite laminates showed a much higher BFS (1682MPa) than all other single and layered samples.

5.2 Occlusal Fracture Resistance "Crunch The Crown Test":

Producing zirconia crowns veneered with composite and testing the fracture resistance was carried out to assess the force that the completed restoration can withstand before failure. This differs to the disc test due to the irregular shape of the crown, which give rise to a test of the bond and the material properly after processing into the crown form.

5.2.1 Materials and methods:

Crowns were fabricated using a CAD/CAM system to produce the zirconia substructure and a silicone matrix was used to produce the veneer overlay, in order to make the crowns as consistent as possible. The crowns were tested using a universal testing machine (Lloyd LRX universal testing machine) having a ball indenter pressing occlusally in the middle of crown (fossa) at a crosshead speed of 1 mm/min using a 2000N load cell and 100.6% sensitivity. The first fracture was recorded either by watching, hearing or machine sensor before recording final load at crown failure by testing machine.

Table 12: Main materials used in the fracture resistance test.

Type	Brand name	Composition
Zirconia	VITA In-Ceram® YZ CUBES for CEREC®	Zirconium dioxide (ZrO_2), yttrium oxide (Y_2O_3) 5%, Hafnium oxide (HfO_2) < 3%, aluminium oxide (Al_2O_3) and silicon dioxide (SiO_2) <1% (weight percentage)
Composite	VITA VMLC	UDMA, TEGMA, Silica, primary paricle (40-50 nm)
Die stone	Dentona esthetic-base gold	Type IV extra-hard dental stone.
Primer	Monobond® Plus	Alcohol solution of: 3-methacryloxyprophyl-trimethoxysilane, phosphoric acid methacrylate and sulphide methacrylate.
Cement	Pavavia 21, Kuraray Co. Japan	Catalyst Paste and Universal Paste. More information available in Appendix.

An ideal preparation and a silicone mould were used to produce stone casts for the test from a master model. All test models were produced by pouring dental stone after mixing with water in a vacuum mixer.

VITA In-Ceram® YZ blocks were used to produce the coping using CEREC® CAD/CAM system. To gain an ideal design of substructure, the prepared tooth along with neighbouring teeth were scanned using the CEREC scanner. The CEREC software was used to produce a cutback substructure design according to the scanned data of the opposing dentition. After the milling process had finished (Figure 31), the sample was removed from the milling chamber and the substructure separated using a round cutting disc in a micromotor. The sprue was evened with the rest using diamond bur in medium speed. The coping in this stage is over sized and brittle, so cautious handling is important to avoid cracks. The Zirconia sample was then heat treated (sintering) using a furnace (Figure 17) with temperature rising 10°C per minute until it reached 1530°C were it was held for two hours before cooling to room temperature at the same rate. After this treatment, the sample had reduced in size to the desired measurement, approximately 20%.

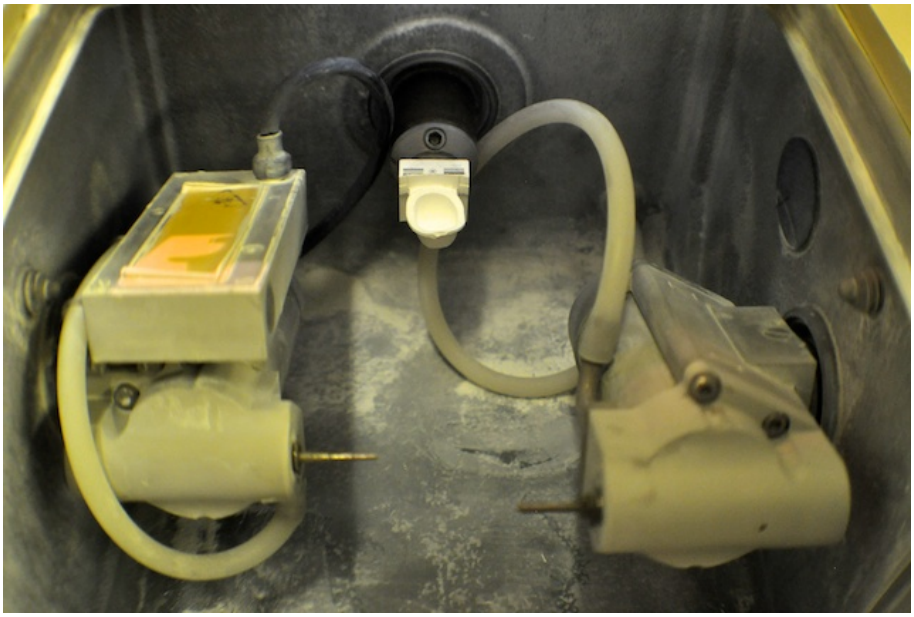


Figure 31: Zirconia coping inside CEREC milling chamber.

The veneering composite was added by using universal primer after lightly shot-blasting the sintered zirconia surface with 50µm particle size Al_2O_3 under 40psi with a distance of about 5mm. The coping was cleaned by rinsing with

water and left to dry. A thin layer of the universal primer (Monobond® plus) is applied on the outer surface of coping using a disposable brush (Figure 32) and left for 10 minute to set.

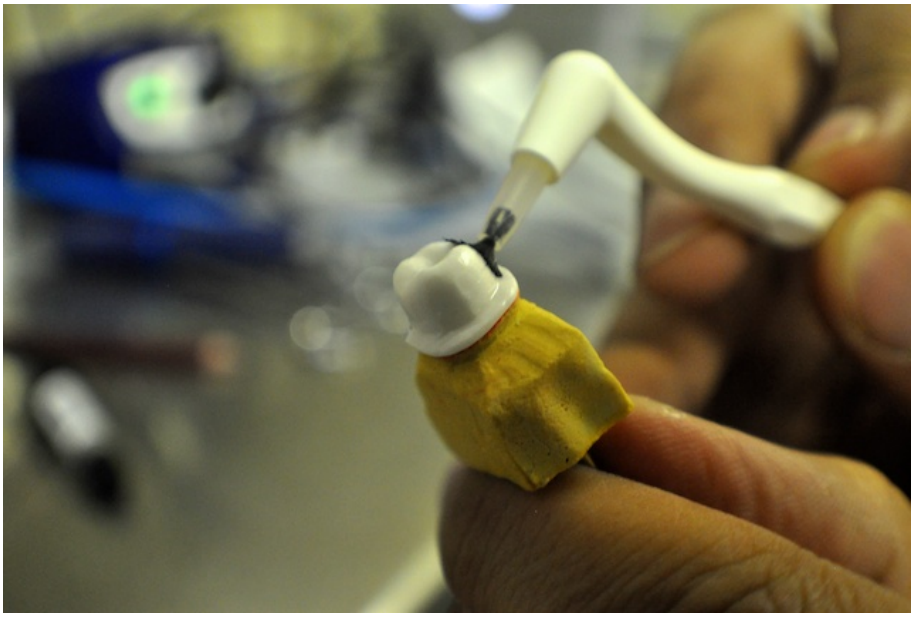


Figure 32: Adding a layer of universal primer using a disposable brush.

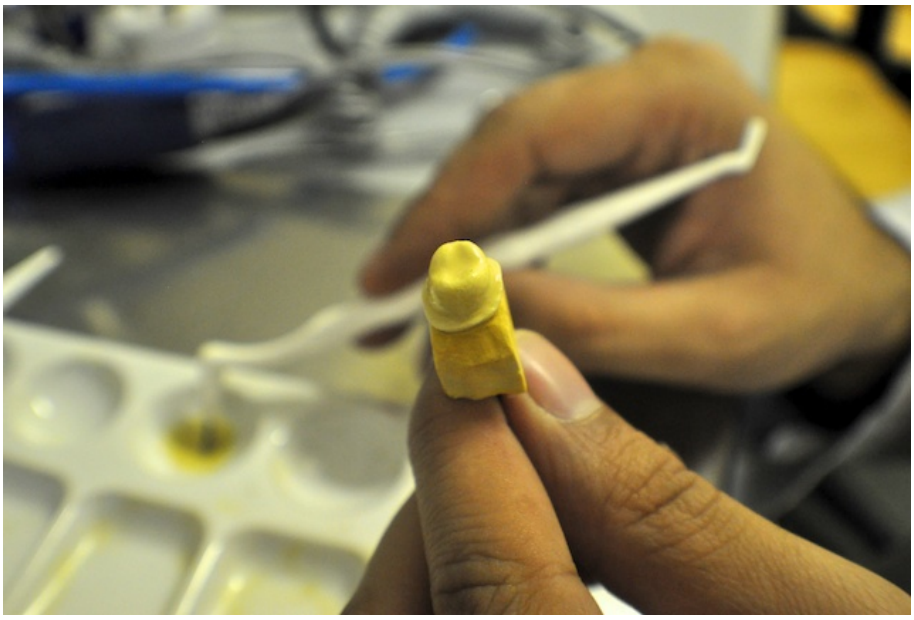


Figure 33: Opaque layer on zirconia coping.

An opaque composite was first added to create the initial bond, which is a powder, and liquid mixed in 1:1 ratio. A layer of composite opaque is painted on top of the primer surface (Figure 33) and light cured using hand held curing unit for one minute on each side (five sides).

After that the dentine layer is applied on top of the opaque, which is a paste like materials comes in a black plastic tubes to prevent it from being composed to light. Using a silicon impression and a small spatula, the outer contact surface of the crown is shaped (Figure 34). Drops of modelling liquid are used to ease up shaping the tooth by simply wetting the spatula. After the desired shape is built-up comes the polymerising step by light curing each side for one minute. After composite sets, adding and removing composite is possible by grinding using a carbide bur in a medium speed micro-motor. In most cases, a second layer of dentine is added and light cured. Finally finishing and polishing of the crown is done using a rubber silicon wheel and brush wheel with polishing gel resulting in a finely polished tooth (Figure 35).

Figure 34: Shaping outer surface before polymerising the composite using spatula.

Figure 35: Completed crown after polishing.

The crowns were cemented on stone models using Panavia resin cement (Kuraray Co. Japan) after the fitting surface was shot blasted with Al_2O_3 and rinsed with water and dried afterwards. The cement is a two-paste system that is mixed and packed inside fitting surface before fixing on prepared tooth. The excess mix was cleaned around the margin and an OXYGUARD gel was applied on it.

Tested groups:

Twenty zirconia/composite crowns were produced differing only in the thickness of the zirconia:

Group 1: Zirconia 1.0mm and 0.5mm of composite (Figure 36).

Group 2: Zirconia 0.7mm and 0.5mm composite (Figure 37).

All composite veneers were built the minimum thickness indicated by the manufacturer for posterior applications: 0.5mm according to manufacturer manual (VITAVM®LC Working Instructions) to optimise the strength of the restoration.

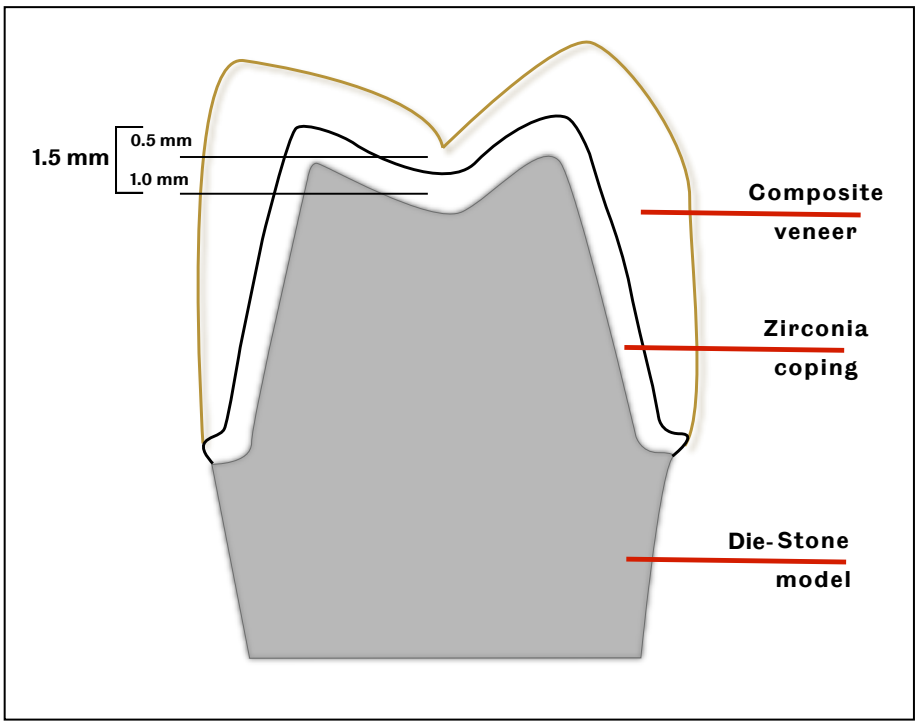


Figure 36: 1st group 1.0mm zirconia and 0.5mm composite.

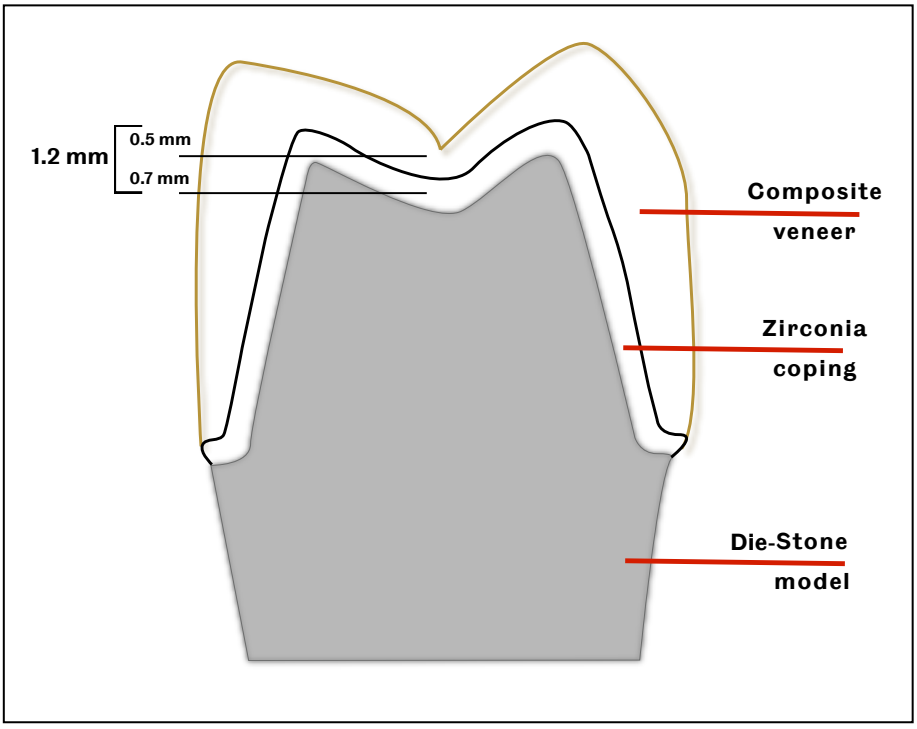


Figure 37: 2nd group 0.7mm zirconia and 0.5mm composite.

5.2.2 Results:

The test was carried out and the failure was recorded in two levels: first fracture and load at restoration failure.

Most samples demonstrated first fracture within the composite veneer, with only some samples fracturing completely at once.

In the first group 1.0mm zirconia, the 10 crowns samples showed an average load at fracture of 1470N with an average of first fracture recorded in 6 crowns of about 1100N (Table 13).

Similar results were found in the second group with the 0.7mm zirconia coping, an average load at fracture of 1460N. First fracture was recorded for all ten samples with an average of around 1000N (Table 14).

The test in all twenty crowns specimens was carried to major fracture and few minor veneer chipping of were observed. All samples demonstrated the composite veneer was still bonded to zirconia coping (Figure 40 and Figure 41) with the exception of one (Figure 42).

Figure 38: Tested crowns of group 1 with 1.0mm thick zirconia coping.

Figure 39: Tested crowns of group 2 with 0.7mm thick zirconia coping

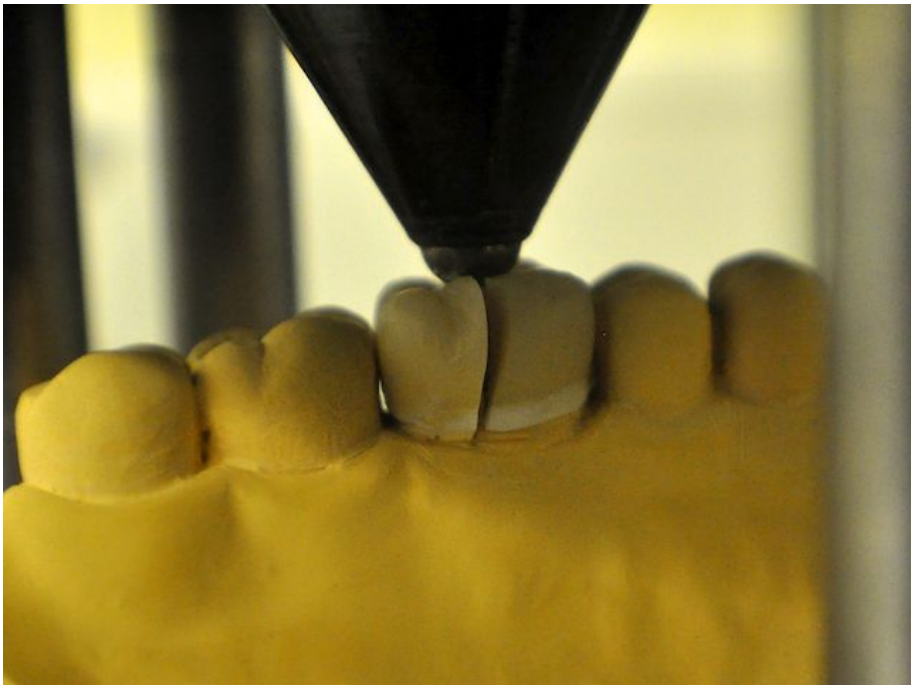


Figure 40: Ball indentor positioned in middle of occlusal surface of crown just after fracture.

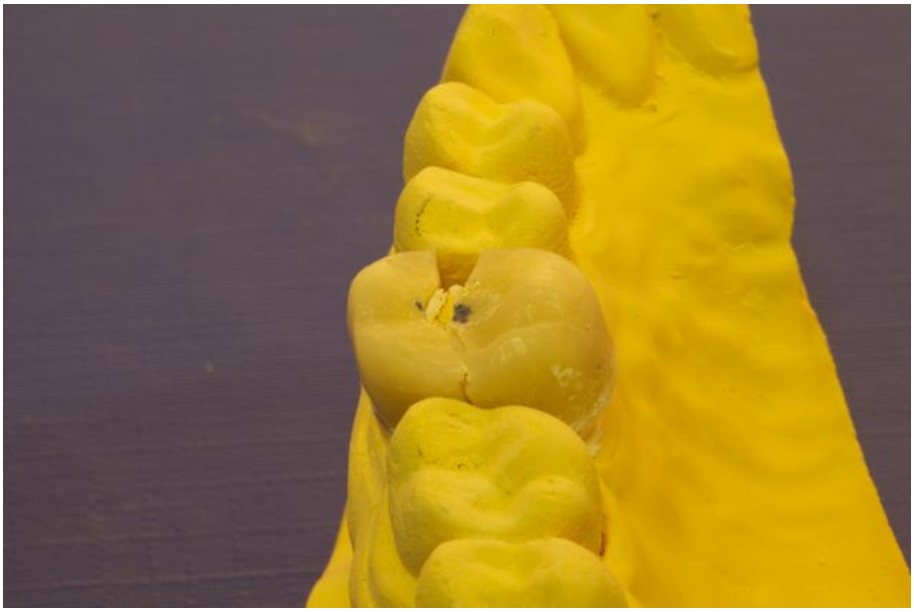


Figure 41: Fracture line in crown with veneer and coping still bonded to each other.

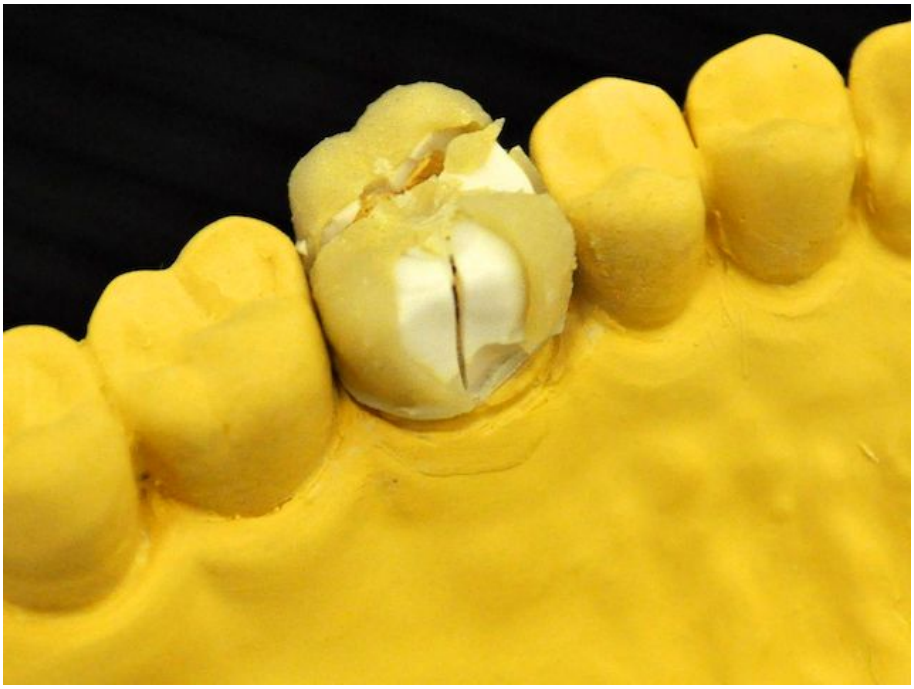


Figure 42: Fractured crown with major delamination between composite veneer and zirconia coping.

Table 13: Fracture loads N for first group tested 1.5mm thick crowns (1.0 zirconia and 0.5 composite).

No.	1st Group	
	1st recorded fracture	Failure
1	700	1562.8
2	1300	1577.7
3	1900	2101.7
4	1100	1642.4
5	-	1858.4
6	1000	907.44
7	1100	1098.4
8	-	1325.3
9	-	1225
10	-	1474.2
Avg	1183.3	**1477.3**
STD	402	355

Table 14: Fracture loads N for second group tested 1.3mm thick crowns (0.7 zirconia and 0.5 composite).

No.	2nd Group	
	1st recorded fracture	Failure
1	1000	1167.5
2	1400	1572
3	800	1152.2
4	740	975.11
5	500	1639
6	659	1355
7	1124	2037
8	1113	1387.2
9	1300	1371
10	1500	1997.4
Avg	1013.6	**1465.3**
STD	333.3	350.5

5.2.3 Discussion:

The so called “Crunch The Crown test” configurations and fabrication process differ between one research and another; Variations include using ball or a bar to apply load (Casson *et al*., 2001). With the differences effecting the occlusal fracture resistance results of dental restorations and may explain the big variation between different investigations (Al-Makramani et al., 2009). It has been stated that the structure and thickness of coping and veneer may affect the fracture resistance of crowns along with mechanical properties of coping/veneer material (Sundh and Sjogren, 2004).

In this study, standardising samples was carried out as much as possible in the hand-built outer veneer since the coping is machined using CAD/CAM. All specimens were fabricated as a clinically expected full contour crown in diameter, shape and cemented to the die as described by (Kelly, 1999). The idealised crown was measured in all sides and recorded with special attention to middle fossa of occlusal surface were ball is placed.

The results gained out of such in-lab based test cannot be directly applied into oral cavity since there are differences in magnitude, direction and repetition of load. Similarly there are differences in the supporting structure and the environment, for example this was a dry condition test.

The results were divided into first fracture, second fracture and finally load during complete restoration failure. First and second fractures were recorded visually and testing machine sensor (Kelly, 1999). However bite force inside human mouth and precisely in posterior region differ but average bite force of 500N is considered depending on various research (Tinschert *et al*., 2001). Research by (Casson *et al*., 2001) tested the fracture load of 10 human extracted teeth mounted in die stone loaded using a bar in cross speed of 1 mm/min and recorded an average of 754N with a standard deviation of 150.

As for the test conditions, the composite zirconia based crowns showed an average resistance higher than the average human bite. In most samples, a minor chipping of the composite veneer occurred which was recorded as the first and second fracture and the lowest recorded first fracture was 500N in

one the second group crowns. In contrast, the highest first fracture was recorded in one of the first group crowns with 1900N. The rest of the crowns just fractured at once through the veneer and coping.

A similar study by (Zahran et al., 2008) tested the fracture resistance of all-ceramic crown made out of yttrium-stabilized zirconium oxide and feldspathic ceramic gave comparable results to composite zirconia based crowns. Zahran's 0.7mm zirconia copings veneered with a 0.8mm VM9 feldspathic porcelain (n=10) gave an average fracture resistance to a ball indenter in a crosshead speed of 1 mm/min of about 1460N with a standard deviation of around 490. The other all-ceramic (n=10) samples were made of VITA mark II with a thickness of 1.5mm in middle fossa showed an average of about 1270N and SD of around 100.

5.3 Finite Element Analysis:

In this research, the use of stress analysis was to give an idea of what happened in the different tested samples and how the different materials in our laminated samples reacted as one structure.

5.3.1 Materials and methods:

ANSYS 11.0 (ANSYS, Inc. USA) programme was used to design the specimens and analyse the stress on them. The geometry was built and a virtual load was applied to the structure and the results are shown in pictured colouring different parts of the tested structure.

Discs:

For the disc samples, the first step for building a virtual specimen was to determine the structure properties such as the materials' elastic modulus and Poisson's ratio, by choosing elastic than isotropic from the sliding menu on the left of screen. Also from preferences, structural and h+method was chosen and then by pressing element edit: solid and choose: 8 node 185 for the 3D structure.

The disc sample was then drawn manually by creating the key points and connecting them and verifying the areas before rotating the shape 180° to creating a half disc 3D shape. The supporting ring was taken into mind hence the disc surface was divided into three invisible sections in order to select one of them as the place of support before applying the load (Figure 43). After building section, the structure is then meshed (Figure 43). After meshing comes the solution step, which starts by defining the load, apply, structure, displacement on area (bottom ring supported area) and clicking on al DOF.

The load was created along the Y-axis in the shape of circle of approximately 4mm diameter as the actual test using the ball indenter. All the drawing was according to the samples, supporting ring and ball indenter diameters. The material details: elastic modulus and Poisson's ratio were used in the calculation.

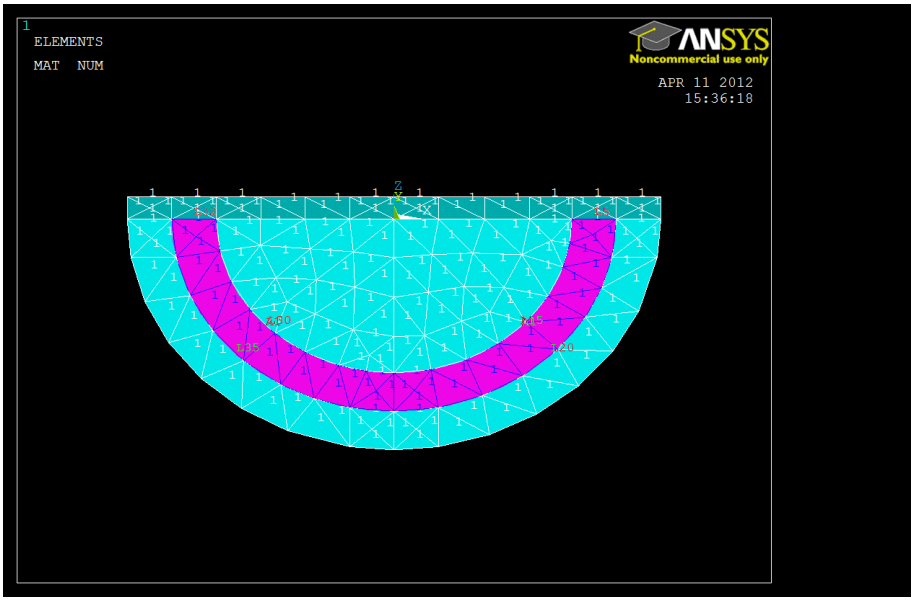


Figure 43: Screen shot of disc bottom layer shape after meshing and the supported area by ring is shown in purple.

The same steps are done with bi-layered discs taking in account the diameter differences. Coming to the solution stage, defining the load by determining the displacement place and type, which in this case the lower supported by ring surface was chosen in all DOF. The load was applied on the designed geometry vertically along the Y-axis (occlusally) simulating the fracture test. The force is then set on the Y-axis in a minus value in Newton's and then solve by choosing current LS and a message will appear when the solution is done. There are various ways to look at the result of the force applied on the structure. In this study, a 1st principal stress was chosen from general post icon and a colourful picture of the compressed structure is shown with clear deformation caused by force. Different colours represent the amount of compression (blue) and stresses (red) in different parts of the structure with a colours guide below showing the values in MPa.

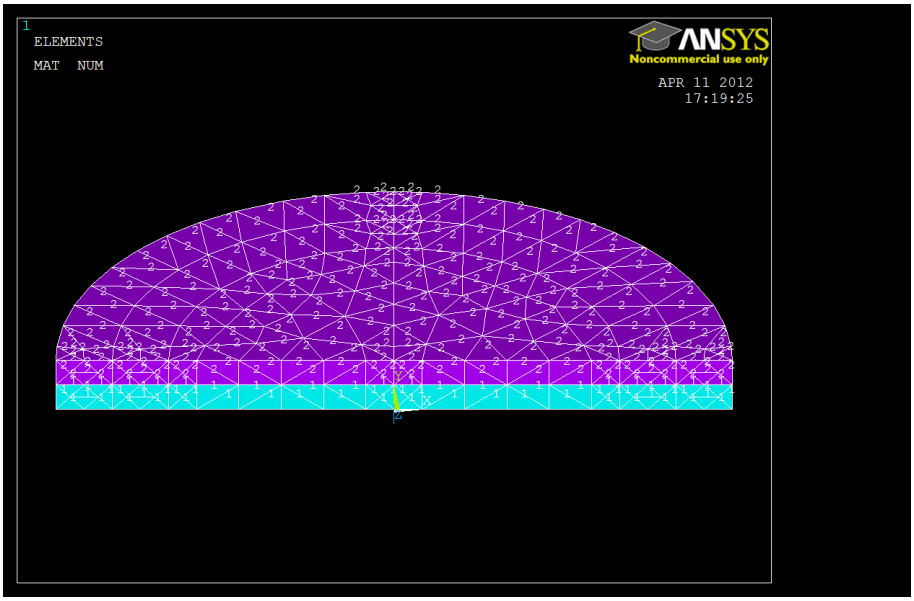


Figure 44: Screen shot after meshing the two layers: layer 1: zirconia in light green and layer 2: composite in purple.

Crowns:

To design the crown, a screen shot was taken of the zirconia coping design the CEREC design software and edited with a mesh to create a Y and Z axis (Figure 45). Depending on the actual crown thicknesses (1.5mm), the outer veneer is schematically drawn (0.5mm). Manually, different points were recorded for the die, cement layer, zirconia coping and composite veneer and Y and Z-axis coordination for each point was recorded in excel files. The coordinates were transferred to a note document after typing in: prep7:/. Saving the numbers in such form allowed easy transfer of the shape into the ANSYS program. Doing so creates the key point in the shape of a quarter of a crown as in the hard copy taken CEREC screen. The points were connected to form 4 areas of die, cement, coping and veneer.

The quarter of crown is then rotated 180° to create 3D half of a crown. The properties of each material were typed in and assigned with its area (Figure 46). The load was defined by choosing displacement on all DOF of the base of structure, and than determining the force. The force was applied occlusally in an approximately 4mm diameter circle along the Y-axis. The drawn circle created a number of scattered nodes, which the applied force is equally divided between. Two amounts of loads were chosen, 500N as an average of different natural bite force (see Discussion: 5.2.3) and 1400N as the average

fracture recorded with crunch-the-crown test done in-lab. For displaying the results, 1st principal stress was chosen to point the stress distribution in the structure after applying virtual load to it with a colour guide below showing stresses values in MPa.

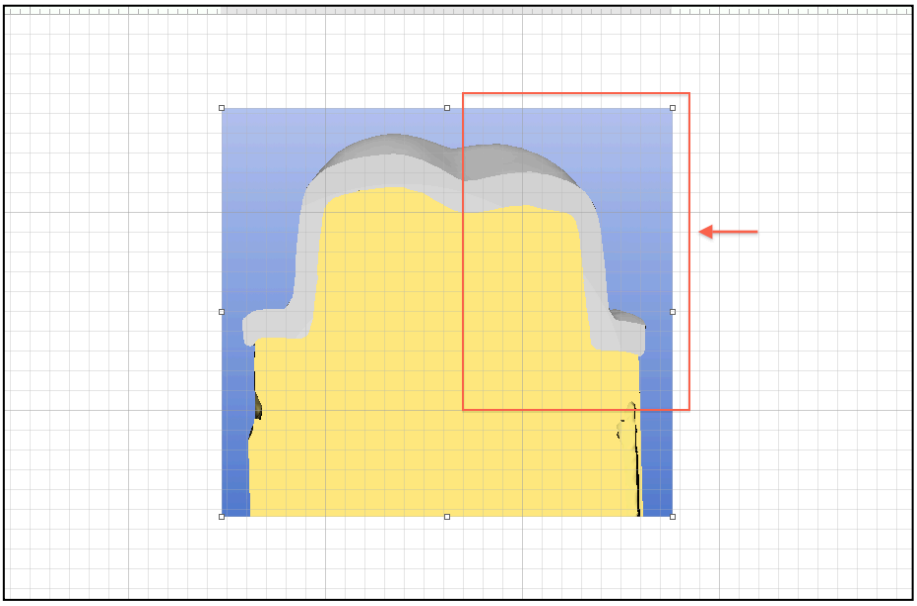


Figure 45: Cross section of the coping design taken from CEREC after adding meshing, and the half that was used for numbering boxed in red.

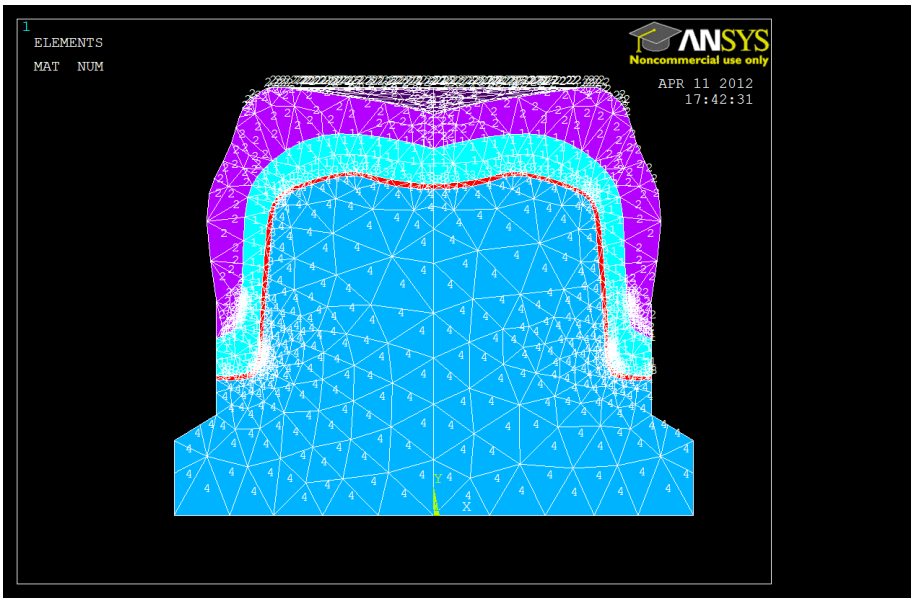


Figure 46: Screen shot showing the structure different layers after assigning them and meshing.

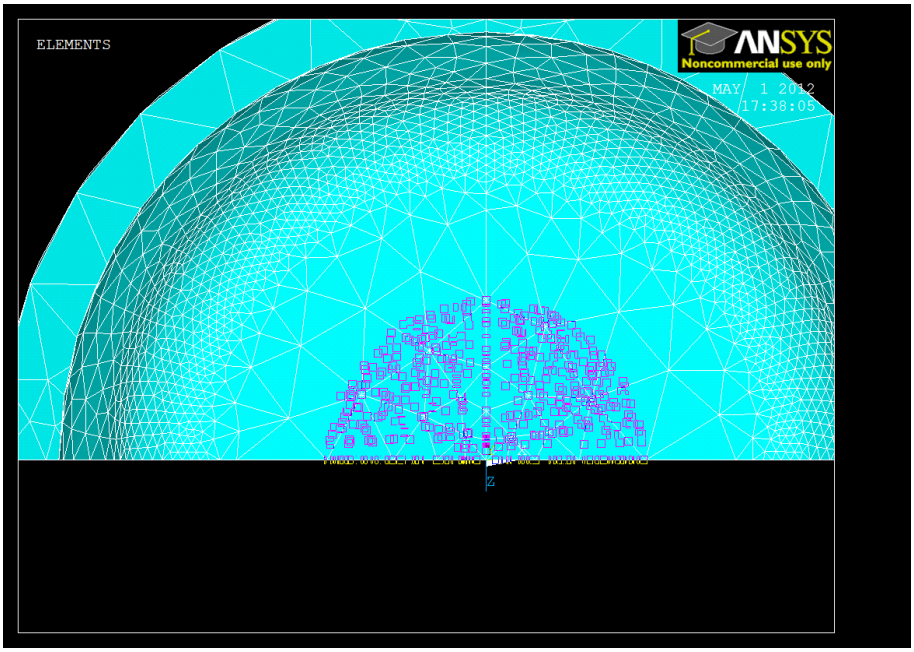


Figure 47: Circle of where the force is applied on the scattered nodes.

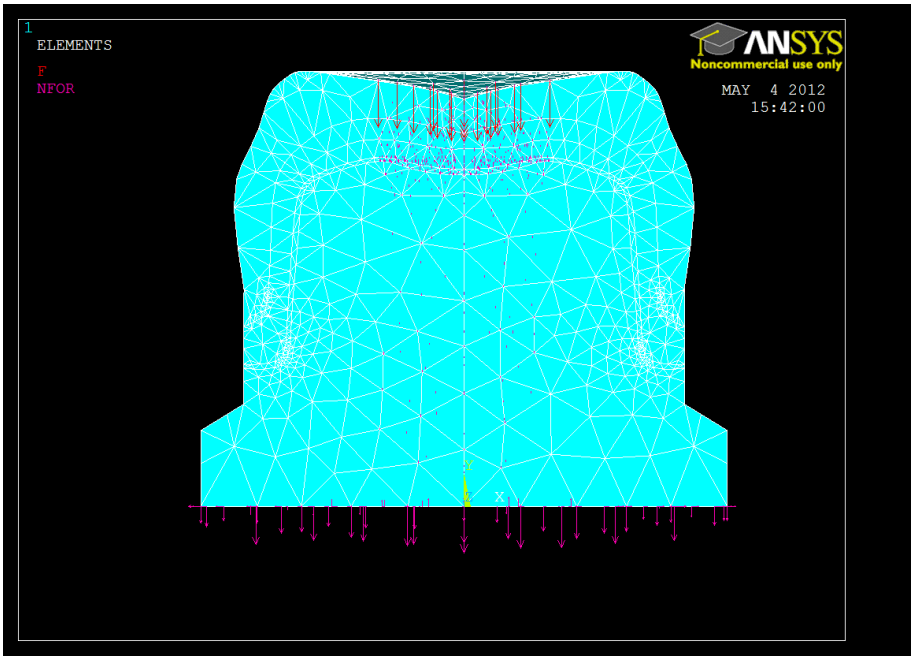


Figure 48: Screen shot showing the force applied occlusally (upper arrows) and where the structure base is fixed (lower arrows).

Table 15: Properties of materials used in FEA.

Material	Elastic modulus (Gpa)	Poisson's ratio	**Reference**
Zirconia	210	0.32	VITA manual
Composite	4.5	0.28	VITA manual
Ceramic	65	0.20	VITA manual
Die (dentine)	18.6	0.32	(Zarone *et al.*, 2006)
Cement	18.6	0.28	(Zarone *et al.*, 2006)

5.3.2 Results:

After applying the load to the designed structure, the result can be seen in different ways depending on the type of material and the user's investigation. For this study, our attention was to observe the stress generated in our bench tested laminate samples and compare them. The 1st stress principal was chosen from general post than nodal solution drop list, to reveal compressive stress and tensile stress, which is one of the causes for ceramic restoration failure (Mollers *et al*., 2012). A coloured deformed structure is then shown with a colour guide below the highest and lowest stress generated in the 3D sample after the a virtual force.

3D virtual disc:

Starting with three single layered discs, the virtually load was determined as the average fracture load of each material group in the in-lab tests. All samples were supported as the ring supporting area and the vertical load was divided equally on selected nodes occlusally. The loads were 50N, 80N and 500N on discs of composite, ceramic and zirconia respectively. All three single layered discs showed a similar stress distribution having the maximum tensile stress (in red) in the centre of the bottom of the disc and some part of top surface opposing the supporting ring. The compressive stress (in blue) where concentrated in the bottom surface in contact with the supporting ring. In a composite of a 4.5Gpa elastic modulus, the maximum tensile stress was around 16MPa. With a stiffer ceramic (E modulus of 65Gpa) the maximum tensile stress was 25MPa under a higher load. The stiff core material (zirconia, E-210Gpa) showed higher tensile stress of around 160MPa under 500N load.

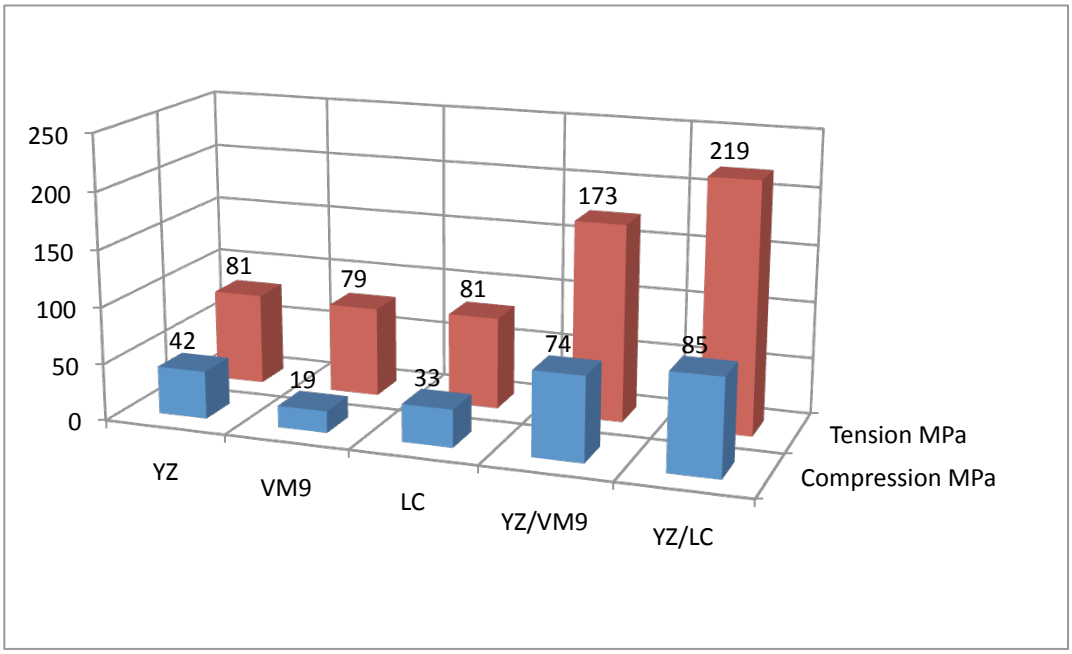


Figure 49: Tensile and compressive stresses (MPa) in single and Bi layered discs under 250N load.

The FEA showed a stress distribution in the bi-layered discs similar to the single ones for the base layer (zirconia) but with a higher tensile stress in the top surface when veneered with composite rather than veneered with the stiffer ceramic veneer. The ceramic veneer seems to gain higher tensile stress than the composite one in the range of 50-90MPa against steady compressive stress level of around 17MPa for composite veneer. All the previous stresses were with different loads, and when comparing them when using same loads of 250N the results were higher tensile stress in zirconia base layer when veneered with composite (220MPa) against less stress zirconia (170MPa) when veneered with ceramic.

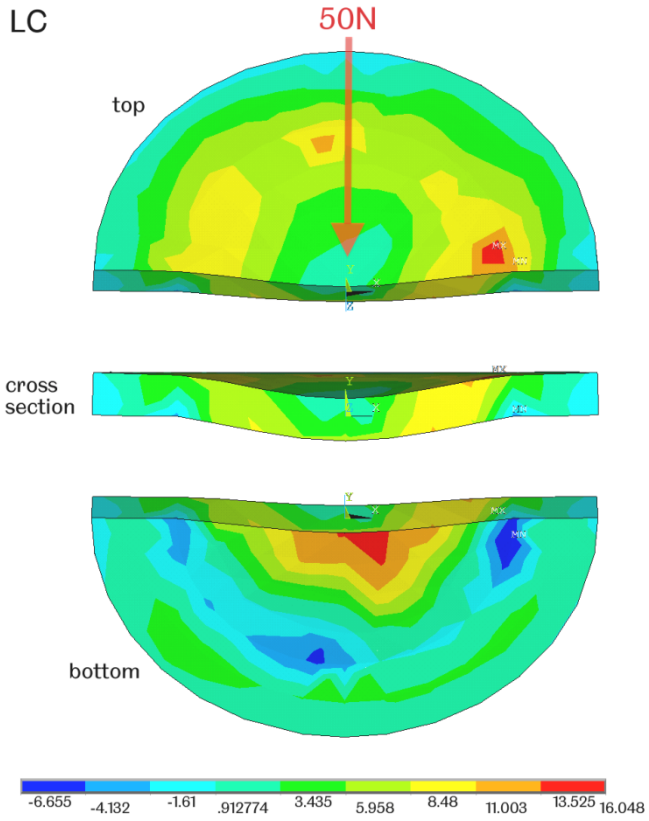


Figure 50: 1st principal stress distribution of composite disc (top, bottom and cross section view).

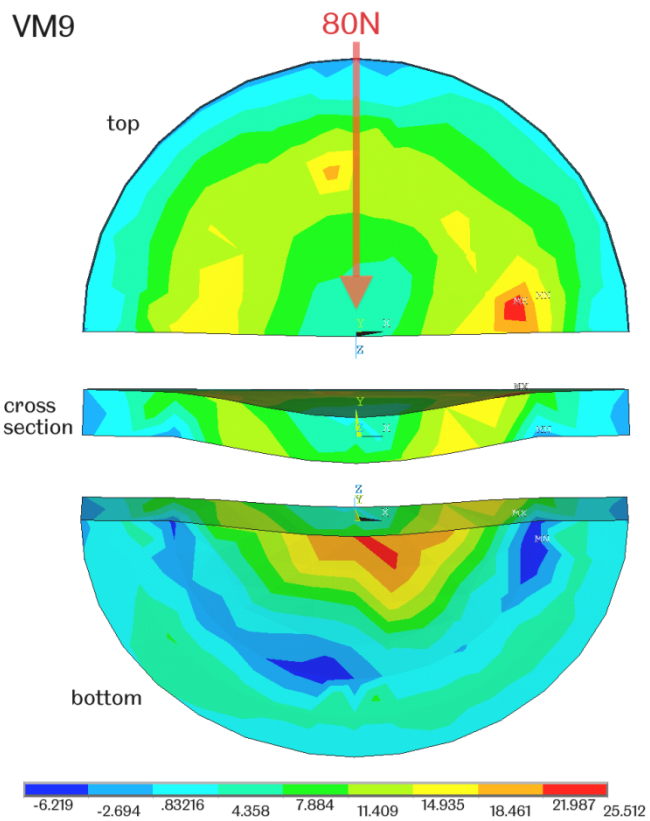


Figure 52: 1st principal stress distribution of ceramic disc (top, bottom and cross section view).

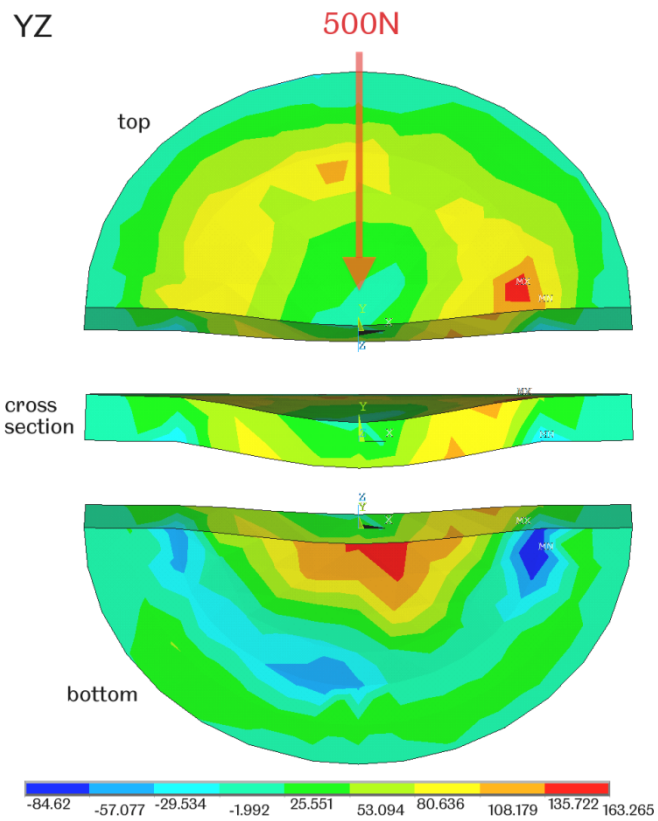


Figure 51: 1st principal stress distribution of zirconia disc (top, bottom and cross section view).

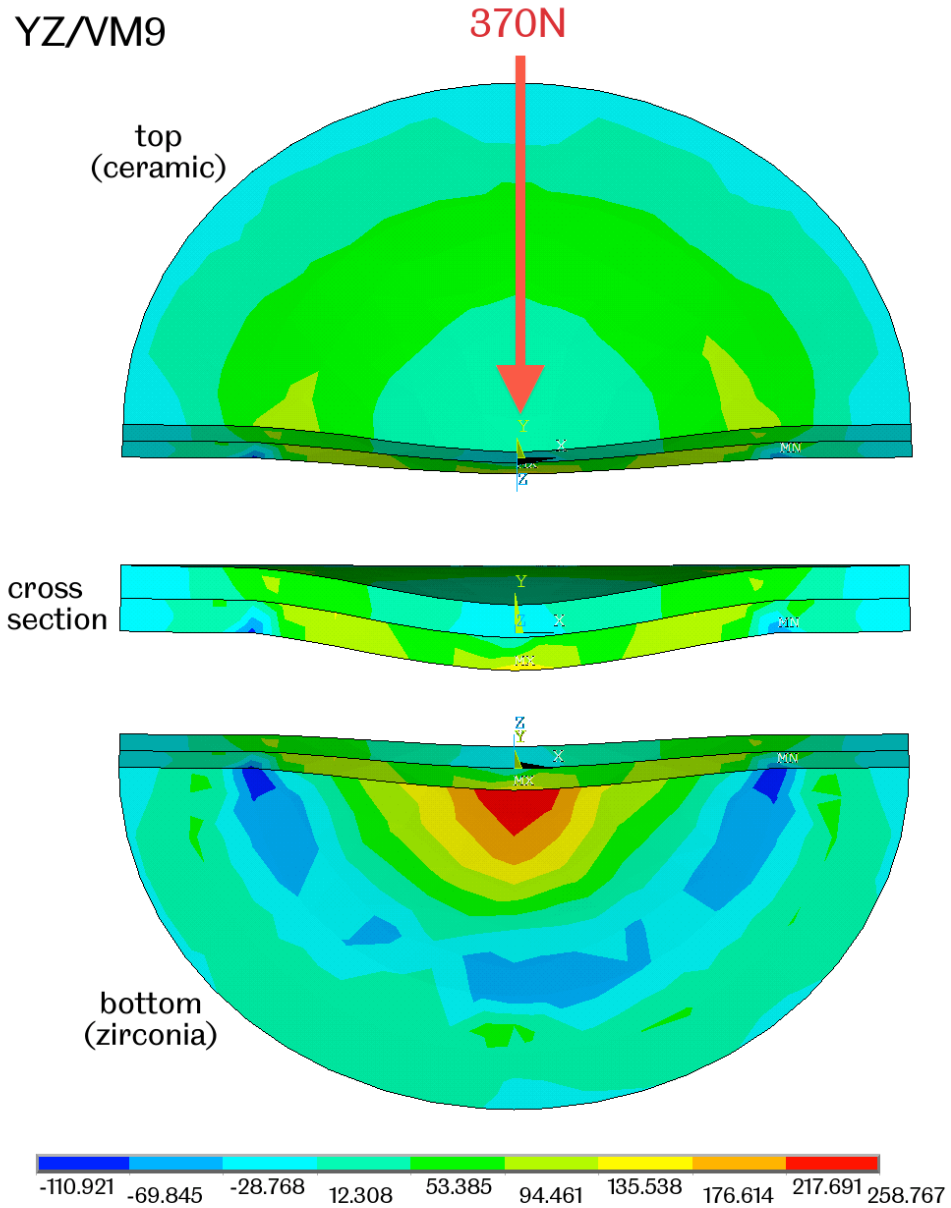


Figure 53: 1st principal stress distribution of zirconia/composite disc (top, bottom and cross section view).

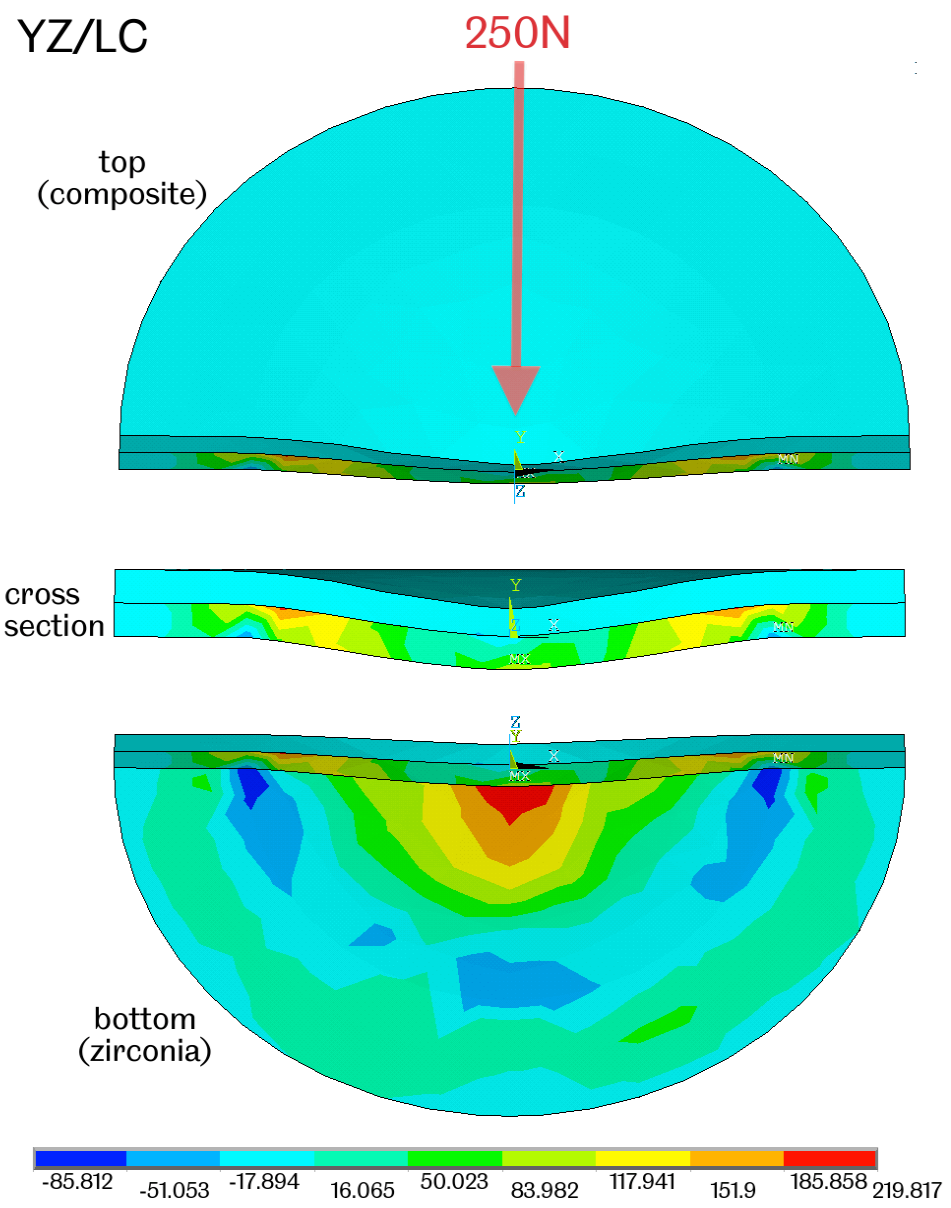


Figure 54: 1st principal stress distribution of zirconia/ceramic disc (top, bottom and cross section view).

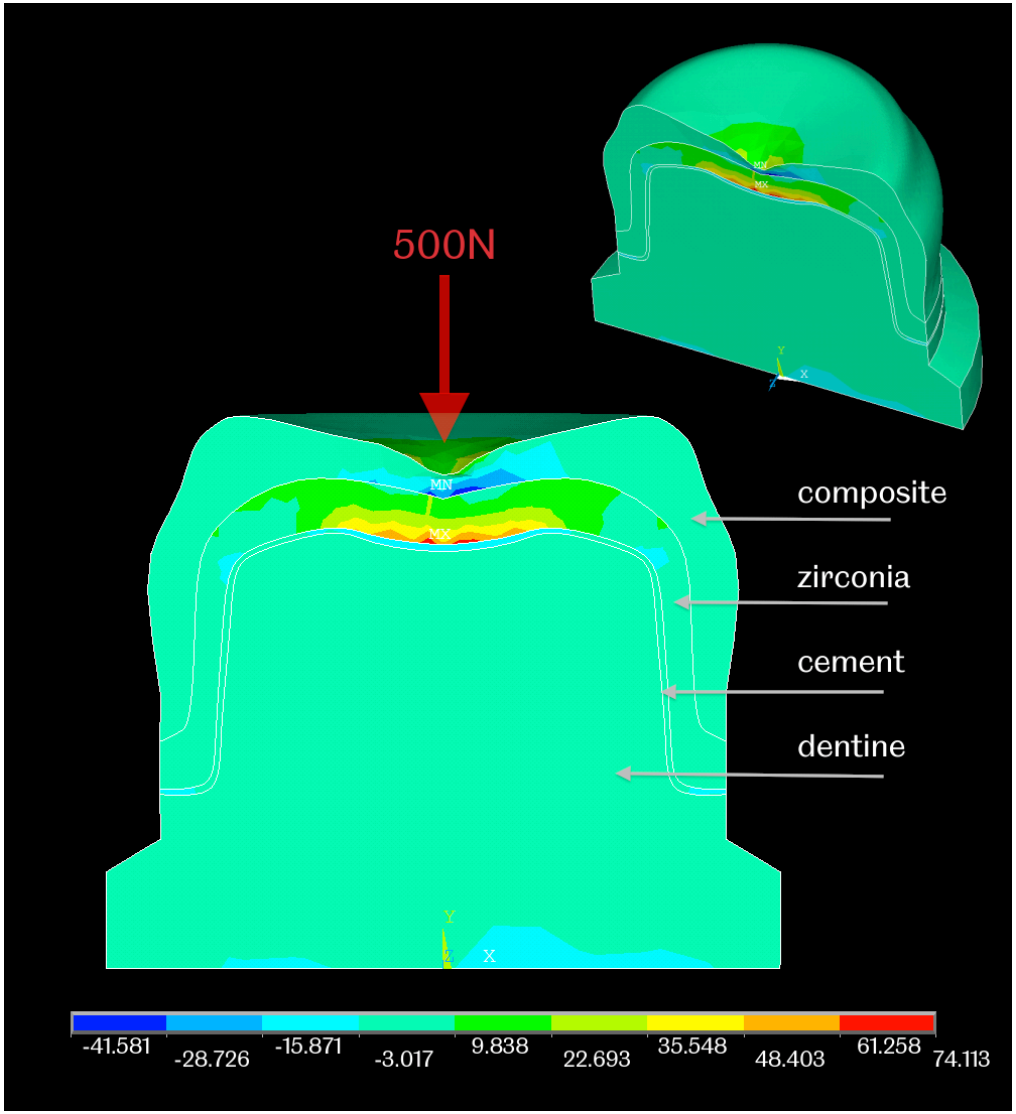


Figure 56: Cross sectional view of 1st principal stress (MPa) distribution of zirconia/composite crown and sphere after virtual load of 500N

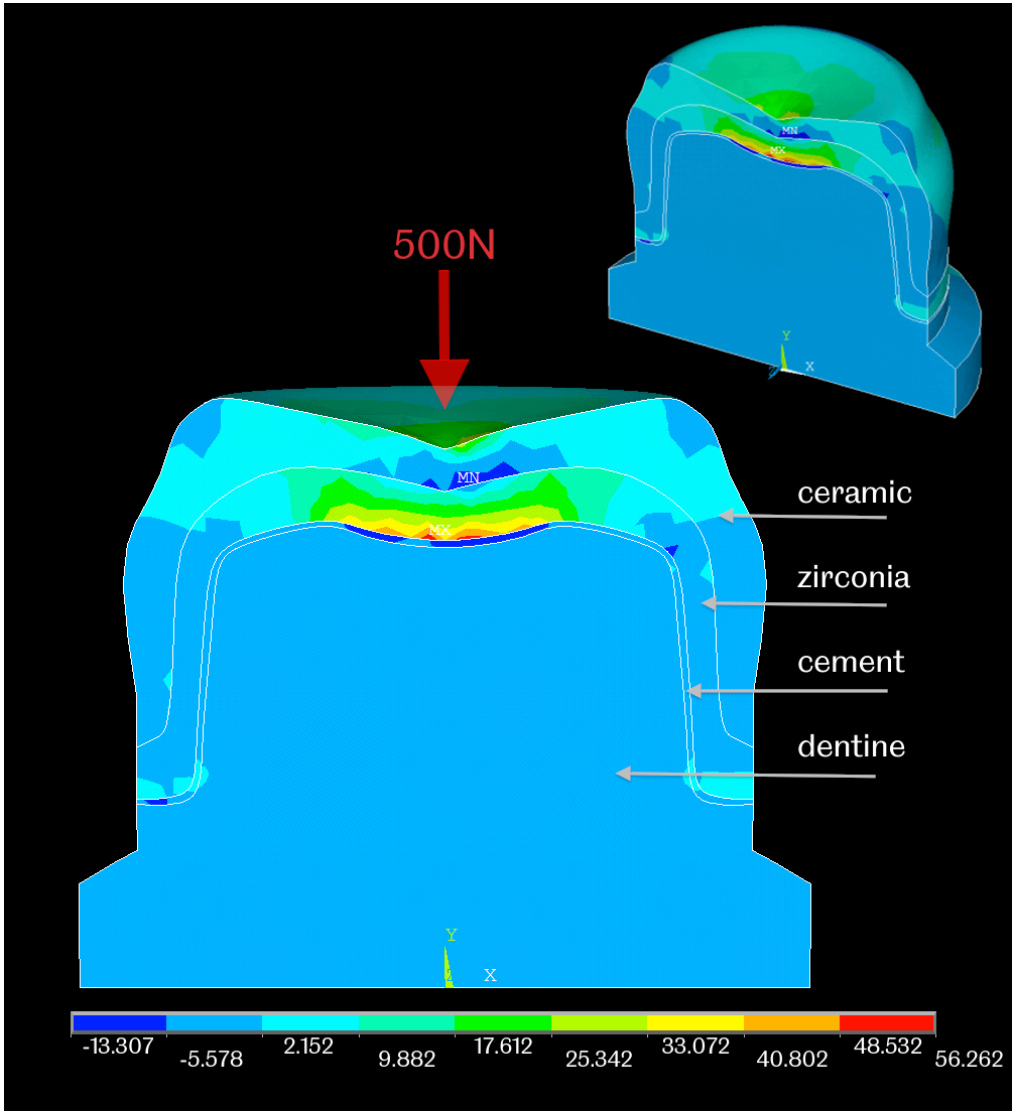


Figure 55: Cross sectional view of 1st principal stress (MPa) distribution of zirconia/ceramic crown and sphere after virtual load of 500N

3D virtual crown:

The 1st principal stress distribution on crown and sphere was created after applying an imitated load of 500N occlusally in the middle fossa evenly distributed on the selected nodes. With crown veneered with a 4.5Gpa stiff composite, the highest tension point was shown under the loading area in the bottom of the zirconia coping in the range of 70MPa, and peaks around 35MPa in the composite veneer. Also in the base of the composite veneer the highest compression point was created around loading region of about -40MPa and the cement layer settling in between the tensile and compressive stresses peak.

Under the same circumstances, the same virtual load was created on stiffer (65Gpa) ceramic veneered crown resulting in a high tension in the bottom of zirconia around 56MPa and compressive stress peaking at about -13MPa in the bottom of the veneer and in the cement layer. The highest tension in the ceramic veneer was recorded around the loading region of about 30MPa. The compressive stress appears more in the ceramic veneered crown than composite one in dentine, cement, zirconia and ceramic. But the tension in zirconia is higher when covered with composite rather than a stiffer ceramic.
The load was also applied on the zirconia coping alone which showed the same stress distribution tensile stress zone at the base (87MPa) as when veneered with composite or ceramic but differing in a compression peaking in the top of coping at about -32MPa.

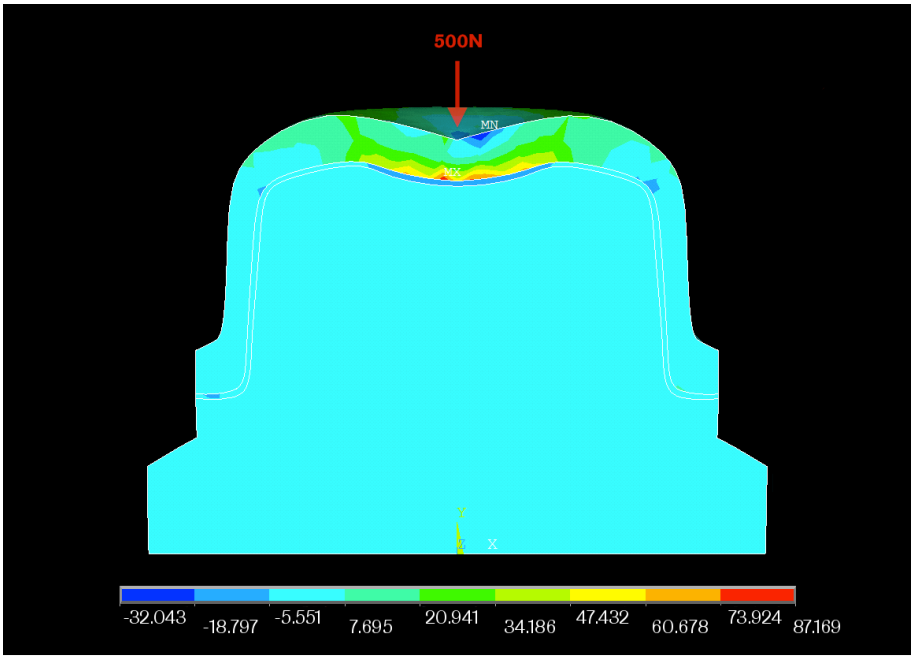


Figure 57: Zirconia coping 1st stress distribution under 500N load.

Another batch was done but with an occlusal load of 1400N as the average fracture resistance of the in-lab test (crunch-the-crown). Almost the same stress distribution was found but with a higher figures as expected. Between the three samples the highest tensile stress was recorded in zirconia un-veneered coping, after that comes the composite veneered and the least stressed was the ceramic veneered crown. As for compressive stresses, the highest record was with the crown veneered with composite after that comes the un-veneered zirconia and the least compression was with the ceramic veneered crown.

In both structures (disc and crown) where the zirconia was veneered with composite, the tension was concentrated in the bottom of zirconia layer. This stress was found in almost in the same region with both zirconia/ceramic disc and crown but less stressed. In table 15, the stresses (tensile or compressive) at the base of each top and bottom layers are summarised for disc and crowns of both composite and ceramic veneered zirconias.

Table 16: The stress (MPa) in the base of each layer in disc and crown samples.

	Zirconia/Ceramic		**Zirconia/Composite**	
	Zirconia	Ceramic	Zirconia	Composite
Disc 250N	170	-19	219	-17
Crown 500N	56	-13	74	-41

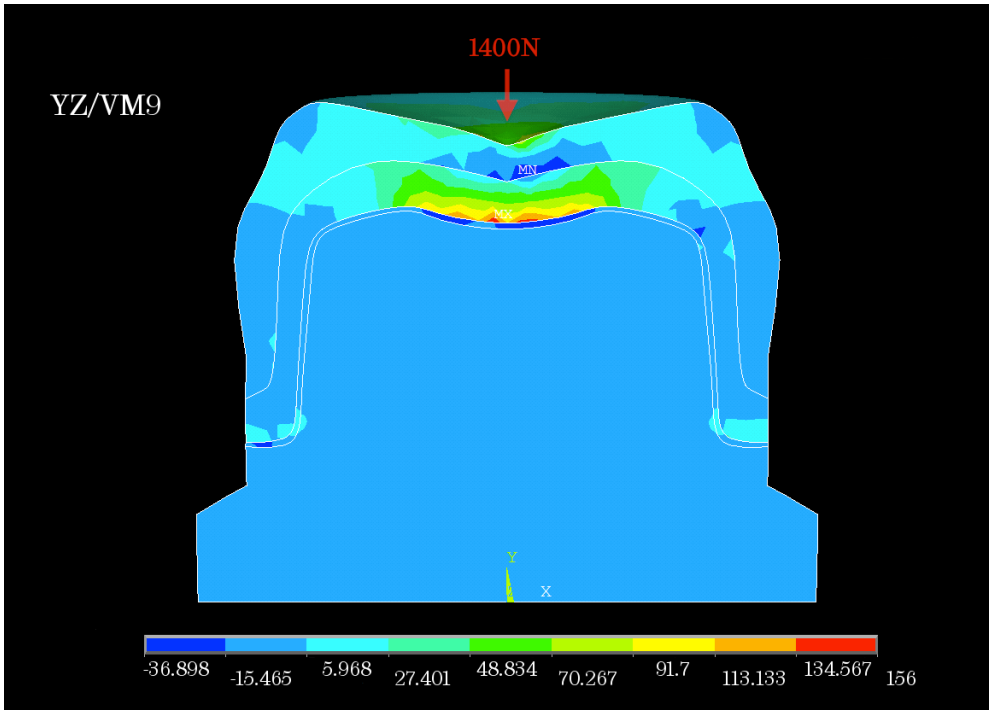


Figure 59: Zirconia-Ceramic crown under 1400N load

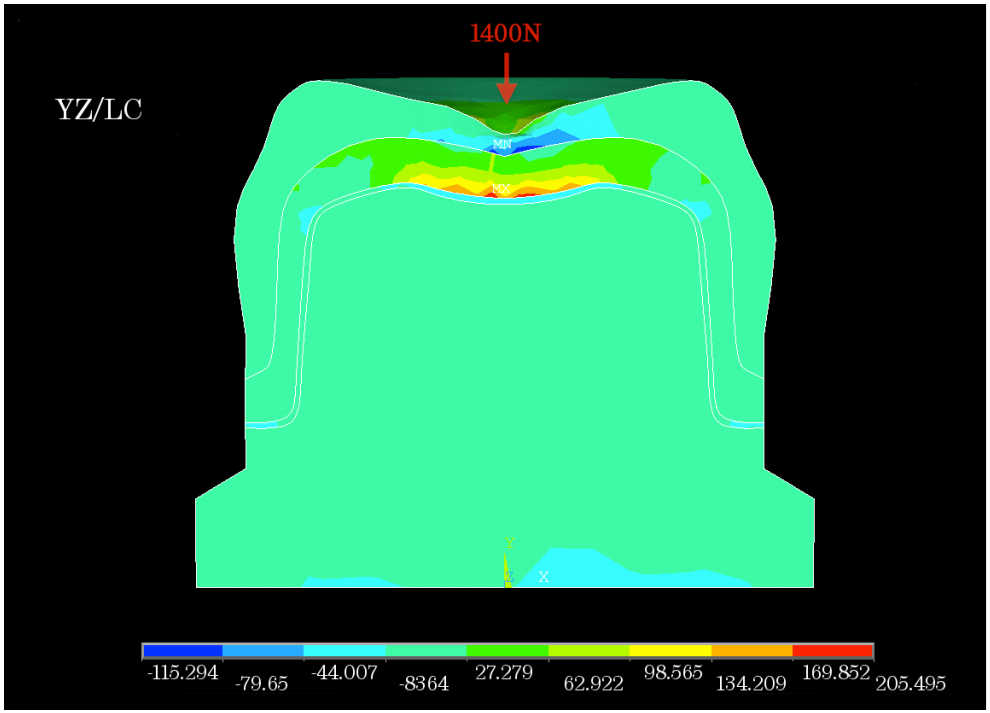


Figure 58: Zirconia-Composite crown under 1400N load

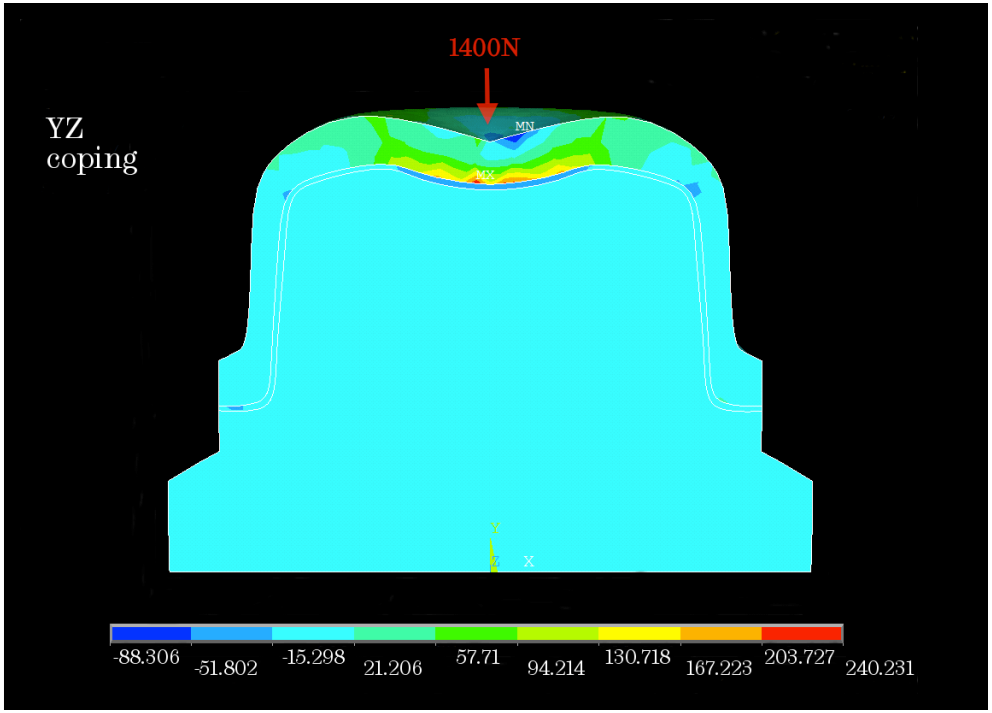


Figure 60: Zirconia coping under 1400N load

5.3.3 Discussion:

Finite element analysis was used to simulate the in-lab tests done on our tested restorative materials in different shapes to display the stress points after applying load on those structures. The virtual schematic specimens do not necessarily reflect the actual samples due to the fabrication process which goes through different stages mainly by hand (Mollers *et al*., 2011). In this FEA study, it was assumed that a good bond between the different layers in the virtual bi-layered discs and veneered crowns, regardless any faults in actual model. Checking the stress zones is essential in most application fields since the stress, even if below failure point, are considered as a major cause of crack propagation hence system failure (Zarone *et al*., 2006).

The FEA for single layered discs showed a 1st stress principle correspondingly distributed having the highest tension in the base of disc (Figure 50, Figure 51 and Figure 52). With the bi-layered structure, the base zirconia layer showed a similar tension in the base when veneered with ceramic and composite and even more on top surface with composite veneer (Figure 54 and Figure 53). This observation matches what other studies revealed that: low stiff veneer passes the load to the core material and as a consequence a higher stress to the core is generated (Mollers et al., 2011) (Ausiello et al., 2002).

It is believed that restoration failure is caused by high tensile stress areas that a crack is then initiated from and grows to the outer veneer (Mollers *et al*., 2011). The same observation was founded in zirconia based laminate discs and crowns having the highest tension point in the bottom of zirconia base layers.

Different effects on the zirconia layer have been observed between composite and ceramic veneers. When veneered with composite, higher tensile stress is generated at the base of zirconia in both disc and crown samples under the same virtual occlusal load. With ceramic veneer, stress was distributed in different levels having the highest point under where the load was applied. Such stress was not found in composite layers almost steady stress was shown.

Relatively similar to the bi-layered discs, the virtual veneered crown showed a higher stress to zirconia core when veneered with lower modulus composite than ceramic veneered ones.

6 General Discussion:

Many studies discuss the idea of improving the strength of high strength all-ceramic restorations or their veneering material (Schmitter *et al.*, 2012) since fracturing while functioning inside the mouth is the biggest challenge for such systems. Researchers focused on the veneer chipping off, and how the CTE dissimilarity may contribute in system failure and as a consequence, some studies suggested to work on improving the veneer of all-ceramic restoration since the zirconia has such a high level of strength (Coelho *et al.*, 2009). Fractured ceramic veneer can be repaired intra-orally using composite with the risk of strength reduction and colour mismatch of the veneer (Hammond, 2009). Different ways were investigated in order to enhance all-ceramic restoration by using a strong material for the framework such as partially stabilized zirconium oxide blocks with yttrium oxide (zirconia for short), but such methods do not rule out veneer's contribute in restoration fracture resistance, as found by (Guazzato *et al.*, 2004b). In other words, zirconia frameworks benefits the high strength performance but it has to be taken in mind that the veneering material does affect the strength of whole restoration. Veneering zirconia substrate is a key point for achieving an aesthetic restoration since it is considered as an opaque material (Zhang *et al.*, 2012) and abrasive to the opposing teeth. Ceramic veneers have approved their aesthetics, but the nature of such materials that they cannot withstand high tensile stresses which eventually cause ceramic to fracture (Casson *et al.*, 2001). Ceramic veneer chipping rate were higher with zirconia substrate than recorded with metal frameworks (Sailer *et al.*, 2006).

Some have tried veneering zirconia copings with CAD/CAM milled lithium disilicate ceramic (360MPa), which did improve the restoration's fracture resistance compared to conventional ceramic (100MPa) veneered zirconia (Schmitter *et al.*, 2012). It was concluded that using of less stiff restorative material, such as the low modulus composite, generate stress in tooth and restoration substrate (Ausiello et al., 2002) . However, (Zarone et al., 2006)

have concluded that using materials such as composite with a lower elasticity, closer to that of the natural tooth, may lower stresses generated between layers and therefore simulate natural teeth reaction.

The problem is complex because the clinical indications for restorations differ and therefore some questions can arise such as:
Is the currently used all-ceramic restoration not strong enough to with stand bite and chewing forces? Aren't some of such systems contraindicated in strong bite zones to avoid damaging opposing natural teeth? In other words, crown should be weaker than the tooth in order to act as 'fuse box' that will break before the tooth.
Or is their major drawback the veneer chipping and delaminating off the outer surface?
Do we really require all the strength in a restoration? Or perhaps it is over toughened and over engineered to what's needed to function inside a human bite, especially with people with a strong bite. Or maybe all the previous questions depends on the case and application conditions?

Different tests were used in this study: Fracture Resistance (discs and crowns), BFS and FEA. Testing samples in-lab in load-to-failure methods does not reflect the actual failure mechanism that occurs to the restoration intra-orally. This failure is determined by the stress zones that are highly influenced by the dissimilarity in geometry and modulus of elasticity (Thompson *et al.*, 2011). For natural teeth and crowns, such tests are performed in order to test the fracture resistance of those samples but contrast in methods between different studies is considered when comparing results (Casson *et al.*, 2001).
Finally, The FEA is considered a useful tool in dental research helping in predicting what mechanical application on restoration and teeth that contribute in improving materials properties and restoration designs (Ausiello et al., 2002).

In all performed in-lab and computerised virtual evaluations, the findings should be taken with regard to the differences between the tests controlled environment and the restoration in action inside human's mouth.

The bonding between zirconia coping and composite veneer appears adequate in these tests but requires further investigation that will simulate a wider range in conditions mimicking oral cavity and chewing forces.
In the literature, some concluded that mechanically modifying zirconia surface may cause weakness of material and some assumed the opposite. Specifically talking about shot-blasting the sintered zirconia with alumina particles (Qeblawi *et al*., 2010). Two groups of 0.5mm zirconia were tested, one shot-blasted with Al_2O_3 50μ particles and the other unmodified, showed a slightly higher BFS of the shot-blasted group. Regardless of it improving the strength or not, it does appear to weaken the zirconia. Under the dry test conditions, using a universal primer on shot-blasted zirconia surface showed an observable bond between fractured parts of discs with the composite still bonded to zirconia.
Testing the three single materials showed an expected fracture resistance and BFS results as for veneer (composite and ceramic) and core (zirconia) materials. When veneering the zirconia substrate, in this case disc specimens, the YZ/VM9 (1.0mm) showed a higher fracture resistance than ceramic alone and un-veneered 0.5mm zirconia. The less stiff composite veneer resulted in lower fracture resistance with YZ/LC and YZ/Sin laminates compared to ceramic veneered ones. This is in accordance with the veneers stiffness. Bearing in mind that the resin veneers had an elastic modulus of 4.5 and 3.1Gpa, it did in fact raise the fracture resistance of a 0.5mm zirconia from about 120N to 240N when veneered with 0.5mm composite.

The BFS results of bi-layered discs, two different equations were used and both showed different results. In both calculations zirconia/composite laminates showed a higher BFS than zirconia/ceramic ones even though the fracture load was higher with ceramic veneered ones. This is in accordance

with findings of FEA of bi-layered laminates, where the 1st principal stress distribution showed a high tensile and compressive stresses generated in zirconia when veneered with composite rather than ceramic with same virtual occlusal load. Similar findings of the higher tension restoration substrate were found in other studies that used low stiff veneering materials to veneer metal substructures (Mollers *et al*., 2011). The FEA of crowns showed the highest tensile stress zone being in the base of zirconia coping under the area of load in both ceramic and composite veneered samples. This may explains what researchers observed previously that the crack begins from the base of the bottom layer and continues to the top surface (Guazzato *et al*., 2004a).

7 Conclusion:

In general, zirconia/composite laminate disc specimens showed comparable results to currently used zirconia/ceramic laminates and certainly superior strength than full composite structure, however:

Veneering zirconia with composite may cause high tension in the zirconia compared to a zirconia veneered with ceramic (from FEA).

Using universal primer did enhance bonding between composite and zirconia when applied after shot blasting sintered zirconia surface using 50µ Al_2O_3 particles. The zirconia and composite showed high bond even after fracture and breaking into small segments.

In this study, it was possible to produce full crown using the same methods used in dental laboratory practice made out of a zirconia coping and veneered using light-cured composite. Such crowns did withstand fracture loads close to that experienced with different types of fixed restorations as found in various studies.

For the FEA with the assumption of a good bond between zirconia and veneer, virtual discs and crowns showed high tensile stress located at the base of zirconia coping under the area of load with ceramic veneered samples and even higher with composite veneered samples. This can explain fracture initiating from the base layer of the specimens, as observed by other researchers as well.

8 Further Work:

- More tests need to be carried out to assess substructure and veneer bonding reliability under different conditions.
- Other tests mimicking oral environment should be carried out on zirconia/composite crowns before applying it in clinics.
- Produce three unit fixed bridge using same methods and subject them to different assessments.
- Use different toughened copings with other types of composites veneers in the same manner and compare it with current results.
- The current used equations to calculate Biaxial Flexural Strength for bi-layered structures needs to be explored more in the case were lower elastic material is the top layer facing the load.

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10 Appendix:

Appendix table 1: Fracture resistance of 1.0mm zirconia discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (gf)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	53788.98286	0.736526169	0.097922812	1753074.612	527.6699219	0.736526169	0.097922812
2	1	45856.97974	0.791299679	0.069253798	1756948.906	449.8569712	0.791299679	0.069253798
3	1	61649.83914	0.701209784	0.122743633	2086404.368	604.7849219	0.701209784	0.122743633
4	1	53514.98378	0.596173081	0.090465183	1756678.856	524.9819909	0.596173081	0.090465183
5	1	51322.90282	0.763491125	0.083780863	2061483.654	503.4776766	0.763491125	0.083780863
6	1	28002.66287	0.497803112	0.029057252	1740150.911	274.7061228	0.497803112	0.029057252
7	1	48942.6958	0.862917204	0.082574825	1827757.17	480.1278457	0.862917204	0.082574825
8	1	57281.26394	0.818323371	0.099963755	1890044.505	522.5402499	0.846653433	0.114096956
9	1	54398.54984	0.711636604	0.095263251	1911504.763	533.649774	0.711636604	0.095263251
10	1	56645.46171	0.511938815	0.097023042	1830795.742	555.6919793	0.511938815	0.097023042

Appendix table 2: Fracture resistance of 1.0mm zirconia discs with pre-sinter roughening (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (gf)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	37176.24614	0.820255073	0.056197282	2061301.599	364.6989746	0.820255073	0.056197282
2	1	38260.15742	0.973965095	0.058234384	1712369.497	375.3321443	0.973965095	0.058234384
3	1	43594.75444	0.998711234	0.073190759	1684226.859	427.6645411	0.998711234	0.073190759
4	1	49557.40454	0.877023294	0.087986505	1617134.338	486.1581386	0.877023294	0.087986505
5	1	44390.98171	0.809854877	0.071538322	1619104.615	435.4755306	0.809854877	0.071538322
6	1	51304.25229	0.860003997	0.094712594	1758958.54	503.294715	0.860003997	0.094712594
7	1	37442.09202	0.941312476	0.052892268	1589881.152	367.3069227	0.941312476	0.052892268
8	1	42798.24743	0.619986786	0.066930293	1839318.468	419.8508073	0.619986786	0.066930293
9	1	45028.83499	0.846762654	0.067900681	1854094.584	441.7328712	0.846762654	0.067900681
10	1	48995.27462	0.716733176	0.077597932	1809081.043	480.6436441	0.716733176	0.077597932

Appendix table 3: Fracture resistance of 1.0mm ceramic (Mark II) discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (gf)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	8353.139571	0.438343737	0.013899056	1663839.499	81.94429919	0.438343737	0.013899056
2	1	8493.327058	0.335406623	0.010821061	3022320.108	83.31953844	0.335406623	0.010821061
3	1	8374.822857	0.201196478	0.006842915	2236850.79	82.15701223	0.201196478	0.006842915
4	1	8673.752924	0.235503473	0.007933141	1627534.347	96.88733567	0.207993928	0.007907529
5	1	7969.822626	0.153019098	0.005267402	1603705.924	78.18395996	0.153019098	0.005267402
6	1	9091.64382	0.300694272	0.009914654	3535697.049	89.18902588	0.300694272	0.009914654
7	1	9035.519632	0.196676306	0.00698469	1493792.404	88.63844759	0.196676306	0.00698469
8	1	8652.67599	0.230802593	0.007747054	1615885.658	84.88275146	0.230802593	0.007747054
9	1	9876.38488	0.207993928	0.007907529	1861761.272	96.88733567	0.207993928	0.007907529
10	1	8760.733237	0.379868504	0.012063756	1765851.27	85.94279306	0.379868504	0.012063756

Appendix table 4: Fracture resistance of 1.0mm composite (LC) discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (N)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (kN/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	59.70545741	0.430709556	0.008012929	352.83379	59.70545741	0.430709556	0.008012929
2	1	52.75304083	0.46831821	0.007117798	396.7319579	52.75304083	0.46831821	0.007117798
3	1	63.94719476	0.468070891	0.008644656	439.2880578	63.9166717	0.472186905	0.008907815
4	1	60.67150879	0.441239099	0.007985903	356.3595679	60.67150879	0.441239099	0.007985903
5	1	59.00524442	0.471523993	0.008227892	319.1308994	59.00524442	0.471523993	0.008227892
6	1	47.8265085	0.576926507	0.007263013	344.0615419	47.8265085	0.576926507	0.007263013
7	1	44.11333839	0.524656442	0.005909195	280.2272246	44.11333839	0.524656442	0.005909195
8	1	48.25358156	0.511497983	0.006467559	291.0482718	48.25358156	0.511497983	0.006467559
9	1	69.55023829	0.552582932	0.011768886	284.8420505	69.55023829	0.552582932	0.011768886
10	1	54.54206593	0.44087414	0.006786648	405.1872294	54.54206593	0.44087414	0.006786648

Appendix table 5:Fracture resistance of 1.0mm composite (Sin) discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (N)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (kN/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	100.7133239	0.527584173	0.015859184	484.8524576	100.7133239	0.527584173	0.015859184
2	1	79.14252285	0.430783774	0.010532076	456.4875332	79.14252285	0.430783774	0.010532076
3	1	86.5053434	0.541632886	0.013499599	400.1751296	86.5053434	0.541632886	0.013499599
4	1	85.86630449	0.419531488	0.013233061	368.9954149	85.86630449	0.419531488	0.013233061
5	1	69.32794187	0.364633779	0.008485173	406.6283004	69.32794187	0.364633779	0.008485173
6	1	80.72008399	0.360179606	0.010267893	498.4348776	80.72008399	0.360179606	0.010267893
7	1	99.34987817	0.391740349	0.015878753	412.4692216	99.34987817	0.391740349	0.015878753
8	1	110.522583	0.362792394	0.018265886	488.2931319	110.522583	0.362792394	0.018265886
9	1	92.6080684	0.32716523	0.013862149	425.9914092	92.6080684	0.32716523	0.013862149
10	1	98.78536514	0.449912133	0.014349476	475.9004182	98.78536514	0.449912133	0.014349476

Appendix table 6: Fracture resistance of 0.5mm zirconia discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (N)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (kN/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	174.9962053	0.556076394	0.018518523	1113.799215	174.9962053	0.556076394	0.018518523
2	1	128.8346595	0.376787441	0.009792953	1494.928129	128.8346595	0.376787441	0.009792953
3	1	115.9840698	0.616078159	0.01058239	1131.488515	115.9840698	0.616078159	0.01058239
4	1	112.240502	0.684233845	0.008852866	1177.320426	112.240502	0.684233845	0.008852866
5	1	144.8329251	0.564682572	0.012568413	1367.932177	144.8329251	0.564682572	0.012568413
6	1	125.0432005	0.502366211	0.010536067	1143.131924	125.0432005	0.502366211	0.010536067
7	1	93.16711324	0.512609314	0.00562961	1085.78257	93.16711324	0.512609314	0.00562961
8	1	161.6988331	0.761194866	0.016915611	1152.893058	161.6988331	0.761194866	0.016915611
9	1	109.4097311	0.561879817	0.008431381	1098.363936	109.4097311	0.561879817	0.008431381
10	1	118.1850586	0.650389206	0.009208029	1433.666919	118.1850586	0.650389206	0.009208029

Appendix table 7: Fracture resistance of sand blasted and primed 0.5mm zirconia discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (gf)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	192.363021	0.557391455	0.021909966	1381404.816	192.363021	0.557391455	0.021909966
2	1	213.8759436	0.406836176	0.023805279	1146796.059	213.8759436	0.406836176	0.023805279
3	1	137.9113061	0.382329285	0.012879695	1308966.328	137.9113061	0.382329285	0.012879695
4	1	166.6074219	0.37035523	0.017022098	1098032.4	166.6074219	0.37035523	0.017022098
5	1	225.4606934	0.402112352	0.026352509	1249901.715	225.4606934	0.402112352	0.026352509
6	1	225.3651123	0.414616096	0.026684067	1178987.822	225.3651123	0.414616096	0.026684067
7	1	159.4968475	0.370052481	0.01590661	1117891.114	159.4968475	0.370052481	0.01590661
8	1	183.679291	0.347203919	0.020879142	986131.6147	183.679291	0.347203919	0.020879142
9	1	138.8554688	0.32170439	0.01225586	1047448.979	138.8554688	0.32170439	0.01225586
10	1	178.7087865	0.383851766	0.018265659	1516833.695	178.7087865	0.383851766	0.018265659

Appendix table 8: Fracture resistance of 1.0mm YZ/VM9 laminate discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (gf)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	43225.60376	0.747303569	0.066297098	1724188.231	424.0431729	0.747303569	0.066297098
2	1	45142.14861	0.508122178	0.072004004	2187137.146	442.8444778	0.508122178	0.072004004
3	1	41575.48888	1.035795561	0.063253991	1711013.569	407.855546	1.035795561	0.063253991
4	1	38800.30764	0.633307854	0.054273312	1609972.304	380.6310179	0.633307854	0.054273312
5	1	37415.70022	0.467389722	0.050642197	1788361.101	367.0480192	0.467389722	0.050642197
6	1	39163.36454	0.471581875	0.05421982	1682730.997	384.1926061	0.471581875	0.05421982
7	1	30338.0066	0.467220069	0.034823296	1677437.763	297.6158447	0.467220069	0.034823296
8	1	35131.09217	0.469968516	0.050084197	1697240.71	344.6360142	0.469968516	0.050084197
9	1	40321.18094	0.698930121	0.063234739	2286986.835	395.550785	0.698930121	0.063234739
10	1	33937.21198	0.624534295	0.044939774	1694614.579	332.9240495	0.624534295	0.044939774

Appendix table 9: Fracture resistance of 1.0mm YZ/LC laminate discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (N)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	238.0926514	1.007585278	0.038128184	1307716.186	238.0926514	1.007585278	0.038128184
2	1	210.9507756	0.948338322	0.031996673	1152185.956	210.9507756	0.948338322	0.031996673
3	1	229.5322861	0.967652415	0.035073785	1094913.602	229.5322861	0.967652415	0.035073785
4	1	240.3625993	0.982294439	0.037663084	1398091.604	240.3625993	0.982294439	0.037663084
5	1	299.0870845	1.065839607	0.055941102	1175919.132	299.0870845	1.065839607	0.055941102
6	1	249.0617108	0.999456359	0.040103314	1183692.127	249.0617108	0.999456359	0.040103314
7	1	257.0673008	0.982068038	0.041034869	1174499.327	257.0673008	0.982068038	0.041034869
8	1	200.2590637	0.98007323	0.03088501	976091.7779	200.2590637	0.98007323	0.03088501
9	1	251.2755713	1.094685393	0.040594795	1155134.62	251.2755713	1.094685393	0.040594795
10	1	253.3631838	1.020064264	0.041160574	1401513.522	253.3631838	1.020064264	0.041160574

Appendix table 10: Fracture resistance of 1.0mm YZ(roughened)/LC laminate discs (n=10) and other parameters.

Ref	*Speed (mm/min)*	*Maximum Load (N)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	161.4912495	0.964767286	0.022823961	1072845.778	134.3687358	1.008637907	0.028149829
2	1	234.285493	1.026905271	0.04065251	1030667.208	234.285493	1.026905271	0.04065251
3	1	243.8303295	1.065091745	0.038741853	1226280.226	243.8303295	1.065091745	0.038741853
4	1	272.7722509	1.02419458	0.046578193	1347719.044	272.7722509	1.02419458	0.046578193
5	1	232.768519	0.997599711	0.037382327	1210284.121	232.768519	0.997599711	0.037382327
6	1	190.3423855	0.921593846	0.029552132	1170195.028	190.3423855	0.921593846	0.029552132
7	1	193.8642659	1.029800972	0.028350957	1129210.399	193.8642659	1.029800972	0.028350957
8	1	169.5363846	0.945943882	0.023712837	1254375.159	169.5363846	0.945943882	0.023712837
9	1	152.4530087	0.895875158	0.021106986	1030065.22	152.4530087	0.895875158	0.021106986
10	1	243.6806033	0.956682117	0.040735194	1051799.694	243.6806033	0.956682117	0.040735194

Appendix table 11: Fracture resistance of 1.0mm YZ(roughened)/LC laminate discs (n=10) and other parameters, (no primer).

Ref	*Speed (mm/min)*	*Maximum Load (N)*	*Deflection at Maximum Load (mm)*	*Work to Maximum Load (J)*	*Stiffness (N/m)*	*Load at Break (N)*	*Deflection at Break (mm)*	*Work to Break (Nm)*
1	1	175.8651017	0.57978819	0.023286271	1062088.862	175.8651017	0.57978819	0.023286271
2	1	158.3460574	0.708236498	0.018738699	1407699.952	158.3460574	0.708236498	0.018738699
3	1	148.8162112	0.453425984	0.028070711	901639.1348	148.8162112	0.453425984	0.028070711
4	1	175.6783481	0.413674767	0.03700663	1045264.913	175.6783481	0.413674767	0.03700663
5	1	210.2002937	0.283203571	0.026330979	1397793.161	210.2002937	0.283203571	0.026330979
6	1	143.8100527	0.431996947	0.014501277	2540783.89	114.1423586	0.499193999	0.020950522
7	1	147.9193045	0.413209339	0.017808661	1006569.888	131.7354757	0.49270054	0.026443032
8	1	209.3425527	0.4190453	0.027889773	1218615.019	209.3425527	0.4190453	0.027889773
9	1	148.2961638	0.420118184	0.019700992	976402.2888	148.2961638	0.420118184	0.019700992
10	1	141.5306879	0.512164705	0.01940403	996333.827	141.5306879	0.512164705	0.01940403

One-way ANOVA: MK2_1, LC_1, Sin_1 and YZ_1 disc groups Fracture Resistance (N).

```
Source  DF       SS      MS       F      P
Factor   3  1332919  444306  219.35  0.000
Error   36    72920    2026
Total   39  1405839

S = 45.01   R-Sq = 94.81%   R-Sq(adj) = 94.38%


                          Individual 95% CIs For Mean Based on
                          Pooled StDev
Level   N    Mean  StDev  --------+---------+---------+---------+-
MK2_1  10   85.41   5.19    (-*-)
LC_1   10   56.03   7.97  (-*-)
Sin_1  10   90.35  12.32    (-*-)
YZ_1   10  497.75  88.66                               (-*-)
                          --------+---------+---------+---------+-
                                150       300       450       600

Pooled StDev = 45.01
```

One-way ANOVA: MK2_1, LC_1, Sin_1, YZ/VM9_1, YZ/LC_1, YZ/Sin_1 and YZ 0.5-1 disc groups Fracture Resistance (N).

```
Source  DF      SS      MS       F      P
Factor   6  796512  132752  146.04  0.000
Error   63   57269     909
Total   69  853781

S = 30.15   R-Sq = 93.29%   R-Sq(adj) = 92.65%


                           Individual 95% CIs For Mean Based on
                           Pooled StDev
Level      N    Mean  StDev  ------+---------+---------+---------+---
MK2_1     10   85.41   5.19     (-*)
LC_1      10   56.03   7.97  (-*-)
Sin_1     10   90.35  12.32     (-*-)
YZ/VM9_1  10  377.60  43.69                                  (-*-)
YZ/LC_1   10  242.77  27.13                    (-*-)
YZ/Sin_1  10  225.51  53.33                   (-*)
YZ 0.5-1  10  128.44  25.13          (-*-)
                           ------+---------+---------+---------+---
                               100       200       300       400

Pooled StDev = 30.15
```

One-way ANOVA: YZ_1 and YZ-Rough-1 disc groups Fracture Resistance (N).

```
Source  DF      SS     MS     F      P
Factor   1   22803  22803  4.40  0.050
Error   18   93289   5183
Total   19  116093

S = 71.99   R-Sq = 19.64%   R-Sq(adj) = 15.18%

                                 Individual 95% CIs For Mean Based on
                                 Pooled StDev
Level        N    Mean  StDev  ----+---------+---------+---------+-----
YZ_1        10  497.75  88.66               (---------*---------)
YZ-Rough-1  10  430.22  50.05  (---------*---------)
                                 ----+---------+---------+---------+-----
                                   400       450       500       550
Pooled StDev = 71.99
```

One-way ANOVA: YZ 0.5-1 and YZ 0.5 primed_1 disc groups Fracture Resistance (N).

```
Source  DF     SS     MS      F      P
Factor   1  14469  14469  17.20  0.001
Error   18  15138    841
Total   19  29606

S = 29.00   R-Sq = 48.87%   R-Sq(adj) = 46.03%

                                       Individual 95% CIs For Mean Based on
                                       Pooled StDev
Level             N    Mean  StDev  ------+---------+---------+---------+---
YZ 0.5-1         10  128.44  25.13  (------*-------)
YZ 0.5 primed_1  10  182.23  32.41                    (-------*-------)
                                       ------+---------+---------+---------+---
                                           125       150       175       200
Pooled StDev = 29.00
```

One-way ANOVA: YZ/VM9_1, YZ/LC_1, YZ/Sin_1 and YZ 0.5-1 disc groups Fracture Resistance (N).

```
Source  DF      SS      MS      F      P
Factor   3  315459  105153  68.72  0.000
Error   36   55089    1530
Total   39  370548

S = 39.12   R-Sq = 85.13%   R-Sq(adj) = 83.89%

                                Individual 95% CIs For Mean Based on
                                Pooled StDev
Level      N    Mean  StDev  -------+---------+---------+---------+--
YZ/VM9_1  10  377.60  43.69                                  (--*--)
YZ/LC_1   10  242.77  27.13               (--*--)
YZ/Sin_1  10  225.51  53.33             (--*--)
YZ 0.5-1  10  128.44  25.13  (--*--)
                                -------+---------+---------+---------+--
                                     160       240       320       400

Pooled StDev = 39.12
```

One-way ANOVA: YZ/VM9_1, YZ/LC_1 and YZ/Sin_1 disc groups Fracture Resistance (N).

```
Source  DF      SS     MS      F      P
Factor   2  138695  69347  37.90  0.000
Error   27   49405   1830
Total   29  188100

S = 42.78   R-Sq = 73.73%   R-Sq(adj) = 71.79%


                            Individual 95% CIs For Mean Based on
                            Pooled StDev
Level      N    Mean  StDev  -------+---------+---------+---------+--
YZ/VM9_1  10  377.60  43.69                           (----*----)
YZ/LC_1   10  242.77  27.13     (---*----)
YZ/Sin_1  10  225.51  53.33  (----*---)
                            -------+---------+---------+---------+--
                                 240       300       360       420
Pooled StDev = 42.78
```

One-way ANOVA: Sin_1 and YZ/Sin_1 disc groups Fracture Resistance (N).

```
Source  DF      SS     MS      F      P
Factor   1   91336  91336  60.97  0.000
Error   18   26965   1498
Total   19  118300

S = 38.70   R-Sq = 77.21%   R-Sq(adj) = 75.94%


                            Individual 95% CIs For Mean Based on
                            Pooled StDev
Level      N    Mean  StDev  -------+---------+---------+---------+--
Sin_1     10   90.35  12.32  (----*----)
YZ/Sin_1  10  225.51  53.33                             (----*----)
                            -------+---------+---------+---------+--
                                 100       150       200       250

Pooled StDev = 38.70
```

One-way ANOVA: LC_1 and YZ/LC_1 disc groups Fracture Resistance (N).

```
Source  DF      SS      MS       F      P
Factor   1  174352  174352  436.06  0.000
Error   18    7197     400
Total   19  181549

S = 20.00   R-Sq = 96.04%   R-Sq(adj) = 95.82%


                            Individual 95% CIs For Mean Based on
                            Pooled StDev
Level     N    Mean  StDev  ---+---------+---------+---------+------
LC_1     10   56.03   7.97  (-*--)
YZ/LC_1  10  242.77  27.13                               (-*--)
                            ---+---------+---------+---------+------
                              60       120       180       240

Pooled StDev = 20.00
```

One-way ANOVA: MK2_1 and YZ/VM9_1 disc groups Fracture Resistance (N).

```
Source  DF      SS      MS       F      P
Factor   1  426862  426862  440.97  0.000
Error   18   17424     968
Total   19  444286

S = 31.11   R-Sq = 96.08%   R-Sq(adj) = 95.86%


                            Individual 95% CIs For Mean Based on
                            Pooled StDev
Level      N    Mean  StDev  ----+---------+---------+---------+-----
MK2_1     10   85.41   5.19  (--*-)
YZ/VM9_1  10  377.60  43.69                                (-*-)
                            ----+---------+---------+---------+-----
                              100       200       300       400

Pooled StDev = 31.11
```

One-way ANOVA: YZ, MK2, LC and Sin disc groups Biaxial Flexural Strength in MPa.

```
Source  DF       SS       MS       F      P
Factor   3  5501005  1833668  241.39  0.000
Error   36   273469     7596
Total   39  5774474

S = 87.16   R-Sq = 95.26%   R-Sq(adj) = 94.87%


                          Individual 95% CIs For Mean Based on
                          Pooled StDev
Level   N    Mean  StDev  --------+---------+---------+---------+-
YZ     10  1011.2  173.1                               (-*-)
MK2    10   178.5   10.7    (-*-)
LC     10   113.7   12.2  (-*-)
Sin    10   178.8   12.7    (-*-)
                          --------+---------+---------+---------+-
                                300       600       900      1200

Pooled StDev = 87.2
```

One-way ANOVA: YZ and YZ-Rough disc groups Biaxial Flexural Strength in MPa.

```
Source  DF      SS     MS     F      P
Factor   1   54612  54612  2.82  0.110
Error   18  348679  19371
Total   19  403290

S = 139.2   R-Sq = 13.54%   R-Sq(adj) = 8.74%


                            Individual 95% CIs For Mean Based on
                            Pooled StDev
Level      N    Mean  StDev  --------+---------+---------+---------+-
YZ        10  1011.2  173.1              (----------*----------)
YZ-Rough  10   906.7   93.7  (----------*----------)
                            --------+---------+---------+---------+-
                                  880       960      1040      1120

Pooled StDev = 139.2
```

One-way ANOVA: YZ 0.5 and YZ 0.5 primed disc groups Biaxial Flexural Strength in MPa.

```
Source  DF      SS     MS     F      P
Factor   1   87490  87490  2.53  0.129
Error   18  621717  34540
Total   19  709207

S = 185.8   R-Sq = 12.34%   R-Sq(adj) = 7.47%

                                    Individual 95% CIs For Mean Based on
                                    Pooled StDev
Level           N    Mean  StDev  --+---------+---------+---------+-------
YZ 0.5         10  1005.6  186.9  (------------*-----------)
YZ 0.5 primed  10  1137.9  184.8               (------------*-----------)
                                    --+---------+---------+---------+-------
                                    900      1000      1100      1200

Pooled StDev = 185.8
```

One-way ANOVA: YZ, YZ-Rough, YZ 0.5 and YZ 0.5 primed disc groups Biaxial Flexural Strength in MPa.

```
Source  DF       SS     MS     F      P
Factor   3   269306  89769  3.33  0.030
Error   36   970396  26955
Total   39  1239702

S = 164.2   R-Sq = 21.72%   R-Sq(adj) = 15.20%

                                    Individual 95% CIs For Mean Based on
                                    Pooled StDev
Level           N    Mean  StDev  ---+---------+---------+---------+------
YZ             10  1011.2  173.1          (--------*--------)
YZ-Rough       10   906.7   93.7  (--------*-------)
YZ 0.5         10  1005.6  186.9          (--------*--------)
YZ 0.5 primed  10  1137.9  184.8                    (--------*--------)
                                    ---+---------+---------+---------+------
                                     840         960       1080       1200Pooled
StDev = 164.2
```

PANAVIA 21 Paste (Catalyst Paste and Universal Paste) Principal ingredients:

(1) Catalyst Paste

• 10-Methacryloyloxydecyl dihydrogen phosphate

• Hydrophobic aromatic dimethacrylate

• Hydrophobic aliphatic dimethacrylate

• Silanated silica filler

• Colloidal silica

• Catalysts

(2) Universal Paste

• Hydrophobic aromatic dimethacrylate

• Hydrophobic aliphatic dimethacrylate

• Hydrophilic aliphatic dimethacrylate

• Silanated titanium oxide

• Silanated barium glass filler

• Catalysts

• Accelerators

• Pigments

Source: Kuraray Europe GmbH