

**EFFECT OF FATIGUING AND THERMOCYCLING ON THE MECHANICAL AND  
OPTICAL PROPERTIES OF DIFFERENT GENERATIONS OF ZIRCONIA**

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## **ABSTRACT**

Islam Abd Alraheem: effect of fatiguing and thermocycling on the mechanical and optical properties of different generations of zirconia  
(Under the direction of Terry Donovan and Taiseer Sulaiman)

This study was conducted to test the bi-axial flexural load and optical properties of fully stabilized monolithic zirconia (FSZ) after fatigue loading and thermocycling. Partially stabilized monolithic zirconia (PSZ) was tested as a control.

BruxZir® Anterior Solid Zirconia ,which is fully stabilized (FSZ) and BruxZir® Shaded Zirconia, which is partially stabilized (PSZ) were used in this study.

Bi-axial flexural loads of PSZ was higher than FSZ at both thicknesses. FSZ has higher translucency than the PSZ. The 0.7 mm thickness groups were more translucent, had lower light blockage percentage and bi-axial flexural load than the 1.2 mm thickness groups. A significant number of the FSZ specimens did not survive the fatiguing protocol. All PSZ specimens survived the fatiguing. Fatiguing and thermocycling had no statistically significant effect on the bi-axial flexural loads of the PSZ. None of the changes in the optical properties after fatiguing is clinically significant.



To my wonderful parents, Mohammad Abd Alraheam and Firial Basyouniy and my amazing siblings who are always supportive of my decisions and career. Thank you for your unconditional love. Words are inadequate to express my gratitude for all of your hard work and sacrifices. I will always strive to be the daughter you deserve.

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## **LIST OF ABBREVIATIONS AND SYMPOLS**

°C	Degree of Celsius
CAD/CAM	Computer aided design/computer-assisted manufacturing
CIE	The International Commission on Illumination
CR	Contrast ratio
CTC	Coefficient of thermal contraction
FSZ	Fully stabilized zirconia
FDPs	Fixed dental prosthesis
GERD	Gastroesophageal reflux disease
HCl	Hydrochloric acid
HIP	Hot isostatic pressuring
hrs	Hours
K	Kalvin
LTD	Low temperature degradation
Mg-PSZ	Magnesium cation doped partially stabilized zirconia
mins	Minutes
MPa	Mega-pascal
n	Number of specimens
N	Newton
PSZ	Partially stabilized zirconia
SD	Standard deviation
SE	Standard error
secs	Seconds

t→m	Tetragonal to monoclinic transformation
TP	Translucency parameter
Y-TZP	Yttria-stabilized tetragonal zirconia polycrystals
ZTA	Zirconia toughened alumina

## **CHAPTER 1: REVIEW OF THE LITERATURE**

### **1. Introduction**

Full cast gold and porcelain fused to metal restorations have been the gold standard in restorative dentistry for many years. After introducing newer and stronger ceramic systems, all porcelain restorations became more popular especially in the anterior zone. Manufacturers are aggressively seeking to develop and market stronger and more esthetically pleasing materials. Unfortunately, no randomized clinical trials have been conducted prior to introducing these new materials to the market.

Two of the most popular and recent ceramics introduced to the market are lithium disilicate (IPS e.MAX, Ivoclar) and zirconia (multiple manufacturers). Both of those materials can be used in a layered or monolithic restorations. Layered versions provide better control of material esthetics. Lithium disilicate, an etchable glass-ceramic material is naturally more translucent and has less flexural strength than zirconia material because of its amorphous glass phase.<sup>[1-3]</sup> The absence of the glass phase in zirconia materials reduces the translucency (increases the opacity) of monolithic zirconia restorations, which is an esthetic concern, but results in a greatly increased flexural strength.<sup>[4]</sup> Manufacturers are now reporting that recent zirconia material formulations have greater translucency and, therefore, better esthetics while still maintaining most of the strength. Improving the translucency of newer zirconia materials has been accomplished by increasing the percentage (8-12%) of yttria which renders the crystal structure of the zirconium dioxide in a fully stabilized cubic form at room temperature.

Traditional zirconia has a lower percentage (2-4%) yttria which results in a crystal structure of the zirconia dioxide that is partially stabilized at room temperature.<sup>[5]</sup> Fully stabilized zirconia, with its improved translucency, is claimed by manufacturers to have a flexural strength higher than that for lithium disilicate glass-ceramic materials. Currently, dental laboratories are increasing the use of fully stabilized zirconia in the fabrication of fixed partial dentures and crowns. However, there is no clinical evidence-base upon which to make this decision. Since conducting randomized clinical trials is time consuming and expensive, *in vitro* testing using laboratory conditions that may be more clinically relevant is clearly indicated to help identify the potential behavior and performance of this material in the oral environment.

Multiple *in vitro* studies had been conducted to test the flexural strength and optical properties of the translucent zirconia comparing it with the conventional zirconia. Hydrothermal aging was the most common aging regime had been used <sup>[3, 6-8]</sup> Aging for clinically relevant thickness specimens using chewing simulator would represent more clinically relevant aging regime. A clinically relevant specimen's thickness is recommended of the bi-axial flexural strength test.

This research study was conducted to test the bi-axial flexural load and the optical properties of fully stabilized monolithic zirconia (FSZ) after fatiguing, thermocycling and acidic challenge. Partially stabilized monolithic zirconia (PSZ) was tested as a control.

## **2. Literature review**

### **2.1. History of dental ceramics**

Dental porcelains were first introduced in Europe in 1774 by the Parisian dentist Nicholas Dubois de Chemant, who used porcelain formulations to fabricate complete dentures.<sup>[9]</sup> de

Chemant worked in England to improve the translucency of dental porcelain. He was successful in obtaining more translucent formulation by adding a feldspathic rich component. Later in 1808, individual denture teeth containing platinum pins were created and used to fabricate more esthetic and repairable dentures. The coefficient of thermal contraction (CTC) of platinum was more compatible with dental porcelain which helped in the prevention of crack formation in the denture teeth upon cooling.

In 1962, a major revolutionary advance in porcelain formulation was discovered and named Glass Ceramic. This material was fabricated by melting and quickly cooling feldspar rock with more than 11% Potassium. The uniqueness of the thermal expansion of the various phases resulted in increased formation of the amorphous glassy phase of the ceramic. (Kelly/Orlowski) Reheating the glass ceramic significantly increased the thermal expansion due to the formation of a new crystalline component called Leucite. Modifications to this formulation were done to create a dental ceramics with an expansion coefficient compatible to most dental alloys. Dental alloys generally have an expansion coefficient ranging from 12 to 14 ( $\times 10^{-6}/^{\circ}\text{C}$ ). Compatible ceramics have 17 to 25 mass % of leucite.<sup>[10]</sup>

Leucite crystals were added later to powdered ceramics as well as to the first pressed ceramics. Adding leucite increases the strength of the ceramics moderately without severely compromising the translucency due to its similar refractive index to that of feldspathic glass. Leucite reinforced feldspathic ceramic is one of the most esthetically pleasing dental ceramics, it has been extensively used and studied in dentistry.<sup>[9] [11]</sup>

Excessive shrinkage during fabrication of ceramic restorations created a major challenge for ceramist and glass ceramic engineers. It was discovered that a reduction in the porosity of the



ceramic was necessary to limit shrinkage. <sup>[9]</sup> From the mid of 1980s to the late 1990s, seven different techniques were developed to fabricate dental ceramic crowns<sup>[9]</sup>:

- 1) Lost wax technique in which ceramic powder combined with polymer binder was melted, pressed into the mould space (created during the wax burnout), expanded and then crystallized during firing the firing cycle ( Cerestore; Johnson & Johnson, New Brunswick, NJ, USA)<sup>[12, 13]</sup>.
- 2) Refiring a special glass after casting it into a lost wax mould to form crystals within the glass ( glass ceramic “DICOR”; Dentsply International, York, PA, USA) The name DICOR was derived from a collaborative effort between Dentsply International and Corning Glass Works (now known as Corning Incorporated).
- 3) Alumina oxide, magnesium aluminate spinel and zirconia/ alumina sintering followed by glass infiltration into the ceramic ( In- Ceram; Vita Zahnfabrik, Bad Sackingen, Germany)<sup>[14]</sup>.
- 4) Filled glass solid ingots that were melted and pressed ( leucite or lithium disilicate) into a lost wax mould (Empress; Ivoclar Vivadent, Schaan, Liechtenstein)<sup>[15]</sup>.
- 5) Milling full density blocks of ceramics to the final restoration size using computer aided fabrication (CEREC; Sirona, Bensheim, Germany)<sup>[16]</sup>.
- 6) Milling an oversized die followed by pressing of Alumina powder then sintering the ceramic to obtain the final restoration size (Procera; Nobel Biocare, Zurich, Switzerland)<sup>[17]</sup>.
- 7) Soft milling of lightly sintered blocks of zirconia and alumina followed by sintering to obtain the final size ( Cercon, Lava, Vita YZ, Ivoclar e. max zir CAD)<sup>[18]</sup>.

## **2.2. Concepts in ceramics science**

Ceramics can be considered a “composite” material, meaning made of a composition of two or more materials. Kelly classified dental ceramics according to their structure into three types: (1) predominantly glassy materials, (2) particle filled glasses, and (3) polycrystalline

ceramics.<sup>[2, 9, 19]</sup> Highly esthetic dental ceramics have an increased glass component (or phase) which limits their flexural strength. Reduction of the glass phase results in higher strength ceramic. Increasing the crystalline phase to fully crystalline, i.e. polycrystalline, is useful for the fabrication of the ceramic substructure of dental restorations.

### **2.2.1. Predominantly Glassy Ceramics**

Ceramics that are predominately glass are optimal for mimicking the optical properties of enamel and dentin. The glasses in dental ceramics are an amorphous mixture of mined minerals called feldspar and are based on silica (silicon oxide) and alumina (aluminum oxide)<sup>[19]</sup>. Adding cations such as sodium and potassium alter important properties of the glass, e.g. by decreasing the firing temperature or increasing thermal expansion/ contraction behavior<sup>[9]</sup>. These materials perform best when fused to higher strength metal or polycrystalline substructures.

### **2.2.2. Particle Filled Glass Ceramics**

Particle filled glass ceramics have improved mechanical properties and altered optical properties such as opalescence, color and opacity. The addition of 17-25 mass % leucite filler particles (leucite) to the base glass composition increases the thermal expansion/contraction coefficient of the ceramic to a level higher than that for dental alloy substructure to which it was being fused during fabrication. Increased contraction of the glass ceramic during cooling of the metal-ceramic restoration, as compared with the metal substructure, resulted in a net compression of the glass ceramic (generally referred to as “porcelain”). Such modification created porcelains that are thermally compatible during firing with dental alloys.<sup>[10, 20]</sup>

John Mclean in 1965 reported on the strengthening of a feldspathic glass via addition of aluminum oxide particles.<sup>[20]</sup> Adding higher percentage of leucite (40 to 55 mass%) or other fillers material such as aluminum oxide (55 mass %) which are uniformly dispersed throughout

the glass, a phenomenon called “ dispersion strengthening”, created moderate increase in ceramic strength<sup>[9, 20]</sup>. It was hoped that restorations fabricated from these materials would no longer require metal or polycrystalline substructures. Leucite was chosen as a filler material due to its high thermal/ contraction coefficient and its index of refraction being very close to that of feldspathic glasses; which is critical for maintaining translucency. In addition, leucite etches at much faster rate than the base glass, which results in a very rough surface area helpful in the establishment of an effective micromechanical bond.<sup>[9]</sup>

Commercially available ceramics utilizing leucite fillers include a group that are pressed into mould at high temperature (OPC, Pentron; Empress Esthetic, Ivoclar Vivadent and Finesse All-Ceramic, Dentsply Prosthetics) and a group provided as a powder for traditional porcelain build up (OPC Plus, Pentron; Fortress, Mirage Dental Systems).

#### **2.2.2.1 Glass ceramics (special subset of particle filled glasses):**

Special heat treatment of the glass restoration causes the growth of the filler particles inside the glass, a process termed “creaming”.<sup>[9]</sup> The crystal phase is derived chemically from the atoms of the amorphous glass phase. The first commercial glass ceramic available was DICOR in which the crystalline phase was comprised of 55 vol % crystalline mica.<sup>[21]</sup> A more recently introduced glass ceramic contains 70 vol % crystalline lithium disilicate (E. max Press and E.max CAD, Ivoclar-Vivadent).<sup>[9]</sup>

#### **2.2.3. Polycrystalline ceramics**

Fired (or sintered) polycrystalline ceramics have practically no amorphous glass phase such that the crystals are closely packed to each other, e.g. In- ceram alumina and zirconia materials. The close proximity of the crystals results in greater resistance to fracture propagation

and laboratory testing reveals high toughness and strength. However, the high toughness and strength of these ceramics made use of this material for dental prosthesis fabrication very challenging. Researchers recognized that polycrystalline ceramics are more readily milled in their pre-sintered state. However, the process of sintering resulted in significant and calculable shrinkage of the ceramic because of the makeup and precisely characterized ceramic powders.<sup>[9, 17, 18]</sup> Therefore computer aided manufacturing became necessary and useful in the fabrication of dental prosthesis from these hard materials. In general, specific software has been used to process 3-D data digitally captured from tooth structure or a wax model of the final restoration. The software has been used to either create an enlarged die, upon which ceramic is packed (Procera, NobelBiocare, Zurich, Switzerland), or create an oversized restorations from a partially fired ceramic powder blocks ( Cercon, Dentsply Prosthetics; Lava, 3M-ESPE; Y-Z, Vita Zahnfabrik).

Polycrystalline ceramics are very strong but they are much more opaque because of the absence of the amorphous glass phase. These materials serve as substructure copings upon which glass ceramics are veneered which may result in improved esthetics of the dental restoration. Dental ceramics fabricated from zirconium oxide (Zirconia) hold a unique place amongst polycrystalline oxide ceramics due to excellent mechanical properties.

### **2.3. Transformation toughened zirconium oxide**

A considerable amount of research, since the 1970s, has been accomplished on the “transformation toughening” properties of zirconium oxide materials. This unique material property of molecular transformation at the initiation of fracture propagation leading to increased material toughness earned zirconium oxide the name “Ceramic Steel”.<sup>[22, 23]</sup>

In 1988, zirconia ceramic became available for hip replacement surgeries. Early experimental studies showed encouraging results and high biocompatibility. However, in one French hospital, zirconia hips were abandoned due to the lower survival rate obtained after 8 years (63%), compared with the alumina femoral heads, which had 93% survival rate at 9 years.

<sup>[24]</sup> The introduction of zirconia based ceramics as restorative dental materials has generated considerable interest in the dental community. Laboratory studies have reported that zirconia materials have the highest mechanical properties of all dental ceramics. Unfortunately, due to the metastability of tetragonal zirconia, stress generating surface treatments such as grinding or sandblasting cause transformation of the zirconium oxide from a tetragonal to monoclinic molecular organization. This molecular transformation results in an associated increase in volume and formation of surface compressive stresses. The increased surface compression stress enhances flexural strength but alters the phase integrity of the material. The altered phase integrity increases susceptibility to aging.<sup>[22, 25]</sup> The drawbacks of this aging process are multiple and include 1) surface degradation with grain pullout, 2) microcracking and 3) low temperature strength degradation. Low temperature degradation (LTD) is a well-documented phenomenon, exacerbated notably by the presence of water <sup>[22, 26-31]</sup>. LTD has been shown to be indirectly associated with a series of femoral head prostheses failures in late 1990s and early 2000s.<sup>[24, 30]</sup>

### **2.3.1. Types of Zirconia ceramics available for dental applications**

The three main types of zirconia are used today in dentistry are as follows: 1) yttrium cation doped tetragonal zirconia polycrystals (3Y-YZP), 2) magnesium cation doped partially stabilized zirconia (Mg-PSZ) and 3) zirconia toughened alumina (ZTA).<sup>[22]</sup>

### 2.3.1.1. Yttrium cation doped tetragonal zirconia polycrystals (3Y-TZP)

3Y-TZP contains 3 mol% Yttria ( $\text{Y}_2\text{O}_3$ ) as a stabilizer.<sup>[32]</sup> While the stabilizer  $\text{Y}^{3+}$  cations and  $\text{Zr}^{4+}$  are randomly distributed over the cationic sites, electrical neutrality is achieved by the creation of oxygen vacancies.<sup>[22, 33, 34]</sup>

The particle size of 3Y-TZP strongly affects its mechanical properties.<sup>[35-37]</sup> Particle size less than one micrometer ( $<1\mu\text{m}$ ) results in 3Y-TZP becoming less susceptible to spontaneous tetragonal to monoclinic ( $t \rightarrow m$ ) transformation.<sup>[38]</sup> Moreover, below a certain particle size ( $\sim 0.2\mu\text{m}$ ), the transformation is not possible, leading to reduced fracture toughness.<sup>[39]</sup> On the other hand, a particle size above  $1\mu\text{m}$  causes 3Y-TZP to be less stable and more susceptible to spontaneous  $t \rightarrow m$  transformation, leading to significant volume increase, resulting in a reduction in fracture toughness. Therefore, it appears that  $1\mu\text{m}$  is the critical particle size for 3Y-TZP.

According to that, the sintering conditions that result in particle size have a strong impact on both stability and mechanical properties of the final ceramic product.<sup>[40]</sup> Higher sintering temperatures and longer sintering times lead to larger particle sizes.<sup>[22, 37, 41, 42]</sup> The final sintering temperatures of 3Y-TZP vary between  $1350$  and  $1550^\circ\text{C}$  depending on the manufacturer. In addition, variation of sintering temperatures during firing may possibly have an influence on the particle size and phase stability of 3Y-TZP used dental applications. (Figure 1.1) shows the molecular structure of Y-TZP.

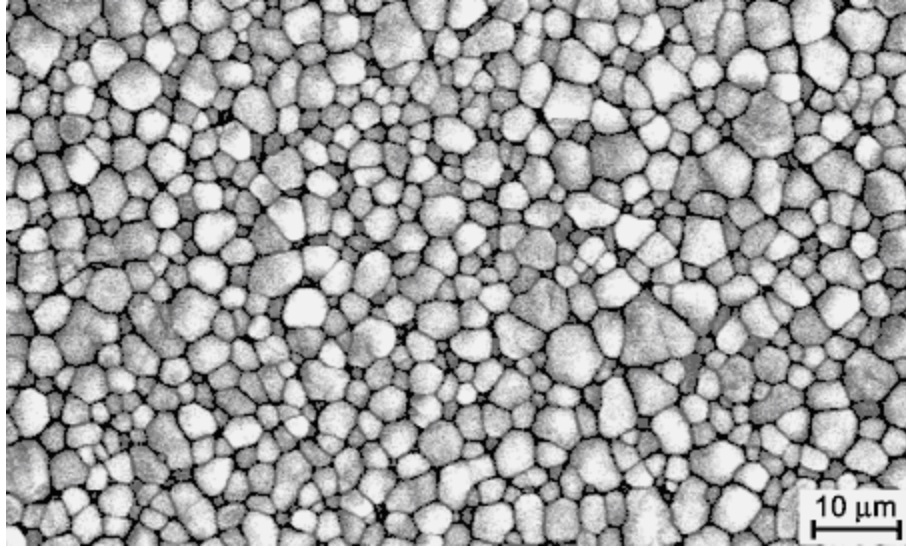


Figure. 1.1: Microstructure of polycrystalline tetragonal zirconium dioxide (TZP)

#### 2.3.1.2. Glass infiltrated zirconia toughened alumina (ZTA)

Zirconia toughened alumina (ZTA), which is alumina matrix combined with zirconia, is another approach to harness the benefit of stress induced zirconia transformation..<sup>[43, 44]</sup> One commercially available dental product, In-Ceram Zirconia (Vident™, Brea, CA), was developed by adding 33 vol.% of 12 mol% ceria-stabilized zirconia (12Ce-TZP) to In-Ceram Alumina.<sup>[22, 45]</sup>

In-Ceram Zirconia restorations may be fabricated by either slip casting or soft machining. Initial sintering has to be done at 1100°C for 2 hours. Prior to that, a porous ceramic mixture has to be infiltrated with amorphous glass so that the glass phase represents approximately 23 vol% of the final product. It was found that less shrinkage resulted from slip cast technique, but the amount of porosity was between 8 and 11% more than that of sintered 3Y-TZP.<sup>[22, 46,</sup>

<sup>47]</sup> Increased porosity likely explains the lower mechanical properties of In-Ceram zirconia when compared to 3Y-TZP dental ceramics<sup>[47]</sup>.

### **2.3.1.3. Partially stabilized zirconia (Mg-PSZ)**

Magnesia stabilized zirconia (Mg-PSZ) has not been successful in biomedical applications due mainly to the presence of surface porosity associated with a large particle size (30-60 $\mu$ m) that can result in excessive wear.<sup>[22, 32, 35]</sup> The MgO amount in the composition of commercial materials in the range of 8-10 mol%.<sup>[35]</sup> High sintering temperature (1680 and 1800°C) and strictly controlled cooling cycles are needed to control the fracture toughness of Mg-PSZ.<sup>[48-50]</sup> The molecular composition of this material is less stable, which can result in early t $\rightarrow$ m transformation such that there is inadequate tetragonal form of the zirconia to transform and toughen the material upon subsequent fracture formation. The higher presence of the monoclinic form results in lower mechanical properties.<sup>[22, 51]</sup> Denzir-M (Dentronic AB) is an example of Mg-PSZ ceramic currently available for hard machining of dental restorations.<sup>[52]</sup>

## **2.3.2. Different generations of zirconia in dentistry**

### **2.3.2.1. Conventional zirconia/ First generation**

Fifteen years ago, tetragonal partially stabilized zirconia was developed. Also known as conventional zirconia, this material may be characterized as having a high light refractive index. The extremely high number of interfaces created by numerous very small crystal structures through which the light has to pass causes the material to be optically opaque. Blanks of this generation can be industrially dyed or non-dyed. The dyed blanks are useful in the production of natural tooth shades. The non-dyed blanks have a white monochromatic color, which can be non-esthetic in many clinical situations. To overcome this disadvantage, milled porous restorations may be immersed in a coloring oxides before sintering, or, brushes can be used to apply a color



gradient to the restorations. Non-porous sintered zirconia copings or frameworks may be surface stained prior to veneering with feldspathic porcelain.<sup>[53]</sup>

One of the drawbacks of the veneered zirconia is chipping (delaminating) of the veneering ceramics. Difference in the coefficients of thermal expansion of the framework material and veneering ceramic may result in inner tensile stress within the restoration upon cooling. This inner tensile stress along with the external compressive stresses from the masticatory forces may exceed the deformation capability of the material and result in delamination and fracture. Since the veneering ceramic is weaker than the zirconia framework, it is more prone to fracture. This assumption is supported by clinical and laboratory studies, where a fracture is never observed in the interface between framework and veneer, but always within the veneering ceramic “cohesive fracture within the veneering ceramic”.<sup>[53, 54]</sup>

According to Heintze, the low thermal conductivity of the zirconia framework could prevent the complete firing of the veneering ceramic, it could also result in an unfavorable temperature distributions leading to the development of internal stresses, which might result in chipping of the veneering ceramic secondary to cyclic loading.<sup>[54]</sup> The veneering ceramic should always be supported by the zirconia framework to minimize the chipping rate. Ferrari M et al reported -in a recently published 3 year clinical trial- higher chipping of veneering ceramic in patient's group who had the flat design occlusal preparation, in which the veneering porcelain is not completely supported by the zirconia core. On the other hand, 0% chipping was observed in the other patient's groups where anatomically guided frameworks were prepared to give complete support for the veneering porcelain.<sup>[55]</sup>

To overcome the problem of chipping of the veneering ceramics, monolithic zirconia restorations were introduced into dentistry. Monolithic zirconia is considered as a good

alternative to the conventional ceramic veneered (i.e. “layered”) zirconia restorations. Since no space is needed for the veneering ceramics, less tooth reduction is required for monolithic zirconia restorations. Furthermore, manufacturing cost may be reduced by automatic designing and milling of full anatomical contour restorations by CAD/CAM technology. However, a major disadvantage of monolithic zirconia is the opacity of the final restorations.

Different approaches have been used to enhance the translucency of the monolithic zirconia restorations. One approach manufacturers have used is via the fabrication of zirconia blanks with multiple, differently-shaded layers. Dental technicians can use the computer software to position the restorations in the right color zone in the blank before milling it. Other approach has been the modification of the sintering temperature of the first generation zirconia (3Y-TZP).<sup>[53]</sup> It was found that an improvement in the translucency may be accomplished through variation of the sintering temperature, the dwell time and the timing and rate of temperature increases and decreases. Higher sintering temperature results in larger particle size, which improves the translucency but compromise the strength of the material. Due to the negative effect on the strength, the first generation of monolithic zirconia failed to become established. Generally, the optimum sintering temperature is less than 1600°C for all zirconia-based materials used in dentistry.<sup>[53, 56]</sup>

#### **2.3.2.2 Second generation**

The second generation of zirconia was introduced in 2012 to 2013. The number and size of alumina oxide ( $\text{Al}_2\text{O}_3$ ) particles were reduced and re-positioned in the zirconia framework. The particles of the ( $\text{Al}_2\text{O}_3$ ) takes place on the boundaries of zirconia particles. Which allows more light transmittance, renders the material more translucent with good long term stability and strength. These findings have been confirmed by laboratory studies that have shown this

generation is not only more translucent but also has high strength at baseline and after fatiguing than the first generation of zirconia based dental materials. <sup>[57]</sup>

### **2.3.2.3. Third generation**

The third generation of zirconia was introduced in 2015. The goal behind introducing this generation is to provide a zirconia restorations with levels of translucency similar to those fabricated from glass ceramic materials. This zirconia is meta-stabilized in tetragonal phase and contains up to 53% cubic phase and, as such, is described as fully stabilized zirconia with a mixed cubic/tetragonal structure. The full level of stabilization is achieved by adding more yttrium oxide (~9.3wt%/ 5mol %) resulting in (5Y-TZP).<sup>[53]</sup> The larger the particle size of the cubic phase renders the material more porous, which results in less light scattering at the particle boundaries and therefore more translucency of the material. The isotropic nature of the cubic crystals results in evenly emitted light in all spatial directions. Which improves the translucency.<sup>[53]</sup> However, movement from partially to fully stabilized zirconia lowers the fracture toughness and may subject this zirconia formulation to increased low temperature degradation secondary to the reduced ( $\text{Al}_2\text{O}_3$ ) particles. <sup>[58]</sup>

Nordahl et al. found that there are no differences in fracture load between second and third generation zirconia.<sup>[59]</sup> Third generation zirconia reported higher translucency than other zirconia generation in one research study, but it wasn't as translucent as the lithium disilicate glass-ceramic IPS e.max. <sup>[60]</sup>

Light permeability of multilayer color zirconia block was evaluated by Udea et al, they showed that four layered, pre-colored, zirconia blanks offer advantages in terms of esthetics compared to monochromatic materials. Precise positioning of individual restorations in the blank

is essential for replicating the natural shade of the tooth being restored.<sup>[61]</sup> Studies testing third generation zirconia are scarce and more research is needed in this area.<sup>[53]</sup>

### **2.3.3. General properties of zirconia**

#### **2.3.3.1 Transformation toughening**

Zirconium oxide has been known for its unique phenomenon of transformation toughening. In general, three crystallographic forms of unalloyed zirconia have been identified depending on the temperature. At room temperature and upon heating up to 1170°C, the symmetry is monoclinic. The molecular form is tetragonal between 1170 and 2370°C and becomes cubic above 2370°C and up to the melting point.<sup>[22, 40, 62]</sup>

Tetragonal to monoclinic transformation upon cooling is accompanied by a substantial increase in volume (around 4.5%), which is sufficient to lead to catastrophic failure. Alloying pure zirconia with stabilizing oxides such as CaO, MgO, Y<sub>2</sub>O<sub>3</sub> or CeO<sub>2</sub> controls (or limits) the stress induced t→m transformation and allows the stabilization zirconia in the tetragonal form at room temperature. Initial surface flaws (cracks) in the material result in t→m transformation which yields effective arresting of crack propagation and, therefore, high fracture toughness.<sup>[22, 23, 48, 63]</sup> As a crack propagates towards the core of the material, tensile stresses develop around the tip of the crack, transforming the tetragonal particles to monoclinic. Monoclinic particles are larger in size, which results in the development of an internal compressive stress that opposes the crack at its tip and limits further crack propagation (Figure 1.2). The net effect in a localized increase in fracture toughness. The transformation toughening phenomena increases the zirconia's flexural and tensile fracture resistance. Excessive grinding, temperature changes and stresses may exceed the compressive strengths around the crack tip, and the crack will propagate

towards the core of the material leading to complete failure of the zirconia. Therefore, the transformation toughening phenomena may be beneficial to the strength of zirconia at first, but as the fatiguing forces and stresses in moist environments increase, the zirconia strength may theoretically become questionable. A decrease of mechanical properties after various aging regimes was not observed in the Stawarczyk et al research study, where they tested conventional and second generation zirconia materials.<sup>[57]</sup> Yet the definitive answer awaits quantitative clinical assessment.

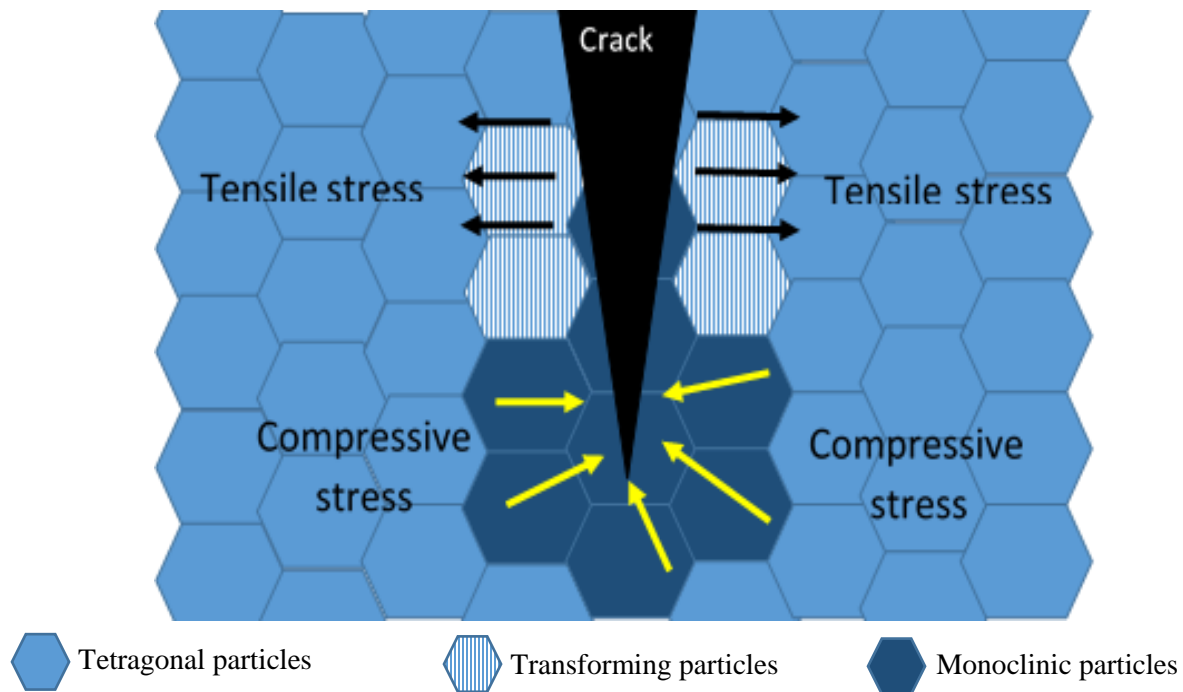


Figure 1.2: Schematic illustration of the transformation toughening phenomenon.

### 2.3.3.2. Physical properties

Due to the transformation toughening phenomena, zirconia ceramics have the highest strength and toughness among all the current dental ceramics. The properties of most material

used in dentistry, as well as enamel and dentin, are summarized in (Table 1.1). Fully stabilized zirconia ceramics (third generation) had lower flexural strength compared to the conventional partially stabilized zirconia (first generation). According to Stawarczyk et al 2016, the flexural strength of first generation zirconia is around 917 MPa. Third generation zirconia ceramics have been found to have a flexural strength between 618 and 718 MPa in the same research study.<sup>[57]</sup>

Conventional zirconia has been reported to have a high fracture toughness ranging from 6.3 to 11.5 MPa<sup>[64, 65]</sup> Zirconia was reported as a chemically stable material, which leads investigators to consider it as a biocompatible dental material.<sup>[32]</sup> Sulaiman et al 2015 tested the acidic resistance of partially stabilized zirconia and found that acid had no significant effect on this generation of zirconia.

**Table 1.1: Properties of zirconia in comparison with enamel, dentin, and other materials used in fixed prosthetic dentistry.**

<b>Material</b>	<b>Flexural Strength (MPa)</b>	<b>Elastic modulus (GPa)</b>	<b>Hardness (VHN)</b>	<b>Reference</b>
<b>Enamel</b>	0.7-1.3 (fracture toughness, MPa m <sup>1/2</sup> )	92	272	(Hassan et al., 1981; Xu et al., 1998; Wongkhantee et al., 2006) <sup>[66-68]</sup>
<b>Dentin</b>	1-2 (fracture toughness, MPa m <sup>1/2</sup> )	20	43	(Imbeni et al., 2004; Xu et al., 1998; Wongkhantee et al., 2006) <sup>[67-69]</sup>

<b>Gold alloy</b> Au-Pd	611 (Tensile strength)	103	220	(O'Brien, 2008) <sup>[70]</sup>
<b>Gold alloy</b> Type IV	759 (Tensile strength)	99	250	(O'Brien, 2008) <sup>[70]</sup>
<b>Feldspathic porcelain</b> (Vita VMK 68)	83	57	703	(Tinschert et al., 2000) <sup>[64]</sup>
<b>Leucite reinforced glass ceramic</b> (Empress 2)	283	96	550	(Aboushelib et al., 2007a) <sup>[65]</sup>
<b>Lithium disilicate</b> (IPS e.max)	360	95	5800	(Ivoclar, Vivadent)
<b>Alumina (Al<sub>2</sub>O<sub>3</sub>)</b>	500	380	2200	(Piconi and Maccauro, 1999) <sup>[32]</sup>
<b>Zirconia</b> (Y-TZP)	800-1500	210	1200	(Piconi and Maccauro, 1999) <sup>[32]</sup>

### 2.3.3.3. Low temperature degradation

The excitement of the physical aforementioned properties of zirconia comes with a potential threat to the long term performance of this material. Low temperature degradation (LTD) was first reported by Kobayashi et al. He reported that Y-TZP ceramics in a humid

environment may suffer from a slow t→m phase transformation, leading to microcracking and reduction in zirconia strength.<sup>[71]</sup>

Some investigators agreed to the following feature of LTD: (1) the tetragonal to monoclinic transformation proceeds from the external surface to the interior (2) water or a humid environment causes t→m phase transformation (3) stabilizing content and particle size has an important effect on the transformation process (4) degradation is time dependent and progresses more rapidly at temperatures between 65°C and 500°C, the maximum rate occurring at 250°C.<sup>[30, 58, 72-74]</sup>

Papanagiotou reported gradual dissolution of Yttria over time due to LTD, which could decrease the tetragonal phase stability and long term clinical performance of zirconia restorations.<sup>[72]</sup> The theoretical models currently proposed to explain LTD in a humid environment are based on (1) the formation of zirconium hydroxides at the surface from water chemisorption, resulting in the accumulation of strain energy and t→m transformation or (2) the depletion of the yttrium oxide stabilizer by formation of yttrium hydroxides from the reaction between water (H<sub>2</sub>O) and Yttria (Y<sub>2</sub>O<sub>3</sub>), leading to t→m transformation.<sup>[29-31, 72, 75, 76]</sup>

It was reported by Grant et al. that the hot isostatically pressed (HIP) 3Y-TZP has a lower susceptibility to LTD than the non-HIP 3Y-TZP material.<sup>[77]</sup> However, the machining of HIP 3Y-TZP for dental applications is likely to modify subsequent susceptibility to aging.

LTD has been shown to be indirectly associated with a series of femoral head prostheses failures in late 1990s and early 2000s.<sup>[24, 30]</sup> Further research is required to identify if LTD will impact the clinical performance of dental zirconia restorations.



*In vitro* studies have been designed to measure the effect of aging on the mechanical properties of different zirconia's generation. In one research study the mechanical properties of three different brands of second generation zirconia (monolithic zirconia) were compared to a conventional zirconia. The results showed that conventional zirconia exhibited major benefits in the mechanical properties compared to the monolithic materials. Another interesting finding in the same research study was none of the tested materials showed decrease of mechanical properties after various aging regimes.<sup>[57]</sup>

#### **2.3.4. Manufacturing zirconia restorations in dentistry**

There are two main methods to fabricate zirconia restoration in dentistry, soft machining of presintered blanks or hard machining of blanks already sintered and processed by hot isostatic pressing temperature under high pressure.

##### **2.3.4.1. Soft Machining**

Soft machining of presintered 3Y-TZP is becoming increasingly popular in dentistry and multiple manufacturers have made this material available. Restoration fabrication requires that 1) the die, the tooth or the wax pattern be scanned, 2) an enlarged restoration be designed by a specific software (CAD) and 3) machine milling of a presintered ceramic blank, 4) high temperature sintering and resultant controlled shrinkage (~25% for 3Y-TZP) to the desired restoration size. The enlarged design of the restoration will compensate for the shrinkage the material will undergo during sintering.<sup>[22]</sup>

The powder of the 3Y-TZP contains a binder that makes it suitable for pressing. This binder will be eliminated during pre-sintering heat treatment. This process has to be controlled carefully by manufacturers, especially the heating rate and the pre-sintering temperature. Too fast heating rate will eliminate the binder and produce burn out products that can lead to cracking of the blanks. Therefore, a slow heating rate is preferred. Hardness and the machinability are affected by the pre-sintering temperature. An adequate hardness is needed for the handling of the blanks but, if too high, might limit machinability.

Careful measurement of the density of the blanks is critical so that the appropriate compensating shrinkage is applied during final sintering.<sup>[22, 78]</sup> The final density of the presintered blanks is about 40% of the theoretical density ( $6.08\text{g/cm}^3$ ). Sintering conditions are product specific. Currently available systems utilizing soft machining of 3Y-TZP for dental restorations are Cercon (Dentsply International), Lava<sup>TM</sup> (3M<sup>TM</sup> ESPE<sup>TM</sup>), Procera zirconia (Nobel Biocare<sup>TM</sup>), YZ cubes for Cerec Inlab (Vident<sup>TM</sup>) and IPS e.max ZirCAD (Ivoclar Vivadent).

Disadvantage of soft milling of zirconia include the need for high-cost, specialized sintering ovens for the subsequent sintering and the somewhat lower flexural strengths.<sup>[53, 56]</sup> However, the strength of these materials remain multiple times greater than the minimum normal of 100MPa. Indeed, depending on the zirconia level of stabilization, the flexural strength may even reach values greater than 1,000 MPa.<sup>[53]</sup> Sintering the restorations after production through soft milling prevents the stress induced transformation from tetragonal to monoclinic phase unless grinding adjustments are needed or sandblasting is performed.

#### 2.3.4.2. Hard Machining

Fabrication of dental restorations by hard machining of 3Y-TZP and Mg-PSZ is possible using at least two systems, Denzir (Cadesthetics AB) and DC-Zirkon (DCS Dental AG). Pre-sintering at temperatures below 1500°C to reach a density of at least 95% of the theoretical density is used to prepare Y-TZP blocks. The blocks are then processed by hot isostatic pressing (HIP) at temperatures between 1400 and 1500°C under high pressure in an inert gas atmosphere. Very high density exceeding 99% of the theoretical density is reached after HIP treatment. Specially designed milling system are then used to mill the blocks. Milling these high strength blocks requires heavier milling machines and may limit the durability of the milling burs.

Furthermore, more surface defects are introduced into the milled restoration which may result in clinically significant t→m transformation. [22, 79, 80] Low temperature degradation is more critical when the material has surface defects which induce residual stresses.

Grinding or sandblasting of 3Y-TZP restorations may lead to t→m transformation and the formation of surface flaws that could be detrimental to the long term performance and is not recommended by most manufacturers. On the contrary, milling fully sintered zirconia (hard machining) has been shown to result in formation of a significant amount of monoclinic phase zirconia. [22, 47]

It has been shown in many in-vitro studies that both sandblasting and sharp indentations, even at very low loads, have a detrimental effect on the long term performance of 3Y-TZP. [5] [81, 82] Multiple studies have reported that annealing at 900°C for 1h or relatively short heat treatments in the temperature range 900-1000°C for 1 min induce the reverse transformation from monoclinic to tetragonal forms which may enable a return to optimal fracture toughness. During

fabrication of layered dental restorations, firing the veneering porcelain is likely to promote the reverse transformation. However, it is debatable whether the reversibility of the transformation provides healing of the previously introduced flaws.<sup>[83, 84]</sup>

The mechanical properties of 3Y-TZP ceramics are higher than all the available dental ceramics. It's flexural strength in the range of 800-1000MPa and the fracture toughness is 6-8 MPa. M. <sup>0.5</sup> range.<sup>[83, 85]</sup>

### **2.3.5. Main applications of zirconia in dentistry**

#### **1) Crowns and multiunit fixed partial dentures**

Crowns and multiunit fixed partial dentures fabricated from zirconia are less costly than porcelain fused to metal restorations. In addition, zirconia materials may be comparable or even provide more optimal esthetic results. Therefore, there has been a major shift toward use of these materials in dentistry.

#### **2) Complete arch implant prostheses**

Review of multiple *in vitro* studies suggest that that monolithic zirconia prostheses may provide improved wear, high esthetic quality, and improved biofilm accumulation over implant prostheses.<sup>[86-88]</sup> However, evidence from clinical studies supporting these claims have not been published.<sup>[89]</sup>

#### **3) Implants**

Brånemark in 1952 accidentally discovered that Titanium could be completely integrated in bone in a way that meant the metal piece could not be removed from it. Brånemark called the discovery “osseointegration”. That discovery opened a new era in dentistry that changed the treatment planning options for patients who are seeking replacement of their missing teeth.<sup>[90]</sup> Titanium has been proven to be biocompatible and, when used as a dental implant, has

shown high long term survival rates in multiple studies.<sup>[90, 91] [92] [93]</sup> However, one of the drawbacks of titanium implants is the gray color of the implant. Exposure of implant threads can be esthetically problematic for many patients, especially those who have a high smile line and thin soft tissue biotype. To overcome the color limitation of titanium implants, zirconia implants have been marketed by multiple manufacturers. *In vitro* studies have shown that direct bone contact on the zirconia implants with modified surface, (acid etched yttrium-stabilized tetragonal polycrystals), was comparable to titanium implants.<sup>[94, 95]</sup> A recently published clinical research study reported three years results on 105 zirconia implants. The author reported a 100% implant survival rate and 95.4% success rate.<sup>[96]</sup> Long term survival statistics of zirconia implants are not yet available.

#### **4) Implant abutments**

Currently, the majority of implant manufacturers offer zirconia abutments for aesthetic implant-supported restorations. Prefabricated or customized abutment forms are available and can be prepared in the dental laboratory either by the technician or by utilizing CAD/ CAM techniques. Zirconia abutments are successors to the densely sintered high-purity alumina ( $\text{Al}_2\text{O}_3$ ) abutments. Zirconia abutments are superior to the alumina abutments in terms of radio-opacity and resistance to fracture.<sup>[97, 98]</sup> Zirconia abutments have also been modified with titanium inserts so as to provide an internal connection of metal between the abutment and the implant. Concerns over the ability of the various types of zirconia abutments to withstand occlusal loads have remained. Several studies reported a mean loading force of approximately 206 N and maximum biting forces of up to 290 N in the aesthetic zone.<sup>[99, 100]</sup>

In an *in vitro* research study, the fracture loads of titanium-reinforced zirconia and pure alumina abutments were compared. After fatigue for 1.2 million cycles and static loading, the

median fracture loads were 294 N, 239 N, and 324 N for the zirconia abutments with titanium inserts, alumina, and titanium abutment groups, respectively. The authors concluded that titanium-reinforced zirconia abutments perform in a similar manner to metal abutments, and can therefore be recommended as an aesthetic alternative for the restoration of single implants in the anterior region.<sup>[101]</sup> No studies looked at the minimum thickness recommended for zirconia abutments. However, it was assumed that the minimum thickness of zirconia abutments needs to be 0.5 mm or more to withstand a functional load.<sup>[22, 102]</sup>

### **2.3.6 Clinical performance of zirconia restorations**

The clinical success rate of layered zirconia restorations has not been well established. However, there is an agreement between the studies that chipping of the veneering ceramic is the main mechanical complication encountered in these restorations. A systematic review that compare the clinical performance of soft- and hard-milled zirconia frameworks reported that fracture of Y-TZP substructures mostly occurred in fixed partial dentures. However, this was found to be rare, and only reported in five studies on two zirconia brands: Cercon and IPS e.max ZirCAD, both brands are soft machining zirconia.<sup>[103]</sup> Heintze et al 2010 reported in his systematic review that the frequency of chipping was 54% for the zirconia supported FDPs and 34% for porcelain fused to metal FDPs.<sup>[54]</sup> In another research study, the cumulative survival rate of single posterior 3Y-TZP crowns was reported to be a promising 88.8%. In addition, chipping of the veneering ceramics was observed in 3% of the crowns and none of the zirconia cores fractured.<sup>[104]</sup>

In a prospective cohort research study done by Sailer et al, conventional zirconia frameworks were used to replace 1 to 3 posterior teeth. After 5 years, 50% of the fixed partial dentures had to be replaced because they were deemed clinically unacceptable. The survival rate

for the rest of the prostheses at 5 years was 73.9%. Secondary caries was found in 21.7% of the fixed partial dentures, and chipping of the veneering ceramic was reported in 15.2%.<sup>[105]</sup>

Nicolaisen et al reported 100% survival rate for both metal ceramic and all ceramic FDPs in an *in vitro* research study. However, he also reported success rate of 76% and 71% for metal ceramic and all ceramic FDPs, respectively. All the complications were associated with chipping of the veneering ceramic.<sup>[106]</sup> According to a recently published systematic review, the current evidence regarding complete-arch implant-supported monolithic zirconia fixed dental prostheses revealed reproducible high prosthesis survival in the short term, the mean follow up time of the prosthesis was more than 1 year.<sup>[89]</sup>

In prospective clinical studies, it was observed that zirconia abutments would not cause technical or biological problems, at least over short or intermediate observation periods (40-48 months).<sup>[102, 107, 108]</sup> These findings were confirmed by a randomized clinical trial with 36 months follow up.<sup>[109]</sup>

To the author's knowledge, no clinical data are currently available on the clinical performance of second and third generation (monolithic) zirconia restorations.

### **2.3.7. Optical properties of zirconia material**

Mimicking the appearance of the natural dentition requires knowledge of the optical behavior of teeth, restorative materials and the science of color perception.

### 2.3.7.1. The main definitions for appearance related to teeth and restorative materials:<sup>[110, 111]</sup>

#### 1) Color attributes

The Munsell color system is the oldest color order system and has been used in dentistry to define color in terms of value, hue and chroma.<sup>[112]</sup> The Munsell color solid can be described as a sphere or a cylinder. A colorless or achromatic axis extends through the center of the cylinder, pure white at the top, pure black at the bottom. A series of grays, progressing from black to white in equal visual steps, connects these extremities. Colors (Hues) are arranged around this axis, and within each Hue, the colors are arranged in scales according to their lightness/darkness (Value) and their purity or strength (Chroma).<sup>[113] [114] [115]</sup>

The International Commission on Illumination (CIE) described the conversion of the spectrophotometric measurements to three-color parameters (CIE L\* a\* b\*) where L\* refers to the lightness, a\* redness to greenness and b\* yellowness to blueness. In addition it knowledge of the spectrum of light from the illuminant, how that light is transmitted or reflected from the object, and the human's ability to recognize hue, value and chroma is also required.<sup>[116]</sup> In the CIElab\* system a formula is used to calculate color differences:

$$\Delta E_{ab} = [(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)^2]^{1/2}$$

This “ΔE” value became pivotal in color science, for both industry and dentistry.<sup>[111]</sup> The CIE has selected incandescent (A) lamplight and daylight (D65) as standard illuminants, representing the most common illuminating lights in daily life.<sup>[117]</sup> The most important illuminant is D65 with a correlated temperature of 6500K.<sup>[118, 119]</sup>



## 2) Geometric attributes

- **Scattering:** the process by which light passing through granular, fibrous or rough surface matter is redirected throughout a range of angles.<sup>[110, 111]</sup>
- **Opacity:** the property of a material to hide what is behind it.<sup>[110, 111]</sup>
- **Transparency:** the property of a material by which a negligible portion of the transmitted light undergoes scattering.<sup>[110, 111]</sup>
- **Translucency:** the property of a material by which a major portion of the transmitted light undergoes scattering.<sup>[110, 111, 116]</sup>

## 3) Optical attributes

- **Opalescence:** the process by which a material appears yellowish-red in transmitted light and blue in the scattered light perpendicular to the transmitted light. The phenomenon is named after the appearance of opal stone.<sup>[110, 111, 120]</sup>
- **Fluorescence:** the process by which electromagnetic radiation of one spectral region is absorbed and reradiated at other, usually longer, wavelengths.<sup>[110, 111]</sup>
- **Metamerism:** the phenomenon whereby the color of specimens match when illuminated by light of one spectral composition despite differences in spectral reflectance of the specimens, and that consequently may not match in light of some other spectral composition.<sup>[111, 112]</sup>
- **Gloss:** The property of a surface which involves specular reflection and is responsible for lustrous or mirror-like appearance.<sup>[111, 112]</sup>

Standardization of color science in dentistry requires the establishment of a reference value for the evaluation of research study results in terms of  $\Delta E$ . Differentiation between perceptibility (the difference that can be identified by the human eye) and acceptability (the difference that is considered tolerable) was proposed.<sup>[111]</sup> Vichi et al proposed 3 different

intervals for distinguishing color differences.  $\Delta E$  values of less than 1 unit were regarded as not appreciable by the human eye;  $\Delta E$  values greater than 1 and less than 3.3 units were considered appreciable by skilled operators, but clinically acceptable;  $\Delta E$  values greater than 3.3 were considered perceivable by untrained observers (e.g. patients), and for that reason were regarded as not acceptable. [111, 121-123] Despite great efforts on the part of researchers to identify a  $\Delta E$  value for clinically acceptable differences, the establishment of a widely accepted limit is still controversial.

According to the rules of physics, the optical behavior of any material is dependent on many factors (Halliday & Resnick, 1993):

- 1) Light refraction: *“the change in the direction of the light wave due to change in the transmission medium”*.
- 2) Light dispersion: *“a wavelength-dependent refractive index, causes different colors to refract at different angles, splitting white light into a rainbow”*.
- 3) Light transmission: *“the fraction of incident light at a specified wavelength that passes through a sample”*.
- 4) Light absorption: *“the way in which the energy of the wavelengths is taken up by matter”*.

The amount of light that is absorbed, transmitted or reflected depends on the crystalline content, chemical nature, and particle size of the restorative material. Light will be reflected if the particle size is larger than the incident wavelength.

#### **2.3.7.2. Measurement of Translucency**

Translucency parameter (TP) and Contrast ratio (CR) are the most common parameters used to measure the translucency of dental materials. TP can be defined as the color difference

between a uniform thickness of a specimen over a white and black background. Translucent materials have high TP value.<sup>[124]</sup> CR is the ratio of the reflectance of a specimen over a black background to that over a white background of a known reflectance.<sup>[119, 125, 126]</sup> CR of 0 value is considered to be the most translucent and CR of 1 is the most opaque.<sup>[127]</sup> The following equation is used to calculate the TP values of a material:

$$TP = [(LB^* - LW^*)^2 + (aB^* - aW^*)^2 + (bB^* - bW^*)^2]^{1/2}$$

Letters “B” and “W” stand for color coordinates over the black and white backgrounds, respectively.

CR values are calculated according to the following equation:

$$CR = YB/YW$$

YB: represents the spectral reflectance of light of the specimen over a black background.

YW: represents the spectral reflectance of light of the specimen over a white background.

Multiple machines have been invented to measure color and translucency of dental and industrial materials. These include spectrophotometers, spectroradiometers and colorimeters.

Spectrophotometers are amongst the most accurate, useful and flexible instruments for overall color matching and color matching in dentistry.<sup>[128, 129]</sup> They measure the amount of light energy reflected from an object at 1-25 nm intervals along the visible spectrum.<sup>[128, 130, 131]</sup> The main components of the spectrophotometer are 1) a source of optical radiation, 2) a measure of dispersing light, 3) an optical system for measuring, 4) a detector and a means of converting light obtained to a signal that can be analyzed and interpreted.<sup>[128]</sup> One research study showed that spectrophotometers had 33% more accuracy and objective matching in 93.3% of the cases as compared with human interpretation of color.<sup>[128, 132]</sup>

Spectroradiometers measure the spectral energy of a source. Spectral radiance and irradiance are expressed by luminance (cd/m<sup>2</sup>) and illuminance (lux) units, respectively.<sup>[112]</sup>

Measurements of spectrophotometer and spectroradiometer were found to be comparable in an *in vitro* research study done by Lim et al.<sup>[133]</sup>

Colorimeters measure tri-stimulus values and filter light in red, green and blue areas of the visible spectrum. However, aging of the filter can negatively affect accuracy.<sup>[128]</sup> Colorimeters are useful to quantify color differences between two tested specimens with convenience and simplicity.<sup>[112]</sup>

Whenever the light passes through a translucent material some of the light will scatter to the edges without being absorbed. The lost light which is not detected by the spectrophotometer results in loss of accuracy in color measurements.<sup>[134]</sup> This loss of light is referred to as the *edge loss phenomenon*. When measuring the translucency of a material the edge loss phenomena should be taken into consideration.<sup>[126, 135]</sup> It is worth mentioning that zirconia material is not translucent so the effect of the edge loss on the measurements of translucency of zirconia samples is minimum.

### **2.3.7.3 Teeth and restorative material translucency**

In a natural tooth, translucency can be seen through the incisal and proximal aspects due to the presence of high proportion of enamel compared to the underlying dentin. Light transmission is less in the cervical aspect of the teeth due to thicker dentin layer.<sup>[119]</sup> The color of the dentin has the greatest influence on the overall color of a tooth. Dentin only allows an average of 52.6% light transmittance. Enamel, on the other hand, is considered the color modifier, allowing 70.1% light transmittance.<sup>[136]</sup>

At 1 mm thickness Dentin has a CR of 0.60 and a TP of 16.4. The CR of enamel is 0.55 and the TP of enamel is 18.7 at the same thickness.<sup>[137]</sup> Dental materials varies in their translucency. Multiple studies reported on the CR and TP of different ceramic materials. CR of feldspathic porcelain at 1.1 mm thickness is close to 0.78.<sup>[138]</sup> TP of different ceramics were reported to be 19.87 for IPS Empress Esthetic, 16.19 for E. max press, 15.91 for Ceram and 14.49 for E. max CAD at 0.5mm thickness.<sup>[139]</sup> Zirconia has low translucency and high opacity. The CR of the zirconia was reported to be close to 1.<sup>[4, 7, 119]</sup> Heffernan et al found that the CR of 0.5mm thick samples of ceramics and non-ceramic materials to be in this order from the most to the least translucent: Vitadur Alpha (0.60) > Empress (0.64) > In-Ceram Spinell (0.67) = Empress 2 (0.68) > Procera (0.72) > In-Ceram Alumina (0.87) > In-Ceram Zirconia (1.00) = SF52 alloy (1.00).<sup>[4, 119]</sup>

Material color, thickness and surface texture affect the translucency of the material. Darker restorations absorb more light and transmit less, which increases opacity.<sup>[140]</sup> It has been reported that increased restoration thickness increases the opacity and decreases the TP.<sup>[1, 141]</sup> In one *in vitro* research study all of the zirconia ceramics evaluated showed some degree of translucency, however, it was less sensitive to thickness compared to that of the glass ceramics.<sup>[11]</sup>

### **2.3.8 Zirconia's resistance to acidic oral environments**

Dental erosion is a multifactorial disease affects significant number of patients. In many cases crowning the patient's teeth using acid resistant restorative materials is the best treatment option. This is especially true when the source of the erosion is intrinsic acid and control of the condition is difficult to obtain as in GERD patients.<sup>[142, 143]</sup> Many research studies have shown association between dental erosion and GERD. A research study done by Meurman et al examined

117 patients with GERD found erosion in 24%. Another research study examined 198 GERD patient found erosion in 68%.<sup>[144-146]</sup>

The behavior of the zirconia restorations in *in vitro* acidic and erosive conditions was studied by Sulaiman et al. It was concluded that acidic challenge had minimum effect on zirconia's optical properties.<sup>[147]</sup> Bi-axial flexural strength of zirconia after acidic challenge has yet to be investigated.

#### **2.4. Guidelines for teeth preparation for full coverage restorations**

The success and longevity of full coverage restorations are highly dependent on the design of the tooth preparation. According to Goodacre et al, teeth should be prepared so that they meet the following characteristics: 10 to 20 degree total occlusal convergence, a minimal occluso-cervical dimension of 4 mm for molars and 3 mm for other teeth, and an occluso-cervical to facio-lingual dimension ratio of 0.4 or greater. Facio-proximal and linguo-proximal line angles should be preserved whenever possible.<sup>[148]</sup>

The amount of occlusal and axial reduction should be determined according to the material being used to fabricate the indirect restorations.<sup>[148]</sup> Historically, full cast restorations allow for the most conservative preparations. Casting alloys are known to be strong in relatively thin thickness. Optimum preparation can be performed with a 0.5 mm axial reduction and 1-1.5 mm occlusal reduction. When esthetics is of a concern, use of porcelain fused to metal or zirconia restorations may be more appropriate. However, to obtain pleasing esthetic results, more aggressive reduction is needed to provide enough room for the layering porcelain. A reduction of 1-1.2 mm axial and 1.5-2 mm occlusal is indicated for such restorations. Recently introduced monolithic zirconia restorations are designed and milled in one piece thereby omitting the need for layering (veneering) porcelain.

The promising strength of zirconia materials enable clinicians to be more conservative in tooth preparation. The minimum thickness recommended for monolithic zirconia restorations is vague. Load to failure testing of zirconia crowns with different occlusal thickness: 2.00mm, 1.5mm, 1.0mm and 0.5mm showed that the fracture resistance values of all the specimens exceeded the maximum physiological occlusal loads in molar region. Complete fracture was observed only in 1 crown with a thickness of 0.5mm.<sup>[149]</sup> Monolithic zirconia crowns exhibited higher fracture loads than monolithic lithium disilicate crowns, layered zirconia crowns and metal ceramic crowns in load to failure test study. The author of that study suggested that the fracture resistance of monolithic zirconia crowns with a thickness of at least 1.0 mm can be equal to metal ceramic crowns. He also suggested that doubling the monolithic zirconia core from 0.6 mm to 1.5 mm increases the fracture resistance of this restorative system threefold.<sup>[150]</sup> A major concern with this load to failure study is that ceramic materials fail by fracture propagation over time with cyclic loading so these results may not be clinically relevant.

The question of whether zirconia material can be considered as a substitute for metal alloys is yet to be studied. More clinically relevant research studies are needed to verify the minimum thickness of zirconia material that can be used without compromising the strength of the final restoration.

## **CHAPTER 2: RESEARCH STUDY**

### **1. Specific Aims of This Research Study:**

Specific Aim 1) Evaluate the effect of reduced thickness of fully stabilized monolithic zirconia (FSZ) and partially stabilized monolithic zirconia (PSZ) on the bi-axial flexural load after fatigue loading and thermocycling.

Specific Aim 2) Evaluate the effect of reduced thickness of FSZ and PSZ on the optical properties, Translucency Parameter (TP), Contrast Ratio (CR) and Percent Light Blockage (%LB) after fatigue loading and thermocycling.

Specific Aim 3) Evaluate the effect of acidic challenge on the bi-axial flexural load of FSZ and PSZ.

Specific Aim 4) Evaluate the effect of acidic challenge on the optical properties (TP, CR, %LB) of FSZ and PSZ.

### **2. The null hypothesis:**

1) There is no statistically significant difference in the mean of the bi-axial flexural load between different thicknesses of FSZ and PSZ prior to and after fatigue loading and thermocycling.

2) There is no statistically significant difference in the mean of the optical properties between different thicknesses of FSZ and PSZ prior to and after fatigue loading and thermocycling.



- 3) Fatigue loading and thermocycling have no statistically significant effect on the bi-axial flexural load of FSZ and PSZ.
- 4) Fatigue loading and thermocycling have no statistically significant effect on the optical properties of FSZ and PSZ.
- 5) Material thickness has no statistically significant effect on the mean of the bi-axial flexural load after fatigue loading and thermocycling.
- 6) Material thickness has no statistically significant effect on the mean of the optical properties after fatigue loading and thermocycling.
- 7) There is no statistically significant effect of acidic challenge on the mean of the bi-axial flexural load of FSZ and PSZ.
- 8) There is no statistically significant effect of acidic challenge on the mean of the optical properties of FSZ and PSZ.

### **3. Material and Methods**

#### **3.1. Material used**

BruxZir high and low translucency zirconia (Glidewell dental laboratory, Newport Beach, California, USA) were used in this study. The high translucency zirconia has 5-6 mol % yttria (third generation/ FSZ). The low translucency zirconia has 2-3 mol % yttria (conventional/ PSZ).

### 3.2. Preparation of the specimens

Zirconia cylinders ( $17.5 \pm 0.05$  mm in diameter) were milled from green stage machinable FSZ and PSZ pucks by Glidewell dental laboratory. Disk shaped specimens were sectioned from the cylinders using a diamond disk (NTI® serrated diamond discs, double sided, Coarse, green, 45.00 mm diameter, 0.30 mm thickness) mounted on straight handpiece at 15,000 rotation per minute (rpm). A specimen holder and a manually fabricated jig were used to control the cutting. Two different thicknesses were prepared, 1.5 mm and 0.9 mm. Final thickness of the specimens was obtained by sequentially grinding using silicon carbide grinding paper 380, 600, 800 and 1200 (Buehler, Illinois, USA). The final thickness ( $\pm 0.05$  mm) was measured using a digital caliper (Digimatic Micrometer, Mitutoyo corporation, Japan). All specimens were sintered according to the manufacturer's instructions. The sintering temperature was  $1580^{\circ}\text{C}$  and  $1530^{\circ}\text{C}$  for the PSZ and FSZ, respectively. The first holding time was 60 minutes at  $1200^{\circ}\text{C}$  followed by a second holding time of 150 minutes at  $1580^{\circ}\text{C}$  (for the PSZ) and  $1530^{\circ}\text{C}$  (for the FSZ). The cooling rate was  $15^{\circ}\text{C} / \text{minute}$ .

After sintering, specimens were polished with sequential diamond polishers ([BruxZir® Polishing and Adjustment Kit](#)) connected to a latch contra-angle handpiece at standardized speed of 15,000 rpm (NSK, Volvere Vmax, Japan) and standardized time and pressure in the same direction. The pressure was standardized using a pressure plate (Taiwan) connected to a computer software (ToothAbrasion). The software helped in standardizing the applied force for all the specimens in the range of 150-250 N (Figure 2.1). The specimens were ultrasonically cleaned in distilled water for 5 min and air-dried for 20 s. the final diameter of the specimens was 14 mm ( $\pm 0.2$  mm). The final thicknesses were 1.2 mm and 0.7 mm ( $\pm 0.05$  mm)

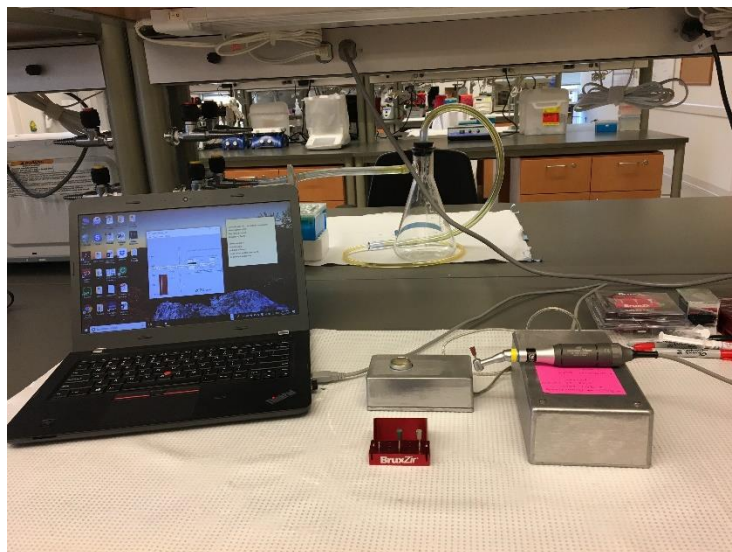


Figure 2.1. The assembly of the pressure plate.

### **Experimental groups:**

Group 1: FSZ, 1.2 mm thick, n=20

Groups 2: FSZ, 0.7 mm thick, n=20

Group 3: PSZ, 1.2 mm thick, n=20

Group 4: PSZ, 0.7 mm thick, n=20

### **3.3. Bi-axial flexural load testing**

Half of the specimens were loaded to failure using an Instron testing machine (Instron 4411, SINTECH, MTS system corporation, USA) to obtain the base line bi-axial flexural loads for each group. The other half was tested after fatiguing.

The specimens were tested dry at room temperature. Specimens were aligned on three symmetrically based steel balls (4.5 mm in diameter), 120° apart on 11 mm diameter circle. The load was applied on the center of the top surface of the specimen by the piston (diameter 1.4 mm) at a speed of 1mm/min until fracture occurred. The assembly of the apparatus is shown in (Figure 2.2). Results were recorded with Testworks, MTS, US.



Figure 2.2. The Bi-axial flexural load testing assembly

### 3.4. Optical measurements

Pre- and post- fatiguing TP, CR and %LB measurements were recorded for each specimen using a reflection spectrophotometer (Ci7600 Benchtop Spectrophotometer, X-Rite, USA) according to the CIE 1976  $L^*a^*b^*$  color scale relative to the CIE standard illuminant D65 ( Figure 3). The spectrophotometer was calibrated according to the manufacturer instructions over a standard illuminant (white background and black background) for TP and CR measurements using an aperture size of 10 mm. Measurements were done on black background and white background. The measurements of the  $L^*a^*b^*$  values were obtained for each specimen and averaged. TP and CR were calculated according to the following equation:

$$TP = [(LB^* - LW^*)^2 + (aB^* - aW^*)^2 + (bB^* - bW^*)^2]^{1/2}$$

Letters “B” and “W” stand for color coordinates over the black and white backgrounds, respectively.

CR= YB/YW where

YB represents the spectral reflectance of light of the specimen over a black background and

YW represents the spectral reflectance of light of the specimen over a white background.

The spectrophotometer was calibrated to measure transmission according to manufacturer instructions using the transmission kit. The %LB was recorded for each sample pre and post fatiguing. All measurements were recorded with Color iQC and Color iMatch software, X-Rite, USA.

The largest piece of the fractured specimens was measured in the spectrophotometer after fatiguing and thermocycling. An overestimated translucency measurements were expected when the fractured piece was smaller than the aperture size (10 mm).



Figure 2.3. Ci7600 Benchtop Spectrophotometer, X-Rite.

### 3.5. Fatiguing protocol

Specimens were mounted for fatiguing in a chewing simulator (SD Mechatronik, Germany). Specimens were coated with separator (Rubber-Sep, Kerr, Orange, CA, USA) and fixed on a glass slap with silicone glue stick. The glass slap was then coated with Vaseline petroleum jelly to facilitate the separation of the glass slap from the epoxy resin later (Figure 4). The specimen holders were coated with petroleum jelly as well. Epoxy resin (Buehler, USA) was mixed according to manufacturer instructions and poured into the specimen holders of the chewing simulator. The glass slap with the specimen was seated horizontally over the epoxy resin before it hardened. After 24 hours, the glass slap was removed. The epoxy resin supporting the specimen was inspected for any bubbles. Bubble detection required remounting of the specimen.

Fatiguing of the specimens was accomplished by cyclic loading and thermocycling. Cyclic loading was accomplished by exposing the specimens to a load of 110N for 1.2 million cycles at 1.4 Hz. This level of cyclic loading is estimated to represent around 5 clinical years.<sup>[151]</sup> The motion of the opposing was set to be vertical. Steatite material was used as an opposing material. Thermocycling between 5 and 55°C in distilled water with 30 second dwell time was simultaneously accomplished. When specimen experienced fracture during fatiguing, cyclic loading was stopped but thermocycling was continued and was not interrupted.

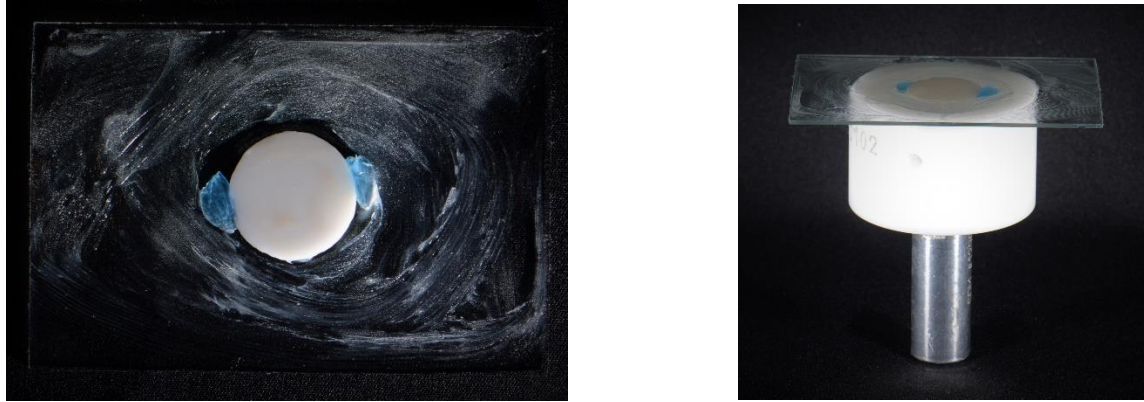


Figure 2.4. Mounting the specimens in the chewing simulator holder.

### 3.6. Post fatigue testing of specimens

Specimens after fatiguing were stored dry until post fatiguing TP, CR and %LB measurements were done for all the specimen. All specimens were loaded to failure in Instron testing machine to determine post fatiguing bi-axial flexural load.

### 3.7. Acidic challenge

Using the same methodology mentioned previously, ten additional specimens were prepared from both of the FSZ and PSZ ( $14 \text{ mm} \pm 0.2$  diameter,  $1.2 \pm 0.05 \text{ mm}$  thick). TP, CR and %LB were measured using the spectrophotometer. Artificial gastric acid was prepared according to Hunt and McIntyre's method.<sup>[110]</sup> Hydrochloric acid (HCl) 0.06 M (0.113% solution in deionized water) was prepared. The solution was adjusted to pH of (1.2) using a pH meter (Mettler Toledo, 8603, Schwerzebach, Switzerland). The specimens were immersed in a 5 ml of the simulated gastric acid for 7 days in a 37°C incubator (MaxQMini 4000, Barnstead Lab-Linename, USA). pH was monitored every day to ensure stability. The specimens were then rinsed with copious amounts of deionized distilled water (pH 7.0) and then stored dry at 25 °C. The TP, CR, %LB and bi-axial flexural load were measured after the acidic challenge.

## **4. Statistical analysis**

### **4.1. Bi-axial flexural load**

A factorial analysis of variance (anova) was used to assess the effect of material, thickness, and intervention (before and after fatiguing) on biaxial flexure load. Pairwise interactions of material by intervention and thickness by intervention were initially included in the model. Neither interaction was statistically significant ( $P = 0.09$  and  $0.15$  respectively) and both were therefore removed from the statistical model.

To assess the effect of acidic challenge, a two way anova including material, intervention, and pairwise interaction. The pairwise interaction was not statistically significant ( $P = 0.9121$ ) and was therefore removed from the statistical model.

### **4.2. Optical measurements**

For fatiguing, a repeated measures analysis of variance with one within specimen factor, time representing before and after fatiguing, and two between specimen factors, material and thickness, was used to assess TP, CR and %LB. The interaction between material and thickness was not included in the model for acidic challenge. Only time and material were included in the model.

## **5. Results**

### **5.1. The bi-axial flexural load results**

The bi-axial flexural load values of all the tested groups are summarized in (Table 2.1).



### **5.1.1. Effect of fatiguing**

Material type, thickness and exposure to fatigue had a statistically significant effect on the mean bi-axial flexural load (Material:  $P < 0.0001$ ; Thickness:  $P < 0.0001$ ; Intervention:  $P = 0.004$ ). The mean for FSZ material controlling for thickness and intervention was 250.8 N (95%CI: 185.7, 315.8) while the mean for PSZ material was 725.18 N (95%CI: 677.0, 773.4) (Table 2.2). For thickness, the mean for 0.7 mm thickness controlling for material and intervention was 221.6 N (95%CI: 161.1, 282.2) while for 1.2 mm the mean was 754.3 N (95%CI: 702.7, 805.9) (Table 2.3). Before fatiguing, the mean controlling for material and thickness was 547.9 N (95%CI: 499.7, 596.1) and after the mean was 428.03 N (95%CI: 363.0, 493.1) (Table 2.4). FSZ specimens did not survived the fatiguing protocol where 84.6% of the 0.7mm thick specimens and 36.4% of the 1.2 mm thick specimens fractured during fatiguing. According to that a statistical analysis is not valid for the post fatiguing flexural load values. All specimens of the PSZ groups survived the fatiguing protocol (Figure 2.5).

### **5.1.2 Acidic challenge**

The mean of the bi-axial flexural load of the materials before and after acidic challenge was not statistically significant ( $P = 0.11$ ). The mean for FSZ material was 375.8 N while the mean for PSZ material was 1162.5 N. The effect of material controlling for the acidic challenge intervention was statistically significant ( $P < 0.0001$ ).

## **5.2. Optical Properties**

The reported mean (SD) of the TP, CR, %LB of the different groups are shown in (Table 2.5).

### **5.2.1. Translucency parameters (TP)**

#### **5.2.1.1. Effect of fatiguing**

The pattern of change with fatiguing was not statistically significantly different for the two materials ( $P = 0.25$ ). The overall difference between the two materials was statistically significant ( $P < 0.0001$ ) with FSZ material having a higher TP, on average, than PSZ material both before and after fatiguing. For FSZ, the average before was 13.16 (SE: 0.11) and after 14.35 (SE: 0.18) while for PSZ the average before was 12.16 (SE: 0.11) and after 13.0 (SE: 0.18) (Table 2.6). There was a statistically significant difference in the pattern of change for the two thicknesses ( $P < 0.0001$ ). Before fatiguing, the average TP difference between the two thicknesses was 2.88 (95% CI: 2.57, 3.18) with 0.7 mm having an average TP of 14.10 (SE: 0.11) and 1.2 mm an average of 11.22 (SE: 0.11) while after fatiguing, the average difference between the two thicknesses was 4.33 (95% CI: 3.82, 4.83) with 0.7 mm thickness showing a slight increase in TP to 15.84 (SE: 0.18) while 1.2 mm thickness did not change perceptively (average 11.51; SE: 0.18) (Table 2.7).

#### **5.2.1.2. Acidic Challenge**

The pattern of change with acidic challenge was not statistically significantly different for the two materials ( $P = 0.59$ ). The overall difference in the mean (SD) TP between the two materials was statistically significant ( $P < 0.0001$ ) with FSZ material having a higher TP, on average, than PSZ material both before and after the challenge. For FSZ, the average before was 11.98 (SE: 0.11) and after 12.54 (SE: 0.12) while for PSZ, the average before was 10.5 (SE: 0.11) and after 11.15 (SE: 0.12).

## **5.2.2. Contrast ratio (CR)**

### **5.2.2.1. Effect of fatiguing**

The pattern of change with fatiguing was statistically significantly different for the two materials ( $P = 0.04$ ). Before fatiguing, the mean for FSZ material was 0.68 (SE: 0.006), controlling for thickness, while the mean for PSZ material was 0.70 (SE: 0.006). Although the difference between means was small, it was statistically significant ( $P=0.004$ ). After fatiguing, the mean for FSZ material was 0.65 (SE: 0.004) while the mean for PSZ material was 0.70 (SE: 0.004). The difference after fatiguing was statistically significant ( $P < 0.0001$ ). The change for FSZ material indicated a decrease, on average, for CR while there was no change, on average, for PSZ material (Table 2.8). The pattern of change over time was not statistically significantly different for the two thickness ( $P=0.56$ ). For both 0.7 and 1.2 mm thickness, the change indicated a slight decrease of  $\sim -0.01$ , on average. The main effect of thickness, controlling for material, was statistically significant ( $P < 0.0001$ ) with 0.7 mm having a higher CR, on average, than 1.2 mm both before and after fatiguing. For 0.7 mm, the average before was 0.65 (SE: 0.006) and after 0.64 (SE: 0.004). For 1.2 mm, the average before was 0.73 (SE: 0.006) and after 0.72 (SE: 0.004) (Table 2.9).

### **5.2.2.2. Effect of acidic Challenge**

The pattern of change with acidic challenge was not statistically significantly different for the two materials ( $P = 0.62$ ). The overall difference between the two materials was statistically significant ( $P < 0.0001$ ) with FSZ material having a lower CR, on average, than PSZ material both before and after the challenge. For FSZ, the average before was 0.70 (SE: 0.0035)

and after 0.69 (SE: 0.003) while for PSZ, the average before was 0.75(SE: 0.0035) and after 0.74 (SE: 0.003).

### **5.2.3. Percentage of light blockage (%LB)**

#### **5.2.3.1. Effect of fatiguing**

The pattern of change with fatiguing was statistically significantly different for the two materials ( $P < 0.0001$ ) and for the two thicknesses ( $P < 0.01$ ). The difference in means for the two materials was 2.59 (85% CI: 1.69, 3.49) before fatiguing with FSZ material having a %LB average of 57.16 (SE: 0.31) and PSZ an average of 59.75(SE: 0.32). After fatiguing the difference in means was 6.10 (95% CI: 5.49, 6.71) with FSZ material having a blockage percentage average of 55.97 (SE: 0.21) and PSZ an average 62.07 (SE: 0.22). FSZ material had a decrease in the percentage of light blockage after fatiguing while PSZ had an increase (Table 2.10). The difference in means for the two thicknesses was 7.11 (95%CI: 6.22, 8.01) before fatiguing with 0.7 mm having a mean of 54.90 (SE: 0.31) and 1.2 mm an average of 62.01(SE: 0.32). After fatiguing, the difference in means was 5.88 (94%CI: 5.28, 6.49) with 0.7 mm having an average transmission of 56.08 (SE: 0.21) and 1.2 mm an average of 61.96 (SE: 0.22). The 0.7 mm thickness had an increase on average while 1.2 mm had a slight decrease (Table 2.11).

#### **5.2.3.2. Effect of acidic challenge**

The pattern of change with acidic challenge was statistically significantly different for the two materials ( $P < 0.001$ ). The difference in means before the challenge was 4.54 (94%CI: 3.82, 5.26) with FSZ material having a lower %LB mean (59.98; SE: 0.24) than PSZ material (64.52; SE: 0.24). After the challenge, the difference in means was 3.33 (95%CI: 2.58, 4.08)

with FSZ showing very little change on average (59.37; SE: 0.25) while PSZ material decreased (62.71; SE: 0.25).

**Table 2.1. The reported mean and (SD) of the bi-axial flexural load of the different groups. (B: baseline, F: post fatiguing, A: post-acid challenge)**

Groups (material/ thickness/ condition)	Biaxial flexural load (mean/ SD)
FSZ/ 1.2 /B	401.49/ 76.39
FSZ/ 1.2/ F	402.76/ 54.89
FSZ/ 1.2/ A	350.04/ 34.07
FSZ/ 0.7/ B	257.95/ 33.42
FSZ/ 0.7/ F	316.95/ 83.51
PSZ/ 1.2/ B	1185.07/ 107
PSZ/ 1.2/ F	998.16/ 96.2
PSZ/ 1.2/ A	1140.16/ 129.18
PSZ/ 0.7/ B	364.58/ 53.82
PSZ/ 0.7/ F	370.4/ 46.42

**Table 2.2. The calculated biaxial flexural load means of the different materials controlling for thickness and intervention (fatiguing).**

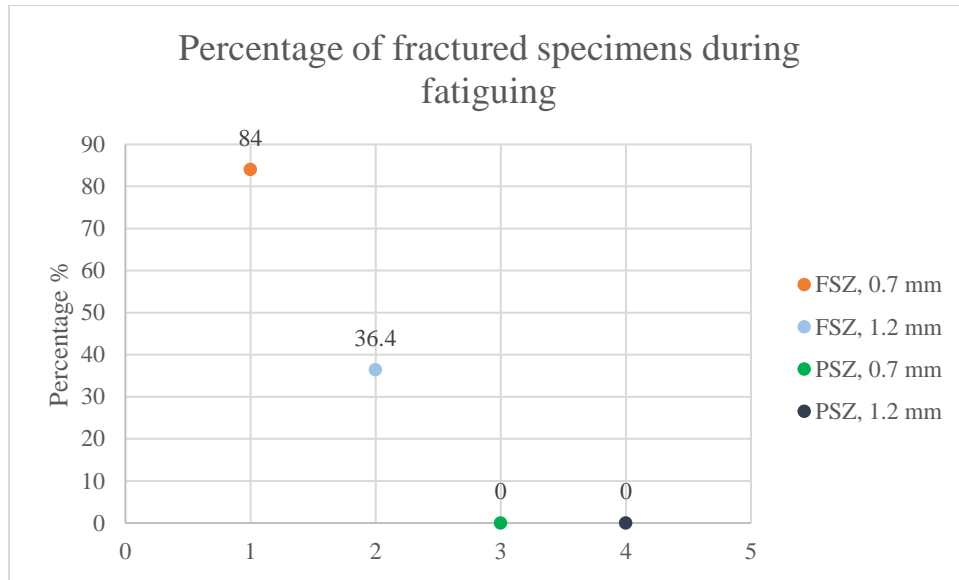
Material	Biaxial flexural load (N)	95% Confidence Intervals	
FSZ	250.8	185.7	315.8
PSZ	725.2	677.0	773.4

**Table 2.3. The calculated biaxial flexural load means of the different thicknesses controlling for material and intervention (fatiguing).**

Thickness	Biaxial flexural load (N)	95% Confidence Intervals	
0.7 mm	221.6	161.1	282.2
1.2 mm	754.3	702.7	805.9

**Table 2.4. The calculated biaxial flexural load means pre and post fatiguing controlling for material and thickness.**

Intervention	Biaxial flexural load (N)	95% Confidence Intervals	
Pre-fatiguing	547.9	499.7	596.1
Post-fatiguing	428.0	363.0	493.1



**Figure 2.5. The percentage of fractured specimens during fatiguing.**

**Table 2.5. The reported mean and (SD) of the TP, CR, and %LB of FSZ abd PSZ (F: fatiguing groups, A: acidic challenge groups)**

Groups	Pre- fatiguing/Acid			Post- fatiguing/Acid		
	TP/SD	CR/SD	%LB/SD	TP/SD	CR/SD	%LB/SD
FSZ/. 1.2/ F	11.9/ 0.36	0.7/ 0.01	60.57/ 0.97	11.94/ 1.17	0.69/0.03	59.38/ 1.01
FSZ/.1.2/ A	11.98/ 0.43	0.70/ 0.01	59.98/ 0.89	12.54/ 0.50	0.69/0.01	59.38/ 1.15
FSZ/ 0.7/ F	14.41/ 0.53	0.65/ 0.01	53.22/1.45	16.86/ 0.78	0.61/0.02	52.87/ 1.53
PSZ/ 1.2/ F	10.54/ 0.56	0.75/ 0.01	63.48/ 0.94	11.08/ 0.30	0.74/0.01	64.82/ 0.63
PSZ/ 1.2/ A	10.53/ 0.23	0.75/ 0.01	64.52/ 0.62	11.14/ 0.23	0.74/0.01	62.71/ 0.50
PSZ/ 0.7/ F	13.78/ 0.25	0.65/ 0.05	56.02/ 0.71	14.92/ 0.3	0.66/0.01	59.32/ 0.39

**Table 2.6. The calculated TP means pre and post fatiguing controlling for thickness.**

Material	Pre-fatigue mean TP	95% Confidence Intervals		Post- fatigue mean TP	95% Confidence Intervals	
FSZ	13.2	12.9	13.4	14.4	13.9	14.7
PSZ	12.2	11.9	12.4	13.4	12.6	13.36

**Table 2.7. The calculated TP means pre and post fatiguing controlling for material.**

Thickness	Pre- fatigue mean TP	95% Confidence Intervals		Post- fatiguing mean TP	95% Confidence Intervals	
0.7 mm	14.1	13.9	14.3	15.8	15.5	16.2
1.2 mm	11.2	11.0	11.4	11.5	11.2	11.9

**Table 2.8. The calculated CR means pre and post fatiguing controlling for thickness.**

Material	Pre- fatiguing mean CR	95% Confidence Intervals		Post- fatiguing mean CR	95% Confidence Intervals	
FSZ	0.68	0.66	0.69	0.65	0.64	0.66
PSZ	0.70	0.69	0.72	0.7	0.69	0.71

**Table 2.9. The calculated CR means pre and post fatiguing controlling for material.**

Thickness	Pre- fatiguing mean CR	95% Confidence Intervals		Post- fatiguing mean CR	95% Confidence Intervals	
0.7 mm	0.65	0.64	0.66	0.64	0.63	0.64
1.2 mm	0.73	0.71	0.74	0.72	0.71	0.72



**Table 2.10. The calculated percentage of light blockage means pre and post fatiguing controlling for thickness.**

Material	Pre- fatiguing mean %LB	95% Confidence Intervals		Post-fatiguing mean %LB	95% Confidence Intervals	
FSZ	57.2	56.5	57.8	55.9	55.5	56.4
PSZ	59.8	59.1	60.4	62.1	61.6	62.5

**Table 2.11. The calculated percentage of light blockage means pre and post fatiguing controlling for material.**

Thickness	Pre- fatiguing mean %LB	95% Confidence Intervals		Post- fatiguing mean %LB	95% Confidence Intervals	
0.7 mm	54.9	54.3	55.5	56.1	55.7	56.5
1.2 mm	62.0	61.4	62.7	62.0	61.5	62.4

## 6. Discussion

The effect of stabilization level and thickness of zirconia on pre- and post- fatigue bi-axial flexural load and translucency, with and without an acid challenge were tested in this *in-vitro* research study.

One of the most popular zirconia brands currently being used in the market was selected to be the test material (BruxZir High and Low Translucency Zirconia, Glidewell Dental Laboratory, Newport Beach, California, USA). Since the level of stabilization has a direct effect on the flexural strength of the material, both the partially stabilized monolithic (PSZ) referred to as “low translucency zirconia” and the fully stabilized monolithic (FSZ) referred to as “high

translucency zirconia” were tested and compared in this study. The PSZ contains 2-3 mol % yttria while the FSZ has 5-6 mol % yttria.

The PSZ groups were considered the control groups. The research study design included two clinically relevant thickness (1.2 and 0.7 mm). Sorrentino et al used an *in vitro* research study in which monolithic zirconia crowns with three different thicknesses (1.5, 1.0, 0.5 mm) were loaded to failure. The minimum thickness of monolithic zirconia crowns evaluated was 0.5mm. The level of stabilization and the yttria concentration of the tested materials were not reported. All of the crowns showed cohesive microcracks of the zirconia core. Only 1 crown with a thickness of 0.5 mm had a complete fracture.<sup>[149]</sup> Therefore, based on the results of that study, the minimum thickness of the specimens in our study was chosen to be 0.7mm. The maximum was selected to be 1.2 mm because the ISO 6872 standard for the bi-axial flexural strength requires the specimen thickness to be 1.2 mm.

The shape of the specimens was designed to facilitate establishing the aims of the study and according to ISO 6872 for the bi-axial flexural strength load test. The standards require a disk shape specimen that is 14.0 mm in diameter and 1.2 mm in thickness. In addition, disk shape specimens facilitate the optical measurements. Optical measurements cannot be accurately recorded on a non-flat surface such as natural teeth contours.

It is known that dental materials fail intraorally due to fatiguing over time in the humid oral environment. Testing dental materials in a conditions simulating the oral environment is required before adapting any new material in dentistry. The literature reports various protocols to mimic fatigue processes experienced by materials in the oral environment. The general consensus is that fatiguing materials through cyclic loading and thermocycling, via use of a

chewing simulator, may be considered the most clinically relevant fatiguing protocol.<sup>[151]</sup> If the material shows promising results after fatiguing, clinical trial should be designed to test the performance of the material intraorally.

Multiple pilot studies were done to determine the best fatiguing parameters that matched the goal of the present study. Cyclic loading parameters included a 0.5 mm indentation, vertical movement (up- down), 1.4 Hz, 110 N and 1.2 million cycles. Thermocycling was done simultaneously with fatiguing at 5 and 55 °C, 30 second dwell time, 10,000 cycles. These parameters approximate 5 years clinical service.

The result obtained from this study showed that FSZ is superior to PSZ in terms of translucency and less light blockage percentage. However, PSZ exhibited greatly increased bi-axial flexural load compared to the FSZ ( $P < 0.0001$ ). PSZ groups reported mean bi-axial flexural load of 1185.07, 364.58 N at 1.2 and 0.7 mm thickness, respectively. On the other hand, FSZ reported 401.49, 257.95 N at 1.2 and 0.7 mm thickness, respectively. All of the groups had a base line bi-axial flexural loads exceeding the normal physiological mastication loads (50- 250 N).<sup>[149, 152]</sup> Only the PSZ at 1.2 mm thickness had bi-axial flexural load equal to the reported parafunctional occlusal loads (500-900N) in molar regions.<sup>[149, 152]</sup> However, the fact that FSZ specimens did not survive the fatiguing protocol indicates that even though if the baseline loads tolerated by this material exceed the normal physiological load, the survival of this material intraorally is questionable. The manufacturer's recommendation for the minimum occlusal thickness of the PSZ used in this study is  $> 0.7$ mm when used in the anterior region and  $> 0.9$  mm when used in the posterior region. The recommendation for FSZ is a minimum of 0.9 mm occlusal thickness of a restoration whether used in anterior or posterior region (Prismatik Dentalcraft, Inc. A wholly owned subsidiary of Glidewell laboratories).

The observation of fractured FSZ specimens during fatiguing at less than 200,000 cycles (equivalent to < 1 year of clinical service) raises potential caution when considering the use of this material at reduced thickness. The percentages of the specimens fractured during fatiguing of the FSZ were 36.4% at 1.2 mm, 84.6% at 0.7 mm thickness. None of the PSZ specimens fractured during fatiguing at either thicknesses. According to the results in this study, the assumption that zirconia is a metal substitute cannot be affirmed for the new zirconia generations, where the bi-axial flexural strength of fatigued FSZ at reduced thicknesses is not as great as PSZ. The manufacturer of the BruxZir Anterior does not recommend using it in less than 0.9 mm occlusal thickness.

Stawarczyk et al reported four point flexural strength values for second generation monolithic zirconia materials that ranged between 611 - 784 MPa and 867 - 928 MPa for conventional (PSZ) zirconia. Specimen's dimensions were 4.0 mm (length), 4.0 mm ( $\pm 0.2$ ) mm (width) and 3.0 ( $\pm 0.2$ ) mm (thickness). Second generation zirconia had significantly less four-point flexural strength than PSZ. Elsaka et al, in a comparable research study reported four-point flexural strength value of 509- 676 MPa for third generation (FSZ) materials (yttria content 8.5-9.5%) and 960 MPa for the PSZ (yttria content 4.5%).<sup>[153]</sup> Biaxial flexural strength values of four ceramic core materials in 0.5 mm thick disk shaped specimens were tested in a research study. The results showed that the bi-axial flexural strength (MPa) of different types of ceramic including conventional (PSZ) zirconia were as following: IPS Empress 2 ( $355.1 \pm 25.7$ ), VITA In-Ceram Alumina ( $514.0 \pm 49.5$ ), VITA In-Ceram Zirconia ( $592.4 \pm 84.7$ ), Cercon Base Zirconia ( $910.5 \pm 95.3$ ).<sup>[7]</sup> In another research, four different brands of translucent Y-TZP were subjected to artificial aging up to 200 hours in steam at 134°C at 0.2 MPa. After aging the t-m transformation in each specimen was quantified and the specimens were tested in 4 point flexure.

The specimens were designed as thin bars (22×3×0.2 mm). The results of that study showed that artificial aging (hydrothermal) resulted in LTD as shown by an increase in the monoclinic phase fraction for all specimens. The four point flexural strength changed from a mean  $\pm$ SD of 1612  $\pm$ 197 MPa to all fractured during aging for Prettau; 1248  $\pm$ 73.5 MPa to all fractured during aging for BruxZir Anterior; 1052  $\pm$ 84.2 to 1099  $\pm$ 70 MPa  $\pm$ 130 for Katana HT13; and from 875  $\pm$ 130 to 909  $\pm$ 70 MPa for Katana ML.<sup>[8]</sup>

The bi-axial flexural strength ISO standard recommends using specimen dimensions that are more clinically relevant. The standards require a disk shape specimen, 14.0  $\pm$  2.00 mm in diameter, 1.2  $\pm$  0.2 mm thickness. In this research study, third generation zirconia (FSZ), showed statistically significantly less bi- axial flexural load than conventional zirconia and experienced fracture during fatiguing (P< 0.0001). The thinner the specimens were, the more fractures occurred.

To the best of our knowledge, there are currently no research studies that reported on the bi-axial flexural load of third generation zirconia after fatiguing in a chewing simulator. All of the previously mentioned studies reported flexural strength not biaxial flexural strength. However, it is worth mentioning that the results of this study relatively validate and agree with the results of the previously discussed studies.

An *in vitro* research study by Nakamura et al suggested that conventional monolithic zirconia crowns with a chamfer width of 0.5 mm and occlusal thickness of 0.5 mm can be used in the molar region.<sup>[154]</sup> In this research study none of the conventional zirconia specimens fractured during the fatiguing protocol. Our observation validates Nakamura et al's results.

The order of the material from the most to the least translucent according to the TP and CR measures was as follows: FSZ (0.7 mm), PSZ (0.7 mm), FSZ (1.2mm), PSZ (1.2mm). Higher light blockage was recorded in PSZ groups, 62%, 56% at 1.2 mm, 0.7 mm thickness respectively. FSZ had 60.2%, 52.5% light blockage at 1.2mm, 0.7 mm respectively.

Reducing the specimen's thickness resulted in improvement of translucency and light transmission for both types of zirconia ( $P < 0.0001$ ). The findings of this study confirm what previous studies found about the inverse relation between the thickness of the restorative material and the translucency. <sup>[1, 138]</sup>Wang et al reported that the TP values of zirconia specimens ranged from 5.5 to 15.1 when the thickness decreased gradually from 1.00 mm to 0.4 mm. He also found that zirconia translucency was less sensitive to thickness compared to that of glass ceramics.<sup>[1]</sup>

After fatiguing, there were statistically significant changes in the optical measurements in some of the groups. These changes might not be clinically significant. The difference in the TP value ( $\Delta E$ ) was less than 3.3 in all material and thickness combinations.  $\Delta E$  values greater than 1 and less than 3.3 units were considered appreciable by skilled operators, but clinically acceptable. A value less than 1 is not noticeable by a skilled operator.

The increase in the translucency of the 0.7 mm FSZ group can be partially caused by experimental errors. The majority of the specimens in this group were fractured during fatiguing. If the measured fractured piece was not fitting the aperture size perfectly as the pre- fatigued specimens, the recorded measures may not be as accurate. One recently published study showed that BruxZir anterior solid zirconia at 1 mm thickness has shown stability in light transmission after a hydrothermal aging protocol. <sup>[6]</sup>

It was found in this study that acidic challenge did not significantly affect the bi-axial flexural load ( $P= 0.11$ ) of both FSZ and PSZ. The pattern of change in the optical properties was not statistically significantly different between the two materials. Sulaiman et. al. found in their study that TP values increased significantly for the PSZ but not for the FSZ after acidic challenge for 96 hours in a 37 °C incubator.

The null hypotheses that fatiguing had no statistically significant effect on PSZ was accepted. All other null hypotheses were rejected.

## **7. Limitations of the study**

One of the major limitations of this study is that we tested only one brand of zirconia material. It is well known that not all zirconia materials are the same. FSZ and PSZ from multiple manufacturers have different formulations and chemical composition, rendering different physical and optical properties between these materials. Thus, the results of this research study cannot be generalized or extrapolated to zirconia materials from different manufacturers.

Another limitation is the specimen design. Ideally, crown shaped specimens cemented on natural teeth or standardized dies would have represented a more clinically relevant scenario. In this study, the zirconia disks were mounted flat in epoxy resin. It is known that ceramic crowns are brittle and more prominent to fracture when they are not cemented or adhesively bonded over the teeth. Even though zirconia is stronger and less brittle than all of the ceramic materials currently available for use in dentistry, it may be expected that higher levels of fracture resistance occur If zirconia is cemented or bonded to supporting the supporting enamel or dentin substrate. The potential exists that the findings of this research study under-estimate the actual

clinical bi-axial flexural load of the tested materials. However, this assumes successful and durable bonding to natural tooth substrates under ideal clinical conditions.

Clinical studies are needed to ascertain the level of clinical relevance if this *in vitro* study.

## **8. Conclusions**

- 1) Different generations of zirconia according to yttria concentration display different mechanical and optical properties. Specifically, third generation (FSZ) is significantly weaker and slightly more translucent than the conventional (PSZ).
- 3) The notion that zirconia is a metal substitute cannot be affirmed for the FSZ evaluated, the bi-axial flexural loads at reduced thickness are not as great as the PSZ.
- 4) Reducing the material thickness minimally enhances the optical properties of the PSZ and FSZ materials evaluated.
- 5) Acidic environment has no significant effect on the bi-axial flexural load or optical properties of both PSZ and FSZ.



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