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# Influence of different thickness on the flexural strength of zirconia ceramics

Master thesis

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#### SUMMARY

Influence of different thickness on the flexural strength of zirconia ceramics.

The aim of this project was to evaluate and compare the different thickness of zirkonzahn ceramics and its mechanical properties of ceramics, which have generated interest in restorative dentistry because of their high strength and high resistance to fracture.

Three different thickness and 10 samples of zirkonzahn ceramic were selected for this study (15x0.3mm), (15x0.5mm), (15 x0.7mm) Three control discs of the green stage Zirconia block were cut with saw (leitz 1600) and sintered in zirkonzahn sintering oven start at 20- 1500 °C. All discs were polished with diamond paste. The cycle of all processes take 8 hrs and this procedure were provided by (zirkonzahn Furnace). The surface of the Zirconia specimens were ground with 600, 800, 1200 (Silver carbide abrasives) under water cooling and then with 120  $\mu$ m Al2O3 particles are blasted to 0.5 MPa pressure from a distance of 10 mm, 15 seconds.

The evaluation data were performed by NCSS (Number Crunchers statistical system) 2007&PASS 2008 Statistical Software (Utah, USA) program. The evaluation of operating data, comparisons between groups one-way analysis of variance (one way ANOVA) test and Tukey HDS test was used for the determination of the group that lead to differences. Pearson correlation analysis was used for assessing the relationships between parameters p <0.05 level were evaluated.

The mean biaxial flexural strengths of ten discs are presented in all groups. The highest mean biaxial flexural strength found at 0.7mm than at 0.5 mm and lowest value was found at 0.3mm. A cording to our results; increasing the core thickness of Zirconia from 0.3 to 0.5mm flexural strength was increased 7%. But core thickness did not differ between 0.5mm and 0.7mm, Which revealed no significant difference between the fracture strength values of the test groups (P>.05).

In this study the Increasing the core thickness from 0.3 to 0.5 has shown 7% increase at the flexural strength and the Increasing the core thickness from 0.5 to 0.7 mm has shown no differences at the flexural strength.

Result of this in-vitro study shows that increasing the thickness of zirconia from 0.3 mm to 0.5 or 0.7 mm it does not appear to be significant effect on flexural strength.

Keyword: Zirconia, flexural strength, core thickness.

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#### **1. Introduction**

#### 1.1 Dental ceramics:

Ceramic comes from the Greek Keramos, which means pottery or burnt stuff. Historically, three basic types of ceramic materials have been developed (1). Earthenware is fired at low temperatures and is relatively porous. Stoneware, which appeared in China in about 100 B.C., is fired at a higher temperature than earthenware; in both materials, firing increases strength and renders it more impervious to water (1). The third material is porcelain, obtained by fluxing white china clay with China stone to produce white translucent stoneware. This porcelain was developed in King-te-tching in China in about 1000 A.D. and is much stronger than earthenware and stoneware (1). The French apothecary, Alexis Duchateau, introduced the first ivory denture in 1774 (1, 2). However, the denture became badly stained, porous and absorbed mouth fluids (1). In collaboration with Duchateau, the first porcelain tooth material was patented in 1789 by a French dentist, Nicholas Dubois de Chemant (2). The product was improved from the previous version that was produced in 1774; however, the method of attaching the teeth to a denture base was still ineffective (2). In 1808, Fonzi, an Italian dentist, invented a terrometallic porcelain tooth that was held in place by a platinum pin or frame (1, 2). Planteau, a French dentist, introduced porcelain teeth to the United States in 1817, and, Peale, an artist in Philadelphia, developed a baking process for them in 1822 (2). Dr. Charles Land introduced the first successful fused feldspathic porcelain inlay and crowns to dentistry in 1886 (3).

Land described a technique for fabricating ceramic crowns using a platinum foil as a substructure with the high controlled heat of a gas furnace. These crowns exhibited excellent aesthetics but the low flexural strength of porcelain resulted in a high incidence of failure (2). Since then, feldspathic porcelains with reliable chemical bonding have been used in metal-ceramic prostheses for more than 35 years. However, feldspathic porcelains have been too weak to use reliably in the construction of all-ceramic crowns without a cast-metal or metal foil coping (2). Furthermore, their firing shrinkage causes significant discrepancies in fit and adaptation of margins. Significant improvement in the fracture resistance of porcelain crowns was introduced by McLean and Hughes, who developed a high-alumina reinforced porcelain restoration in 1965 (4). They fabricated alumina of 95% purity as an aluminous core ceramic consisting of a glass matrix containing 45-50% Al2O3 (5). Because of the inadequate translucency of the aluminous porcelain core material, a veneer of feldspathic porcelain was required to achieve acceptable aesthetics. Aluminous porcelain crowns provided low flexural strength of approximately 131 MPa; therefore, this type of porcelain crown was only used for anterior restorations. Recently, there has been development in both dental ceramic materials and fabrication techniques. For example, higher strength substructure materials such as lithiumdisilicate, alumina, and zirconia have been used. Additionally, fabrication techniques such as slip-casting and copy milling techniques have been improved.

#### 1.2. Classification of dental ceramics

Can be classified of dental ceramics according to their fusion temperature, chemical composition, application, processing method, and substructure material (2,6). According to the firing temperature, dental ceramics can be divided into high-fusing (1300°C), medium fusing(1101-1300°C), low fusing (850-1100°C), and ultra-low fusing (<850°C) ceramics (7). This classification was employed more intensively with earlier dental ceramic compositions, which contained three major ingredients: feldspar, quartz, and clay (or kaolin) (6). The fusion temperature is dictated by the relative amount of these three ingredients. In addition, ceramics can be classified by chemical composition, such as feldspathic porcelain (high and low leucite), glassceramic (lithium disilicate and mica), and core reinforcement (alumina and magnesia) (8); by application such as denture teeth, metal ceramics, veneers, crowns, inlays anterior bridges, and posterior bridges; by processing method such as pressable, sintering, machining, or casting; or by substructure material such as cast metal, glass-ceramic, CAD/CAM porcelain, or sintered ceramic core (2). A classification of dental ceramics in this review will focus on types of ceramics, which are metal ceramic and all-ceramic and then the latter group will be classified by the processing method.

## 1.2.1 Metal ceramic (Porcelain-fused-to-metal)

Ceramic-metal restorations consist of a cast metallic framework (or core) on to which at least two layers of ceramic are baked. The first layer to be applied is the opaque layer, consisting of ceramic, rich in opacifying oxides (6). Its role is to mark the darkness of the oxidized metal framework to achieve adequate aesthetics (6). The next layer is opacious dentine then dentine and enamel to obtain an aesthetic appearance similar to that of a natural tooth. After it has been built, the ceramic-metal crown is sintered in a porcelain furnace. The alloys used for casting the substructure are usually gold-based, containing tin and indium. Gold-palladium silverpalladium, and nickel-chromium alloys were initially developed as lower cost alternatives (6). It has been shown that metal-bonded ceramic crowns are up to three times stronger than conventional all-ceramic crowns (9). One of the common causes of failure with this system is the separation of the ceramic from the metal due to an interfacial breakdown of the metalceramic bond. Most ceramics have a coefficient of thermal expansion less than that of metals. The metal tries to contract more than the ceramic during cooling because of its higher coefficient of expansion. If the mismatch between the metal and ceramic is too big, internal stresses may be created during cooling, which in turn may cause the ceramic to fracture (9). The coefficient of thermal expansion of feldspathic glasses used in the construction of the porcelain jacket crowns is 7-8 ppm.°C-1. This value is lower than alloys, which are typically in the range of 14-16 ppm.°C-1 (9). To overcome the mismatch in thermal coefficients, soda (Na2O) and potash (K2O) are added to the ceramic composition to increase the thermal expansion (6, 9). The addition of these oxides leads to the formation of the crystalline phase in the glassy ceramic (9). This crystalline phase is known as tetragonal leucite. Additionally, the introduction of soda and potash leads to a reduction in the firing temperature, which reduces distortion due to creep of the alloy (9).

#### 1.2.2 All ceramics

#### 1.2.2.1 Alumina-reinforced porcelain jacket crown

The alumina-reinforced feldspathic core was developed by Hughes and McLean in 1965 (4). The material consists of a feldspathic glass containing 45-50% alumina. The alumina ceramic is strengthened by dispersion of a crystalline phase in the glassy matrix (6). Traditionally, the core was baked on a platinum foil and later veneered with matched-expansion porcelain; however, it is now more commonly baked directly on a refractory die (6). The alumina particles are stronger than the glass and more effective at preventing crack propagation than quartz (9). However, the flexural strength of feldspathic porcelain is at best 60 MPa, which is raised to 120-150 MPa

for the aluminous core porcelain. This strength is insufficient in posterior sites and is suitable only in anterior sites (9).

## 1.2.2.2 Leucite-reinforced feldspathic porcelain

Leucite-reinforced feldspathic porcelain contains 45% by volume tetragonal leucite, which acts as a reinforcing phase (6) The thermal contraction mismatch between leucite (22 to 25 x 10-6.°C-1) and the glassy matrix (8x10-6.°C-1) results in the development of tangential compressive stresses in the glass around the leucite crystals, which can act as crack deflectors with increased resistance to crack propagation (6). However, the flexural strength is still low at about 104 MPa (6).

#### 1.2.2.3 Magnesia based core porcelain

Magnesia core material is compatible with the high expansion porcelain normally bonded to metals. Magnesia has a higher expansion coefficient than alumina (MgO: 13.5x10-6.°C; Al2O3: 8x10-6.°C). Strengthening is achieved by dispersion of the magnesia crystals in a vitreous matrix, and also by crystallization within the matrix. Additionally, a magnesia core can be significantly strengthened by glazing (6). Normally, a magnesia core has a modulus of rupture strength of 131 MPa after firing; however, the strength can be doubled to 269 MPa by applying glaze (6, 8). Another advantage of this material is that it is considered to be aesthetically superior to a PFM and provides no metal margin. However, it is only used for anterior crowns and is unsuitable for use as a fixed partial denture restoration (8).

#### 1.2.2.4 Castable ceramics

The first commercially available castable ceramic material was Dicor ceramic, which was introduced in the early 1980s. It is a micaceous glassceramic, which contains 45% volume glass and 55% crystalline tetra silicate mica. It is produced by a conventional lost-wax investment techniques and glass casting (10). A full contour transparent glass crown was cast at 1350°C then heat treated at 1075°C for 10 hours to form a partial crystallization (55%) of fluorine mica silicate (2). The crystals not only create an opaque material out of a transparent crown; they also increase the fracture resistance and strength of the ceramic. It has been reported to have a flexural strength of 152 MPa (8).

#### 1.2.2.5 Slip-cast ceramic

The core is made from fine grained alumina particles that are mixed with water to form a suspension referred to as a 'slip'. The slip is then placed on a gypsum die, which absorbs the water from the slip by capillary action (11). This is then heated in a furnace to produce a sintered coping or framework. It is later infiltrated with glass at 1100°C for 4 hours to eliminate porosity and to strengthen the core (12). The rest of the crown is then formed by traditional firing using body porcelain over the core (8). Examples of materials that have used this technique are In-Ceram Alumina, In-Ceram Spinell, In-Ceram Zirconia (Vita Zahnfabrik, Bad Säckingen, Germany). In-Ceram Alumina was developed by Sadoun as a core material in 1988 (11). In-Ceram Alumina was introduced as a dental glass-infiltrated core material containing about 70% alumina infiltrated with 30% sodium lanthanum glass for crown and bridge substructures (2). In-Ceram Spinell was introduced as an alternative to In-Ceram Alumina. It contains magnesium and aluminum oxide. This type of In- Ceram seems to improve translucency in the final restoration (2, 13), while In-Ceram Zirconia is a zirconia-based dental ceramic containing approximately 30 wt% zirconium dioxide and 70 wt% aluminum oxide. In-Ceram Zirconia is available as either slip or dry-pressed. The flexural strength values of the glass-infiltrated core materials are approximately 350 MPa for In-Ceram Spinell, 500 MPa for In-Ceram Alumina, and 700 MPa for In-Ceram Zirconia (2). Although the strength of the glass-infiltrated alumina cores is high, the alumina cannot be etched and saline treated for resin bonding; therefore, it cannot bond easily to tooth structure (8). Also, this material is relatively costly and has a long processing time (8). In-Ceram ceramic can be used for single anterior and posterior crowns (In-Ceram Alumina and In-Ceram zirconia), anterior three-unit bridges (In-Ceram Alumina), and three-unit posterior bridges (In-Ceram Zirconia) (2).

#### 1.2.2.6 Pressable ceramics

The method for producing pressable ceramics, which was first described by Wohlwend 1989 (14), utilises the lost wax technique. A wax pattern of the restoration is invested in a phosphate-bonded investment material. Following the burn out procedure, a glass-ceramic is pressed into the mould at a temperature of 1050°C in a custom furnace. An example of the material used is leucite-reinforced feldspathic porcelains strengthened by incorporating leucite (K2O Al2O3 4SiO2) crystals, approximately 45% volume, in the glass matrix (15). Flexural strength for these materials has been reported to be 120 MPa. Conventional feldspathic porcelains designed for metal ceramic restorations contain 12 to 25% volume leucite and have a flexural strength in the range of 60 MPa. The increase in strength has been achieved through a heat treatment that enhances the formation of a highly crystallized microstructure and resists crack propagation under stress (15). Also, large pore formation can be avoided due to the better distribution of the crystalline phase within the glass matrix the final restoration can use either the leucite-reinforced core material alone or a 2-layer all-ceramic crown veneered with low fusing porcelain (15). Some of the most representative pressable glass ceramics are IPS Empress (EM1) and Empress 2 (EM2) (16). EM1 is a leucite-reinforced glass ceramic produced in the early 1990s (14). It obtains its strength from the finely dispersed leucite crystal reinforcement and is recommended for restoring single units including veneers, inlays, onlays, and anterior crowns (16, 17).

The strength values of EM1 range from 95 to 180 MPa and the fracture toughness is approximately 1.3 MPa.m1/2 (18). In 1998, Ivoclar released EM2, which is lithium disilicate-reinforced glass ceramic processed with the same procedure and equipment used for EM1. EM2 has been recommended for core material suitable for 3 unit-fixed partial dentures up to second premolar (16, 18). The chemical composition of EM2 is 60 % by weight lithium disilicate, which represents the main crystalline content (16). The improved mechanical properties of this material compared to most other pressable ceramics are attributed to its chemical composition, which comprises dense multi-elongated lithium disilicate crystals within the glass matrix. In such a structure, a crack would be trapped by these distributed crystals, resulting in improved strength and fracture toughness (16). The strength and fracture toughness values of EM2 have been reported to range from 340-400 MPa and 2 to 3.3 MPa.m1/2, respectively (17).

#### 1.2.2.7 Machinable ceramic or CAD/CAM systems

Traditional methods for the production of indirect restorations usually include impression taking die or model making and final manufacture. Recent technology developments have enabled digitization and replication of the tooth surface by using CAD/CAM (computer assisted design/computer assisted machining) technology, which can facilitate a restoration. After the tooth is prepared, it is scanned by an optical scanner and a restoration is designed with the aid of a computer and milled by a milling machine (19). An advantage of the CAD/CAM system is that it can produce a restoration in one visit, which not only minimizes the inaccuracies of the hand/laboratory fabrication process (20); it can also reduce the time taken to produce the restoration (21). Additionally, a temporary restoration is not

used; therefore, there can be no loss or re-cementation of temporary restorations, leading to a reduction in laboratory fees. Another advantage is that disposable supplies, such as impression material, wax, stone, and temporary bridge resin, can be eliminated. Clinicians can also spend the majority of their time on tooth preparation and on seating of the final restoration (21). In addition, the system can produce a higher strength core material compared to conventional ceramic (22). However, CAD/CAM requires costly equipment and a trained dentist. Furthermore, the system produces larger internal gaps compared with conventional methods ranging from 100-200 µm (23, 24); and marginal gaps ranged from 50-80 µm (25, 26). The latest versions of CAD/CAM systems such as CEREC® 3 have been improved to provide smaller marginal gaps, 53-67 µm for crowns (27) and 39.1-52.2 µm for inlays (28). However, one study reported the marginal gap of CEREC® 3 was between 75 and 102 μm when resin composite was used (29). These findings are far from the theoretically based requirement of cementation film thickness, which should be between 25 and 40 µm (30). Zirconia crown framework was reported to have marginal opening ranging between 36.6 to 45.5 µm; however, the internal gaps are still large (50-75 µm for axial walls and 74-100 µm for occlusal gaps) (31). During the last 10 years, the use of CAD/CAM in ceramic manufacture has increased significantly, e.g. the Cerec, Celay, Procera, and Lava systems.

# 1.3 Classification of CAD/CAM systems

#### 1.3.1 CEREC Systems

CEREC stands for computer-assisted Ceramic Reconstruction. The CEREC CAD/CAM (Siemens/Sirona Dental Systems, Bensheim, Germany) method was first developed at the University of Zurich by Mörmann and Brandestini in 1980. CEREC 1 functioned for the first time in 1985 to produce the first chair side inlay and the material was Vita Mark I feldspathic ceramic (Vita Zahnfabrik) (32). In 1994, CEREC 2 was launched to give more options for restorations such as 1-3 partial and full crowns and copings. It was equipped with an additional cylinder diamond enabling formgrinding for partial and full crowns instead of using only a diamond-coated wheel like CEREC 1. The software design was still displayed twodimensionally. In 2000, CEREC 3 omitted the wheel and introduced a twobur system, consisting of a cylindrical diamond and tapered burs. It divided the system into an acquisition/design unit and a machining unit. The software for three dimensional virtual display became available in 2003 using CEREC 3 & in Lab, which makes the handling illustrative and easy both in the office and the laboratory (32). The step bur that was introduced in 2006, reduced the diameter of the top one-third of the cylindrical bur to a small diameter tip enabling high precision form-grinding with reasonable bur life (32). The CEREC system can produce a restoration in a single appointment (20). To make a restoration with the CEREC CAD/CAM chair side system (Sirona), the following sequence is carried out. First, a titanium dioxide powder is applied to the patient's prepared tooth to provide contrast for the optical scanner. The prepared tooth is scanned with an optical probe and the image is stored in a computer as a positive digital XYZ data model. The restoration is designed on a monitor screen, a block of a machinable glassceramic is selected by shade and the restoration is milled at the chair side. The materials used with the CEREC system are Vita Mark II fine-grained, feldspathic ceramic (Vita Zahnfabrik, Bad Säckingen, Germany), ProCAD leucite reinforced ceramic (Ivoclar Vivadent, Amherst, N.Y.) and Paradigm<sup>TM</sup> MZ100 composite block (3M ESPE). Four types of strong and aesthetic CEREC in Lab materials are also available: SPINELL<sup>TM</sup>, ALUMI-NA<sup>TM</sup>, ZIRCONIA<sup>TM</sup> and new Yttrium-stabilized Zirconia (YZ<sup>TM</sup>). The latest material is Invizion<sup>TM</sup>, which is milled on CEREC in Lab system and combines VITA In-Ceram YZ cubes with the VITAMV9 ceramic veneering material.

#### 1.3.2 Celay System

The Celay system (Mikrona Technologie, Spreitenbach, Switzerland), introduced in 1992, is a machinable ceramic system that is capable of milling inlays, onlays, and veneers from prefabricated industrial ceramic blocks. A technique for manufacturing crowns was then introduced in 1993 using Vita-Celay alumina blocks (33). Direct intra-oral or indirect patterns may be used to make ceramic restorations (34). Celay enables the dentist to produce a restoration during either a single treatment, or a two-session procedure (35).

# 1.3.3 DCS System

The Digitizing Computer System (DCS) can be mechanically programmed to produce a computer-milled restoration using three-dimensional computer models (36). The DCS Precedent system comprises a Preciscan laser Scanner and Precimill CAM multibtool milling center. The DCS Dent form software automatically suggests connector sizes and pontic forms for bridges. It can scan 14 dies simultaneously and mill up to 30 framework units in 1 fully automated operation. Materials used with DCS include porcelain, glass ceramic, In-Ceram, dense zirconia, metals, and fiberreinforced composites. This system is one of the few CAD/CAM systems that can mill titanium and fully dense sintered zirconia (37).

# 1.3.4 CICERO System (Computer Integrated Ceramic Reconstruction)

Computer-aided design (CAD) and computer-aided manufacturer (CAM) in dentistry offer an alternative to lessen the variability of colored reproduction in the fabrication of ceramic restorations. However, most of the CAD/CAM systems depend on the process of milling a restoration from porcelain blocks, which may make them less aesthetic then layered restorations. Therefore, improvement in the cosmetic appearance of this type of CAD/CAM restoration is made manually (38). CICERO systems can produce a ceramic coping and 2 porcelain layers (opaque and translucent porcelain) with determined thickness, which may improve the predictability of the final shade of the restoration (38). All-ceramic restorations produced by the CICERO CAD/CAM system consist of a glass impregnated aluminum oxide ceramic core (Synthoceram, Elephant Dental BV) with a thickness 0.6 to 0.8 mm and veneering porcelain which combines 2 porcelains, a less translucent version and a more translucent version. The 2 types of veneering porcelains can be applied in 16 different shades and various thickness ratios (38). The CICERO method of crown fabrication consists of optically digitizing a gypsum die, designing the crown layer build-up, and subsequently pressing, sintering, and milling consecutive layers of a shaded high-strength alumina-based core material, a layer of dentin porcelain, and a final layer of incisal porcelain. Final finishing is performed in the dental laboratory (39). The CICERO system differs from the CEREC and DCS systems in that the ceramic is sintered and milled in a centralized laboratory whereas the CEREC and DCS are produced from ceramic blocks either in the dental laboratory or in the dental practice (39).

#### 1.3.5 Procera All Ceramic System

Procera/All Ceram (Nobel Biocare, Goteborg, Sweden) was first described by Anderson and Odén (40). The Procera All Ceram crown is composed of densely sintered, high-purity aluminum oxide core combined with compatible All Ceram veneering porcelain (41). This ceramic material contains 99.9% alumina, and its hardness is one of the highest among the ceramics used in dentistry (2). Procera All Ceram can be used for anterior and posterior crowns, veneers, onlays, and inlays. A unique feature of the Procera system is the ability of the Procera scanner to scan the surface of the prepared tooth and transmit the data to a milling unit to produce an enlarged die through a CAD/CAM process. The core ceramic form is dry-pressed onto the die, and then sintered and veneered. Thus, the usual 15-20% shrinkage of the core ceramic during sintering will be compensated by constructing an oversized ceramic pattern, which will shrink during sintering to the desired size to accurately fit the prepared tooth (2). Some studies confirm that Procera restorations have high strength and excellent longevity (42). After 5 and 10 years, a cumulative survival rate of 97.7% and 93.5% was reported. The mean flexural strength for Procera alumina and zirconia is 639 (43) and 1158 MPa respectively (44).

#### 1.3.6 Cercon

The Cercon system is commonly referred to as a CAM system because it does not have a CAD component (37). After the die is prepared, a wax pattern of coping or pontic with a minimum thickness of 0.4 mm is made. The system scans the wax pattern and mills a zirconia bridge coping from pre sintered zirconia blanks in an enlarged size to compensate for the 20% shrinkage. The processing time for milling is approximately 35 minutes for a crown and 80 minutes for a 4-unit bridge (2). The coping is then sintered in the Cercon heat furnace (1,350°C) for 6 to 8 hours (37). This method of milling presintered blanks reduces milling time and increases the service life of the unit and instruments. The mean flexural strength of Cercon core using three-point bending test was 1305 MPa and the fracture strength of Cercon crown from one study (45), which was fabricated simulating a maxillary central incisor, was 1850 MPa.

# 1.3.7 LAVA TM system

The Lava system was introduced in 2002. The Lava<sup>™</sup> All-Ceramic System utilizes CAD/CAM technology to produce a densely sintered and high-strength zirconia framework with 3 mol% yttria partially stabilized zirconia

polycrystal content. The dental laboratory includes a special scanner (Lava scan computerized milling machine (CAM) (Lava Form), and a sintering oven (Lava Therm) plus CAD/CAM software technology. Tooth preparations and any edentulous areas are scanned by a contact- free optical process that uses white light triangulation. The entire scanning process takes approximately 5 minutes for a crown preparation and 12 minutes for 3units FPD (46). After scanning, the crown or bridge framework is designed on the computer and subsequently milled from a green blank (partially sintered zirconia block). This green blank is much softer than a sintered zirconia, allows reduced milling times, minimal tool wear, and demands less load from the milling unit (47). Additionally, in order to compensate for shrinkage during the sintering process (20-25%), the CAM produces an enlarged framework structure. The average milling time for crown coping is approximately 35 minutes for a crown preparation and 75-90 minutes for a 3-unit FPD substructure (46, 47). After milling, the framework can be colored in 7 shades by the dental technician before sintering. Sintering is accomplished using the special automated oven, which is programmed to run for 8 hours, including heating and cooling phases. Yttrium stabilized zirconia is used for the Lava framework because of its high strength (47) and was reported to have a flexural strength exceeding 1000 MPa (48).

### 1.4 Zirconium

(Zr) is a metal with the atomic number 40. It was first discovered in 1789 by the German chemist Martin Klaproth. The material has a density of 6.49g/cm<sup>3</sup>, a melting point of 1852°C and a boiling point of 3580°C. It has a hexagonal crystal structure and a grayish color. Zirconium does not occur in nature in a pure state. It can be found in conjunction with silicate oxide with the mineral name Zircon (ZrO2 x SiO2) or as a free oxide (Zirconia, ZrO2) with the mineral name Baddeleyite (49). These minerals cannot be used as primary materials in dentistry because of impurities of different metal elements that color their mass and natural radio nuclides like urania and thoria, which make them radioactive. In order to produce pure zirconia powders, complex and time-consuming processes that result in an effective separation of such elements are used. The material can be used after purifying as a ceramic biomaterial (49).

#### 1.4.1 Structure of Zirconia

Pure Zirconia (ZrO2) has a high melting point (2680°C) and low thermal conductivity. However, its polymorphism restricts its widespread use in the ceramics industry. ZrO2 occurs in three crystallographic forms: monoclinic (M), tetragonal (T) and cubic (C). During a heating process, zirconia undergoes a phase transformation process (49). The monoclinic form is stable at room temperature and up to 1170°C. Above this temperature, it transforms into the denser tetragonal phase with a 5% volume decrease and the creation of cracks within its structure (50). The tetragonal form is stable between 1170 and 2370°C. At temperatures higher than 2370°C, ZrO2 acquire cubic crystal structure. Reversely, during cooling a T-M transformation.

mation takes place in a temperature range of about 100°C below 1070°C, with a volume expansion of approximately 3-4% and the generation of stresses that originate cracks in ZrO2-ceramics (49). The stresses induced during these phase transformations result in crack formation (50).

#### 1.4.2 Advantages of Zirconia

Advanced ceramic materials such as zirconia have great potential as substitutes for traditional materials in many biomedical applications. Since the end of the 1990s, the form of partially stabilized zirconia has been promoted as suitable for dental use due to its excellent strength and superior fracture resistance as result of an inherent transformation toughening mechanism. In addition, zirconia bio ceramic presents enhanced biocompatibility, low radioactivity, and interesting optical properties. The introduction of computer-aided design/computer-aided manufacturing (CAD/CAM) techniques has increased the general acceptance of zirconia in dentistry. However, some fabrication procedures such as grinding, polishing, sandblasting, heat treatment, and veneering of the fine-grained metastable zirconia microstructures may affect the long-term stability and success of the material by influencing its aging sensitivity. Compared to metals, dental ceramics show better biocompatibility and improved esthetics (51). These favorable properties are due to the capability of transmission of light through the ceramic mass. In addition, dental ceramics exhibit diminished plaque accumulation, low thermal conductivity, resistance to corrosion and color stability. On the other hand, insufficient mechanical stability and strength caused by brittleness and low tensile strength of these materials limit their range of indication (51). In the 1990s, only small FPDs made of glass-infiltrated alumina porcelain were recommended for the replacement of a single missing tooth in the anterior area (52). Today, a few all-ceramic materials, like glass-infiltrated alumina (In Ceram Alumina, In Ceram Zirconia), glass ceramics (Empress2) high-strength ceramics (e.g. zirconium- and high-aluminum oxide-based ceramics), can be used for the fabrication of all-ceramic FPDs with appropriate resistance (52, 53, 54, 55, 56). The mechanical properties of high-strength ceramics make them appropriate as potential core materials for all-ceramic restorations in high stress-bearing areas (57).

#### 1.4.3 Manufacturing of Zirconia:

Today, zirconia is being manufactured under optimized industrial conditions and can be designed for its processing by computer-aided design/manufacturing (CAD/CAM) technologies (57). High quality allceramic restorations can be obtained. On the other hand, the assessment of its physical and chemical properties, the long-term behavior of zirconiabased restorations and the fabrication techniques are essential before recommending for daily practice. Loss of teeth can affect a person's appearance and functions such as eating and speaking. There is thus a need for prosthetic rehabilitation to improve quality of life. For many patients, a fixed dental restoration is preferred, and a common restoration is a porcelain-fused-to-metal bridge retained by teeth or implants. Metal-based restorations can potentially cause adverse reactions though, and this is cause for the search for alternative materials. All-ceramic materials are characterized by strong atomic bonds that make them reluctant to react with the environment, and thus unlikely to cause adverse reactions. All-ceramic materials have other attractive material properties and excellent aesthetic properties and have been successfully used in dentistry, mostly for smaller anterior restorations. Ceramics, however, do not withstand tensile forces as well as metals, and are susceptible to brittle fractures with the connector area being especially prone to fracture. More recently, a new type of ceramic material, based on zirconium dioxide, has been developed. Yttria-stabilized tetragonal zirconia polycrystal, Y-TZP, has a unique ability to resist crack propagation by being able to transform from one crystalline phase to another, and the resultant volume increase stops the crack and prevents it from propagating. This material has the potential to be used for larger restorations and in the molar area.

### 1.4.4 Dental application of Zirconia:

In 1968, MacCulloch was the first to use glass ceramics in dentistry.

The interest in esthetic all-ceramic restorations were renewed with the introduction of a castable glass ceramic (Dicor, Dentsply/York Division, York, Penn., USA). The material contained tetracyclic fluormica crystals that increased strength and resistance to crack propagation. Despite the enhanced mechanical properties, the material was not strong enough for the fabrication of posterior all-ceramic FPDs (58). In the mid-1970s, special shoulder porcelain masses were developed and applied in collarless metalceramic restorations to overcome the esthetic problems of metal-ceramic restorations (59, 60, 61). Forming metal frameworks by electroforming pure gold was another technique to keep the FPD framework as thin as possible, providing natural warm color beneath ceramics (62). In 1982, McLean introduced the platinum foil-reinforced alumina FPD, in order to reduce the frequent problem of fracture at the connector area. Oxidation of the tin coating provided a mechanism for bonding of porcelain and the traditional cast-metal framework was eliminated. Its application was recom-

mended only for the replacement of single anterior teeth (63). Because of the high failure rate at the connector sites, this restorative option was not feasible for the fabrication of bridges and was limited to the fabrication of jacket crowns (64). In the past two decades, several approaches have been suggested to enhance the strength of ceramics. These approaches have generated toughened ceramics with microstructure that substantially differs from that of conventional feldspathic porcelains. Their common feature is a considerable crystalline phase in the glassy matrix that contributes to their physical, mechanical and optical properties. Particle size and distribution, nature and amount of the crystalline phase affect the fracture behavior of these ceramics. In addition, mismatches in thermal expansion coefficients among various phases can cause localized stresses at phase boundaries improving the overall toughness (65). In 1989, In-Ceram Alumina glassinfiltrated ceramics (Vita Zahnfabrik, Bad Sackingen, Germany) were introduced. The material has a 70% crystalline content in its mass. This made it possible to fabricate frameworks for three-unit anterior FPDs (66). The IPS-Empress® I (Ivoclar-Vivadent, Schaan, Liechtenstein), which utilizes the principle of leucite crystal dispersion, was marketed in 1991. The later developed IPSEmpress 2 (Ivoclar-Vivadent, Schaan, Liechtenstein), a lithium disilicate glass ceramic with 66% crystalline content, showed a flexural strength 3 times greater than that of Empress I. With this material, it is possible to fabricate small all-ceramic FPDs that can replace a single missing tooth anterior to the second premolar (67). Further efforts to enhance the strength of ceramic cores were made by adding leucite (Optec HSP, Jeneric/Pentron, Wallingford, USA), aluminum oxide (Hi-Ceram, Vita Zahnfabrik), or zirconium dioxide crystals (Mirage II, Mirage Dental Systems, Chameleon Dental) to conventional feldspathic porcelains. However,

the resultant ceramic did not meet the requirements for the fabrication of FPDs (57).

# 1.5 physical properties of zirconia based dental ceramics:

#### 1.5.1 Transformation-toughening mechanism:

In the presence of a small amount of stabilizing oxides, it is possible to obtain PSZ ceramics at room temperature with a tetragonal phase only, hence called Tetragonal Zirconia Polycrystal (TZP). The finely dispersed tetragonal ZrO2 grains within the cubic matrix, provided that they are small enough, can be maintained in a metastable state that is able to transform into the monoclinic phase (50). This phenomenon can be explained through the lower surface energy of the tetragonal ZrO2 particles and the constraint of the rigid matrix on them that opposes their transformation to the less dense monoclinic form. This process gives rise to a powerful crackinhibiting and strengthening mechanism- the transformation-toughening mechanism (68). The tetragonal ZrO2-grains can transform into the monoclinic phase when the constraint exerted on them by the matrix is relieved, i.e. by a crack advancing in the material (49). At the edge of the crack, the compressive stress field, this is associated with a volume expansion of 3-5% of the transformed tetragonal grains, acts in opposition to the tensile stress field that promotes the propagation of the crack (Figure 1.1). The fracture energy is dissipated in the T-M transformation, characterized as a marten site-like transformation that occurs in quenched steel, as well as in the process of overcoming the compression stress of matrix due to the volume expansion. The progression of the crack is inhibited and the toughness of ZrO2-ceramics is enhanced (49).



Figure 1.1 Schematic drawing of the stress-induced transformation-toughening mechanism in TZP (Source: Piconi and Maccauro, 1999).

The addition of approximately 2-3% mol yttria (Y2O3) as a stabilizing agent in zirconia allows the sintering of fully tetragonal fine-grained zirconia ceramic materials made of 100% small metastable tetragonal grains and called Y-TZP (Yttrium-Tetragonal Zirconia Polycrystals) (50). The fraction of the T-phase retained at room temperature is dependent on the processing temperature, the yttrium content, the size of grains and the grade of constraint exerted on them by the matrix (Figure 1.2). The mechanical properties of TZP ceramics depend also on these parameters (49). Addition of Y2O3 in higher concentrations produces a fully stabilized zirconia ceramic with a cubic phase only and lower fracture strength (69).



Figure 1.2 Phase relationship in the zirconia-yttria systems according to Composition and processing temperatures (m:monoclinic, t:tetragonal, c:cubic), (Source: Piconi and Maccauro, 1999).

# 1.5.2 Biocompatibility of Y-TZP:

A small percentage of the population is hypersensitive to dental alloys containing noble and base metals, such as palladium and nickel (79). In vitro and in vivo studies have confirmed the high biocompatibility of Y-TZP when high purity zirconia-powders are used. These powders are purified from their radioactive content. As a result, no local (cellular) or systemic adverse reactions to the material have been reported (49, 50, 71, 72, 73). Recent studies have demonstrated that fewer bacteria accumulate around Y-TZP than titanium (74, 75). These findings have lead to the suggestion that zirconium oxide may be a suitable material for manufacturing implant abutments with a low colonization potential (75). Aging of Y-TZP The long-term stability of ceramics depends on the subcritical crack growth and the stress corrosion caused by water. In the mouth, saliva reacts with glass, resulting in the decomposition of the glass structure and an increase of crack propagation. Glass-free, polycrystalline microstructures, as Y-TZP, do not exhibit the latter phenomenon but, unfortunately, ZrO2-ceramics are prone to age in the wet environment, showing degradation of their mechanical properties. The mechanical performance of ZrO2 as a function of time is of particular concern in YTZP for biomedical applications. Low temperature degradation (LTD) of zirconia, known as aging, happens because of the progressive spontaneous transformation of the tetragonal phase into monoclinic and results in the degradation of the mechanical properties of the material (49). It is well established that when YTZP is in contact with water or vapor (69), body fluid, or during steam sterilization (49), a slow T-M transformation occurs, which leads to surface damage. In addition, nonaqueous solvents with a chemical structure similar to water can also destabilize Y-TZP, causing strength degradation (69, 76).

#### 1.5.3 Fracture resistance after fatigue:

All ceramic crown and bridge restorations are subjected on a daily basis to masticatory loading which places the restoration under repeated loading throughout its service-life. Repetitive stresses during the chewing cycle may lead to fatigue of the material and eventually fractures when they are exposed to the oral environment (77). Previous studies showed that ceramic containing glass such as ProCAD, In- Ceram, and IPS Empress had a decrease in fracture strength after cyclic loading (77, 78, 79). Chen et al (1999) reported a significant decrease in fracture strength and considerable increase in failure probability of ceramic crowns (Vita Mark II, ProCAD and IPS Empress I, II) when subjected to the cyclic loading at 50,000 cycles using a force of 200 N. Similarly, the fracture strength of In-Ceram and IPS Empress was significantly reduced after 10,000 cyclic using 300 N

forces (79). One study reported that fatigue (20,000 cycles) slightly reduced the flexural strength of glass infiltrated zirconia (In-Ceram Zirconia) and high purity alumina (Procera All Ceram) but it was not statistically significant (43). On the other hand, the reduction of the fracture strength of high purity zirconia ceramic after cyclic loading has not been reported. A recent study showed that when using high forces of 500, 700 and 800 N at low numbers of cycles (2,000 cycles) and also low force of 80 N at 10,000 and 100,000 cycles, there was no significant difference in the mean biaxial flexural strength between unloaded and zirconia discs that had undergone cyclic loading in both dry and wet conditions (80).

#### 1.5.4 Industrial procedures for the fabrication of zirconia blanks

Raw materials are selected and processed in the form of powders. With cold isostatic pressing, the powders are shaped in ceramic pre-forms. Cold isostatic pressing is the most accepted procedure as a shaping technique of Y-TZP. In this way, stable, chalk-like green-stage objects with a very high primary density are produced. The green objects are further stabilized and condensed up to about 95% of the theoretical density (50) through sintering without pressure in the oxidized atmosphere of a special furnace (57), forming pre-sintered oxide-ceramic blanks. Additional compression can be achieved with Hot Isostatic Post compaction (HIP) that takes place at 1000bar and 50°C below the sintering temperature (57). With this procedure, residual porosity is removed and densely sintered oxide-ceramic blanks are produced. "HIP-ping" of Y-TZP results in a grey-black material that usually requires subsequent heat treatment to oxidize and restore the whiteness of the material (81). Complete densification occurs with only

limited grain growth, resulting in improved strength (50). These densely sintered blanks are highly opaque, which makes it necessary to veneer them for a better esthetic result (57). Hot isostatic-pressed Y-TZP samples have been found to show slower T-M transformation than hot-pressed bar samples (unHIPped) (82).

#### 1.5.5 Zirconia as a framework material

Zirconium dioxide ceramics are being used in dentistry as framework materials for the fabrication of posterior FPDs. These frameworks can be fabricated mainly with the help of a CAD/CAM system by means of milling of a ZrO2-block. These blocks can be milled either in the green stage, the presintered stage or the completely sintered stage. Green stage ZrO2-blocks can be milled using dry carbide burs, pre-sintered ZrO2-blocks can be milled using carbide burs under cooling liquid, and milling of completely sintered ZrO2- blocks requires the use of diamonds under cooling liquid (83). Frameworks made from green and pre-sintered ZrO2 are milled in an enlarged form to compensate for the shrinkage that occurs during sintering, which usually equals 20-25% for partially sintered frameworks (56). The milling process is faster and the wear and tear of hardware is less than when milling from a fully sintered blank. The frameworks are subsequently post-sintered in special furnaces (about 1500°C) to reach the fully sintered stage. In addition, the color of ZrO2 can be individualized through adding some oxides to the green-stage framework (e.g. Vita shade concept, Vita Zahnfabrik, Bad Sackingen, Germany) (83). Compared to the previous method, milling of fully sintered ZrO2-blocks is a time consuming process that causes greater wear of the diamond burs and is more expensive. Hence,
green-stage ZrO2 should be considered advantageous. Completely sintered zirconia has usually a 5% yttria-content (i.e. TZP Bio HIP material, Metoxit, Thayngen, Switzerland) (57). Apart from CAD/CAM technology (See also 2.4 C. Hardware), new innovative techniques have been presented for the production of ZrO2-frameworks.

## 1.5.6 Subcritical crack growth resistance:

Brittle materials are susceptible to time-dependent failure under static loads, caused by the subcritical growth of cracks to critical lengths (84). The subcritical crack growth refers to environmentally enhanced crack propagation at subcritical stress levels. The propagation of the pre-existing natural defects occurs at low rates (slow crack growth), and causes delayed failure of ceramics when the flaw size reaches a critical value (85). The subcritical parameter A and n are usually examined for estimating crack growth resistance and lifetime of materials. Where A is a constant in metre per second and n is an exponent, which in most ceramics has a value >10 (84). The n value can vary from 20-100 (86, 87, 88) dependent on the test methods. Previous studies reported that two common methods, double torsion and flexural tests, show a difference in subcritical n values of zirconia ceramics (86, 89). Li and Pabst (1980) reported that the n value of asreceived ZrO2 specimens tested using the double torsion test (n=80) was higher than the flexural test (n=51).

# 1.6 Influence of Surface treatment on Zirconia

# 1.6.1 Effect of Sandblasting & grinding on strength of Zirconia

A requirement for the successful function of low strength ceramic restorations over the years is an adequate adhesion between ceramic and tooth substance. To achieve the best fit between the prosthetic work and the prepared tooth structure, a final adjustment by dental grinding may be required. Sandblasting is frequently used to improve the bond between the luting agent and the prosthetic material. Several researchers have studied the effect of surface treatments such as grinding sandblasting, and polishing on the strength and microstructure of zirconia (68, 90, 91, 92, 93). Garvie et al (1975) first reported that grinding increased the strength of zirconia compared with a polishing process. This difference was associated with an increase in the monoclinic content at the surface on grinding while polishing removed some of the monoclinic material and this reduced the surface strain. A similar reduction of the strength can be achieved by annealing, which removes the strain without reducing the surface content of monoclinic phase (68). Denry and Holloway found grinding with a 20 micron diamond bur increased the biaxial flexural strength of zirconia because a rhombohedral phase and strained tetragonal phase was formed, which can increase resistance to crack propagation but may be associated with surface and subsurface damage (93). Kondoh (2004) suggest that the shoulder or hump observed in XRD peak, Which others indicated it was rhombohedral phase, was caused by lattice distortion of tetragonal phase Kosmac et al (1999) reported that grinding using a coarse grit (150  $\mu$ m) diamond bur at high rotation speed lowered the mean strength and reliability whereas

sandblasting improved the mean strength at the expense of lower reliability. Therefore, grinding has the ability to introduce compressive stress and enhance the transformation toughening of the zirconia surface with a resultant increase in strength (68). However, coarse grinding or severe grinding introduces deep surface flaws which can lower the strength and reliability of zirconia (48, 92). On the other hand, sandblasting is considered to be a more gentle process and less material is removed from the surface. Some studies found that sandblasting increased the strength of Y-TZP ceramics (91, 92). They suggested that sandblasting is an efficient technique for strengthening zirconia based ceramic in clinical practice. However, Curtis et al. (2005) found alumina abrasion regimes did not significantly alter the mean biaxial flexural strength of zirconia specimens.

#### 1.6.2 Manufacturing and sintering process

Manufacturing and sintering processes have a major effect on the properties of zirconia ceramic. The sintering temperature and the duration of the sintering process have been reported to affect grain size and phase content, which influence the strength of zirconia (94, 95). Ruiz and Ready proposed that the grain size increased with increasing sintering temperature, which led to an increase in fracture toughness, owing to larger transformation zones. However, no significant difference in biaxial flexural strength of various grain size zirconia ceramics was reported in this study. This contrasts with the results of Casellas et al (2001) who found that a decrease in grain size gave a slight increase in flexural strength. This is attributed to greater phase transformation around the crack in coarser microstructures than smaller grain materials. The factors caused by the manufacturing and sintering processes affecting the property of zirconia are flaws or defects and crack initiation, which can lead to early restoration failures (15). The strength of ceramic specimens and prostheses depend on the size of microscopic cracks and pores. One article (96) reported that the presence of numerous surface flaws, including submicroscopic Griffith flaws can lead to failure. This flaw can act as stress concentrators when the object is under load, with microscopic stress occurring at the flaw tip and causing fracture as soon as a critical breaking stress is reached (96). Crack initiation may be influenced by the quality of the ceramic surface and the internal structure of ceramic materials, which can provide resistance to crack growth (15). The strength of material is dependent on the size of the pre-existing initiating cracks present in a particular sample or component (97). In addition, a large number of cracks together with a low fracture toughness of materials will limit the strength of ceramics and cause a large variability in strength (97).

# 1.6.3 Aging of Y-TZP:

The long-term stability of ceramics depends on the subcritical crack growth and the stress corrosion caused by water. In the mouth, saliva reacts with glass, resulting in the decomposition of the glass structure and an increase of crack propagation. Glass-free, polycrystalline microstructures, as Y-TZP, do not exhibit the latter phenomenon<sup>-</sup> but, unfortunately, ZrO2ceramics are prone to age in the wet environment, showing degradation of their mechanical properties. The mechanical performance of ZrO2 as a function of time is of particular concern in YTZP for biomedical applications. Low temperature degradation (LTD) of zirconia, known as aging, happens because of the progressive spontaneous transformation of the tetragonal phase into monoclinic and results in the degradation of the mechanical properties of the material (49). It is well established that when YTZP is in contact with water or vapor (69), body fluid, or during steam sterilization (49), a slow T-M transformation occurs, which leads to surface damage. In addition, non-aqueous solvents with a chemical structure similar to water can also destabilize Y-TZP, causing strength degradation (69, 76).

### 1.6.4 Polishing:

The process of polishing develops scratches that induce residual stresses in the material. The influence of polishing on the aging sensitivity of zirconia is contradictory and relates to the type and amount of these stresses. Rough polishing produces a compressive surface stress layer beneficial for the aging resistance, while smooth polishing produces preferential transformation nucleation around scratches, due to tensile residual stresses caused by elastic/plastic damage (98). Fine polishing after grinding may remove the compressive layer of monoclinic phase from the surface, while further polishing may minimize the size of flaws and result in greater flexural strength (90).

# 1.7 Manufacturing of Zirconia:

CAD/CAM (Computer-Aided Design/Computer-Aided Manufacturing) designates the three-dimensional planning and design of a work piece on the screen of a computer with subsequent automated production by a computer-controlled machine tool (99). This technology enables the milling of ceramic materials in room temperature, under technical processes that yield

homogenous material structures, where voids, flaws and cracks are reduced to a minimum (57). Every specific system involves a certain coded framework material and the compatible veneering material. Regardless of the CAM system and the materials used, the CAD program can also function separately for the design of the restoration (83). Current CAD/CAM systems consist basically of three components:

A. Three-dimensional scanning of the surface: The surface of the model (made from wax or dental stone) is 3-D scanned and a dot matrix of measuring points is generated, providing an image of the original working model. The number of measurement points is no indications of the quality of the dot matrix (100). There are three types of digital 3-D scanning devices for dental use:

I) Mechanical scanner: These systems detect the surface of the prepared tooth on the die, using a sphere (Procera), needle or pin (Precedent DCS until 1997).

II) Intraoral scanner: An image of the prepared tooth and the anatomic structures of the adjacent teeth can be recorded and provide a digital image (Cerec 3D, Vincron; Evolution 4D)

III) Optical scanner: An optical reading of the surface of the die is possible with a white or colored light or with laser-beam projection (100).

B. Software for computer-aided design: The dental framework is designed virtually with the help of 3-D software on the digital image of the prepared teeth. The occlusal and proximal relations of the prepared teeth are not considered when designing a framework for a crown or a FPD (100). Few systems allow the design of the occlusal surface of a complete restoration.

The 3-D volume model is saved in a specific data format, then sent to a production unit (CAM). Most dental CAD/CAM systems operate as closed data systems, which restrict the use of the generated data to the specific CAM device. In a newer group of CAD/CAM systems, the 3-D volume model is transferred from CAD to CAM in a neutral data format, which allows free choice among different producing centers and CAM systems (83).

C. Hardware (CAM): The manufacturing units are located either in the dental laboratory or in a specialized producing center. Most systems are able to produce larger than 4-unit FPDs. According to the technique used, CAM technologies for FPDs can be divided into three groups:

- I) Subtractive technique from a solid block this technique is the most commonly applied. The contour of the framework is cut out of an industrially prefabricated, solid material block using burs, diamonds or diamond disks (101,102). The material block can be in the green stage, partially sintered, or may require further processing (sintering, glass infiltration). The precise fit of the restoration depends on the size of the smallest usable tool for each material.
- II) Additive technique by applying material on a die

In this technique, the powder material is applied directly on the die of the master model. With the Procera system (Nobel Biocare, Goteborg, Sweden), powder material is applied on an enlarged metal die with compaction under pressure. The framework, which is in the green stage, is removed from the die and sintered to correct size (1550°C). The outside contour of the coping is created by a computer-aided milling process, while the powder is on the metal die in the green stage (83). The WOL-CERAM-EPC-CAM-System (Wol-Dent, Ludwigshafen, Germany) uses electrophoresis to produce ZrO2-frameworks. The sintering shrinkage is minimal (0.1%) (103). The outside contour of the frameworks is shaped by a CAM process. The frameworks are removed from the die and sintered during firing. (83,104).Pure aluminum- or ZrO2-frameworks with high density and purity can also be fabricated in a production center (ce.novation, Inocermic, Hermsdorf, Germany) from fine-dispersed nanoceramic powders with a particle size of 100 nm (83,101,103).

III) Solid free form fabrication

In this method, the selective laser sintering technique allows the buildup of ZrO2- frameworks in a powder bed. Heat-fusible powder materials are applied sequentially, or layer by layer, on the spots those are indicated from the CAM-model, and are then selectively sintered by a laser to form the framework. With this system, the frameworks can be designed in the dental laboratory (CAD) and fabricated in a producing center (Bego Medifacturing-System, Bego Medical, Bremen, Germany) (83,103). The technique is still new and needs further development.

# 2. Materials and Methods

#### 2.1 Material

Yttria (3 mol %) partially stabilized zirconia polycrystal (3Y-TZP) ceramics used in this study. The manufactures of zirconia ceramic is zirkonzahn (zirkonzahn SRL City; Italy). 3 different thickness and 10 samples of zirkonzahn ceramic were selected for this study (15x0.3mm), (15x0.5mm), (15 x0.7mm) fig.2.1. Three control discs of the green stage Zirconia block were cut with saw (leitz 1600) and sintered in zirkonzahn sintering oven start at 20- 1500 °C temperature using rise time of 3h and kept at 1500 °C for 2h. All discs were polished with diamond paste. The final sintering temperature was 1500°C for 5 hrs and the cycle of all processes take 8 hrs and the cooling rate 8°C/ min this procedure were Provided by fig 2.1.5 (zirkonzahn Furnace) .The surface of the Zirconia specimens were ground with 600, 800, 1200 (Silver carbide abrasives) under water cooling and then with 120  $\mu$ m Al2O3 particles are blasted to 0.5 MPa pressure from a distance of 10 mm, 15 seconds. The three different thickness (0.3, 0.5, and 0.7) and diameter (15mm) of all sintered discs were measured with digital micrometer (Mitutoyo Crop, JAPAN) fig.2.1.2 before the fracture test. (Mitutoyo Crop, JAPAN) calipers are used to measure thickness of samples. By cleaning them of any debris, you can assure that they can be calibrated correctly. Simply using precision gauge blocks to check their accuracy every day can also let you know when it is time to recalibrate them to assure that your parts are being measured correctly. Clean the Mitutoyo calipers off with acetone or any other cleaning fluid suitable for precision instruments. (121)



Fig. 2.1.1 Super high quality measuring Caliper device.



Fig.2.1.2 Zirkonzahn samples



Fig 2.1.3 Zikonzahn milling machine



Fig.2.1.4 Zirkonzahn Furnace (ZIRKONOFEN 600)

# 2.2 Methods

For the biaxial flexural strength test a universal testing machine (Model 3345, Instron, Canton, Mass, USA). Fig (2.2.1) The manufactures by standardizing zikonzahn discs were (testing machine Model, 3345, Instron, Canton, Mass, USA). The method adapted was the one recommended by the International Standard Organization [123] because the test standardizes specimen thickness, diameter, shape, and roughness. Disc specimens were supported on 3 ball spheres (3.4mm in diameter) equally Spaced on a circle with a diameter of 15.7 mm & center- loaded by a steel piston (with a flat area of 1.4mm in diameter ground along the contact surface) until Fracture occurred at a crosshead speed of o.5mm/min. Fig (2.2.2) The ultimate load that Caused specimen fracture was recorded in Newton (N). Failure stress was calculated using the equation listed in ISO 6872 with Poisson Ratio value of 0.25 for all materials. Ten specimens of each group were subjected to a biaxial flexural strength test; each disc specimen was placed centrally on three hardened steel balls (with the diameter of 3mm, positioned 120° apart on a support circle with a diameter of 11 mm). The load to failure (N) of each specimen was recorded and the biaxial flexural strength (MPa) was calculated using the following equations;

(1)-(3) according to the ISO 6872 standard

$$S = -0.2387 \ \underline{P(X - Y)}{d \ 2}$$
 (1)

Where S is the maximum tensile stress in Pascal's, P the total load causing fracture in Newton's and d is the specimen thickness at fracture origin in millimeters. X and Y were determined as following:

$$X = (1+v)\ln\left(\frac{r_2}{r_3}\right)^2 + \left[\frac{(1-v)}{2}\right]\left(\frac{r_2}{r_3}\right)^2$$
(2)

$$Y = (1 + v) \left[ 1 + \ln \left( \frac{r_2}{r_3} \right)^2 \right] + (1 - v) \left( \frac{r_1}{r_3} \right)^2$$
(3)

In which: v is Poisson's ratio. If the value for the ceramic concerned is not known, a Poisson's ratio = 0.25 is used; r1 the radius of support circle, in millimeters; r2 the radius of loaded area, in millimeters; r3 the radius of specimen, in millimeters; d is the specimen thickness at fracture origin, in millimeters.



Fig. 2.2.1 Universal testing machine (Model,3345,Instron,Canton,Mass,USA).



Fig. 2.2.2 Tip of the universal testing machine in stu.

# 2.3 Statistical evaluation

Bending strength values obtained from the experiment after experiment in our study groups, the mean and standard deviation values were calculated. Parameters in the evaluation of operating data, comparisons between groups one-way analysis of variance (one way ANOVA) test and Tukey HDS test was used for the determination of the group that lead to differences. Pearson correlation analysis was used for assessing the relationships between parameters. Significance p <0.05 level were evaluated. (Number Cruncher test were used to analyze data NCSS. p<0.05 was the significance level. Statistical System) 2007&PASS 2008 Statistical Software (Utah, USA) program was used.

# **3. Results**

For all specimens, maximum load at failure was measured with biaxial flexural strength test (piston-on-three balls). The mean biaxial flexural strengths of ten discs are presented in all groups in table (3.1). The highest mean biaxial flexural strength found at 0.7mm (338.Mpa), than at 0.5 mm (337.Mpa) and lowest value was found at 0.3mm (315.Mpa Graph. (3.1)

0.3 mm		0.5 mm		0.7 mm	
N	Мра	N	Мра	N	Мра
49.11	324	127.2	302	259.2	314
55.82	368	144. 5	343	299.5	363
43.65	288	145.7	346	268.6	325
45.99	303	160.2	380	248.0	300
46.9	309	133.5	317	292.8	355
48.41	319	140.7	334	271.4	329
49.5	326	140.0	332	285.4	346
49.88	329	155.7	369	270.5	327
45.93	303	116	275	328.1	397
43.41	286	156.1	371	267	323
MEAN	316		337		338

Table 3.1 The load at the point of fracture.



Graph 3.1 The relation between thickness of zirconia & fracture strength (Mpa).

Thicness of zirkonzahn	Mean	S.d ±	Max. load	Min. load
0.3 mm	316	15,6	368	286
0.5 mm	337	16,2	380	275
0.7 mm	338	21,3	397	205

Table 3.2 Three point bending test (Mpa)

There is a small difference in the values of biaxial flexural strength between 0.3, 0.5 mm &0.7mm Which revealed no significant difference in the three different thickness of zirconia groups (table 3.3). A cording to our results; increasing the core thickness of Zirconia from 0.3-to 0.5mm flexural strength was increased 7%. But core thickness did not differ between 0.5mm and 0.7mm.

	Table1: Fracture strength evaluation	
	Fracture strength (Mpa)	<i>F;</i> + <i>p</i>
Thickness	Mean±Sd	
0.3 mm	316±15,53	F:2,520; p:0,099
0.5 mm	337±21,28	
0.7 mm	338±16,17	
0.3 mm/0.5 mm ++p	0,205	
0.3 mm/0.7 mm ++p	0,111	
0.5 mm/0.7 mm ++p	0,939	

<sup>+</sup> One way ANOVA Test

<sup>++</sup> Tukey HSD test

Table 3.3.Statistical result of the tests



Graph 3.2 fracture strength of zirconia (Mpa)

There was no statistically significant differences between the fracture strength values of the test groups (P>.05).

# 4. Discussion:

### 4.1 Flexural strength

Usually, complex stress distributions that are induced by compressive, tensile and shear stresses are present in most specimens. However, brittle materials are much weaker in tension than in compression (105). Therefore, tensile strength is generally considered as the more meaningful property for brittle materials for assessment of the failure potential of dental restorations (105). Flexural strength is a fracture-related mechanical property since it is a measure of the resistance of restorations to tensile forces. Materials with high flexural strength provide restorations with less susceptibility to bulk fracture (106). Different methods are available to assess the flexural strength of ceramic materials. These test methods included three-point bending, four-point bending and biaxial flexural tests

# 4.1.1 Uni-axial strength (three-point or four-point flexure test)

Three-point and four-point flexure tests have also been used for strength evaluation of brittle materials and metal-ceramic structures (105). For these uni-axial flexure tests, the principle stress on the lower surfaces of the specimens is tensile, and it is usually responsible for crack initiation in brittle materials.

#### 4.1.2 Four point bending

The four-point flexure test is a method of assessing mechanical properties of materials. It has been used for strength evaluation of single component brittle materials and bilayered structures such as glass veneer on core ceramic specimens and metal-ceramic structures (97). For the four-point flexure test, rectangular specimens are supported by two 3-mm-diameter rods set 21 mm apart. The load is applied by two rods that are set 7 mm apart. A cross-head loading rate of 0.2 mm/min is used. The maximum tensile stress is calculated by the equation  $\sigma=PL/wt2$  2.1 Where is *P* is the applied force at failure, *L* is the length of outer span, *w* is the width of the specimen, and *t* is the thickness of the specimen. Ban and Anusavice (1990) studied flexural strength of zinc phosphate cement for orthodontics, feldspathic opaque porcelain, feldspathic body porcelain and light cured composite resin using biaxial flexural test compared with four-point bending test. The results showed that groups submitted to four-point bending test exhibited lower strength values than biaxial flexural test.

#### 4.1.3 Three point bending

The three-point flexural test is one of the standards for strength testing of dental ceramics Shetty *et al* (107) studied the flexural strength of glass-ceramic comparing among 4-point, 3-point bending and biaxial flexural test (ball-on-ring test). They found mean strength values from 4-point and 3-point bending were similar whereas ball-on-ring tests exhibited higher mean strength than uni-axial tests. Although the three-point and four-point flexural tests are one of the standard tests of dental materials, the strength results may be affected by edge flaws and defect (16,105,108). Conse-

quently, the strength value obtained from these methods is lower than the actual strength of the materials (108). This problem is overcome by the use of biaxial flexural strength test because it is not affected by edge flaws and defects (16,109,110) as they are not directly loaded (110). Therefore, the biaxial test should produce less variation in data for strength determination compared with uni-axial tests (109). This study was undertaken to demonstrate the possible change in strength of different thickness of zirconia discs after sintering procedure .This is of interest because questions have arisen whether the properties of some green stage milled zirconia can reach higher strength by changing the thickness of Zikonzahn discs. The biaxial flexural tests were performed first. Flexural strength is the resistance of a specimen to a flexural load at the moment of fracture. Biaxial flexural testing is recognized as a reliable technique and method of choice (ISO 6872) (1) for studying brittle materials since the maximum tensile stress occurs within the central loading area and edge failures are eliminated. (105) In this study, there were no significant differences (P > .05) between the biaxial flexural strengths of all of the groups. In addition, the obtained biaxial flexural strength values of the experimental group samples met the requirements of ISO standard 6872, which recommends a minimum flexural strength of 100 MPa for this type of ceramic restorative material. Additionally, the experimental group samples possessed biaxial flexural strengths that are comparable to other all-ceramic materials. However, further experiments need to be conducted to determine if the observed flexural strengths of the experimental ceramics are sufficient for clinical use. Zirconia ceramic was extended into dentistry in the early 1990s as endodontic posts (111) and more recently as implant abutments (112,113) and hard framework cores for crowns and fixed partial dentures (114,115,116). Zirconia has unique a characteristic called transformation toughening, which can give it higher

strength and toughness compared with other ceramics. The major issue concerning zirconia ceramics is their sensitivity to low temperature degradation (LTD)(117) Ageing occurs by a slow surface transformation from metastable tetragonal phase to a more stable monoclinic phase in a humid environment such as humid air, water vapor and aqueous fluids at a relatively low-temperature (118) ranging from 65-500°C (69). The tetragonal to monoclinic transformation can be of benefit due to the compressive layer on the surface of the ceramic, which improves its properties. Biocompatibility of zirconia has been studied both in vitro and in vivo, but most of the biocompatibility studies concentrated on femoral ball head implantation. In vitro tests used cell cultures with cells such as fibroblasts, blood cells (49) and osteoblast cells .In vitro tests using cell cultures were performed on ceramic materials indifferent physical forms such as powders and dense ceramics (49). Some of the *in vivo* tests investigated implanting zirconia ceramics into bone and soft tissue. Scarano et al (2003) reported high biocompatibility and osteoconductivity of zirconia implant. Therefore, it can be concluded that there appears to be no cytotoxicity of zirconia ceramic when tested both in vitro and in vivo.

#### 4.2. Factor effect of flexural strength

4.2.1 Sintering temperature:

The sintering temperature and the duration of the sintering process have been reported to affect grain size and phase content, which influence the strength of zirconia (94, 95). Ruiz and Ready proposed that the grain size increased with increasing sintering temperature, which led to an increase in fracture toughness, owing to larger transformation zones. However, no significant difference in biaxial flexural strength of various grain size zirconia ceramics was reported in this study. This contrasts with the results of Casellas et al (2001) who found that a decrease in grain size gave a slight increase in flexural strength. This is attributed to greater phase transformation around the crack in coarser microstructures than smaller grain materials. In addition, All-ceramic crowns and fixed prostheses have had many false starts across the past several decades. Most dentists have frustrating memories of placing beautiful all-ceramic restorations, only to have them fracture after a few months or years of service. An obvious exception to this phenomenon is the success of pressed ceramic restorations for the anterior portion of the mouth. Millions of beautiful crowns and fixed prostheses using pressed ceramic have served successfully for many years, primarily in the anterior portion of the mouth. However, dentists long have sought stronger all-ceramic restorations for crowns and fixed prostheses in both the anterior and the posterior portions of the mouth.

In the past several years, numerous brands of zirconia-based all-ceramic crowns and fixed prostheses have been introduced to dentistry. Some popular brands in the United States are Cercon (Dentsply, Milford, Conn.), Everest (KaVo, Lake Zurich, Ill.), IPS e.max (Ivoclar Vivadent, Amherst, N.Y.) and Lava (3M ESPE, St. Paul, Minn.). These materials are having a significant effect on the fixed Prosthodontics laboratory industry, as well as on practitioners and their patients. Many clinical reports and research articles have been published on use of zirconium oxide milled by computer-aided design/computer-aided manufacture procedures, sintered and used to create substructures for ceramic that subsequently is layered or pressed on the external surfaces. (146,147) When restoring a single maxillary central

incisor and attempting to achieve an optimum esthetic result, most dentists prefer to make the restoration from all-ceramic materials. Most dentists would agree that all-ceramic crowns and fixed prostheses usually have a better appearance than do PFM restorations.

# 4.2.2 Firing times and ceramic thickness:

A- A study found that there were significant color difference varies with respect to the firing times and dentin ceramic thicknesses. Most all ceramic systems consist of a ceramic core with a thickness of 0.5 to 1.0 mm and approximately 1.0 to 1.5 mm of space available for veneering ceramic. (125) In the current study, the specimens had ceramic thicknesses of 0.5, 1, or 1.5 mm, with a core thickness of 1 mm. L\* values, which reflect the brightness of the specimens, decreased for both systems as the total thickness of the specimens increased.

**B**- Antonson and Anusavice (130) studied the effect of change in the thickness of ceramics on the contrast ratio of dental core and veneering ceramic, and concluded that the contrast ratio was dependent on the type of the material tested.

C- Heffernan *et al.* (122,128) described the influence of core material thickness on its translucency and the influence of core plus ceramic veneer thickness on the overall translucency of specimens. Shokry. (128) demonstrated that L\* values decreased for leucite reinforced and spinell ceramics as the total thickness increased. The results of the present study are in agreement with the previous studies (124,128,129,131,132) since the thickness of the layered ceramic influenced the final shade, partially due to the

translucency, as the thicker ceramic disks were less translucent. (131) An increase in the number of firings resulted in an appreciable increase in L\* values that resulted in darker specimens for 0.5 mm and 1.5 mm thicknesses used in the present study. However it decreased for 1 mm thickness specimens. (133,134) In the current study, mean color differences caused by various dentin thicknesses and repeated firings were below 3.7  $\Delta E$  units, which is rated as a match in the oral environment. (126,127) Ceramic systems in the present study exhibited visual color changes during firing and demonstrated that changes in the thickness and repeated firings of ceramic have an effect on the final shade.

One recent study reported that, since 1998, (155) studies have demonstrated a 90% or greater success rate using zirconia-based prostheses.(152,153) zirconia with a 0.5-mm layer thickness served as a framework for primarily posterior 3 - to 5- unit fixed partial dentures. Failures were attributed to cracking or crazing of the veneering porcelain, but bulk fractures were uncommon.(152) The clinical success of zirconia based posterior prostheses reaffirms the structural potential and processing possibilities of this material.(152)The superior mechanical properties of zirconia allow clinicians to reconsider established preparation guidelines for the design of single anterior teeth copings, and consider variations such as reducing the coping thickness from 0.5 mm to 0.3 mm and changing finish line preparations from chamfer (CHA) to minimally invasive knife edge (KNE) margins. In vitro research evaluating the influence of processing variables on fracture resistance of all-ceramic restorations has revealed highly divergent failure loads of 450 to 1600 N for zirconia single crown copings, depending on coping thickness, marginal design, and applied luting agent.(154,156) In general, higher fracture loads can be produced by increasing the coping thickness or by using adhesive luting materials instead of retentive cementation.(149,150,157) However, conventional cementation is recommended by the manufacturers.(157) Improved resin adhesion has been reported when using tribochemical silica coating and silanization of the zirconia frameworks, or when using phosphate-modified resin luting agents.

Fracture resistance evidence can also be provided by microscopic fracture analysis. Fractography, in this context, represents an analytical tool for assessing fractured surfaces for the purpose of locating fracture origins and elucidating fracture patterns. (158,159) Reasons for fracture can be assessed from different crack modes such as cone, radial, or fatigue crack pathways.(160,161) One minor influence on fracture resistance is the design of the finish line.(162) However, marginal integrity and the degree of seating of a restoration is dependent upon the finish line of the preparation.

The configuration of the gingival margins dictates the shape and bulk of the restorative material. The preferred gingival finish line for metal ceramic restorations is the CHA preparation. (148) For all-ceramic bilayer restorations, the CHA and shoulder preparation are recommended.(148,151) These finish line configurations are reported to transfer a minimum of masticatory stress from the coping into the veneering porcelain, which in turn might help to reduce clinical failures due to crazing, cracking, or chipping of the veneering porcelain. (148,162,163) In addition to vertical loading, the induced shear stress could account for the reduced fracture performance of CHA 0.5 and CHA 0.3 preparations Bindl et al(155) observed extreme differences between the fracture initiation (697 N) and catastrophic fracture forces(1607 N) due to increased toughness and extensive crack propagation of the cemented copings. Crack propagation within a coping is accelerated by high shear stresses under vertical loading, reducing the catastrophic fracture forces.

# 4.2.3 Sandblasting and Grinding:

The results of the present investigation revealed that surface grinding and sandblasting exhibit a counteracting effect on the strength of yttria stabilized tetragonal zirconia (Y-TZP) ceramic. Surface grinding using a coarse grit (150 mm) diamond burr at a high rotation speed lowered the mean strength and reliability, whereas sandblasting improved the mean strength, at the expense of lower reliability. During grinding, tens of microns of material were removed by a single pass as the burr was moved back and forth across the surface, and sparks were commonly observed, indicating that both stresses and temperatures were high during this operation. Nevertheless, the amount of transformed zirconia on ground surfaces was low as was the calculated depth of the surface compressive layer contributing to the strengthening of the material. Previous work has shown that severe machine grinding is less effective in initiating the  $t \rightarrow m$  transformation in TZP materials (135,136). In addition, Swain and Hannink (137) reported that hand ground Ce-TZP surface contained about five times more monoclinic zirconia than severely machine ground surface of the same material. This, they argued, is because extensive heat is generated during severe machine grinding in spite of a stream of coolant that was directed near the cutting edge during grinding. As a result, locally developed temperatures exceeded the  $m \rightarrow t$  transformation temperature and the reverse  $m \rightarrow t$  transformation occurred. In contrast, the forward  $t \rightarrow m$  transformation was retained upon hand grinding at lower speed and grinding force, which was not associated with extensive heat generation. Therefore, based on almost negligible amounts of transformed zirconia upon grinding under conditions used in our work, we assume that the locally developed temperatures exceeded the  $m \rightarrow t$  transformation temperature (about 7008C) above which

the tetragonal zirconia is thermodynamically stable. On the other hand, due to high stresses developed during grinding, severe surface cracks must have been formed which lowered the strength and reliability of the material. Blasting, in contrast, is considered to be a more gentle process, during which considerably less material is removed from the surface. In spite of lower stresses occurring during sandblasting, the thickness of the transformed surface layer was found to be larger than in ground samples, indicating that not only stresses, but also the locally developed temperatures during sandblasting were lower. The calculated transformed zone depth, roughly corresponding to the average grain size of sintered Y-TZP ceramic, implies that only those tetragonal zirconia grains have transformed into the monoclinic symmetry during sandblasting, which are forming the very surface layer. Since these grains are not hydrostatically constrained as those in the bulk of the material, they can readily transform under the tresses caused by impacting alumina particles. Although the thickness of the surface compressive layer formed during sandblasting is very small, in comparison with the thickness of the test specimens, it is effective in increasing the strength of the Y-TZP materials. The length of surface flaws, which are introduced by sandblasting, does not seem to exceed largely the thickness of the compressive surface layer, otherwise the strength of the material would have been reduced instead of being increased. Recently published results by Peterson et al. (145), who studied strength degradation of various dental ceramic materials upon indentation damage, support this assumption. According to these authors, the contact damage in Y-TZP, which is closely equivalent to impact damage caused by sand blasting, is manifested in surfacelocalized, incompletely developed ring cracks which do not extend into the bulk of the material. In order to illustrate the counteracting effect of surface grinding and sandblasting on flexural strength of Y-TZP, an effective value for the mean critical defect size ccr for each group was calculated, the mean critical defect size of ground and sandblasted specimens should be regarded only as an effective length of strength controlling defects, which would result in an equivalent mean strength of the material without any residual surface stresses. Bearing this in mind, For instance, the calculated ccr value for the as-sintered fine grained Y-TZP is smaller than that in the coarse grained material, which is a commonly reported observation, and relates to the inherent flaw population generated during sintering of fine and coarse powders (138,139). Surface grinding, dry or wet, increases the Surface grinding, dry or wet, increases the effective critical defect size by generating surface cracks which can be readily observed by SEM examination of fracture surfaces. Similarly, greater flaw tolerance of tougher course grained TZP ceramics upon indentation was observed by Readey et al. (140). By using water spray during grinding, the calculated ccr of dry ground specimens was reduced by about 30%, indicating lower stresses during wet grinding. In comparison with Si3N ceramics, a further reduction in ccr can be expected by using a finer diamond burr (141). Sandblasting followed by dry grinding appears to be at least as detrimental surface treatment as dry grinding alone. It results in the highest calculated ccr in the coarse grained material and next highest ccr value in the fine grained Y-TZP, with highest statistical variability in the effective critical flaw size in both materials. If, however, dry grinding was followed by sandblasting, the effective mean critical defect size was reduced to one third of its initial value that is reflected in significant strengthening of ground Y-TZP ceramics. Microscopic examination of these samples revealed that a substantial erosive wear through chipping has occurred during sandblasting which largely leveled the surface of ground material. The SEM image of an eroded surface also indicates that some of the larger grinding induced cracks were removed from the surface during sandblasting. However, since lateral crack chipping is a most prevalent mechanism involved in erosive wear (144), lateral cracks were expected instead, which was later confirmed by fractographic examination. In most cases, failure of these samples was initiated from a surface crack linked to subsurfacecracks,. Notice that the critical flaw size, which initiated failure of this fine grained specimen, is evidently larger (50 mm) than the effective value for ccr reported for this particular group (14.8 mm). The difference between the true and calculated effective flaw size is believed to be due to surface compressive stresses introduced during sandblasting such that the reduced *c*cr mainly reflects the contribution of surface compressive layer to the mean strength of Y-TZP ceramic. This effect was even more pronounced when the surface of the specimens had not been previously damaged by grinding. However, large scatter in strength values, as reflected in decreased Weibull modulus of the sandblasted samples, indicates that during this operation the ceramic surface was not only constrained but also damaged. Under clinical conditions, where the material is exposed to thermal and mechanical cycling in a chemically active aqueous environment over long periods, these impact flaws may grow to become stress intensifiers, facilitating fracture initiation at lower levels of applied stress. Since coarse grained ceramics exhibit stronger crack resistance toughening, it is likely to expect the effect to be more pronounced in fine grained Y-TZP. The influence of various mechanical treatments on the reliability of sintered Y TZP ceramic can best be discussed in terms of the Weibull parameters. If the clinical failures of Y-TZP dental ceramic were primarily due to local over loadings on bending, the largest number of accidental failures could be expected with the dry ground—fine or coarse grained—material which was sandblasted prior to grinding. By omitting sandblasting prior to grinding and by using water

spray during this operation, the performance of the material should be improved but not to the extent observed on sandblasting of already ground Y-TZP ceramic. The formation of a compressive surface layer during subsequent sandblasting largely compensates for the grinding-induced surface cracks by pushing the normalized strength (s0) of the material toward the higher values. Although, sandblasting does not increase the Weibull modulus of the already ground Y-TZP ceramic, the increased s0 results in acceptable low failure probability of the material up to the bending stress level of about 500 MPa. According to the results, the as-sintered coarse and fine grained material can be exposed to approximately 700 MPa bending stress level without any serious risk of failure. Since, coarse grained Y-TZP is also less susceptible to grinding induced defects; it appears that tougher coarse grained material might be suggested for dental application of zirconia ceramic requiring surface treatment, in spite of somewhat lower mean strength before surface treatments. SEM micrograph of the surface of fine grained dry ground 1 sandblasted Y-TZP showing remaining grinding scratches and sharp sandblasting secures. The predominant mechanism of material removal during sandblasting was surface chipping. Failure originated from a 50 mm deep surface pit. It should be pointed out, that the strength of damaged zirconia is still higher than the strength of other fullceramic systems even without any surface treatment (142, 143). Under clinical conditions, however, dental restorations are exposed to stress and chemical interactions that are more complex and deleterious than those used in our strength degradation tests. It is, therefore, reasonable to expect first accidental failures to occur at stress levels lower than those reported in our study.

#### 4.3. Biaxial flexural strength

The biaxial strength testing has been used for many years (119) and is considered a more reliable method of assessing the strength of brittle dental ceramic materials than uni-axial flexural tests (15, 30, 105). There are several advantages claimed for biaxial flexural testing of discs compared with uni-axial testing of bars including ease of test piece preparation and ability to test a large effective surface area (119,120). Additionally, the test provides an equibiaxial stress distribution, which is more searching for defects than a uni-axial distribution as found in a bar test (119). The specimen is loaded on the opposite sides with radically symmetrical bending forces (120) and the maximum tensile stress occurs at the central loading area rather than at the edges. Hence, the cracks propagate from the centre of the disc toward the edges (105; 109; 120). The 'edge effect' on the strength data is eliminated or minimized (105, 109, and 120). Biaxial strength testing has some disadvantages compared with uni-axial testing. First, it is unclear what the optimum test geometry in terms of test piece aspect ratio and edge overhang should be. The use of thin plate analytical solutions for the stress field may have limitations, especially regarding thin materials such as electrolyte membranes Morrell et al. (119).

A wide variety of loading arrangements have been developed for biaxial flexure tests such as ring-on ring, piston-on-ring, ball-on ring, ring-on-ring, and piston-on three balls. A difference in geometry for the strength tests such as uni-axial (3 or 4 point bending test) and biaxial test leads to a variation in strength values. Ban and Anusavice (1990) studied failure stress of four brittle materials (zinc phosphate, opaque porcelain, body porcelain and resin composite) using biaxial flexural test (piston on three balls) compared

with a four point flexural test. They found that the mean strength obtained from biaxial flexural test was higher than a four-point flexural test. Shetty *et al* (1981) reported that there was no significant difference between the strength value of glass ceramic tested by three and four point bending; however, biaxial flexural test (balloon- ring) provided a higher strength value in comparison. One of the reasons that the different geometries provide different results is the specimen preparation. The specimens for uniaxial flexural test are prepared in bar shape whilst those for biaxial tests are prepared in disc shaped. Undesirable edge fracture could occur when preparing bar specimens and this may ultimately affect strength.

# 5. Conclusion

In this study the following conclusions are drawing:

1-Increasing the core thickness from 0.3-to 0.5 has shown 7% increase at the flexural strength.

2-Increasing the core thickness from 0.5 to 0.7 mm has shown no differences at the flexural strength.

Result of this in-vitro study shows that increasing the thickness of zirconia from 0.3 mm to 0.7 mm it does not appear to be significant effect on flexural strength. In the future, in-vivo studies have to be carried out to validate the mean of this in-vitro study.
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