Effect of Speed Firing Protocol on Physical and Mechanical Properties of Cubic Phase Containing Zirconia Used as Implant Restorations

Suchada Kongkiatkamon

A Thesis Submitted in Fulfillment of the Requirements for the Degree of Doctor of Philosophy in Oral Health Sciences
Prince of Songkla University
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Author	Ms. Suchada. Kongkiatkamon
Major Program	Oral Health Sciences

Major Advisor

.....
(Assoc. Prof. Dr. Chaimongkon Peampring)

Examining Committee :

.....Chairperson
(Assist. Prof. Dr. Pisaisit Chaijareenont)

.....Committee
(Assoc. Prof. Dr. Chaimongkon Peampring)

.....Committee
(Assoc. Prof. Dr. Tool Sriamporn)

.....Committee
(Prof. Dr. Chidchanok Loothanakul)

.....Committee
(Assoc. Prof. Dr. Sasiwimol Sanohkan)

The Graduate School, Prince of Songkla University, has approved this thesis as fulfillment of the requirements for the Doctor of Philosophy Degree in Oral Health Sciences

.....
(Asst. Prof. Dr. Thakerng Wongsirichot)
Acting Dean of Graduate School

This is to certify that the work here submitted is the result of the candidate's own investigations. Due acknowledgement has been made of any assistance received.

.....Signature
(Assoc. Prof. Dr. Chaimongkon Peampring)
Major Advisor

.....Signature
(Ms. Suchada Kongkiatkamon)
Candidate

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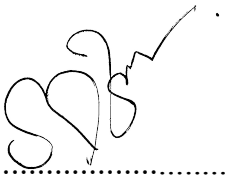

.....Signature
(Ms. Suchada Kongkiatkamon)
Candidate

ชื่อวิทยานิพนธ์ **อิทธิพลของการเผาผนึกอย่างด่วนที่ส่งผลต่อคุณสมบัติทางกายภาพ และคุณสมบัติทางกลของของเซอร์โคเนียชนิดโปร่งแสงที่มีองค์ประกอบของผลึกคิวบิกในงานทันต กรรมรากเทียม**

ผู้เขียน นางสาวสุชาดา ก้องเกียรติกมล

สาขาวิชา วิทยาศาสตร์สุขภาพช่องปาก

ปีการศึกษา 2565

บทคัดย่อ

วัตถุประสงค์: ในการทดลองส่วนแรกเพื่อศึกษาโครงสร้างจุลภาค และลักษณะวัฏภาค คุณสมบัติทางแสงและคุณสมบัติเชิงกลของเซอร์โคเนียชนิดโปร่งแสงที่มีองค์ประกอบของคิวบิกที่ได้รับการเผาผนึกที่แตกต่างกันคือ การเผาผนึกด้วยวิธีปกติ และการเผาผนึกอย่างด่วน ในการทดลองส่วนที่สองเพื่อทำการศึกษาลักษณะของพื้นผิว การแตก และการต้านทานต่อการล้าของครอบฟันเซอร์โคเนียชนิดโปร่งแสงที่มีองค์ประกอบของคิวบิก ที่ได้รับการเผาผนึกที่แตกต่างกันคือ การเผาผนึกด้วยวิธีปกติ และการเผาผนึกอย่างด่วน

วิธีการวิจัย: ในส่วนแรก ได้เตรียมชิ้นงานจากเซอร์โคเนียเซรามิกให้เป็นรูปทรงแผ่นกลมทั้งหมด 60 ชิ้น (แบ่งเป็นสองกลุ่ม โดย 30 ชิ้นสำหรับการเผาผนึกแบบปกติ และ 30 ชิ้นสำหรับการเผาผนึกแบบด่วน) เซอร์โคเนีย 15 ชิ้นจากทั้งสองกลุ่มจะถูกจำลองการสลายตัวด้วยอุณหภูมิต่ำ หลังจากนั้นชิ้นงานทั้ง 4 กลุ่มจะถูกนำมาศึกษาลักษณะโครงสร้างจุลภาคในแต่ละกลุ่มด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราดเพื่อวัดขนาดอนุภาค และศึกษาการเปลี่ยนวัฏภาคของเซอร์โคเนียด้วยเครื่องวัดการเลี้ยวเบนของรังสีเอ็กซ์ นอกจากนั้นความแตกต่างของคุณสมบัติทางแสงของแต่ละกลุ่มจะถูกประเมินด้วยเครื่องวัดการดูดกลืนแสง นอกจากนี้ใช้ยังการประเมินความต้านทานการแตกหักในการเปรียบเทียบคุณสมบัติเชิงกลของชิ้นงานแต่ละกลุ่ม ในการทดลองส่วนที่สอง ครอบฟันบนรากเทียมแบบเจาะรูทั้งหมด 60 ชี่ถูกทำขึ้นมาจากเซอร์โคเนียด้วยวิธีการใช้คอมพิวเตอร์ในการออกแบบและผลิต (Katana STML Block) ชิ้นงานถูกแบ่งเป็น 2 กลุ่ม คือกลุ่มที่ได้รับการเผาผนึกแบบปกติ (30 ชิ้น) และกลุ่มที่ได้รับการเผาผนึกแบบด่วน (30 ชิ้น) โดย 15 ชิ้น จากทั้งสองกลุ่มนำไปผ่านการให้แรงเป็นรอบที่อุณหภูมิห้อง ชิ้นงานทุกกลุ่มจะถูกศึกษาลักษณะพื้นผิว และลักษณะรอยแตกด้วยกล้องจุลทรรศน์อิเล็กตรอนแบบส่องกราด นอกจากนี้ความต้านทานต่อการแตกหักเพื่อเปรียบเทียบคุณสมบัติเชิงกลยังได้รับการศึกษาอีกด้วย

ผลการศึกษา: ในการทดลองส่วนแรกพบว่า ชิ้นงานเซอร์โคเนียภายหลังการจำลองการสลายตัวด้วยอุณหภูมิต่ำ ทั้งในกลุ่มที่ได้รับการเผาผนึกแบบปกติ และแบบด่วนมีลักษณะโครงสร้างจุลภาคที่ไม่แตกต่างกัน ในกลุ่มที่ได้รับการเผาผนึกแบบปกติพบว่ามีความใสมากกว่ากลุ่มที่ผ่านการเผาผนึกแบบด่วน จากการเปรียบเทียบระหว่างกลุ่มที่ความเชื่อมั่นที่ 95 เปอร์เซ็นต์พบว่าทุกกลุ่มให้ค่าความใสแตกต่างกันอย่างมีนัยยะสำคัญ ยกเว้นกลุ่มที่ผ่านการเผาผนึกแบบด่วนที่ถูกจำลอง และไม่ได้ถูกจำลองการสลายตัวด้วยอุณหภูมิต่ำ ทั้งนี้ยังพบว่าในกลุ่มที่ผ่านการเผาผนึกอย่างด่วนนั้นมีค่าความต้านทานต่อการหักสูงกว่ากลุ่มที่ได้รับการเผาผนึกปกติ ในการทดลองส่วนที่สองพบว่าลักษณะของพื้นผิวที่แตกในกลุ่มที่ผ่านการเผาผนึกอย่างด่วนมีลักษณะสม่ำเสมอ และรอยแตกมีความยาวที่สั้นกว่าในกลุ่มที่ผ่านการเผาผนึกแบบปกติ นอกจากนี้ยังพบว่าในกลุ่มที่ผ่านการเผาผนึกแบบด่วนที่ไม่ได้นำไปผ่านการให้แรงเป็นรอบ มีค่าความต้านทานต่อการแตกหักสูงที่สุด

บทสรุป: เซอร์โคเนียที่ผ่านการเผาผนึกแบบปกติจะพบว่ามีขนาดอนุภาคที่ใหญ่ ซึ่งส่งผลให้มีค่าความใสที่มากกว่าเซอร์โคเนียที่ผ่านการเผาผนึกแบบด่วน แต่เซอร์โคเนียที่ผ่านการเผาผนึกแบบด่วนจะมีค่าความต้านทานต่อการแตกหัก และดัดงอที่สูงกว่า ซึ่งแสดงให้เห็นว่าการเผาผนึกแบบด่วนเป็นวิธีที่เหมาะสมสำหรับการใช้งานในคลินิก ในตำแหน่งที่ต้องการความแข็งแรงสูงอาจจะพิจารณาเผาผนึกเซอร์โคเนียแบบด่วน ในขณะที่บริเวณที่ต้องคำนึงถึงความสวยงามเป็นหลักยังแนะนำให้เผาผนึกเซอร์โคเนียแบบปกติอยู่

คำสำคัญ : เซอร์โคเนียเซรามิก, คุณสมบัติทางแสง, คุณสมบัติเชิงกล, การสลายด้วยอุณหภูมิต่ำ, ผลึกรูปคิวบิก

Thesis Title Effect of Speed Firing Protocol on Physical and Mechanical Properties of Cubic Phase Containing Zirconia Used as Implant Restorations

Author Ms. Suchada Kongkiatkamon

Major Program Oral Health Sciences

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ABSTRACT

Objectives: First part was to evaluate the surface structure, phase determination, translucency, and strength (flexural) of the zirconia (Katana STML Block and Disc) between the regular sintering (RS) and the speed sintering (SS) with and without low-temperature degradation (LTD). The second part aimed to compare the surface structure, cracks, and load-bearing capacity in zirconia screw-retained implant crowns between RS and SS protocols with and without cyclic loading (fatigue).

Materials and methods: For the first part, a total of 60 zirconia discs (30 per group; RS and SS). The zirconia discs were subjected to RS and SS with and without LTD. Scanning electron microscopy (SEM) was done to characterize the zirconia specimens and the zirconia grain size. Furthermore, the zirconia specimens were analyzed for elemental analysis using energy dispersive spectroscopy (EDS) and phase identification using X-ray diffraction. The zirconia specimens were analyzed for the translucency measurements and biaxial flexural strength testing. For the second part, a total of 60 screw-retained crowns were fabricated from zirconia (Katana STML Block) by the CAD/CAM system. Then, 30 crowns were subjected to the RS protocol and 30 crowns to the SS protocol. Cyclic loading was done in half zirconia crowns (15 crowns in each group) using a chewing simulator at room temperature. SEM was done to study the surface of the crowns and the cracks in the crowns of the RS and SS protocols, with and without fatigue. Load to failure was also evaluated.

Results: For the first part, the zirconia specimens with and without LTD in RS and SS presented a similar surface structure. RS showed more translucency compared to SS. Multiple comparisons of the translucency parameter were a significant difference (p value < 0.05) among various groups except for the comparison between SS and SS with LTD. The RS sintering showed bigger gain sizes and slightly more translucency compared to SS. The SS showed higher biaxial flexural strengths compared to RS. For the second part, for the SS group, the crack surface looks more uniform, and the crack lines are present at a short distance compared to RS. It showed that the SS group showed the maximum fracture load, followed by the RS, SS with fatigue, and RS with fatigue groups. The fracture load in various groups showed significant differences.

Conclusions: The RS showed a bigger grain size and slightly more translucency compared to SS. The SS showed higher biaxial flexural strengths compared to RS. This shows that SS can be considered a suitable method of sintering zirconia. Hence, when biaxial flexural strength is required, SS can be considered; however, when better translucency is required, RS is recommended.

Keyword : dental ceramics zirconia, translucency, mechanical properties, low temperature degradation, cubic zirconia

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This success would not have been possible without them.

Ms. Suchada Kongkiatkamon

Contents

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List of Abbreviations/Symbols

Y-TZP	Yttrium-stabilized tetragonal zirconia polycrystal
t	Tetragonal
m	Monoclinic
c	Cubic
FPDs	Fixed partial dentures
LTD	Low-temperature degradation
CR	Contrast ratio
T_t%	Total percentage of transmittance of light
TP	Translucency parameter
CAD/ CAM	Computer-aided design/ Computer-aided manufacturing
T	Vita YZ T material
ST	Vita YZ ST material
XT	Vita YZ XT material

Lists of Published Papers and Proceedings

List of Paper

1. Kongkiatkamon S, Peampring C. Comparison of Regular and Speed Sintering on Low-Temperature Degradation and Fatigue Resistance of Translucent Zirconia Crowns for Implants: An In Vitro Study. *Journal of Functional Biomaterials* 2022; 13: 281. doi: 10.3390/jfb13040281
2. Kongkiatkamon S, Peampring C. Effect of Speed Sintering on Low Temperature Degradation and Biaxial Flexural Strength of 5Y-TZP Zirconia. *Molecules* 2022; 27: 5272. doi: 10.3390/molecules27165272

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Paper 1

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Comparison of Regular and Speed Sintering on Low-Temperature Degradation and Fatigue Resistance of Translucent Zirconia Crowns for Implants: An In Vitro Study

Suchada Kongkiatkamon, Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Resources, Software, Writing – original draft and Chaimongkon Peampring, Conceptualization, Funding acquisition, Investigation, Project administration, Supervision, Validation, Visualization, Writing – review & editing*

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Paper 2

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Molecules

Effect of Speed Sintering on Low Temperature Degradation and Biaxial Flexural Strength of 5Y-TZP Zirconia

Suchada Kongkiatkamon and Chaimongkon Peampring*

Jan Janczak, Academic Editor

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Introduction

1. Introduction to Ceramic in Dentistry

All-ceramic dental restorations and prostheses are widely used at present because they are potential alternative to the metal-ceramic prostheses because of their esthetics, strengths, stability, and biocompatibility.[1-3] Zirconia (ZrO_2), a type of dental ceramic, is glass-free polycrystalline (96–99%) in nature in which all atoms are compacked into regular crystalline structure.[4] Zirconia has excellent physicochemical properties, such as excellent hardness, fracture toughness, flexural strength, biocompatibility, and esthetic. Hence, its application has been expanding in dentistry.[4]

Zirconia is polymorphic biomaterial and exists in basic three forms: monoclinic (m), tetragonal (t) , and cubic (c).[5,6] It is present in the m form at the room temperature zirconia and it is stable up to 1170◦C. When heated above the temperature of 1170◦C, a transformation results to the t phase which is stable up to 2370◦C.[7] Above the temperature of 2670 ◦C, zirconia assumes its c form.[8] On cooling from high to low temperatures, the c–t transformation occurs.[9] Conversely, the c–m transformations and t–m are caused from high-volume expansions (3%–5%). Pure zirconia results cracks from large stresses during cooling. To avoid these problems, oxide additives, e.g., calcium oxide (CaO), cerium oxide (CeO_2), magnesium oxide (MgO), and yttria (yttrium oxide, Y_2O_3) are added to stabilize the t or c phase of zirconia.

The main shortcomings of zirconia restorations are chipping of veneering porcelain and the inadequate adhesion.[10] Sometimes, airborne-particles can cause damage to the zirconia material and jeopardize the long-term outcome of the zirconia restorations. Another shortcomings of zirconia restorations is their less translucency, weak veneered with porcelain over zirconia cores due to the failure of the adhesion between the two materials.[11] In consideration to these problems, recently, new zirconia restorations (which is also known as the third generation) are introduced in the market with an intent to solve these problems of zirconia. More scientific evidences are needed for their applications in clinical dentistry.

The zirconia from different manufacturing processes has different processes related to microstructure. The Y-TZP undergoes low temperature degradation (LTD) which may cause damage of the restoration or even failure of the implant.[12] The aging of zirconia results due to the transformation of the stable t phase to the m phase (m–t transformation) in the presence of water or water vapor at relatively low temperatures (approximate 30°C up to 400°C) a phenomenon known as hydrothermal or LTD.[13,14] LTD has a adverse effect on the mechanical behavior of zirconia.[13] LTD becomes faster by the combination of stress and moisture.[15,16] In addition, the LTD is also related with other factors, such as type and amount of stabilizer [17], residual stress [17,18], density [19], sintering temperature and fabrication method and hydrothermal consitions [20,21], and surface finish [22].

This study will compare the surface structure, phase determination, translucency, and biaxial flexural strength of zirconia blocks and discs between regular sintering and speed sintering. In addition, this study will also compare the surface/ cracks determination and load-bearing capacity in zirconia screw-retained implant crowns between the regular sintering and the speed sintering.

2. Literature Review

2.1 Dental Ceramic and Zirconia Restorations

All-ceramic restorations are commonly used in dental clinics due to their good esthetic properties.[1] They are potential alternative to metal-ceramic restorations due to their esthetics, and biocompatibility.[2,3] Types of dental ceramic include glass ceramic, glass infiltrated ceramic, and oxide ceramic (Figure 1).[23]

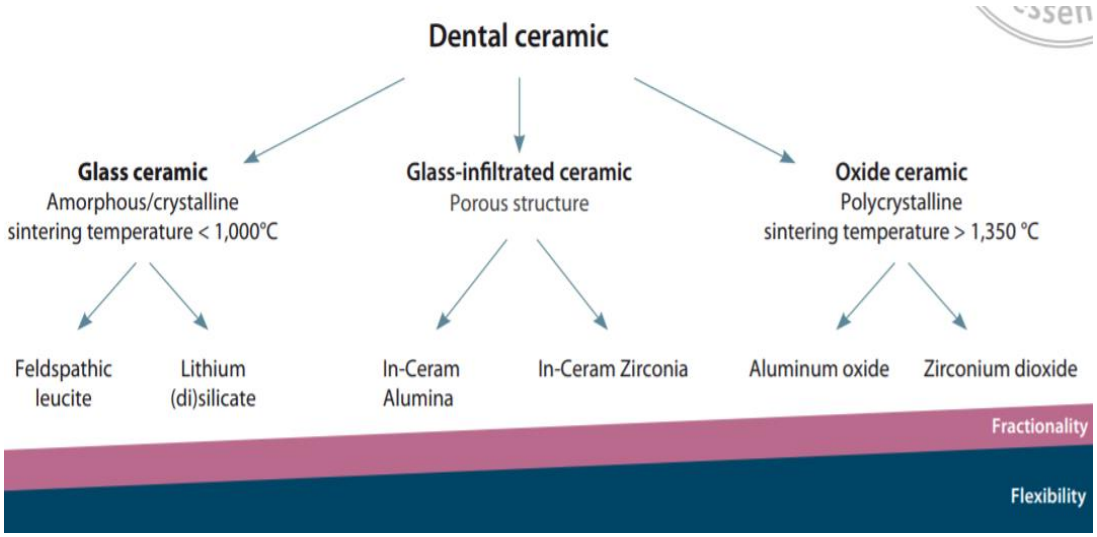


Figure 1. Types of dental ceramics.[23]

Zirconia (ZrO_2), a type of dental ceramic, is used in biomedical applications since the 1960s and it is used as a long-span restoration. It is glass-free polycrystalline (96%–99%) ceramics in which all atoms are packed into regular crystalline arrays.[4] Zirconia has excellent physicochemical properties, such as excellent biocompatibility, hardness, fracture toughness, flexural strength, and satisfactory esthetic (Figure 2). Hence, its application has been expanding in dentistry.[4] Zirconia restorations play important in dental implantology as they meet the functional and aesthetic need of the patients. Evidently, 3 mol% stabilized t zirconia polycrystalline (3Y-TZP) are being used as a favorable restoration due to their good mechanical properties such as high strength, biocompatibility, and esthetics.[2,24,25] Nevertheless, the zirconia core is less translucent than the glass-infiltrated ceramic.[26-28]

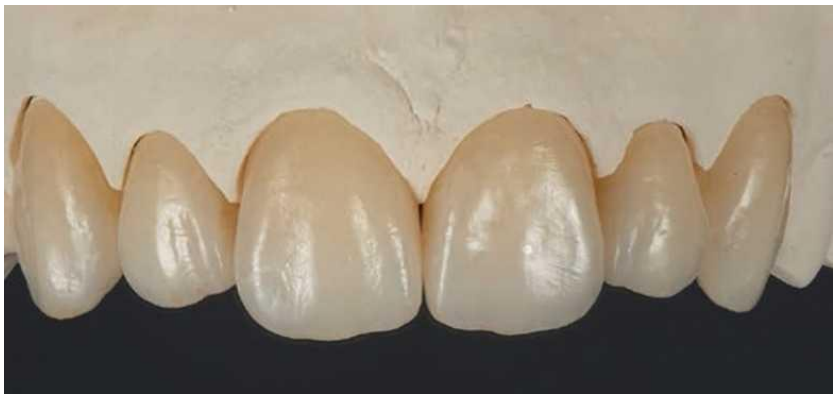


Figure 2. All-ceramic restorations.

Zirconia is polymorphic biomaterial which exists in three forms: m, t, and c (Figure 3).[5,6] It is present in its m form at the room temperature and it is stable up to 1170◦C. Above 1170◦C, a transformation occurs to the t phase that is stable up to 2370◦C.[7] Above the temperature of 2670 ◦C, zirconia assumes its c form.[8] On cooling to low temperatures, the c–t transformation occurs.[9]

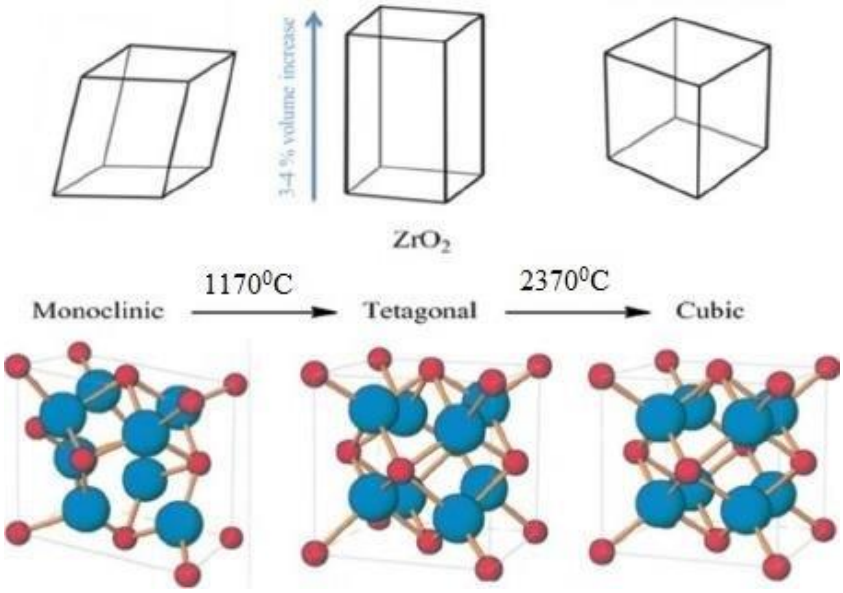


Figure 3. Polymorphic transformation of zirconia.[5]

Zirconia is generally produced from zircon mineral ($ZrSiO_4$) using decomposition agents.[9] Highly pure t zirconia is produced from the sol-gel process which includes dissolving acid by zirconium salt and nucleation and growth of zirconia particles. With this process, Y_2O_3 can be integrated during the fabrication process for the stabilization of t zirconia.. Finally, a high level of homogeneity and required particle size can be obtained. Furthermore, additives to zirconia such as MgO, CaO, CeO_2, and Y_2O_3 are used to stabilize the high-temperature phases and are known as partially stabilized zirconia (PSZ).[29] Yttria is used as a stabilizer but full stabilization is achieved with yttria t zirconia particles (Y-TZP). Thus, 8, 5, 3 and 2 mol% yttria fully

stabilized zirconia are found in market. The yttria stabilizer particle sizes may range from 0.5 μm to 0.1 μm. Table 1 shows the chemical composition and technical properties of third generation zirconia.[30]

Table 1. The composition and properties of third generation zirconia.[30]

Details	Contents
Chemical Composition	
• $ZrO_2 + HfO_2$	87–92%
• Yttrium oxide (Y_2O_3)	8–11%
• Other oxides	0–2%
Properties	
• Bending strength	557 MPa
• Coefficient of thermal expansion (25–500 °C)	$9.7 \pm 0.2\ 10^{-6}K^{-1}$
• Translucency	43%

Various uses of zirconia in dentistry include abutments, crowns (full and partial), implant material, veneer, orthodontic brackets, and posts. Zirconia dental prosthesis can be produced from a zirconia disc or block (Figure 4).

Figure 4. Fabrication of zirconia dental prosthesis from zirconia disc and block.

In dentistry, the use of metallic parts results may cause unfavorable esthetic results from grayish color discoloration of ceramic crowns and gingiva and may cause allergy. These problems led to the production of esthetic zirconia and other ceramic materials. Zirconia implants are esthetically pleasing with no dark discoloration around the gingiva and option for people with known metal allergies. Zirconia also can be used in implant dentistry such as prosthetic crowns, abutments, and dental implants (Figure 5). A zirconia implant material retains less plaque and calculus compared to titanium.[31,32] It has been shown zirconia implants can be used as an alternative to titanium with a superior aesthetics, biocompatibility, and soft-tissue response.[33,34] But the zirconia is more brittle with lower fracture and flexural strength than titanium. Zirconia implants with a small diameter are prone to fracture.

Figure 5. Various applications of zirconia in dental dentistry.

Zirconia is divided into three groups according to the yttria content as follows.[35]

1. First Group: This group is strong, 3 mole % Y-TZP (3Y-TZP, mainly tetragonal) (IPS e.max® ZirCad LT and MO, , 3M; BruxZir®, Glidewell Laboratories; Ivoclar Vivadent; Lava™ Plus and KATANA™ HT, Kuraray Noritake).
2. Second Group: This group is more translucent, 4 mole % Y-TZP (4Y-TZP) (IPS e.max ZirCAD MT; Zpex® 4, Kraun; and KATANA™ ST/STML).
3. Third Group: This group is most translucent, 5 mole % Y-TZP (5Y-TZP) with reduced mechanical properties (Lava Esthetic; Cercon® XT, KATANA™ UT/UTML; BruxZir Anterior; Dentsply Sirona; and Zpex Smile),

The properties also vary according to the % Y-TZP as shown in Table 2.

Table 2. Types of zirconia-based on the yttria content.

	3 Y-TZP	4 Y-TZP	5 Y-TZP
Phase	Mainly tetragonal	Tetragonal and cubic	More cubic and less tetragonal
Flexural strength	Maximum	High	Lower
Fracture toughness	Maximum	High	Lower
Translucency	White opaque	Some translucency	Moderate translucency
Esthetic	Low esthetic	Moderate esthetic	High esthetic

An example of third generation zirconia restoration is shown in Figure 6. They have light refraction similar to that of the natural teeth, integrated translucency, and no laborious pretreatment but only polishing or glazing required, and available in various shades.[30] But the fracture toughness and flexural strength are slightly lower than first and second generation zirconia. The c phase zirconia having increasing yttria content (5Y-TZP) have shown the best effect of translucency as a result of 9.35 wt.% yttria-stabilized zirconia powder and 46% t zirconia and 54% c zirconia.[36,37]

Figure 6. Dental restoration made from third generation zirconia.[30]

Various mechanical and chemical treatment methods are proposed on zirconia surfaces to bond the zirconia to the resin-based materials. Phosphoric acid (30%–40%) is used to clean and remove contaminants from the bonding surface of prosthetic restoration.[10] Aluminum oxide air abrasion increases the bonding area for mechanical interlocking and surface wettability of zirconia. Other protocols for the surface treatment of zircona are plasma technology, hydrofluoric acid (>40%), and coupling agents (zirconia primers, metal primers, and silanes).

2.2 Sintering of Zirconia and Zirconia Restorations

Zirconia restorations can be fabricated by two methods. One method is by partially sintered state from soft machining followed by sintering cycle and another method is fully sintered state by using hard machining.[38,39] The soft machining produces less accurate frameworks due to the shrinkage during sintering. But, the hard machining results t–m transformation and can introduce cracks, and can cause milling machine wear.[40]

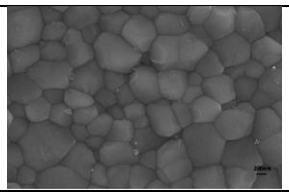
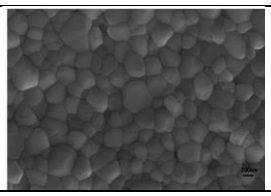
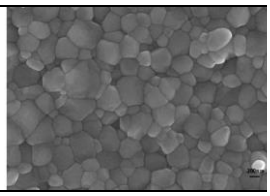
The sintering of zirconia allow densification without causing simultaneous grain growth necessary for the microstructural refinement.[41] Pretreatment is done at a low temperature and then at higher-temperature and then cooling. This allows refinement of the microstructure and present better properties.[42]

The details on the conventional, rapid, and super-speed sintering is shown in Table 3. Table 4 shows various mechanical properties of zirconia according to the types of sintering of zirconia (conventional, rapid and super-rapid).[43]

Table 3. Various tested materials, sintering protocols for zirconia.[43]

	Sintering Details	Sintering time
Conventional sintering (CS)	Heating at 2 °C/min to 1480 °C, then dwelling for 120 min, then cooling at 30 °C/min down to 200 °C.	15 h
Super-speed sintering-1 (S-1)	Heating at 400 °C/min to 1200 °C, then at 190 °C/min to 1580 °C, dwelling for 2 min, then cooling at 310 °C/ min down to room temperature.	12 min
Super-speed sintering-2 (S-2)	Heating at 400 °C/min to 1200 °C, dwelling for 5 min, then at 190 °C/min to 1580 °C, dwelling for 10 min, followed by cooling at 310 °C/ min down to room	25 min

Table 4. Properties of zirconia of various sintering protocols.[43]

Properties	Conventional sintering	Rapid sintering	Super-speed sintering
Grain size (μm)	0.6	0.59	0.32
Hardness (GPa)	13.4 (0.2)	13.3 (0.1)	13.6 (0.2)
Flexural strength (MPa)	1172 (73)	1151 (149)	1038 (183)
Fracture toughness (MPa・$m^{1/2}$)	5.89 (0.18)	5.92 (0.09)	5.33 (0.34)
Presence of a stable monoclinic phase	Monoclinic phases 10%	Monoclinic phases 5%	No obvious monoclinic phase
Structure under scanning electron microscopy			

2.3 Failures of Zirconia

Failure of zirconia/ceramic materials result from the surface or volume flaws. The pores can act as a stress concentration during cooling and lead to the formation of microcrack. [45-47] Hoffman and Rödel [46] showed that the critical stress for microcrack formation decreases with increasing grain size and the failure is caused by the stress concentrations around the pore before a microcrack can form.

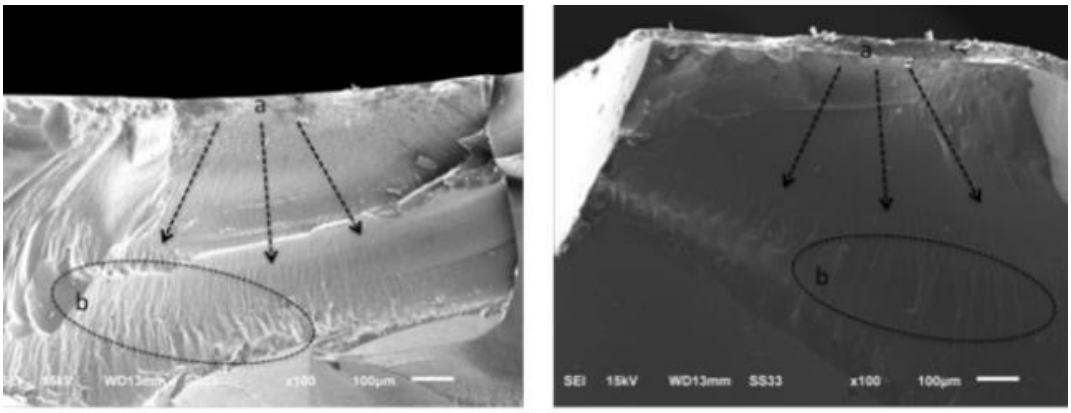
It has been found that when monolithic zirconia restoration is exposed to the humid environment of saliva, it may be deteriorated by low temperature degradation because of the zirconia structure.[48] Regarding the aging and degradation of zirconia, some theories have explained. Yttria is added in zirconia to achieve phase stability and

it is finished through a reaction with water resulting in t to m (t–m) transformation.[48-50] This transformation is caused when the zirconia bond (Zr-O) is disturbed by water (O_2 fills the O_2 vacancies), thereby creating stress due to the diffusion of OH^- and forming lattice defects.[51,52] This results in microcracks and grain pull-out produced on zirconia surfaces. Water can penetrate cracks and induce the surface degradation of zirconia.[50]

2.4 Low Temperature Degradation (LTD)

The aging process results from the transformation of the stable t phase to the m phase (t–m transformation) in the presence of water or water vapor at relatively low temperatures (approximately 30°C up to 400°C) a phenomenon known as hydrothermal or LTD.[13,14] The LTD of t zirconia was first reported by Kobayashi *et al*. in 1981.[54] Since then, much research has been performed on the degradation of zirconia ceramics.[55] A thinner Y-TZP specimen presents surface defects and microcracks after aging and decreases the flexural strengths.[55]

LTD is increased by stress and moisture in an environment (Figure 7).[15,16] In addition, the LTD can also be associated with other factors, type and amount of stabilizer [17], residual stress [17,18], density [19], sintering temperature and fabrication method [20,21], surface finish [22], and different hydrothermal conditions.



(A) (B)

Figure 7. Scanning electron microscopy images of polished monolithic (A) and glazed monolithic (B) fractured specimens, crack propagation starts at the occlusal surface.[16]

The LTD results in a detrimental influence on the long-term mechanical behavior of zirconia ceramics in the oral cavity.[13] The stresses the near grains and microcracks appear allowing water to penetrate and the process to progress, resulting in a remarkably surface roughness increase and decrease in hardness and strength thereby severely degrading the zirconia properties.[56-58] In addition, a recent study by Kengtanyakich and Peampring [59] showed that hydrothermal aging in the autoclave for 8 hours caused a significant decrease in the fracture toughness and low-temperature

degradation affected the surface properties (surface roughness and hardness) of 3Y-TZP zirconia material, owing to the spontaneous t-m transformation.

In addition, the mechanical properties of the Y-TZP can dramatically reduce due to LTD. Flinn et al.[60] evaluated the LTD behavior of Y-TZP materials by accelerated aging specimens in steam at 134°C, 2 bar. It also showed that following 200 hours of aging of Y-TZP could cause a significant transformation from t-m crystal structure, which statistically significant decrease in the flexural strength.[60,61]

2.5 Effect of Sintering Protocol on Zirconia Properties

The sintering of zirconia can affect its microstructure and properties.[62,63] Various types of sintering can be done for the zirconia from conventional to super-fast. Table 4 shows the review of some studies (tested materials, experimental design, sintering protocols, and testing for zirconia).

Studies have been done on the effect of the changes in sintering time and temperature on the grain size, biaxial flexural strength, and translucency of zirconia.[25,64-66] Li et al.[43] found super-speed sintered at 1580 °C dental zirconia with a dwelling time of less than 20 min results acceptable clinical performances for one-visit application. In 20 minutes, a longer dwelling time can present better transmittance and mechanical properties of zirconia (Table 5). Kaizer et al. [44] mentioned that fast sintering protocols results better microstructural, physical, and wear properties of monolithic zirconia (Table 6).

Table 5. Results of mechanical properties of dental zirconia ceramics after conventional and super-speed sintering.[43]

	Conventional sintering (CS)	Super-speed sintering-1 (S-1)	Super-speed sintering-2 (S-2)	Super-speed sintering-3 (S-3)
Grain size (μm)	0.63	0.32	0.51	0.59
Flexural strength (MPa)	1172(73)	1038(183)	1087 (124)	1151(149)
Hardness (GPa)	13.4(0.2)	13.6(0.2)	13.4(0.3)	13.3(0.1)
Fracture toughness ($MPa \cdot m^{1/2}$)	5.89(0.18)	5.33 (0.34)	5.49(0.17)	5.92(0.09)

Table 6. Results of mechanical properties of zirconia crowns after long-term and super-speed sintering.[44]

	Long-term sintering (LT)	Speed sintering (S)	Super-speed sintering (SS)
Grain size (μm)	0.66	0.50	0.59
Flexural strength (MPa)	13.3 (0.1)	13.1 (0.2)	13.1 (0.2)
Hardness (GPa)	579.7 (130.6)	622.3 (82.7)	904.27 (115.7)
Translucency	4.3 (04)	4.2 (0.5)	4.6 (0.4)

Kim *et al*.[64] studied the effects of the sintering conditions of dental zirconia (Lava and KaVo) on grain size and translucency. The dwelling time was 20 min for microwave sintering (MS) and 20 min, 2, 10, and 40 hours for conventional sintering (CS). They concluded that different sintering conditions resulted in differences in grain size and light transmittance. Shorter sintering times result in more translucent dental zirconia restorations. Other important parameters are the composition type, particle size, and processing method can affect the final microstructure. Figure 8 shows the schematic representation of the conventional and two-step sintering technique.[67]

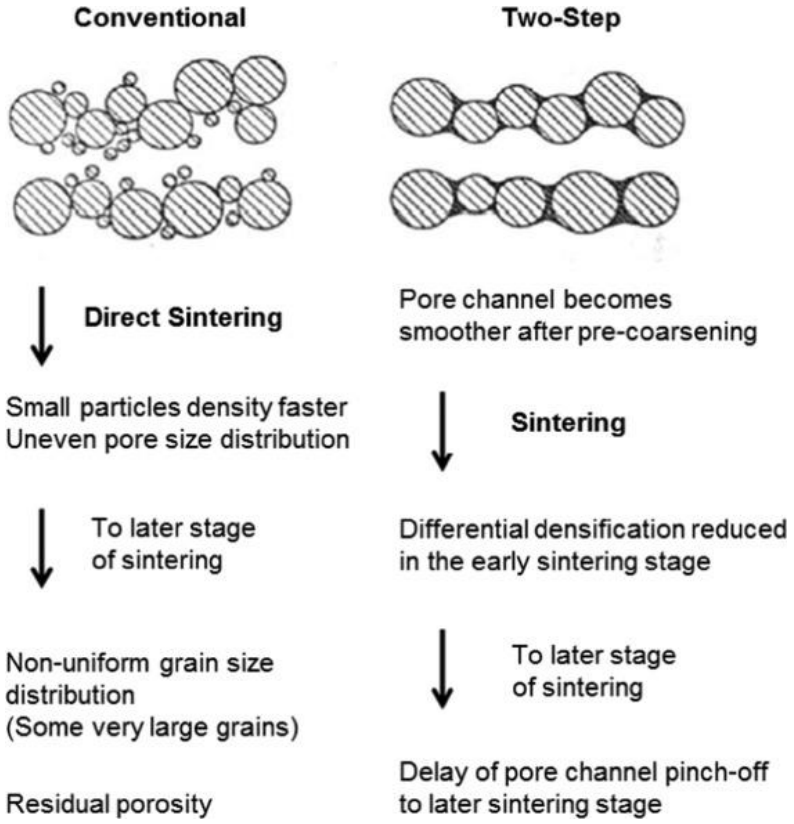


Figure 8. Conventional and two-step sintering and microstructural refinement.[67]

2.6 Screw-Retained Implant Restoration

Implant restorations (screw-retained or cemented retained on standard or customized abutments) are two types of restoration that show similar outcomes in clinical studies.[69] The fracture load of implant-supported restorations is an important factor in clinical success. Mokhtarpour et al. [70] evaluated the effect of two techniques for screw hole preparation (screw hole preparation before sintering and preparing screw hole manually after sintering) on the fracture load and found both techniques in implant-supported zirconia-based crowns decreased the fracture load.

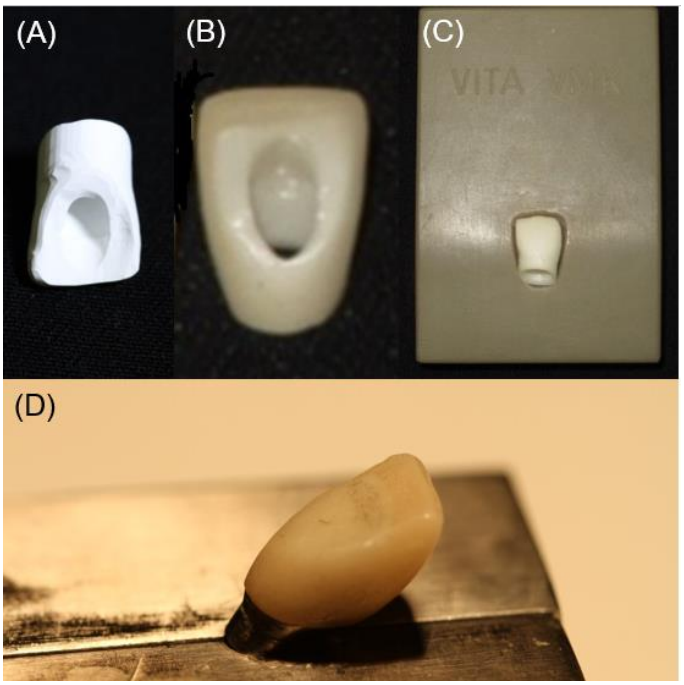


Figure 9. Screw access hole preparation on the fracture load of implant-supported zirconia-based crowns.[70] Milled zirconia with screw hole before sintering (A) and after sintering and testing (C-D).

As the zirconia from different manufacturing processes has different process-related microstructures, there is a need to assess their aging sensitivity. The Y-TZP can undergo LTD and can result restoration damage or failure of the implant.[12]

3. Rationale

It is important to study the surface structure, phase determination, translucency, and biaxial flexural strength of zirconia blocks and discs between the regular sintering and the speed sintering. In addition, it is also important to compare the surface/ cracks determination and load-bearing capacity in zirconia screw-retained implant crowns between the regular sintering and the speed sintering. But there is no literature on these topics.

4. Research Questions

Is there a difference in the surface structure, phase determination, translucency, and biaxial flexural strength of cubic containing zirconia between the regular sintering and the speed sintering?

Is there a difference in the surface/crack determination and load-bearing capacity in zirconia screw-retained implant crowns between the regular sintering and the speed sintering?

5. Conceptual Framework

The conceptual framework of this study is shown below (Figure 10).

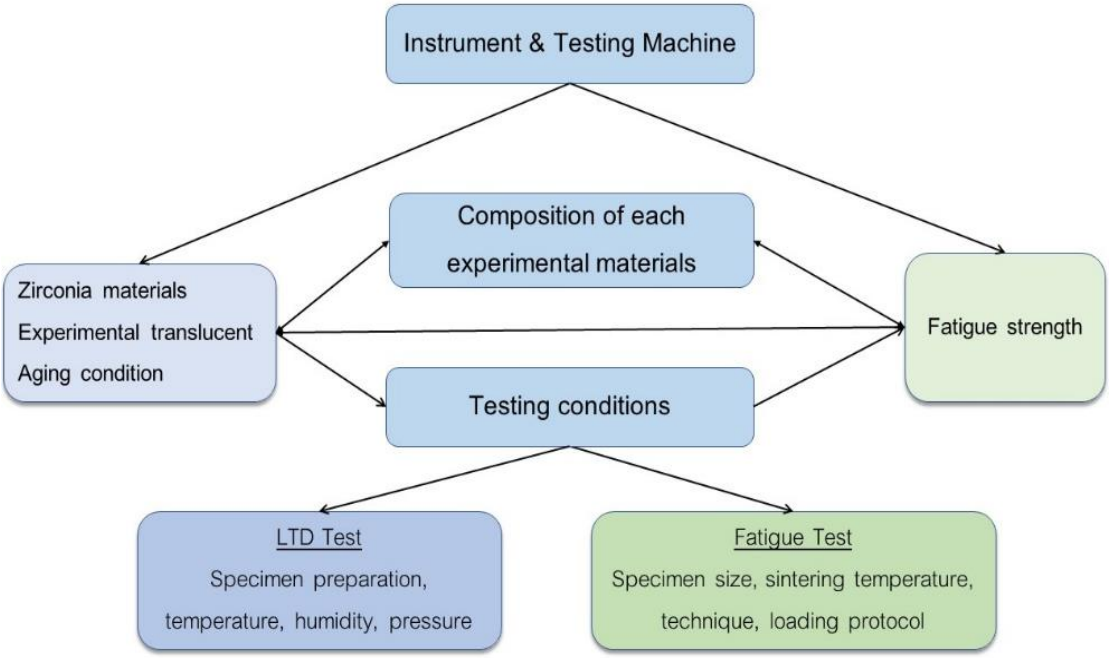


Figure 10. Conceptual framework of this study

Objectives

The research objectives are as follows.

The first objective is to compare the surface structure, phase determination, translucency, and biaxial flexural strength of cubic containing zirconia between the regular sintering and the speed sintering with and without low temperature degradation.

The second objective is to compare the surface/ cracks determination and load-bearing capacity in zirconia screw-retained implant crowns between the regular sintering and the speed sintering with and without cyclic loading (fatigue).

Result and Discussion

Results

1. Results of Zirconia Disc

1.1 Surface Structure and Elemental Analysis

The SEM images of the zirconia specimens are shown in Figure 11 and the results of the grain size (μm) in each group are shown in Table 7 and Figure 12. It showed that regular sintering showed a higher grain size compared to speed sintering. The LTD zirconia specimens showed a similar structure to no LTD in both regular and speed sintering groups.

Table 7. Results of the grain sizes of the zirconia specimens in various groups.

Groups	Mean (μm)	SD (μm)
Regular	3.206	1.257
Regular LTD	3.442	1.820
Speed	1.822	0.452
Speed LTD	1.689	1.053

SD = Standard deviation.

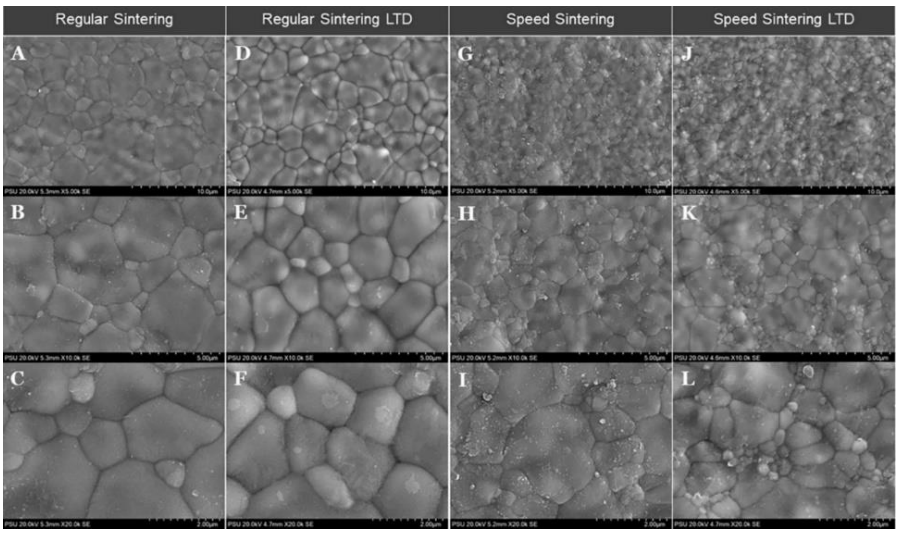


Figure 11. Scanning electron microscopy images of the zirconia specimens for regular sintering and speed sintering with and without low-temperature degradation (LTD). Regular sintering (A–F) and speed sintering (G–I) from low magnification to high magnification.

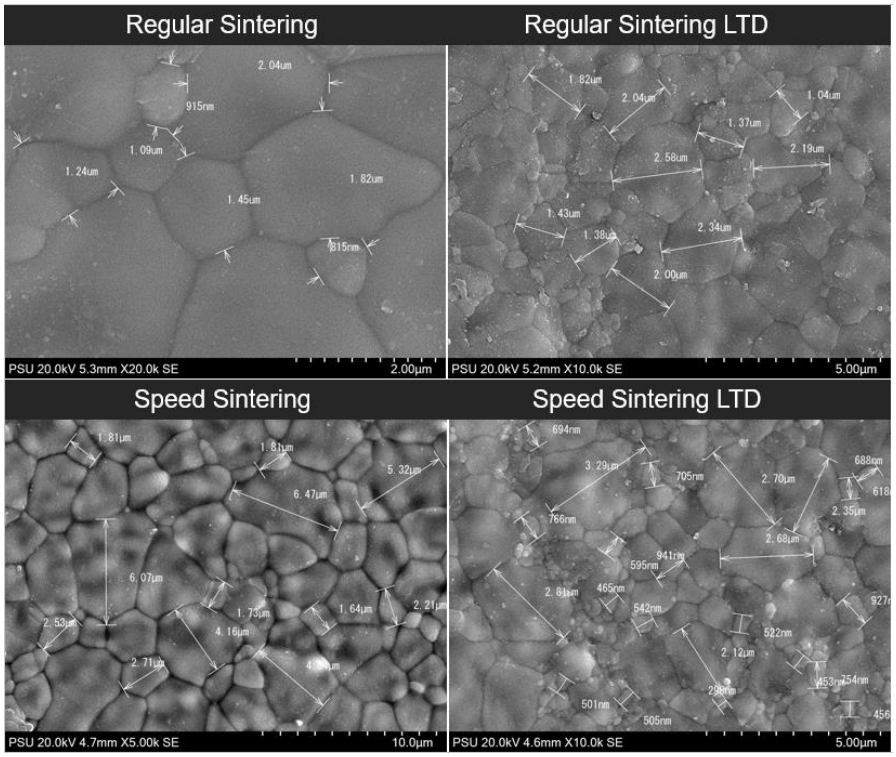


Figure 12. Grain size (μm) as measured from the scanning electron microscopy images of the zirconia specimens for the regular sintering and speed sintering with and without low-temperature degradation (LTD).

Table 8 shows the multiple comparisons of the grain size of the zirconia specimens among various groups among groups. The regular sintering showed a significantly bigger grain size (p value < 0.05) compared to the speed sintering LTD and regular sintering LTD compared to the speed sintering and speed sintering LTD. There was no difference in the flexural strength between the regular sintering vs regular sintering LTD (p value = 0.975) and speed sintering vs speed sintering LTD (p value = 0.995).

Table 8. Multiple comparisons of the grain size of the zirconia specimens among various groups.

(I) Groups	(J) Groups	Mean Difference (I-J)	*p* value
Regular	Regular LTD	−0.236	0.975
	Speed	1.383	0.075
	Speed LTD	1.517	0.042 *
Regular LTD	Regular	0.236	0.975
	Speed	1.6192	0.026 *
	Speed LTD	1.7532	0.014 *
Speed	Regular	−1.383	0.075
	Regular LTD	−1.6192	0.026 *
	Speed LTD	0.134	0.995
Speed LTD	Regular Sintering	−1.517	0.042 *
	Regular Sintering LTD	−1.753	0.014 *
	Speed Sintering	−0.134	0.995

* The significant difference at the 0.05 level. Multiple comparisons done using Scheffe analysis.

The EDS elemental analysis of the zirconia specimens is shown in Figure 13 and Table 9. Figure 13 shows the EDS spectra and Table 9 shows the elemental analysis of the zirconia specimens in atomic %. The speed sintering presented slightly higher Zr. LTD showed slightly lower C and O with higher Y, Zr, and Hf.

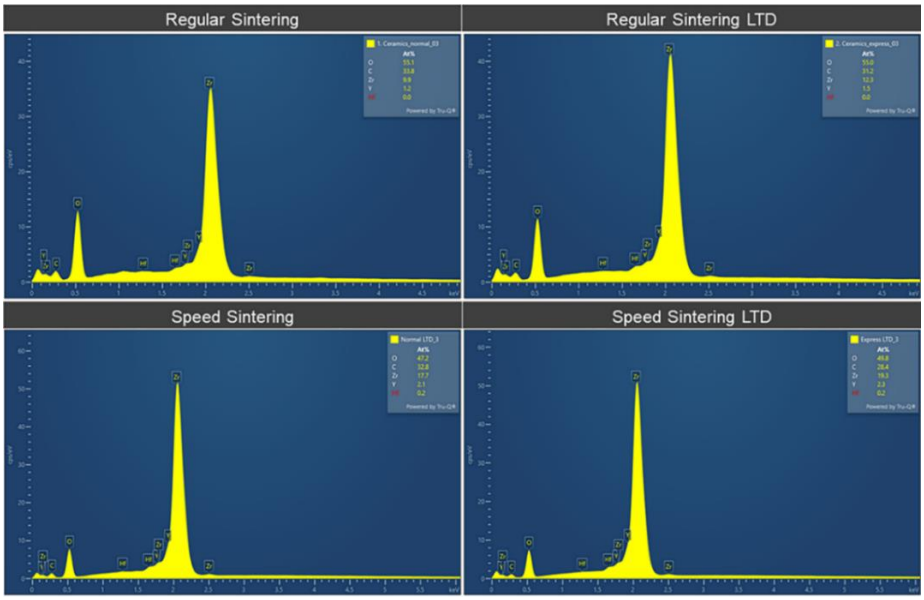


Figure 13. EDS Spectra of the zirconia specimens for the regular speed sintering and speed sintering with and without low-temperature degradation (LTD).

Table 9. Results of the EDS elemental analysis of the zirconia specimens for the regular speed sintering and speed sintering with and without low-temperature degradation (LTD).

Zirconia Specimens	C (Mean ± SD) (Atomic %)	O (Mean ± SD) (Atomic %)	Y (Mean ± SD) (Atomic %)	Zr (Mean ± SD) (Atomic %)	Hf (Mean ± SD) (Atomic %)
Regular Sintering	35.073 ± 1.355	53.773 ± 1.505	1.220 ± 0.051	9.833 ± 0.238	0.016 ± 0.004
Regular Sintering LTD	32.973 ± 1.682	47.436 ± 0.947	2.076 ± 0.084	17. 376 ± 0.6930	0.1433 ± 0.024
Speed Sintering	34.260 ± 2.692	53.123 ± 1.831	1.346 ± 0.097	11.163 ± 0.906	0.013 ± 0.004
Speed Sintering LTD	28.190 ± 0.190	50.093 ± 0.339	2.316 ± 0.02	19.236 ± 0.182	0.166 ± 0.012

SD = Standard deviation.

1.2 Phase Structure

The XRD analysis (Figure 14) of the zirconia specimens shows that the zirconium specimens showed that the peak positions for the spectra correspond to the t phase for zirconium yttrium oxides (ZrO_2).

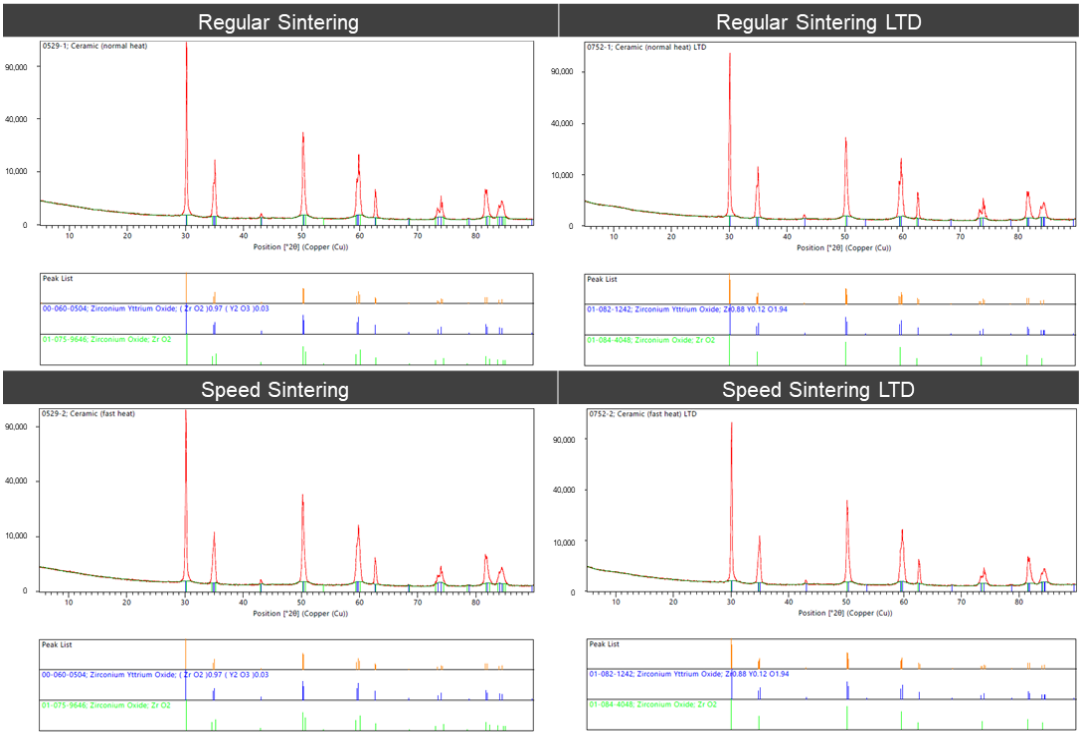


Figure 14. X-ray diffraction (XRD) analysis of the zirconia specimens for regular sintering and speed sintering with and without low-temperature degradation (LTD).

1.3 Translucency

The results of the transparency are shown in Table 10. Regular sintering showed more translucency compared to speed sintering. Multiple comparisons of the contrast ratio between the groups show that there was a significant difference (p value < 0.05) between the various groups. However, the translucency parameter presented a significant difference (p value < 0.05) between the various groups except for between speed sintering vs speed sintering LTD (p value = 0.931).

Table 10. Results of the contrast ratios transparency, and biaxial flexural strength of the zirconia specimens.

Zirconia Specimens	Contrast Ratio (Mean ± SD)	Translucency (Mean ± SD)	Flexural strength (MPa) (Mean ± SD)
Regular Sintering	0.787 ± 0.034	10.581 ± 0.798	466.41 ± 22.898
Regular Sintering LTD	0.734 ± 0.028	12.443 ± 1.173	471.85 ± 20.789
Speed Sintering	0.833 ± 0.021	9.052 ± 0.618	500.5 ± 23.432
Speed Sintering LTD	0.824 ± 0.252	9.263 ± 0.775	513.63 ± 19.909

SD = Standard deviation.

1.4 Biaxial Flexural Strength

The results of the flexural strength (MPa) are shown in Table 10. It showed that speed sintering and speed sintering LTD showed higher biaxial flexural strengths. Table 11 shows the multiple comparisons of flexural strength (MPa) among the groups. The regular sintering showed significantly lower flexural strength (p value ≤ 0.01) compared to the speed sintering and speed sintering LTD. Similarly, the regular sintering LTD showed a significantly lower flexural strength (p value < 0.005) compared to the speed sintering and speed sintering LTD. However, there was no difference in the flexural strength between the speed sintering and speed sintering LTD (p value = 0.444).

Table 11. Multiple comparisons of the biaxial flexural strength of the zirconia specimens among various groups.

(I) Groups	(J) Groups	Mean Difference (I-J)	*p* value
Regular Sintering	Regular Sintering LTD	−5.436	0.926
	Speed Sintering	−34.097 *	0.001 *
	Speed Sintering LTD	−47.219*	<0.0001 *
Regular Sintering LTD	Regular Sintering	5.436	0.926
	Speed Sintering	−28.661 *	0.008 *
	Speed Sintering LTD	−41.783 *	<0.0001 *
Speed Sintering	Regular Sintering	34.097 *	0.001 *
	Regular Sintering LTD	28.661 *	0.008 *
	Speed Sintering LTD	−13.122	0.444
Speed Sintering LTD	Regular Sintering	47.219 *	<0.0001 *
	Regular Sintering LTD	41.783 *	<0.0001 *
	Speed Sintering	13.122	0.444

* The sigificant difference at the 0.05 level. Multiple comparisons done using Scheffe analysis.

2. Results of Implant Crown

2.1 Fractographic Analysis

The surface of various zirconia crowns of various sintering groups is shown in Figure 15. It shows that, for all samples, the crack lines run from the top (occlusal) to the bottom (gingiva). Indeed, the arrest lines are perpendicular to the crack propagations. For the SS group, the material looks more uniform, and the crack lines are present at shorter distances compared to RS; this concurs with the result that SS provides higher strength than RS. Once the material was fatigued, more crack lines were presented. The direction of crack lines in the fatigue group is presented in different directions compared to the non-fatigue group. In this study, all zirconia crowns were presented with bulk catastrophic material as shown in Figure 16.

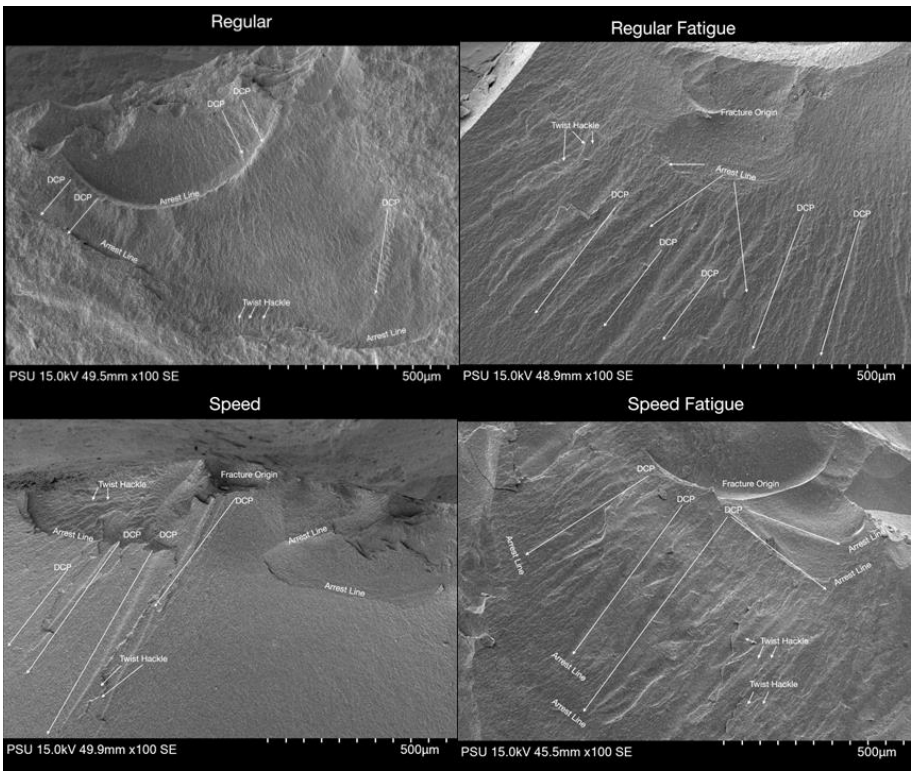


Figure 15. Surface structure in SEM of the zirconia crowns of various sintering groups.

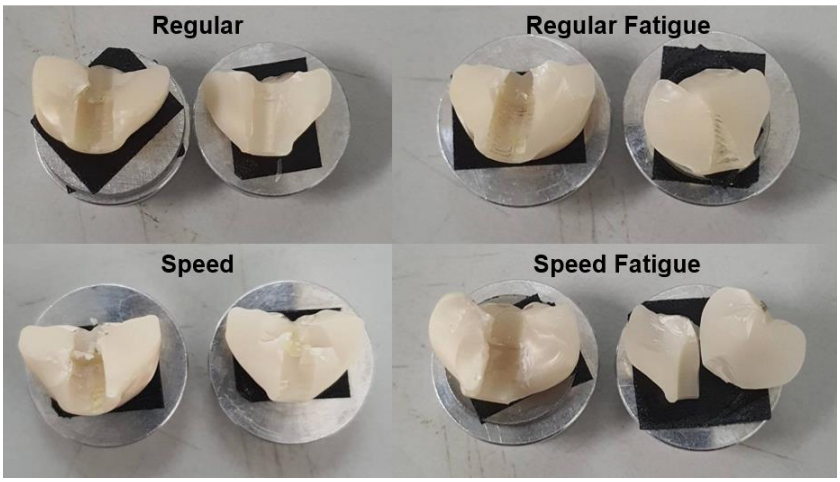


Figure 16. Fracture of the zirconia crowns of various sintering groups.

2.2 Load to Failure

The descriptive statistics of the fracture load are shown in Table 12. It shows that the speed group showed the maximum fracture load, followed by the regular, speed fatigue, and regular fatigue groups. The multiple comparisons show that the fracture load was a significant difference among the various groups; regular vs regular fatigue, regular vs speed, regular vs speed fatigue, regular fatigue vs speed, regular fatigue vs speed fatigue, and speed vs speed fatigue (Table 13).

Table 12. Descriptive statistics of fracture load.

Sintering	N	Mean (N)	Std. Deviation	Std. Error	Minimum	Maximum
Regular	15	3223.60	76.11	19.65	3146.00	3456.00
Regular fatigue	15	2143.06	135.93	35.09	1783.00	2324.00
Speed	15	3664.46	140.90	36.38	3276.00	3787.00
Speed fatigue	15	2450.06	128.26	33.12	2290.00	2790.00

Table 13. Multiple comparisons of the fracture load among the groups.

(I) Groups	(J) Groups	Mean Difference (I-J)	*p* Value
Regular	Regular fatigue	1080.53	<0.001 *
	Speed	−440.86	<0.001 *
	Speed fatigue	773.53	<0.001 *
Regular fatigue	Regular	−1080.53	<0.001 *
	Speed	−1521.40 *	<0.001 *
	Speed fatigue	−307.00	<0.001 *
Speed	Regular	440.86	<0.001 *
	Regular fatigue	1521.40	<0.001 *
	Speed fatigue	1214.40	<0.001 *
Speed fatigue	Regular	−773.53	<0.001 *
	Regular fatigue	307.00	<0.001 *
	Speed	−1214.40	<0.001 *

* The mean difference is significant at the 0.05 level.

Figure 17 shows the Weibull distribution for the zirconia crowns. The 63.3% value was also marked, for which the strength according to the analysis is σ_0 = 3244 MPa for regular, σ_0 = 2190 MPa for regular fatigue, σ_0 = 3765 MPa for speed, and σ_0 = 2457 MPa for speed fatigue (Table 14). It shows that only the regular group shows an R^2 of less than 65%. The sintering protocol with a larger Weibull module and durability increases reliability.

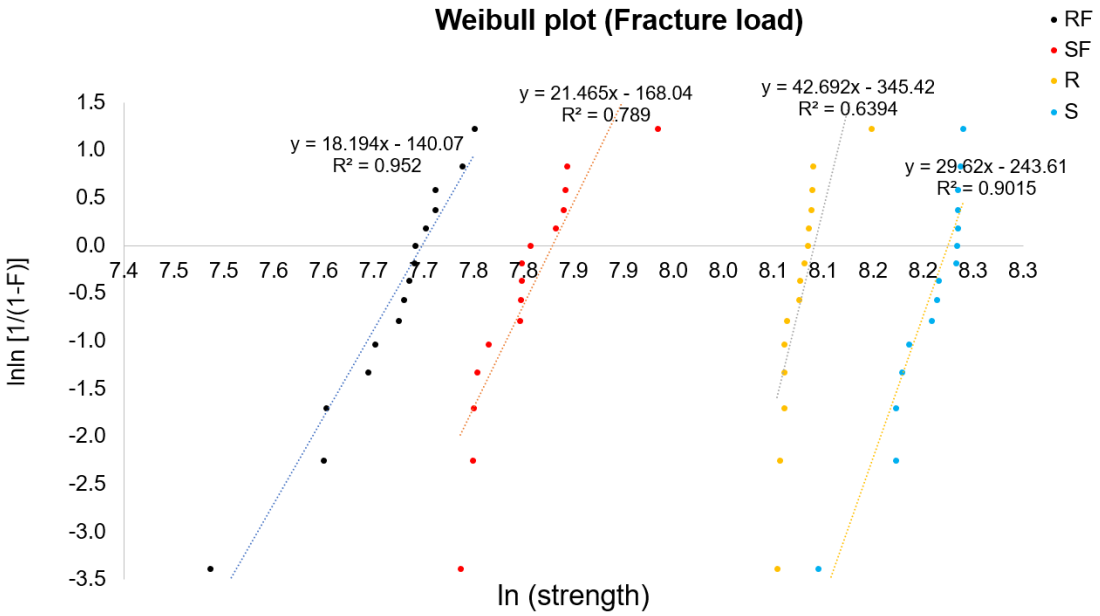


Figure 17. Weibull plot for the strength of the zirconia crowns of various sintering groups. R = regular, S = speed sintering, RF = regular fatigue, SF = speed fatigue.

Table 14. Results of Weibull distribution analysis.

Sintering Protocol	N	Characteristic strength σ_o [MPa]	Coefficient of determination R2	Weibull modulus m
Regular	15	3244	0.64	42.69
Regular fatigue	15	2190	0.95	18.19
Speed	15	3765	0.90	29.62
Speed fatigue	15	2457	0.79	21.46

Discussion

1. Zirconia Disc

Zirconia has good wear resistance and good color stability.[71] There have developed various sintering methods to enhance the properties and esthetics of zirconia. As there was no study that studied the speed sintering with aging or LTD. Sintering times with LTD aging affects the structural, optical, and mechanical properties of zirconia.[72] Hence, it was important to study the surface structure, phase determination, translucency, and flexural strength of zirconia between regular sintering and speed sintering. So, this research was done to compare the surface structure, phase determination, translucency, and flexural strength of zirconia blocks and discs (5Y-TZP) between the regular speed sintering and the speed sintering with and without LTD. In this study, we rejected the null hypothesis as there was a difference in the surface structure, grain size, contrast ratio, and biaxial flexural strength in zirconia between the regular sintering and the speed sintering with and without LTD. In this study, regular sintering with LTD showed the most translucency. From XRD, there was no difference in all groups. All the peaks were the same; however, the translucencies are different, which could be due to the difference in the grain size.[73] In addition, in this study, for speed sintering, we can notice a smaller grain size. The smaller grain of the m lattice may cause less light transmission. Liu et al.[74] also found conventionally-sintered Y-PSZ had a larger average grain size than speed-sintered Y-PSZ.

Furthermore, Kilinc et al.[72] studied the various sintering methods and aging on the grain size, flexural strength, and translucency of the zirconia and they found that the sintering method and aging significantly influence the translucency. The flexural strength and grain sizes were influenced by aging only ($p < 0.001$). Aging times increased the grain size but prolonged sintering with 120 min of aging negatively influenced the translucency of zirconia. The increase in grain size compared to our research might be due to the shorter aging time (60 min and 120 min), but in our research, the samples were subjected to 122 °C under 2-bar pressure for 8 h. Hence, prolonged aging reduces grain size.

In this study, the size of the zirconium samples before sintering was 1.226 times bigger than the size of the zirconium samples after sintering. This bigger size before sintering is to compensate for the shrinkage following the sintering (enlargement factor of 1.226). In addition, the dimensions of the zirconia samples of both sintering protocols were similar. Ahmed et al.[75] also found that there is no difference in the linear and dimensional changes between standard and fast sintering methods.

Liu et al.[76] studied the optical properties of two generations of rapid sintered translucent zirconia (3Y-TZP and 5Y-TZP) and they found that rapid sintering resulted in reduced lightness but did not affect the surface roughness. They concluded that rapid sintering is a practical method of reducing the production time of zirconia restorations.

In our study, flexural strength was affected by the sintering method. These results could be due to the smaller grain size of zirconia from speed sintering. These

results were similar to Ersoy et al.[77], who found that both a high and low sintering temperature combination increases the zirconia's flexural strength, but there were no visible differences in the grain size in the zirconia specimens. Stawarczyk et al.[66] mentioned that the grain size of zirconia increases with the sintering temperatures and the sintering temperature significantly affects the flexural strength.

When heating or LTD, the m phase will transform into a t and c phase according to the temperature, and when cooling, it will transform back to m, which is not strong. Producing stable sintered zirconia ceramic is difficult as the large volume change occurs while transition from t to m (approximately 5%). Hence, yttria is added to stabilize in the t phase.[78,79]

Arcila et al.[80] characterized the microstructure of three types of zirconia; 3Y-TZP (yttria-t *zirconia* polycrystal), 4Y-PSZ, and 5Y-PSZ (yttria-partially stabilized zirconia) and compared their hardness, fracture resistance, and fatigue and flexural strength. Three zirconia used were 3Y-TZP (Vita YZ HT), 4Y-PSZ (Vita YZ ST), and 5Y-PSZ (Vita YZ XT). The 4Y-PSZ and 5Y-PSZ specimens presented some surface defects under the SEM, whereas the 3Y-TZP demonstrated a greater grain consistency on the surface. They concluded that despite the structural differences, 4Y-PSZ and 3Y-TZP had similar fatigue. The 5Y-PSZ had the least mechanical strength.

Finally, in this study, only one brand of zirconia was used, and different manufacturers may have presented different grain sizes for sintering zirconia. Future studies can be done to study the grain size and its chemistry.

2. Implant Crown

We analyzed the zirconia's survival using the Weibull approach. A low Weibull modulus shows more variability in the strength (σ_c), whereas a high Weibull modulus signifies higher reliability of zirconia material. Weibull's theory is based on the weakest link theory whereby failure is the result of the weakest line break in the chain.[81]

The crack propagation mechanism in zirconia materials differs according to the type of material composition and sintering protocols. For the two material types, it was found that an intergranular fracture is seen in 3Y-TZP and a combination of intergranular and intragranular crack propagation is seen in 5Y-TZP.[82] When a micro-crack is created during the manufacturing process, the failure will start.

Our results were similar to the results obtained by some previous studies.[83-85] Jerman et al.[85] found that the Weibull modulus of the three thermomechanically aged materials was negatively influenced by high-speed sintering; hence, they also mentioned that high-speed sintering is presents an alternative to conventional sintering.

Arcila et al. [80] studied the microstructure of 3 yttria partially stabilized (3Y-PSZ) disc-shaped zirconia and compared the fracture resistance, hardness, and fatigue flexural strength of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. They demonstrated that all zirconia materials show similar compositions but vary the yttria content (5Y-PSZ > 4Y-PSZ > 3Y-TZP). They concluded that despite the microstructural differences, 3Y-TZP and

4Y-PSZ showed similar fatigue behavior whereas 5Y-PSZ had the least fatigue behavior.

Ordoñez Balladares et al.[86] compared the fracture strength of monolithic Zr dioxide after sintering in two different furnaces: CEREC SpeedFire (fast sintering) and InFire HTC Speed (slow sintering) produced from CAD/CAM. They found that the CEREC SpeedFire presented 1222.8 N fracture strength, whereas InFire HTC Speed showed 1068.5 N fracture strength, but no significant differences among the groups. The different furnaces did not affect the strengths of the zirconia and there is time saving when using rapid sintering. However, speed-sintered restorations may have limited reliability. According to a previous article, super-speed sintering is considered an option [87]; however, in the esthetic area where translucency is required, super-speed sintering might not be suitable.

The thickness reduction of zirconia and fatigue affects the failure load of monolithic zirconia crowns. Prott et al.[88] mentioned that less thickness of the crown leads to less strength, even if the failure loads surpass the chewing forces. Fatigue significantly lessened the failure load of 0.5 mm 3Y-TZP crowns.

Regarding the limitations of this research, we should note the limited sample size. For the regular group, the R^2 might be higher if we cut out the samples or add more samples. This research is preliminary. Further research can be carried out to analyze the chemical chain or strengthening phenomena in zirconia. In addition, in this research, we did not compare the shades of the zirconia block. Further research can be done to try different block colors and see if there are any differences in translucency results. Also, the color change of zirconia should be investigated and compared between regular sintering and speed sintering.

Concluding remarks

Within the limitations of this study, the following are conclusions.

1. There was a difference in surface structure, translucency, and biaxial flexural strengths. Regular sintering showed a bigger grain size and slightly more translucency compared to speed sintering. The speed sintering showed higher biaxial flexural strengths compared to regular sintering. This shows that speed sintering can be considered a suitable method of sintering zirconia. Hence, when biaxial flexural strength is required, speed sintering can be considered; however, when better translucency is required, regular sintering is recommended.
2. Surface flaws are the failure origin, and the crack lines ran from the occlusal to the bottom. Handling of the zirconia plays an important role in the longevity of the zirconia restorations. It was found that the speed sintering group showed the maximum fracture load followed by the regular, speed fatigue, and regular fatigue groups. These values provide reference fracture load values for the implant crown and fixed partial denture and are used to assess the durability of zirconia in dentistry.

Bibliography

1. Kelly, J.R.; Nishimura, I.; Campbell, S.D. Ceramics in dentistry: Historical roots and current perspectives. *The Journal of prosthetic dentistry* **1996**, *75*, 18-32.
2. Anusavice, K.J. Recent developments in restorative dental ceramics. *Journal of the American Dental Association (1939)* **1993**, *124*, 72-74, 76-78, 80-74.
3. Grech, J.; Antunes, E. Zirconia in dental prosthetics: A literature review. *J Mater Res Technol* **2019**, *8*, 4956-4964.
4. Tanzi, M.C.; Farè, S.; Candiani, G. Chapter 4 - biomaterials and applications. In *Foundations of biomaterials engineering*, Tanzi, M.C.; Farè, S.; Candiani, G., Eds. Academic Press: 2019; pp 199-287.
5. Ivvala, J.; Sharma, V.; Kuriachen, B. *A review on the selection of dental implant material and suitable additive manufacturing technology in dentistry*. 2019.
6. Saridag, S.; Tak, O.; Alniacik, G. Basic properties and types of zirconia: An overview. *World Journal of Stomatology* **2013**, *2*, 40-47.
7. Fischman, G. 1.3.4b - structural ceramic oxides. In *Biomaterials science*, 4th ed.; Wagner, W.R.; Sakiyama-Elbert, S.E.; Zhang, G.; Yaszemski, M.J., Eds. Academic Press: 2020; pp 319-326.
8. Piconi, C.; Maccauro, G. Zirconia as a ceramic biomaterial. *Biomaterials* **1999**, *20*, 1-25.
9. Farid, S.B.H. 1 - overview. In *Bioceramics: For materials science and engineering*, Farid, S.B.H., Ed. Woodhead Publishing: 2019; pp 1-37.
10. Vitti, R.P.; Catelan, A.; Amaral, M.; Pacheco, R.R. 14 - zirconium in dentistry. In *Advanced dental biomaterials*, Khurshid, Z.; Najeeb, S.; Zafar, M.S.; Sefat, F., Eds. Woodhead Publishing: 2019; pp 317-345.
11. Malkondu, Ö.; Tinastepe, N.; Akan, E.; Kazazoğlu, E. An overview of monolithic zirconia in dentistry. *Biotechnol Biotechnol Equip* **2016**, *30*, 644-652.
12. Reveron, H.; Fornabaio, M.; Palmero, P.; Fürderer, T.; Adolfsson, E.; Lughi, V.; Bonifacio, A.; Sergo, V.; Montanaro, L.; Chevalier, J. Towards long lasting zirconia-based composites for dental implants: Transformation induced plasticity and its consequence on ceramic reliability. *Acta biomaterialia* **2017**, *48*, 423-432.
13. Kohorst, P.; Borchers, L.; Strempel, J.; Stiesch, M.; Hassel, T.; Bach, F.-W.; Hübsch, C. Low-temperature degradation of different zirconia ceramics for dental applications. *Acta biomaterialia* **2012**, *8*, 1213-1220.
14. Guo, X. Property degradation of tetragonal zirconia induced by low-temperature defect reaction with water molecules. *Chem Mater* **2004**, *16*, 3988-3994.
15. Cotes, C.; Arata, A.; Melo, R.M.; Bottino, M.A.; Machado, J.P.B.; Souza, R.O.A. Effects of aging procedures on the topographic surface, structural stability, and mechanical strength of a zro2-based dental ceramic. *Dental materials : official publication of the Academy of Dental Materials* **2014**, *30*, e396-e404.

16. Lameira, D.P.; Silva, W.A.B.e.; Silva, F.A.e.; De Souza, G.M. Fracture strength of aged monolithic and bilayer zirconia-based crowns. *BioMed Res Int* **2015**, *2015*, 418641.
17. Camposilvan, E.; Marro, F.G.; Mestra, A.; Anglada, M. Enhanced reliability of yttria-stabilized zirconia for dental applications. *Acta biomaterialia* **2015**, *17*, 36-46.
18. Monaco, C.; Tucci, A.; Esposito, L.; Scotti, R. Microstructural changes produced by abrading y-tzp in presintered and sintered conditions. *J Dent* **2013**, *41*, 121-126.
19. Lucas, T.J.; Lawson, N.C.; Janowski, G.M.; Burgess, J.O. Effect of grain size on the monoclinic transformation, hardness, roughness, and modulus of aged partially stabilized zirconia. *Dental materials : official publication of the Academy of Dental Materials* **2015**, *31*, 1487-1492.
20. Stawarczyk, B.; Frevert, K.; Ender, A.; Roos, M.; Sener, B.; Wimmer, T. Comparison of four monolithic zirconia materials with conventional ones: Contrast ratio, grain size, four-point flexural strength and two-body wear. *J Mech Behav Biomed Mater* **2016**, *59*, 128-138.
21. Śnieżek, E.; Szczerba, J.; Stoch, P.; Prorok, R.; Jastrzębska, I.; Bodnar, W.; Burkel, E. Structural properties of mgo–zro2 ceramics obtained by conventional sintering, arc melting and field assisted sintering technique. *Materials & Design* **2016**, *99*, 412-420.
22. Pereira, G.K.R.; Silvestri, T.; Camargo, R.; Rippe, M.P.; Amaral, M.; Kleverlaan, C.J.; Valandro, L.F. Mechanical behavior of a y-tzp ceramic for monolithic restorations: Effect of grinding and low-temperature aging. *Mater Sci Eng C* **2016**, *63*, 70-77.
23. Stawarczyk, B.; Keul, C.; Eichberger, M.; Figge, D.; Edelhoff, D.; Lümkemann, N. Three generations of zirconia: From veneered to monolithic. Part i. *Quintessence international (Berlin, Germany : 1985)* **2017**, *48*, 369-380.
24. Conrad, H.J.; Seong, W.J.; Pesun, I.J. Current ceramic materials and systems with clinical recommendations: A systematic review. *The Journal of prosthetic dentistry* **2007**, *98*, 389-404.
25. Hjerppe, J.; Närhi, T.; Fröberg, K.; Vallittu, P.K.; Lassila, L.V. Effect of shading the zirconia framework on biaxial strength and surface microhardness. *Acta odontologica Scandinavica* **2008**, *66*, 262-267.
26. Heffernan, M.J.; Aquilino, S.A.; Diaz-Arnold, A.M.; Haselton, D.R.; Stanford, C.M.; Vargas, M.A. Relative translucency of six all-ceramic systems. Part i: Core materials. *The Journal of prosthetic dentistry* **2002**, *88*, 4-9.
27. Heffernan, M.J.; Aquilino, S.A.; Diaz-Arnold, A.M.; Haselton, D.R.; Stanford, C.M.; Vargas, M.A. Relative translucency of six all-ceramic systems. Part ii: Core and veneer materials. *The Journal of prosthetic dentistry* **2002**, *88*, 10-15.
28. Brodbelt, R.H.; O'Brien, W.J.; Fan, P.L. Translucency of dental porcelains. *J Dent Res* **1980**, *59*, 70-75.
29. Farid, S.B.H. 2 - structure, microstructure, and properties of bioceramics. In *Bioceramics: For materials science and engineering*, Farid, S.B.H., Ed. Woodhead Publishing: 2019; pp 39-76.

30. Katana* zirconia stml. Https://www.Bego.Com/cad-cam-solutions/materials/ceramic/katana-zirconia-stml/?Fbclid=iwar16ehpx-z-hvl3vqlwccjwv3icma_sg0e8qdrs2ysvb8k89bu8hxe0frny.
31. Apratim, A.; Eachempati, P.; Krishnappa Salian, K.K.; Singh, V.; Chhabra, S.; Shah, S. Zirconia in dental implantology: A review. *J Int Soc Prev Community Dent* **2015**, *5*, 147-156.
32. Özkurt, Z.; Kazazoğlu, E. Zirconia dental implants: A literature review. *The Journal of oral implantology* **2011**, *37*, 367-376.
33. Sivaraman, K.; Chopra, A.; Narayan, A.I.; Balakrishnan, D. Is zirconia a viable alternative to titanium for oral implant? A critical review. *Journal of prosthodontic research* **2018**, *62*, 121-133.
34. Depprich, R.; Zipprich, H.; Ommerborn, M.; Naujoks, C.; Wiesmann, H.P.; Kiattavorncharoen, S.; Lauer, H.C.; Meyer, U.; Kübler, N.R.; Handschel, J. Osseointegration of zirconia implants compared with titanium: An in vivo study. *Head & face medicine* **2008**, *4*, 30.
35. Zhang, Y. Making yttria-stabilized tetragonal zirconia translucent. *Dental Materials* **2014**, *30*, 1195-1203.
36. Zhang, F.; Inokoshi, M.; Batuk, M.; Hadermann, J.; Naert, I.; Van Meerbeek, B.; Vleugels, J. Strength, toughness and aging stability of highly-translucent y-tzp ceramics for dental restorations. *Dental materials : official publication of the Academy of Dental Materials* **2016**, *32*, e327-e337.
37. Peuchert, U.; Okano, Y.; Menke, Y.; Reichel, S.; Ikesue, A. Transparent cubic-zro2 ceramics for application as optical lenses. *J Eur Ceram Soc* **2009**, *29*, 283-291.
38. Tinschert, J.; Natt, G.; Hassenpflug, S.; Spiekermann, H. Status of current cad/cam technology in dental medicine. *International journal of computerized dentistry* **2004**, *7*, 25-45.
39. Luthardt, R.G.; Holzhüter, M.S.; Rudolph, H.; Herold, V.; Walter, M.H. Cad/cam-machining effects on y-tzp zirconia. *Dental materials : official publication of the Academy of Dental Materials* **2004**, *20*, 655-662.
40. Kohorst, P.; Brinkmann, H.; Li, J.; Borchers, L.; Stiesch, M. Marginal accuracy of four-unit zirconia fixed dental prostheses fabricated using different computer-aided design/computer-aided manufacturing systems. *European journal of oral sciences* **2009**, *117*, 319-325.
41. Mayo, M.J. Processing of nanocrystalline ceramics from ultrafine particles. *Int Mater Rev* **1996**, *41*, 85-115.
42. Chu, M.-Y.; De Jonghe, L.C.; Lin, M.K.F.; Lin, F.J.T. Precoarsening to improve microstructure and sintering of powder compacts. *J Am Ceram Soc* **1991**, *74*, 2902-2911.
43. Li, L.; Zhao, C.; Du, Z.; Qiu, Y.; Si, W. Rapid-sintered dental zirconia for chair-side one-visit application. *Int J Appl Ceram Technol* **2019**, *16*, 1830-1835.
44. Kaizer, M.R.; Gierthmuehlen, P.C.; Dos Santos, M.B.; Cava, S.S.; Zhang, Y. Speed sintering translucent zirconia for chairside one-visit dental restorations: Optical, mechanical, and wear characteristics. *Ceram Int* **2017**, *43*, 10999-11005.

45. Zimmermann, A.; Hoffman, M.; Flinn, B.D.; Bordia, R.K.; Chuang, T.-J.; Fuller Jr., E.R.; Rödel, J. Fracture of alumina with controlled pores. *J Am Ceram Soc* **1998**, *81*, 2449-2457.
46. Hoffman, M.; Rödel, J. Suggestion for mechanism of strengthening of 'nanotoughened' ceramics. *J Ceram Soc Japan* **1997**, *105*, 1086-1090.
47. Zimmermann, A.; Rödel, J. Generalized orowan-petch plot for brittle fracture. *J Am Ceram Soc* **1998**, *81*, 2527-2532.
48. Amat, N.F.; Muchtar, A.; Amril, M.S.; Ghazali, M.J.; Yahaya, N. Effect of sintering temperature on the aging resistance and mechanical properties of monolithic zirconia. *Journal of Materials Research and Technology* **2019**, *8*, 1092-1101.
49. Rekow, E.D.; Silva, N.R.F.A.; Coelho, P.G.; Zhang, Y.; Guess, P.; Thompson, V.P. Performance of dental ceramics: Challenges for improvements. *Journal of dental research* **2011**, *90*, 937-952.
50. Chevalier, J.; Cales, B.; Drouin, J.M. Low-temperature aging of y-tzp ceramics. *Journal of the American Ceramic Society* **1999**, *82*, 2150-2154.
51. Dehestani, M.; Adolfsson, E. Phase stability and mechanical properties of zirconia and zirconia composites. *Int J Appl Ceram Technol* **2013**, *10*, 129-141.
52. Yang, H.; Ji, Y. Low-temperature degradation of zirconia-based all-ceramic crowns materials: A mini review and outlook. *J Mater Sci Technol* **2016**, *32*, 593-596.
53. Sato, T.; Shimada, M. Transformation of yttria-doped tetragonal zro2 polycrystals by annealing in water. *J Am Ceram Soc* **1985**, *68*, 356-356.
54. Kobayashi, K.; Kuwajima, H.; Masaki, T. Phase change and mechanical properties of zro2-y2o3 solid electrolyte after ageing. *Solid State Ionics* **1981**, *3-4*, 489-493.
55. Zhuang, Y.; Zhu, Z.; Jiao, T.; Sun, J. Effect of aging time and thickness on low-temperature degradation of dental zirconia. *J Prosthodont* **2019**, *28*, e404-e410.
56. Haraguchi, K.; Sugano, N.; Nishii, T.; Miki, H.; Oka, K.; Yoshikawa, H. Phase transformation of a zirconia ceramic head after total hip arthroplasty. *The Journal of bone and joint surgery. British volume* **2001**, *83*, 996-1000.
57. Lawson, S. Environmental degradation of zirconia ceramics. *J Eur Ceram Soc* **1995**, *15*, 485-502.
58. Alghazzawi, T.F.; Lemons, J.; Liu, P.R.; Essig, M.E.; Bartolucci, A.A.; Janowski, G.M. Influence of low-temperature environmental exposure on the mechanical properties and structural stability of dental zirconia. *J Prosthodont* **2012**, *21*, 363-369.
59. Kengtanyakich, S.; Peampring, C. An experimental study on hydrothermal degradation of cubic-containing translucent zirconia. *The journal of advanced prosthodontics* **2020**, *12*, 265-272.
60. Flinn, B.D.; deGroot, D.A.; Mancl, L.A.; Raigrodski, A.J. Accelerated aging characteristics of three yttria-stabilized tetragonal zirconia polycrystalline dental materials. *The Journal of prosthetic dentistry* **2012**, *108*, 223-230.
61. Flinn, B.D.; Raigrodski, A.J.; Mancl, L.A.; Toivola, R.; Kuykendall, T. Influence of aging on flexural strength of translucent zirconia for monolithic restorations. *The Journal of prosthetic dentistry* **2017**, *117*, 303-309.

62. Ebeid, K.; Wille, S.; Hamdy, A.; Salah, T.; El-Etreby, A.; Kern, M. Effect of changes in sintering parameters on monolithic translucent zirconia. *Dent Mater J* **2014**, *30*, e419-e424.
63. Chevalier, J. What future for zirconia as a biomaterial? *Biomaterials* **2006**, *27*, 535-543.
64. Kim, M.-J.; Ahn, J.-S.; Kim, J.-H.; Kim, H.-Y.; Kim, W.-C. Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency. *The journal of advanced prosthodontics* **2013**, *5*, 161-166.
65. Jiang, L.; Liao, Y.; Wan, Q.; Li, W. Effects of sintering temperature and particle size on the translucency of zirconium dioxide dental ceramic. *Journal of materials science. Materials in medicine* **2011**, *22*, 2429-2435.
66. Stawarczyk, B.; Ozcan, M.; Hallmann, L.; Ender, A.; Mehl, A.; Hämmerlet, C.H. The effect of zirconia sintering temperature on flexural strength, grain size, and contrast ratio. *Clinical oral investigations* **2013**, *17*, 269-274.
67. Lin, F.J.T.; de Jonghe, L.C.; Rahaman, M.N. Microstructure refinement of sintered alumina by a two-step sintering technique. *Am Ceram Soc* **1997**, *80*, 2269-2277.
68. De Souza, G.M.; Zykus, A.; Ghahnavyeh, R.R.; Lawrence, S.K.; Bahr, D.F. Effect of accelerated aging on dental zirconia-based materials. *J Mech Behav Biomed Mater* **2017**, *65*, 256-263.
69. Rosentritt, M.; Hahnel, S.; Engelhardt, F.; Behr, M.; Preis, V. In vitro performance and fracture resistance of cad/cam-fabricated implant supported molar crowns. *Clinical oral investigations* **2017**, *21*, 1213-1219.
70. Mokhtarpour, H.; Eftekhar Ashtiani, R.; Mahshid, M.; Tabatabaian, F.; Alikhasi, M. Effect of screw access hole preparation on fracture load of implant-supported zirconia-based crowns: An in vitro study. *J Dent Res Dent Clin Dent Prospects* **2016**, *10*, 181-188.
71. Abhay, S.S.; Ganapathy, D.; Veeraiyan, D.N.; Ariga, P.; Heboyan, A.; Amornvit, P.; Rokaya, D.; Srimaneepong, V. Wear resistance, color stability and displacement resistance of milled peek crowns compared to zirconia crowns under stimulated chewing and high-performance aging. *Polymers* **2021**, *13*, 3761.
72. Kilinc, H.; Sanal, F.A. Effect of sintering and aging processes on the mechanical and optical properties of translucent zirconia. *Journal of Prosthetic Dentistry* **2021**, *126*, 129.e121-129.e127.
73. Sulaiman, T.A.; Abdulmajeed, A.A.; Shahramian, K.; Lassila, L. Effect of different treatments on the flexural strength of fully versus partially stabilized monolithic zirconia. *Journal of Prosthetic Dentistry* **2017**, *118*, 216-220.
74. Liu, H.; Inokoshi, M.; Nozaki, K.; Shimizubata, M.; Nakai, H.; Cho Too, T.D.; Minakuchi, S. Influence of high-speed sintering protocols on translucency, mechanical properties, microstructure, crystallography, and low-temperature degradation of highly translucent zirconia. *Dental Materials* **2022**, *38*, 451-468.
75. Ahmed, W.M.; Troczynski, T.; Stojkova, B.J.; McCullagh, A.P.; Wyatt, C.C.; Carvalho, R.M. Dimensional changes of yttria-stabilized zirconia under different preparation designs and sintering protocols. *Journal of Prosthodontics* **2020**, *29*, 699-706.

76. Liu, Y.-C.; Lin, T.-H.; Lin, Y.-Y.; Hu, S.-W.; Liu, J.-F.; Yang, C.-C.; Yan, M. Optical properties evaluation of rapid sintered translucent zirconia with two dental colorimeters. *Journal of Dental Sciences* **2022**, *17*, 155-161.
77. Ersoy, N.M.; Aydoğdu, H.M.; Değirmenci, B.; Çökük, N.; Sevimay, M. The effects of sintering temperature and duration on the flexural strength and grain size of zirconia. *Acta biomaterialia odontologica Scandinavica* **2015**, *1*, 43-50.
78. Yanagida, H.; Koumoto, K.; Miyayama, M. *The chemistry of ceramics*. John Wiley & Sons: Oxford, UK, 1996.
79. Leib, E.W.; Vainio, U.; Pasquarelli, R.M.; Kus, J.; Czaschke, C.; Walter, N.; Janssen, R.; Müller, M.; Schreyer, A.; Weller, H.; Vossmeyer, T. Synthesis and thermal stability of zirconia and yttria-stabilized zirconia microspheres. *Journal of Colloid and Interface Science* **2015**, *448*, 582-592.
80. Arcila, L.V.C.; Ramos, N.C.; Campos, T.M.B.; Dapieve, K.S.; Valandro, L.F.; de Melo, R.M.; Bottino, M.A. Mechanical behavior and microstructural characterization of different zirconia polycrystals in different thicknesses. *Journal of Advanced Prosthodontics* **2021**, *13*, 385-395.
81. Nemeth, N.N.; Gyekenyesi, J.P.; Jadaan, O.J. *Lifetime reliability prediction of ceramic structures under transient thermomechanical loads*; 10/01, 2005; p 212505.
82. Liu, C.; Eser, A.; Albrecht, T.; Stournari, V.; Felder, M.; Heintze, S.; Broeckmann, C. Strength characterization and lifetime prediction of dental ceramic materials. *Dental materials : official publication of the Academy of Dental Materials* **2021**, *37*, 94-105.
83. Mayinger, F.; Pfefferle, R.; Reichert, A.; Stawarczyk, B. Impact of high-speed sintering of three-unit 3y-tzp and 4y-tzp fixed dental prostheses on fracture load with and without artificial aging. *The International journal of prosthodontics* **2021**, *34*, 47-53.
84. Wiedenmann, F.; Pfefferle, R.; Reichert, A.; Jerman, E.; Stawarczyk, B. Impact of high-speed sintering, layer thickness and artificial aging on the fracture load and two-body wear of zirconia crowns. *Dental materials : official publication of the Academy of Dental Materials* **2020**, *36*, 846-853.
85. Jerman, E.; Wiedenmann, F.; Eichberger, M.; Reichert, A.; Stawarczyk, B. Effect of high-speed sintering on the flexural strength of hydrothermal and thermo-mechanically aged zirconia materials. *Dental materials : official publication of the Academy of Dental Materials* **2020**, *36*, 1144-1150.
86. Ordoñez Balladares, A.; Abad-Coronel, C.; Ramos, J.C.; Martín Biedma, B.J. Fracture resistance of sintered monolithic zirconia dioxide in different thermal units. *Materials* **2022**, *15*, 2478.
87. Kongkiatkamon, S.; Peampring, C. Effect of speed sintering on low temperature degradation and biaxial flexural strength of 5y-tzp zirconia. *Molecules (Basel, Switzerland)* **2022**, *27*, 5272.
88. Prott, L.S.; Spitznagel, F.A.; Bonfante, E.A.; Malassa, M.A.; Gierthmuehlen, P.C. Monolithic zirconia crowns: Effect of thickness reduction on fatigue behavior and failure load. *The journal of advanced prosthodontics* **2021**, *13*, 269-280.

Appendices

Paper 1

Journal of *Functional Biomaterials*

MDPI

Article

Comparison of Regular and Speed Sintering on Low-Temperature Degradation and Fatigue Resistance of Translucent Zirconia Crowns for Implants: An In Vitro Study

Suchada Kongkiatkamon and Chaimongkon Peampring *

Department of Prosthetic Dentistry, Faculty of Dentistry, Prince of Songkla University, Hat Yai 90110, Songkhla, Thailand
* Correspondence: mongkon.5c@gmail.com

Abstract: Background: Although there are a few studies which compare fast and slow sintering in normal zirconia crowns, it is essential to compare the cracks and load-bearing capacity in zirconia screw-retained implant crowns between regular and speed sintering protocols. This research aimed to compare the surface structure, cracks, and load-bearing capacity in zirconia screw-retained implant crowns between regular sintering (RS) and speed sintering (SS) protocol with and without cyclic loading (fatigue). Methods: A total of 60 screw-retained crowns were fabricated from zirconia (Katana STML Block) by the CAD/CAM system. Then, 30 crowns were subjected to the RS protocol and 30 crowns were subjected to the SS protocol. Cyclic loading was done in half zirconia crowns (15 crowns in each group) using a chewing simulator CS-4.8/CS-4.4 at room temperature. The loading force was applied on the middle of the crowns by a metal stylus underwater at room temperature with a chewing simulator at an axial 50 N load for 240,000 cycles and lateral movement at 2 mm. Scanning electron microscopy was done to study the surface of the crowns and the cracks in the crowns of the regular and speed sintering protocols, with and without fatigue. Results: For the speed sintering group, the surface looks more uniform, and the crack lines are present at a short distance compared to regular sintering. The sintering protocol with a larger Weibull module and durability increases the reliability. It showed that the Speed group showed the maximum fracture load, followed by the regular, speed fatigue, and regular fatigue groups. The fracture load in various groups showed significant differences. Conclusions: It was found that the speed group showed the maximum fracture load followed by the regular, speed fatigue, and regular fatigue. The crack lines ran from occlusal to bottoms (gingiva) and the arrest lines were perpendicular to the crack propagations.

Keywords: all-ceramic; zirconia; dental materials; firing; sintering; speed sintering; prosthetic dentistry; biaxial flexural strength; Weibull

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1. Introduction

Zirconia ceramics are the choice of materials for esthetic dental restorations and prosthetic crowns and they are also commonly used for the fabrication of implant crowns [1]. Recently, the translucency of zirconia materials has improved, resulting in more esthetic restorations in prosthetic dentistry [2,3]. In addition, zirconia has high strength and high wear resistance, can resist masticatory forces, and is widely used for the stress-bearing posterior teeth [4–7]. Zirconia restorations also presents good biocompatibility [8,9]. Currently, the use of digital technologies, computer-aided design, and computer-aided manufacturing (CAD/CAM) have allowed the simplified production of precise and durable implant components resulting in high clinical success rates and outcomes [10,11]. The precise fit of the crown is affected by the design of the implant [12–14].

Translucent zirconia can be obtained by increasing translucency and decreasing opacity. Optimal translucency in zirconia can be obtained by increasing grain size, optimizing grain boundary region, and increasing yttria content to produce partially stabilized zirconia

with increased amounts of nonbirefringent cubic phase [15,16]. In addition, the opacity in zirconia can be decreased by sintering additives (i.e., alumina), reducing oxygen vacancies, defects, and pores, and controlling the sintering environment (pressure and temperature) [15,17]. It was found that as the translucency in zirconia increased, the mechanical properties (especially the flexural strength) were reduced [18].

Zirconia is subjected to various processes such as milling, grinding, and polishing during the manufacturing process. From these processes, surface and volume flaws are formed which differ according to the location, size, and orientation, resulting in variable strength [19–22]. These process-related microstructures and flaws can affect the fracture strengths of the restoration. Furthermore, the zirconia can undergo low-temperature degradation (LTD) and can result in damage to implant restoration [23].

Zirconia stabilized with 5 mol% yttria content (5Y-TZP) and possesses excellent mechanical properties and biocompatibility [24,25]. Although there are few studies which compare fast and slow sintering in normal zirconia crowns, it is essential to compare the cracks and load-bearing capacity in zirconia screw-retained implant crowns between regular and speed sintering protocols, and there are no studies focused on this comparison. Hence, this research compared the surface structure, cracks, and load-bearing capacity in zirconia screw-retained implant crowns fabricated from third generation zirconia under regular and speed sintering protocols, with and without cyclic loading (fatigue).

2. Materials and Methods

2.1. Construction of Screw-Retained Zirconia Crowns

The study overview is shown in Figure 1. A total of 56 sample sizes is calculated from the G*Power software with a power of 90%. A total of 60 screw-retained zirconia crowns were fabricated from the zirconia material (Katana STML Block as shown in Table 1) by the CAD/CAM system as explained by Nogueira et al. [26]. At first, dental implants (Astra Tech EV, diameter 4.2 mm) were placed in the dentoform at the mandibular first molar region. The implants were placed at the center of the tooth and platform 3 mm below the gingival line. All crowns were of the same size (4 mm thick measured from the Ti base abutment outer surfaces and 1.74 mm measured from the top of the Ti base abutment).

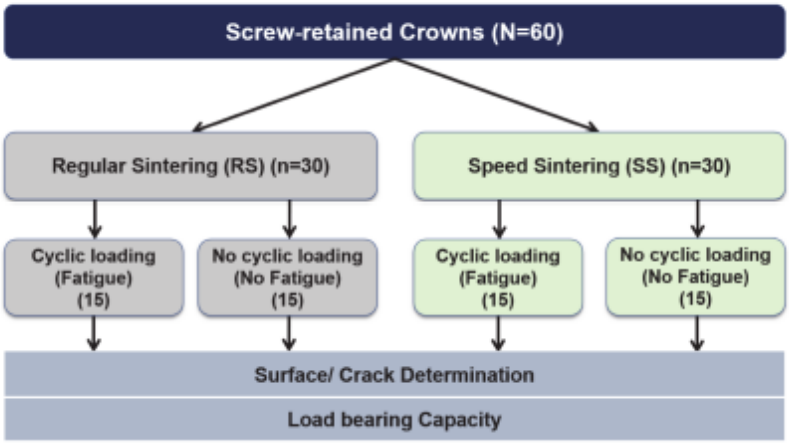

Figure 1. Overview of the study.

Scan post and scan body were utilized to transfer the information and position to the CAD software by using inLAb CAD Software 22.0 (Dentsply Sirona, Germany) (Figure 2). Ti-base GH 1 mm was selected. Sixty Crowns were milled by using MCXL and then sintered by using speed sintering (SS) protocol (n = 30) and regular sintering (RS) protocol (n = 30).

Table 1. Details of the zirconia used in this study [25,27].

Composition	
$ZrO_2 + HfO_2$	88–93 wt.%
Y_2O_3	7–10 wt.%
Other oxides	0–2 wt.%
Properties	
Flexural strength	748 MPa
Translucency	38%
Coefficient of thermal expansion	$9.8 \pm 0.2 \times 10^{-6}$ K^{-1}

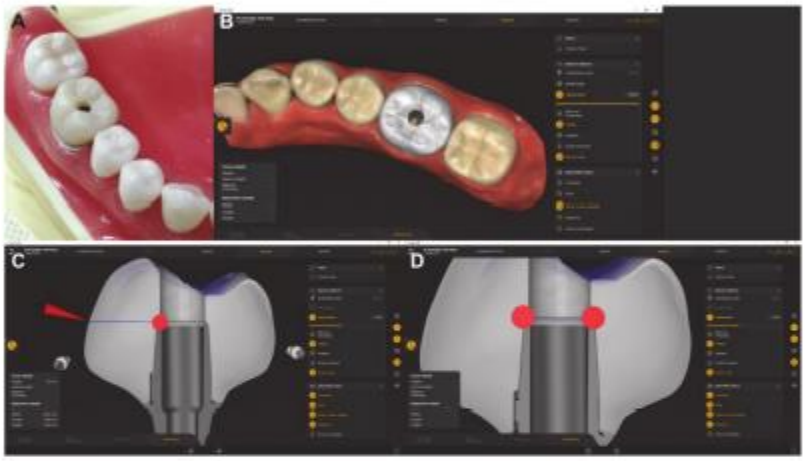

Figure 2. Screw-retained zirconia crowns designed from Omnicam software and completed screw-retained zirconia crown. (**A**) = study model, (**B**) = occlusal view in the software, (**C**) = sectional view of the implant crown, and (**D**) = screw hole measurement.

The zirconia screw-retained crowns were fabricated from zirconia block, then scanned by Cerec Omicam (Dentsply Sirona, Bensheim, Germany), designed by inLAb CAD Software 22.0 (Dentsply Sirona, Bensheim, Germany), and finally milled with an MCXL (Dentsply Sirona, Bensheim, Germany) milling unit (Figure 2). Then the crowns were sintered following the respective manufacturer's recommendations. For the RS group, the total thermal cycle, sintering time, and dwell temperature were 6.8 h, 2 h, and 1550 °C, respectively, by using inFire HTC Furnace (Dentsply Sirona, Bensheim, Germany). For the SS group, the total thermal cycle, sintering time, and dwell temperature were calculated based on each specimen, which was 30 min, 16 min, and 1560 °C, respectively, by using SpeedFire oven (Dentsply Sirona, Bensheim, Germany) [28]. In each group, half of the crowns were again subjected to fatigue using LTD by putting the autoclave at 122 °C under −2 bar pressure for 8 h, and half of the crowns were again subjected to no fatigue.

2.2. *Cementation of Screw-Retained Zirconia Crowns*

All crowns were cemented to Ti-base. Ti base and Zirconia crowns were sandblasted (50-micron 1 bar) for 5 s. Single Bond Universal (3MESPE, Saint Paul, MN, USA) was utilized to apply both crowns and Ti-base surfaces. Rely X Ultimate (3MESPE, Saint Paul, MN, USA) was utilized to cement following the manufacturer's instructions. Screw-retained crowns were torqued at 25 Ncm to implant analog. Teflon tape and resin composite Z350 (3MESPE, Saint Paul, MN, USA) were used to close access screw holes of 2.6 mm diameter.

2.3. Cyclic Loading of Screw-Retained Zirconia Crowns

The zirconia crowns (15 crowns in each group) were subjected to cyclic loading using the chewing simulator CS-4.8/CS-4.4 (CS 4.8, SD Mechatronic GmbH, Feldkirchen-Westerham, Germany) at room temperature. The technical specifications included traverse paths of the Z-axis approx. 120 mm, and X-axis approx. 50 mm, speed ranged between 1–60 mm/s in both axes, dimensions including thermocycling unit was 190 × 200 × 80 cm (length × height × depth), and weight load of maximum 10 kg per sample chamber. The loading force was applied on the middle of the crowns by a metal stylus underwater at room temperature with a chewing simulator at an axial 50 N load for 240,000 cycles (frequency of 1 Hz) and lateral movement at 2 mm (Figure 3A) [29].

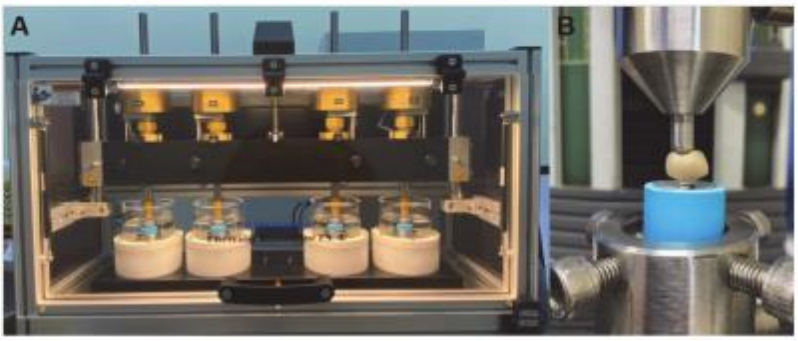

Figure 3. Chewing stimulator and flexural strength testing. (**A**) = Chewing stimulator and machine, (**B**): universal testing machine and set up of biaxial test method for the zirconia disc.

Then, the surface, cracks, and load-bearing capacity of the zirconia crowns were studied under regular and speed sintering protocols with and without fatigue.

2.4. Load to Failure

All zirconia crowns (fatigued and non-fatigued) were subjected to a single load-to-failure using the universal testing machine (Lloyd instruments, Model LRX-Plus, AMETEK Lloyd Instrument Ltd., Hampshire, UK) according to ISO 6872 [30]. An indenter consisting of a steel ball (6 mm diameter) was aligned at the same contact point as the dynamic loading with an increasing speed of 1 mm/min. Cracks on zirconia crowns and crown fractures (chipping cohesive fractures and/or bulk catastrophic fractures) were analyzed (Figure 3B).

2.5. Surface Characterization and Fractographic Analysis

The surface structures of the zirconia specimens were characterized using scanning electron microscopy (SEM) (FEI Quanta 400, FEI, Czech) of zirconia specimens (3 samples for each group). The specimens were submitted to sputter-coating with a gold-palladium alloy. The images were observed with an accelerating voltage of 20 kV in various magnifications. Selected crowns of each group were studied for the fractographic analysis with SEM (EVO10, Carl Zeiss, Oberkochen, Germany).

2.6. Weibull's Analysis

For the zirconia crowns, a probabilistic approach was done to analyze the strength and lifetime. The fracture strength of zirconia is done by the Weibull formula as shown in Equation (1) [31]:

$$P_f = 1 - \exp[-(\sigma_c - \sigma_0)^m] \quad (1)$$

where P_f = fracture probability, σ_0 = characteristic strength, σ_c = fast-fracture strength without subcritical crack growth, and m = Weibull modulus which is the slope of the regression line in the $\ln\sigma_c - \ln \ln [1/1 - P_f)]$ diagram [32]. σ_0 relates to the stress point

where 63.2% of the crowns would fracture. The fast-fracture strength of each crown was calculated by the formula shown in Equation (2) [20]:

$$\ln \ln \frac{1}{1 - P_f} = m \ln \sigma_c - m \ln \sigma_0. \quad (2)$$

2.7. Statistical Analysis

Descriptive statistics were calculated, and the results were compared among the sintering groups. The statistical analysis was done using SPSS 20.0 (SPSS Inc., IBM Corporation, Chicago, IL, USA) to see the significant differences (p value = 0.05). One-way ANOVA with multiple comparisons using the Scheffe analysis was done to compare the results among various groups.

3. Results

3.1. Fractographic Analysis

The surface of various zirconia crowns of various sintering groups is shown in Figure 4. Figure 4 shows that, for all samples, the crack lines run from the top (occlusal) to the bottom (gingiva). Indeed, the arrest lines are perpendicular to the crack propagations. For the SS group, the material looks more uniform, and the crack lines are present at shorter distances compared to RS; this concurs with the result that SS provides higher strength than RS. Once the material was fatigued, more crack lines were presented. The direction of crack lines in the fatigue group is presented in different directions compared to the non-fatigue group. In this study, all zirconia crowns presented with bulk catastrophic material as shown in Figure 5.

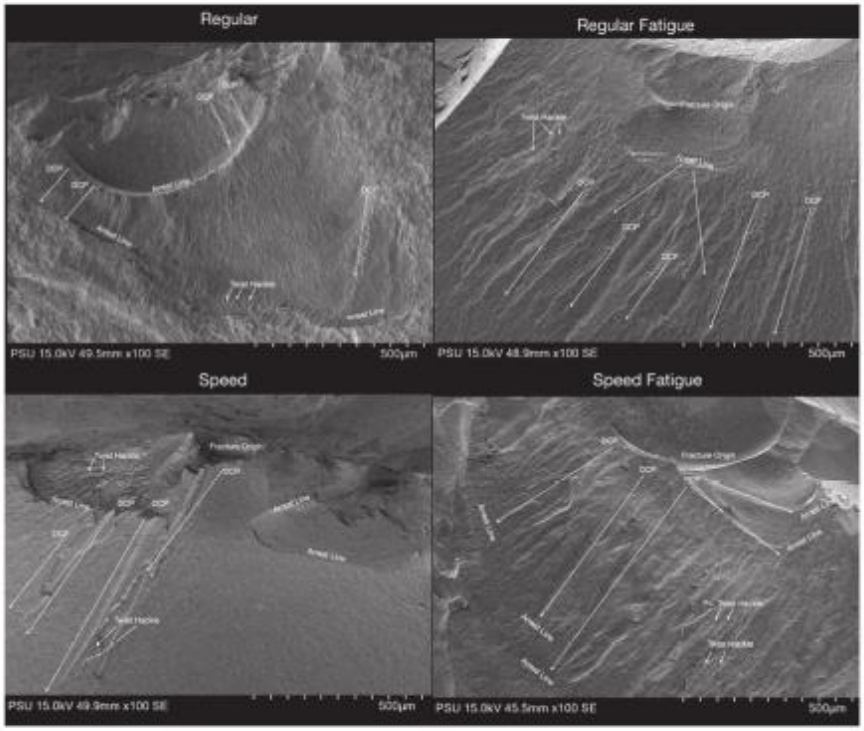

Figure 4. Surface structure in SEM of the zirconia crowns of various sintering groups.

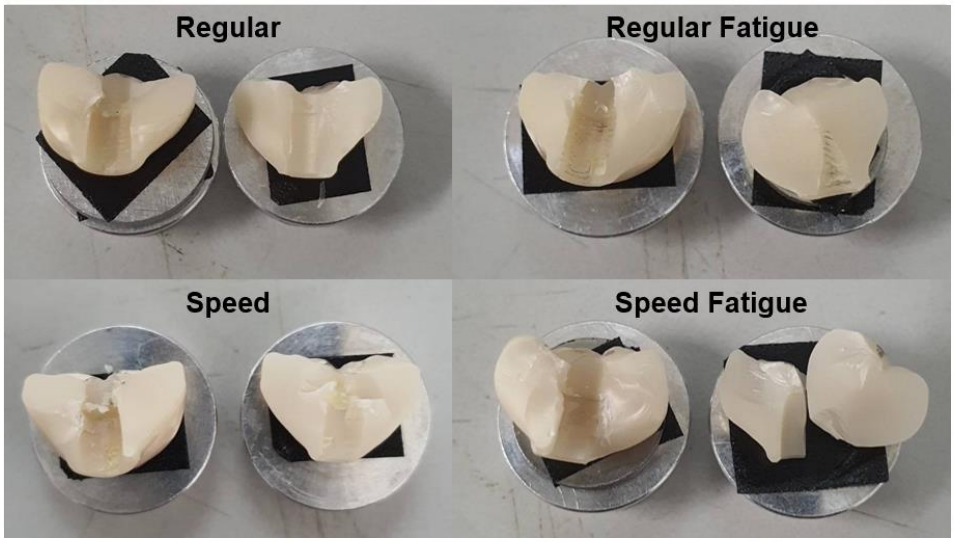

Figure 5. Fracture of the zirconia crowns of various sintering groups.

3.2. *Load to Failure*

The descriptive statistics of the fracture load are shown in Table 2. It shows that the speed group showed the maximum fracture load, followed by the regular, speed fatigue, and regular fatigue groups. The multiple comparisons show that the fracture load was a significant difference among the various groups; regular vs. regular fatigue, regular vs. speed, regular vs. speed fatigue, regular fatigue vs. speed, regular fatigue vs. speed fatigue, and speed vs. speed fatigue (Table 3).

Table 2. Descriptive statistics of fracture load.

Sintering	N	Mean (N)	Std. Deviation	Std. Error	Minimum	Maximum
Regular	15	3223.60	76.11	19.65	3146.00	3456.00
Regular fatigue	15	2143.06	135.93	35.09	1783.00	2324.00
Speed	15	3664.46	140.90	36.38	3276.00	3787.00
Speed fatigue	15	2450.06	128.26	33.12	2290.00	2790.00

Table 3. Multiple comparisons of the fracture load among the groups.

(I) Groups	(J) Groups	Mean Difference (I-J)	*p* Value
Regular	Regular fatigue	1080.53	<0.001 *
	Speed	−440.86	<0.001 *
	Speed fatigue	773.53	<0.001 *
Regular fatigue	Regular	−1080.53	<0.001 *
	Speed	−1521.40 *	<0.001 *
	Speed fatigue	−307.00	<0.001 *

Table 3. *Cont.*

(I) Groups	(J) Groups	Mean Difference (I-J)	*p* Value
Speed	Regular	440.86	<0.001 *
	Regular fatigue	1521.40	<0.001 *
	Speed fatigue	1214.40	<0.001 *
Speed fatigue	Regular	−773.53	<0.001 *
	Regular fatigue	307.00	<0.001 *
	Speed	−1214.40	<0.001 *

* The mean difference is significant at the 0.05 level.

Figure 6 shows the Weibull distribution for the zirconia crowns. The 63.3% value was also marked, for which the strength according to the analysis is σ_0 = 3244 MPa for regular, σ_0 = 2190 MPa for regular fatigue, σ_0 = 3765 MPa for speed, and σ_0 = 2457 MPa for speed fatigue (Table 4). It shows that only the regular group shows an R^2 of less than 65%. The sintering protocol with a larger Weibull module and durability increases the reliability.

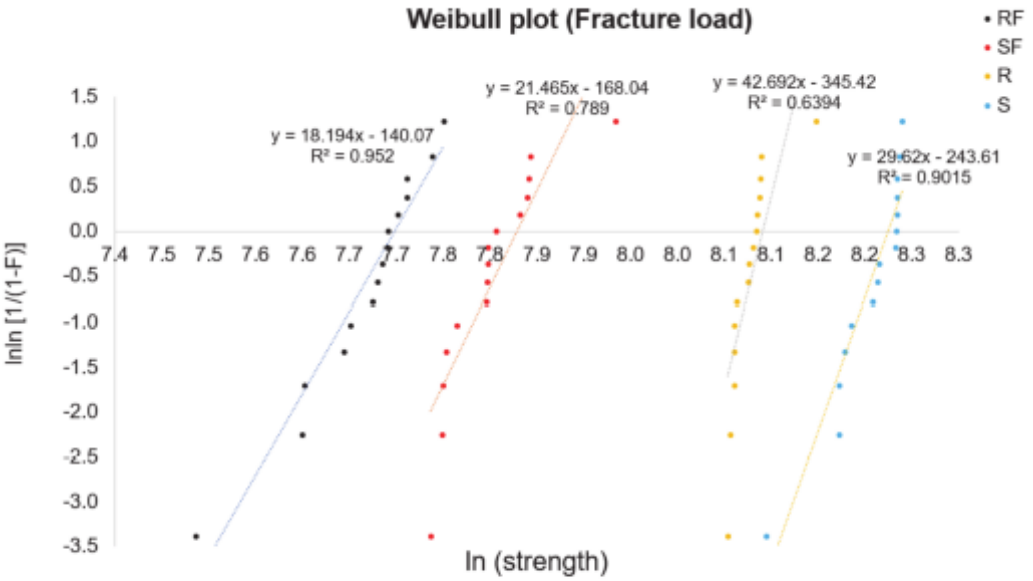

Figure 6. Weibull plot for the strength of the zirconia crowns of various sintering groups. R = regular, S = speed sintering, RF = regular fatigue, SF = speed fatigue.

Table 4. Results of Weibull distribution analysis.

Sintering Protocol	N	Characteristic Strength σ_o [MPa]	Coefficient of Determination R2	Weibull Modulus m
Regular (R)	15	3244	0.64	42.69
Regular fatigue (RF)	15	2190	0.95	18.19
Speed (S)	15	3765	0.90	29.62
Speed fatigue (SF)	15	2457	0.79	21.46

4. Discussion

The zirconia restorations are subjected to the occlusal loads but are below the flexural strength of zirconia in the mouth. However, the pre-existing surface flaws may lead to the propagation of those cracks and eventually cause the fracture of the zirconia restoration [15,33]. We analyzed the zirconia's survival using the Weibull approach. A low Weibull modulus shows more variability in the strength (σ_c), whereas a high Weibull modulus signifies higher reliability of zirconia material. Weibull's theory is based on the weakest link theory whereby failure is the result of the weakest line break in the chain [32].

The crack propagation mechanism in zirconia materials differs according to the type of material composition and sintering protocols. For the two material types, it was found that an intergranular fracture is seen in 3Y-TZP and a combination of intergranular and intragranular crack propagation is seen in 5Y-TZP [20]. When a micro-crack is created during the manufacturing process, the failure will start.

Our results were similar to the results obtained by some previous studies [34–36]. Mayinger et al. [34] studied the impact of high-speed sintering and artificial aging on the fracture load of three-unit zirconia fixed dental prostheses and found promising results for high-speed sintering, as it led to comparable fracture load and similar, or even superior, Weibull modulus results compared to the control group. The 4Y-TZP material presented fracture load results similar to the tried-and-tested 3Y-TZP. Artificial aging did not influence zirconia's resistance to fracture for either 3Y-TZP or 4Y-TZP. Similarly, Jerman et al. [36] investigated the influence of high-speed and conventional sintering on the flexural strength of three zirconia materials, initially and after artificial aging, and found that high-speed sintered HT+ showed higher initial flexural strength than the control group ($p < 0.001$). ZI ($p < 0.001$–0.004) and Zolid ($p < 0.001$–0.007) showed higher flexural strength after thermomechanical aging. In addition, the Weibull modulus of the three thermomechanically aged materials was negatively influenced by high-speed sintering; hence, they also mentioned that high-speed sintering is a valid alternative to conventional sintering protocols.

Arcila et al. [37] studied the microstructure of 3 yttria partially stabilized (3Y-PSZ) disc-shaped zirconia and compared the fracture resistance, hardness, and fatigue flexural strength of 3Y-TZP, 4Y-PSZ, and 5Y-PSZ. They demonstrated that all zirconia materials show similar compositions but vary the yttria content (5Y-PSZ > 4Y-PSZ > 3Y-TZP). They concluded that despite the microstructural differences, 3Y-TZP and 4Y-PSZ showed similar fatigue behavior whereas 5Y-PSZ had the least fatigue behavior.

Ordoñez Balladares et al. [38] compared the fracture strength of monolithic Zr dioxide after sintering in two different furnaces: CEREC SpeedFire (fast sintering) and InFire HTC Speed (slow sintering) produced from CAD/CAM. They found that the CEREC SpeedFire presented 1222.8 N fracture strength, whereas InFire HTC Speed showed 1068.5 N fracture strength, but no significant differences among the groups. The different furnaces did not affect the strengths of the zirconia and there is time saving when using rapid sintering. However, speed-sintered restorations may have limited reliability. According to a previous article, super-speed sintering is considered an option [28]; however, in the esthetic area where translucency is required, super-speed sintering might not suitable.

The thickness reduction of zirconia and fatigue affects the failure load of monolithic zirconia crowns. Prott et al. [39] mentioned that less thickness of the crown leads to less strength, even if the failure loads surpass the chewing forces. Fatigue significantly lessened the failure load of 0.5 mm 3Y-TZP crowns.

Regarding the limitations of this research, we should note the limited sample size. For the regular group, the R^2 might be higher if we cut out the samples or add more samples. This research is preliminary. Further research can be carried out to analyze of the chemical chain or strengthening phenomena in zirconia.

5. Conclusions

Surface flaws are the failure origin, and the crack lines ran from the occlusal to the bottom. Handling of the zirconia plays an important role in the longevity of the zirconia

restorations. It was found that the speed sintering group showed the maximum fracture load followed by the regular, speed fatigue, and regular fatigue groups. These values provide reference fracture load values for the implant crown and fixed partial denture and are used to assess the durability of zirconia in dentistry.

Author Contributions: Conceptualization, S.K.; methodology, S.K. and C.P.; software, S.K.; validation, S.K. and C.P.; formal analysis, S.K. and C.P.; investigation, S.K.; resources, S.K.; data curation, S.K.; writing—original draft preparation, S.K.; writing—review and editing, S.K. and C.P.; visualization, C.P.; supervision, C.P.; project administration, S.K.; funding acquisition, S.K. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

References

1. Mayinger, F.; Buser, R.; Laier, M.; Schönhoff, L.M.; Kelch, M.; Hampe, R.; Stawarczyk, B. Impact of the material and sintering protocol, layer thickness, and thermomechanical aging on the two-body wear and fracture load of 4y-tzp crowns. *Clin. Oral Investig.* **2022**, *26*, 6617–6628. [CrossRef] [PubMed]
2. Zhang, Y.; Lawn, B.R. Novel zirconia materials in dentistry. *J. Dent. Res.* **2018**, *97*, 140–147. [CrossRef] [PubMed]
3. Jung, S.; Moser, M.M.; Kleinheinz, J.; Happe, A. Biocompatibility of lithium disilicate and zirconium oxide ceramics with different surface topographies for dental implant abutments. *Int. J. Mol. Sci.* **2021**, *22*, 7700. [CrossRef]
4. Abhay, S.S.; Ganapathy, D.; Veeraiyan, D.N.; Ariga, P.; Heboyan, A.; Amornvit, P.; Rokaya, D.; Srimaneepong, V. Wear resistance, color stability and displacement resistance of milled peek crowns compared to zirconia crowns under stimulated chewing and high-performance aging. *Polymers* **2021**, *13*, 3761. [CrossRef] [PubMed]
5. Kongkiatkamon, S.; Booranasophone, K.; Tongtaksin, A.; Kiatthanakorn, V.; Rokaya, D. Comparison of fracture load of the four translucent zirconia crowns. *Molecules* **2021**, *26*, 5308. [CrossRef] [PubMed]
6. Heboyan, A.; Marya, A.; Syed, A.U.Y.; Khurshid, Z.; Zafar, M.S.; Rokaya, D.; Anastasyan, M. In vitro microscopic evaluation of metal-and zirconium- oxide-based crowns' marginal fit. *Pesqui. Bras. Odontopediatria Clín. Integr.* **2022**, *22*, e210144. [CrossRef]
7. Sailer, I.; Balmer, M.; Hüsler, J.; Hämmerle, C.H.F.; Känel, S.; Thoma, D.S. 10-year randomized trial (rct) of zirconia-ceramic and metal-ceramic fixed dental prostheses. *J. Dent.* **2018**, *76*, 32–39. [CrossRef]
8. Heboyan, A.; Manrikyan, M.; Zafar, M.S.; Rokaya, D.; Nushikyan, R.; Vardanyan, I.; Vardanyan, A.; Khurshid, Z. Bacteriological evaluation of gingival crevicular fluid in teeth restored using fixed dental prostheses: An in vivo study. *Int. J. Mol. Sci.* **2021**, *22*, 5463. [CrossRef]
9. Shin, H.; Ko, H.; Kim, M. Cytotoxicity and biocompatibility of zirconia (y-tzp) posts with various dental cements. *Restor. Dent. Endod.* **2016**, *41*, 167–175. [CrossRef]
10. Kongkiatkamon, S.; Rokaya, D. Full digital workflow in the esthetic dental restoration. *Case Rep. Dent.* **2022**, *2022*, 8836068. [CrossRef]
11. Humagain, M.; Rokaya, D. Integrating digital technologies in dentistry to enhance the clinical success. *Kathmandu. Univ. Med. J.* **2019**, *17*, 256–257.
12. Abduo, J.; Lyons, K. Rationale for the use of cad/cam technology in implant prosthodontics. *Int. J. Dent.* **2013**, *2013*, 768121. [CrossRef] [PubMed]
13. D'Souza, K.M.; Aras, M.A. Applications of cad/cam technology in dental implant planning and implant surgery. In *Advances in Dental Implantology Using Nanomaterials and Allied Technology Applications*; Chaughule, R.S., Dashaputra, R., Eds.; Springer International Publishing: Cham, Switzerland, 2021; pp. 247–286.
14. Rokaya, D.; Kongkiatkamon, S.; Heboyan, A.; Dam, V.V.; Amornvit, P.; Khurshid, Z.; Srimaneepong, V.; Zafar, M.S. 3d-printed biomaterials in biomedical application. In *Functional Biomaterials: Drug Delivery and Biomedical Applications*; Jana, S., Jana, S., Eds.; Springer: Singapore, 2022; pp. 319–339.
15. Elraggal, A.; Aboushelib, M.; Abdel Raheem, I.M.; Afifi, R.R. Effect of surface treatments on biaxial flexural strength, fatigue resistance, and fracture toughness of high versus low translucency zirconia. *BMC Oral Health* **2022**, *22*, 412. [CrossRef] [PubMed]
16. Zhang, F.; Inokoshi, M.; Batuk, M.; Hadermann, J.; Naert, I.; Van Meerbeek, B.; Vleugels, J. Strength, toughness and aging stability of highly-translucent y-tzp ceramics for dental restorations. *Dent. Mater.* **2016**, *32*, e327–e337. [CrossRef]

17. Baldissara, P.; Wandscher, V.F.; Marchionatti, A.M.E.; Parisi, C.; Monaco, C.; Ciocca, L. Translucency of ips e.Max and cubic zirconia monolithic crowns. *J. Prosthet. Dent.* **2018**, *120*, 269–275. [CrossRef]
18. Kwon, S.J.; Lawson, N.C.; McLaren, E.E.; Nejat, A.H.; Burgess, J.O. Comparison of the mechanical properties of translucent zirconia and lithium disilicate. *J. Prosthet. Dent.* **2018**, *120*, 132–137. [CrossRef]
19. Zhang, Y.; Lawn, B.R.; Rekow, E.D.; Thompson, V.P. Effect of sandblasting on the long-term performance of dental ceramics. *J. Biomed. Mater. Res. B Appl. Biomater.* **2004**, *71*, 381–386. [CrossRef]
20. Liu, C.; Eser, A.; Albrecht, T.; Stournari, V.; Felder, M.; Heintze, S.; Broeckmann, C. Strength characterization and lifetime prediction of dental ceramic materials. *Dent. Mater.* **2021**, *37*, 94–105. [CrossRef]
21. Kosmač, T.; Oblak, C.; Jevnikar, P.; Funduk, N.; Marion, L. The effect of surface grinding and sandblasting on flexural strength and reliability of y-tzp zirconia ceramic. *Dent. Mater.* **1999**, *15*, 426–433. [CrossRef]
22. Guazzato, M.; Albakry, M.; Quach, L.; Swain, M.V. Influence of surface and heat treatments on the flexural strength of a glass-infiltrated alumina/zirconia-reinforced dental ceramic. *Dent. Mater.* **2005**, *21*, 454–463. [CrossRef]
23. Reveron, H.; Fornabaio, M.; Palmero, P.; Fürderer, T.; Adolfsson, E.; Lughi, V.; Bonifacio, A.; Sergo, V.; Montanaro, L.; Chevalier, J. Towards long lasting zirconia-based composites for dental implants: Transformation induced plasticity and its consequence on ceramic reliability. *Acta Biomater.* **2017**, *48*, 423–432. [CrossRef]
24. Mao, L.; Kaizer, M.R.; Zhao, M.; Guo, B.; Song, Y.F.; Zhang, Y. Graded ultra-translucent zirconia (5y-psz) for strength and functionalities. *J. Dent. Res.* **2018**, *97*, 1222–1228. [CrossRef] [PubMed]
25. McLaren, E.A.; Maharishi, A.; White, S.N. Influence of yttria content and surface treatment on the strength of translucent zirconia materials. *J. Prosthet. Dent.* **2021**, *in press*. [CrossRef] [PubMed]
26. Nogueira, L.B.; Moura, C.D.; Francischone, C.E.; Valente, V.S.; Alencar, S.M.; Moura, W.L.; Soares Martins, G.A. Fracture strength of implant-supported ceramic crowns with customized zirconia abutments: Screw retained vs. Cement retained. *J. Prosthodont. Off. J. Am. Coll. Prosthodont.* **2016**, *25*, 49–53. [CrossRef] [PubMed]
27. Inokoshi, M.; Shimizu, H.; Nozaki, K.; Takagaki, T.; Yoshihara, K.; Nagaoka, N.; Zhang, F.; Vleugels, J.; Van Meerbeek, B.; Minakuchi, S. Crystallographic and morphological analysis of sandblasted highly translucent dental zirconia. *Dent. Mater.* **2018**, *34*, 508–518. [CrossRef] [PubMed]
28. Kongkiatkamon, S.; Peampring, C. Effect of speed sintering on low temperature degradation and biaxial flexural strength of 5y-tzp zirconia. *Molecules* **2022**, *27*, 5272. [CrossRef] [PubMed]
29. Pang, Z.; Chughtai, A.; Sailer, I.; Zhang, Y. A fractographic study of clinically retrieved zirconia-ceramic and metal-ceramic fixed dental prostheses. *Dent. Mater.* **2015**, *31*, 1198–1206. [CrossRef]
30. *ISO 6872:2015*; Dentistry–Ceramic Materials. International Standards Organization (ISO): Geneva, Switzerland, 2015; p. 28.
31. Weibull, W. A statistical distribution function of wideapplicability. *J. Appl. Mech.* **1951**, *103*, 293–297. [CrossRef]
32. Nemeth, N.N.; Gyekenyesi, J.P.; Jadaan, O.J. *Lifetime Reliability Prediction of Ceramic Structures under Transient Thermomechanical Loads*; NASA Center for Aerospace Information: Hanover, MD, USA, 2005; p. 212505.
33. Pereira, G.K.R.; Guilardi, L.F.; Dapieve, K.S.; Kleverlaan, C.J.; Rippe, M.P.; Valandro, L.F. Mechanical reliability, fatigue strength and survival analysis of new polycrystalline translucent zirconia ceramics for monolithic restorations. *J. Mech. Behav. Biomed. Mater.* **2018**, *85*, 57–65. [CrossRef]
34. Mayinger, F.; Pfefferle, R.; Reichert, A.; Stawarczyk, B. Impact of high-speed sintering of three-unit 3y-tzp and 4y-tzp fixed dental prostheses on fracture load with and without artificial aging. *Int. J. Prosthodont.* **2021**, *34*, 47–53. [CrossRef]
35. Wiedenmann, F.; Pfefferle, R.; Reichert, A.; Jerman, E.; Stawarczyk, B. Impact of high-speed sintering, layer thickness and artificial aging on the fracture load and two-body wear of zirconia crowns. *Dent. Mater.* **2020**, *36*, 846–853. [CrossRef]
36. Jerman, E.; Wiedenmann, F.; Eichberger, M.; Reichert, A.; Stawarczyk, B. Effect of high-speed sintering on the flexural strength of hydrothermal and thermo-mechanically aged zirconia materials. *Dent. Mater.* **2020**, *36*, 1144–1150. [CrossRef]
37. Arcila, L.V.C.; Ramos, N.C.; Campos, T.M.B.; Dapieve, K.S.; Valandro, L.F.; de Melo, R.M.; Bottino, M.A. Mechanical behavior and microstructural characterization of different zirconia polycrystals in different thicknesses. *J. Adv. Prosthodont.* **2021**, *13*, 385–395. [CrossRef]
38. Ordoñez Balladares, A.; Abad-Coronel, C.; Ramos, J.C.; Martín Biedma, B.J. Fracture resistance of sintered monolithic zirconia dioxide in different thermal units. *Materials* **2022**, *15*, 2478. [CrossRef]
39. Prott, L.S.; Spitznagel, F.A.; Bonfante, E.A.; Malassa, M.A.; Gierthmuehlen, P.C. Monolithic zirconia crowns: Effect of thickness reduction on fatigue behavior and failure load. *J. Adv. Prosthodont.* **2021**, *13*, 269–280. [CrossRef]

Paper 2

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Effect of Speed Sintering on Low Temperature Degradation and Biaxial Flexural Strength of 5Y-TZP Zirconia

Suchada Kongkiatkamon and Chaimongkon Peampring *

Department of Prosthetic Dentistry, Faculty of Dentistry, Prince of Songkla University, Hat Yai, Songkhla 90110, Thailand
* Correspondence: mongkon.5c@gmail.com

Abstract: Translucent zirconia is becoming the material of choice for the esthetic restorative material. We aimed to evaluate the surface structure, phase determination, translucency, and flexural strength of 5Y-TZP Zirconia (Katana STML Block and Disc) between the regular sintering and the speed sintering with and without low-temperature degradation (LTD). A total of 60 zirconia discs (30 per group; regular sintering and speed sintering) were used in this study. A CAM machine was used to mill cylinders out of the zirconia blanks and then cut into smaller discs. For the speed sintering, the zirconia blocks were milled into smaller discs. The zirconia discs were subjected to regular and speed sintering with and without LTD. Scanning electron microscopy was used to characterize the zirconia specimens and the zirconia grain size. Furthermore, the zirconia specimens were analyzed for elemental analysis using energy dispersive spectroscopy and phase identification using X-ray diffraction. The zirconia specimens were subjected to translucency measurements and biaxial flexural strength testing. The results of the zirconia specimens were compared among the groups. Statistical analysis was completed using SPSS version 20.0 to detect the statistically significant differences (p value = 0.05). A one-way ANOVA with multiple comparisons was performed using Scheffe analysis among the groups. The speed sintering presented smaller grain sizes. The zirconia specimens with and without LTD in regular and speed sintering presented a similar surface structure. Regular sintering showed more translucency compared to speed sintering. Multiple comparisons of the translucency parameter were a significant difference (p value < 0.05) between the various groups except for the comparison between speed sintering and speed sintering LTD. The regular sintering showed bigger gain sizes and slightly more translucency compared to speed sintering. The speed sintering showed higher biaxial flexural strengths compared to regular sintering. This shows that speed sintering can be considered a suitable method of sintering zirconia.

Keywords: dental ceramics; zirconia; Y-TZP; CAD/CAM; low temperature degradation; flexural strengths; grain size; translucency

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1. Introduction

Recently, there has been a substantial development in restorative biomaterials and technologies in dentistry [1–5]. The all-ceramic/zirconia restorations are esthetic restorations and are widely used restorative materials in restorative and prosthetic dentistry [6]. Zirconia is a polymorphic biomaterial having three crystallographic chemical structures: monoclinic, tetragonal, and cubic [7,8]. Yttria-partially stabilized zirconia (Y-PSZ), with a higher yttria content (4–6 mol% yttria) than 3Y-TZP, has been developed as a novel dental zirconia material [9].

The zirconia is present in the monoclinic form at room temperature and when heated; its phase transformation occurs to tetragonal and further heating leads to its cubic form [10–12], as shown in Figure 1. On cooling, cubic–tetragonal transformation takes place.

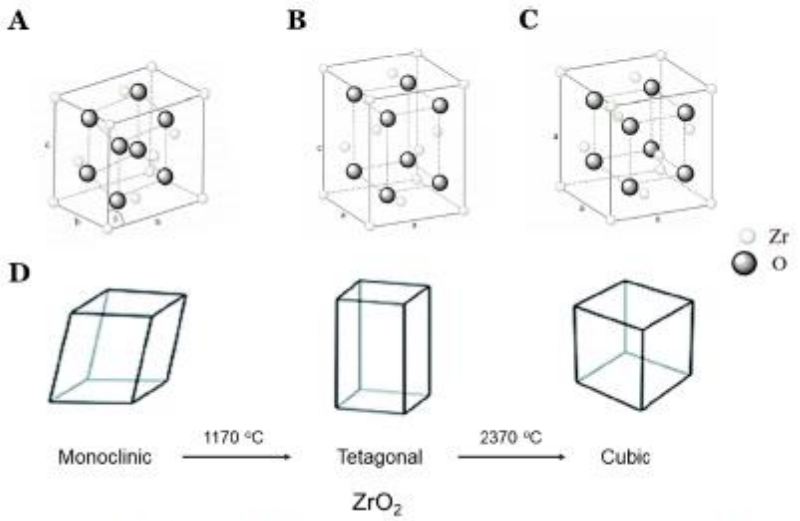

Figure 1. Different phases and phase transformation of zirconia. Monoclinic (**A**), tetragonal (**B**), and cubic structure (**C**). Zirconia can have 3 polymorphs depending on temperature: monoclinic at <1170 °C, tetragonal between 1170 °C and 2370 °C, and cubic at >2370 °C (**D**). Adapted with permission from Ref. [11]. 2019, MDPI, Basel, Switzerland.

Furthermore, zirconia (TZP, tetragonal *zirconia* polycrystal) can be of three types according to the yttria content [13,14]: 3Y-TZP, 4Y-TZP, and 5Y-TZP containing 3 mol%, 4 mol%, 5 mol% Y-TZP, respectively. The 3Y-TZP is strong and mainly tetragonal, 4Y-TZP is more translucent, and 5Y-TZP is the most translucent.

High-precision ceramic prostheses can be produced from computer-aided design and computer-aided manufacturing (CAD/CAM) technology with a more time-efficient and simplified process compared to traditional techniques [15,16]. In addition, milling also facilitates the fabrication of durable ceramic prostheses.

The aging or hydrothermal aging or low-temperature degradation (LTD) of zirconia results in the progressive transformation of the tetragonal to the monoclinic (t–m) phase in the presence of water/water vapor (at approx. 30 °C up to 400 °C). Yttria, a dopant used in zirconia to achieve the stability of the phase, is exhausted following LTD through a reaction in the presence of moisture resulting in the phase transformation [12,17,18].

There are some studies that compared the fast and slow sintering protocol of zirconia [18–22]. However, there is no study that studied speed sintering with LTD. Sintering times with aging can affect the structural, optical, and mechanical properties of zirconia [20]. Hence, the objective of this research is to compare the surface structure, phase determination, translucency, and biaxial flexural strength of zirconia blocks and discs (5Y-TZP) between the regular speed sintering and the speed sintering with and without LTD. The null hypothesis is that there is no difference in the surface structure, grain size, contrast ratio, translucency, and flexural strength in zirconia between regular sintering and the speed sintering with and without LTD.

2. Materials and Methods

Figure 2 shows the overview of this study. The study design was conducted by dividing into 2 sintering protocols: regular sintering (RS) and speed sintering (SS). A total sample size of 56 was calculated from the G*Power software version 3.1.9.7 [23] with the power of the study at 90%. Hence, 60 zirconia discs (30 per group) were selected for this study.

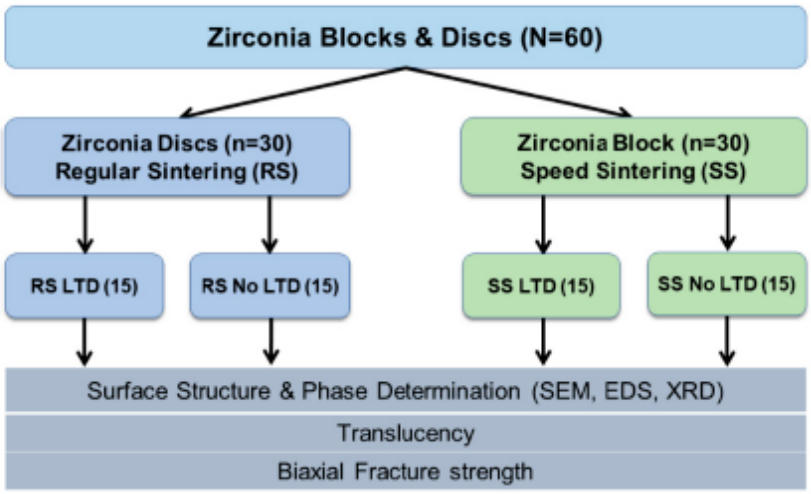

Figure 2. Overview method of the study. LTD = low temperature degradation.

2.1. Specimen Preparation

From the 3rd generation of zirconia (Katana STML Block) (5Y-TZP), specimens were prepared by the CAD/CAM system in both sintering protocols: speed sintering and regular sintering groups (Figures 2 and 3). Thirty zirconia blocks (Katana block 14ZL/STML A3) by the CAD/CAM system (inLAB20) were milled by a CAM machine (MCXL) to produce a disc shape.

For the regular sintering groups, big zirconia discs were milled into a cylinder shape and then cut into smaller discs using a CAM machine (IMES-ICORE Coritec 250i, Germany) (Figure 3A). For the speed sintering, the zirconia blocks (height 17.8 × 19.2 width × length 40 mm) were milled into smaller discs (Figure 3B).

The total thermal cycle, sintering time, and dwell temperature for the regular and speed sintering are shown in Figure 4. For the regular sintering group, the total thermal cycle, sintering time, and dwell temperature for the regular sintering were 6.8 h, 2 h, and 1550 °C, respectively, by using inFire HTC Furnace (Dentsply Sirona, Bensheim, Germany). For the speed sintering group, the total thermal cycle, sintering time, and dwell temperature were calculated based on each specimen, which was 30 min, 16 min, and 1560 °C by using SpeedFire (Dentsply Sirona, Bensheim, Germany).

Dimensions of each zirconia sample were measured with a digital caliper (Mitutoyo; Mitutoyo Co. Ltd., Kawasaki, Japan) before and after sintering. Before sintering, the average size of the zirconia samples was 18.39 mm in diameter and 1.23 mm in thickness. After sintering, the final dimensions for each test were 15 mm in diameter and 1 mm in thickness. The dimensions of the zirconia samples of both sintering protocols were similar.

Then, the specimens were cleaned ultrasonically (Model: 460/M, Elma Schmidbauer GmbH, Singen, Germany) in deionized water for 10 min then air dried individually for 20 s before testing.

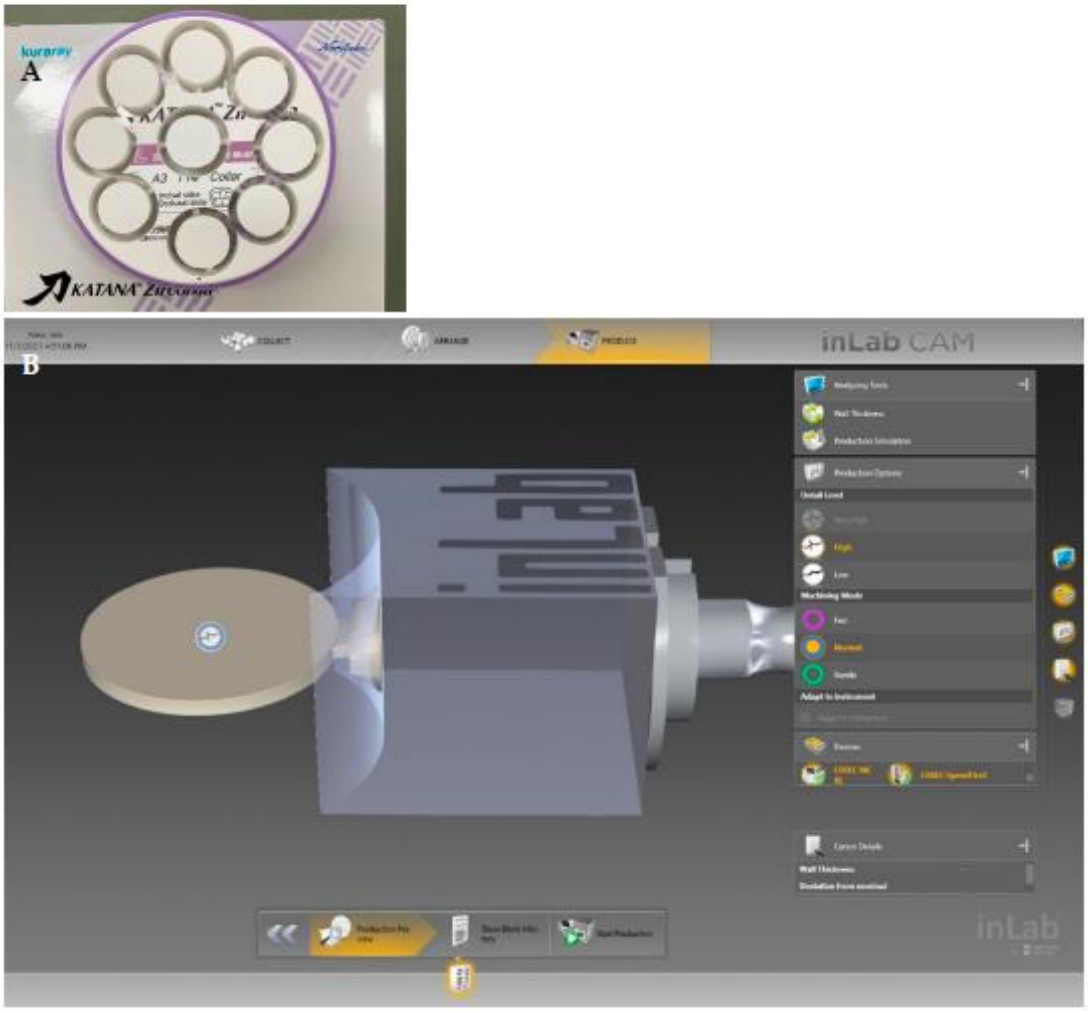

Figure 3. Preparation of the zirconia specimens by milling of big zirconia disc into a cylinder shape and then cutting into smaller discs for the regular sintering (**A**) and milling of the zirconia block into smaller discs for the speed sintering (**B**).

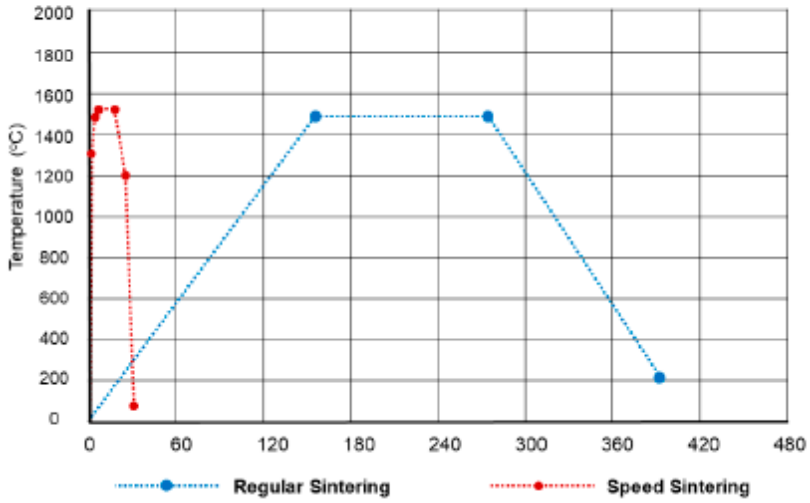

Figure 4. Total thermal cycle, sintering time, and dwell temperature for the regular and speed sintering.

2.2. *Specimen Preparation for Hydrothermal Aging*

Specimens for each group (15 for regular sintering and 15 discs for speed sintering) were subjected to LTD using an autoclave machine (TOMY ES 215/ES-315, Tomy Kogyo Co. Ltd., Nerima, Japan) at 122 °C under 2-bar pressure for 8 h and subjected to the phase transformation of zirconia at the surface. A total of 1 h of steam autoclave at 122 °C under 2-bar pressure has the same effect as 1 y in vivo [24]. Each specimen was subjected to sterilization using the autoclave. After the LTD, all specimens were dried in air for 24 h.

2.3. *Surface Characterization and Phase Structure*

2.3.1. Surface Structure, Grain Size, and Elemental Analysis

The zirconia specimens and the zirconia grain size were characterized using scanning electron microscopy (SEM) (FEI Quanta 400, FEI, Czech). At first, the zirconia specimens were coated with a gold–palladium alloy using sputter-coating and SEM images were obtained with an accelerating voltage of 20 kV with a 1000×, 5000×, 10,000×, 20,000×, and 30,000× magnification. Grain sizes were determined in each group from the SEM and the average grain size was calculated [20]. For measuring the grain size from the SEM, 3 specimens were randomly selected from each group. The five largest and five smallest grains in the center and off-center of each specimen were measured and the mean sizes were calculated and compared.

Furthermore, the zirconia specimens were analyzed for elemental analysis using energy dispersive spectroscopy (EDS) (OXFORD X-MaxN, FEI, Czech) to quantify material composition (zirconium, yttrium, aluminum, hafnium, and other oxides).

2.3.2. Phase Structure

The phase identification of the zirconia specimens was done using X-ray diffraction (XRD) (Philips X'Pert MPD, Philips, Eindhoven, The Netherlands) using Cu-K_α radiation from 0–90° (2θ). Scans were performed at 40 kV, 30 mA, step size of 0.05°/step, and a scan time of 1 s/step. The phase structures of zirconia were defined as a tetragonal unit cell with space group P42/nmc, a monoclinic unit cell with space group P21/c, and a cubic with space group Fm-3m (library data). Then, the phase fraction, the peak position, and composition were measured. The fractions volume of the monoclinic transformation was calculated from the peak intensities (Xm).

2.4. Translucency

The zirconia specimens were subjected to translucency measurements. A spectrophotometer (HunterLab, ColorQuest XE, Hunter Associates Laboratory Inc., Reston, Virginia, USA) with a calibration plate and port size 0.375" was used to record the CIELAB coordinates (L*, a*, and b*) of the zirconia discs. The translucency parameter (TP) was calculated from the difference in color between the same specimen against black and white backgrounds using the following formula (Equation (1)) [25]:

$$TP = [(L^*_B - L^*_W)^2 + (a^*_B - a^*_W)^2 + (b^*_B - b^*_W)^2]^{1/2} \quad (1)$$

where the B and W subscripts are the color coordinates over black and white backgrounds. A higher TP value indicates a higher translucency.

The contrast ratio (CR) was calculated from the spectral reflectance of light (Y) over a black (Y_B) and white (Y_W) background using the Equations (2) and (3) [25]:

$$Y = [(L^* + 16) / 116]^3 \times 100 \quad (2)$$

$$CR = Y_B / Y_W \quad (3)$$

where the Y = spectral reflectance of light of the specimen over a Y_B = black and Y_W = white background and CR = contrast ratio. The lower the CR, the more translucent. For a transparent material, CR is 0.0 whereas, for an opaque material, CR is 1.0.

2.5. Biaxial Flexural Strength

Zirconia discs (30 per group, Ø = 15 and t = 1 mm) were subjected to the flexural strength tests according to ISO 6872:2015 [26]. The treated surface of the specimens was faced down (tensile stress) on three balls (Ø = 4.6 mm) placed in a triangular position 10 mm apart from each other. A circular tungsten piston (Ø = 1.4 mm) of a universal testing machine (Lloyd Instruments, Model LRX-Plus, AMETEK Lloyd Instrument Ltd., Hampshire, UK) was used with an increasing load (1 mm/min) until the catastrophic failure occurred. Then, the flexural strength was calculated from Equation (4):

$$\sigma = [-0.2387P(X - Y)] / b^2 \quad (4)$$

where σ is the maximum tensile stress, P is the total load to fracture, and b is the thickness at fracture origin. X and Y are calculated from Equations (5) and (6):

$$X = (1 + \nu) \ln(r_2/r_3)^2 + \left[(1 - \nu)/2 (r_2/r_3)^2\right] \quad (5)$$

$$Y = (1 + \nu) [1 + \ln(r_1/r_3)^2 + (1 - \nu) (r_1/r_3)^2 \quad (6)$$

where ν is Poisson's ratio (ν = 0.30)[83], r_1 is the radius of the support circle (6.05 mm), r_2 is the radius of the loaded area (0.8 mm), and r_3 is the radius of the specimen (7.5 mm).

2.6. Statistical Analysis

The results of the zirconia specimens were compared among the groups. Descriptive statistics were calculated for each experiment. Statistical analysis was done using SPSS software version 20.0 (SPSS Inc., IBM Corporation, Chicago, IL, USA) to detect statistically significant differences (*p* value = 0.05). One-way ANOVA was used to compare the studied parameters among the groups. Multiple comparisons were done using Scheffe analysis.

3. Results

3.1. Surface Structure and Elemental Analysis

The SEM images of the zirconia specimens are shown in Figure 5 and the results of the grain size (μm) in each group are shown in Table 1. It showed that regular sintering showed a higher grain size compared to speed sintering. The low-temperature degradation

(LTD) zirconia specimens showed a similar structure to no LTD in both regular and speed sintering groups.

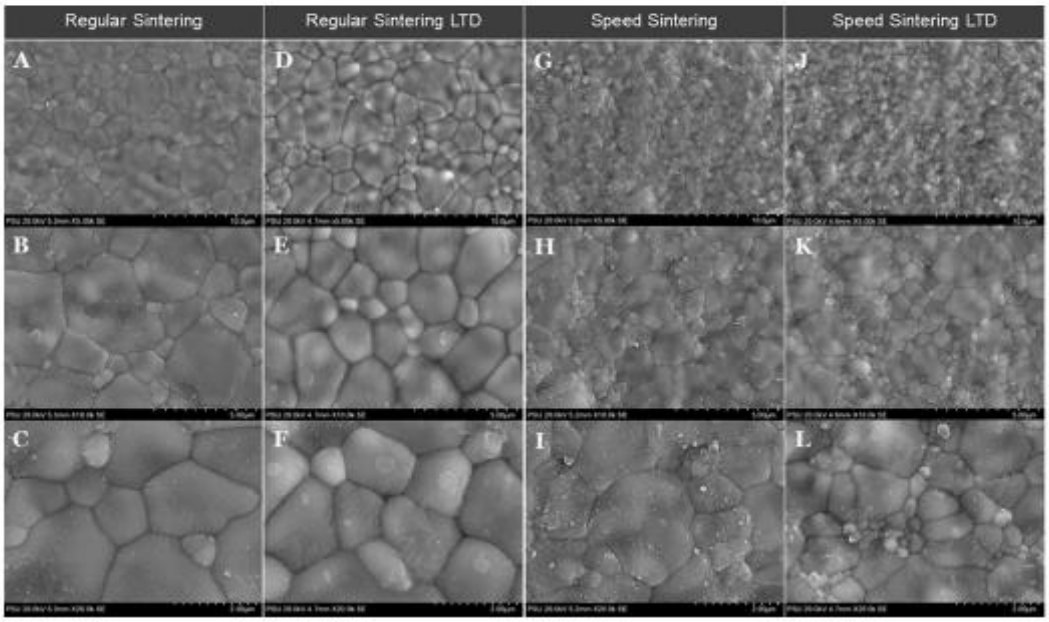

Figure 5. Scanning electron microscopy images of the zirconia specimens for the regular sintering and speed sintering with and without low-temperature degradation (LTD). Regular sintering (**A**–**F**) and speed sintering (**G**–**L**) from low magnification to high magnification.

Table 1. Results of the grain sizes of the zirconia specimens in various groups.

Groups	Mean (μm)	SD (μm)
Regular Sintering	3.206	1.257
Regular Sintering LTD	3.442	1.820
Speed Sintering	1.822	0.452
Speed Sintering LTD	1.689	1.053

SD = Standard deviation.

Table 2 shows the multiple comparisons of the grain size of the zirconia specimens among various groups among groups. The regular sintering showed a significantly bigger grain size (p value < 0.05) compared to the speed sintering LTD and regular sintering LTD compared to the speed sintering and speed sintering LTD. There was no difference in the flexural strength between the regular sintering vs regular sintering LTD (p value = 0.975) and speed sintering vs speed sintering LTD (p value = 0.995).

Table 2. Multiple comparisons of the grain size of the zirconia specimens among various groups.

(I) Groups	(J) Groups	Mean Difference (I − J)	*p* Value
Regular Sintering	Regular Sintering LTD	−0.236	0.975
	Speed Sintering	1.383	0.075
	Speed Sintering LTD	1.517	0.042 *
Regular Sintering LTD	Regular Sintering	0.236	0.975
	Speed Sintering	1.6192	0.026 *
	Speed Sintering LTD	1.7532	0.014 *
Speed Sintering	Regular Sintering	−1.383	0.075
	Regular Sintering LTD	−1.6192	0.026 *
	Speed Sintering LTD	0.134	0.995
Speed Sintering LTD	Regular Sintering	−1.517	0.042 *
	Regular Sintering LTD	−1.753	0.014 *
	Speed Sintering	−0.134	0.995

* The mean difference is significant at the 0.05 level. Multiple comparisons were done using Scheffe analysis.

The EDS elemental analysis of the zirconia specimens is shown in Figure 6 and Table 3. Figure 6 shows the EDS spectra and Table 3 shows the elemental analysis of the zirconia specimens in atomic %. The speed sintering presented slightly higher Zr. LTD showed slightly lower C and O with higher Y, Zr, and Hf.

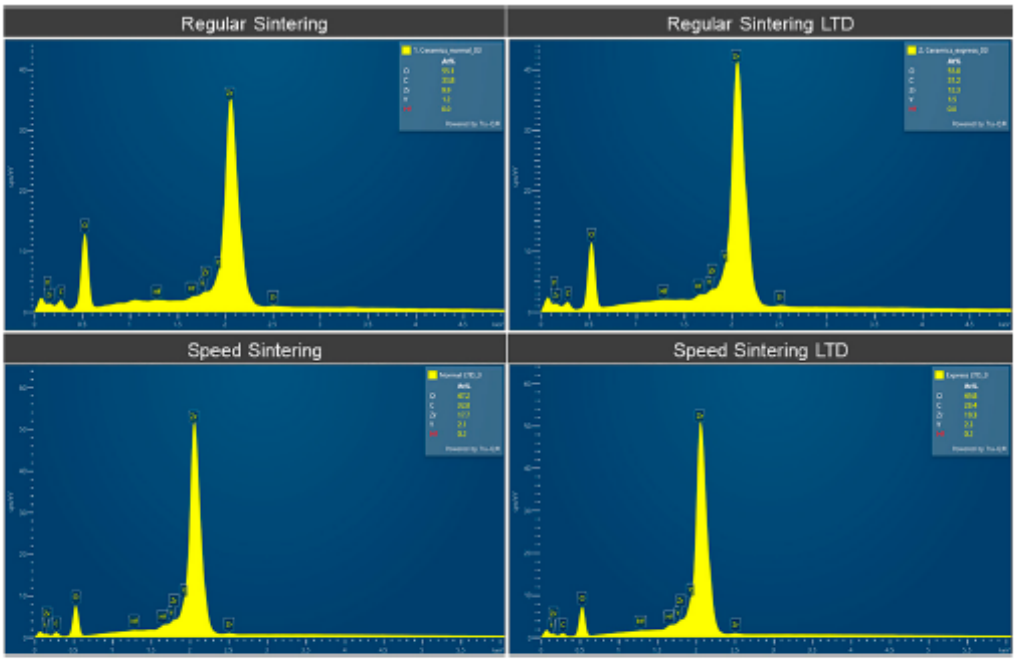

Figure 6. EDS Spectra of the zirconia specimens for the regular speed sintering and speed sintering with and without low-temperature degradation (LTD).

Table 3. Results of the EDS elemental analysis of the zirconia specimens for the regular speed sintering and speed sintering with and without low-temperature degradation (LTD).

Zirconia Specimens	C (Mean ± SD) (Atomic %)	O (Mean ± SD) (Atomic %)	Y (Mean ± SD) (Atomic %)	Zr (Mean ± SD) (Atomic %)	Hf (Mean ± SD) (Atomic %)
Regular Sintering	35.073 ± 1.355	53.773 ± 1.505	1.220 ± 0.051	9.833 ± 0.238	0.016 ± 0.004
Regular Sintering LTD	32.973 ± 1.682	47.436 ± 0.947	2.076 ± 0.084	17. 376 ± 0.6930	0.1433 ± 0.024
Speed Sintering	34.260 ± 2.692	53.123 ± 1.831	1.346 ± 0.097	11.163 ± 0.906	0.013 ± 0.004
Speed Sintering LTD	28.190 ± 0.190	50.093 ± 0.339	2.316 ± 0.02	19.236 ± 0.182	0.166 ± 0.012

SD = Standard deviation.

3.2. *Phase Structure*

The XRD analysis (Figure 7) of the zirconia specimens shows that the zirconium specimens showed that the peak positions for the spectra correspond to the tetragonal phase for zirconium yttrium oxides (ZrO_2).

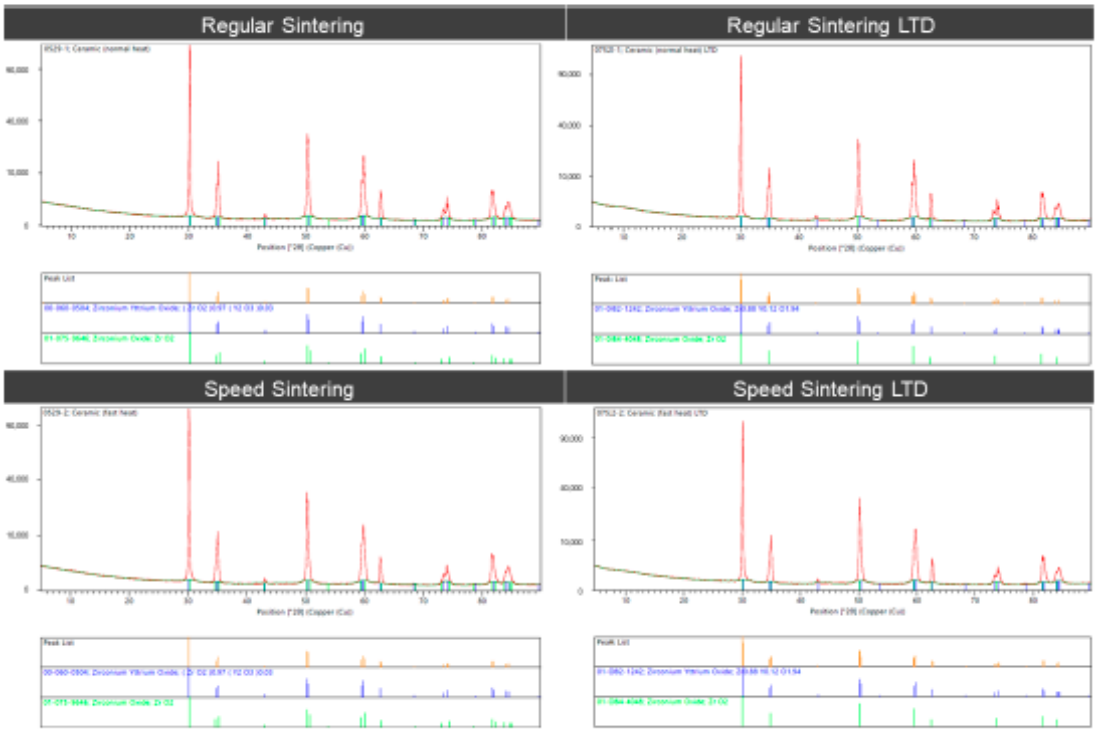

Figure 7. X-ray diffraction (XRD) analysis of the zirconia specimens for the regular sintering and speed sintering with and without low-temperature degradation (LTD).

3.3. *Translucency*

The results of the transparency are shown in Table 4. Regular sintering showed more translucency compared to speed sintering. Multiple comparisons of the contrast ratio between the groups show that there was a significant difference (p value < 0.05) between the various groups. However, the translucency parameter presented a significant difference

(p value < 0.05) between the various groups except for between speed sintering vs speed sintering LTD (p value = 0.931).

Table 4. Results of the contrast ratios transparency, and biaxial flexural strength of the zirconia specimens.

Zirconia Specimens	Contrast Ratio (Mean ± SD)	Translucency (Mean ± SD)	Flexural Strength (MPa) (Mean ± SD)
Regular Sintering	0.787 ± 0.034	10.581 ± 0.798	466.41 ± 22.898
Regular Sintering LTD	0.734 ± 0.028	12.443 ± 1.173	471.85 ± 20.789
Speed Sintering	0.833 ± 0.021	9.052 ± 0.618	500.5 ± 23.432
Speed Sintering LTD	0.824 ± 0.252	9.263 ± 0.775	513.63 ± 19.909

SD = Standard deviation.

3.4. Biaxial Flexural Strength

The results of the flexural strength (MPa) are shown in Table 4. It showed that speed sintering and speed sintering LTD showed higher biaxial flexural strengths.

Table 5 shows the multiple comparisons of the flexural strength (MPa) among the groups. The regular sintering showed significantly lower flexural strength (p value ≤ 0.01) compared to the speed sintering and speed sintering LTD. Similarly, the regular sintering LTD showed a significantly lower flexural strength (p value < 0.005) compared to the speed sintering and speed sintering LTD. However, there was no difference in the flexural strength between the speed sintering and speed sintering LTD (p value = 0.444).

Table 5. Multiple comparisons of the biaxial flexural strength of the zirconia specimens among various groups.

(I) Groups	(J) Groups	Mean Difference (I − J)	p Value
Regular Sintering	Regular Sintering LTD	−5.436	0.926
	Speed Sintering	−34.097 *	0.001 *
	Speed Sintering LTD	−47.219 *	<0.0001 *
Regular Sintering LTD	Regular Sintering	5.436	0.926
	Speed Sintering	−28.661 *	0.008 *
	Speed Sintering LTD	−41.783 *	<0.0001 *
Speed Sintering	Regular Sintering	34.097 *	0.001 *
	Regular Sintering LTD	28.661 *	0.008 *
	Speed Sintering LTD	−13.122	0.444
Speed Sintering LTD	Regular Sintering	47.219 *	<0.0001 *
	Regular Sintering LTD	41.783 *	<0.0001 *
	Speed Sintering	13.122	0.444

* The mean difference is significant at the 0.05 level. Multiple comparisons were done using Scheffe analysis.

4. Discussion

Zirconia has good wear resistance and good color stability [27]. There have been developed various sintering methods to enhance the properties and esthetics of zirconia. As there was no study that studied the speed sintering with aging or LTD. Sintering times with LTD aging can affect the structural, optical, and mechanical properties of zirconia [20]. Hence, it was important to study the surface structure, phase determination, translucency, and flexural strength of zirconia between regular sintering and speed sintering. So, this research was done to compare the surface structure, phase determination, translucency,

and biaxial flexural strength of zirconia blocks and discs (5Y-TZP) between the regular speed sintering and the speed sintering with and without LTD. In this study, we rejected the null hypothesis as there was a difference in the surface structure, grain size, contrast ratio, and biaxial flexural strength in zirconia between the regular sintering and the speed sintering with and without LTD. In this study, the regular sintering with LTD showed the most translucency. From XRD, there was no difference in all groups. All the peaks were the same; however, the translucencies are different, which could be due to the difference in the grain size. A possible scenario for this change might be due to material structural changes due to the degradation of the metal salts within the coloring liquid [28]. In addition, in this study, for speed sintering, we can notice a smaller grain size. The smaller grain of the monoclinic lattice may cause less light transmission. Liu et al. [9] also found similar results that the conventionally-sintered Y-PSZ had a larger average grain size and a smaller fraction of fine grains than those of the speed-sintered specimens.

Furthermore, Kilinc et al. [20] studied the various sintering methods and aging on the grain size, flexural strength, and translucency of the zirconia and they found that the sintering method and aging significantly influence the translucency. The flexural strength and grain sizes were influenced by aging only ($p < 0.001$). Aging times increased the grain size but prolonged sintering with 120 min of aging negatively influenced the translucency of zirconia. The increase in grain size compared to our research might be due to the shorter aging time (60 min and 120 min), but in our research, the samples were subjected to 122 °C under 2-bar pressure for 8 h. Hence, prolonged aging reduces the grain size. Similarly, Alamledin et al. [29], mentioned that accelerated aging had an effect on the translucency of both fully-stabilized cubic and partially-stabilized tetragonal zirconia, causing an increase in the translucency of zirconia.

In this study, the size of the zirconium samples before sintering was 1.226 times bigger than the size of the zirconium samples after sintering. This bigger size before sintering is to compensate for the shrinkage following the sintering (enlargement factor of 1.226). In addition, the dimensions of the zirconia samples of both sintering protocols were similar. Ahmed et al. [30] also mentioned that there is no difference in the linear and volumetric dimensional changes between standard and fast sintering protocols.

Liu et al. [22] studied the optical properties of two generations of rapid sintered translucent zirconia (3Y-TZP and 5Y-TZP) and they found that rapid sintering resulted in reduced lightness but did not affect the surface roughness. They concluded that rapid sintering is a practical method of reducing the production time of zirconia restorations.

In our study, the flexural strength was affected by the sintering method. These results could be due to the smaller grain size of zirconia from speed sintering. These results were similar to Ersoy et al. [21], who found that both a high and low sintering temperature combination increases the zirconia's flexural strength, but there were no visible differences in the grain size in the zirconia specimens. Stawarczyk et al. [31] mentioned that the grain size of zirconia increases with the sintering temperatures and the sintering temperature significantly affects the flexural strength.

When heating or low-temperature degradation (LTD), the monoclinic phase will transform into a tetragonal and cubic phase according to the temperature (Figure 1), and when cooling, it will transform back to monoclinic, which is not strong. Obtaining stable sintered zirconia ceramic products is difficult because of the large volume change accompanying the transition from tetragonal to monoclinic (approximately 5%). Hence, yttria is added to stabilize in the tetragonal phase [32,33]. Yttria-doping reduced the grain growth, stabilized the tetragonal phase, and significantly improved the thermal stability of the particles. In addition, stabilization of the cubic polymorph of zirconia over a wider range of temperatures is accomplished by the substitution of some of the Zr^{4+} ions (ionic radius of 0.82 Å, too small for the ideal lattice of fluorite characteristics for the cubic zirconia) in the crystal lattice with slightly larger ions, e.g., those of Y^{3+} (ionic radius of 0.96 Å). The resulting doped zirconia materials are stabilized zirconia (PSZ) [32].

Arcila et al. [34] characterized the microstructure of three types of zirconia; 3Y-TZP (yttria-tetragonal *zirconia* polycrystal), 4Y-PSZ, and 5Y-PSZ (yttria-partially stabilized zirconia) and compared their hardness, fracture resistance, and fatigue and flexural strength. Three zirconia used were 3Y-TZP (Vita YZ HT), 4Y-PSZ (Vita YZ ST), and 5Y-PSZ (Vita YZ XT). The 4Y-PSZ and 5Y-PSZ specimens presented some surface defects under the SEM, whereas the 3Y-TZP demonstrated a greater grain consistency on the surface. They concluded that despite the structural differences, 4Y-PSZ and 3Y-TZP had similar fatigue. The 5Y-PSZ had the least mechanical strengths.

Finally, in this study, only one brand of zirconia was used, and different manufacturers may have presented different grain sizes for sintering zirconia. Future studies can be done to study more the grain size and its chemistry.

5. Conclusions

This research showed that there was a difference in surface structure, translucency, and biaxial flexural strengths. The regular sintering showed a bigger grain size and slightly more translucency compared to speed sintering. The speed sintering showed higher biaxial flexural strengths compared to regular sintering. This shows that speed sintering can be considered a suitable method of sintering zirconia. Hence, when biaxial flexural strength is required, speed sintering can be considered; however, when better translucency is required, regular sintering is recommended.

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References

1. Kontonasaki, E.; Rigos, A.E.; Ilia, C.; Istantsos, T. Monolithic Zirconia: An Update to Current Knowledge. Optical Properties, Wear, and Clinical Performance. *Dent. J.* **2019**, *7*, 90. [CrossRef]
2. Kontonasaki, E.; Giasimakopoulos, P.; Rigos, A.E. Strength and aging resistance of monolithic zirconia: An update to current knowledge. *Jpn. Dent. Sci. Rev.* **2020**, *56*, 1–23. [CrossRef]
3. Humagain, M.; Rokaya, D. Integrating Digital Technologies in Dentistry to Enhance the Clinical Success. *Kathmandu Univ. Med. J. (KUMJ)* **2019**, *17*, 256–257.
4. Amornvit, P.; Rokaya, D.; Peampring, C.; Sanohkan, S. Confocal 3D Optical Intraoral Scanners and Comparison of Image Capturing Accuracy. *Comput. Mater. Contin.* **2021**, *66*, 303–314. [CrossRef]
5. Rokaya, D.; Kongkiatkamon, S.; Heboyan, A.; Dam, V.V.; Amornvit, P.; Khurshid, Z.; Srimaneepong, V.; Zafar, M.S. 3D-Printed Biomaterials in Biomedical Application. In *Functional Biomaterials: Drug Delivery and Biomedical Applications*; Jana, S., Ed.; Springer: Singapore, 2022; pp. 319–339. [CrossRef]
6. Kongkiatkamon, S.; Booranasophone, K.; Tongtaksin, A.; Kiatthanakorn, V.; Rokaya, D. Comparison of Fracture Load of the Four Translucent Zirconia Crowns. *Molecules* **2021**, *26*, 5308. [CrossRef] [PubMed]
7. Saridag, S.; Tak, O.; Alniacik, G. Basic properties and types of zirconia: An overview. *World J. Stomatol.* **2013**, *2*, 40–47. [CrossRef]

8. Bocanegra-Bernal, M.H.; de la Torre, S.D. Phase transitions in zirconium dioxide and related materials for high performance engineering ceramics. *J. Mater. Sci.* **2002**, *37*, 4947–4971. [CrossRef]
9. Liu, H.; Inokoshi, M.; Nozaki, K.; Shimizubata, M.; Nakai, H.; Cho Too, T.D.; Minakuchi, S. Influence of high-speed sintering protocols on translucency, mechanical properties, microstructure, crystallography, and low-temperature degradation of highly translucent zirconia. *Dent. Mater.* **2022**, *38*, 451–468. [CrossRef]
10. Piconi, C.; Maccauro, G. Zirconia as a ceramic biomaterial. *Biomaterials* **1999**, *20*, 1–25. [CrossRef]
11. Sorrentino, R.; Navarra, C.O.; Di Lenarda, R.; Breschi, L.; Zarone, F.; Cadenaro, M.; Spagnuolo, G. Effects of Finish Line Design and Fatigue Cyclic Loading on Phase Transformation of Zirconia Dental Ceramics: A Qualitative Micro-Raman Spectroscopic Analysis. *Materials* **2019**, *12*, 863. [CrossRef]
12. Rekow, E.D.; Silva, N.R.F.A.; Coelho, P.G.; Zhang, Y.; Guess, P.; Thompson, V.P. Performance of Dental Ceramics: Challenges for Improvements. *J. Dent. Res.* **2011**, *90*, 937–952. [CrossRef] [PubMed]
13. Zhang, Y. Making yttria-stabilized tetragonal zirconia translucent. *Dent. Mater.* **2014**, *30*, 1195–1203. [CrossRef] [PubMed]
14. Abdulmajeed, A.; Sulaiman, T.; Abdulmajeed, A.; Bencharit, S.; Närhi, T. Fracture Load of Different Zirconia Types: A Mastication Simulation Study. *J. Prosthodont. Off. J. Am. Coll. Prosthodont.* **2020**, *29*, 787–791. [CrossRef]
15. Padrós, R.; Giner, L.; Herrero-Climent, M.; Falcao-Costa, C.; Ríos-Santos, J.V.; Gil, F.J. Influence of the CAD-CAM Systems on the Marginal Accuracy and Mechanical Properties of Dental Restorations. *Int. J. Environ. Res. Public Health* **2020**, *17*, 4276. [CrossRef] [PubMed]
16. Abduo, J.; Lyons, K. Rationale for the Use of CAD/CAM Technology in Implant Prosthodontics. *Int. J. Dent.* **2013**, *2013*, 768121. [CrossRef]
17. Chevalier, J.; Cales, B.; Drouin, J.M. Low-Temperature Aging of Y-TZP Ceramics. *J. Am. Ceram. Soc.* **1999**, *82*, 2150–2154. [CrossRef]
18. Amat, N.F.; Muchtar, A.; Amril, M.S.; Ghazali, M.J.; Yahaya, N. Effect of sintering temperature on the aging resistance and mechanical properties of monolithic zirconia. *J. Mater. Res. Technol.* **2019**, *8*, 1092–1101. [CrossRef]
19. Ordoñez Balladares, A.; Abad-Coronel, C.; Ramos, J.C.; Martín Biedma, B.J. Fracture Resistance of Sintered Monolithic Zirconia Dioxide in Different Thermal Units. *Materials* **2022**, *15*, 2478. [CrossRef]
20. Kilinc, H.; Sanal, F.A. Effect of sintering and aging processes on the mechanical and optical properties of translucent zirconia. *J. Prosthet. Dent.* **2021**, *126*, 129.e1–129.e7. [CrossRef]
21. Ersoy, N.M.; Aydoğdu, H.M.; Değirmenci, B.; Çökük, N.; Sevimay, M. The effects of sintering temperature and duration on the flexural strength and grain size of zirconia. *Acta Biomater. Odontol. Scand.* **2015**, *1*, 43–50. [CrossRef]
22. Liu, Y.-C.; Lin, T.-H.; Lin, Y.-Y.; Hu, S.-W.; Liu, J.-F.; Yang, C.-C.; Yan, M. Optical properties evaluation of rapid sintered translucent zirconia with two dental colorimeters. *J. Dent. Sci.* **2022**, *17*, 155–161. [CrossRef]
23. Faul, F.; Erdfelder, E.; Lang, A.G.; Buchner, A. G*Power 3: A flexible statistical power analysis program for the social, behavioral, and biomedical sciences. *Behav. Res. Methods* **2007**, *39*, 175–191. [CrossRef] [PubMed]
24. Kim, J.W.; Covel, N.S.; Guess, P.C.; Rekow, E.D.; Zhang, Y. Concerns of hydrothermal degradation in CAD/CAM zirconia. *J. Dent. Res.* **2010**, *89*, 91–95. [CrossRef] [PubMed]
25. Della Bona, A.; Nogueira, A.D.; Pecho, O.E. Optical properties of CAD-CAM ceramic systems. *J. Dent.* **2014**, *42*, 1202–1209. [CrossRef] [PubMed]
26. *ISO 6872*; 2015 Dentistry—Ceramic Materials. International Standards Organization (ISO): Geneva, Switzerland, 2015; p. 28.
27. Abhay, S.S.; Ganapathy, D.; Veeraiyan, D.N.; Ariga, P.; Heboyan, A.; Amornvit, P.; Rokaya, D.; Srimaneepong, V. Wear Resistance, Color Stability and Displacement Resistance of Milled PEEK Crowns Compared to Zirconia Crowns under Stimulated Chewing and High-Performance Aging. *Polymers* **2021**, *13*, 3761. [CrossRef]
28. Sulaiman, T.A.; Abdulmajeed, A.A.; Shahramian, K.; Lassila, L. Effect of different treatments on the flexural strength of fully versus partially stabilized monolithic zirconia. *J. Prosthet. Dent.* **2017**, *118*, 216–220. [CrossRef]
29. Alamledin, A.M.; Shokry, T.E.; El Kouedi, A.Y. Effect of Accelerated Aging on the Translucency of Monolithic Zirconia at Minimal Thickness. *Al-Azhar J. Dent. Sci.* **2020**, *23*, 7–14.
30. Ahmed, W.M.; Troczynski, T.; Stojkova, B.J.; McCullagh, A.P.; Wyatt, C.C.; Carvalho, R.M. Dimensional Changes of Yttria-stabilized Zirconia under Different Preparation Designs and Sintering Protocols. *J. Prosthodont.* **2020**, *29*, 699–706. [CrossRef]
31. Stawarczyk, B.; Ozcan, M.; Hallmann, L.; Ender, A.; Mehl, A.; Hämmerlet, C.H. The effect of zirconia sintering temperature on flexural strength, grain size, and contrast ratio. *Clin. Oral Investig.* **2013**, *17*, 269–274. [CrossRef]
32. Yanagida, H.; Koumoto, K.; Miyayama, M. *The Chemistry of Ceramics*; John Wiley & Sons: Oxford, UK, 1996.
33. Leib, E.W.; Vainio, U.; Pasquarelli, R.M.; Kus, J.; Czaschke, C.; Walter, N.; Janssen, R.; Müller, M.; Schreyer, A.; Weller, H.; et al. Synthesis and thermal stability of zirconia and yttria-stabilized zirconia microspheres. *J. Colloid Interface Sci.* **2015**, *448*, 582–592. [CrossRef]
34. Arcila, L.V.C.; Ramos, N.C.; Campos, T.M.B.; Dapieve, K.S.; Valandro, L.F.; de Melo, R.M.; Bottino, M.A. Mechanical behavior and microstructural characterization of different zirconia polycrystals in different thicknesses. *J. Adv. Prosthodont.* **2021**, *13*, 385–395. [CrossRef] [PubMed]

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Name Mrs. Suchada Kongkiatkamon

Student ID 6310830008

Educational Attainment

Degree	Name of Institution	Year of Graduation
DDS	Srinakharinwirot University, Thailand	2006
MS	University of Illinois at Chicago, United States	2012

Scholarship Awards during Enrolment

.....

.....

Work – Position and Address

Prosthodontist, Bangkok Hospital, Bangkok, Thailand

.....

List of Publication and Proceeding

1. Kongkiatkamon S, Peampring C. Comparison of Regular and Speed Sintering on Low-Temperature Degradation and Fatigue Resistance of Translucent Zirconia Crowns for Implants: An In Vitro Study. *Journal of Functional Biomaterials* 2022; 13: 281. doi.org/10.3390/jfb13040281
2. Kongkiatkamon S, Booranasophone K, Tongtaksin A, Kiatthanakorn V, Rokaya D. Comparison of Fracture Load of the Four Translucent Zirconia Crowns. *Molecules* 2021;26: 5308. doi.org/10.3390/molecules26175308
3. Kongkiatkamon S, Peampring C. Effect of Speed Sintering on Low Temperature Degradation and Biaxial Flexural Strength of 5Y-TZP Zirconia. *Molecules* 2022;27: 5272. doi.org/10.3390/molecules27165272
4. Kongkiatkamon S, Wongkornchaowalit N, Kiatthanakorn V, Tonphu S, Kunanusont C. Quantitative Fit Test of a 3D Printed Frame Fitted Over a Surgical Mask: An Alternative Option to N95 Respirator. *International Journal of Dentistry* 2022;2022: 1270106. doi: 10.1155/2022/1270106