Factors affecting fracture of zirconia dental crowns

Laboratory studies on retrievals and crown-shaped specimens

Anneli Skjold

Thesis for the degree of Philosophiae Doctor (PhD)
University of Bergen, Norway
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Scientific environment

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The main supervisor for the project was Professor Marit Øilo. Co-supervisors were Professor Nils Roar Gjerdet, late Associate Professor Harald Gjengedal and Associate Professor Christian Schriwer.

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Abstract

Objective

The use of zirconia material in dental use has increased significantly over the last 20 years. Laboratory tests that examine material strength and toughness indicate that the available zirconia materials have sufficient mechanical properties to tolerate clinical use. However, clinical studies show that zirconia-based crowns fail due to mechanical failure such as fracture and chipping. There is little information available on the underlaying cause and possible factors affecting a crown fracture.

Aim

The main aim of this thesis was to examine fracture details of zirconia-based crowns fractured during clinical use and to identify factors that could affect fracture of zirconia dental crowns.

Material and methods

A convenience sample of clinically fractured zirconia-based crowns submitted by general dentists were analyzed by fractographic method. Two laboratory studies assessed how different factors such as crown material thickness, preparation type, production method, material composition and aging procedures affected load at fracture for zirconia crowns.

Conclusions

Fractographic analysis showed that most fractured crowns had fracture origin at the crown margin and thus indicating an area of increased load during clinical use. Zirconia material thickness and material composition had an effect on load at fracture. However, aging procedures simulating short-term clinical use did not affect the load at fracture values, regardless of material composition.

List of Publications

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List of abbreviations

ZrO_2 – zirconium dioxide (zirconia)

Y_2O_3 – yttrium oxide (yttria)

3Y-TZP - 3mol% yttria stabilized tetragonal zirconia polycrystal

PSZ - partially stabilized zirconia

FSZ - fully stabilized zirconia

3Y-Z – zirconia with 3 mol% added yttria

4Y-Z - zirconia with 4 mol% added yttria

5Y-Z – zirconia with 5 mol% added yttria

FDP - fixed dental prostheses

PTT - phase transformation toughening

LTD - low temperature degradation

CAD/CAM – computer aided design/computer aided manufacturing

HIP - hot isostatic pressing

CTE - linear coefficient of thermal expansion

RCT - randomized controlled clinical trial

SM – soft-machined

HM – hard-machined

VHN - Vickers hardness (number)

SEM - scanning electron microscope

1. Introduction

1.1 Dental ceramics

Dental ceramics can roughly be organized into three groups: (I) porcelain (feldspathic); (II) glass-ceramics; and (III) polycrystalline oxide ceramics (1). Dental porcelain derives principally from feldspar-quartz-kaolin compositions and mimic well the optical properties of natural teeth (2, 3). Their three-dimensional network of atoms has no regular pattern and have an amorphous (glassy) structure. Dental feldspathic porcelains have low strength and resistance to crack propagation, that in turn limit their clinical indications to low stress-bearing restorations (4-7).

Glass-ceramics are strengthened feldspathic porcelains Lithium disilicate-based ceramics are achieved by a “ceraming” process, where crystals are precipitated under controlled heat-treatments from homogeneous glass through the nucleation and growth processes (8-11). Clinical indications of glass-ceramics include onlays, inlays, crowns and as veneering layer. Such reinforced feldspathic porcelain restorations have shown good long-term success rates when bonded to and supported by sufficient amount of tooth structure (12, 13).

Polycrystalline ceramics contains no amorphous components, and the atoms are densely packed into regular arrays and are the strongest dental ceramics (1, 14). During packing and firing from powder form into fully dense material, the polycrystalline ceramics shrink around 30% by volume (1). Thus, two key developments were important in enabling polycrystalline ceramics in dental use: (1) the availability of highly controlled starting powders and (2) the application of CAD/CAM technology (1). The two types of polycrystalline ceramics used in dental industry are aluminium oxide (Al_2O_3) and zirconium dioxide (ZrO_2), where zirconium dioxide has become predominant in later years.

1.2 Zirconia

Zirconium dioxide (ZrO_2), also called zirconia, is a polycrystalline oxide ceramic available as a white, high temperature fusing crystalline powder (15). Single crystals group into a grain and grains group into monoliths. Each grain can be oriented randomly and forms grain boundaries where grains meet one another. At ambient pressure, pure zirconia can assume three crystallographic phases/crystal structures depending on the temperature: monoclinic (*m*) at room temperature, tetragonal (*t*) above 1,170 °C and cubic (*c*) above 2,370 °C (14) (Figure 1).

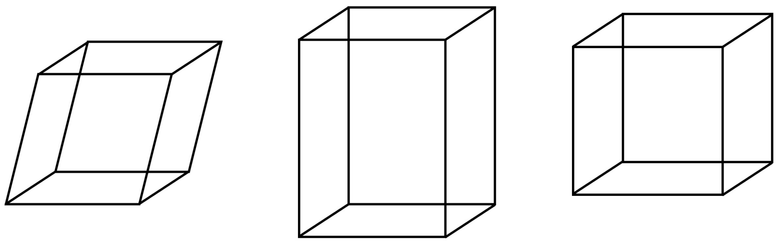


***Figure 1**. Three crystallographic phases/crystal structures of zirconia: (a) monoclinic phase; (b) tetragonal phase; (c) cubic phase.*

During cooling the process is reversed (*c-t-m*). The transformation from *t*-phase to the *m*-phase results in increase in volume (~ 4%) and leads to excessive high tension and the development of unwanted cracks in the zirconia structure. Retention of tetragonal structure can be achieved by addition of stabilizing oxides such as CaO, MgO, Y_2O_3 or CeO_2 (14). Of the various stabilizing oxides, yttria (Y_2O_3) has shown to provide a combination of high strength and toughness. At the microstructural level, the composition of tetragonal and cubic phases of zirconia material depends on the amount of added yttria (Figure 2). Addition of approximately 3% mol (5.2 wt%) yttria (Y_2O_3) results in an yttria stabilized tetragonal zirconia polycrystal (3Y-TZP) which has metastable tetragonal phase at room temperature and has become the most widely used dental ceramic for prosthetic restorations (14, 16).

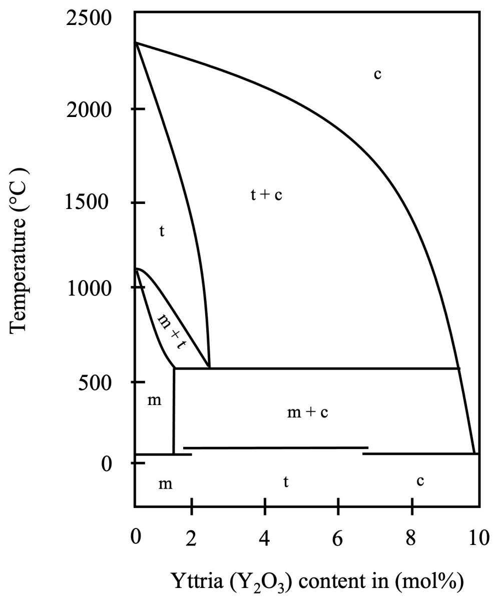


Figure 2. *Zirconia-yttria phase diagram. The figure is modified from Piconi and Maccauro (1999).*

1.2.1 Phase transformation toughening (PTT) and low temperature degradation (LTD) of zirconia

3Y-Z is a metastable zirconia material consisting of both tetragonal and cubic phases. Upon impact of stresses, the tetragonal zirconia crystals convert to monoclinic shape. During this action, due to larger volume of the monoclinic zirconia crystals, an increase in volume of 4-5% occur. This mechanism is known as *t-m* phase transformation toughening (PTT) (14, 17-20). This *t-m* transformation has been considered advantageous in 3Y-Z since it prevents further the propagation of micro-cracks and fractures within the material by compressive stress around the crack tip (20-22).

However, the *t-m* transformation may also be induced in the humid environment, even in the absence of mechanical stress. At room temperature, the *t-m* transformation proceeds slowly and spreads gradually along the surface of the zirconia. Increased amount of monoclinic phase causes a deterioration of mechanical properties and responsible for ageing of the metastable zirconia (14, 17-24). This mechanism is known as low-temperature degradation (LTD) and is affected by several factors, such as percentage and distribution of stabilizing oxides, crystals dimension mechanical stress and manufacturing techniques. However, zirconia materials with higher proportion of cubic phase are less prone to LTD due to absence of *t-m* transformation (22, 25).

1.2.2 Production methods

Dental zirconia restorations can be fabricated by CAD/CAM technology with two possible methods, either by soft-machining or by hard-machining. Soft-machining is based on milling of pre-sintered zirconia blanks fabricated by cold isostatic pressing a mixture of zirconia powder. After the milling, the zirconia framework is sintered at high temperatures to obtain the desired density and mechanical properties. The sintering induces shrinkage of approximately 25% and can thus cause changes in volume and dimensions of the zirconia restoration (14, 17-19). Nevertheless, soft-machining has become the most commonly used manufacturing method due to its cost effectiveness.

By hard-machining, the restorations are milled from fully sintered zirconia blanks produced with hot isostatic pressing (HIP) at 1,400°C-1,500 °C. Although, an oversizing of the framework before the milling is not necessary in this method, several disadvantages need to be noted. Complex processing with longer milling time and wear of the machinery lead to higher cost. In addition, due to mechanical stress during milling, a certain amount of monoclinic phase transformation can occur in the zirconia frameworks which can have an adverse effect on the material properties (21).

1.2.3 Restoration design

Zirconia can be fabricated as bi-layered construction with a veneering layer or as monolithic restorations. Due to the opaque appearance of the 3Y-TZP zirconia a veneering layer of porcelain was needed to achieve desirable aesthetics. Thus, the early all-ceramic restorations were produced as a bi-layered construction. Although this production method led to aesthetically pleasing restorations, excessive amount of healthy tooth substance needed to be removed in order to provide enough space for the two layers. In addition, cohesive fractures of the veneering layer, known as “chipping” occurred frequently during clinical use (26-28). Zirconia materials with higher translucency can be produced as monolithic crowns that are less affected by cohesive fractures of the veneered ceramics (26, 27). In addition, since the entire restoration consists of high-strength ceramic, the need for tooth substance removal is less than with bi-layered restorations. The minimum thickness suitable for monolithic Y- TZP restorations is 0.5 mm; whereas for veneered restorations, the total thickness ranges between 1.0 and 1.5 mm (26, 27).

1.2.4 Zirconia generations

Zirconia materials can currently be divided into several types/generations according to their mechanical and optical properties (29).

First-generation 3Y-TZPs contained 0.25 wt% alumina (Al_2O_3) sintering aid and exhibited strength above 1 GPa in flexure (30). Due to its high flexural strength, it was used as a substitute for metal as framework materials in porcelain-veneered crowns and fixed dental prostheses (FDPs) (14, 17). However, 3Y-TZP zirconias exhibited high opacity due to inherent birefringence and light scattering from grain boundaries, pores and additive inclusions. They appear white and opaque and are thus contradicted for the fabrication of monolithic restorations.

In order to sufficiently improve the translucency of monolithic zirconia materials, the concentration of alumina additive was reduced, and the amount of pores was eliminated by sintering at a higher temperature (31). However, this led to modest improvements in translucency and thus these second generation zirconias still had insufficient aesthetics for use in the anterior region as monolithic restorations (32). In the next stage of monolithic zirconia development, more yttria was added. Higher amount of yttria resulted in partially or fully stabilized zirconias, 4 mol% (4Y-PSZ) or 5 mol% (5Y-FSZ), with higher fraction of cubic phase which has larger grain size than the tetragonal phase. Increase in grain size results in increased translucency. However, higher fraction of cibuc phase leads to reduced fracture toughness and flexural strength due to lack of t-m phase transformation (33). Significant reduction in strength and toughness in successive, more translucent generations, were reported by independent studies (25, 30).

In addition, sintering parameters such as temperature and holding time affect the zirconia microstructure and thus result in altered mechanical and optical properties of the final zirconia material (34-36). High sintering temperature and longer sintering time results in increased grain size (35, 36). Increased grain size leads to enhanced translucency, however the flexural strength reduces at same the time (37). The nomenclature of the different zirconia types is somewhat confusing and overlapping. Thus, to avoid confusion, the terms 3Y-Z and 5Y-Z will be used further in the text to identify the zirconia types with 3mol% and 5mol% yttria.

1.3 Clinical status

In the early 2000s, the first-generation of yttria-stabilized tetragonal zirconia polycrystal (3Y-Z) was introduced in prosthodontics as an alternative to metal-ceramic restorations (38) that were considered as the gold standard in restorative dentistry (39, 40). The metal-based restorations had insufficient aesthetic appearance such as dark margins and possibility of allergic reactions to alloy components (41, 42). The zirconia-based restorations had high strength and biocompatibility, however due to its white appearance the early zirconia materials were only used as frameworks for porcelain-veneered restorations (38). The initial clinical studies focused on comparison of clinical survival between metal-based and zirconia-based dental restorations, especially on fixed dental prostheses (28, 43, 44). A randomized controlled trial showed that the survival of zirconia-based and metal-based single crowns is similar over a follow-up period of 5 years, with estimative cumulative survival (97,7% and 97,4%) for zirconia-based and metal-based single crowns respectively (45). Chipping fractures of the veneering ceramic in both materials were the main failure modes, but no significant differences in aesthetic, functional and biological outcomes was demonstrated between the two groups (45). Also, several other studies showed that, porcelain chipping (cohesive failure) in veneered zirconia crowns was the major complication mode (15, 28, 46-48). The high rate of porcelain chipping has become one of the major drawbacks of veneered zirconia-based restorations. Possible reasons for of this include insufficient support of the veneering material by the framework design, excessive occlusal forces, improper clinical handling, mismatch of the linear coefficient of thermal expansion (CTE), and unfavorable surface and heat treatments (49). In order to improve mechanical performance of veneered restorations, the compatibility of CTE between veneering ceramics and zirconia material is of importance (50, 51).

Substructure design, surface quality, and firing parameters during the veneering process are considered as important factors influencing porcelain chippings. Recently published data show that the chipping failure of zirconia crowns can be reduced with adjusted framework desin. A simplistic and nonanatomical modeling of the zirconia core may result in inappropriate support of the veneering ceramic (52). Therefore, an optimized substructure design, as well as an optimized surface, reduced the number and size of chipping incidents *in vitro* (53). Anatomical support provided by a zirconia core results in uniform thickness of the layering material that better resist the load during mastication (53).

In an attempt to reduce the chipping rate in zirconia-based restorations, different approaches on veneering technique were explored. In a 5-year randomized controlled clinical study examining effect of overpressing ceramic on zirconia-based and metal-based single crowns, showed that chipping rate in both material types were low (45). An another 2-year follow-up study examining clinical performance of zirconia-based crown veneered with CAD/CAM-produced lithium disilicate ceramic showed that no technical complications such as veneer fracture were detected (54). Although these studies show promising results, they are based on small data sets and short follow-up time.

Monolithic zirconia fixed dental prostheses have gained popularity as a metal-free option (48). However, translucent zirconia with high amount of cubic phase, have shown to have lower fracture strength compared to previous generations of zirconia (55). Thus, one can expect increased fracture occurrences of monolithic translucent zirconia crowns during clinical use. The available clinical studies on monolithic zirconia crowns show that short-term survival and success rates are high, 90,5% and 92,8% respectively (56). Fracture rate of monolithic zirconia crowns fabricated at a dental laboratory and investigated for 7,5 years, was reported to be 0,54%, which was lower than layered zirconia single crowns (2.83%) (57, 58). However, there is limited information available regarding the clinical outcomes for monolithic zirconia restorations due to short follow-up time and small sample sizes (56, 59, 60). An overview over some clinical studies on zirconia-based crowns are presented in table 1 (45, 46, 48, 54, 61-75).

Table 1. *Overview of some of the clinical studies that are available on zirconia-based single crowns. Key information such as type of clinical study, time length, sample size, number of crowns, placement of the crowns and number of observed fracture failures are summarized. * - randomized controlled study.*

|Year	Author	Time span	Sample	No. Rest.	Place-ment	Fracture, veneer/core
2009	Cehreli et al *	2 yr	slip-cast glass-infiltrated alumina/zirconia crowns vs CAD/CAM zirconia crowns	30	post	1/1
2011	Tartaglia et al	3 yr	zirconia-based crowns vs FDPs	238	ant/post	0/0
2012	Örtorp A et al	5 yr	zirconia-based crowns	216	ant/post	6/0
2013	Monaco c et al *	1-5 yr	zirconia-based crowns: different core and veneer materials	1132	ant/post	62/0
2014	Gherlone E et al	3 yr	zirconia-based crowns produced by intraoral digital impressions	86	ant/post	26/0
2015	Seydler et al	2 yr	zirconia med LD_2 veneer vs litium disilicate monolithic	60	post	0/0
2015	Tartaglia et al	7 yr	zirconia-based bi-layer crowns and FDPs	150	ant/post	3/0
2015	Nejatidanesh et al	3-7 yr	zirconia-based crowns	556	ant/post	45/
2017	Monaco et al *	5 yr	zirconia-based vs metal-based with overpressing veneer on endodontically treated teeth	90	post	5/1
2017	Dogan et al	5-yr	zirconia-based anterior crowns with customized zir copings	20	ant	0/0
2017	Bomicke et al	3-yr	monolithic vs buccal partially veneered zirconia crowns	162		1/0
2018	Miura s et al	up to 12 yr	zirconia-based, bi-layer	137	ant/post	16/0
2018	Hansen T et al	1-3 yr	monolithic ziconia crowns	84	ant	4/1
2018	Kitaoka A et al	2 yr	monolithic zirconia crowns	26	post	0/0
2019	Koenig V et al	2 yr	second generation monolithic zirconia restorations (crowns and FDPs)	95	post	1/1
2019	Tang Z et al	up to 96 W	monolithic zirconia crowns	49	post	0/0
2019	Cagidiaco E et al *	4 yr	zirconia slice vs chamfer	50	post	9/0
2020	Hammoudi W *	up to 6 yr	pressed litium disilicate crowns vs translucent zirconia crowns	713	ant/post	2/0
2020	Miura S et al	3.5 yr	monolithic zirconia crowns	40	ant/post	0/2

The necessity for long term clinical studies lasting longer than 5 years is clearly shown in the case of shrinkage-free $ZiSiO_4$ ceramic that was developed during second half of 2000. *In vitro* tests that were done to confirm clinical applicability of a novel $ZrSiO_4$ (zircon) based shrinkage-free ceramic, showed that the mechanical properties such as flexural strength, fracture toughness and chemical solubility were adequate for recommendation for clinical use (76, 77). Thereafter a prospective randomized controlled trial for posterior crowns showed high survival rate (89,8%) after an observation of 2 years (78). However, at 5 years follow-up of the same posterior $ZrSiO_4$ restorations, the survival rate was reduced to 73,2% compared to the control group (92,3%). Thus, the study concluded that use of $ZrSiO_4$-ceramic crowns in posterior position was not recommended (79). This case shows that new material development in dentistry should be followed up closely and often clinical performance confirmed with long-term studies. Nevertheless, new materials are being introduced in the field of dental ceramics at a rapid pace where a complete examination of the new materials is lagging. Thus, it is even more important to consider reports from the clinicians. Several cases of early failure of zirconia crowns during clinical use have been reported by clinicians, despite good results from clinical trials.

1.4 Laboratory testing

Clinical trials are time consuming and costly. During the development of new or modified dental materials, basic tests are performed to investigate clinical applicability by assessing mechanical properties such as strength, toughness and hardness. The setup for such standardized tests is given in international standards guidelines (80-82). Such tests are cost effective and can be performed in a controlled laboratory setting (22, 55, 83). The disadvantage of such tests, however, is that the tests are simplified and often use standardized bars and discs (25, 84-86). Thus, the results do not necessarily fully simulate a clinical situation. At the same time, the results of such laboratory tests provide a minimum of information that indicate whether a material is suitable for clinical use or not and whether to proceed to clinical trials. However, strength values of the ceramic materials can change, when testing complex geometry of anatomically shaped ceramic material (87). Some types of tests such as load to fracture, aging tests and hydrothermal cyclic loading tests have been widely used in dental ceramics. Recently, guidelines have become available with aims to provide practical guidance in test methods of dental ceramic systems (88-90). Over time, many laboratory tests have been performed that examine dental ceramic systems and try to simulate clinically similar situations, but the test method setup has varied greatly so direct comparison of the results is difficult. Although a laboratory test on anatomically formed ceramic restorations provide more clinically relevant information than the standardized disc tests, it will not be able to provide full insight into the effect of all the factors present in a clinical situation (91, 92).

1.5 Fractography in dental material testing

Since the late 2010s, the use of fractography has been gradually introduced in the field of dental ceramics. Fractographic analysis of cracked ceramic restorations can serve as a validation method for laboratory tests. Fractography is used in industrial material technology, where the method is used to map the causes of failure and breakage in material technology (93, 94). Recently, recommended guidelines have been published on how fractographic examination of clinically fractured crowns can be performed (90). This method involves examining fracture surfaces and using signs of the fracture surface to provide information about where the fracture started, how it propagated and possibly identifying the root cause (94-98). Studies examining the fracture mechanism of clinically fractured ceramic restorations provide information on crack initiation and propagation and the possible cause of fracture onset (99-101). These studies showed that most fractures started at the crowns´ marginal edge, which was not entirely consistent with the results from laboratory tests where fractures started from the occlusal surface with contact damage from the loading indentor. By using these findings, the quality of both laboratory and clinical studies could be improved. It is common that a certain time need to pass before a new method is implemented fully in research field, but several recent clinical studies have used fractographic analysis to elucidate cause of failure (56, 67, 98).

2. Aims

The main aim of the present work has been to identify factors affecting fracture of zirconia-based dental crowns in order to improve the clinical survival time.

The specific aims were:

- ***Study I***: to investigate the applicability of fractography as a method for analysis of fractured zirconia-based crowns and assess the fracture pattern and identify cause of failure in zirconia-based crowns failed in clinical use.

- ***Study II***: to analyze effect of different preparation and crown margin designs on load at fracture for bi-layer zirconia-based crowns.

- ***Study III***: to investigate effect of the aging tests that simulates a potential early failure of monolithic zirconia crowns with different material compositions (3Y and 5Y).

3. The hypothesis

Several factors may have an effect on the clinical survival time of zirconia crowns. Different production methods, variation in the amount of added yttria and varying zirconia material thickness can affect the clinical survival time for zirconia crowns differently. The hypotheses were:

H0: Thin or uneven crown margins will reduce the fracture resistance of all types of zirconia crowns. Hard-machined crowns are more fracture resistant than soft-machined crowns. Furthermore, increased yttria content will reduce the fracture resistance, but increase the aging resistance of soft-machined zirconia.

4. Material and methods

The present work consisted of three studies. In study I, retrieved zirconia-based crowns were examined using fractographic analysis to determine fracture pattern and origin areas. In studies II and III, zirconia-based cores and crowns were subjected to *in-vitro* tests in order to examine the effects of various factors on the crowns´fracture resistance. In figure 3, an overview information on material, sample size and methods for each of the studies are given.

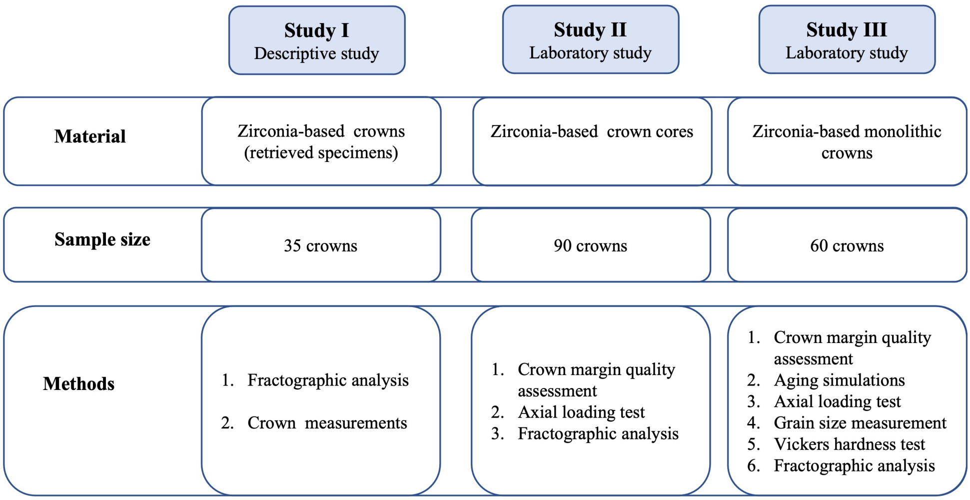


Figure 3. *An overview information on material, sample size and methods for each of the studies.*

4.1 Material and sample selection

4.1.1 Study I – Retrieved specimens

The sample size consisted of retrieved crowns and crown fragments failed during clinical use due to chipping or total/partial fracture. The specimens were submitted by public and private dentists. Supplementary information on crown function time, cement type, zironia type and brand names was not available. The specimens were submitted without any patient-related information and thus did not require any ethical concidérations and approvals. Thirty-five, bi-layered (n=15) and monolithic (n=20), zirconia-based crowns were retrieved. Both anterior and posterior crowns were represented among the bi-layered and monolithic crowns (Table 2). Four monolithic anterior crowns with incisal chipping were retrieved from the same patient and their fracture details were obtained by replica technique (94). The rest of the fractured crowns were all retrieved from separate individuals. Three of the crowns had been cemented on implant abutments and remaining crowns had been tooth-supported.

Table 2. *Almost an even distribution of anterior and posterior crown types in the bi-layered and monolithic crowns.*

	Bi-layered	Monolithic
Anterior (incisive and canine)	8	7
Posterior (premolar and molar)	7	13
Total	n = 15	n = 20

4.1.2 Study II and III – laboratory studies

In both study II and III, crown-shaped specimens were subjected to several laboratory tests in order to examine the effect of different variables on load at fracture for bi-layered and monolithic zirconia crowns. An overview information on sample sizes and material selection is presented in figure 4. In study II, the effects of different crown margin designs and production methods on load at fracture were examined. In study III, the effects of aging simulations in two different zirconia materials were investigated.

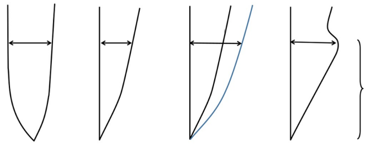
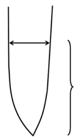
	Study II	Study III
Sample size	90 crowns	60 crowns
Sample groups	9 groups	6 groups
Crown type	Bi-layered crown cores	Monolithic crowns
Preparation type	 chamfer  slice	 chamfer
Crown margin design	 no collar  margin collar	 no collar
Crown margin thickness (in cross section)	 0.80 mm; 0.56 mm control; 0.50 mm slice; 0.63 mm slice with veneer; 0.70 mm slice with collar	 0.80 mm; 0.70 mm
Production type	Hard and soft-machined	Soft-machined
Zirconia type	3Y-Z	3Y-Z and 5Y-Z
Zirconia brand names and manufacturers	- Soft-machined: Zerion HSC zirconium (Straumann) - Hard-machined: Denzir (Denzir Cad.esthetics)	- 3Y-Z: DDBioZX² (Dental Direkt) - 5Y-Z: DDcubeX² (Dental Direkt)
Abutment tooth material	Epoxy resin: EpoFix (Struers)	Composite resin: SDR flow+® (Dentsply Sirona)
Cementation material	Zinc phosphate oxide cement: De Trey Zink (Dentsply)	Resin cement: RelyX Unicem (3M)

Figure 4. *An overview information on sample size, preparation types, crown margin designs and materials in study II and III. Measurements of the wall thickness of the four margin designs in study II are shown in cross section. The thickness is measured at the same height as the upper limit of the cervical collar (0.80 mm). The choice of zirconia materials was based on which zirconia materials were available on the market and at dental technician at the time.*

Sample selection – study II

In study II, a combination of two different preparation types with two crown margin designs resulted in three different groups (control, slice and slice with collar) (Figures 4 and 5). In addition, an extra group of crowns with reduced occlusal thickness (0.4 mm) were produced on the slice preparation with a 0.8 mm high cervical collar (Figure 5). In order to examine the effect of different zirconia production methods (hard-machined vs soft-machined), the four crown design groups were produced in both hard-machined and soft-machined zirconia cores. An additional group of ten hard-machined crowns on slice preparations were veneered with porcelain, to assess the effect of the veneering procedure on load at fracture values (Figure 5).

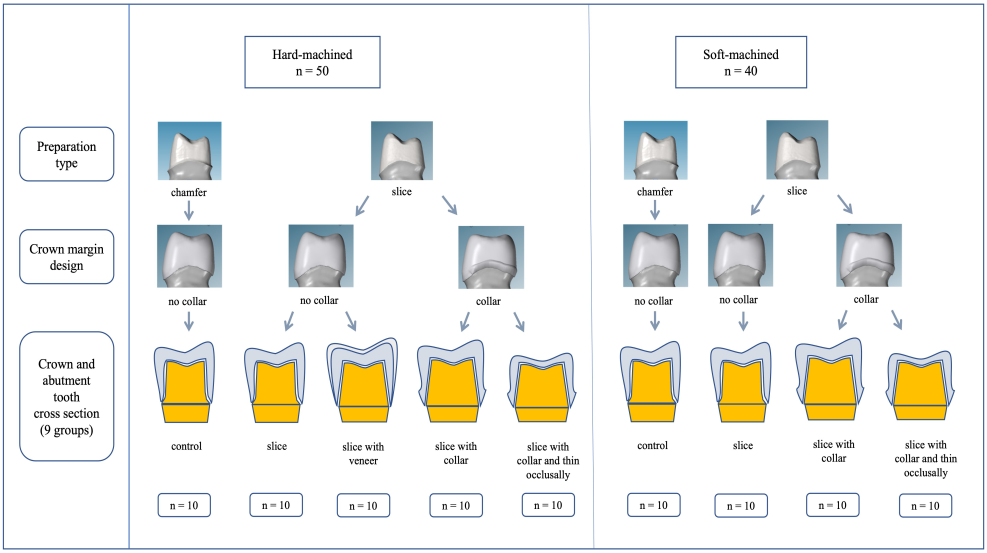


Figure 5. *Flow chart of sample selection in laboratory study II. Ten crowns each from both soft and hard-machined zirconia, were manufactured for each of the four designs: control, slice preparation without collar, slice preparation with collar normal and thin occlusal thickness. In addition, 10 hard-machined crowns made for the slice preparation were veneered and glazed with feldspathic porcelain.*

Sample selection – study III

In study III, a total of 60 monolithic zirconia crowns were made from two different zirconia materials: super high translucent zirconia (5Y-Z, n = 30) and high translucent zirconia (3Y-Z, n = 30). Chamfer preparation type was used. The sample size of ten crowns in each group was based on the results from the pilot studies and in consideration of the non-normality of the distribution of fracture values from similar laboratory studies on zirconia crowns. The crowns from each material were divided into three groups of ten crowns in each: dynamic loading, hydro-thermal exposure, and a control group of untreated specimens, in order to examine the effect of short-term aging procedure on monolithic zirconia crowns (Figure 6).

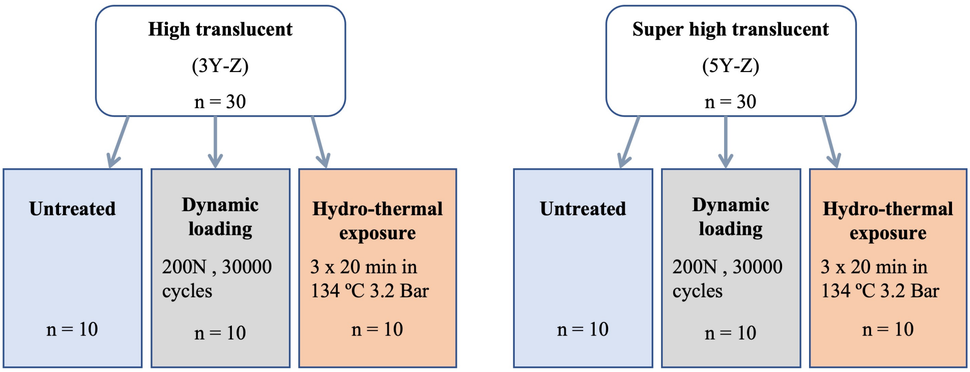


Figure 6. *In laboratory study III, the crowns from each material were divided into three groups of ten crowns each: dynamic loading, hydro-thermal exposure and a control group of untreated specimens.*

Abutment material and cementation procedure – study II and III

In study II, the crowns were cemented with zinc phosphate oxide cement (De Trey Zink; Dentsply DeTrey, Konstanz, Germany) on individual epoxy abutment models of the preparations (EpoFix; Struers, Ballerup, Denmark). Zinc phosphate oxide cement was chosen to minimize the bonding effect of the cement. Excess cement was removed and after a 5-min setting time, the crowns were stored in distilled water at 37 ºC for 24h.

In study III, individual abutments in a polymer composite restorative material (SDR flow+; Dentsply Sirona, Charlotte, NC, USA) were made for each crown. The abutments were stored for a period of 45 days prior cementation. The crowns were cemented with a self-adhesive resin cement (RelyX Unicem Aplicap Capsule; 3M ESPE, St. Paul, MN, USA) with occlusal pressure of 50N for 10s. Excess cement removed and after a 5-min setting time, the crowns were stored in distilled water at 37 ºC for 24h.

4.2 Test methods

The test methods used in this thesis are presented below. Information on which test method was performed in each study is listed in figure 3 and presented in the order in which they were performed.

4.2.1 Fractographic analysis

In study I, the retrieved specimens were cleaned prior to fractographic analysis in order to remove remnants of biofilm and tartar. The crown fragments were cleaned for fifteen minutes in an ultrasonic bath containing 1% enzymatic detergent solution followed by rinsing with distilled water and airdrying.

The specimens in all three studies were inspected to determine the directions of crack propagation and identify the location of the fracture origin. Fractographic analysis was performed by first getting an overview of all the fragments of the fractured crown. Then assembling the fragments to reconstruct the original crown shape to aquire an overview of the fracture pattern. The fracture propagation and fracture origin area are determined by by interpreting fractographic features such as compression curls, wake hackle, arrest lines, crack branching and hackle lines (Figure 7) (90). The fracture surfaces were first examined in optical microscopy (Leica M205 C, Leica Microsystems, Wetzlar, Germany) then in a scanning electron microscope (SEM) (Phenom XL Desktop SEM, Thermo Fisher Scientific, Waltham, MA, USA) in secondary emission mode sputter-coated specimens (gold thickness of 30Å). Images taken under microscopy were used to assemble fractographic maps of the matching pieces of each specimen where orientation, crack propagation and details of the fracture origin and other findings are included (Figure 7). The crown margin in the region of origin was inspected in detail from all sides. Defects at the crown margin and contamination by veneering material on the crown inner surfaces were noted.

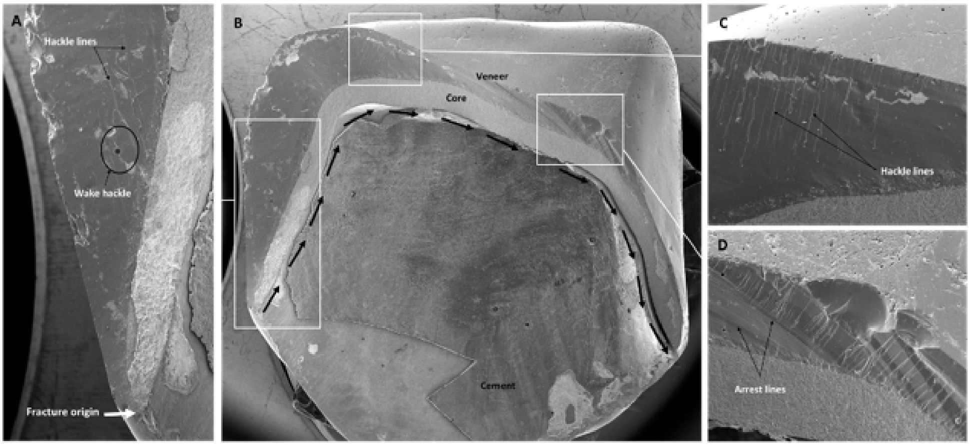


Figure 7. *A fractographic map of a bi-layered incisor zirconia crown fractured during clinical use (B, magnification x10) with the fracture origin at the crown margin, white arrow (A, magnification x30). Several fractographic features are visible in the veneer-layer such as wake hackle (A, black circle), hackle lines (C, magnification x30) and crack arrest lines (D, magnification x30). The direction of crack propagation is show with bold black arrows (A). SEM images by second emission mode.*

4.2.2 Crown measurements

In study I, the zirconia thickness was measured in three different distances from the crown margin in specimens with fracture origin at the crown margin. Crown axial wall heights in the same area were also measured (Figure 8). The thickness and axial wall heights measurements were compared with corresponding measurements on the opposite side of the same crown.

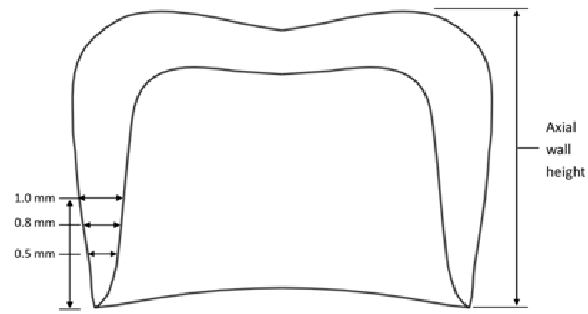


Figure 8. *A schematic illustration of the measurement points for zirconia thickness and wall height (cross-section).*

4.2.3 Crown margin quality assessment

In both laboratory studies, each crown was examined in an optical light stereomicroscope (Leica M205C, Heerbrugg, Switzerland) to assess their margin quality. Observed defects such as fractures and flaws were graded according to a severity scale, on a Likert scale, as follows: 1, optimal margins without flaws; 2, minor chips and flaws; 3, multiple chips and flaws; 4, continuous flaws or uneven margins; 5, large defects visible without a microscope (102) (Figure 9).

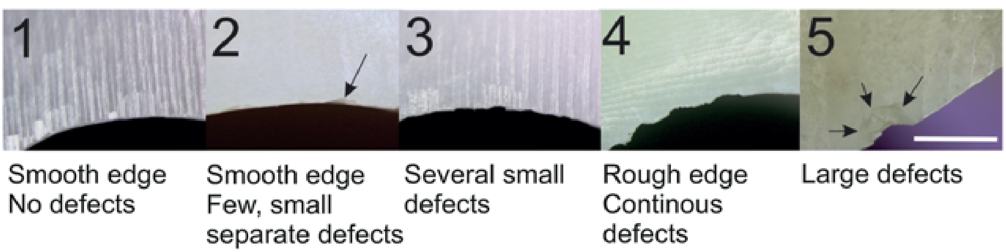


Figure 9. *Images 1-5 represents each grade on a scale for assessing crown margin quality. The horizontal white bar indicates 0.5 mm. Black arrows highlights defects. (Photo source: Schriwer C).*

4.2.4 Aging simulation – dynamic loading

In study III, ten crowns from each zirconia material type were subjected to 30,000 loading cycles of 60–200 N at 1 Hz in water (37°C) by pneumatically operated pistons (Festo). The crowns were loaded in the middle of occlusal surface with a spherical stainless-steel tip with 3 mm diameter. In order to reduce contact damage, a thin electro-tape was applied between the indenter and the crowns. After each period of 10,000 cycles, the crowns were examined for cracks or fractures in optical stereomicroscopy at 10x magnification.

4.2.5 Aging simulation – hydro-thermal exposure

In study III, ten crowns from each zirconia material type were exposed to three cycles in autoclave (134°C at 3.2 bar) (Tuttnauer3150EL; Tuttnauer, Breda, Netherlands). Each cycle lasted for 20 min and between each cycle, the crowns were dried at room temperature (21°C) and inspected for flaws in an optical stereomicroscope.

4.2.6 Axial load testing

In both studies II and III, the crowns were loaded occlusally with a horizontal cylindrical steel indenter of 13 mm in diameter. In order to avoid contact damage on the occlusal surface, a 2-mm-thick ethylene propylene diene rubber disc of hardness 90 Shore A (EPDM 90) was used as a cushioning layer between the indenter and crown. The cylinder was placed centrally at the occlusal fossa to ensure even distribution of load between the cusps (Figure 10). The load was applied in a servo hydraulic testing system at 0.5 mm/min until fracture occurred (MTS 852 MiniBionix II; MTS Systems, Eden Prairie, MN, USA). The crowns were immersed in water at room temperature during load testing. For the crowns in study II, the loading procedure was halted at 3,300 N due to limitation of epoxy abutment material.

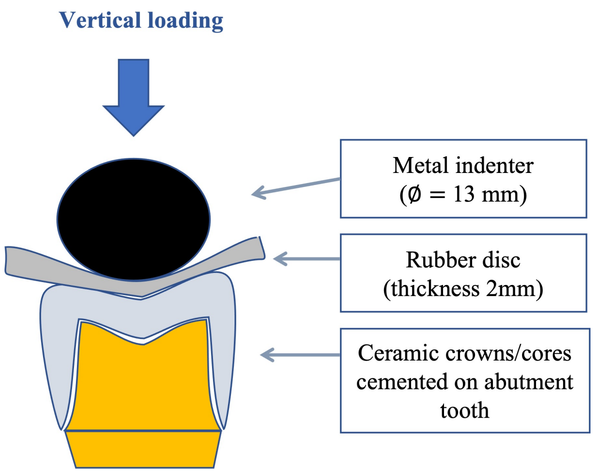


Figure 10. *The test method set up for axial load testing procedure.*

4.2.7 Hardness and damage tolerance measurements

In study III, in order to assess and compare microhardness of the two diferrent zirconia materials, the Vickers hardness (VHN) was measured after the aging procedures and axial loading of the crowns were completed. In addition, the average diagonal of the indent (a), as well as length of the cracks radiating from the indents (c), were measured (Figure 11). Calculation of the ratio between the crack length and the diagonal length of the indent (*c/a*-ratio) was used as an indicator of the crack propagation resistance (damage tolerance) (50). For both zirconia material types, two crowns from each of three test groups were randomly selected. The fractured crown parts were embedded in epoxy resin (EpoFix; Struers, Ballerup, Denmark) and cut to a flat surface with a diamond cutter. All resin blocks underwent a series of grinding and polishing steps with cleansing in ultrasonic bath between each grinding and polishing round to obtain a flat, polished surface suitable to perform the indentation test. Fifteen measurements were made on each crown using a microhardness tester (Zwick Roell Indentec; ZwickRoell, Ulm, Germany) with load of 150 g applied for 10 s at three different regions (axial wall and occlusal) with five measurements in each region.

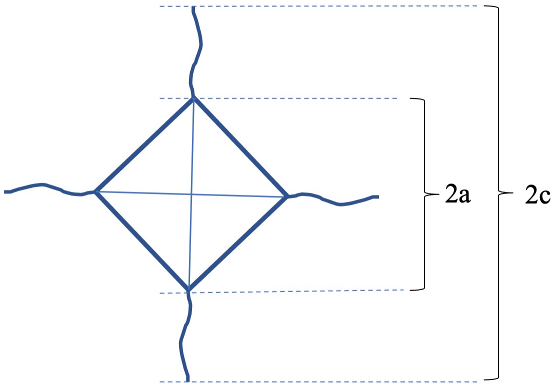


Figure 11. *Schematic illustration of the measurementsperformed for damage tolerance calculation, where 2a is the diagonal length of the indent and 2c is the total crack length.*

4.2.8 Grain size measurements

In study III, the grain sizes for 3Y-Z and 5Y-Z zirconias were measured by the Feret´s diameter method. The longest diameter of the grains was measured and compared in SEM micrographs at magnification of 29,500 (Figure 12).

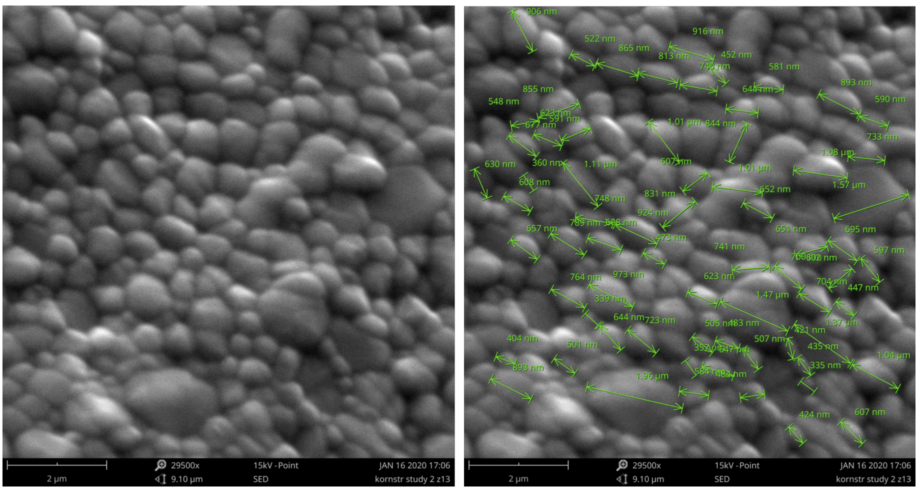


Figure 12. *Varying grain sizes and composition for high translucent (3Y-Z) zirconia crown, left (29,500x). Measurents of the longest diameter of the grains, right.*

4.3 Statistical analyses

In study I, distribution rate of different failure modes of the retrieved crowns, according to the location of the restoration (anterior versus posterior) and fabrication method (monolithic versus bi-layered) were compared and analyzed with chi-squared test (α = 0.05).

In studies II and III, due to limited number of specimens in each group and a tendency for skewed data, non-parametric methods were used to analyze the load at fracture values. The Kruskal-Wallis test was used for overall comparison and the Mann–Whitney U-test was used for between-group testing, with correction for multiple groups. Assessment of correlations was performed using Spearman's rank test. Significance was set to α = 0.05. The normality of the data was tested by Shapiro-Wilk test. The data were analyzed with a statistical software system (Stata 15, StataCorp, College Station, TX, USA).

4.4 Reliability and validity assessments

4.4.1 Fractographic analyses

Two operators performed the fractographic examination of every crown in all three studies. The interpretation of the fracture patterns and location of fracture origins were performed independently and blinded for the other operators results. In cases of discrepancy, both operators performed a joint analysis and came to an agreement (3 cases). If the operators could not reach agreement, the fracture was deemed to be uncertain (2 cases).

4.4.2 Microscope – calibration of size measurement

Prior to the descriptive analysis, the optical light stereomicroscope was controlled, and accuracy of the measurements calibrated with a micrometer scale with 10 µm divisions (Reichert Analystical Intruments, Vienna, Austria).

4.4.3 Crown margin quality – test retest

In both studies II and III, the test-retest of margin quality asessment showed good strength of agreement with Kappa analysis (0.8) in inter and intra-operator reliability and repeatability (103).

4.4.4 Axial load testing

Prior to axial loading of the crowns in laboratory studies, the servo hydraulic testing system (MTS 852 MiniBionix II; MTS Systems, Eden Prairie, MN, USA) was calibrated and the test set up was controlled.

4.4.5 Hardness – test training and assessment

Prior to the hardness and damage tolerance measurements of the fractured crowns in laboratory study III, the calibration of the microhardness tester (Zwick Roell Indentec; ZwickRoell, Ulm, Germany) was performed by the laboratory technician. Ten measurements on polished zirconia surfaces from 4 different fractured crowns were performed for training purposes and measurement accuracy validation.

4.4.6 Pilot tests

Three pilot tests were performed in order to determine best suited abutment material and short-term aging procedure in study III.

Firstly, 15 monolithic 5Y-Z crowns were subjected to 100,000 loading cycles of 60–200 N at 1 Hz in water (37°C). The load was applied occlusally in the middle of the occlusal surface by pneumatically operated pistons (Festo, Esslingen, Germany). The crowns were mounted on metal abutments with silicone material (Speedex Medium; Coltène/Whaledent, Altstätten, Switzerland) as substitute for cement. Ten of the crowns fractured before reaching 20,000 cycles. The remaining five crowns did not fracture. Thus, the number of dynamic loadings were set to 30 000 cycles in the main test.

The second pilot test investigated the choice of appropriate composite material for individually made abutments. Four monolithic crowns, two from each zirconia type (3Y-Z and 5Y-Z) were cemented on abutments from three different composite materials: (i) SDR flow+ (Dentsply Sirona, Charlotte, NC, USA); (ii) Filtek Supreme XTE (3M ESPE, St. Paul, MN, USA); or (iii) Tetric Evo Ceram (Ivoclar Vivadent, Schaan, Liechtenstein). The abutments were prepared by increments of two layers (SDR flow+) or four layers (Filtek S and Tetric Evo C), with light curing protocols at room temperature of 23°C and 40% humidity according to the manufacturer's recommendations. Each individual crown was used as mold for the associated abutment. The crowns were subsequently loaded axially until fracture. All crowns had fracture values between 1,801 N and 4,883 N, which is considered clinically relevant since these fracture values were higher than the recorded maximum bite force values given in the literature. After the crowns were fractured during axial loading, all the composite abutments were assessed for vertical deformations. The deformation was assessed by measuring changes in the composite material height visually in an optical stereomicroscope (Leica M205 C; Leica Microsystems, Wetzlar, Germany) at 10x magnification. The least deformations were observed in abutments made of SDR flow+, thus this composite material was chosen to be used in the actual study.

In the third test, four monolithic crowns, two from each zirconia type (3Y-Z and 5Y-Z), were cemented on individually made composite abutments (SDR flow+) with self-adhesive cement (RelyX Unicem Aplicap Capsule; 3 M ESPE) according to the manufacturer's recommendation. Then, the crowns were subjected to 30,000 loading cycles of 60–200 N at 1 Hz in water (37°C) and subsequently loaded axially until fracture.

5. Results

5.1 Study I - Descriptive fractographic analysis

An overview of the main results in study I are given in figure 13.

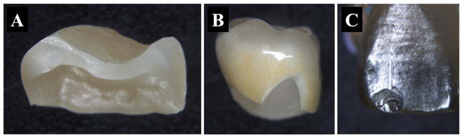
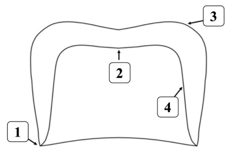
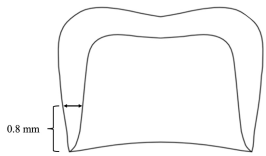
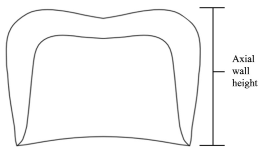
Failure modes	Three failure modes: A. Total fracture – 25 B. Semilunar fracture – 6 C. Incisal chipping – 4	
Fracture propagation pattern	Two main patterns: 1) Margin to margin – 23 2) Occlusal to margin – 2	
Fracture origin	Four origin areas: 1) Crown margin – 23 2) Occlusal intaglio – 2 3) Incisal – 4 4) Inner axial wall – 2	
Defect at the fracture origin	Two main defects types: 1) Manual adjustment – 4 2) Material defects – 4	
Thickness measurements (mean value at 0.8mm from the crown edge)	At the fracture origin area: - Bi-layered – 0.454 mm - Monolithic – 0.403 mm At the opposing area: - Bi-layered – 0.541mm - Monolithic – 0.603mm	
Axial wall height measurements	At the fracture origin area: - Mean – 5.261 mm - Min – 2.187 mm - Max – 9.756 mm At the opposing area: - Mean – 6.789 mm - Min – 4.339 mm - Max – 10.538 mm	

***Figure 13**. The fractographic analysis showed three main failure modes in the retrieved crowns. Fracture propagation pattern and fracture origin areas were identified. For eight of the crowns with fracture origin at the crown margin, defects in the crown material and manual post-production adjustments were noted at the origin area.*

5.1.1 Fractographic analysis

Overall examination of retrieved clinically fractured zirconia-based crowns showed three failure modes: total bulk fracture (n=25), semilunar fracture at the crown margin (n=6) and incisal chipping (n=4) (Figure 13). Fracture origins were identified for all the crowns except for four crowns due to missing fragments. Four different fracture origin areas were observed: at the crown margin (n=23), the inner surface of axial wall (n=2), the occlusal intaglio area (n=2) and the outer incisal surface (n=4) (Figure 13). The crowns with fracture start at the crown margin were equally distributed between bi-layered (n=12) and monolithic (n=11) crowns, and between anterior (n=9) and posterior (n=14) crowns. Only two crowns had fracture start at the occlusal intaglio area (Figure 13). Two other crowns that had fracture start at the inner axial wall were both previously cemented on implants. Defects at fracture origin area that may have acted as fracture initiators were observed for eight of the crowns. No definite conclusions could be made based on the statistical analysis due to low number of specimens.

5.1.2 Crown measurements results

The cross-sectional measurements at the crown margin are given in table 3. Overall, the measurements showed that margin thickness was 20% thinner at the fracture origin area compared to the area where fracture ended. The axial wall height measurement values varied depending on the type of the tooth type (Figure 13). The measurements of axial wall height showed that crown wall height at the fracture origin were 30% shorter than the opposing wall.

Table 3*. Crown margin thickness measurements at three different points, at both fracture origin area and opposing area. Measurements for bi-layered and monolithic crowns are given separately.*

Crown type		Measurement points					
		At the fracture origin area			At the opposing area		
		0.5 mm	0.8 mm	1.0 mm	0.5 mm	0.8 mm	1.0 mm
Bi-layered	Min (mm)	0.247	0.309	0.332	0.188	0.216	0.274
	Max (mm)	0.586	0.641	0.670	0.843	1.145	1.390
	Median (mm)	0.404	0.454	0.469	0.446	0.541	0.534
Monolithic	Min (mm)	0.173	0.286	0.186	0.305	0.389	0.422
	Max (mm)	0.795	1.015	1.184	1.278	0.871	1.018
	Median (mm)	0.348	0.403	0.493	0.494	0.603	0.626

5.2 Studies II and III – laboratory studies

An overview of the main results in study II and III are given in figure 14.

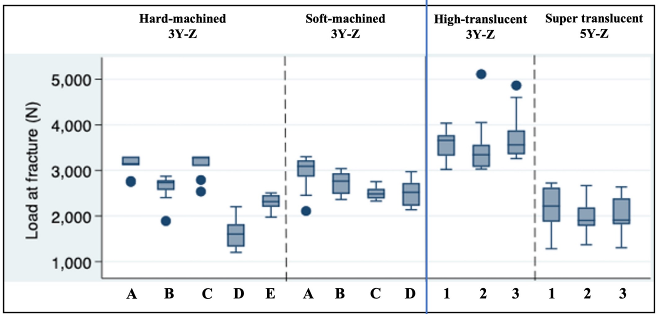
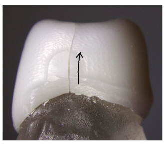
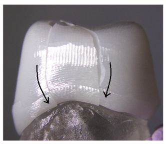
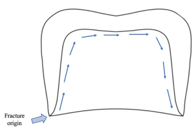
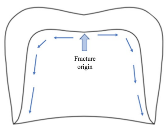
	Study II	Study III
Crown margin quality (median value on a 1-5 Likert scale)	Hard-machined: - Chamfer – 2 - Slice with collar – 3 - Slice no collar – 2 Soft-machined: - Chamfer – 2 - Slice with collar – 3 - Slice no collar - 3	3Y-Z: - Median – 3 - Min – 1 - Max – 5 5Y-Z: - Median – 3 - Min – 2 - Max – 5
Load at fracture values (N) given in box-plot	 **A** – chamfer **B** – slice no collar **C** – slice with collar **D** – slice with collar and thin occlusal **E** – slice with collar and veneer	**1** – control **2** – dynamic loading **3** – hydro-thermal exposure
Vickers hardness (HV) and c/a ratio	No data available	3Y-Z: - HV: mean – 1321 - c/a ratio: 1.5 5Y-Z: - HV: mean – 1357 - c/a ratio: 2.5
Grain size (nm)	No data available	3Y-Z: - Mean – 668 - Min – 250 - Max – 1960 5Y-Z: - Mean – 775 - Min – 208 - Max – 1880
Fracture propagation pattern	For both studies: 1) Margin to margin 2) Occlusal to margin  Margin to margin	 Occlusal to margin
Fracture origin area	For both studies – two fracture origin areas: 1) Crown margin 2) Occlusal intaglio  Origin at crown margin	 Origin at occlusal intaglio

Figure 14. *Crown margin quality was measured on a 1-5 Likert scale. The box plot of the load at fracture values of all the groups are shown. Microhardness test and grain size measurements were not performed in study II.*

5.2.1 Load at fracture values

In the study II, 11 of 90 crowns did not fracture, mainly crowns with chamfer preparation design. The load fracture values of the hard-machined crowns on chamfer preparation and crowns on slice preparation with outer collar and standard occlusal thickness had significantly higher values than the rest of the hard-machined groups. In the soft-machined groups, the crowns on chamfer preparation had significantly higher load at fracture values than the two groups with a cervical collar of both types of occlusal thickness (Figure 14).

In the study III, all the crowns fractured during axial loading post aging procedures. Each high translucent (3Y-Z) group had significantly higher load at fracture values than the corresponding super high translucent (5Y-Z) groups (Figure 14).

Comparing between the groups of both studies, high translucent groups had significantly higher load at fracture values than the groups of study II. Load at fracture values of the super translucent zirconia groups were in the same range of the study II.

5.2.2 Fractographic analysis

In both laboratory studies, two main fracture propagation patterns were observed: either fracture starting at the crown margin (Figure 14 and 15) or at the occlusal intaglio area (Figure 14 and 15). No crowns were fractured due to contact damage between the indenter and outer occlusal surface. In study II, most of the crowns had fracture origin at the occlusal intaglio surface (n=57) and for the remaining crowns the fracture originated at the crown margin (n=22). In study III, 3Y-Z crowns had an even distribution of the two types of fracture modes whereas most of the 5Y-Z crowns had the fracture origin at the crown margin. There was no correlation between fracture origin area and crown margin defects noted prior testing. The 5Y-Z crowns showed a predominantly trans-granular fracture mode, whereas inter-granular fracture modes were more prevalent in 3Y-Z crowns. The grains of the 3Y-Z were more rounded and homogeneous in size than the grains of 5Y-Z. There was a statistically significant difference in grain size between the two material types ($P < 0.0008$).

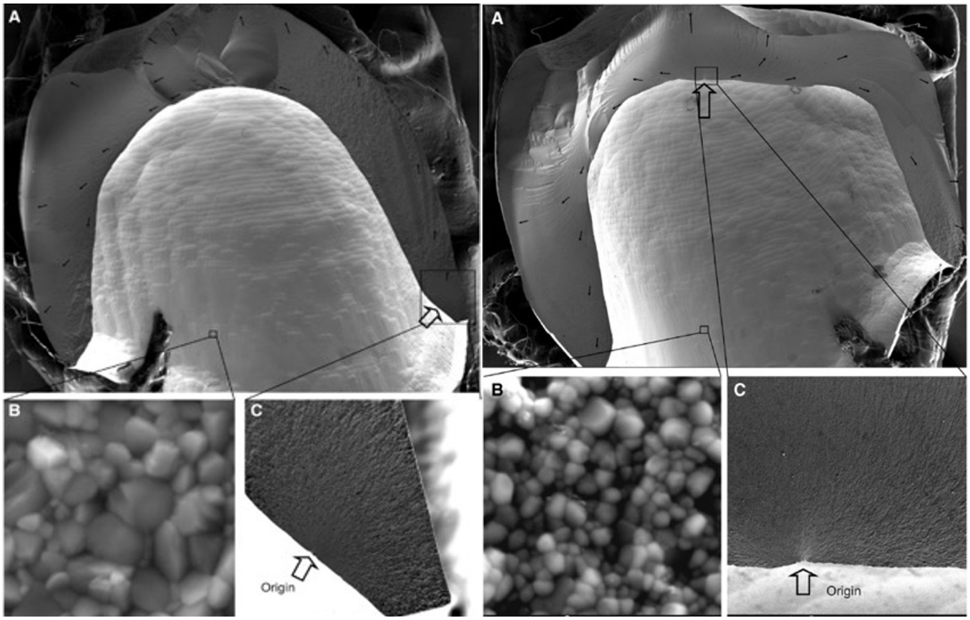


Figure 15. *Scanning electron images of fracture surfaces of a 5Y-Z crown with fracture origin at the crown margin (on the left) and a 3Y-Z crown with fracture origin at the occlusal intaglio area (on the right). Crack propagation is shown with black arrows in A. The fracture origin is shown with open arrows in A and C. Grain size and composition is shown in B (50,000x). SEM images by second emission mode.*

5.2.3 Hardness and damage tolerance

In study III, the Vickers hardness (VHN) value for the super high translucent (5Y-Z, mean value =1357) zirconia was significantly higher than the high translucent (3Y-Z, mean value = 1321) zirconia material. This indicates that the 5Y-Z is harder than the 3Y-Z. The total crack lengths of the 5Y-Z zirconia material were longer than the the 3Y-Z zirconia material. The calculated *c/a*-ratio values were significantly higher for the 5Y-Z. No significant differences in *c/a*-ratio were observed between the different test groups within each zirconia material type.

6. Discussion

Both biological and mechanical factors can lead to failure of the crown. The fractographic analysis in study I provide information on fracture mechanism of the retrieved specimens. The findings show that most of the fractures had started at the crown margin and thinner zirconia material at the origin area compared to opposing side. This indicates that the crown´s design and material thickness are of great importance. Which is further supported by the findings in study II and III, where different margin thickness and design as well as zirconia material type had a significant effect on load at fracture values. Overall fracture values of hard-machined zirconia crowns were not significantly higher than the soft-machined crowns. However, load at fracture values in hard-machined crowns were more affected more by variation in zirconia material thickness, than in soft-machined crowns. Increased yttria content did not affect the load at fracture values during short-term aging procedures. However, the high translucent (3Y-Z) crowns had significantly higher load at fracture than super translucent crowns (5Y-Z). However, one should be aware that in this thesis the effect of other factors such as cement type, crown surface treatment prior to cementation and sintering procedures were not investigated. Therefore, prior to concluding on which factors have an effect on the crown's survival in clinical use, the effect of the above-mentioned factors should be investigated further.

6.1 Fracture origin and pattern

Fractographic analyses of clinically failed all-ceramic dental restorations provide an approach to the determination of failure origin (104, 105). The findings in study I indicates that the crown margin in zirconia-based crowns is an area that is highly prone to fracture initiation during clinical use due to potential stress accumulation. Previuos fractographic studies of clinically failed all-ceramic restorations showed that all or majority of the fractures originated at the crown margin and usually from the approximal area close to the most coronally curvature of the margin (96, 97, 99). Majority of the crowns in study I had fracture propagation direction form margin to margin, including 11 crowns with fracture origin at the approximal area close to the most coronally curvature. The location of the fracture origins indicate that fractures are initiated by tension at the cervical margin. However, crown axial wall height measurements showed that crown wall height were 30% shorter than the opposing wall. This indicates that the tension at the cervical margin exerts stress not only at the approximale area, but around the entire crown circumference. Tensile stress at the crown margin can occur due to several stress situations, such as dentine expansion during occlusal loading, wedging forces or hoop stress from cementation (105-107). Modelling of fracture behaviour in biomaterials and engineering analysis demonstrated theoretical stress concentration at the cervical regions of teeth (108-110). In such areas with stress concentration, the presence of defects at the crown margin can act as a fracture initiators. A third of the crowns in the descriptive study with fracture origin at the crown margin, had defects in the zirconia material at the fracture origin. Similar observations were made in a previous study, which also showed that crowns had pores, contamination, or incomplete sintering that acted as fracture origins (99). Crowns fractured in both the laboratory studies had fracture origins either at the crown margins or at the occlusal intaglio area. A similar fracture pattern and origin in both descriptive study and laboratory studies suggest that the failure mode of the crowns in the laboratory tests simulate the clinical failure of the zirconia crowns to a certain degree.

6.2 Effect of crown design

Although clinical follow-up studies have shown that chipping of the veneer material is the most common technical complication, few cases of chipping were observed among the retrieved crowns. Crowns with chipping in clinical cases are most likely repaired *in situ* rather than replaced. The effect of bi-layer design versus monolithic design cannot be identified based on the present results as there was an even distrubution of monolithic and bi-layered zirconia crowns. The crown margin thickness seems to have an effect as, 17 crowns out of 23 crowns had fracture origin at the crown margin, had thinner margin thickness at fracture origin area than at the fracture ending site. The sample size was, however, too small to draw any conclusions on this causality. It seems that the thin crown margins were due to slice preparation of the abutment teeth.

However, the crown margin thickness was not even: both slice and chamfer preparations were often used in the same crown. In such crowns, the fracture started in the thinner part of the crown margin indicating that uneven margin thickness is unfortunate. The effect becomes even more apparent in bi-layered crowns, where the core is even thinner. The differing failure modes between bi-layered and monolithic zirconia crowns indicate that crown margin design is of importance for clinical survival. It is, however, not possible to quantify the effect based on clinical retreavals and this variable should be assessed *in vitro* in a controlled setting as perfomed in study II. This interpretation is further supported by load at fracture results in study II, where veneered crowns fractured at lower loads than the identical non-veneered crowns. However, only one group of ten crowns was veneered in this study and thus there is not enough data to conclude on. The findings of the study II showed that the crowns with chamfer design fractured at a higher load than did the crowns with slice design. The increased margin thickness in the collar resulted in an increase of almost 20% in load at fracture compared with crowns with slice preparation and uniform thickness. The additional collar results in fracture loads equal to the crowns made for the chamfer design. Similar findings are observed in studies that examined effect of preparation designs at the crown margin and material thickness, showing that higher load at fracture with increased zirconia thickness (106-117). However, one study did not find a strengthening effect of a cervical collar (118). Slice preparation may be associated with biological benefits as it requires less removal of sound tooth substance. The findings in study II indicate that a slice preparation with a modified crown design may increase both technical and biological success. In addition, reduced occlusal thickness resulted in weaker crowns, as expected based on previous studies (107, 110-112). This observation is supported by the measurements of the crown thickness in study I, where zirconia material was 20% thinner at the area where fracture originated compared to fracture ending area.

6.3 Effect of material composition (3Y-Z/5Y-Z)

The results in study III showed that the high translucent 3Y-Z had significantly higher load at fracture values than the equivalent groups of super high translucent 5Y-Z, which also were observed in previous studies (119-122). The material composition and larger grain size that enhance the translucency of the material are the most likely reasons for the reduced strength of the 5Y-Z (25, 123). The increase in translucency of the tested 5Y-Z was achieved by increasing the amount of yttria to 5 mol% (<10 wt%) and reducing the amount of alumina to <0.01 wt%, and thus achieving a higher proportion of cubic phase crystals. A higher proportion of cubic phase in 5Y-Z (approx. 50%) reduces the fracture toughening property due to reduced *t-m* phase transformation (21, 89). Almost all of the 5Y-Z crowns had fracture origins at the crown margin, whereas the 3Y-Z crowns had an even distribution of the two fracture modes. The observed fracture origins at the crown margin did not correlate with the margin defects identified prior to aging and testing procedures. This may indicate that the occurrence of margin defects is not one of the main causes of the clinical failure of zirconia crowns. The difference in grain size and crack propagation between 3Y-Z and 5Y-Z indicates that the difference in material composition has an effect on sintering quality and on difference in fracture mode.

A previous study showed that the flexural strength of 3Y-Z ceramic decreased only after the specimens were subjected to both mechanical and thermomechanical cycling (119). Furthermore, a study that compared the effects of mechanical and hydrothermal aging on microstructure and biaxial flexural strength of monolithic 5Y-Z and 3Y-Z showed that 5Y-Z had the lowest biaxial flexural strength values and it was affected when mechanical cycling was involved (121). Monoclinic transformation was observed in 3Y-Z when exposed to hydrothermal aging alone or in combination with mechanical cycling. No monoclinic transformation was found in any of the treatments for 5Y-Z (121).

6.4 Effect of production method and post-production handling

In study I, material defects were observed at the crown margin that may have acted as fracture initiators. These defects may occur as flaws within the material created during sintering processes or as margin flaw, that were introduced by post-production processing, such as manual adjustment of the crown margin. Material defects such as pores, contamination, or incomplete sintering can have acted as fracture initiators (99). This indicates that extra attention and care should be taken during CAD/CAM machining and any post-production manual adjustment of the zirconia crowns. Load at fracture values in study II, indicate that different crown margin designs had more effect in hard-machined crowns than in soft-machined crowns. Based on previous studies, a larger difference in load at fracture values were expected between the two different production methods (102). This difference may be a result of microscopic-level changes in the zirconia material created during the production by the two different methods. A final sintering of the soft-machined zirconia may have a reducing effect on the microscopic defects that may have occurred during milling. In addition, the post-sintering adjustment necessary on the margins of the soft-machined crowns to achieve optimal fit and crown emergence profile can affect fracture resistance. Manual adjustment of the margins could at least have had a reinforcing effect on the core by introducing a *t-m* phase transformation toughening. On the other hand, post sintering margin adjustment of the soft-machined crowns can also introduce new defects. Machining by diamond grinding is shown to be a major source of failure-inducing flaws in dense ceramics. Further studies are needed to add to the understanding of the connection between processing and fracture strength.

6.5 Effect of aging procedures

Clinical follow-up studies have shown that complete fracture of zirconia-based restorations can occur as early as within the first 2 years of clinical function (44). Fatigue treatments such as thermal exposure and dynamic cyclic loading were conducted to simulate a realistic clinical situation. In study III, the effect of short-term aging on two different zirconia dental materials was investigated. The effects of hydrothermal exposure and dynamic loading on zirconia monolithic crowns were assessed separately. The overall results show that the high translucent 3Y-Z had significantly higher load at fracture values after aging procedure than the equivalent groups of super high translucent 5Y-Z, which is also observed in previous studies (119-122). The material composition and grain size that enhance the translucency of the material are the most likely reasons for the reduced strength of the 5Y-Z (56, 57).

The current hydro-thermal exposure procedure aimed to simulate accelerated aging corresponding to approximately 3 years of clinical use. The hydro-thermal procedure had, however, no significant effect on load at fracture values in any of the zirconia material types. A possible explanation is that the duration of the autoclave procedure was not sufficiently long to cause degradation of the zirconia materials. According to ISO 13356:2015, zirconia should not present more than 25% of monoclinic phase when submitted to autoclave aging for 134°C and 2 bar for 5 h, which is supposed to be 15–20 yr of clinical aging. A review of the literature shows that several studies on accelerated aging of zirconia have focused on long-term effects, which shows that zirconia weakens during autoclave aging tests (22, 119-121). Material degradation in 3Y-Z occurs due to tetragonal-to-monoclinic phase transformation, and the longer the zirconia is exposed to high temperatures, the higher the proportion of tetragonal phase that transforms into monoclinic phase. Aging studies performed on standardized disks of 3Y-Z confirm that the fraction of t - m phase transformation increases with aging time (55). However, such phase transformation processes do not occur in zirconia materials with higher amounts of yttria (5Y-Z) due to their high content of cubic phase (55, 121). Thus, aging degradation similar to that occurring in 3Y-Z material does not occur in 5Y-Z materials. In theory, due to the material composition, the 5Y-Z materials should not be affected by autoclave aging. However, the results of this study show that time length of a hydrothermal aging in autoclave may have an effect on aging resistance in 5Y-Z materials. Therefore, it is reasonable to assume that an extended duration of the aging procedure would produce larger differences between the zirconia materials. However, 'aging' test periods should not be so long as to be clinically unrealistic, which may be the case with autoclave treatments with long durations. The reported early clinical failures of zirconia restorations most likely have explanations other than aging, such as poor damage tolerance, insufficient design parameters of the restoration or preparation, undesirable load distribution in the oral cavity, or existing flaws in the zirconia material.

6.6 Effect of laboratory test set up

There are both advantages and disadvantages associated with performing laboratory tests. Ideally, it is desirable that a test set up simulate mechanisms that occur during a clinical situation. However, multiple factors act simultaneously during a clinical case. Thus, laboratory tests examining the effect of each factor separately, are recommended. However, care should be taken when choosing the test method and test set up, in order to achieve clinically relevant results. Number of groups to be compared in the test can be influenced by the number of different factors to be examined. Thus, it is desirable to avoid including too many groups, but sufficient number of groups. However, such simplified and standardized test methods will not be able to show the possible effects of several factors that occur during a clinical situation. Thus, it is not always possible to conclude directly to clinical recommendations based on the laboratory tests results.

6.6.1 Load to fracture test

The results of current and previous descriptive studies show that production method, handling, design and material insufficiences have an influence on the fracture of ceramic crowns (96, 99, 100). It is clear that several factors affect simultaneously. Thus, laboratory studies are performed in order to examine the effect of each factor separately. Choice of indenter design and size can affect the clinical relevance of the test results. Wedging effect of the indenter, occlusal contact damage and formation of Hertzian cone crack are examples of clinically irrelevant fracture mechanism due to indenter design and size (124-130). Use of an alternative indenter design, an inverse V-shaped two-plane indenter, resulted in clinically relevant fracture pattern with fracture origin at the cervical area of the crowns (92). Abutment material choice and cementation technique can have an effect on fracture resistance (129-132). Ideal material choice for abutment would However, currently, there is no standarized testing method. The results of both laboratory studies indicates that the material choice of the supporting materials has an effect on load at fracture values. The crowns in study III had higher fracture values than the crowns in study II, although lower fracture values were expected for the 5Y-Z crowns. The observed discrepancy can partially explained by the difference in supporting materials.

6.6.2 Aging test

Chewing simulators are typically used to mimic the clinical masticatory process and to produce relevant long-term cyclic fatigue resistance data from non-clinical specimens. However, the experimental settings in such approaches need to be carefully adjusted in order to create some damage accumulation without causing catastrophic failures (133, 134). Reported loads during normal function vary considerably, and there is no consensus on the loads present *in vivo* or the best way to replicate these *in vitro*. Some authors use lower loads, 100–200 N, while others use loads in the range of 500–800 N (135). The testing conditions should not be arbitrarily chosen, but instead be determined a priori in a pilot study. According to ADM guidance on fatigue principles and testing, aging conditions should not be so severe as to be clinically unrealistic (89). In the present study, after performing a pilot test, preloading in the fatigue test was set at 200 N to avoid using either too low or too high loads. The choice of spherical stainless-steel tip for cyclic loading was based on the desire to avoid contact damage on the crowns during cyclic loading (136, 137).

7. Clinical relevance

In this thesis, a descriptive analysis of retrieval specimens was performed with the aim to gain insight into the underlying cause of failure of zirconia crowns fractured in clinical use. An example of a similar retrieval analysis used can be seen at The Norwegian Arthroplasty Register (138). The aim of the register is to detect poor prostheses, cements and surgical procedures as early as possible and to provide increased expertise in the field and monitoring of the effects of implants and surgical procedures in order to improve the quality of treatment. A similar approach to clinically failed ceramic restorations could contribute to further development and knowledge in the field of dental ceramics.

The analyses of the retrievals in study I showed that care should be taken when designing the zirconia-based single crowns. The material thickness of zirconia at certain areas such as crown margin is of importance with regard to crown failure in clinical use. The findings indicate that thin crown margin designs should be avoided, as it involves reduced fracture resistance due to less material thickness and increased risk of defects. Defects such as chippings at the crown margins can act as a fracture initiation site. The overall load to fracture results of the laboratory studies shows that both bi-layered and monolithic zirconia-based single crowns are viable solutions for clinical use. Care should be taken when planning to use zirconia materials with higher content of yttria in areas of high load. The fractography method can be a useful tool for determining the causes of clinically failed zirconia-based crowns. In addition, the fractographic method can be used as a validation method of laboratory tests that attempt to simulate clinically relevant situations.

8. Future aspects

It is a continuous challenge to develop short-term *in vitro* models that can realistically predict clinical behaviour of dental restorations. Particularly, simulation of ageing processes in zirconia needs to be refined. In addition, an increased use of the fractography method may contribute to enhance the performance and applicability of more clinically relevant laboratory studies. Computerized multifactorial approaches, including finite element analyses, could be explored in depth. However, such models require input of "real-life" data, preferably from long-term randomized controlled clinical trials.

9. Conclusions

The main aim of the present work has been to identify factors affecting fracture of zirconia-based dental crowns in order to improve the clinical survival time.

Fractographic analysis of the clinically failed zirconia crowns showed that crown margin area is an area prone to fracture initiation. Factors such as presence of defects due to production method, post-production adjustments and contamination by veneering material can act as fracture initiators.
Variation in zirconia material thickness affect the load at fracture values more than differences in production method of 3Y-TZP bi-layered crowns. Load at fracture values in hard-machined crowns are more affected more by variation in zirconia material thickness, than in soft-machined crowns.
Short-term aging procedure had no significant effect on load at fracture values of high and super translucent zirconia crowns. High translucent crowns had significantly higher load at fracture than super translucent crowns.

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Effect of margin design on fracture load of zirconia crowns

Anneli Skjold, Christian Schriwer, Marit Øilo
Department of Clinical Dentistry, Faculty of Medicine, University of Bergen, Bergen, Norway

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Zirconia-based restorations are showing an increase as the clinicians' preferred choice at posterior sites because of the strength and esthetic properties of such restorations. However, all-ceramic restorations fracture at higher rates than do metal-based restorations. Margin design is one of several factors that can affect the fracture strength of all-ceramic restorations. The aim of this study was to assess the effect of preparation and crown margin design on fracture resistance. Four groups of bilayer zirconia crowns (with 10 crowns in each group) were produced by hard- or soft-machining technique, with the following four different margin designs: chamfer preparation (control); slice preparation; slice preparation with an additional cervical collar of 0.7 mm thickness; and reduced occlusal thickness (to 0.4 mm) on slice preparation with an additional cervical collar of 0.7 mm thickness. Additionally, 10 hard-machined crowns with slice preparation were veneered and glazed with feldspathic porcelain. In total, 90 crowns were loaded centrally in the occlusal fossa until fracture. The load at fracture was higher than clinically relevant mastication loads for all preparation and margin designs. The crowns on a chamfer preparation fractured at higher loads compared with crowns on a slice preparation. An additional cervical collar increased load at fracture for hard-machined crowns.

Marit Øilo, Department of Clinical Dentistry, Faculty of Medicine, University of Bergen, Aarstadveien 19. N-5009 Bergen, Norway

E-mail: marit.oilo@uib.no

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All-ceramic dental restorations are increasingly preferred over metal-based restorations (1, 2). The early all-ceramic crowns had lower mechanical strength, which limited their use to the anterior region (1, 2). Polycrystalline ceramics, such as zirconia containing only crystalline particles, allow use of all-ceramic restorations even at posterior teeth (3, 4). In order to improve the esthetics of zirconia, the crowns can be produced as bilayer crowns with an esthetic veneering layer over the zirconia core. However, the rate of failure of bilayer crowns is still higher than for metal-based crowns (4–9). The most frequent clinical failures are chipping and delamination of the veneering layer. Core fractures represent a low percentage of the failures observed in zirconia-based bilayers in clinical studies (4). The amount of core fractures may, however, be underestimated because of the lack of a sufficient number of clinical studies with an observation period extending beyond 3 yr. Personal communication with dental technicians and practicing dentists indicate that core failure is more common than the scientific evidence indicates. Core fracture can be related to several factors, such as material composition, production method, design, and thickness of the core.

In vitro studies have been performed in order to achieve a better insight into the behavior of modern high-strength zirconia crowns (10, 11). The fracture modes observed with conventional in vitro fracture strength tests often differ from the fracture modes observed with clinically failed zirconia-based bilayer crowns (11–13). Differences between clinical observations and in vitro tests are evident in both fracture strength values and crack initiation sites, as well as in crack propagation patterns (14–17). The fracture pattern of crowns fractured during clinical use demonstrates fracture origins in the cervical margin of the crown (15, 18–20) or from the intaglio surface of the crown (21–23) (Fig. 1). Margin failures often occur at the proximal area of the crown, where the finish line curves toward the occlusal surface over the gingival papilla (24).

Margin failures may be related to the design and thickness of the crown margins. Multiple studies have been performed in order to evaluate the effect of margin design on load at fracture, but the results are inconclusive. Several studies find that the margin design has an effect on the fracture resistance (24–29), while others see no such effect (30, 31). The large differences in study design among these trials complicate comparison and conclusions on this issue. Clinical recommendations for margin design are based on previous experiences with all-ceramic crowns and also on the design of metal-based crowns. It is not evident

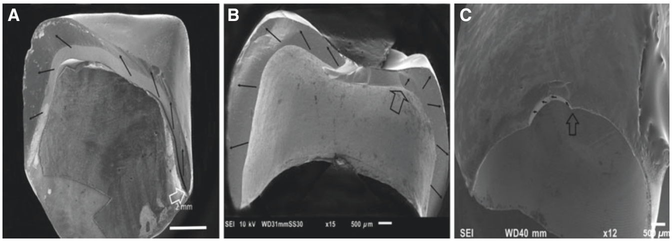

Fig. 1. Three different fracture modes have been observed with zirconia-based crowns fractured in clinical use (ongoing retrieval study). (A) Total fracture in two to three pieces, usually originating in the proximal margin area. (B) Total fracture in three pieces or more, with the fracture originating in the intaglio occlusal wall in a thin region of the crown. (C) Margin chip, usually observed on buccal or lingual flange. Open arrows indicate fracture origin. Black arrows indicate direction of crack propagation.

whether these recommendations are optimal for the modern high-strength zirconia restorations. Most manufacturers of dental ceramics advise dentists to remove up to 0.5–1.5 mm of tooth substance to make room for a bilayer ceramic crown. A preparation depth of 1.5 mm increases the risk of negative effects on tooth vitality (32, 33). Based on mechanical properties, zirconia crowns can probably be made using a minimal invasive slice preparation technique, as suggested by some manufacturers and in scientific papers (34, 35). However, it is still uncertain which design provides optimal balance between crown strength and tooth vitality.

The aim of this study was to analyze the effect of different preparation and crown margin designs on load at fracture for bilayer zirconia crowns.

Material and methods

Crown preparation and margin designs

Two artificial models of premolar teeth were prepared for all-ceramic crowns with an axial wall taper of 10 degrees and rounded edges. Two different finish line designs were prepared. One model had a circumferential chamfer preparation of 0.5 mm depth; the other had a circumferential slice preparation (Fig. 2).

Impressions of each preparation were taken using an A-silicone impression material (Affinis; Coltene, Altsätten, Switzerland), and stone models of the preparation were scanned. The three-dimensional (3D) digital files were used for designing the cores (Fig. 3A–C). The cores were designed and produced by a dental technician according to the protocol. Four different designs were made: uniform-thickness (0.5 mm) crowns on the chamfer preparation [control (C)]; uniform-thickness (0.5 mm) crowns on the slice preparation (S); uniform-thickness (0.5 mm) crowns on the slice preparation with an additional 0.8 mm high cervical collar in the mesiolingual-distal region (S + Col); and finally, reduced occlusal thickness (to 0.4 mm) crowns on the slice preparation with a 0.8 mm high cervical collar (S + Colr) (Fig. 3D).

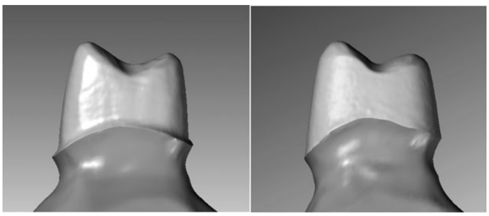

Fig. 2. Two different types of preparations used in the test (lateral view).

Ten crowns of each material were manufactured for each of the four designs according to instructions from the manufacturers. One set of 40 crowns was produced using a soft-machining technique (SM) (Zerion HSC zirconium oxide ceramic single unit; Institut Straumann, Basel, Switzerland) and another set of 40 crowns was produced, with the same specifications, using a hard-machining technique (HM) (Denzir Y-TZP; Denzir Cad.esthetics, Skellefteå, Sweden). In addition, 10 HM crowns made for the slice preparation were veneered and glazed with feldspathic porcelain (S + V) (Fig. 4).

The cusps were designed with equal heights and aligned to prevent the crowns from tilting backwards during loading. The central fossa was designed to be rounded and shallow on the occlusal surface to increase the contact area between the crown and the indenter in order to disperse the load evenly and avoid localized contact damage.

Precementation examinations of margin defects

The crowns were examined at 10× magnification in an optical stereomicroscope (Leica M205 C; Leica Microsystems, Wetzlar, Germany) for signs of marginal defects. All defects or irregularities were registered and graded on a scale of 1–5, according to severity, on a Likert scale, as follows: 1, optimal margins without flaws; 2, minor chips and flaws; 3, multiple chips and flaws; 4, continuous flaws or uneven margins; and 5, large defects visible without a microscope (36). The SM crowns were manually adjusted externally at the margin after delivery from the technician to achieve an acceptable marginal thickness edge equivalent with the margins of the HM crowns. The margin adjustment was performed using a dental handpiece (KaVo K-Control; Kavo, Biberach an den Riss, Germany) according to the manufacturer's recommendations. The crowns were re-examined to ensure that no defects had occurred during the adjustments.

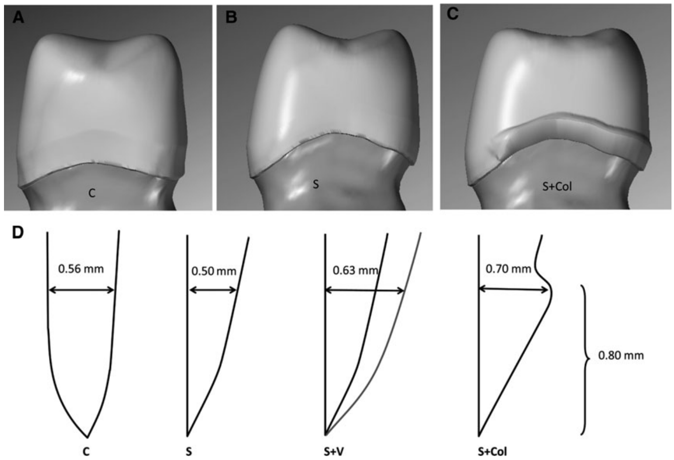

Fig. 3. Three different margin designs (lateral view): chamfer preparation with uniform thickness (control) (A); slice preparation with a partial cervical collar (B); and slice preparation with a uniform thickness (0.5 mm) (C). In addition, one set of 10 crowns identical to those in panel B were made with reduced occlusal thickness (0.4 mm). (D) Measurements of the wall thickness of the four margin designs are shown (in cross section). The thickness is measured at the same height as the upper limit of the cervical collar. The different designs are chamfer (C), slice (S), slice with veneering layer (S + V), and slice with cervical collar (S + Col).

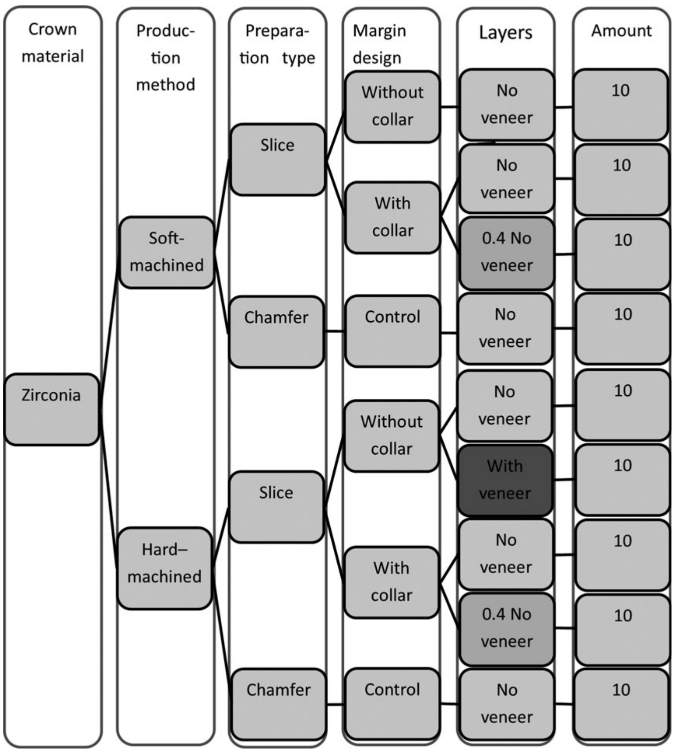

Fig. 4. Flow chart of the crown production. The veneered crowns are indicated with darker shadow.

Load testing

The crowns were cemented to epoxy models (EpoFix; Struers, Ballerup, Denmark) of the preparations with zinc phosphate oxide cement (De Trey Zink; Dentsply DeTrey, Konstanz, Germany). The cement was chosen to minimize the bonding effect of the cement. Excess cement was removed, and after a 5-min setting time the crowns were placed in distilled water at 37°C for 24 (range ± 2) h. The crowns were subsequently loaded centrally at the occlusal fossa with a horizontal cylindrical steel indenter of 13 mm in diameter, cushioned with a 2-mm-thick ethylene propylene diene rubber disc of hardness 90 Shore A (EPDM 90) to avoid contact damage. The cylinder was placed centrally to ensure even distribution of load between the cusps. The load was applied in a servo hydraulic testing system at 0.5 mm min^{-1} until fracture occurred (MTS 852 MiniBionix II; MTS Systems, Eden Prairie, MN, USA). The procedures were performed while the crowns were immersed in water at room temperature. Load at fracture was recorded. At 3,300 N, the procedure was halted because of equipment limitation. The fractured crowns were analyzed, using fractographic methods, to identify the fracture origin and direction of crack propagation (16, 37). The fracture initiation area was compared with the crown margins before fracture, to assess whether pre-existing defects were the fracture initiators or not.

Statistical analysis

As a result of the limited number of specimens in each group and a tendency for skewed data, non-parametric methods were used to analyze the results. The Kruskal–Wallis test was used for overall comparison and the Mann–Whitney *U*-test was used for between-group testing, with correction for multiple groups. Assessment of correlations was performed using Spearman's rank test. Significance was set to $\alpha = 0.05$.

Results

After the loading test, 11 of 90 crowns did not fracture, mainly in the control groups with a chamfer preparation design (Figs 5 and 6, Table 1). Their load value was set to 3,300 N. Overall comparison of all data shows significant differences among the nine test groups ($P = 0.0001$).

In the HM groups, there were significant differences between all groups except between the control group (chamfer) and the group with a cervical collar of standard occlusal thickness. By contrast, in the SM groups, there were no significant differences except between the control group (chamfer) and the two groups with a cervical collar for both types of occlusal thicknesses.

Fractographic analysis revealed that no crowns fractured because of contact damage between the indenter and the crowns, as detected by light microscopy. Crowns fractured either from the crown margin ($n = 22$, Figs 7 and 8) or from the intaglio surface of the crown ($n = 57$, Fig. 9). Most crowns fractured into two or three pieces. Twenty-four of 90 crowns fractured into four or five pieces.

There was no statistically significant correlation between fracture mode and load at fracture overall ($r = 0.26$) or within groups ($r = 0.39$ for HM groups and $r = 0.04$ for SM groups). There was no statistically significant correlation between margin defects and load at fracture ($r = 0.25$).

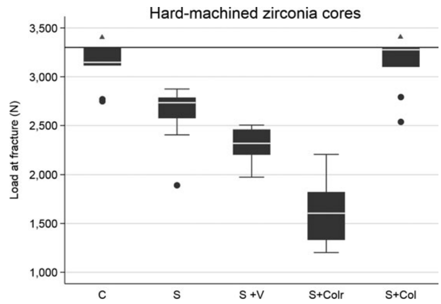

Fig. 5. Box plot of the five hard-machined groups with different margin designs. Further load was stopped at 3,300 N (horizontal reference line at *y*-axis) as a result of equipment limitation. The line within boxes represents the median, and the bottom and top of boxes represent the first and third quartiles, respectively. The maximum and minimum whiskers represent 1.5 interquartile ranges. There were statistically significant differences among all the groups, except for comparison between the chamfer (C) group and the slice with cervical collar (S + Col) group (shown with triangles). S, slice; S + Colr, slice with collar and reduced occlusal thickness.

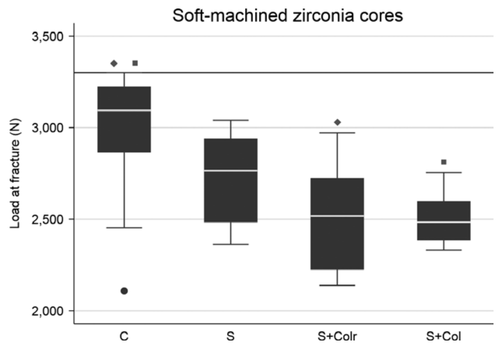

Fig. 6. Box plot of the four soft-machined groups with different margin designs. Further load was stopped at 3,300 N (horizontal reference line at *y*-axis) as a result of equipment limitation. The line within boxes represents the median, and the bottom and top of boxes represent the first and third quartiles, respectively. The maximum and minimum whiskers represent 1.5 interquartile ranges. Statistically significant differences between groups are marked with corresponding squares or diamonds. C, chamfer; S, slice; S + Colr, slice with cervical collar and reduced occlusal thickness; S + Col, slice with cervical collar.

Discussion

The overall results indicate that preparation design, crown design, and manufacturing method affect load at fracture. The different design variables assessed in this

Table 1

Overview of the fracture origin and margin quality evaluation

Production method	Margin designs	Fracture origin			Margin defect mean value
		Cervical	Intaglio occlusal surface	Non-fractured crowns	
Hard-machined	Chamfer (control)	5	–	5	1.6
	Slice with cervical collar	6	3	1	2.8
	Slice with cervical collar and reduced occlusal thickness	–	10	–	3.2
	Slice no collar	1	8	1	1.5
	Slice with collar and veneering	7	2	1	2
Soft-machined	Chamfer (control)	–	7	3	2.5
	Slice with cervical collar	–	10	–	2.6
	Slice with cervical collar and reduced occlusal thickness	1	9	–	2.9
	Slice no collar	2	8	–	3
Total		22	57	11	

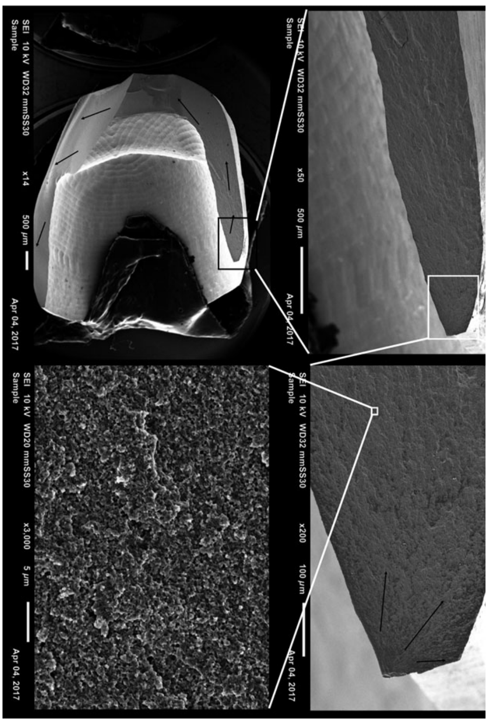

Fig. 7. Fractographic map of a non-veneered soft-machined crown with slice preparation without a cervical collar. Black arrows show direction of crack propagation originating at the cervical edge of the crown.

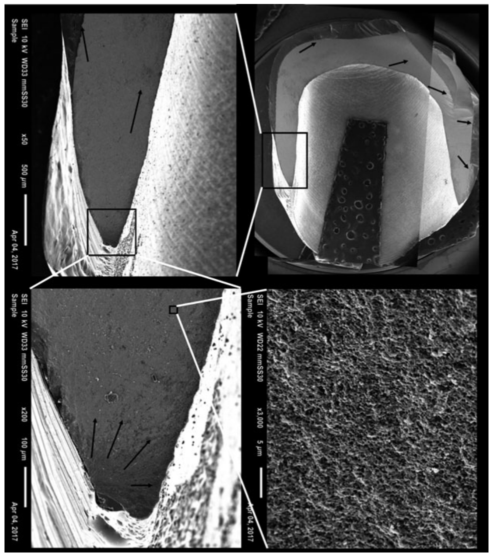

Fig. 8. Fractographic map of a veneered hard-machined crown with slice preparation without a cervical collar. Black arrows show direction of crack propagation originating at the cervical edge of the crown.

study are not independent. An alteration in one of the design variables also causes change in other variables. Furthermore, the effect of each variable differed between the two materials tested. Nonetheless, some effects of the different variables can be isolated.

The finding that the chamfer design crowns fractured at a higher load than did the slice design crowns in both production-method groups indicates that the commonly recommended chamfer margin design gives the strongest crowns. This is in accordance with previous studies (12, 27, 38–44). The increased thickness in the crown margin probably explains this result. Thick crown walls can, however, compromise tooth vitality by requiring increased drilling depth (33, 45, 46). The finding that all crowns fractured at a higher level than maximal mastication forces indicates that slice preparation design might result in sufficiently strong crowns (47). Further studies must be performed to evaluate this in a clinical setting.

The increased margin thickness in the collar resulted in an increase of almost 20% in load at fracture compared

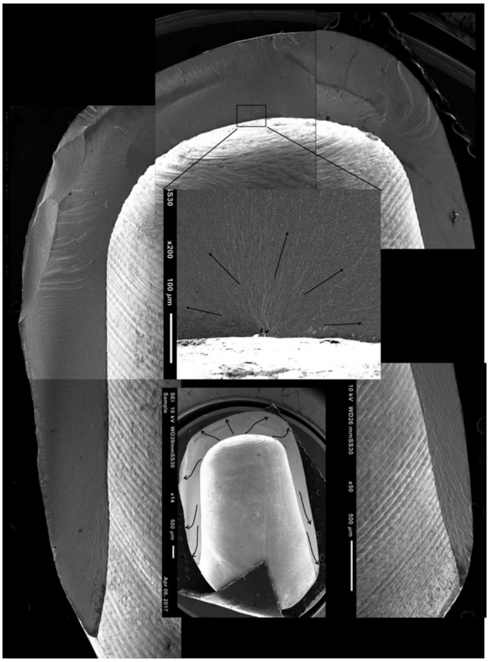

Fig. 9. Fractographic map of a non-veneered hard-machined crown with slice preparation without a cervical collar. Blue arrows show direction of crack propagation originating at the intaglio occlusal surface of the crown.

with uniform thickness slice preparation for the HM group. This is in accordance with previous findings (48–50). The additional collar results in fracture loads equal to the crowns made for the chamfer design. However, another study did not find a strengthening effect of a cervical collar similar to the results for the SM group in the present study (51). Slice preparation may be associated with biological benefits as it requires less removal of sound tooth substance. On the other hand, an over-contoured crown margin may compromise gingival and periodontal health (52). Thus, a highly polished zirconia margin with no veneer may be less plaque retentive than a zirconia core veneered down to the crown margins because the latter will be bulkier. These findings indicate that a slice preparation with a modified crown design may increase both technical and biological success.

Reducing the occlusal thickness resulted in weaker crowns as expected based on previous studies (27, 40–43, 47). This was, however, only statistically significant for the HM group. The results indicate that a change in occlusal thickness may have a greater effect on the load at fracture values than cervical thickness. The veneered crowns fractured at statistically significantly lower loads than the identical non-veneered crowns. This indicates that post-treatment of zirconia cores adversely affects fracture strength (53, 54). Only one HM group was veneered in this study; further studies are needed to assess the effect also on other zirconia cores.

Based on previous studies, a larger difference in load at fracture between HM and SM crowns had been expected (36). Our findings cannot fully explain why this is not the case in the present study. The results indicate that different margin designs have a more pronounced effect on the fracture resistance in HM zirconia cores for bilayer all-ceramic crowns than in SM zirconia cores. This difference may be a result of microscopic-level changes in the zirconia material occurring during the production by the two different methods. A final sintering of the SM zirconia may have a reducing effect on the microscopic defects that may have occurred during milling. Furthermore, the post-sintering adjustment necessary on the margins of the SM crowns to achieve optimal fit and crown emergence profile can affect fracture resistance. The SM restorations are milled with slightly over-contoured margins in the computer-aided design/computer-aided manufacturing (CAD/CAM) unit in order to reduce the risk of margin chips. Manual adjustment of the margins could theoretically have had a reinforcing effect on the core by introducing a *t-m* phase transformation toughening effect at the molecular level. On the other hand, post sintering margin adjustment of the SM crowns can also introduce new defects, although none was observed in this study. The finding that only three crowns had fracture origins in pre-existing defects indicates that margin flaws are not very detrimental to crown strength. It can, however, not be excluded that pre-existing defects not detectable by light microscopy may contribute to crack initiation (18). Further studies of the effect of margin quality are needed to assess this.

The difference in fracture modes among the test groups indicates that both different production methods and designs affect fracture initiation and crack propagation. All fractured crowns in the present study fractured either from the margin or at the intaglio surface, as have been previously reported to be clinically relevant fracture patterns. The rubber disc placed between the indenter and the zirconia cores during loading prevented visible contact damage. It is reasonable to expect, however, that some contact damage has occurred at a microscopic level, although the effect of this is not obvious in the present study. The finding that more than two-thirds of the crowns fractured from the intaglio occlusal surface indicates that occlusal thickness is also important for crown survival. This is in accordance with previous studies in which thinner walls resulted in weaker crowns (41, 44, 55). The finding that the crowns with reduced occlusal thickness had lower fracture loads further supports this point. The loading test configuration may explain the large proportion of fractures originating in the occlusal intaglio surface. The cylindrical indenter used to load the bicuspid zirconia cores may have created a wedging effect in addition to the axial load. Furthermore, the cement layer immediately beneath the loading area may have been crushed during loading, resulting in a local failure of the support of the zirconia core and leading to tensile stress and subsequent cracks at the intaglio surface. The cement used in this study was chosen in order to study zirconia cores without adhesive effects from the cement. This could produce somewhat different results from clinical situations and with other cements. The failure mode is, however, similar to clinical failures observed in an ongoing retrieval study, as seen in Fig. 1.

Previous studies reveal that alterations in margins design affect the cervical stress distribution (38, 49). The lowest load at fracture was 1,202 N, which is higher than the maximally measured biting force of 800–1,000 N (56). Considering the fact that the crowns in this study were not subjected to aging prior to testing and that they were produced and cemented under optimal conditions, the results can, nevertheless, be considered clinically relevant and indicate that zirconia-based crowns can fracture during normal mastication forces if the design is poor or if there is a defect in a tensile stress location.

Within the limitations of this study, it can be concluded that zirconia crowns made for a chamfer preparation fracture at significantly higher loads than similar crowns made for a slice preparation design. Both preparation designs resulted in crowns that fractured at loads above normal mastication forces. A slice preparation with a modified cervical collar crown design may increase both technical and biological success. The veneering process decreases the fracture resistance significantly, just as reduced wall thickness reduces load at fracture.

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Effect of artificial aging on high translucent dental zirconia: simulation of early failure

Anneli Skjold, Christian Schriwer, Nils R. Gjerdet, Marit Øilo
Department of Clinical Dentistry, Faculty of Medicine, University of Bergen, Bergen, Norway

Skjold A, Schriwer C, Gjerdet NR, Øilo M. Effect of artificial aging on high translucent dental zirconia: simulation of early failure.
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Higher yttria content enhances the translucency and appearance of dental zirconia materials. Alterations in material composition also affect mechanical properties. The aim of this study was to compare the fracture load after artificial short-term aging of monolithic, full-contour zirconia crowns with different amounts of yttria-stabilization. Sixty crowns (thirty super high translucent crowns (5Y-Z) and thirty high translucent crowns (3Y-Z)) were produced to fit a model of a premolar with a shallow chamfer preparation. The crowns were cemented with self-adhesive resin cement on composite abutments. For each zirconia type, three groups of crowns ($n = 10$) were allocated to: (i) cyclic loading (200 N, 1 Hz, 30,000 cycles), (ii) hydrothermal aging (3 × 20 min, 134°C 3.2 bar), or (iii) no treatment (control). Surviving crowns from the aging process were quasistatically loaded until fracture. The 3Y-Z crowns had statistically significantly higher fracture values (3,449 N) than the 5Y-Z crowns (1,938 N). The aging procedures did not affect load at fracture. Fractographic analysis showed that fractures started either at the crown margin or at the occlusal intaglio area. Higher yttria content leads to a reduction in material strength and damage tolerance, and this should be reflected in recommendations for clinical use.

Anneli Skjold, Department of Clinical Dentistry, Faculty of Medicine and Dentistry, University of Bergen, Aarstadveien 19, N-5009 Bergen, Norway

E-mail: anneli.skjold@uib.no

Key words: crown dental; dental ceramics; fatigue; monolithic; prosthodontics

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Two decades ago, yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) was introduced in dentistry as a tooth colored material for fixed dental prostheses. Since then, Y-TZP has been widely used due to its biocompatibility, good dimensional and chemical stability, and high fracture toughness (1-6). Crystals of zirconia can appear in three types of lattice structures or phases: monoclinic (m), tetragonal (t), and cubic (c). The monoclinic phase dominates at room temperature, but increased temperature and pressure leads to its transformation into tetragonal and, finally, cubic crystals. In order to arrest a reversion of the process upon cooling, yttria is added as a stabilizing oxide. Addition of 3–4 mol% yttria results in predominantly t-phase crystals, usually called 3Y-TZP, and has the highest fracture toughness and flexural strength of all dental ceramics (7). However, due to its opacity, the early versions of 3Y-TZP required an outer veneering layer in order to achieve a clinically acceptable appearance. The veneering layer has inferior mechanical strength and is prone to fracture (8-10), delamination, and chipping (4,11-17) and thus shortens the clinical lifetime of the restorations and increases the maintenance burden.

In order to eliminate the need for the veneering layer, zirconia with higher translucency and more natural tooth colors were developed (12,15). This provided the opportunity to produce monolithic zirconia restorations. One of the methods to improve the translucency is by increasing the amount of yttria stabilizer. An increased amount of yttria leads to a larger proportion of crystals remaining in cubic phase after cooling, often called partially or fully stabilized zirconia (PSZ or FSZ). Different and overlapping terminologies are used for zirconia materials with different amounts of yttria. In this paper, the terms 3Y-Z and 5Y-Z are used for 3 and 5 mol% yttria-stabilized zirconia, respectively. Increased amounts of cubic phase lead to higher translucency, but reduced fracture toughening properties (9,18,19). Dentists and dental technicians have reported early clinical failures of high translucent zirconia restorations. On the other hand, zirconia material with a higher cubic phase content seems to be less exposed to low temperature degradation (LTD) due to the lack of observed tetragonal-to-monoclinic phase transformation (20).

Low temperature degradation can be provoked by accelerated aging tests in autoclaves, where the pressure of water vapor, temperature, and elapsed time are the experimental variables (21). Aging protocols involving 134°C up to five hours are widely used to assess long-term survival. In vitro correlations of the required time for the aging acceleration test and its correspondence with the longevity of yttria-stabilized ceramic in clinical conditions are challenging. In light of the reported early clinical failures, it is of great interest to assess the short-term effects as well.

The aim of this study was to evaluate and compare the fracture strength and damage tolerance of monolithic crowns manufactured from two different dental zirconias (3Y-Z and 5Y-Z), after short-term aging through chewing simulation and hydrothermal exposure. The null hypothesis to be tested was that the fracture strength and damage tolerance of zirconia would not be affected by the content of yttria or by artificial aging.

Material and methods

Pilot tests

In order to determine abutment material and aging procedure, three pilot tests were performed. Firstly, 15 monolithic 5Y-Z crowns were subjected to 100,000 loading cycles of 60–200 N at 1 Hz in water (37°C). The load was applied occlusally in the middle of the occlusal surface by pneumatically operated pistons (Festo, Esslingen, Germany). The crowns were mounted on metal abutments with silicone material (Speedex Medium; Coltène/Whaledent, Altstätten, Switzerland) as substitute for cement. Ten of the crowns fractured before reaching 20,000 cycles. The remaining five crowns did not fracture. The second pilot test investigated choice of appropriate composite material for individually made abutments. Four monolithic crowns, two from each zirconia type (3Y-Z and 5Y-Z) were cemented on abutments from three different composite materials: (i) SDR flow+ (Denstply Sirona, Charlotte, NC, USA); (ii) Filtek Supreme XTE (3M ESPE, St. Paul, MN, USA); or (iii) Tetric Evo Ceram (Ivoclar Vivadent, Schaan, Liechtenstein). The abutments were prepared by increments of two (SDR) or four (Filtek S and Tetric Evo C) layers, with light curing protocols at room temperature of 23°C and 40% humidity according to the manufacturer's recommendations. Each individual crown was used as mold for the associated abutment. The crowns were subsequently loaded axially until fracture. All crowns had fracture values between 1,801 and 4,883 N, which is considered clinically relevant since these fracture values were higher than the recorded maximum bite force values given in the literature (22). After the crowns were fractured during axial loading, all the composite abutments were assessed for vertical deformations. The deformation was assessed by measuring changes in the composite material height visually in an optical stereomicroscope (Leica M205 C; Leica Microsystems, Wetzlar, Germany) at 10x magnification. The least deformations were observed in abutments made of SDR, so this material was chosen for the rest of the study. In the third test, four monolithic crowns, two from each zirconia type (3Y-Z and 5Y-Z), were cemented on individually made composite abutments (SDR flow+) with self-adhesive cement (RelyX Unicem Aplicap Capsule; 3 M ESPE) according to the manufacturer's recommendation. Then, the crowns were subjected to 30,000 loading cycles of 60–200 N at 1 Hz in water (37°C) and subsequently loaded axially until fracture. All the crowns fractured at values similar to clinical failures (1,823–4,716 N).

Crown design and preparation

An artificial mandibular premolar (Kavo EWL Model teeth; KaVo Dental, Biberach an der Riss, Germany) was prepared with shallow chamfer preparation design for a monolithic crown with minimum crown thickness of 0.5 mm, as recommended by the manufacturer (Fig. 1). An impression of the preparation was taken with an A-silicone impression material (Affinis; Coltène/Whaledent) and the stone model of the preparation was scanned using an intraoral scanner (TRIOS intraoral scanner; 3Shape, Copenhagen, Denmark). The three-dimensional (3D) digital file was used for designing the monolithic crowns according to the protocol and instructions from the manufacturer (Dental Direkt, Spenge, Germany).

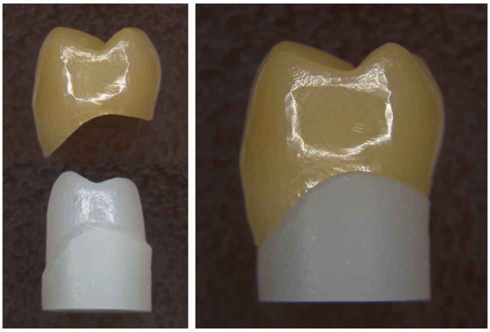

Fig. 1. Monolithic zirconia crown based on a preparation with shallow chamfer. [Colour figure can be viewed at wileyonlinelibrary.com]

A total of 60 monolithic zirconia crowns were made from two different zirconia materials: super high translucent zirconia (5Y-Z, $n = 30$) and high translucent zirconia (3Y-Z, $n = 30$) (Table 1). The sample size of ten crowns in each group was based on the results from the pilot studies and in consideration of the non-normality of the distribution of fracture values from similar in vitro studies on zirconia crowns.

The cusps were designed with equal heights and aligned to prevent the crowns from tilting during loading. The central fossa was designed rounded and shallow on the occlusal surface to increase the contact area between the crown and the indenter in order to disperse the load evenly and avoid localized contact damage.

Pre-cementation examinations

All crowns were examined for signs of marginal defects using a 10 × magnification in the optical stereomicroscope. All defects or irregularities were registered and graded according to severity on a Likert scale. The scale of 1–5 is defined as follows: 1- optimal margins without flaws; 2- minor chips and flaws; 3- multiple chips and flaws; 4- continuous flaws or uneven margins; and 5- large defects visible without a microscope (23). None of the crowns were discarded after the margin quality assessment.

Crown cementation

The individual abutments were made from a polymer composite restorative material (SDR flow+) prepared by increments of two layers according to the manufacturer's recommendations at a room temperature of 23°C and 40% humidity, and using light curing (20 s) of each layer with an additional 20 s after removal from the mold. Each individual crown was used as mold for the associated abutment. The abutments were stored individually in

Table 1

Overview over zirconia materials tested in the study

Abbreviation	3Y-Z		5Y-Z	
Product name and manufacturer	High Translucent DD Bio ZX^2 Dental Direkt, Spenge, Germany		Super High Translucent DD cube X^2 Dental Direkt, Spenge, Germany	
Chemical composition	$ZrO_2 + HfO_2 + Y_2O_3$	≥99.0	$ZrO_2 + HfO_2 + Y_2O_3$	≥99.0
	Y_2O_3	<6	Y_2O_3	<10
	Al_2O_3	≤0.15	Al_2O_3	≤0.01
	Other oxides	<1	Other oxides	<1
Physical specifications	Density after sintering [g cm^{-3}]	>6.0	Density after sintering [g cm^{-3}]	>6.0
	CTE (25–500°C) [10^{-6} K^{-1}]	~10	CTE (25–500°C) [10^{-6} K^{-1}]	~10
	Fracture toughness (SEVNB) [MPa$\sqrt{m}$]	>10	Fracture toughness (SEVNB) [MPa$\sqrt{m}$]	>4.0
	Fracture toughness (SEPB) [MPa$\sqrt{m}$]	4.0	Fracture toughness (SEPB) [MPa$\sqrt{m}$]	2.4
	Flexural strength [MPa] (4-point test)	1100	Flexural strength [MPa] (4-point test)	750
	E modulus [GPa]	>200	E modulus [GPa]	>200
Type and indications	Type 2, class 5, ≥4 units, max 2 connected pontics		Type 2, class 4, ≤3 units	
Cubic phase	~0%		~50%	

plastic containers at room temperature for a period of 45 d before cementation.

Prior to cementation, the zirconia crowns were thoroughly cleaned. Visible debris was removed with air blowing, each crown was rinsed with distilled water and air dried, and finally, rinsed with alcohol and air dried. No other treatment of the crowns' intaglio surface was performed. All crowns were attached to the abutments with a self-adhesive resin cement (RelyX Unicem Aplicap Capsule; 3 M ESPE) according to the manufacturer's recommendation with occlusal pressure of 50 N for 10 s. Excess cement was removed with plastic spatula to avoid surface damage after initial light curing for 2 s. Each surface of the crown was then light cured for 20 s. After setting for 5 min, the crowns were placed in distilled water at 37°C for 24 (±2)h.

Test groups

The crowns from each material were divided into three equal groups of ten: cyclic loading, hydrothermal aging, and a control group of untreated specimens (Fig. 2).

Cyclic loading group

Ten crowns from each material were subjected to 30,000 loading cycles of 60–200 N at 1 Hz in water (37°C) using a spherical stainless-steel tip (ø = 3 mm). The load was applied occlusally in the middle of the occlusal surface by pneumatically operated pistons (Festo). A thin electrotape was applied between the indenter and the crowns to limit contact damage. The crowns were examined visually by optical stereomicroscopy at 10x magnification for cracks or total fractures after each period of 10,000 cycles.

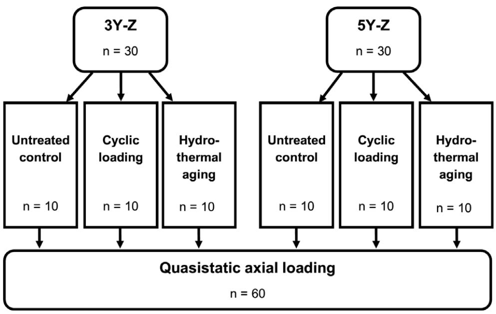

Fig. 2. Flow chart of the test groups.

Hydrothermal aging group

Ten crowns from each material type were exposed to three autoclave cycles of 134°C for 20 min at 3.2 bar (Tuttnauer 3150EL; Tuttnauer, Breda, Netherlands). This specific autoclave aging protocol was chosen for a period of 1 h to simulate aging corresponding approximately 3–4 yr of in vivo exposure at 37°C (24,25). Between each cycle, the crowns were dried at room temperature (21°C) and inspected for flaws at 10× magnification in an optical stereomicroscope. The crowns were cemented on the abutments after the hydrothermal exposure was completed to avoid possible material changes of the polymer abutment teeth models in the autoclave.

Untreated control group

Ten crowns from each material type were cemented without further treatment and thereafter stored in distilled water at 37°C until all the other test groups had completed the aging procedures.

Quasistatic axial loading

All sixty crowns were subsequently subjected to axial loading until fracture (23,26-28). The crowns were loaded centrally at the occlusal fossa with a horizontal cylindrical steel indenter (ø = 13 mm), cushioned with a 2 mm thick ethylene propylene diene rubber disc of hardness 90 Shore A (EPDM 90) to avoid contact damage. The indenter was placed centrally to ensure even distribution of load between the cusps. For the crowns that were subjected to cyclic loading, the placement of the indenter covered the area where cyclic loading had been applied. The load was applied in a servo hydraulic testing system (MTS 852 MiniBionix II; MTS Systems, Eden Prairie, MN, USA) at

0.5 mm min^{-1} until fracture occurred, and the load was recorded. The specimens were immersed in water at 37°C during testing.

Fractographic and grain size analysis

The fractured crowns were analyzed by fractographic methods to identify direction of crack propagation and fracture origin (29). The crowns were examined by optical stereomicroscopy at 10x and 20x magnifications. To illustrate the microstructural features and to determine the fracture origin, selected crowns were further examined by scanning electron microscopy (Phenom XL Desktop SEM, Thermo Fisher Scientific, Waltham, MA, USA). Before sputter coating with gold, the selected crowns were cleaned in ultrasonic bath for 15 min, then rinsed with alcohol and subsequently air dried. Fracture surface features were assessed at magnifications between 100x–5,000x. Identification of the fracture origins and measurements of the zirconia grain sizes were performed at magnifications of 10,000x–50,000x. The Feret's diameter method, which involves measuring the longest diameter of the grains, was used to measure and compare the zirconia grain sizes in the 20,000x SEM micrographs ($n = 4$) (30). The fracture initiation area was compared with the margin quality assessment made prior to the cementation, in order to assess whether pre-existing defects were the fracture initiators.

Hardness and damage tolerance measurements

Vickers hardness (VHN) was measured after the aging procedures and quasistatic loading of the crowns were completed. For both material types, two crowns from each of three test groups were randomly selected. The fractured crown parts were embedded in epoxy resin (EpoFix; Struers, Ballerup, Denmark) and cut to a flat surface with a diamond cutter. All resin blocks underwent a series of grinding (starting at 80 grit, 220 grit, and 1,200 grit MD-Allegro, Struers) and polishing steps with cleansing in ultrasonic bath for 5 min with 2% Deconex (Borer Chemie, Zuchvil, Switzerland) between each grinding and polishing round to obtain the a flat, polished surface suitable to perform the indentation test.

Fifteen measurements were made on each crown using a microhardness tester (Zwick Roell Indentec; ZwickRoell, Ulm, Germany) with load of 150 g applied for 10 s at three different regions (axial wall and occlusal) with five measurements in each region. The average diagonal of the indent (a), as well as length of the cracks radiating from the indents (c), were measured. Damage tolerance was calculated as the c/a ratio (31).

Statistical analysis

The normality of the data was tested by Shapiro-Wilk test. For the load at fracture values, the Kruskal Wallis test was used for overall comparison with Bonferroni adjustment for multiple comparisons. The Mann-Whitney U-test was used to test differences between two groups. Spearman's rank test was used to assess correlations among variables. The level of significance was set to $\alpha = 0.05$. The data were analyzed with a statistical software system (Stata 15; StataCorp, College Station, TX, USA).

Results

Margin defect

Crowns in both material types had a median value of the margin defect of three (Table 2). There were no statistically significant differences in the severity of the crown margin defects between the two different materials ($P > 0.05$). Most of the defects were observed at the mesio-buccal area of the crown margin.

Table 2

Fracture origin, Vickers hardness, c/a ratio, grain size and margin defect values of the different test groups

	3Y-Z			5Y-Z			
Material type	Untreated control	Cyclic loading	Hydrothermal aging	Untreated control	Cyclic loading	Hydrothermal aging	Significance
Fracture origin							
Crown margin	7	5	4	8	9	9	n.s.
Occlusal intaglio	3	5	6	2	1	1	
Vickers hardness							
Mean (SD)		1321 (19.60)			1357 (22.18)		$P < 0.001$
c/a ratio							
Mean (SD)		1.5 (0.12)			2.5 (0.17)		$P < 0.001$
Grain size (nm)							
Mean (SD)		668 (220)			775 (329)		$P < 0.0008$
Min		250			208		
Max		1960			1880		
Margin defect*							
Median		3			3		n.s.
Min		1			2		
Max		5			5		

*1–5 on Likert scale

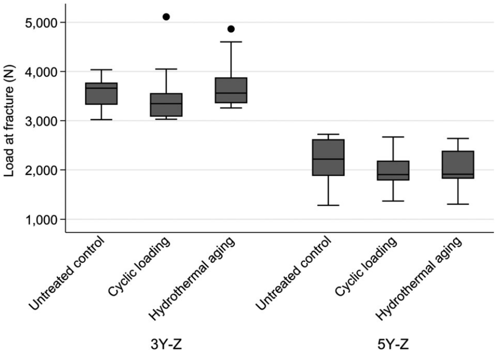

Fig. 3. Boxplot diagram of load at fracture (N) of the test groups. The line within boxes represents the median, the bottom and top of boxes represent the first and third quartiles, respectively. The whiskers represent 1.5 interquartile ranges. Outliers are shown with dots. Statistically significant differences were found between corresponding groups from each zirconia type ($P < 0.005$). There were no statistically significant differences among the groups within each zirconia type.

Effect of artificial aging on load at fracture

None of the crowns subjected to cyclic loading had cracks or total fractures after 30,000 cycles. All the crowns fractured during axial quasistatic loading. All three groups of high translucent (3Y-Z) crowns had significantly higher fracture values than the corresponding groups of super high translucent (5Y-Z) crowns ($P < 0.005$) (Fig. 3). There were no statistically significant differences in fracture load among the three aging subgroups within each material type.

Fractographic and grain size analysis

Two main fracture modes were observed (Table 2). The fractures started either at the crown margin (Fig. 4) or at the occlusal intaglio surface (Fig. 5). No occlusal contact damage leading to crown fracture was observed. Most of the 5Y-Z crowns had the fracture origin at the crown margin, whereas there was an even distribution of the two types of fracture modes in the 3Y-Z crowns. There was no correlation between fracture origin and pre-registered crown margin defects. For the 3Y-Z crowns, there was no correlation between fracture mode and load at fracture. However, for the 5Y-Z crowns, there was a moderate positive correlation between fracture mode and load at fracture (Spearman′s rho: 0.49, $P = 0.0059$). There was a tendency for fracture origins to be at the crown margin in the cases of higher fracture loads. The 5Y-Z crowns showed a predominantly trans-granular fracture mode, whereas inter-granular fracture modes were more prevalent in 3Y-Z crowns (Figs. 4 and 5). The grains of the 3Y-Z were more rounded and homogeneous in size than the grains of 5Y-Z. There was a statistically significant difference in grain size between the two material types ($P < 0.0008$) (Table 2).

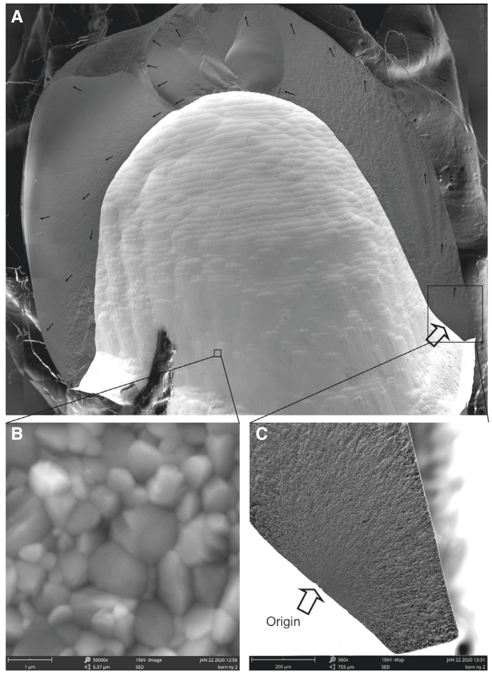

Fig. 4. Scanning electron image of fracture surface of 5Y-Z crown with fracture start at the crown margin. Crack propagation is shown with black arrows in A. The fracture origin is shown with open arrows in A and C. Grain size and composition is shown in B (50,000x).

Hardness and damage tolerance

There was a statistically significant difference ($P < 0.001$) in VHN values between the two different zirconia materials (Table 2). Also, a statistically significant difference was found in the crack length ratio (c/a) between the different zirconia materials ($P < 0.001$). However, no statistically significant difference was found in the crack length ratio between the test groups.

Discussion

This study investigated the effect of accelerated aging on two different zirconia dental materials. The effects of hydrothermal aging and cyclic loading on monolithic crowns were assessed separately. The overall results show that the high translucent 3Y-Z had significantly higher load at fracture values than the equivalent groups of super high translucent 5Y-Z,

which is in accordance with previous studies (32-35). The material composition and grain size that enhance the translucency of the material are the most likely reasons for the reduced strength of the 5Y-Z (36,37). The increase in translucency of the tested 5Y-Z was achieved by increasing the amount of yttria to 5 mol%

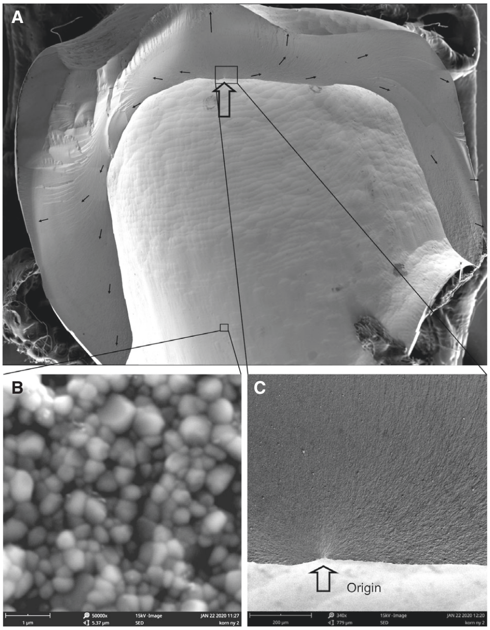

Fig. 5. Scanning electron image of fracture surface of 3Y-Z crown with fracture start at the occlusal intaglio area. Crack propagation is shown with black arrows in A. The fracture origin is shown with open arrows in A and C. Grain size and composition is shown in B (50,000x).

(<10 wt%) and reducing the amount of alumina to <0.01 wt%, and thus achieving a higher proportion of cubic phase crystals. A higher proportion of cubic phase in 5Y-Z (approx. 50%) reduces the fracture toughening property due to reduced tetragonal-to-monoclinic transformation (21,38), which is also evident by the two-thirds reduction in damage tolerance (c/a ratio) in the present study (31).

The current hydrothermal aging procedure aimed to simulate accelerated aging corresponding to approximately 3 yr of clinical use (24,25). The hydrothermal aging procedure had, however, no significant effect on load at fracture values in any of the zirconia material types. A possible explanation is that the duration of the autoclave procedure was not sufficiently long to cause degradation of the zirconia materials. According to ISO Standard 13356-2015, zirconia should not present more than 25% of monoclinic phase when submitted to autoclave aging for 134°C and 2 bar for 5 h, which is equivalent to 15–20 yr of clinical aging (38). A review of the literature shows that several studies on accelerated aging of zirconia have focused on long-term effects, which shows that zirconia weakens during autoclave aging tests (39-43). These studies show that hydrothermal aging in autoclave at 134°C and 2 bar for varying durations (hours) results in material degradation and reduced strength of the yttria-stabilized tetragonal zirconia polycrystal (3Y-Z). Material degradation in 3Y-Z occurs due to tetragonal-to-monoclinic phase transformation, and the longer the zirconia is exposed to high temperatures, the higher the proportion of tetragonal phase that transforms into monoclinic phase (33). Aging studies performed on standardized disks of 3Y-Z confirm that the fraction of tetragonal-to-monoclinic phase transformation increases with aging time (44). However, such phase transformation processes do not occur in zirconia materials with higher amounts of yttria (5Y-Z) due to their high content of cubic phase. Thus, aging degradation similar to that occurring in 3Y-Z material does not occur in 5Y-Z materials (34,45,46). Studies assessing the effect of autoclave aging on monolithic 5Y-Z and 3Y-Z have shown that 5Y-Z has the lowest biaxial flexural strength values, but no low temperature degradation was found after autoclave aging (34,47). Another study examining the effect of autoclave aging on two types of 5Y-Z materials (DD cubeX^2 and Prettau Anterior), revealed a difference in flexural strength between the two zirconia materials (48). DD cubeX^2 had a statistically significantly higher mean flexural strength than Prettau Anterior, both before and after artificial aging for 10 h. However, DD cubeX^2 showed a statistically significant reduction in flexural strength after artificial aging. These results differ from the results of our study, which may be explained by the difference in time length of the aging procedure. On the other hand, a study that examined a shorter time length of autoclave treatment (3 h) showed that the fracture strength of monolithic zirconia crowns decreased significantly (42).

In theory, due to the material composition, the 5Y-Z materials should not be affected by autoclave aging. However, the results of this study show that time length of a hydrothermal aging in autoclave may have an effect on aging resistance in 5Y-Z materials. Therefore, it is reasonable to assume that an extended duration of the aging procedure would produce larger differences between the zirconia materials (43). However, ‘aging’ test periods should not be so long as to be clinically unrealistic, which may be the case with autoclave treatments with long durations (38). The reported early clinical failures of zirconia restorations most likely have explanations other than aging, such as poor damage tolerance, insufficient design parameters of the restoration or preparation, undesirable load distribution in the oral cavity, or existing flaws in the zirconia material (26,49).

Reported loads during normal function vary considerably, and there is no consensus on the loads present in vivo or the best way to replicate these in vitro. Some authors use lower loads, 100–200 N, while others use loads in the range of 500–800 N (50). Chewing simulators are typically used to mimic the clinical masticatory process and to produce relevant long-term cyclic fatigue resistance data from non-clinical specimens. However, the experimental settings in such approaches need to be carefully adjusted in order to create some damage accumulation without causing catastrophic failures (51,52). The testing conditions should not be arbitrarily chosen, but instead be determined a priori in a pilot study. In the present study, after performing a pilot test, preloading in the fatigue test was set at 200 N to avoid using either too low or too high loads. The choice of spherical stainless-steel tip for cyclic loading was based on the purpose of avoiding contact damage on the crowns during cyclic loading (53,54). The smaller size of the sphere was chosen for easier remounting of the crowns after each cyclic loading round. The finding that all the crowns survived the cyclic loading was surprising given that in our second pilot study several crowns fractured before 30,000 cycles were reached (26). However, the test set up in the pilot differed slightly from that used in the main study. The crowns in the pilot study were mounted on metal abutments with silicone material as substitute for cement, while the crowns in the cyclic loading test were cemented with self-adhesive resin cement on composite abutments. These changes in the test protocol were made in order to more closely simulate the clinical situation. However, the difference in results between the pilot test and the main cyclic loading test indicates that choice of abutment and cement material can have a significant effect on the mechanical aging tests. In this study, the zirconia crowns were subjected to 30,000 cycles of cyclic loads in an attempt to simulate early clinical failure, which differs from most studies on mechanical cyclic loading, where the number of cycles may reach as many as 2,000,000 cycles (42,50,52,55,56). However, according to ADM guidance on fatigue principles and testing, aging conditions should not be so severe as to be clinically unrealistic (38). In a study examining the effect of artificial aging on 3Y-Z (DD Bio ZX^2) and 5Y-Z (DD cubeX^2) through chewing simulation with simultaneous thermocycling, it was found that 5Y-Z crowns had significantly lower fracture resistance than 3Y-Z (55). However, in this study, the artificial aging consisted of 1,200,000 cycles. A previous study showed that the flexural strength of 3Y-TZP ceramic decreased only after the specimens were subjected to both mechanical and thermomechanical cycling (32). Furthermore, a study that compared the effects of mechanical and hydrothermal aging on microstructure and biaxial flexural strength of monolithic 5Y-Z and 3Y-Z showed that 5Y-Z had the lowest biaxial flexural strength values and it was affected when mechanical cycling was involved (34). Monoclinic transformation was observed in 3Y-Z when exposed to hydrothermal aging alone or in combination with mechanical cycling. No monoclinic transformation was found in any of the treatments for 5Y-Z (34).

Anatomically shaped crowns were chosen to ensure an in vitro setup mimicking the clinical situation as closely as possible (28,57). In addition, a polymer-based abutment material with tooth-like properties was chosen in order to use materials with a comparable modulus of elasticity with dentin (58). In a study that investigated aging procedures, the zirconia crowns were cemented on metal abutments, resulting in a relatively high fracture resistance (55). However, no fractographic analysis of the fracture mode was performed, and, therefore it is impossible to confirm if the crowns fractured in a similar way as clinically fractured crowns. Self-adhesive cementation with RelyX Unicem Aplicap Capsule was chosen due to several desirable properties (easy delivery, optimal consistency, one-step self-adhesive, easy removal of excess, and moisture-tolerance) and also due to improved fracture resistance (59). The dual curing properties of the cement leads to sufficient curing, taking into account a possible incomplete light curing. According to a meta-analysis on bonding effectiveness to zirconia ceramics, the cement choice appeared less critical, as long as a composite cement was used for adhesive luting of zirconia ceramics (60).

The fractographic analyses show that all the crowns had fracture modes similar to clinically fractured crowns (28). The fractures started either at the crown margin or at the occlusal intaglio surface. The fracture modes were similar to clinically fractured crowns, indicating that the load at fracture values are clinically relevant. The fracture modes of the crowns indicate that difference in material composition may have an effect on the crack propagation. Almost all of the 5Y-Z crowns had fracture origins at the crown margin, while the 3Y-Z crowns had an even distribution of the two fracture modes. The observed fracture origins at the crown margin did not correlate with the margin defects identified prior to aging and testing procedures. This may indicate that the occurrence of margin defects is not one of the main causes of the clinical failure of zirconia crowns. However, a previous study on the effects of margin curvature on load at fracture of ceramic crowns showed that stress concentration along the crown margin varies (26). Thus, deleterious crown fracture may start at a margin defect if the defect is localized at a high stress area of the crown margin. For a premolar crown, the stress concentration tends to be localized at the mesial and distal approximal curvature. The margin defects observed in the present study were in general small (grade 3 on the Likert scale), few, and localized mainly at the mesio-buccal area. There was, however, no obvious shortcomings in the original prepared abutment tooth or in the design of the crown that could explain the predominant localization of the flaws in this region.

The finding that 5Y-Z crowns had predominantly trans-granular fracture mode whereas inter-granular fracture modes were more prevalent in 3Y-Z crowns, indicates that the difference in material composition has an effect on sintering quality. The two-thirds reduction in damage tolerance supports this. The exact underlying mechanism of the difference in fracture mode needs to be further investigated.

In conclusion, a higher concentration of yttria is associated with a decrease in the strength and damage tolerance of the zirconia material. The 3Y-Z crowns had significantly higher load at fracture values both before and after the aging procedures. The short-term hydrothermal aging and cyclic loading procedures did not affect the load at fracture for any of the crowns. A prolonged aging test might affect the load at fracture values of the zirconia crowns differently. Fracture values of 5Y-Z crowns indicate that care should be taken when applying the 5Y-Z material in areas of high stress.

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Conflicts of interest – The authors declare no conflict of interests.

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