On dental ceramics and their fracture
A laboratory and numerical study

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Abstract

Background Surface treatments and irregularities in the surfaces may affect the fracture of ceramics. The effects of various treatments on the surface texture of different types of ceramic cores/substructures was therefore qualitatively, quantitatively and numerically evaluated. Since fractures in ceramics are not fully understood, the fracture behavior in dental ceramic core/substructures was also studied using both established laboratory methods and newly developed numerical methods.

Methods The surfaces of dental ceramic cores/substructures were studied qualitatively by means of a fluorescence penetrant method and scanning electron microscopy, quantitatively evaluated using a profilometer and also numerical simulation. In order to study fracture in zirconia-based fixed partial denture (FPD) frameworks, fractographic analysis in combination with fracture tests and newly developed two-dimensional (2D) and three-dimensional (3D) numerical modeling methods were used. In the numerical modeling methods, the heterogeneity within the materials was described by means of the Weibull distribution law. The Mohr–Coulomb failure criterion with tensile strength cut-off was used to judge whether the material was in an elastic or failed state.

Results Manual grinding/polishing could smooth the surfaces on some of the types of dental ceramic cores/substructures studied. Using the fluorescence penetrant method, no cracks/flaws apart from milling grooves could be seen on the surfaces of machined zirconia-based frameworks. Numerical simulations demonstrated that surface grooves affect the fracture of the ceramic bars and the deeper the groove, the sooner the bar fractured. In the laboratory tests the fracture mechanism in the FPD frameworks was identified as tensile failure and irregularities on the ceramic surfaces could act as fracture initiation sites. The numerical modeling codes allowed a better understanding of the fracture mechanism than the laboratory tests; the stress distribution and the fracture process could be reproduced using the mathematical methods of mechanics. Furthermore, a strong correlation was found between the numerical and the laboratory results.

Conclusion Based on the findings in the current thesis, smooth surfaces in areas of concentrated tensile stress would be preferable regarding the survival of ceramic restorations, however, the surfaces of only some of the ceramic cores/substructures could be significantly affected by manual polishing. The newly developed 3D method clearly showed the stress distribution and the fracture process in ceramic FPD frameworks, step by
step, and seems to be an appropriate tool for use in the prediction of the fracture process in ceramic FPD frameworks.

Keywords: Dental ceramics, Finite element analysis, Fixed partial denture, Fracture, Numerical modeling, Surface treatment.
Abbreviations and definitions

2D: Two-dimensional

3D: Three-dimensional

CAD: Computer aided design

CAM: Computer aided manufacturing

Dep: Direction of crack propagation

FPD: Fixed partial denture

FEA: Finite element analysis

HIPing: Hot isostatic pressing is a method used to densify a material, whereby heat and pressure are imposed simultaneously and the pressure is applied from all directions via a pressurized gas such as argon or helium. (Adapted from the glossary bank in Richerson, 2006)

PFM: Porcelain fused to metal

Ra: describes the average roughness value for a surface that has been traced by a profilometer.

SEM: Scanning electron microscope

Yttria: Y$_2$O$_3$.

Y-TZP: Yttria tetragonal zirconia polycrystals is a Y$_2$O$_3$-ZrO$_2$ essentially in tetragonal phase.
List of papers

The original papers that comprise this doctoral thesis are listed below and will be referred to in the text by their Roman numerals.


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Introduction

Teeth are an integral part of the facial structure and in most cultures they symbolize youth, health, beauty, and dignity. Loss of teeth can, therefore, create physical and functional problems and missing teeth can cause psychological and social disturbances. Thus, the desire to replace missing teeth is ancient and man has long sought durable and esthetic dental restoration materials, resulting in the use of many different materials for making artificial teeth. For example, during the 18th century human teeth, animal teeth, and ivory were commonly used. Human teeth, however, were scarce, animal teeth were unstable in human saliva and ivory contained pores that easily became stained. Out of curiosity it can be mentioned that in an attempt to make his denture constructed of hippopotamus ivory taste less foul president George Washington used to soak it in port (Guerini, 1909). In 1774, the French pharmacist Alexis Duchateau introduced ceramic as a restoration material in dentistry. He was dissatisfied with his ivory dentures which were badly stained, whereas the glazed ceramic utensils he used every day as a pharmacist resisted wear and staining. From that the idea of using porcelain as a dental restoration material was born and, the first set of ceramic dentures was manufactured in cooperation with porcelain manufacturers at the Guerhard factory in Saint Germain-en Laye, France (Jones, 1985).

Brief review of ceramics in dentistry

The first ceramic item was probably born when people mixed clay with water and accidentally fired it and then discovered that the material became hard. Ceramics cover inorganic and non-metallic materials, which are formed by the action of heat and are considered to be biocompatible (Depprich et al., 2008), have a good esthetic appearance, high resistance to wear and distortion (Ghazal et al., 2008), low plaque accumulation (Hahn et al., 1993), low thermal conductivity and low solubility (Chai et al., 2007). That is, properties that have made ceramics among the most inert material known and especially interesting as a biomaterial.

The development of improved ceramic systems for dentistry has been carried on for centuries. For example, at the end of the 19th century the so-called porcelain Jacket crown, mainly based on feldspar ceramics, was invented (Moffa, 1988). The introduction of the porcelain Jacket crown coincides with the introduction of electric ovens, which facilitated the firing process of porcelain restorations (Jones, 1985). Although these crowns were highly esthetic, the density change and shrinkage during the firing process and the low strength of the restorations were problems often related to this type of crown.
To improve the mechanical properties porcelain fused to metal (PFM) systems were developed and introduced in the 1950s and soon became popular. Single crowns and FPDs made using the PFM technique were popular because the system provided, among other things esthetic acceptability, ductility, strength and toughness. Around 1960 it was found that adding leucite (KAlSi$_2$O$_6$) to the porcelain affected the coefficient of thermal expansion, which resulted in better matches between the veneer porcelain and the substructure (Kelly et al., 1996) and to an even wider use of various PFM systems.

Although the PFM systems provided dental restorations that derived advantages from both the porcelain and the alloys, such restorations were still built on a metal alloy base, tended to look opaque and monochromatic and often had a dark grayness at the gingival line. All of which have encouraged the development of all-ceramic core/substructure materials with improved strength and mechanical properties and during the past few decades such new systems have been introduced into the market (Andersson & Odén, 1993; Kappert et al., 1995; Hornberger, 1996; Höland et al., 2000; Sundh & Sjögren, 2006).

Classification of dental ceramics
Generally, dental ceramics could be classified in three main groups: silica-based ceramics, hybrid ceramics and oxide ceramics (Munz & Fett, 1999; Milleding, 2001).

Silica-based ceramics
The main content in silica-based ceramics is SiO$_2$ and the main feature is the glassy-amorphous phase (Munz & Fett, 1999). Examples of silica-based ceramics are IPS Empress, IPS Empress 2 and the recently introduced IPS e.max Press (Ivoclar, Schaan, Liechtenstein). IPS Empress is a heat-pressed leucite-reinforced feldspathic all-ceramic system (Blatz et al., 2003). The desired color and shading of IPS Empress are achieved either by means of staining the surface of the restoration using pigmented color or by the so-called layering technique (Brochu & El-Mowafy, 2002). IPS Empress 2 and IPS e.max Press are based on a core of a heat-pressed ceramic material, which contains lithium disilicate (Li$_2$Si$_2$O$_5$) as the main crystalline phase (Albakry et al., 2003; Madina et al., 2009) and veneered with a sintered glass-ceramic (Blatz et al., 2003). In addition, the IPS Empress CAD (leucite-reinforced) and the IPS e.max CAD (lithium disilicate) are intended for fabricating restorations using the computer aided design/computer aided manufacturing (CAD/CAM) technique (Charlton et al., 2008; Guess et al., 2009; Fasbinder et al., 2010).
**Hybrid ceramics**

Hybrid ceramics contain a high volume fraction of crystalline oxides in a glassy matrix (Milleding, 2001). Examples of hybrid ceramics are the In-Ceram Alumina and the In-Ceram Zirconia systems (Vita Zahnfabrik, Bad Säckingen, Germany). The core of the In-Ceram Alumina system is made of lanthanum glass-infiltrated alumina, whereas the core of the In-Ceram Zirconia system is created from a glass-infiltrated alumina structure, in which 34-wt% partially stabilized zirconia is added and infiltrated with lanthanum glass. The cores can be manufactured either by the so-called slip casting technique or by means of the CAD/CAM technique. The latter employs prefabricated In-Ceram blanks made using the so-called dry-pressing technique (Apholt et al., 2001; Denry & Kelly, 2008). The partially sintered In-Ceram cores are veneered with a feldspar-based porcelain (Apholt et al., 2001).

**Oxide ceramics**

Oxide ceramics are dominated by a crystalline phase with only a small glassy phase (Munz & Fett, 1999). Among the reasons for the interest in this type of ceramics is that they exhibit better mechanical properties than other bioceramics (Christel et al., 1989; Hannink et al., 2000). Densely-sintered oxide ceramics became available for dental applications as a result of the development of the CAD/CAM technique and have aroused particular interest in dentistry during the past few decades (Andersson & Odén, 1993; Sjölin et al., 1999).

One oxide ceramic, which has shown better flexural strength and fracture resistance than other dental ceramics is zirconia (Christel et al., 1989; Hannink et al., 2000). Today zirconia is, therefore, among the most interesting dental ceramics materials for cores/substructures for all-ceramic crowns and FPDs. Pure zirconia is monoclinic at room temperature and transforms to tetragonal phase at about 1000 °C, causing volume changes and creating cracks within the structure (Christel et al., 1989). The volume change associated with this transformation makes it impossible to use pure zirconia in many applications. However, adding stabilizers, such as CaO, MgO or Y₂O₃ to the zirconia structure could result in a more stable solution (Christel et al., 1989; Piconi & Maccio, 1999). To reach fully stabilized zirconia enough stabilizer should be added to the zirconia structure and it became a cubic solid solution (Richerson, 2006). Today, the zirconia-based ceramics used in dentistry is partially stabilized (Denry & Kelly, 2008), meaning that it contains a mixture of zirconia polymorphs (Richerson, 2006). The partially stabilized zirconia commonly used for dental applications is yttria tetragonal zirconia polycrystals (Y-TZP) (Denry & Kelly, 2008; Al-Amleh et al., 2010).
Zirconia-based ceramic restorations for dental applications are mainly processed with hard or soft machining (Filser et al., 2001; Denry & Kelly, 2008; Øilo, 2008; Al-Amleh et al., 2010). Hard machining uses sintered blocks processed by hot isostatic pressing (HIPing) and HIPing has been said to be effective in reducing porosity and increasing the density of ceramic materials and could improve the mechanical properties of the material (Tsukuma & Shimada, 1985; Richerson, 2006). Soft machining mainly uses pre-sintered prefabricated blocks and is often named as ‘green-zirconia’. In this way an enlarged core/substructure for a crown or FPD is machined from the pre-sintered block and subsequently sintered to the desired dimensions before veneering (Filser et al., 2001; Al-Amleh et al., 2010). Examples of CAD/CAM systems using HIPed zirconia blocks are the Denzir ceramic products (Cad.esthetics, Skellefteå, Sweden), DC-Zirkon (DCS Dental AG, Allschwil, Switzerland), and Digizon (AmannGirrbach AG, Pforzheim, Germany). Systems using pre-sintered ‘green-zirconia’ blocks include Lava Zirconia (3M ESPE, St Paul, MN, USA), NobelProcera Zirconia (Nobel Biocare AB Göteborg, Sweden), IPS e.max ZirCAD (Ivoclar Vivadent, Schaan, Liechtenstein) and In-Ceram YZ (Vita Zahnfabrik, Bad Säckingen, Germany)(Sundh & Sjögren, 2006; Denry & Kelly, 2008; Al-Amleh et al., 2010).

Thus, one of the major struggles in dental ceramic research during the past few decades has been to improve the strength of the ceramic materials. Although dental ceramic FPDs have shown promising results in clinical follow-up studies (Sailer et al., 2007; Molin & Karlsson, 2008; Schmitt et al., 2009; Al-Amleh et al., 2010; Roediger et al., 2010), ceramics in general remain brittle and fractures in ceramic material often start from defects within and/or on the surface of the material (Munz & Fett, 1999) resulting in failure that may have very serious consequences.

Moreover, manufacturing techniques and the subsequent handling of dental ceramics may introduce surface defects and irregularities and could have a negative impact on the strength of dental ceramic restorations (de Jager et al., 2000; Lohbauer et al., 2008). It was, therefore, of interest to investigate whether CAM machining introduces surface irregularities and/or defects in dental ceramic FPD frameworks and to evaluate the effects of introduced surface irregularities on fractures of ceramic frameworks. In addition, it has long been well-known (McLean & Hughes, 1965) and verified in a number of studies (de Jager et al., 2000; Fischer et al., 2003; Quinn, 2007; Lohbauer et al., 2008) that surface treatments and surface texture affect the fracture of ceramics. For example it has been shown that the rougher the surface, the lower the flexural strength of dental ceramics (de Jager et al., 2000; Fischer et al., 2003) and it has been stated that highly polished porcelain can be stronger than glazed porcelain (Giordano et al.,
Therefore, it was of importance to investigate the possibility of smoothing out and reducing the roughness on the surfaces of newly introduced ceramic core/substructure materials.

One in vitro technique commonly used to study fracture in dental ceramics is the fractographic analysis method (Scherrer et al., 2006; Quinn, 2007; Scherrer et al., 2008). However, using this in vitro technique, the stress distribution during the fracture and the fracture process in the ceramics cannot be fully understood and cannot be clearly followed. In addition, a survey of the literature revealed no article clearly showing the fracture process of ceramic FPD frameworks, stress distribution, fracture initiation and propagation in ceramic FPD frameworks. Therefore, it was of particular interest in the present thesis to investigate the possibility of using the mathematical methods of mechanics to reproduce the stress distribution and the fracture process in dental ceramics and in this way achieving a better understanding of the fracture mechanism and behavior of ceramic restorations. With a better understanding of the fracture behavior in dental ceramic materials, the material properties could be used more advantageously and ceramic restoration design could be improved, thus prolonging the life of dental ceramic restorations.
Aims

The specific aims of the present thesis were

- to identify the occurrence of cracks/flaws on the surfaces of CAD/CAM machined zirconia-based FPD substructures;
- to investigate whether manual grinding and polishing could reduce surface irregularities and smooth out the surfaces of newly introduced ceramic cores/substructures;
- to examine the fracture process in CAD/CAM machined three-unit zirconia-based FPD frameworks after static loading to fracture in a laboratory environment;
- by means of the mathematical methods of mechanics to analyze the fracture process in ceramic FPD frameworks under static loading to fracture.
Brief description of the techniques used in the present thesis

Techniques used to study the surfaces of the ceramics
Flaws and defects located on the surface of ceramic materials can often act as stress concentrators and are common origins of fracture in ceramic materials (Quinn, 2007). Therefore, it is of importance to identify the occurrence of defects on the ceramic surfaces and to study the effect of these defects on the fracture of ceramic constructions and to evaluate the possibility of smoothing out and reducing irregularities on the surfaces of ceramic core/substructure materials.

Qualitative analysis of the surfaces
Conceivable qualitative analysis techniques that could be used to study the surface of the ceramics are stereomicroscopy, scanning electron microscopy (SEM) and the fluorescence penetrant method. The advantages of stereomicroscopy are that it presents an image that is the correct way up, which makes it easy to correlate to a specimen held in the hand and the surface features can be clearly seen despite the roughness or curvature of the fracture surfaces. With SEM, high-resolution close-ups of a specimen can be captured and compositional information obtained (Quinn, 2007). The fluorescent penetrant method (Fig. 1) is a non-destructive method for detection of defects on a material and is said to be superior to stereomicroscopy and SEM in identifying cracks and defects on the surface of a material (Fischer et al., 2002).

Fig. 1. Image of an In-Ceram Zirconia FPD framework, made using the slip-casting technique, subjected to the fluorescence penetrant method. After the examined specimen was treated with a fluorescence penetrant liquid, it was observed using a stereomicroscope while simultaneously illuminated with UV-light. The light areas indicate defects on the surface of the framework.
Numerical modeling
To study the effect on fracture of irregularities/grooves on the ceramic surfaces a newly developed 3D numerical simulation code was applied. Models of introduced milling grooves of varying depths in a ceramic bar were simulated and the effects of the grooves on the fracture of the ceramic bar were determined.

Quantitative analysis of the surfaces
One way in which to quantitatively analyze the surface condition is to measure the surface roughness by means of a profilometer. In the present work, the parameter $Ra$ was used to describe the mean roughness value of a number of measurements within a sampling length, giving the average deviation of the trace from the determined centre line (European Standard EN 623–4:2004).

Techniques used for fracture analysis
When a brittle material fractures, no or little apparent plastic deformation takes place before fracture, and once a fracture is initiated, it often rapidly propagates leading ultimately to total catastrophic failure (Quinn, 2007). In the present thesis both laboratory and numerical methods were used to analyze fracture in ceramic FPD frameworks.

Fracture test
Zirconia-based frameworks (Fig 2) were cemented with zinc phosphate cement on to stainless steel abutments in a stainless steel socket. The frameworks were then statically loaded to fracture in a universal testing machine (Tinius Olsen H10K-T, Tinius Olsen, Horsham, PA, USA). The load was applied at the central part of the substructures’ pontic by means of a hardened steel ball with a diameter of 6 mm. The loads in Newton required to fracture the substructures were automatically recorded.
Fractographic analysis
Fractographic analysis is the integration of knowledge from a variety of sources to solve the puzzle of how fracture occurred. The integrated knowledge involves, among others things, understanding the fracture mechanics, recognition of crack pattern, identification of the fracture origin and also awareness of the background information such as the process technique, the microstructure and test conditions (Quinn, 2007). It is a well-established method used in the laboratory for identification of fracture origin and direction of crack propagation (dcp) in a fractured specimen. When a ceramic material fractures, different characteristic fracture features can often be observed on the fracture surfaces and can be converted into interpretable information. Since fractographic analysis is a method that has been used for many years, the characteristic fracture features seen have been defined and known terminologies have emerged. The definition of fracture features that were sought for in the present work were adopted from National Institute of Standards and Technology guide textbook (Quinn, 2007) and from previous articles addressing failure analysis of ceramics (Scherrer et al., 2006; Scherrer et al., 2008) and are defined as below.

Arrest line is a sharp line on the fracture surface defining the crack front shape of an arrested or momentarily hesitated crack prior to resumption of crack propagation under a more or less altered stress configuration.
Compression curl is a telltale feature of flexural fracture. A crack starts and grows perpendicularly to the tensile surface of a specimen or component loaded in bending, when the crack reaches the compressive side of the specimen, it leaves a curved lip, just before fracture occurs.

Fracture mirror is a relatively smooth region surrounding and centered on the fracture origin.

Fracture origin is the source from which fracture begins.

Hackle is a line on the surface running in the local direction of cracking, separating parallel, but noncoplanar portions of the crack surface.

Twist hackle is hackle that separates portions of the crack surface, each of which has rotated from the original crack plane in response to a lateral rotation or twist in the axis of principal tension. Usually crack propagation is in the direction of fine to coarse hackle.

Numerical modeling
Mechanical problems in everyday life can often be simplified and described using a mathematical model (Aris, 1978; Bender, 1978). Once the problem has been analyzed, the mechanism can be found and after appropriate idealization, mechanical modeling can be built up. There are two main methods for solving a mathematical model. One is the analytical method and the other is the numerical method. The analytical method is accurate but limited in use. In most cases, it is difficult to find analytical solutions to the problems. The numerical calculation method, however, can solve more problems in an approximate manner and achieve rather satisfactory results (Hildebrand, 1956). The development of computer technology/techniques increased the speed of calculation leading to wider use of the numerical calculation method.

There are various kinds of numerical calculation methods but the finite element analysis (FEA) method is one that is commonly used. The FEA method is a numerical technique for finding approximate solutions of partial differential equations and integral equations (Ottosen & Petersson, 1992). It has been used in technical fields for many years and has also been applied in biomechanical studies in dentistry (Ausiello et al., 2004; Ichim et al., 2007; Bergkvist et al., 2008; Tsumita et al., 2008).

In the numerical modeling codes used in the present thesis, two basic concepts, stress and strain, are frequently used and some basic knowledge about stress and strain is presented below. The definition of stress could be simply defined as a force $F$ divided by its acting area $A$. Problems are usually three dimensional (3D), but in some special cases they can be greatly
simplified to two dimensional (2D), i.e. in plane stress or in plane strain problems (Chandrupatla & Belegundu, 1991). The plane in the 2D case is usually named the $xy$-plane, resulting in the stress-components $\sigma_x$, $\tau_{xy}$, $\sigma_y$.

The plane stress problem assumes that the object is infinitively thin. The plane strain problem is based on the assumption that the object discussed is infinitively thick in the direction of the non-existing $z$ direction. Although these two extreme 2D cases deviate a lot from reality, they are easier to use than 3D and in some cases they can still give guidance in solving the problem.

Stresses in 3D involve the $xy$-, $xz$- and the $yz$ planes, which results in the stress-components $\sigma_x$, $\sigma_y$, $\sigma_z$, $\tau_{xy}$, $\tau_{xz}$, $\tau_{yz}$ at one physical point, in which $\sigma_x$, $\sigma_y$, $\sigma_z$ are the normal stresses and $\tau_{xy}$, $\tau_{xz}$, $\tau_{yz}$ are the shear stresses. Imagine that all these stresses are related within a specific cube; while the cube rotates around the physical point, meaning that the direction of $x$, $y$, $z$-axes also rotate the six stress components will, correspondingly, be changed. It will finally arrive at a state where all of the shear stresses vanish and only the normal stresses are left. In this state, these axes are called the principal axes of stresses and the stress-components relevant to them are called principal stresses and denoted as $\sigma_1$, $\sigma_2$, $\sigma_3$. Usually $\sigma_1$ is the maximum principal stress, $\sigma_2$ intermediate principal stress and $\sigma_3$ minimum principal stress (Jaeger & Cook, 1976).

Strains are used to describe material deformation and engineering strain was used in the present thesis because only small deformations are present in brittle materials until failure. In this case, linear strain $\varepsilon$ is defined as the length change divided by its length. Shear strain is equal to the ratio between the amount by which an object is skewed and its length. The strains in 3D are defined similarly to the stresses. That is, the strain-components $\varepsilon_x$, $\varepsilon_y$, $\varepsilon_z$, $\gamma_{xy}$, $\gamma_{xz}$, $\gamma_{yz}$, at one physical point, in which $\varepsilon_x$, $\varepsilon_y$, $\varepsilon_z$ are called the normal strains, $\gamma_{xy}$, $\gamma_{xz}$, $\gamma_{yz}$ are the shear strains, and $\varepsilon_1$, $\varepsilon_2$, $\varepsilon_3$ are the principal strains. The above-mentioned concepts and symbols are used in the present thesis.

Ceramics are rather homogeneous materials, but often contain flaws. These flaws can be located both on the surface and within the material. These flaws, such as cracks, pores, defects or hard inclusions may influence the stress distribution in the material and induce further fracture initiation and propagation (Quinn, 2007). In numerical modeling, material heterogeneity must be taken into consideration because it greatly influences the failure. Based on earlier studies (Liu, 2004), the Weibull distribution method has been shown to be a satisfactory tool for describing the distribution of the defects in brittle materials such as rocks. Since ceramics is a brittle material, the Weibull distribution law (Weibull, 1951) was utilized in both Papers III and IV in the present thesis to assign the values of the heterogeneity parameters and to give the elements varying mechanical
The Weibull distribution law is described by the following formula:

\[ q(\sigma) = \begin{cases} \frac{m}{\sigma_0} \left( \frac{\sigma}{\sigma_0} \right)^{m-1} \exp\left( -\left( \frac{\sigma}{\sigma_0} \right)^m \right), & \sigma \geq 0 \\ 0, & \sigma < 0 \end{cases} \]

where \( q(\sigma) \) is the probability function of \( \sigma \), \( \sigma \) is the element parameter that can be compressive strength, tensile strength or Young’s modulus of the element, \( \sigma_0 \) is the mean value of the element parameter and \( m \) is the Weibull modulus. The Weibull modulus is a homogeneous index of a material. A larger \( m \) implies a more homogeneous material and vice versa. When \( m \) trends to infinity, the variance of the value \( \sigma \) among the elements approaches zero, meaning that the material simulated becomes absolutely homogeneous. In the present thesis the specific heterogeneity distribution in physical space after generation of finite elements was achieved by the Monte Carlo method (Liu, 2004).
Materials and methods

The materials and methods are briefly described in this section, more detailed descriptions can be found in each original paper.

Paper I

Specimen preparations
Four identical stylized three-unit ceramic substructures intended for dental all-ceramic FPDs were CAM machined from HIPed Y-TZP pre-fabricated blanks. Stainless steel models with a circumferential chamfer (120°) placed in pairs in a holder were used as master models for the FPD frameworks. The distance between the centers of the master models was 17.5 mm, which approximately corresponds to the distance from a second lower molar to a second lower premolar. Using the firing schedule of a feldspar-based veneer porcelain, the specimens were heat-treated in a way similar to that used when veneering.

Screening for surface flaws using the fluorescent penetrant method
Before the zirconia substructures were mechanically loaded, the surfaces of the ceramic specimen were screened out using a fluorescent penetrant liquid and investigated in a stereomicroscope while simultaneously illuminated with UV-light. A more detailed description can be found in Paper I.

Identification of fracture features
After screening using the fluorescent penetrant method, the zirconia substructures were loaded to fracture in a universal testing machine. After fracture of the specimens, the fracture surfaces were analyzed in a stereomicroscope. Thereafter, the fracture surfaces of the specimens were gold-coated and subsequently re-examined in a stereomicroscope. Interesting fractographic features were digitally photographed. Characteristic fracture features such as arrest line, fracture origin, fracture mirror, hackle and twist hackle were sought. Finally, selected areas on one of the specimens were examined in a scanning electron microscope (SEM).

Paper II

Specimen preparations
Two types of silica-based ceramics, hybrid ceramics and oxide ceramics, respectively, were selected for the study. The silica-based ceramics were represented by a feldspar-based ceramic (Vita Mark II) and a lithium disilicate glass-ceramic (IPS Empress 2). The hybrid ceramics were
represented by a lanthanum glass-infiltrated alumina (In-Ceram Alumina) and a zirconia reinforced lanthanum glass-infiltrated alumina (In-Ceram Zirconia). The oxide ceramics were represented by a densely sintered alumina (Procera AllCeram) and a HIPed Y-TZP ceramic (Denzir). Ten specimens of each material were manufactured in accordance with the manufacturers’ instruction. Five specimens of each material were then randomly selected.

**Surface treatments and measurements**
The surface roughness was measured using a profilometer (Surtronic 3; Rank Taylor-Hobson, Leicester, England), where the parameter Ra was calculated. The surface roughness determined before grinding and polishing the specimens was used as baseline. After grinding with diamond burs of three different coarsenesses (medium, fine and extra fine), the surface roughness was re-measured. Finally, after polishing with Sof-Lex Discs with four different grain sizes (coarse, medium, fine and superfine), the surface roughness was re-measured using the profilometer. All grinding, polishing and measuring was conducted by the same person. In addition, selected specimens were qualitatively studied in SEM.

**Statistical analysis**
In order to determine the difference between the various core materials within each surface condition, one-way analysis of variance (ANOVA), supplemented by Scheffe’s multiple-comparison test, was used. In order to determine the effect of grinding and polishing within each core material, one-way ANOVA supplemented by Bonferroni multiple-comparison test was used. A $P$-value of $< 0.05$ was considered significant.

**Paper III**
The R-T$^{2}$D code used in Paper III is a 2D numerical modeling method utilized to examine the fracture mechanism and process in brittle materials and was developed at the Division of Rock Engineering, Luleå University of Technology, Luleå, Sweden in cooperation with Northeastern University, Shengyang, China in order to study rock fragmentation process. The R-T$^{2}$D code is based on the rock failure process analysis (RFPA) code and the finite element analysis (FEA) method (Liu, 2004). The R-T$^{2}$D code was used in the present work to simulate the fracture process in a three-unit HIPed Y-TZP FPD framework. The model consisted of HIPed Y-TZP ceramics for the framework, and stainless steel for the abutments and the loading head. The selection of the materials, the shape and the dimensions of the numerical model were based on a physical model used in a previous laboratory study (Sundh et al., 2005) and the problem was simplified to a 2D plane strain condition. In total 99 000 elements were
generated. The HIPed Y-TZP ceramic was assumed to be a heterogeneous material and the heterogeneity was described using the Weibull distribution law. The stainless steel was assumed to be a homogeneous material. In this study, the maximum principal stress described the compressive stress and the minimum principal stress described the tensile stress. Hooke’s law was applied to describe the relationship between stress and strain until failure occurred. The Mohr-Coloumb criterion with tensile strength cut-off was applied as the failure threshold. To simulate the loading of the FPD framework a displacement increment of 0.002 mm per step was statically applied on the loading head, which indicated increased loading. After failure, linear damage fracture mechanics was used.

**Paper IV**

**Laboratory test**
One specimen of a three-unit HIPed Y–TZP FPD framework of identical size and geometric design as the frameworks tested in Paper I was studied. The frameworks, which had been fabricated using computer aided designing/computer aided manufacturing (CAD/CAM), were used as the base for the size and geometric form of the framework studied in Paper IV. The diameter of the connecting areas between the abutments and the pontic was 3 mm. The distance between the centers of the abutments was 17.5 mm. The framework was cemented with zinc phosphate cement on to stainless steel abutments in a stainless steel socket. Using a universal testing machine the specimen was then statically loaded, occlusally, until fracture occurred.

**3D numerical simulation**
The numerical simulation code used in Paper IV was developed at the Institute of Mechanics, Chinese Academy of Sciences in Beijing, China. It is based on GiD, a 3D graphic pre-and post-processor for computer simulation and Finite Element Program Generator, which is a platform for generating finite element modeling codes. Using this 3D numerical modeling code the stress distribution and fracture process in a simulated three-unit ceramic FPD framework of the same shape and size as the framework in the laboratory test were studied.

The geometric form of the framework in the laboratory test was designed by a CAD program. For the geometric shape in the numerical simulation, representative nodes were selected from the CAD data and the coordinators of the nodes were entered into GiD to generate finite elements for the solid model of the framework. In total 302,200 elements were generated. The framework and the abutments were simulated as HIPed Y-TZP and soft-layers were made under the abutments. All the materials were assumed to be heterogeneous and the heterogeneity was described using the Weibull
distribution law. In this study, the maximum principal stress described the tensile stress and the minimum principal stress described the compressive stress. Hooke’s law was applied to describe the relationship between stress and strain until failure occurred. The Mohr-Coloumb criterion with tensile strength cut-off was applied as the failure threshold. To simulate the loading of the framework, displacement of the loading areas was applied, 0.002 mm/step. After failure, the material modulus were reduced by a degradation factor R with R>>1.

In addition, the effect of surface grooves/irregularities on the fracture of ceramic FPD frameworks was examined. To evaluate the effect, a cylinder-shaped zirconia-based bar was created with one groove introduced in the high tensile stress concentrated area on the surface. In total 198 655 elements were generated and grooves with different depths were simulated.
Results

Paper I

Using the fluorescence penetrant method, no other cracks/defects apart from grinding grooves could be detected on the surface of the frameworks after CAM machining. After fracture, compression curls (Fig. 3) were seen in all specimens indicating that the fracture started at the gingival side.

Fig. 3. The image of the buccal view of a gold-coated HIPed Y-TZP substructure after fracture. The arrows mark the fracture. The compression curl is indicated by white arrows.

When the fracture surfaces were studied in the stereomicroscope white spots were detected around the loading area (Fig. 4). One possible explanation for this color change could be that the loading equipment indentation caused cracks in the loading area and the presence of cracks caused different light reflection and gave a white-colored appearance. After gold coating, the specimens were re-examined in the stereomicroscope.

Fig. 4. Stereomicroscope image of the fracture surface of a specimen before gold coating. The white spots are most certainly cracks within the thickness of the Y-TZP ceramic resulting from the crunching of the substructure and reflecting the light differently. Contact damage, arrest lines and twist hackle can be seen on the top (occlusal) surface of the specimen.
Mussel shell-like patterns that were most likely caused by the contact loading by the steel ball can be seen (Figs 4, 5, 6 a-c, 7a). Their appearance suggested that oblique loading had been performed on the lingual and buccal cusps and that the cracks seem to have hesitated at the last arrest lines. The fracture origin could not be identified in the stereomicroscope and even though a fracture mirror could be localized in the gingival portion of the FPD substructure in the stereomicroscope, it was difficult to capture on a photograph because of the uneven topography of the specimens.

One specimen with the largest number characteristic fracture features was selected for further SEM analysis. With a higher magnification in the SEM evaluation, the characteristic fracture patterns, indicating the direction of crack propagation (dcp) such as hackle, twist hackle, fracture origin and fracture mirror, were identified. Twist hackle was present close to the occlusal part (Figs. 5, 6d, 6e) indicating that the dcp passed the central part of the specimen and went from the gingival portion of the specimen upwards approaching the occlusal part. Mainly intergranular fracture could be observed (Fig. 7C). The fracture origin could be observed as a very small (<10 µm) flaw (Fig. 7C) located to one of the parallel milling grooves at the gingival side of the FPD substructure.
Fig. 6. Scanning electron microscope images derived from the black squared areas in Fig. 5. Characteristic fracture features are indicated by white arrows. The black arrows mark the direction of crack propagation (dcp) on the fractured surfaces.
Fig. 7. (A) Scanning electron microscope image of the fractured surface (12X). The black arrow marks the location of the fracture initiation site. The main crack propagation direction is indicated by a red arrow and this crack originated at the gingival (tensile) side. Cracking resulting from the occlusal loading with the steel ball is indicated by blue arrows and the cracks seem to stop at the level of the last arrest lines. (B) Scanning electron microscope image of fracture surface (200X). The fracture mirror is indicated by black arrows. (C) Scanning electron microscope image of the fracture origin (1000X). The fracture origin, a < 10 µm flaw, is shown by a black arrow. Mainly intergranular fracture can be observed.
Fig. 8. Images of the assembled pieces of the fractured specimen. White arrows indicate chipping and fracture. This specimen was sectioned to make it possible to examine the fractured framework in the SEM. Thus, the white area in the upper part of the specimen in Figures A–C is the section of the zirconia surface facing one of the abutments, after the specimen had been sectioned. ‘Ling’ indicates the lingual direction of the specimen. (A) Buccal view. (B) Gingival view. (C) Occlusal view.
In summary, the prime critical crack responsible for catastrophic failure of the zirconia-based FPD substructure started at the gingival part as a result of tensile stresses. The fracture origin was identified as a $<10$ µm flaw in association with a milling groove. The crack propagated towards the occlusal side meeting the crack event starting from the occlusal indentation loading sites and moving downward over one third of the surface.

**Paper II**

In Fig. 9 the medians, first and third quartiles are presented. At baseline, the IPS Empress 2 specimens had the roughest surface, whereas Denzir and Procera AllCeram surfaces were the smoothest (Fig. 9). Among the In-Ceram Alumina specimen defects on the surface could be identified using SEM (Fig. 10). After grinding, all of the cores became rougher compared to baseline (Table 1) except for IPS Empress 2 which became smoother, confirmed by the SEM evaluation (Fig. 11). Polishing with Sof-Lex discs made all of the core materials smoother compared to their state after grinding, with the exception for Procera AllCeram and In-Ceram Alumina (Table 2).

![Box plot diagram illustrating the surface roughness (Ra, µm) of the ceramic core materials evaluated. N=5 in each test group. Data are presented as medians and first and third quartiles. A horizontal line within the box signifies the median. The maximum and minimum values are illustrated by the upper and lower strokes. (*) marks extreme outliers. (O) marks outliers.](image-url)
Fig. 10. Scanning electron microscope image of an In-Ceram Alumina specimen. At baseline (a), after grinding with diamond burs (b), and after polishing (c). The arrows indicate defects on the surface (200X).

Fig. 11. Scanning electron microscope image of an IPS Empress 2 specimen. At baseline (a), after grinding with diamond burs (b), and after polishing (c) (200X).
Table 1. Summary of statistical analyses of surface roughness of varying core materials within each surface condition evaluated before grinding (baseline), after grinding with diamond burs (after grinding) and after polishing (after polishing). Mean values ± standard deviation (SD) of the surface roughness obtained are presented. \textit{a} denotes results of one-way ANOVA and Scheffe’s multiple-comparison test. Groups with the same letter was not statistically significantly different (p < 0.05).

<table>
<thead>
<tr>
<th>Surface condition</th>
<th>Specimens</th>
<th>Mean surface roughness ± SD (Ra, (\mu m))</th>
<th>\textit{a}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>Vita Mark II</td>
<td>1.34 ± 0.27</td>
<td>A</td>
</tr>
<tr>
<td></td>
<td>Denzir</td>
<td>0.63 ± 0.29</td>
<td>B</td>
</tr>
<tr>
<td></td>
<td>Procera AllCeram</td>
<td>0.56 ± 0.25</td>
<td>B</td>
</tr>
<tr>
<td></td>
<td>IPS Empress 2</td>
<td>4.15 ± 0.54</td>
<td></td>
</tr>
<tr>
<td></td>
<td>In-Ceram Alumina</td>
<td>1.00 ± 0.19</td>
<td></td>
</tr>
<tr>
<td></td>
<td>In-Ceram Zirconia</td>
<td>1.30 ± 0.37</td>
<td>A</td>
</tr>
<tr>
<td>After grinding</td>
<td>Vita Mark II</td>
<td>1.78 ± 0.40</td>
<td>C</td>
</tr>
<tr>
<td></td>
<td>Denzir</td>
<td>1.40 ± 0.20</td>
<td>D, E</td>
</tr>
<tr>
<td></td>
<td>Procera AllCeram</td>
<td>1.35 ± 0.31</td>
<td>D, E</td>
</tr>
<tr>
<td></td>
<td>IPS Empress 2</td>
<td>1.27 ± 0.21</td>
<td>E</td>
</tr>
<tr>
<td></td>
<td>In-Ceram Alumina</td>
<td>1.89 ± 0.46</td>
<td>C</td>
</tr>
<tr>
<td></td>
<td>In-Ceram Zirconia</td>
<td>1.50 ± 0.36</td>
<td>D</td>
</tr>
<tr>
<td>After polishing</td>
<td>Vita Mark II</td>
<td>0.88 ± 0.37</td>
<td>F</td>
</tr>
<tr>
<td></td>
<td>Denzir</td>
<td>0.94 ± 0.25</td>
<td>F</td>
</tr>
<tr>
<td></td>
<td>Procera AllCeram</td>
<td>1.33 ± 0.55</td>
<td>G</td>
</tr>
<tr>
<td></td>
<td>IPS Empress 2</td>
<td>0.85 ± 0.28</td>
<td>F</td>
</tr>
<tr>
<td></td>
<td>In-Ceram Alumina</td>
<td>1.77 ± 0.47</td>
<td></td>
</tr>
<tr>
<td></td>
<td>In-Ceram Zirconia</td>
<td>1.24 ± 0.30</td>
<td>G</td>
</tr>
</tbody>
</table>
Table 2. Summary of the statistical outcome of the surface roughness within each ceramic core material before grinding (baseline), after grinding with diamond burs (after grinding) and after polishing (after polishing). > indicates statistically significantly higher surface roughness. Results of one-way ANOVA and Bonferroni multiple-comparison test (P < 0.05).

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Comparison within each core material</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vita Mark II</td>
<td>After grinding &gt; Baseline &gt; After polishing</td>
</tr>
<tr>
<td>Denzir</td>
<td>After grinding &gt; After polishing &gt; Baseline</td>
</tr>
<tr>
<td>Proceras AllCeram</td>
<td>After grinding, After polishing &gt; Baseline</td>
</tr>
<tr>
<td>IPS Empress 2</td>
<td>Baseline &gt; After grinding &gt; After polishing</td>
</tr>
<tr>
<td>In-Ceram Alumina</td>
<td>After grinding, After polishing &gt; Baseline</td>
</tr>
<tr>
<td>In-Ceram Zirconia</td>
<td>After grinding &gt; Baseline, After polishing</td>
</tr>
</tbody>
</table>

In summary, manual polishing using the Sof-Lex system significantly reduced the surface roughness of the cores of HIPed Y-TZP (Denzir), zirconia reinforced lanthanum glass-infiltrated alumina (In-Ceram Zirconia), lithium disilicate glass-ceramics (IPS Empress 2) and feldspar-based ceramics (Vita Mark II) after grinding with diamond burs, whereas manual polishing of the cores of densely sintered alumina (Procera AllCeram) and lanthanum glass-infiltrated alumina (In-Ceram Alumina) using the Sof-Lex system was ineffective.

**Paper III**

Stress distribution, fracture initiation and propagation in a simulated three-unit zirconia–based FPD framework could be shown using the R-T^2D code. The quasi-photoelastic stress fringe pattern gave a good picture of the stress distributions in the three-unit zirconia–based FPD framework and of the heterogeneity of the materials (Fig. 12). Before fracture, the photoelastic stress fringe pattern was similar to straight lines in the abutments, indicating homogeneous material, whereas in the pontic of the framework, the fringe patterns were less distinct, indicating heterogeneous material (Fig. 12). The compressive stresses were greatest close to the loading area and the wave-like patterns in the lower boundary of the pontic indicated the location of a high concentration of tensile stress. It could also be seen that the tensile stress concentration was larger on the right side than on the left, thus the fracture probably started at the right connector (Fig. 12).
Another feature available in the R-T2D code was acoustic emission (AE). A circle represented a failed element with the centre located in the center of the element. The radius of the circle represents the magnitude of the energy released by that particular element. The color red indicated failure by tension, blue indicated failure by compression. The AE showed that the fracture started gingivally on the right connector and, since the circles were all red, tension was the main cause of the fracture of this simulated framework (Fig 13). It was also shown that all the breakage happened in one single loading level, which is typically a phenomenon of brittle fracture.
Using the R-T2D code allowed the fracture process to be followed step by step and step in step. According to this analysis, the stress distribution varied continuously during the fracture process and the fracture pattern was determined by the stress distribution in the framework (Fig. 14). Comparisons of the fracture pattern between that produced by the R-T2D code and the fracture pattern in an earlier laboratory study of HIPed Y-TZP frameworks of similar shape and size (Sundh et al., 2005) as in the present numerical simulation (Paper III) showed that they closely agreed with each other (Fig. 15). One important difference between the numerical modeling and the laboratory test was that the whole fracture process could be followed step-by-step in the 2D numerical modeling, whereas in the laboratory test the fracture happened suddenly and it was difficult to follow the process.

Fig. 14. Representative views of the maximum principal stress distribution (A), the minimum principal stress distribution (B), and the fracture pattern. The grey scale in the figure indicates the stress level. Arrows mark the brightest color and indicate the highest stress concentration.
Fig. 15. Comparison between the fracture pattern of a three-unit Y–TZP FPD framework in a previous laboratory test using a universal testing machine (A) and the results of the present numerical simulation using the R-T²D code (B). In (A) the fracture is indicated by black arrows. The grey scales in (B) represent the distribution of Young’s modulus; the brighter the color, the higher the value of Young’s modulus. The black line indicates the fracture.

In summary, the 2D numerical simulation using the R-T²D code showed that the crack-initiation site was located in the gingival portion of the framework, the framework fracture was caused by tensile failure, the fracture process could be followed step by step and the fracture pattern obtained from this simulation correlated well with an earlier laboratory test using frameworks of similar size and shape.
**Paper IV**

In Paper IV, not only could the stress distribution in the three-unit zirconia-based FPD framework be simulated and observed but the fracture process could also be followed step by step in 3D. At Step 3, the first fracture initiated at the loading area at the buccal cusp on the occlusal side of the framework could be seen (Fig. 16, Step 3). Another fracture initiated at the gingival embrasure close to the buccal side of the substructure at Step 5 (Fig. 16, Step 5). At Step 6, the substructure was totally fractured, a typical sign of brittle fracture (Fig. 16, Step 6). At Step 18, a high maximum principal stress could be observed at the occlusal part of the connector area close to the left abutment (Fig. 17A3), which could be the reason for the fracture initiation site at the area close to the left abutment of the framework in Step 18 in Fig. 16.

![Figure 16. The fracture propagation in the three-unit framework. The color mark indicates different levels of fracture for each step. No fracture is present when the flag=0. An element has completely failed when the flag=1. Flag between 0 and 1 indicates that an element has partly failed. The color red indicates high flag numbers and blue indicates low flag numbers.](image-url)
Figure 17. Images of representative views of the maximum ($\sigma_1$) and minimum ($\sigma_3$) principal stress distributions. The rainbow colored marker indicates the stress value (MPa) in the three-unit framework for each step. The reddest color indicates the highest stress and the bluest color indicates the lowest stress. Tensile stress is defined with a positive value and compressive stress is defined with a negative value. Therefore, maximum principal stress ($\sigma_1$) describes the tensile stress and minimum principal stress ($\sigma_3$) describes the compressive stress.

For validation, the fracture pattern of the numerically obtained substructure was compared with the result received from the laboratory test. Comparisons between the laboratory result and the numerical study showed that the fracture patterns in the gingival parts of the substructure correlated well with each other (Fig. 18a,d); in the buccal part, the compression curl could be observed in both cases (Fig. 18b,e), whereas the shape of the fracture pattern occlusally differs a little between the two cases (Fig. 18b,e and Fig. 18c,f). Overall the numerical simulation result provides a good guidance as regards the shape of the fracture pattern of the ceramic framework.
Figure 18. Comparison between the fracture patterns obtained in the laboratory test (left column) and in the numerical simulation (right column). Gingival view (a, d). Buccal view (b, e). Occlusal view (c, f). In a, b and c, the color difference is caused by the low angle illumination used to accentuate the fracture. The black arrows indicate the fracture.
To study the effects of surface irregularities on the fracture, grooves of varying depths were introduced at the gingival part of a ceramic cylinder–shaped bar where high tensile stress was located when it was occlusally loaded (Fig. 19). The results revealed that at Step 7 (flag=0.51767) none of the elements had completely fractured in the cylinder-shaped zirconia-based bar without any groove (Fig. 19), whereas in the bar with a groove 0.02 mm deep total fracture of at least one element could be observed at Step 6 (flag=1). With a groove 0.04 mm deep total fracture of at least one element could already be observed at Step 3 (flag=1). Thus, the deeper the groove, the sooner the bar fractured.

Figure 19. Selected steps in the fracture propagation in a cylinder-shaped bar with grooves of varying depths introduced on the surface. Buccal view of the loading model (a). Gingival view of the bar with no grooves (b). Gingival view of the bar with a groove 0.02 mm deep (c). Gingival view of the bar with a groove 0.04 mm deep (d). The color marks to the right indicate different levels of fracture. No fracture is present when the flag=0. An element has failed completely when the flag=1. Flag between 0 and 1 indicates that an element has partly failed. The color red indicates high flag numbers and blue indicates low flag numbers.
In summary, the numerical simulation showed that the first fracture initiated at the buccal cusp in the occlusal area (Fig. 16, Step 3). However, this initiation site was not the one that caused the total catastrophic failure of this FPD framework. The decisive fracture initiated at the gingival embrasure. In addition, the 3D numerical simulation revealed that irregularities on the ceramic surfaces, especially in the areas of concentrated tensile stress, affect the fracture and may have a negative influence on the strength of ceramic restorations.
General discussion

It has been reported previously that flaws and irregularities on ceramic surfaces can often have negative impacts on the strength of ceramic restorations (McLean & Hughes, 1965; de Jager et al., 2000; Fischer et al., 2003; Quinn, 2007; Lohbauer et al., 2008). In the present thesis the surfaces of ceramic substructures were therefore screened out using a fluorescent penetrant method in order to detect surface defects on zirconia substructures (Paper I). In addition, as it is known that the rougher the surface the lower the strength (Fischer et al., 2003), the possibility of smoothing out the surfaces of ceramic cores/substructures using a variety of surface treatments was evaluated with a profilometer (Paper II).

Laboratory tests in combination with fractographic analysis were applied in order to study the fracture behavior in three-unit ceramic FPD frameworks (Paper I). In this way the fracture mechanism in the ceramic frameworks was identified as tensile failure and fracture initiation sites were found both on the occlusal area and in the gingival embrasure. These tests led to an understanding that the basic phenomenon involved in the fracture of a three-unit ceramic FPD specimen is one of mechanics.

Using both mathematical and mechanics approaches to reproduce the fracture of dental ceramics in an environment similar to that in the laboratory tests should lead to better understanding of the fracture mechanism. This idea resulted in the numerical simulations (Papers III and IV) of fracture in ceramics FPD frameworks using recently developed 2D and 3D numerical modeling codes. In these numerical models, heterogeneity within the material, failure criteria of the materials, surface irregularities on the material and loading conditions were formulated mathematically. The simulated results obtained not only presented fracture shapes and patterns which were largely similar to those obtained in the laboratory tests, but also provided information about the stress distribution during the loading process and the fracture initiation and the development step by step (Papers III and IV). These findings revealed that the numerical codes used in the present thesis could possibly partly replace time-consuming, expensive laboratory tests and reduce costs in the design of ceramic FPD frameworks.

Heterogeneity on the ceramic surfaces

As mentioned earlier, fracture in ceramics can emanate from surface defects and/or from volume imperfections. Consequently, since surface treatments, such as manual grinding and CAM machining, are said to cause defects on the surface of ceramics (Denry et al., 1999; Luthardt et al., 2004), evaluation of the occurrence of defects and flaws on ceramic restorations was important. In the present thesis, the fluorescent penetrant method was,
therefore, applied in Paper I in order to ascertain whether there were any flaws on the CAM machined ceramic frameworks. No cracks/defects, apart from milling grooves, could be observed on the surfaces of the zirconia-based frameworks after machining. The possibility, however, cannot be excluded that there were cracks on the surface of the specimens but they were too small to be detected using the current fluorescence method (Luthardt et al., 2004).

In Paper I, the fracture origin was identified as a $< 10 \mu m$ flaw at the gingival embrasure of the machined HIPed Y-TZP framework (Fig. 7C) and the decran parallel along a milling groove on the tensile side (Fig. 8B). In addition, it was shown in Paper I that flexural testing probably activated the parallel machining grooves which acted as strength-limiting flaws. Those findings, among others, motivated interest in studying how varying depths of milling grooves affect the fracture of the zirconia-based frameworks. Thus, in order to study the manner in which such grooves on ceramic surfaces may affect the fracture in ceramic frameworks, grooves of different depths were simulated at the tensile stress area in a cylinder-shaped HIPed Y-TZP bar (Paper IV). The width and depth of a simulated groove was based on the measurements of the machined framework in the laboratory test in Paper IV. For comparison purposes, the cylinder-shaped bar was also loaded with a simulated groove 0.04 mm deep and without any groove. The results in Paper IV revealed that surface irregularities such as grooves, obviously affect the fracture and the deeper the groove the sooner the bar fractured. Based on these findings (Papers I and IV), avoidance of grooves and other irregularities at tensile stress concentration areas of ceramic frameworks is desirable. Consequently, smoothing out the tensile stress areas of the ceramic frameworks might result in stronger and more durable constructions.

**Smoothing out ceramic surfaces**

Fracture in ceramic materials may originate in surface irregularities and defects (Quinn, 2007) and it has been reported in a number of studies that surface roughness affects fracture of ceramics and that there is a linear correlation between roughness and failure stress (McLean & Hughes, 1965; de Jager et al., 2000; Fischer et al., 2003; Lohbauer et al., 2008). These statements have recently been further verified in a study by Nakamura et al. (2010) who demonstrated that when the surface roughness increased the flexural strength decreased and that the surface roughness influenced the Weibull modulus (Nakamura et al., 2010). Thus, increased surface roughness on ceramics has been reported to have a negative impact on fracture strength and failure stress and it is recommended that ceramics should be carefully polished after grinding to achieve very smooth “roughness free” surfaces (Fischer et al., 2003). Accordingly, if polishing can
reduce the surface roughness of the ceramic materials, the risk of material fracture can be reduced.

These findings are of interest because clinical adjustments made by grinding with diamond burs could expose ceramic cores/substructures and introduce milling grooves and/or cracks that could act as stress concentrators and affect the fracture (Quinn, 2007). Therefore, since a number of reinforced core/substructure ceramics with improved mechanical properties have been introduced into dentistry (Andersson & Ödén, 1993; Kappert et al., 1995; Hornberger, 1996; Höland et al., 2000; Sundh & Sjögren, 2004), it was of interest to find out whether various surface treatments, such as manual grinding and polishing, could reduce the surface irregularities of newly introduced ceramic cores/substructures (Paper II).

One way to evaluate the texture of superficial surfaces quantitatively is to determine the surface roughness (European Standard EN 623–4:2004). After polishing with the Sof-Lex discs (Paper II), which in a preparatory study was identified as among the most efficient polishing systems for the ceramics evaluated, the results obtained showed that the surface roughness of the HIPed Y-TZP (Denzir) and the lithium disilicate (IPS Empress 2) were significantly reduced and similar to that of the feldspar-based ceramic (Vita Mark II) which served as control (Fig. 9). These findings indicate that it seems to be possible to achieve relatively smooth surfaces for the Denzir and the IPS Empress 2 cores/substructures by manual polishing which, based on earlier studies (de Jager et al., 2000; Fischer et al., 2003; Lohbauer et al., 2008), should be instrumental in reducing the risk of fracture in the restorations.

In this context, it should be stressed that conflicting effects of surface treatments on the strength of zirconia-based ceramics have been reported (Kosmač et al., 1999; Zhang et al., 2004; Guazzato et al., 2005; Deville et al., 2006). For example, the studies by Guazzato et al. (2005) and Kosmač et al. (1999), showed that sandblast treatment increased the flexural strength of the Y-TZP specimens, whereas the study by Zhang et al. (2004) showed that sandblasting reduced their strength. Moreover, Guazzato et al. (2005) have shown that grinding Y-TZP surfaces can increase the flexural strength, whereas Kosmač et al. (1999) showed that grinding reduced the flexural strength. Damage to the surface has often been given as the reason for the strength reduction and the phase transformation from tetragonal phase to monoclinic phase has often been suggested as the reason for the increased strength (Kosmač et al., 1999; Deville et al., 2006). Hence, it is still not totally clear how and why different surface treatments affect zirconia-based ceramics and more studies have to be performed to analyze this issue further.
Heterogeneity within ceramic materials

One of the reasons for the interest in the heterogeneity of materials is because the occurrence of voids and impurities is not only common on the surfaces but also within materials (Munz & Fett, 1999; Quinn, 2007). In other words, defects could be found within materials which could influence their mechanical properties and failure behavior (Munz & Fett, 1999).

Various methods have been applied to find heterogeneity in materials. For example, in the context of numerical simulations in biomechanics, the large extent of heterogeneity has sometimes been shown by computer tomography (CT) (Magne, 2007; Tajima et al., 2009). That is, after the examined specimen was CT-scanned, the constitution of the examined parts could be identified, such as enamel, dentin or ceramics, and the material properties for each material were then defined. However, this method is relatively expensive, time consuming and it is difficult to identify the dispersed heterogeneity within each material.

Brittleness is the major drawback in ceramics and it often has a large scatter in strength caused by the scatter in the defect size compared with metals (Munz & Fett, 1999) and these properties need to be considered in the design of ceramic components (Munz & Fett, 1999). Since fracture in ceramics can start from surface defects and/or from volume imperfections, it is important to identify the defects or imperfections and to find a proper way of describing them. However, the dispersed volumetric defects are complex and it is difficult to define them exactly. A statistical method, the Weibull distribution law, was therefore applied in the present thesis (Papers III and IV). The normal distribution function is a good approximation when the results depend more or less equally on a large number of events but it is not appropriate for fracture in ceramics where the failure depends more on the most serious flaws (Munz & Fett, 1999). Based on earlier experiments, scientists in various fields have stated that the Weibull distribution law can be an appropriate statistical method for describing the dispersed heterogeneity in brittle materials (Liu, 2004; Quinn & Quinn, 2010). Thus in the present thesis, the Weibull distribution law was applied both in the 2D and the 3D simulations to describe the dispersed heterogeneity within the ceramics (Papers III and IV).

When using the Weibull distribution law, different homogeneity index \( m \) values can cause different failure modes. With a low \( m \) value, an increased number of micro-fractures could be observed and with a high \( m \) value, a decreased number of micro-fractures could be observed and the material behaves in a more brittle fashion (Liu, 2004). Moreover, in the present thesis, the disorder of the probability distribution in physical space was achieved by the Monte Carlo method and a series of random data with a uniform distribution between 0 and 1 was generated. In this way, difference in the mesoscopic structure could be achieved.
Fracture in three-unit ceramic FPD frameworks

In the present thesis the fracture process in three-unit ceramic zirconia-based FPD frameworks was studied. The fracture mechanism was identified as tensile failure, which was confirmed both in the fractographic analysis (Paper I) and in the numerical analyses (Papers III and IV). The 3D numerical simulation (Paper IV) indicated that the first fracture initiation site was located on the occlusal area, but this fracture initiation did not cause the total fracture of the frameworks. Although the fracture initiation has been identified on the occlusal side of the framework, the findings in the numerical simulation in Paper IV revealed that the three-unit zirconia-based framework could still function. However, if the fracture initiation site was located in the gingival part, which is at the area of concentrated tensile stress in the framework, there was a greater risk that it would failed catastrophically. Therefore, it seems likely that by reducing the tensile stress on the frameworks and/or by increasing their tensile stress resistance by other means, the lifetime of three-unit ceramic frameworks could be prolonged.

Failure criterion

The purpose of a failure criterion is to predict/estimate the failure of a material in numerical simulations. A failure criterion frequently used in finite element analysis of ceramic restorations in dentistry is von Mises stress criterion (Ausiello et al., 2004; Eraslan et al., 2005; Oruc et al., 2008; Tsumita et al., 2008). This states that failure occurs when the energy of distortion reaches the same energy as for yielding in uniaxial tension and is a shear stress criterion often used to estimate the yield of ductile materials, such as metals (von Mises, 1913). In this criterion, shear stress is considered the only parameter that effects yielding/fracture. In the case of fracture in brittle materials, in addition to shear stress, tensile stress and confining stress also play important roles, as has been confirmed by earlier laboratory studies (Jaeger & Cook, 1976). Another failure criterion is the Mohr-Coulomb failure criterion, which apart from shear stress, also takes the confining stress and the tensile stress into consideration (Liu, 2004). Thus, the Mohr-Coulomb strength failure criterion would be a more appropriate failure criterion for brittle material, such as ceramics, than the von Mises stress criterion and, therefore, in the present thesis the Mohr-Coulomb strength failure criterion was selected.

A survey of the literature addressing numerical studies of stress distribution and fractures in ceramics in dentistry found only one article (Dejak & Mlotkowski, 2008) that applied the Mohr-Coulomb failure criterion. In that study, the authors claimed that when the Mohr-Coulomb failure value reached 1 an element could be considered as failed. Although they made this statement the calculation in the study continued without any
change in material properties after the Mohr-Coulomb failure value had reached 1 and a Mohr-Coulomb value as high as 2.62 was obtained (Dejak & Mlotkowski, 2008). However, when one element has failed, its material properties should be changed and Hooke’s law should no longer be applied to that element. A new constitutive relationship for the failed element needs to be established because the failed element is incapable of bearing such a high load as before its failure. For this reason the stresses in the whole restoration should be recalculated taking the failed element into consideration. In order to share the load carried by the failed elements, the stresses in the neighboring elements have to be increased.

In the present thesis, the Mohr-Coulomb failure criterion with a tensile strength cut-off was applied. An element was considered failed when the Mohr-Coulomb criterion was met. Instead of Hooke’s law, a new relationship between stress and strain was established for the failed element (Papers III and IV). Recalculations of the stresses of every element in the whole framework were performed by taking the failed element into consideration and in most cases the stress redistribution also brought failure to one or several neighboring elements. The recalculations continued until a new stress equilibrium in the framework was established. This procedure usually causes a chain reaction of element failures and the failed elements describes a trace similar to the real fracture (Papers III and IV).

Comparison of the 2D and the 3D numerical results with laboratory studies

In recent years, numerical simulations such as finite element analysis have been used in dentistry to analyze stress distribution in single teeth and FPDs (Proos et al., 2000; Oh et al., 2002; Romeed et al., 2004; Oruc et al., 2008; Tsumita et al., 2008). However, there are few articles that have validated the results. In the present thesis, the results from the numerical calculations were validated with the results from laboratory tests.

In the present work, the fracture pattern was one of the parameters that was applied in the validation. In the 2D numerical simulation in Paper III, the fracture pattern buccally correlated well with a previous laboratory test in which HIPed Y-TZP frameworks of a similar size and shape to the simulated framework (Paper III) were studied (Sundh et al., 2005). In the 3D numerical simulation in Paper IV, the fracture pattern was compared with the laboratory test in the same paper from three different angles, that is, buccally, occlusally and gingivally. From the gingival point of view, the fracture patterns obtained correlated well with each other and for the occlusal and buccal views, a good guidance concerning the shape of the fracture pattern of the ceramic framework was provided.

In the fractographic analysis in Paper I, the fracture mechanism of the fractured three-unit zirconia-based frameworks was identified and fracture
initiation sites were seen on the occlusal area and in the gingival embrasure, a little towards the buccal side of the framework. Similar locations for fracture initiation sites were also identified in the 3D numerical model. However, the fracture process in the three-unit zirconia-based frameworks could not be completely clarified by the fractographic analysis in Paper I, whereas the 3D numerical simulation in Paper IV revealed that the first fracture initiation site was located on the occlusal area. However, this initial occlusal fracture did not cause the total fracture of the framework, information that could not be elicited from the fractographic analysis.

Both in the 2D and 3D numerical modelings, the stress values for every element in the framework and the stress distribution during the fracture process could be followed step by step. The numerical methods revealed where and why the fracture initiated, how and why the fracture propagated and why the fracture hesitated or changed direction. All this information became available because of the numerical modeling methods but could not be provided by laboratory tests alone.

**Comparison between the 2D and the 3D numerical simulations**

The 2D numerical simulation (Paper III) was carried out using the R-T^2D code (a two dimensional code for studying Tool-Rock Interaction). This code has previously been shown to be an appropriate tool for simulating fracture processes in rocks (Liu, 2004). Since this code could simulate fracture behavior in rocks, the assumption was that it would also be an appropriate method for simulating fracture processes in other brittle materials, such as ceramics. This assumption was verified in Paper III, where it was shown that the 2D code can simulate stress distribution and fracture processes in ceramic FPD frameworks.

Although the basics of the fracture process of three-unit ceramic FPD frameworks could be captured by the 2D numerical simulation (Paper III), the oral cavity is more of a 3D environment. Since the 3D numerically simulated results may have the possibility of better simulating reality than 2D simulation, a new 3D numerical modeling was performed on a three-unit ceramic FPD framework (Paper IV). In this 3D numerical simulation, the fracture initiation site was located on the gingival embrasure and a little towards the buccal side of the framework, which was a more accurate localization of the fracture initiation site than that obtained by the 2D simulation (Paper III). In other words, more information, of greater accuracy, was revealed by the 3D numerical simulation than the 2D numerical simulation.
**Simplifications**

The core/framework is the key part that sustains the load in ceramic FPDs, therefore, only fracture in the framework was studied in the current thesis. In addition, the loading condition in the oral cavity is complex and varies both among individuals and within the same individual, but in the present work only static mechanical loading with an axial load was applied.

In the 3D simulation (Paper IV), it was difficult to accurately simulate the space filled with cement between the abutment and the retainer. Since the stainless steel abutments and the retainers never fractured before the fracture of the framework in the previous laboratory tests (Sundh *et al.*, 2005; Sundh & Sjögren, 2006) or in Papers I and IV, a model with zirconia-based abutments with soft-layers at the bottom was used in Paper IV. Thus, the soft-layers were created under the zirconia abutments to compensate for the load on the zirconia abutments and to avoid fracture of the abutments in the numerical simulation in Paper IV. In addition, since the soft-layers should remain intact during the loading process, high values for tensile and compressive strength were chosen for these layers.

Many factors play a role in fractures of ceramic FPDs in clinical situations. A detailed calculation of the stress distribution and failure, taking all the factors in the real oral situation into consideration, is still not possible, which clearly indicates the need for simplified models for handling the complexity. Simplification of the clinical situation into a laboratory and/or numerical simulation model is, thus, still necessary if meaningful and interpretable results are to be achieved without sacrificing too much accuracy.
Major results and conclusions

- Using the fluorescence penetrant method, no cracks or other defects apart from milling grooves could be detected on the surfaces of CAM machined HIPed Y-TZP-based FPD frameworks.

- The decisive fracture initiation site that caused the catastrophic failure of the zirconia-based FPD frameworks started at the gingival part as a result of tensile stresses. The fracture initiated in association with a milling groove.

- Manual polishing can significantly reduce the surface roughness of HIPed Y-TZP (Denzir), zirconia reinforced lanthanum glass-infiltrated alumina (In-Ceram Zirconia), and lithium disilicate glass-ceramic (IPS Empress 2) cores, whereas densely sintered alumina (Procera AllCeram) and lanthanum glass-infiltrated alumina (In-Ceram Alumina) cores were not significantly affected by the polishing technique used.

- Numerical simulations demonstrated that surface grooves affect the fracture of the ceramic bars and the deeper the groove, the sooner the bar fractured. Thus, surface irregularities on the ceramic surfaces, especially in the areas where tensile stress is great, may have a negative influence on the strength of ceramic restorations.

- Both the fractographic analysis and the numerical methods revealed that the fracture mechanism of three-unit zirconia-based FPD frameworks was tensile failure.

- The fracture pattern obtained by the numerical simulation methods largely correlated with the patterns provided by the corresponding laboratory tests.

- Similar locations for fracture initiation sites were detected both with the 3D numerical simulation code and the fractographic analysis. In addition, the 3D numerical simulation code revealed that the first initial fracture site was located in the occlusal area, information that could not be obtained from the fractographic analysis.

- The newly developed 3D code clearly showed the stress distribution and the fracture process in three-unit zirconia-based FPD
frameworks step by step and seems to be an appropriate tool for use in the prediction of the fracture process in ceramic FPD frameworks.
Suggestions for future research

In the present thesis, the 3D numerical modeling was well able to simulate the fracture process in a three-unit zirconia-based FPD framework. However, as a stylized zirconia-based FPD framework fixed on standardized abutments was used in the present thesis more validation studies are planned that will also consider other parameters, such as: multilayered ceramic FPD that involves veneer ceramics and cements; frameworks of different materials, shape and dimensions; various abutment and loading conditions.

Hopefully, in the future the simulated results from 3D numerical modeling will be sufficiently trustworthy and the numerical simulation code will be used as a design tool for dental frameworks, thereby reducing the number of laboratory tests needed and reducing costs.
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