Strength testing variables in dental ceramics

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CHAPTER 1

Introduction
1.1.1 Strength testing variables in dental ceramics

Understanding the mechanical properties of ceramics and porcelains is essential for the explanation of the effects of applied forces on these materials and their responses to the forces. For that reason an overview of the structural as well as mechanical properties of dental ceramics is presented in this chapter.

1.1.2 Ceramics and porcelains

The first successful applications, in 1774, of ceramic materials in dentistry are all porcelain dentures. Until that moment tooth-colored substitutes of lost teeth had mainly been made from ivory, bone, animal teeth, and even extracted human teeth. Aided by the achievements of science and technology, the translucency and color of dental porcelains were improved and production techniques for dental restorative applications developed. Fused porcelain inlays and crowns were introduced around 1886, however, without any worry over the structural weakness [1, 2].

A milestone in the widespread application of dental porcelains was reached in 1950’s [3] with the invention of porcelains with a high coefficient of thermal expansion due to the addition of leucite, which enabled them to be fused to supporting structures made from gold alloys. This new and layered structure, with the patent title of ‘porcelain fused to metal’ (PFM) crowns [4, 5] and the practical name ‘metal-ceramic’, combined the favorable properties of metals and porcelain. This new technology and combination of materials enabled the fabrication of complete crowns and fixed partial dentures with a reliable durability. The rigid metal framework resists the firing contraction of porcelains and guarantees a precise fit on the prepared tooth. In the next decades metal, porcelain, their bonding, and their fabricating technology were developed further to reach the level of a clinical standard [6-8]. However, metal alloys also have disadvantages with regard to biocompatibility and esthetics. As the main reason to combine ceramics with metals is the lack of strength of the dental porcelains, much effort has been taken to develop strong ceramic materials to replace the metals.

Generally, porcelains belong to ceramics. Ceramics are classified into crystalline and non-crystalline ceramics. Non-crystalline ceramics are composed of glass that has been enriched with alumina or other powders, and are called ‘dispersion-reinforced porcelains’ or ‘glass-ceramics’. This second phase in glass results from devitrification or addition of particles or whiskers. Most crystalline ceramics are only partly crystalline as they are mainly ceramic composites; a glass matrix phase and a reinforcing filler fraction. A special group of dental ceramics is fully crystalline as
they lack a matrix phase. In dentistry the term ‘polycrystalline’ typically refers to ceramics, which are fully crystalline.

In the middle of the 1960’s the introduction of alumina as a reinforcement phase started the era of development of all-ceramic crown systems. First for the all-ceramic Jacket crown made of conventional dental porcelain, which was reinforced with alumina [9-11]. These restorations were only reliable in the anterior, low stress bearing region. Although products, like Hi-ceram, Vitadur N systems, were introduced in the 1970’s [12], it was only in the 1980’s before all-ceramic crown systems came up to clinical acceptability [13]. Among these early representatives were the “shrink-free” Cerestore [14] and a castable glass-ceramic Dicor [15]. Clinical experience with both systems showed that they were still not strong enough for application in the posterior region of the mouth [16-20]. When Empress 1, with its hot-press technique enabling a precise fit of restorations, came on the market the indication area for all-ceramic restoration was extended to the premolar region [21-23].

Prefabricated, i.e. already fired porcelains, glass-ceramics, and CAD/CAM were developed in the 1980’s. Milling the dental restoration from prefabricated ceramic blocks that do not require further sintering, effectively prevents high contraction during firing, and the amount of intrinsic flaws is reduced in this production method. The CAD/CAM-based production method, which is required for this method provides a clean and fast fabrication, and enables chair-side production of small all-ceramic restorations [24].

The pronounced development of full range dental all-ceramic systems began in the 1990’s when materials with enhanced mechanical properties like Empress 2, In-Ceram series, and Procera Allceram with strength’s of 300 to 700 MPa became available.

Empress 2 utilizes the hot-press production technology, equal to Empress 1 and shows an improved strength because it contains more than 60 vol% lithium disilicate interlocking grains. This material can be used in the anterior and premolar region for both crown and three-unit bridge restorations [25, 26].

In-Ceram series is a novel ceramic named ‘interpenetrating phase composite’, which combines glass and crystalline ceramic in three-dimensional and structural continuity. The size characteristics of the powder and the sintering procedure result in a partially sintered porous framework with more than 80 vol% crystals, which is subsequently infiltrated with molten glass by capillary action. Glass offers transmissivity and color as well as eliminates the pores. The main disadvantage of this system is the need to create enough space for the restoration especially at the margin to
obtain enough structural resistance to fracture. The three products in the In-Ceram series are Spinell, Alumina, and Zirconia, in order of increasing strength or decreasing translucency. The recommended indications are anterior crowns, anterior crowns and three-unit bridges combined with posterior crowns, posterior crowns, and three-unit bridges, respectively, according to the trade-off of the mechanical and esthetic qualities. In-Ceram Zirconia, as radio-opaque as metal was the first all-ceramic system recommended for short bridges in the posterior region [27-34].

The first polycrystalline ceramic used in dentistry was the alumina-based Procera AllCeram composed of micrometric pure alumina grains. Applying CAD/CAM technology to produce enlarged green bodies solved the problem of the large sintering shrinkage of polycrystalline ceramics. This kind of alumina is ivory white but to some extent also translucent. Despite the remarkable mechanical properties and clinical results, this technique was not suitable for bridge fabrication due to the dry pressing forming technique on enlarged die models [35-42].

A milestone of this development was reached at the end of the last century when poly-crystalline yttria stabilized zirconia, e.g products like Cercon, LAVA, and Everest, was introduced in dental industry. Zirconia ceramics are usually rather strong, above 900 MPa [43-48]. The main difference from other ceramics is its toughening mechanism, based on grain phase transformation. Depending on the temperature and external pressure zirconia has three crystallographic phases. With pure zirconia only the monoclinic phase (m) is stable at room temperature and pressure. The tetragonal phase (t) exists between 1170°C and 2370°C while the structure becomes cubic above 2370°C. So after pure zirconia is sintered at least at 100°C higher than 1170°C, it undergoes a phase transformation during cooling from the tetragonal to the monoclinic phase, which is accompanied by a volume increase of approximately 4.5%. Dopant additions such as CaO, MgO, Y₂O₃, and CeO₂ dramatically reduce the transformation temperature and stabilize the tetragonal phase at clinical temperatures. Stress may transform the stabilized tetragonal phase to monoclinic. This unique transformation in response to stress is very favorable, as it generates compressive stress, which contributes to crack arrest and superior mechanical properties. Due to the strength and toughness they are suitable as a substrate for veneering porcelain, as recommended for example as copings of crowns and frameworks of bridges in both the anterior and posterior regions. These materials became a most promising candidate for substitution of metal in esthetic restorations. The zirconia ceramics are often white and opaque, but they can be stained to be dentin-colored.
1.2.1 Mechanical aspects of dental ceramics

Essential for the mechanical properties is to understand that most materials applied in dentistry are composite materials in which a matrix material is ‘reinforced’ with filler particles. In glass ceramics the matrix is composed of an amorphous glass phase while the fillers are composed of crystalline glass or ceramic materials like leucite, alumina, or zirconia. The production of dental structures with these materials is achieved by sintering the milled ceramic powders, which are heated until the matrix melts and the filler particles fuse. The sintering causes shrinkage in excess of the thermal shrinkage from cooling to room temperature. The ability of the glass matrix to flow allows this type of materials to be condensed to metal structures without fracturing during cool-down to room temperature. This is not the case for the more homogenous poly-crystalline alumina or zirconia ceramics, in which no matrix phase is present. For this reason, these materials have to be shaped into an enlarged model in the green state, which shrinks down to a precise fitting shape after sintering.

As in general for composites, the tensile strength is much lower than the compression strength. In tensile stress the weakest link in the materials, i.e. the glass phase, is loaded, while in compression the stronger filler is loaded. For poly-crystalline materials the inter-grain interface is probably the weakest link in tensile loading. The structural difference between these types of ceramics is of major importance for understanding their fracture behaviour [49].

1.2.2 Stress-strain relations

The deformation of a material with an applied force is described as a stress-strain curve, which is one of the most important material parameters. From a stress-strain relationship the elastic modulus, fracture strength, and yield strength can be determined. Brittleness can be identified by the lack of deformation before fracture. Ceramics, porcelains, and glasses are brittle materials, which fracture elastically already at little elastic strain [49, 50].

1.2.3 Strength

The strength ($\sigma_f$) of a material refers to the material's ability to resist an applied force. It is defined as the maximum or critical stress that a material can endure before fracture. The incapability of ceramics, as brittle materials, to relieve tensile stresses by plastic deformation at the tip of arising cracks explains their low resistance to tensile forces compared to compressive forces. From this point of view, the tensile strength is more meaningful than the compressive strength for dental ceramics. Routinely, the
tensile fracture strength of brittle materials is measured with 3-point, 4-point or biaxial flexural tests [51-53]. In clinical dentistry, it was proved that stronger restorative ceramics including structurally uni-layered or substrate ceramics, had a higher survival rate [54-58].

For brittle materials as ceramics or porcelains, the outcome of fracture strength tests is a range of widely scattered values. According to ‘Weakest Link’ theory, fracture of a brittle material always starts at the biggest flaw in the loaded area. Due to the uncertainty of the distribution of those flaws in size, type and location in the material, the measured strength depends on the individual composition of a specimen and its flaw characteristics. To assess the strength distribution, failure probability was introduced as a statistic assessment, which is based on Weibull’s theory stating that the strength depends on the weakest element [59]. For ceramic materials, the so-called ‘strength-control’ or ‘strength-limiting flaw’ is the weakest element, capable of initiating ultimate fracture at a given stress, depending on size, shape, direction, and the location where the stress is applied. Using the relationship of probability with strength analysed with the Weibull approach, the ‘failure probability’ or ‘risk of rupture’ of a structural specimen at any applied stress may be predicted. The strength evaluation in which the impact of the tensile stress status (magnitude, distribution) is taken into account on fracture probability also considers the influence of specimen volume and surface area on the chance of a large flaw [60, 61]. For example, for specimens with same size and shape, the measured strength in a 4-point bending test might be lower than in a 3-point test. The Weibull analysis therefore offers a good risk and reliability evaluation for a material as well as a structure. The Weibull modulus characterises the distribution or variability of strength. A lower value of $m$ indicates a greater range of strength values, while a higher $m$ suggests a smaller range, thus a better reliability and safety.

1.2.4 Fracture toughness

With brittle materials fatal fracture is caused by a crack, which propagates through the material until the integrity of structure is lost. As a consequence the strength of a material may be considered as the stress required to initiate and propagate a crack to the fracture point. The driving “force” for a crack to grow is the stress intensity factor $K$ near the crack tip [60-67]. The stress intensity factor $K$ at a given stress $\sigma$ is related firmly as the following equation:

$$K = Y\sigma\sqrt{c} \text{ or } \sigma = \frac{K}{Y\sqrt{c}}$$
where $Y$ is a constant depending on the geometry and location of the crack, crack shape and loading configuration, and $c$ is the radius of the crack tip. The stress intensity factor is positively proportional to stress. When stress intensity factor rises sufficiently, a crack is propagated leading to structural failure. In tensile or mode I (mode I: tensile opening; mode II: parallel sliding; mode III: lateral tearing) loading the critical level of stress intensity factor is denoted as $K_{Ic}$, which indicates the ability of a material to resist crack propagation and its consequent catastrophic failure.

$K_{Ic}$ or the fracture toughness is a quantitative way of expressing the material resistance to brittle fracture and one of the most important material properties in fracture mechanics of brittle materials. For ceramics fracture toughness is therefore more elucidating than the strength. Strength of a brittle material depends on flaw size and its values are statistically scattered as a consequence of the flaw size distribution. Meanwhile, the fracture toughness is theoretically or generally a stable parameter independent of the crack size. If a material has a high fracture toughness it will probably undergo ductile fracture. Brittle fracture is characteristic of materials with low fracture toughness. High fracture toughness materials have improved clinical performance and reliability over low fracture toughness ones [68-70]. In dental ceramics, fracture toughness assessment may help to evaluate the suitability in long-term clinical performance of brittle materials.

1.2.5 Fatigue

Fatigue is another mode of failure, which occurs after repeated or continuous loading at stress levels smaller than the strength measured in one-load-failure tests. For dental ceramics, the term is related to cyclic loading in oral cavity [71, 72]. In applications of structural materials, this delayed failure property is most important for the durability of the structure. Fatal failure, especially for brittle ceramics may occur without a clear symptom [49, 50]. Early all-ceramic restorations failed at a high rate, but at a stress lower than material strength.

Progressive stages of fatigue damage, derived from a detailed classification are given [50] as:

a. Substructural and micro-structural changes, which cause nucleation of permanent damage.

b. The creation of microscopic cracks.

c. The growth and coalescence/fusion of microscopic crack to form a dominant crack that potentially or eventually lead to fatal failure. From a practical standpoint, this
stage is the demarcation between crack initiation and propagation.
d. Stable propagation of the dominant macroscopic cracks.
e. Structural instability or complete fracture.

With knowledge of fracture toughness and strength, it is clear that structural failure due to fatigue loading results from crack growth from initial size till critical in stress concentration areas. During this event smaller cracks may fuse or individual cracks become larger. The growing crack decreases the strength of the structure until the final loading cycle causes complete failure.

1.2.6 Crack initiation

Based on their origin, three categories of crack initiation flaws may be recognized. Intrinsic flaws are pores, agglomerates, inclusions, irregularity of grain distribution, and micro-cracks due to release of thermal residual stresses at grain boundaries and interfaces [49, 50, 71-77]. Scratches, grooves, pits, notches, fissures, usually present on or near surface, are extrinsic sources and often are damage from machining, grinding, and sandblasting. Crack initiation during service is a dynamic process, which arises from defects, which may already be micro-cracks or will lead to micro-cracks. Any defect may grow to complete fracture. Dental ceramics and porcelains are subjected to a mechanically, chemically, thermally, and tribologically rigorous environment [49, 50, 71-85].

1.2.7 Cyclic loadings and cyclic contact damage

Serious loss of ceramic strength may be caused by cracks that arise from accumulated damage in cyclic loading in aqueous environments. This mechanism will be mentioned in the part of crack growth. Cyclic loading may lead to irreversible microscopic deformations, which engenders different failure mode. Under these loading conditions, the accumulated permanent strain inevitably increases pre-present damage. Micro-cracks may nucleate at grain boundaries in single-phase materials, interphase regions in multiphase, and along interfaces between matrix and filler in brittle composites [50].

Cyclic uni-axial contact damage is well modelled in the Hertzian contact test, in which spherical indenters apply a load to a flat surface. A major advantage of controlled contact testing is that simple analytic relations can be derived for critical loads in terms of layer thickness, indenter radius, and basic material properties such as modulus, hardness, toughness, or strength [86].
Under cyclic uni-axial contact loading two basic damage modes are identified. The tensile-driven brittle mode produces single “cone” cracks that initiate from the surface; the other shear-driven quasi-plastic mode leads to distributed micro-cracks from the subsurface Hertzian “yield” zone. The former occurs mainly in brittle glasses, porcelains, and fine-grained ceramics. The quasi-plastic mode is predominant in coarse-grained tougher ceramics such as alumina- and zirconia-based ceramics. Both damage modes increase with cyclic loading; the brittle mode by slow extension of the cone crack, and the quasi-plastic mode by relatively rapid micro-crack coalescence, ultimately leading to the formation of subsurface radial cracks.

In layered all-ceramic structures, a more dangerous mode is interior radial cracking at a layer boundary, which is reported to derive from pre-existing flaws. Bi-axial cyclic contact loading modes like sliding or fretting in wear processes may cause severe surface ageing on the relatively weak veneering porcelains [87]. Uni-axial cyclic contact fatigue studies are well reported in literature [86].

1.2.8 Wear

Wear is another source of unwanted cracks and surface fracture, which may grow under occlusal force with time, while their propagation enhances mechanisms like chemical erosion. Subsequent degradation of the surface microstructure, accumulation of unwanted residual stress, irreversible strain, initiation and growth of cracks are in the process [78-84, 88-90].

Chemical or corrosive wear is caused by aggressive chemical agents like acids, bases, and enzymes, in the food, drinks, and saliva [85]. For example, acid etching and leaching can lead to dissolution of the silica network through ion exchange and Si-O bond network breakdown. This causes both surface loss and roughness with defects leading to a higher vulnerability to wear and stress. Although ceramics and porcelains are very stable materials, the well-known breakage of SiO$_2$ bonds explains the sensitivity of glass-containing ceramics and porcelains to water.

1.2.9 Thermal loading

The defects may be transformed into cracks at an additional stressing mode: repeated driven forces induced by thermal cycles in oral cavity. Changes of temperature especially sudden thermal shocks due to eating, drinking and breathing generate thermal transient stresses [28, 91-99]. Thermal shocking is often performed by moving subjects between two water containers at different temperatures. In laboratory experiments thermal shocking results in clear crack formation and/or
fracture, which is assumed to be driven by repeated thermal transient stress. The temperature range is often defined from 0°C to 60°C, very hot water for oral tissues. This may not be as severe as in other fields of engineering, yet thermal shocking is a potential weakening factor. Several investigations showed that porcelains intended for dental use are less resistant to thermo-cycling fatigue than core materials, especially polycrystalline ones like alumina and zirconia.

1.2.10 Crack growth and its involvement in lifetime predication

The first part of a fatigue process is characterized by subcritical crack growth (SCG) until a critical size is reached and unstable and fast fracture occurs. When the magnitude of SCG is known, it is possible to estimate the service life of a mechanical structure.

The crack growth of brittle materials is difficult to characterize, especially when they are subjected to cyclic loading because several processes are involved [50]:

A) Frictional sliding of the mating surfaces of microscopic cracks.
B) Progressive wear and breakage of the bridging ligaments connecting the faces of cracks and long flaws.
C) Wedging of the crack surfaces by debris such as entrapped particles.
D) Inelastic strain from shear or dilatational transformations such as mechanical twins or martensitic lamellae.

Progressive wear of ligaments, and wedging are more important under cyclic loading [50,100-102].

Although cyclic loading leads to complex processes of crack enlargement, simplified models may help to understand these. Sub-critical crack growth can be explained with a typical (K, v) curve, shown in Figure 1.1.

Region 1. in which crack motion is assumed to be controlled by stress-enhanced chemical processes at the crack tip.
Region 2. where the crack velocity does not depend strongly on the stress intensity factor (n = 0, below). Crack motion is controlled by the diffusion rate of corrosive agents at the crack tip.
Region 3. The crack velocity increases rapidly when K reaches the critical stress intensity factor, Kc. The crack motion is governed by brittle fracture mechanisms, associated with a high exponent of crack propagation.
All three regions may be described by the form [61, 103]

$$v = \frac{dc}{dt} = AK_f^n$$

where $v$ is the crack velocity, $c$ is the crack length, and $A$ is a constant. The propagation exponent $n$, is a characteristic of the resistance to mechanical fatigue. $A$ and $n$ are recognized as SCG parameters. A greater $n$ indicates a better resistance to stress-driven environment-assisted SCG.

Experimental tests may be divided into direct and indirect techniques. Direct techniques directly measure the stable subcritical crack growth speed through a specimen having a big pre-crack such as Double torsion (DT), Compact Tension (CT), and Double Cantilever Beams (DCB) [72, 104-107] and the help of fracture mechanic analysis. Indirect techniques are based on fracture strength measurements [108-110]. However, resistance to crack growth may be crack-size dependent for example with rising R-curve materials. The resistance to growth of large cracks in direct techniques may not be identical to that in small cracks.

Meanwhile indirect techniques may take advantage of the initial surface condition, which is expected to simulate the natural crack system, including flaws. Fracture strength tests in indirect techniques use a controlled applied stress $\sigma_{f0}$ (static fatigue test) or a controlled stress rate $\dot{\sigma}$ (dynamic fatigue test). The combination of these two indirect techniques is often employed for the determination of SCG parameters and for the estimation of clinical lifetime of all-ceramic prosthetic devices using.
The measurement of the fracture strength at different stressing rates allows the determination of the SCG parameters. After mathematic processing, the time to failure at a constant stress $\sigma$ becomes

$$t_f = B\sigma_{f_0}^{n-2}\sigma^{-n}$$

The development of Strength–Probability–Time (SPT) diagrams looks theoretically and practically promising in perspective of structural design, material selection, and as a tool for the improvement and development of materials and their processing.

Integrated with failure probability due to the random distribution of crack size and the consequent dispersion of strength values, SPT diagrams provide a combination of failure probabilities as a function of strength and time [108].

$$\ln \ln \left( \frac{1}{1 - P_f} \right) = \frac{m}{n-2} \ln t_f + \frac{m}{n-2} \ln \frac{\sigma^n}{B} - m \ln \sigma_0$$

$P_f$ is the failure probability. SPT diagrams provide a combination of failure probabilities as a function of strength and time [111-113].

Static fatigue lifetime may be depicted in another manner, focused on the driving force of the stress intensity using the initial value of the stress intensity factor, $K_{i_i}$.

$$t_f = \frac{2\sigma^{-2}}{AY^2(n-2)} \left( K_{i_i}^{2-n} - K_c^{2-n} \right)$$

Note the strong effect of $K_{i_c}$ on $t_f$. Taking $c = (K_i/Y\sigma)^2$, the equation illustrates the effect of the initial crack size $C_i$ on SCG and the lifetime of mechanical parts. The closer it is to the critical size $C_c$, the more rapid the crack grows to a fatal stage.

$$t_f = \frac{2\sigma^{-n}}{AY^n(n-2)} \left( C_i^{2-n} - C_c^{2-n} \right)$$

Unfortunately, the SCG knowledge of cyclic fatigue is far away from the level of static and dynamic fatigue due to its complex failure mechanism. Crack
susceptibility to $K_I$ or $\Delta K_I$ depends on size, as small cracks are more sensitive and advance below $K_{Ih}$ (threshold of $K_I$ for SCG) found for large-crack specimens. Assuming equality of the subcritical crack enhance rate or process under different loading modes, [114] gave a formulation of the number of cycles to failure $N_f$ with substitution of the applied stress $\sigma$ of the static test with an alternating stress $\Delta \sigma$ as the subtraction of the minimum stress $\sigma_{min}$ from the maximum stress $\sigma_{max}$.

$$N_f = \frac{2(\Delta \sigma)^n}{AY''(n-2)} \left( \frac{C_i^{2-n}}{C_c^{2-n}} \right)$$

Another modified numeric estimation of cyclic fatigue lifetime and SCG was developed by Munz and Fret [115],

$$\nu = A(\Delta K)^n = A(1 - R)^n K_i^{n_{\text{max}}} = A^* K_i^{n_{\text{max}}}$$

where $\Delta K_i$ is the amplitude of the applied stress intensity factor ($K_{i\text{max}} - K_{i\text{min}}$), $R$ is the applied stress ratio $K_{i\text{min}}/K_{i\text{max}}$, and $n$ and $A$ (and $A^*$) are the subcritical crack growth parameters for cyclic fatigue.

$$n = \frac{m_{f0}}{m} + 2,$$

$$A = \frac{2K_i^{2-n} \sigma_{f0,0}^{n-2}}{N_{f0} Y''(n-2)(\Delta \sigma)^n} \text{ and } A^* = \frac{2K_i^{2-n} \sigma_{f0,0}^{n-2}}{N_{f0} Y''(n-2)(\sigma_{\text{max}})^n}$$

where $m^*$ and $N_{f0}$ are the Weibull modulus and the characteristic number of cycles to failure from the lifetime distribution, $m_{f0}$ and $\sigma_{f0,0}$ are the Weibull modulus and characteristic strength of the inert strength $\sigma_{f0}$, which are based on

$$\ln \left( \frac{1}{1-P_f} \right) = m \ln \sigma_{f0} - m \ln \sigma_{f0,0}$$

and

$$\ln \left( \frac{1}{1-P_f} \right) = m^* \ln N_f - m^* \ln N_{f0}$$

The numbers of cycles leading to failure at a $\sigma_{\text{max}}$ of about half of $\sigma_{f0}$, are adopted for the Weibull analysis. The prediction for the number of cycles then becomes
$N_f = \frac{2K_{ic}^{2-n} \sigma_{f0}^{n-2}}{A^2 Y^2 (n-2) \sigma_{max}^n}$

or $N_f = B^* \sigma_{f0}^{n-2} (\sigma_{max})^n$, with $B^*$ based on $A^*$, which looks similar to the static fatigue lifetime formulation $t_f = B \sigma_{f0}^{n-2} \sigma^{-n}$, with $N_{f,0}$ merged into $B^*$.

This method employs the SCG data obtained from cyclic loading rather than static and dynamic fatigue tests, which might be a better representation of cyclic SCG, and has been reported in the evaluation of dental ceramics [116-119].

Stress-assisted and corrosion-driven crack growth can be characterized with the knowledge of SCG as described above. By applying engineering tools as finite element analysis and with the help of those formulations, the SPT relation could be established provided that all the necessary basic and SCG properties are available. Fatigue behaviour may also be calculated with the relevant software like CARES/LIFE (Ceramic Analysis and Reliability Evaluation of Structure Life Prediction) (NASA Lewis Research Center, Cleveland, OH, USA) [109, 120, 121], which also accounts for the phenomenon of subcritical crack growth by utilizing the power law [115].

Nevertheless, in dental ceramics, SCG parameters are mainly assessed with dynamic fatigue test and lifetime predictions based on static fatigue theories. Although the parameters are based on experimental data, these results rely on theoretical calculations and the assumption of similarity of SCG behaviour under distinctly different loading conditions. In practice such fatigue estimations seem inadequate for complex conditions like the oral cavity [49, 50, 104-106, 122], despite that several scientific publications [109, 123] confirmed the consistence of SCG parameters from one method or experimental test with other methods and clinical failure trends.

### 1.2.11 S-N curve for strength degradation test

In other experimental fatigue approaches the applied stress, or the residual strength is plotted to the loading time in-service time or number of cycles to failure, resulting in S-N (Stress-Number of cycles) diagrams, which expose initially “natural” surfaces to controlled stresses [50, 72, 124-126]. There are two definitions of “time” which could be introduced in stress fatigue in prosthodontics: total life and half life or endurance limit. Total life is the average number of cycles until failure, while half-life is defined as the number of cycles where 50% of the specimens have failed. The technical problem is that the total-life S-N curve approach may be rather time-consuming. To mitigate this, specimens are often polished or roughened to control the surface profile [108-111, 113, 116-119], in which relatively large surface flaws are
preferred or introduced to get a narrower range of degradation strength or time/cyclic numbers.

Half-life can be assessed with the staircase test design, which is a straightforward approach for endurance limit in which the load for each successive specimen is slightly increased or decreased, depending on success or failure of the previous specimen [72].

Normally in the biomaterial industry, the processing of products like artificial heart valves and hip joints is standardized, the microstructure of the used materials is well controlled and proof testing is routine practice [50]. Characteristic of the custom made processing in dentistry is the individual shape of the structures and that proof testing is not feasible because it is hardly possible to make more than one identical restoration.

1.2.13 Complications in lifetime prediction

Service time prediction (STP) is an achievement in materials engineering, which may be applied in prosthetic dentistry too for the prediction of longevity of prosthetic restorations. Obviously fatigue is a process of time-dependent evolution of damage or damage accumulation. Besides functional loading, restorations in the oral cavity have to function in a wet environment with wear, contact and impact damage, thermal residual stresses due to ceramic and porcelain sintering, and repeated thermal transient stresses from temperature changes. It is hard to couple all factors and failure modes in the acquirement of data of strength degradation as well as lifetime prediction and experimental design. At present, this is an impossible mission.

It can be concluded that masticatory complex loading modes, damaging modes, flaw size and distribution, SCG parameters of small cracks and clinical failure modes should be well investigated and known before a useful prediction of the durability of a dental prosthetic restoration is possible. The study presented in this thesis aims to contribute to this knowledge.

Chapter 2 is a study into the influence of test methods on fracture toughness of a dental porcelain and a soda lime glass. While in chapter 3 three test methods to determine fracture toughness are compared. In chapter 4 the indentation strength in bending method is further evaluated with dental porcelains. Chapter 5 deals with the role of the indentation load at smaller flaw sizes in the indentation-strength fracture toughness. In chapter 6 the strength influencing variables on CAD/CAM zirconia frameworks and in chapter 7 the staircase strength degradation of sandblasted zirconia due to cyclic fatigue are investigated further.
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Chapter 1


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