Mechanical Properties of Ceramics

or

Mechanical Behavior of Brittle Materials

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What you already know and understand!

• Ceramic materials are susceptible to thermal shock, they fail if exposed to too fast temperature changes ($\Delta T/\Delta t$) and locally to too large temperature gradients ($\Delta T/\Delta x$).

• Small component = small $\sigma_{\text{thermal}}$ // Large component = large $\sigma_{\text{thermal}}$

• Low CTE, high $K_{\text{lc}}$, and low $E$ = high $R_S$ (= 1st thermal shock parameter)
  2nd thermal shock parameter → constant heat transfer
  3rd thermal shock parameter → surface heated by constant rate

• Slow Crack Growth (scg): time dependent failure → limited life time

• Crack velocity is influence by humidity → brake up of bonds at crack tip
  e.g. in soda-lime glass by water (Si-O-Si- +H2O → -SiOH + -SiOH).

• SCG parameters can be measured in accordance to state of load (static, dynamic, cyclic, or a combination thereof).

• Correlation between largest failure relevant defect, failure strength and lifetime.

• Link between strength, probability of failure and lifetime: Strength-Probability-Time diagram
Repetition learning targets part 4

• **Creep**: Boundary (diffusion) or lattice (dislocation) mechanism

• **Diffusional creep**:  
  Free surfaces and grain boundaries work as source and assembly point for voids and atoms. Voids diffuse from surfaces under tension to surfaces under compression and matter flows in reverse direction.

• **Grain boundary sliding**:  
  Structure elongates by shifting & twisting of grains

• **Stages of creep**:  
  - primary  
  - secondary (steady state)  
  - tertiary

• Creep should be measured in tension and not in bending.

• **Stain rate** is increasing with  
  - increasing load & temperature and  
  - decreasing grain size.
Aim of chapter & Learning targets

1. Introduction
   “Why mechanical testing …”
2. Stresses at a crack tip
   “Higher than you’d assume …”
3. Griffith law
   “Conditions for failure …”
4. \( K_I \) and \( K_{IC} \)
   “Stress intensity & critical stress intensity …”
5. R-curve
   “Improving toughness …”
6. Properties
   “Knowing what you measure …”
7. Strength
   “Just a value …”
8. Statistic
   “Weibull, a name you’ll never should forget …”
9. Proof testing
   “Make it or …”
10. Fractography
    “Reading fracture surfaces …”
11. Thermal shock
    “Temperature, time and geometry …”
12. Slow crack growth
    “After several years …”
13. SPT diagrams
    “Combining strength, lifetime & statistics …”
14. Creep
    “Temperature makes it move …”
15. Failure maps
    “Finding your way …”

part 5 - Case Study: Lifetime of All-Ceramic Dental Bridges
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Outline

• Introduction
  - Dental bridges in time
  - Why ceramics
  - Mechanical short & longtime properties
  - Aim & Objectives of study

• Production process, various

• Experimental & Results
  - Mechanical short-time properties
  - Lifetime prediction
    - influence of water
    - subcritical crack growth & fatigue

• Lifetime diagrams

• Summary, Conclusion & Outlook
Introduction

Dental bridges in time

Etruscan prosthesis  
200 BC

Metal-porcelain bridge  
1960s

All-ceramic bridge  
1999

Filser, PhD thesis, ETHZ
Introduction

Chillitory

- Low allergenic potential
- Pink esthetic of gums
  - Biocompatibility
  - Low deposition of plaque
- White esthetic of tooth
  - Natural look
  - Imitation of nature
  - Translucent

Why ceramics?

Metallic framework

Ceramic framework

5-unit Bridge in the Molar Region after 3 years in service (2002)

(Courtesy of A. Fehérand I. Sailer, University of Zurich)
High-strength materials are required!

The “failure load” was set to 800 N, 900 N or 250 N / teeth depending on the time in the long-time research project a specific part has been conducted.
Introduction

Mechanical strength of ceramics and metals

Strength of traditional ceramics had to be improved!
Fatigue and subcritical crack growth

Mastication: Cyclic stresses + Water

Fatigue mechanisms decrease the strength over time!
Introduction

New materials

Toughness, $K_{IC}$ (MPa m$^{1/2}$)

Strength, $\sigma_c$ (MPa)

Advanced ceramics

Glass-infiltrated porous aluminas

Glass-ceramics & reinforced porcelains

Conventional porcelains

MK II

Dicor MGC

Li$_2$O.2SiO$_2$ (Empress 2)

Al$_2$O$_3$-Glass (Inceram-Alumina)

Al$_2$O$_3$-ZrO$_2$-Glass (Inceram-Zirconia)

ZrO$_2$ (Y,Mg-PSZ)

Nano-ZrO$_2$ (Y-TZP)

Al$_2$O$_3$

Omega

Empress 1

Empress 2

Conventional porcelains

New materials

Introduction
Aim & Objectives

Systematically evaluate the lifetime under cyclic conditions for selected, representative dental ceramics.

With variables effect of water and effect of coating (porcelain)

Establish guidelines for materials selection, fabrication process and bridge design

Framework:
- Inceram Zirconia (VITA)
  \[ \text{Al}_2\text{O}_3-\text{ZrO}_2-\text{Glass} \]

Framework:
- Zirconia (Cercon, deegudent)
  \[ \text{Nano-ZrO}_2 (Y-TZP) \]

Framework:
- LiSilicate (Ivoclar, Empress II)
  \[ \text{Li}_2\text{O} \cdot 2\text{SiO}_2 \]

Veneer:
- Glass A

Veneer:
- Glass Z

Veneer:
- Apatite

Kübler Empa-HPC, ETHZ MW-II Ceramics-6.5, 2010
Pre-operative view shows the presence of extensive infiltrated composites.

Teeth are prepared.

The four waxed copings are sprued and then invested.

The frameworks after being pressed in the pressing furnace.

View of the finished frameworks on the master model.

The crowns and veneer are again placed on the master model.
Introduction

**Empress-2 Process**

Facial view of the four restorations.

Labial view of the four restorations.

Facial view of the four restorations following immediate operative placement.

*IvoclarVivadent: “i, Vol. 7, Nr. 1, 2005*

Kübler Empa-HPC, ETHZ MW-II Ceramics-6.5, 2010
Introduction

InCeram-Zirconia (Alumina)

Process

- model made of gypsum
- digitizing
- grinding
- crown caps after grinding
- crown caps fitted on the gypsum model
- glass infiltration

A. David, SpectrumInternational • IDS 2003, p. 5-7
Introduction

**InCeram-Zirconia (Alumina) Process**

- infiltrating the porous alumina
- overworking the excess infiltration glass
- veneering the framework with VITADUR® ALPHA
- situation after cementation of the 3-unit bridge

A. David, SpectrumInternational •IDS 2003, p. 5-7
Introduction

Process of Machining in the Soft Sintered State

Cercon® (Degudent) system

Load Bearing Experimental Setup

e.g. 3-unit bridges

- Load
- Teflon disk
- Framework (dental bridge)
- Steel post
- Elastic rubber hose
- Die holder

adapted from
H. Lüthy et al., Dental Materials, 21 (2005) p930
Strength

Load Bearing Capacity and Reliability of 4-Unit Frameworks

H. Lüthy et al., Dental Materials, 21 (2005) p930
Mean Load Bearing Capacity of 3- and 4-unit Frameworks

\[ F = \frac{\sigma \cdot l}{l \cdot e} \]
First conclusions

• Cercon Zirconia is the superior material
• in-vitro load bearing capacity and reliability of Cercon Zirconia 3-unit bridges are superior
• 7 mm² connector area is sufficient for 3-unit Cercon bridges
• 7 mm² connector area is NOT sufficient for Cercon 4-unit bridges
• 4-unit bridges need larger connector areas, or less span width or stronger materials

And what about lifetime?
Subcritical crack growth

How fast does the crack propagate under subcritical conditions?

$$\sigma_c = \frac{K_{IC}}{\gamma \sqrt{a_c}}$$

Griffith's law

Flaw size (μm)

Stress applied

Inert strength

Failure!

Kübler Empa-HPC, ETHZ MW-II Ceramics-6.5, 2010
Theory

Lifetime prediction

\[ v = f(K_i) \]

\[ \sigma_c = \frac{K_{IC}}{\gamma \sqrt{a_c}} \]  
Griffith's law

\[ \sigma_c = \sigma \sqrt{\frac{1}{K_{IC}}} \]

\[ t_f = \left( \frac{2}{AY^2(n-2)K_{IC}^{2-n}} \right) \sigma_c^{n-2} \sigma^{-n} = B \sigma_c^{n-2} \sigma^{-n} \]

For constant stress \( \sigma \)!

De Aza et al., Biomaterials 23, 937 (2002)
Theory
Lifetime

\[ \nu = f(K_I) \] ?

\[ \sigma_c = \frac{K_{IC}}{Y \sqrt{a_c}} \] Griffith's law

\[ \sigma_c = \frac{K_{IC}}{Y \sqrt{a_c}} \] Griffith's law

\[ \frac{1}{h(n, \sigma_m/\sigma_a)} B \sigma_c^{n-2} \sigma_a^{-n} \] Any given stress variation

\[ t_f = B \sigma_c^{n-2} \sigma_a^{-n} \] Constant stress \( \sigma \)

\( \nu = A K_I^n \) Empirical relation

Inert strength

failure (= fracture)

Stress applied

Time (hours)

Stress (MPa)

De Aza et al., Biomaterials 23, 937 (2002)
Theory Lifetime

**Scheme of the ageing process**

a) Nucleation on a particular grain at the surface, leading to microcracking and stresses to the neighbours.
b) Growth of the transformed zone, leading to extensive microcracking and surface roughening.
c) Grain pull-out induced by wear.

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water induced

\[ t \rightarrow m \]

transformation

(degradation of strength)

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\( \sigma \)

Monoclinic

Tetragonal
Experimental Approach

Sample preparation

Set-up fatigue machine

Strength and lifetime measurements

Subcritical crack growth curves

Stresses on posterior bridges

Lifetime diagrams

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Sample preparation

**Experimental Framework**

- **Framework**
- **Framework + veneer**
- **Apatite (veneer)**
- \( \text{Li}_2\text{O}.2\text{SiO}_2 \) (framework)

- inert strength + lifetime in water
- coated + uncoated
- 3 materials + TZP rods

\[ = \]

240 samples (effective 232)

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Detection of sample breakage

Experimental

Aqueous environment
Experimental

- **σ_{\text{max}}**
- Peak stress for accelerated fatigue tests

- **Maximum Stress (MPa)**
- **Time (min)**

- **Failure probability (%)**

- Mechanical strength
- Fatigue

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Results

Determination of SCG behavior e.g. TZP

Inert strength (MPa)

Lifetime (min)

\[ \ln(1/(1-F)) \]

\[ \ln(\sigma_c) \]

\[ \ln(Lifetime) \]

\[ \ln(\ln(Lifetime)) \]

\[ \Delta K / K_{IC} \]

\[ v \]

\[ n = 79.8 \]

\[ n = 66.5 \]

Describes the properties of the material!
Results

Lifetime prediction of dental materials

Materials' properties

20 years with 1’400 cycles/day

Predict lifetime for any bridge!
Results

Fracture mode


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Results

Fatigue and subcritical crack growth

Veneer

\[ 10\text{Ca}^{2+} + 6\text{PO}_4^{3-} + 2\text{OH}^- \]

\[ \uparrow \]

\[ \text{Ca}_{10} (\text{PO}_4)_6(\text{OH})_2 \]

Enamel: Apatite

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Results

Fatigue and subcritical crack growth

Framework

![Graph showing crack velocity vs. stress intensity factor for different materials](image)

- Human dentin (Nalla et al., 2003)
- Enamel: Apatite
- Dentin: ~75% Apatite + 25% collagen
- Li₂O.2SiO₂
- Al₂O₃-ZrO₂-Glass
- Nano-ZrO₂

Stress intensity factor, $K_{I,max}$ (MPa.m$^{1/2}$)
Results

**Lifetime diagram**

20 years with 1,400 cycles/day and \( \text{Load}_{\text{max}} = 130 \text{ N} \)

![Graph showing lifetime diagram with stress and probability of failure](image)

![Graph showing framework strength and stress](image)

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Results

Lifetime diagram: Veneer

Glass A

Glass Z

Apatite

1400 cycles/day

Load_{max} = 130 N

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Results

Lifetime diagram: Framework

Al₂O₃-ZrO₂-Glass

Nano-ZrO₂

Li₂O.2SiO₂

1400 cycles/day

Load_{max} = 130 N
Summary

Framework:

Al$_2$O$_3$-ZrO$_2$-Glass

Veneer: Glass A

1400 cycles/day
Load$_{max} = 130$ N

Framework:

Nano-ZrO$_2$

Veneer: Glass Z

Max Ø (≥ 4 mm)

Framework:

Li$_2$O.2SiO$_2$

Veneer: Apatite

Max Ø (≥ 4 mm)

Veneer and not framework dictates what’s feasible .......

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Courtesy: University of Zürich, Center for Dental & Oral Medicine
Conclusions

Nano-ZrO₂ ceramics and ZrO₂-based ceramic composites can withstand the high stresses applied on posterior bridges during mastication.

Long-term failure due to fatigue and subcritical crack growth can be avoided through proper bridge design.

Lifetime diagrams are suitable tools for the selection of new materials and fabrication technologies for dental restorations.

Apatite-based materials should be avoided in the connector region of posterior bridges due to the pronounced subcritical crack growth.

Ca₁₀(PO₄)₆(OH)₂

\[ 10Ca^{2+} + 6PO_4^{3-} + 2OH^- \]
Outlook

Etruscan prosthesis | Metal-porcelain bridge | All-ceramic bridge

200 BC | 1960s | 2005

Ceramic high strength, non veneer bridge? | Natural tooth genesis mediated by stem cells?

20yy | 2zzz

different materials - strength not anymore the issue - treatment methods shifting -

- wishful
- 1st steps successful
- lots of challenges ahead
Summary:
What you know and understand, now!

✔ Hook’s law …
✔ Stress …
✔ Griffith’s law …  ★
✔ Weibull statistic … ★
✔ Influence of surface, volume, microstructure … ★
✔ R-curve …
✔ Environment …
✔ Sub-critical crack growth …
✔ Static & dynamic fatigue …
✔ Proof-testing …
✔ Deformation & failure @ elevated temperatures …
✔ Thermal shock …

★ Those relations, laws & equations you known by heart, now!