Mechanical Properties of Ceramics
or
Mechanical Behavior of Brittle Materials

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Repetition learning targets part 2

What you already know and understand!

- Fracture toughness can be enhanced by increasing energy required to extend crack.
- Ceramics with R-curve behavior:
  - degradation in strength with increasing flaw size is less severe
  - reliability increases
- Crack deflection, crack bridging, martensitic transformation are mechanisms that enhance $K_{IC_{app}}$.
- Fracture toughness values measured with different test methods may differ.
- Bend test:
  - universal (e.g. strength, fracture toughness)
  - sensitive to surface defects
  - only small volume tested
  - value $\sigma_{3Pt}$ test $>$ value $\sigma_{4PT}$ test
  - specimen sees stress gradient
Repetition learning targets part 2

- All components have **defects** due to fabrication and usage. They have a size from a few μm up to a few 100 μm

- **Strength of a component** is defined by a combination of
  - critical stress intensity factor
  - size of critical defect
  - position of critical defect
  - stress and stress direction the crack sees

- Ceramic materials **fail without warning** even at elevated temperatures $K_{Ic}$ is between 1 MPa $\sqrt{m}$ and 20 MPa $\sqrt{m}$

- The aim is always to **improve** both
  - $\sigma_c$, $\rho_c$, e.g. by improved processing
  - $K_{Ic}$ by increasing fracture energy, e.g. crack bridging

- Strength of ceramics must be described by **statistics** as identical components will not fail at one reproducible strength value.

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Aim of chapter & Learning targets

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| Part 5 - Case Study: Lifetime of All-Ceramic Dental Bridges |

Kübler Enpa HPC, ETHZ MW-II Ceramics 6.3, 2010
Waloddi Weibull: 1887-1979

Swedish engineer famous for his pioneering work on reliability, providing a statistical treatment of:
- fatigue,
- strength and
- lifetime in engineering design.

*The widely-usable, reliable and user-friendly Weibull distribution is named after him.*

Weibull statistic (2)

**Model of the chain with the weakest link (1)**

- a chain is only as strong as its weakest link
- if the strength of the links is distributed evenly then the probability of survival of a chain with length $L$ is defined as:

$$P_s(L) < 1$$

- then the probability of survival of a chain of twice the length ($2L$) is

$$P_s(2L) = P_s(L) \cdot P_s(L) \quad (not +)$$

- because both halves of the chain must survive

$$P_s(2L) < P_s(L)$$
Weibull statistic (3)

Model of the chain with the weakest link (2)

assume probability \( P_F \) of length \( L_0 \) failing at \( \sigma \) is \( R(\sigma) \)

\[ \Rightarrow \quad P_S \text{ of length } L_0 \text{ surviving at } \sigma \text{ is } 1 - R(\sigma) \]

\( P_F \) of length \( \Delta L \) failing at \( \sigma \) is \( R(\sigma) \cdot (\Delta L/L_0) \)

\[ \Rightarrow \quad P_S \text{ of length } \Delta L \text{ surviving at } \sigma \text{ is } 1 - R(\sigma) \cdot (\Delta L/L_0) \]

and \( P_S \) of length \( (L_0 + \Delta L) \) surviving at \( \sigma \) is

\[ P_S(L_0 + \Delta L) = P_S(L_0) \cdot \left[ 1 - R(\sigma) \cdot \frac{\Delta L}{L_0} \right] \]

Weibull statistic (4)

Model of the chain with the weakest link (3)

3-D-analogy

\[ \Delta V \rightarrow dV \]

now: \( dV \rightarrow dV \)

\[ \frac{dP_s(V)}{dV} = -\frac{R(\sigma) \cdot P_s(V)}{V_0} \]

and integrating:

\[ \int \frac{dP_s(V)}{P_s(V)} = -\frac{R(\sigma) \cdot V}{V_0} \]

\[ P_s = \exp\left(-\frac{R(\sigma) \cdot V}{V_0}\right) + C \]

because \( P_s = 1 \) only for \( V = 0 \)

\[ \Rightarrow C = 0 \]
Weibull statistic (5)

if stress varies from place to place

\[ P_s = \exp\left(-\int \frac{R(\sigma)}{V_0} dV\right) \]

\( R(\sigma) = \left(\frac{\sigma - \sigma_c}{\sigma_0}\right)^m \)

\[ m = \text{Weibull modulus} \]
\[ \sigma_0 = \text{characteristic (stress) strength} \]
\[ \sigma_c = \text{stress below which no failure occurs} \]
\[ \sigma = \text{stress at failure} \]

Weibull proposed simple (parametric) solution for probability of failure \( R(\sigma) \):

now it’s possible to calculate \( P_s \) for a component under a mechanical stress:

\[ P_s = \exp\left(-\int \frac{R(\sigma)}{V_0} dV\right) \]

\[ P_s = \exp\left(-\int \left(\frac{\sigma - \sigma_c}{\sigma_0}\right)^m \frac{dV}{V_0}\right) \]

and if whole component stays under the same stress the equation reduces to:

\[ P_s = \exp\left(-\left(\frac{\sigma - \sigma_c}{\sigma_0}\right)^m \frac{V}{V_0}\right) \]
Weibull statistic (7)

and probability of failure: 

\[ P_f = 1 - P_s = 1 - \exp\left(-\left(\frac{\sigma - \sigma_c}{\sigma_o}\right)^m \frac{V}{V_0}\right) \]

if

- stress below which no failure occurs is neglected \( \Rightarrow \sigma_c = 0 \) MPa
- volume is normalized e.g. in standardised test bar \( \Rightarrow V = V_0 \)

\[ P_f = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_o}\right)^m\right) \]

this purely mathematical description doesn’t have at this point a material scientific meaning (… Weibull proposed simple function which fits …)

but \( \sigma_0 \), \( m \) and \( \sigma \)

must somehow correlate with the density of the defects

---

Weibull statistic (8)

\[ P_f = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_o}\right)^m\right) \]

Weibull module \( m \) describes form of failure probability curve

\( m = 0 \)

\( \Rightarrow P_f \) independent of applied stress

\( m = 1 \)

\( \Rightarrow P_f \) exponential asymptotic curve

\( m = \infty \)

\( \Rightarrow P_f \) “step curve”

- with \( P_f = 0 \) if \( \sigma < \sigma_0 \)
- with \( P_f = 1 \) if \( \sigma > \sigma_0 \)
Weibull statistic (9)

- **large m:**
  narrow distribution, small spread
  → reliable material
  
  "tough" ceramic components:
  \( m = 10-40 \)

- **small m:**
  wide distribution, large spread
  → unreliable material
  
  "bad" ceramic components:
  \( m = 1-10 \)

Weibull statistic (10)

**Calculation of \( m \) und \( \sigma_0 \)**

with defined volume, e.g. test bar:

\[
P_f = 1 - \exp \left( -\left( \frac{\sigma}{\sigma_0} \right)^m \right)
\]

rearrange and take twice the logarithm:

\[
\ln \left( \ln \left( \frac{1}{1-P_f} \right) \right) = m \cdot \ln \sigma - m \cdot \ln \sigma_0
\]

\[
y = m \cdot x + C
\]

make a graph with the left term on the y-axis and the right term as x-axis and insert measured values:

- slope of straight line → \( m \)
- \( \ln (\ln(1/(1-P_i))) = 0 \) → \( \sigma_0 \)

\[
\sigma_0 = 0.632
\]
Weibull statistic (11)

**Determination of Weibull-parameter**

- conduct measurements
- classify failures (>30 per class)
- rank results
- assign relative frequency

\[ P = \frac{n - 0.5}{N} \quad \text{or} \quad \frac{n}{N + 1} \]

- draw Weibull-diagram
- calculate \( \sigma_0 \) and \( m \)
- calculate confidence intervals

![Weibull-diagram](image1)

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**Example: ground glass rods**

<table>
<thead>
<tr>
<th>Test No.</th>
<th>Failure strength (MPa)</th>
<th>Rank</th>
<th>Failure strength (MPa)</th>
<th>Failure probability (FP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>178</td>
<td>1</td>
<td>178</td>
<td>0.1</td>
</tr>
<tr>
<td>2</td>
<td>276</td>
<td>2</td>
<td>210</td>
<td>0.2</td>
</tr>
<tr>
<td>3</td>
<td>262</td>
<td>3</td>
<td>235</td>
<td>0.3</td>
</tr>
<tr>
<td>4</td>
<td>296</td>
<td>4</td>
<td>248</td>
<td>0.4</td>
</tr>
<tr>
<td>5</td>
<td>210</td>
<td>5</td>
<td>262</td>
<td>0.5</td>
</tr>
<tr>
<td>6</td>
<td>248</td>
<td>6</td>
<td>276</td>
<td>0.6</td>
</tr>
<tr>
<td>7</td>
<td>235</td>
<td>7</td>
<td>296</td>
<td>0.7</td>
</tr>
<tr>
<td>8</td>
<td>318</td>
<td>8</td>
<td>318</td>
<td>0.8</td>
</tr>
<tr>
<td>9</td>
<td>345</td>
<td>9</td>
<td>345</td>
<td>0.9</td>
</tr>
</tbody>
</table>

\[ \sigma_0 = \exp \left( \frac{C}{m} \right) = 285 \text{ MPa} \]

\[ P = \frac{n - 0.5}{N + 1} \]
Weibull statistic (13)

**Confidence Interval**

Interval for which it can be stated with a given confidence level that it contains at least a specified portion of the population of results (= measure of uncertainty of parameters).

"Cook book"

- determine required confidence level, $1 - \alpha$
  (common practice: 90 % → $\alpha = 0.1$)
- for a given number of test-pieces $N$
  → upper confidence interval limit factor $t_u \, @ \, \alpha/2$
  → lower confidence interval limit factor $t_l \, @ \, (1 - \alpha/2)$
- $t_u$ and $t_l$ are determined from tables (e.g. EN 843-5)
- upper & lower values of $\hat{\sigma}_0$:

  upper limits of confidence interval: $C_u = \hat{\sigma}_0 \exp\left(\frac{t_u}{m}\right)$

  lower limits of confidence interval: $C_l = \hat{\sigma}_0 \exp\left(\frac{t_l}{m}\right)$

“Cook book” for confidence interval for $m$ is identical

Weibull statistic (14)

- typical value: $m = 10$
- probability of failure for components can be calculated without knowing defect density
- weakest components (or sample) determine widely the slope of Weibull line
- statistically relevant Weibull parameters require $\geq 30$ experimentally measured values

- used next to the "naturally" present defects (mainly volume) a 2nd defect population (surface) leads to lower failure stresses
Proof testing (1)

... assuring that no component fails while in use ...

- Proof test stress lower than the design stress and higher than the expected stress in use is applied to components
- This will eliminate "bad" components (samples)
- The lower end of the distribution is therefore cut off and the new distribution isn't a proper Weibull distribution anymore

Components should be used after proof testing @ $\sigma < \sigma_p$

It is possible to calculate the failure rate if $\sigma < \sigma_p$ but there is %-wise only a small improvement in failure.

Proof testing (2)

<table>
<thead>
<tr>
<th>Rank</th>
<th>Failure strength (MPa)</th>
<th>Probability of failure $P_f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1178</td>
<td>0.4</td>
</tr>
<tr>
<td>2</td>
<td>2410</td>
<td>0.2</td>
</tr>
<tr>
<td>3</td>
<td>2350</td>
<td>0.3</td>
</tr>
<tr>
<td>4</td>
<td>2480</td>
<td>0.4</td>
</tr>
<tr>
<td>5</td>
<td>2620</td>
<td>0.5</td>
</tr>
<tr>
<td>6</td>
<td>2760</td>
<td>0.6</td>
</tr>
<tr>
<td>7</td>
<td>2960</td>
<td>0.7</td>
</tr>
<tr>
<td>8</td>
<td>3180</td>
<td>0.8</td>
</tr>
<tr>
<td>9</td>
<td>3450</td>
<td>0.9</td>
</tr>
</tbody>
</table>

Proof stress 240 MPa

$G(\sigma_c)$ is not a Weibull distribution.
Proof testing (3)

Distribution of stress for $F$ and $G$ after proof testing.

What does it mean?

\[
F(\sigma_c) = 1 - \exp \left( - \left( \frac{\sigma_c}{\sigma_p} \right)^m \right)
\]

\[
G(\sigma_c) = 1 - \exp \left( - \left( \frac{\sigma_c}{\sigma_p} \right)^m + \left( \frac{\sigma_p}{\sigma_c} \right)^m \right)
\]

Influence of volume ...

Weibull statistic (15)

\[
P_{15} = \exp \left( - \left( \frac{\sigma_1}{\sigma_0} \right)^m \frac{V_2}{V_0} \right) \quad \text{and} \quad P_{25} = \exp \left( - \left( \frac{\sigma_2}{\sigma_0} \right)^m \frac{V_2}{V_0} \right)
\]

... the influence of volume $V_2$ for a stress $\sigma_2$ can be calculated from volume $V_1$ and stress $\sigma_1$ if $m$ is know:

\[
\frac{\sigma_1}{\sigma_2} = \left( \frac{V_2}{V_1} \right)^{\frac{1}{m}}
\]

example: 3 x 4 x 45 mm bend bar:

- 3-pt BT $V_2 \approx 1 \text{ mm}^3$ → $1.27 \times \sigma_1$
- 4-pt BT - 40 / 20 mm $V_1 \approx 11 \text{ mm}^3$ → $\sigma_1$
- tensile - 3x4x20 mm $V_3 \approx 240 \text{ mm}^3$ → $0.73 \times \sigma_1$
Influence of tested volume or surface

![Weibull statistic diagram](Image)

<table>
<thead>
<tr>
<th>Question (reason)</th>
<th>Answer for ... (application)</th>
</tr>
</thead>
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<tr>
<td>Where did it break from?</td>
<td>Engineering</td>
</tr>
<tr>
<td>Did it crack suddenly or slowly?</td>
<td>Engineering</td>
</tr>
<tr>
<td>Why did it break from here?</td>
<td>QA, process monitoring</td>
</tr>
<tr>
<td>Nature of fracture source?</td>
<td>Material development, QA</td>
</tr>
<tr>
<td>Stress at fracture?</td>
<td>Design</td>
</tr>
<tr>
<td>Environment or fatigue?</td>
<td>Engineering</td>
</tr>
<tr>
<td>Good test?</td>
<td>Material evaluation</td>
</tr>
<tr>
<td>Whose fault?</td>
<td>Commercial, legal</td>
</tr>
</tbody>
</table>

Fractography (1) ... why?

“Improvement through feedback.”
(... cause of failure)

... skill seldom taught academically
– poor ability to interpret reasons for failure
– leads to negative impression of value of ceramic components (liability !)
– leads to wrong conclusions concerning causes of failure (materials versus manner of use/abuse)
Fractography (2)

roman temple rebuild after earth quake

shell like chip

eample glass

Fractography (3)

… old but not well known science

First mention of ceramic fractures by E. Bourry in: A treatise on ceramic industries (first English edition 1901)

“… observation of the structure or homogeneity should consist of the examination of a fracture, either by the naked eye or by a magnifying glass.”

“… it will be advisable to note:
(a) appearance of the fracture, whether granulated, rough or smooth, or with a conchoidal surface.
(b) size of the grains…….
(c) homogeneity…, whether there are any planes of cleavage or scaling, and whether these are numerous and pronounced”

Guide for hobby astronomer ...

• get familiar with the firmament simply by the naked eye and a map ..
• observe satellites and stars with a simple field glass ..
• locate and enjoy details of far away stars and galaxies with a telescope ..
Fractography (4)

Stage 1:
- Objective: Location of origin
- Action: Collect and clean fragments
- Deduction: History of fracture
- Result: Visual inspection

Stage 2:
- Objective: Tentative classification of origin
- Action: SEM inspection
- Deduction: Identify features and locate origin
- Result: Binocular macroscope inspection

Stage 3:
- Objective: Mechanical nature of origin
- Action: SEM inspection
- Deduction: Mechanical circumstances of fracture
- Result: EDX analysis

Flow diagram

Fractography (5)

Fracture patterns in four-point flexural strength test pieces

- **Medium stored energy test piece**
  - Primary fracture in centre with compression curl; secondary fractures caused by impact between test piece and jig parts
- **High stored energy fracture**
  - With multiple cracking near the origin; cracks bifurcate shortly after initiation; fracture origin may be lost in fragmentation
- **Low to medium stored energy fracture**
  - Primary failure close to loading rod; secondary break due to impact with jig parts
  - Four-point bend test piece, tensile face on lower side
- **“low energy”**
  - e.g. porcelain
- **“medium energy”**
  - e.g. fine grained alumina
- **“high energy”**
  - e.g. silicon nitride
- **s**: secondary failure
  - Often due to shock wave
  - 5% increased load @ roller
  - "long" fracture piece hits jig
Fractography (6)

Fracture patterns in ring-on-ring test pieces

1 likely origin zone
2 primary crack

Fractography (7)

Macro-features in flexural test bars

Origin inside body
Origin at or close to surface

Ridge and compressive curl
Hackle
Mist (when visible)
Mirror
Origin

Origin inside, but to one side
Fractography (8)

**Microscopic: ‘fracture lines’ – fine hackle**

Features near fracture origins

- Fracture lines from an extended origin such as a machining flaw
- Fracture lines from a pore associated with an agglomerate
- Fracture lines from a large surface connected pore
- Fracture initiating from both sides of origin in different planes and joining
- Twist due to two parts of crack meeting

Fractography (9)

**Example 1: High purity alumina bend bar**

Optical fractography showing:
1. matched fracture surfaces of a flexural strength test bar
2. mirror region
3. compression side marked by compression curl
4. hackel (appears laterally only)
5. large internal pore
6. tail (wake hackle)

Optical fractography showing:
- tensile surfaces together
**Fractography (10)**

**Example 2: Failure from agglomerate intersected by machining the surface**

1. tensile surface
2. directions of failure
   
   ("rising sun" – use light to illuminate topography)
3. origin region
4. extended void
5. agglomerate

**Fractography (11)**

**Example 3: Chemical inhomogeneity in silicon nitride**

1. tensile surface
2. secondary electron image
3. backscattered electron image
4. high ytterbium (= sintering additive) concentration around pore (EDX)
Fractography (12)

Example 4: Fracture toughness calculated with natural flaw

\[
\text{Al}_2\text{O}_3
\]

\[\sigma_C = 292 \, \text{MPa}\]

\[2a \sim 2c \sim 160 \, \mu\text{m}\]

\[\rightarrow c/a \sim 1 \rightarrow Y \sim 1.13\]

.. go and calculate \[K_{IC}!!\]

\[K_{IC} \sim 3.0 \, \text{MPa} \sqrt{\text{m}}\]

\[K_{IC} \text{ measured in VAMAS / ESIS round robin 3.6 MPa} \sqrt{\text{m}}\]

Possible reasons:
- effective elliptical flaw size is larger ...
- granulate / effective defect isn’t a sharp crack ...

Fractography (13)

Example 5: TZP bend bar failing from large pore

\[\sigma_C = 728 \, \text{MPa}\]

\[a \sim 35 \, \mu\text{m}\]

\[2c \sim 140 \, \mu\text{m}\]

\[\frac{c}{a} \sim 2\]

\[Y_{\text{cent}} = 1.59 > Y_{\text{surf}} = 1.24\]

\[\rightarrow K_{IC} \sim 6.8 \, \text{MPa} \sqrt{\text{m}}\]

Measured:
\[K_{IC} = 4.7 \, \text{MPa} \sqrt{\text{m}}\]

Possible reasons:
- effective elliptical flaw size is smaller ...
- pore isn’t a sharp crack ...

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Learning targets part 3

What you should know and understand, now!

Weibull, a name you’ll never should forget …

• Weibull: mathematical description of failure / survival probability
  \[ P_f = 1 - P_s = 1 - \exp \left( -\frac{\sigma - \sigma_c}{\sigma_0} \right) \]

• Weibull parameter m describes the width of the distribution:
  - small m = large distribution
  - large m = small distribution

• If you talk from “characteristic strength” \( \sigma_0 \), already 2/3 of your components failed!

• The effect of volume and/or surface area on the acceptable stress level can be calculated. (If you want to hide the poor quality of your material use 3-point bend test to get failure stress values.)

• Proof testing will eliminate “bad” components. Lower end of distribution is cut off and new distribution isn’t a proper Weibull distribution anymore.

Reading fracture surfaces …

• Increasing the level of information of a fracture by starting from its history.
• Fracture patterns will lead you to the origin zone.
• Macro- and micro-features point towards the origin.
• Fracture mechanics and fractography combined are strong tools to
  - develop materials
  - optimize procedures and processes
  - construct components
  - improve machining
  - design systems

Guide for fractographer …

• .. get familiar with the failure and its "environment" simply by the naked eye and a map ..
• .. observe large markings and features with a simple optical microscope ..
• .. locate and understand small details with a SEM ..