Materials Science II - 2010, Ceramic Materials, Chapter 6, Part 3

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 σ_{3Pt} test > value σ_{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 √m and 20 MPa √m
- The aim is always to **improve** both
  - σ_c *by reducing a_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.

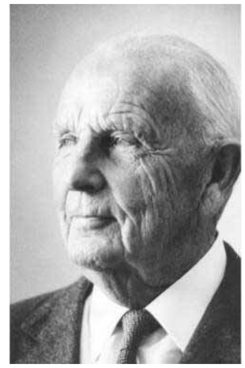
Aim of chapter & Learning targets

Part	No.	Topic	Learning target	
part 1 Crack tip	1.	Introduction	"Why mechanical testing"	learning targets 1
	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"	
part 2 Strength	5.	R-curve	"Improving toughness"	learning targets 2
	6.	Properties	"Knowing what you measure"	
	7.	Strength	"Just a value"	
part 3 Statistics	**8.**	**Statistic**	**"Weibull, a name you'll never should forget"**	**learning targets 3**
	9.	**Proof testing**	**"Make it or"**	
	10.	**Fractography**	**"Reading fracture surfaces"**	
part 4 Time&Temp	11.	Thermal shock	"Temperature, time and geometry"	learning targets 4
	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

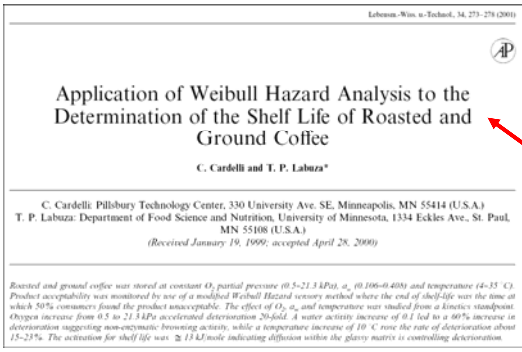
Weibull statistic (1)

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.



Lebensm.-Wiss. u.-Technol., 34, 273–278 (2001)

Application of Weibull Hazard Analysis to the Determination of the Shelf Life of Roasted and Ground Coffee

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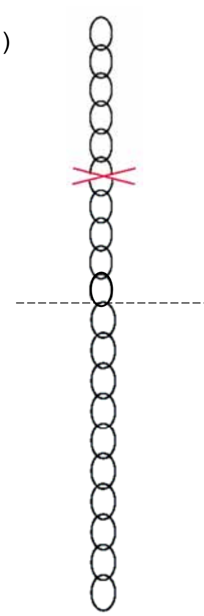
(Received January 19, 1999; accepted April 28, 2000)

Roasted and ground coffee was stored at constant O_2 partial pressure (0.5–21.3 kPa), a_w (0.106–0.408) and temperature (4–35 °C). Product acceptability was monitored by use of a modified Weibull Hazard sensory method where the end of shelf-life was the time at which 50% consumers found the product unacceptable. The effect of O_2, a_w and temperature was studied from a kinetics standpoint. Oxygen increase from 0.5 to 21.3 kPa accelerated deterioration 20-fold. A water activity increase of 0.1 led to a 60% increase in deterioration suggesting non-enzymatic browning activity, while a temperature increase of 10 °C rose the rate of deterioration about 15–23%. The activation for shelf life was ≅ 13 kJ/mole indicating diffusion within the glassy matrix is controlling deterioration.

Today used for many other time-dependant fault mechanisms, too.

Weibull statistic (2)

Model of the chain with the weakest link (1)



- a chain is only as strong as it‘s 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 :

$$P_s(L) \quad < 1$$

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

$$P_s(2L) = P_s(L) \cdot P_s(L) \qquad \textit{(not +)}$$

- 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 σ is $R(\sigma)$

→ P_S of length L_0 surviving at σ is $1 - R(\sigma)$

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

→ P_S of length ΔL surviving at σ is $1 - R(\sigma) \cdot (\Delta L / L_0)$

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

P_S of length L_0 multiplied by P_S of length ΔL

$$P_s(L_0) = 1 - R(\sigma) \qquad P_s(\Delta L) = 1 - R(\sigma) \cdot \frac{\Delta L}{L_0}$$

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

Weibull statistic (4)

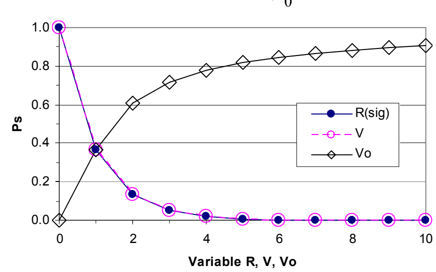
Model of the chain with the weakest link (3)

3-D-analogy →
$$P_s(V_0 + \Delta V) = P_s(V_0) \cdot \left[1 - \frac{R(\sigma) \cdot \Delta V}{V_0} \right]$$

now: $\Delta V \rightarrow dV$
$$\frac{dP_s(V)}{dV} = -\frac{R(\sigma) \cdot P_s(V)}{V_0}$$

and integrating:
$$\int \frac{dP_s(V)}{P_s(V)} = -\frac{R(\sigma) \cdot V}{V_0} \rightarrow P_s = \exp\left(\frac{-R(\sigma) \cdot V}{V_0}\right) + C$$

because $P_s = 1$ only for $V = 0$ → $C = 0$
$$P_s = \exp\left(\frac{-R(\sigma) \cdot V}{V_0}\right)$$



Weibull statistic (5)

probability of V_0 failing at stress σ

if stress varies from place to place

→ probability of survival: $P_s = \exp\left(-\int \frac{R(\sigma) \cdot dV}{V_0}\right)$

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

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

m = Weibull modulus

σ_0 = characteristic (stress) strength

σ_c = stress below which no failure occurs

σ = stress at failure

Weibull statistic (6)

now it's possible to calculate P_S for a component under a mechanical stress:

$$\left(\frac{\sigma - \sigma_c}{\sigma_o}\right)^m \qquad P_s = \exp\left(-\int \frac{R(\sigma) \cdot dV}{V_0}\right)$$

$$\longrightarrow \quad P_S = \exp\left(-\int \left(\frac{\sigma - \sigma_c}{\sigma_o}\right)^m \frac{dV}{V_0}\right)$$

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

$$P_S = \exp\left(-\left(\frac{\sigma - \sigma_c}{\sigma_o}\right)^m \frac{V}{V_0}\right)$$

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* → ***σ_c = 0 MPa***
- *volume is normalized e.g. in standardised test bar* → ***$V = V_0$***

equation reduces to:

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

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

but σ_0 , **m** and σ
must somehow correlate with the density of the defects

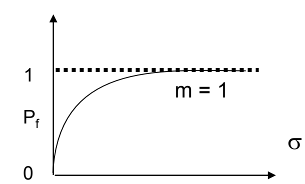
Weibull statistic (8)

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

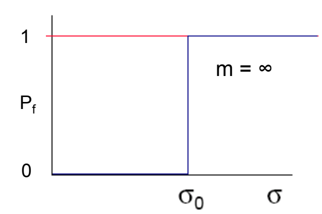
Weibull module m describes form of failure probability curve

m = 0
→ P_f independent of applied stress

m = 1
→ P_f exponential asymptotic curve

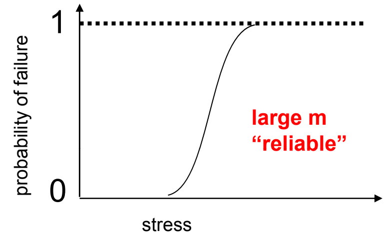


m = ∞
→ P_f "step curve"
- with $P_f = 0$ if $\sigma < \sigma_0$
- with $P_f = 1$ if $\sigma > \sigma_0$

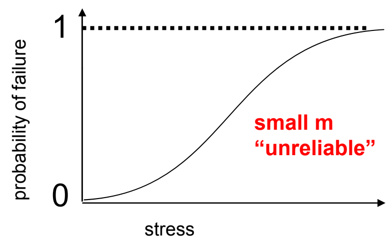


Weibull statistic (9)

- **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 σ_0
with defined volume, e.g. test bar:

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

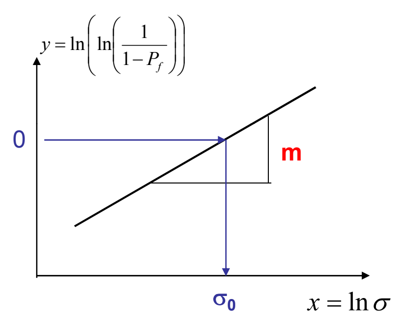
rearrange and take twice the logarithm:

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

$$y = m \cdot x + C$$

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_f))) = 0 → σ_0
= 0.632



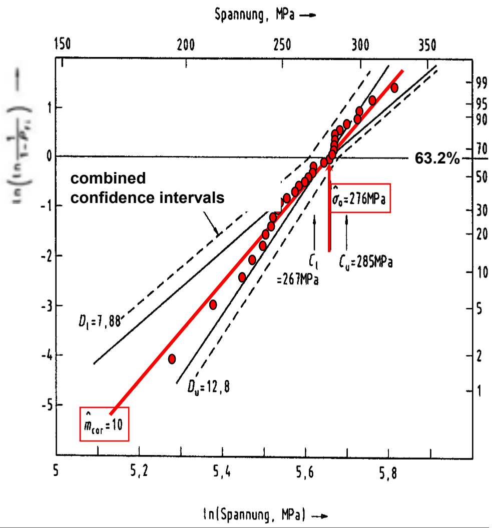
Weibull statistic (11)

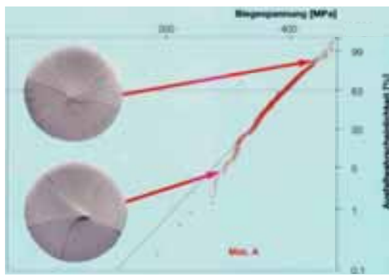
Determination of Weibull-parameter

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

$$P_{fi} = \frac{n-0.5}{N} \quad or \quad \frac{n}{N+1}$$

- draw Weibull-diagram
- calculate σ_0 and m
- calculate confidence intervals





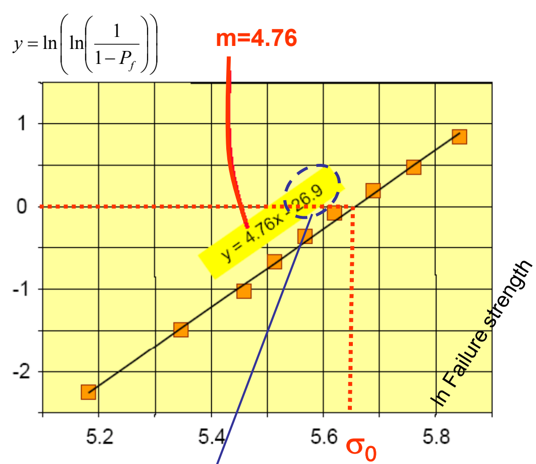
Weibull statistic (12)

Example: ground glass rods

Test No.	Failure strength (MPa)
1	178
2	276
3	262
4	296
5	210
6	248
7	235
8	318
9	345

Rank	Failure strength (MPa)	Failure probability (Pf)
1	178	0.1
2	210	0.2
3	235	0.3
4	248	0.4
5	262	0.5
6	276	0.6
7	296	0.7
8	318	0.8
9	345	0.9

$$P_f = \frac{n}{N+1}$$



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

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

^ = maximum likelihood estimate of Weibull characteristic strength of test piece

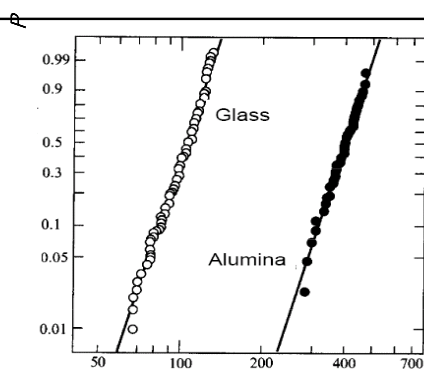
upper limits of confidence interval: $C_u = \hat{\sigma}_0 \exp\left(-\frac{t_u}{\hat{m}}\right)$

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

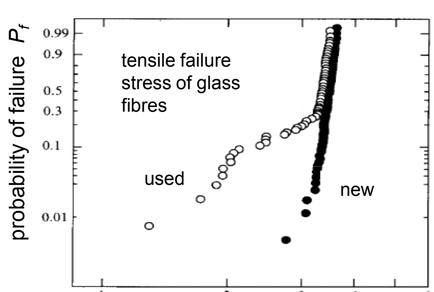
"Cook book" for confidence interval for m is identical

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 ≥ 30 experimentally measured values



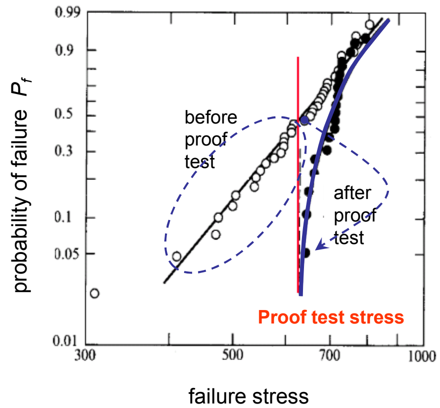
- <u>used</u>
  next to the "naturally" present defects (mainly volume) a 2nd defect population (surface) leads to lower failure stresses



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



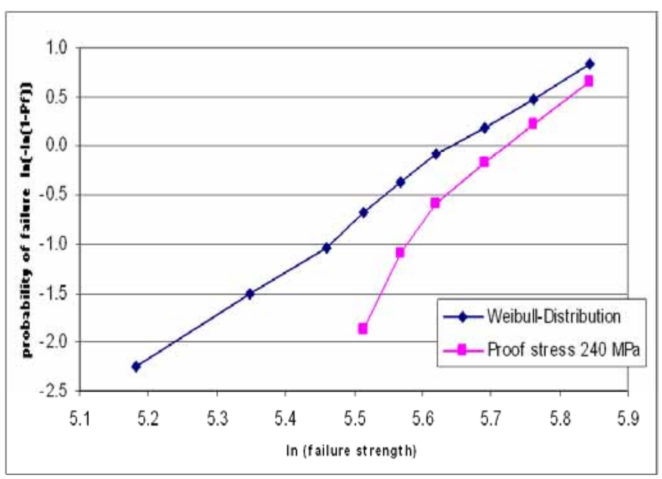
Components should be used after proof testing @ $\sigma < \sigma_P$

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)

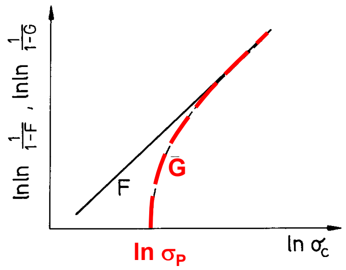
Rank	Failure strength (MPa)	Failure probability $P_f = n/(N+1)$
1	178	0.1
2	210	0.2
3	235	0.3
4	248	0.4
5	262	0.5
6	276	0.6
7	296	0.7
8	318	0.8
9	345	0.9

Proof stress 240 MPa



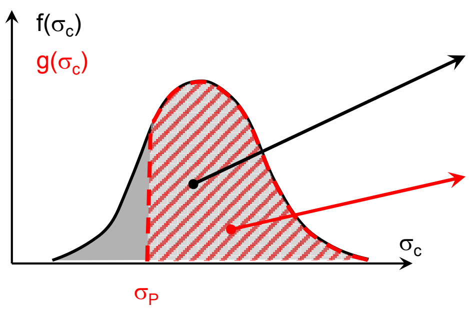
$G(\sigma_c)$ is not a Weibull distribution.

Proof testing (3)



distribution of stress for F and G after proof testing

What does it mean ?



$$F(\sigma_c) = 1 - \exp\left(-\left(\frac{\sigma_c}{\sigma_o}\right)^m\right)$$

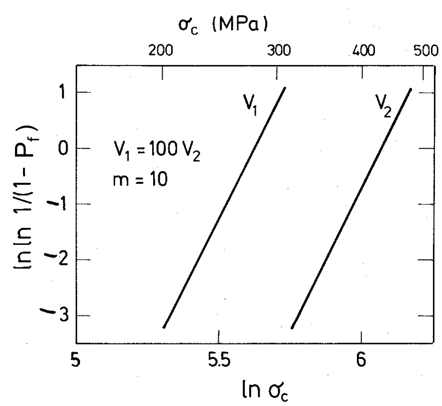
$$G(\sigma_c) = 1 - \exp\left(-\left(\frac{\sigma_c}{\sigma_o}\right)^m + \left(\frac{\sigma_p}{\sigma_o}\right)^m\right)$$

Influence of volume

$$P_{1S} = \exp\left(-\left(\frac{\sigma_1}{\sigma_0}\right)^m \frac{V_1}{V_0}\right) \quad \text{and} \quad P_{2S} = \exp\left(-\left(\frac{\sigma_2}{\sigma_0}\right)^m \frac{V_2}{V_0}\right)$$

… the influence of volume ***V_2*** for a stress ***σ_2*** can be calculated from volume ***V_1*** and stress ***σ_1*** if ***m*** is know:

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

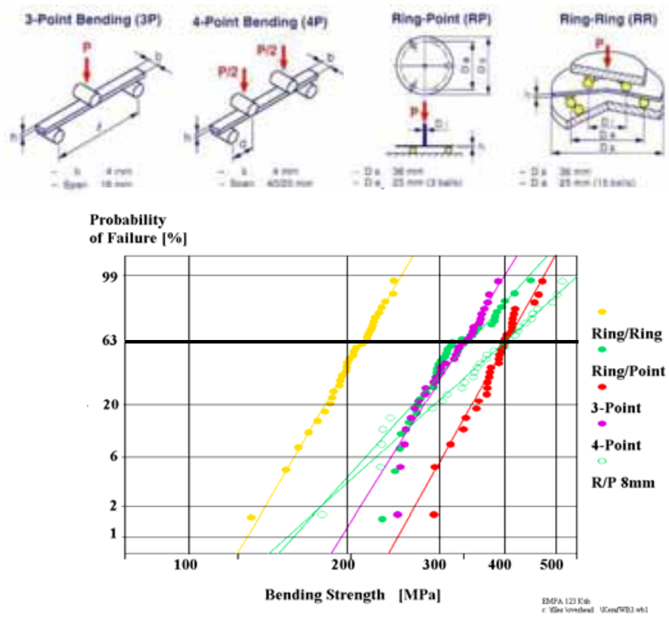


example: 3 x 4 x 45 mm bend bar:

3-pt BT	$V_{2\ eff} \approx$ 1 mm³	→ **1.27** x σ_1
4-pt BT - 40 / 20 mm	$V_{1\ eff} \approx$ 11 mm³	→ σ_1
tensile - 3x4x20 mm	$V_{3\ eff} \approx$ 240 mm³	→ **0.73** x σ_1

Weibull statistic (16)

Influence of tested volume or surface



Fractography (1) why ?

"Improvement through feedback."

(… cause of failure)

Question (reason)	Answer for (application)
Where did it break from?	Engineering
Did it crack suddenly or slowly?	Engineering
Why did it break form here?	QA, process monitoring
Nature of fracture source?	Material development, QA
Stress at fracture?	Design
Environment or fatigue?	Engineering
Good test?	Material evaluation
Whose fault?	Commercial, legal

… 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)*

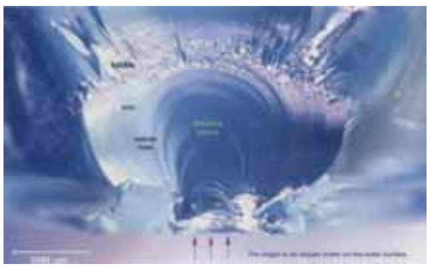
Fractography (2)

roman temple rebuild after earth quake

shell like chip

Garni, Armenia
© Kübler, 2003

example glass



Am. Ceram. Soc. Bulletin

Fractography (3)

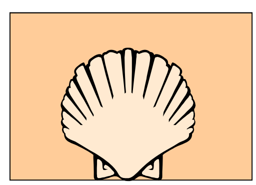
old but not well known science

First mention of ceramic fractures by E. Bourry in: *A treatise on ceramic industries*
(first English editon 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"

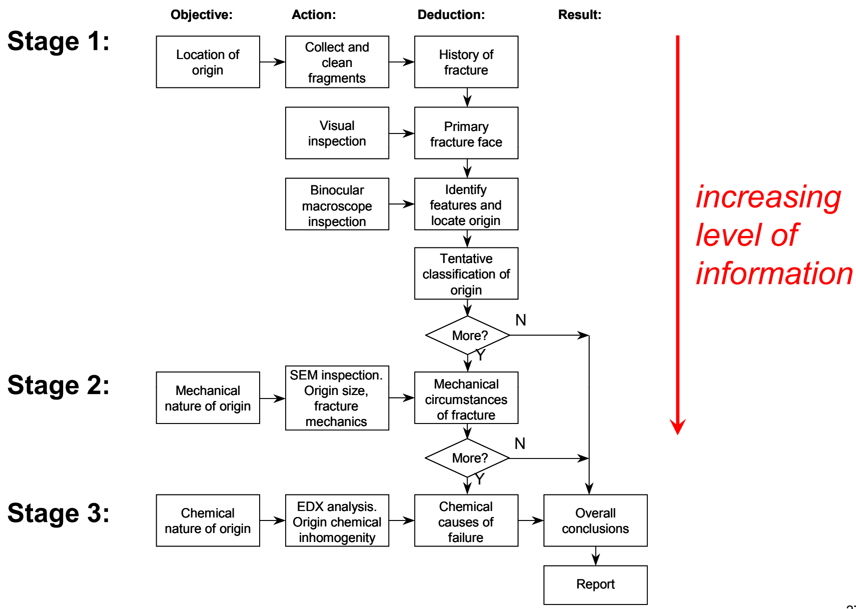


Guide for hobby astronomer …

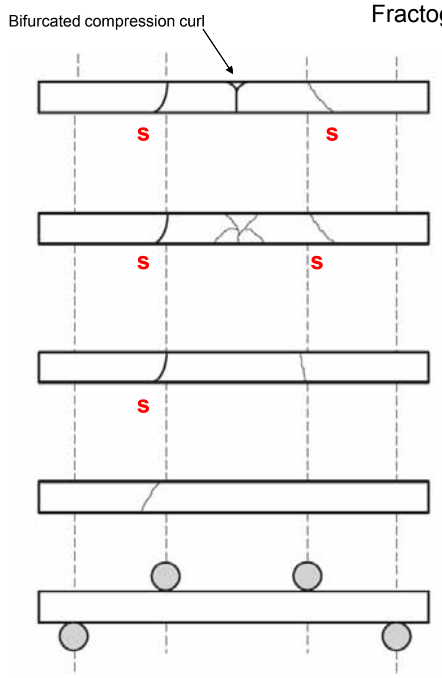
- *.. get familiar with the firmament simply by the naked eye and a map ..*
- *.. observe satellites and stars with a simple field glass ..*
- *.. locate and enjoy details of far away stars and galaxies with a telescope ..*

Fractography (4)

Flow diagram



Fractography (5)



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

Low to medium energy fracture outside the loading span; usually due to larger than normal fracture origin

Four-point bend test piece, tensile face on lower side

Fracture patterns in four-point flexural strength test pieces

"low energy"
e.g. porcelain

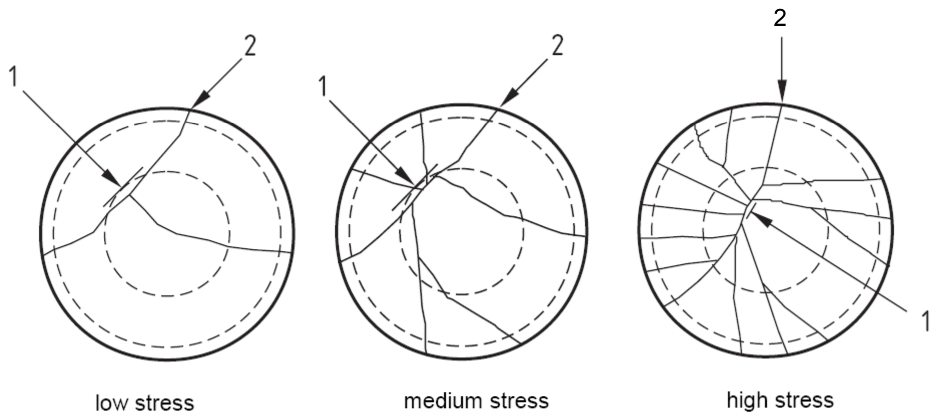
"medium energy"
e.g. fine grained alumina

"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)

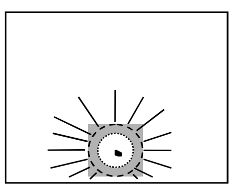
Fracture patterns in ring-on-ring test pieces



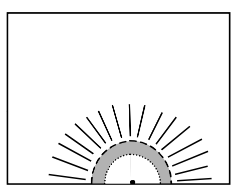
1 likely origin zone
2 primary crack

Fractography (7)

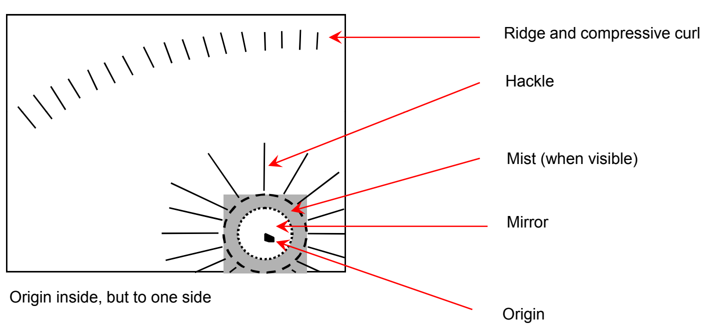
Macro-features in flexural test bars



Origin inside body



Origin at or close to surface

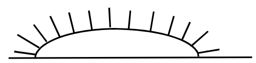


Origin inside, but to one side

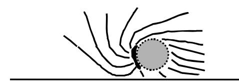
Fractography (8)

Microscopic: ‘fracture lines’ – fine hackle

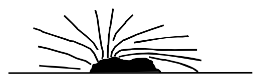
Features near fracture origins



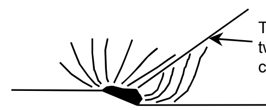
Fracture lines from an extended origin such as a machining flaw



Fracture lines from a pore associated with an agglomerate



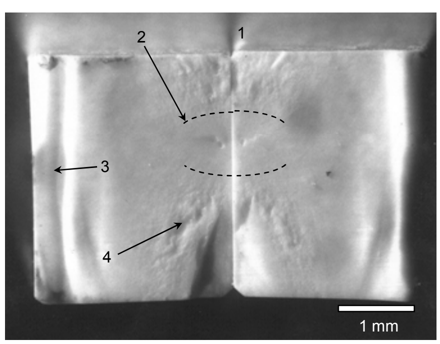
Fracture lines from a large surface connected pore

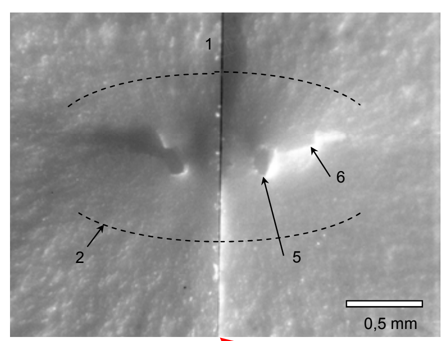


Fracture initiating from both sides of origin in different planes and joining

Fractography (9)

Example 1: High purity alumina bend bar



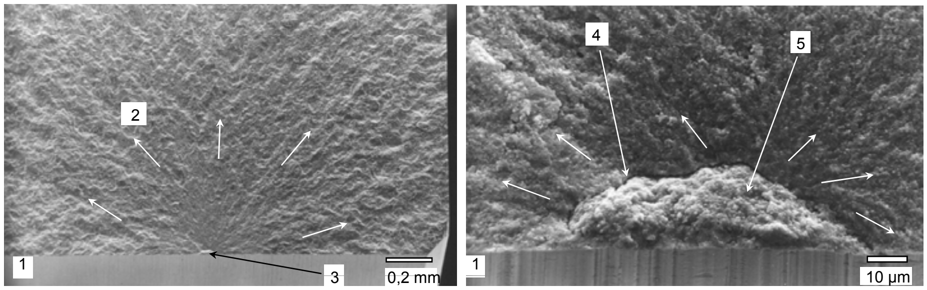


Optical fractography showing:
1 matched fracture surfaces of a flexural strength test bar
2 mirror region
3 compression side marked by compression curl
4 hackel (appears laterally only)
5 large internal pore
6 tail (wake hackle)

tensile surfaces together

Fractography (10)

Example 2: Failure from agglomerate intersected by machining the surface



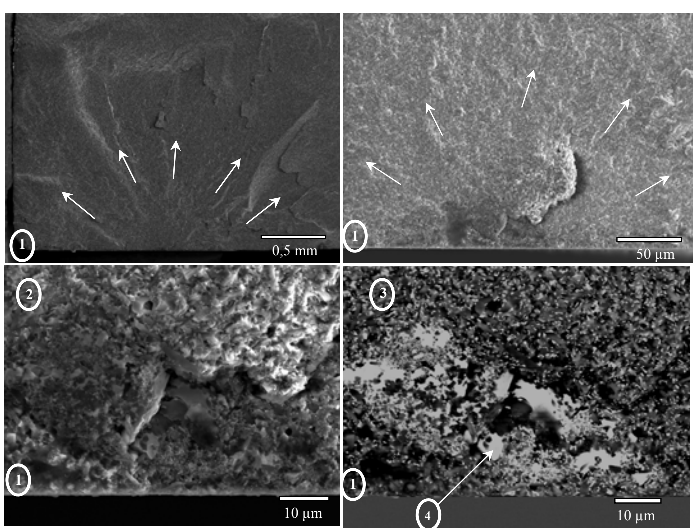
1 tensile surface
2 directions of failure
("rising sun" – use light to illuminate topography)
3 origin region
4 extended void
5 agglomerate

Fractography (11)

Example 3: Chemical inhomogeneity in silicon nitride

(a) Macro:

(b) Meso:



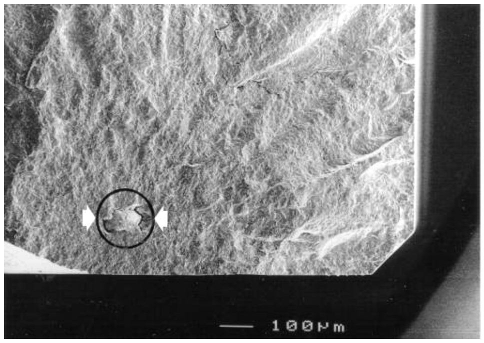
(c) Micro:

(d) BS:

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



Al_2O_3

σ_C = 292 MPa

2a ~ 2c ~ 160 µm

→c/a ~ 1 →Y ~ 1.13

.. ~~go and calculate K_{Ic} !!!~~

K_{Ic} ~ 3.0 MPa √m

K_{Ic} measured in VAMAS / ESIS round robin 3.6 MPa √m

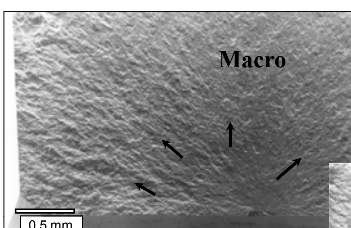
possible reasons:

→ effective elliptical flaw size is larger …

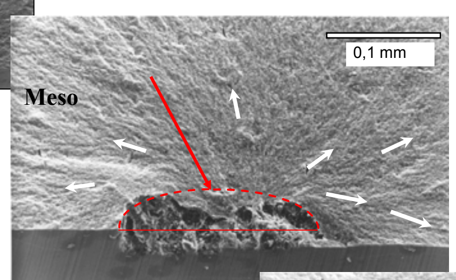
→ granulate / effective defect isn't a sharp crack …

Küb @ Fractography of Glasses and Ceramics III
Alfred University, NY, USA, 29.061995

Fractography (13)



Example 5: TZP bend bar failing from large pore



σ_c = 728 MPa

a ~ 35 µm

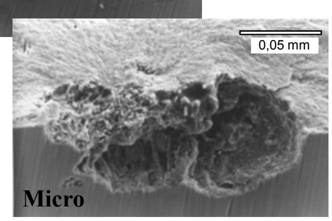
2c ~ 140 µm

c/a ~ 2

Y_{cent} = 1.59 > Y_{surf} = 1.24

→ K_{Ic} ~ 6.8 MPa √m

<u>Measured:</u>
K_{Ic} = 4.7 MPa √m



possible reasons:

→ effective elliptical flaw size is smaller …

→ pore isn't a sharp crack …

Küb @ FAC 2001, Stara Lesná, Slovakiá

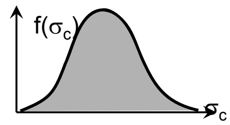
What you should know and understand, now!

Weibull, a name you'll never should forget

- Weibull: mathematical description of failure / survival probability

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

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



- If you talk from "characteristic strength" σ_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.)*

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

- 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 ..*