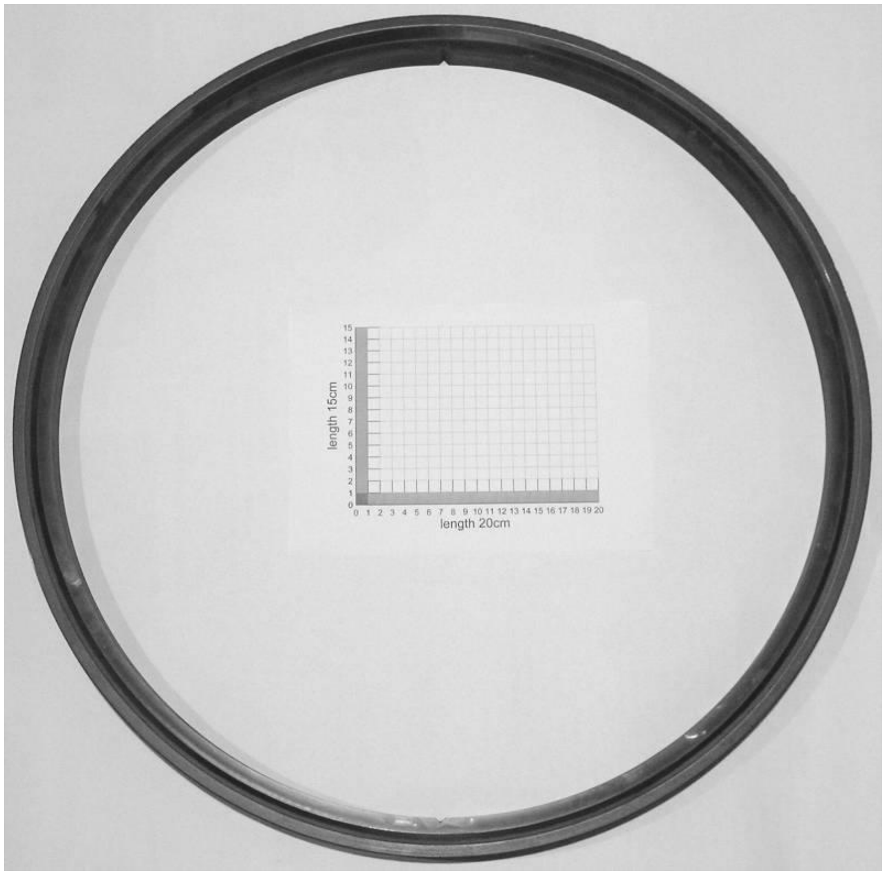
1) Ceramics – oldest structural material
2) Nature of brittleness
3) Statistical nature of strength
4) Ceramics testing
5) Toughening

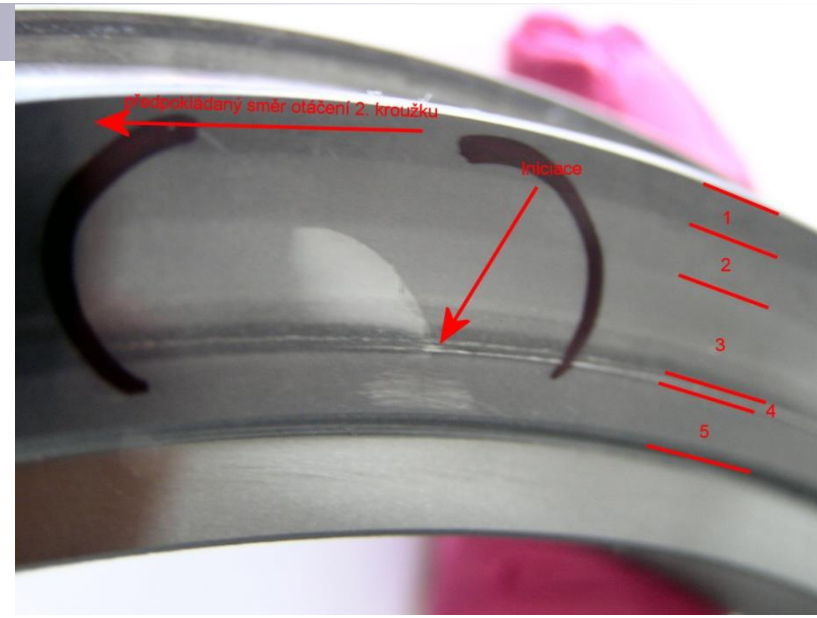
Technological development - renewed use

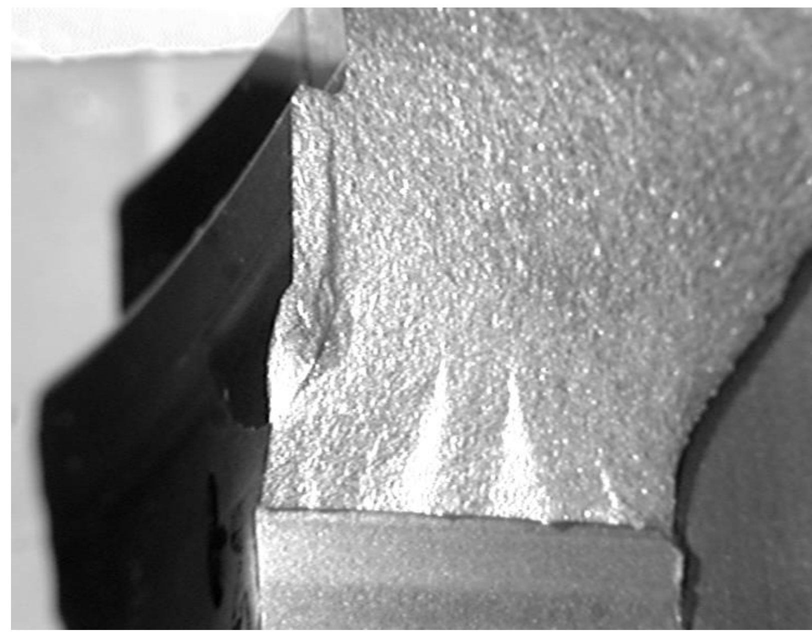
- White ceramics – no mechanical stresses transmitted – resistance against temperature shocks, corrosion and wear
- Building materials – strength properties – dominant role – heavy structures
- Structural ceramics - bioceramics, blades of pumps, valve seat, filtres – light structures
- Tools and grinding materials – tools, tools for high temperatures, manipulation tools

sealing rings

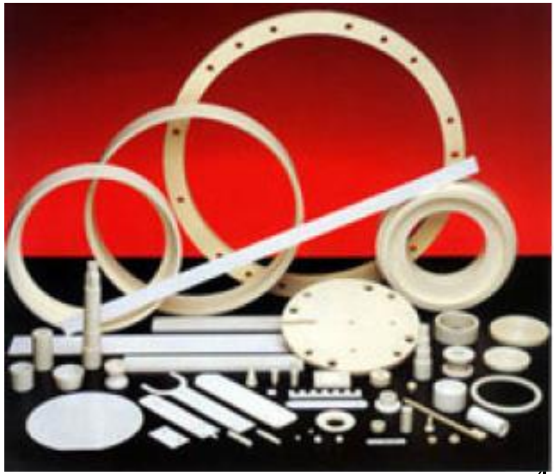
for sea ship shafts







- **Extremelly loaded components**
  - Cutting tools (Al_2O_3/SiC_W)
  - Abrasion resistant components ($Al_2O_3/SiC+ZrO_2$ apod.)
  - Building elements
  - Valve seats
  - Engine components (Si_3Ni_4/SiC… SiC/SiC)
  - Armours (SiAlON /SiC … SiC/SiC)
  - Biocompatible implantants ($CaO.SiO_2$ sklo / C, SiC)
  - Space applications (sklo/C)
- **Synergy effects**
- **Principially new properties**
  - Mechanical and physical properties
  - Autodiagnostics
  - Crack healing (property recovering)



Advantages of ceramics

from designer's poing of view

High specific elastic modulus

Hardness

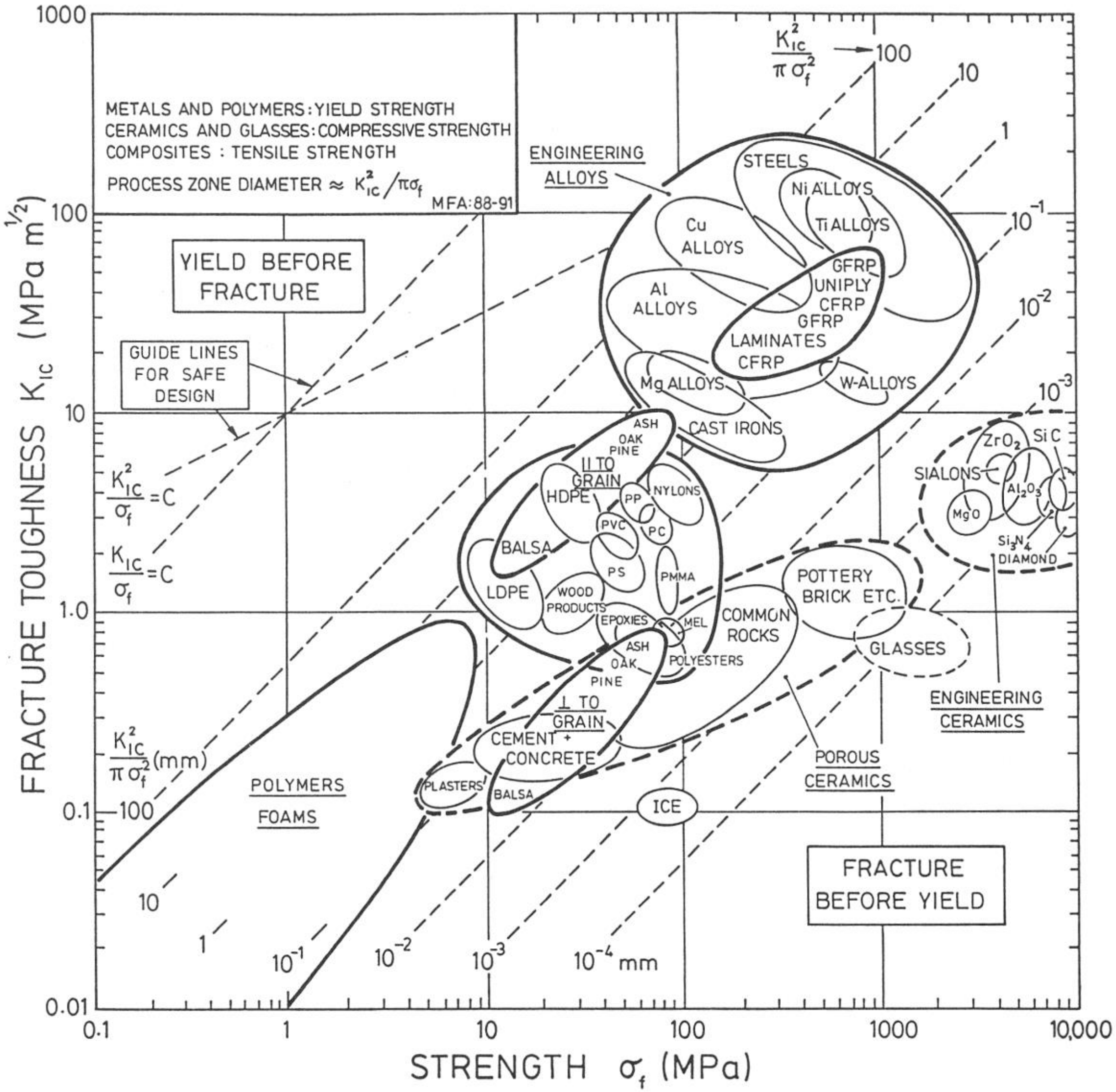
Abrasion resistance

Heat resistance

Corrosion resistance, chemical stabiblity

Consequence: brittleness (thermal shock resistance]

Origin of glass / ceramics brittleness



Comparing to other materials glass based and ceramic based materials are strong but having very poor fracture resistance

Origin of glass / ceramics brittleness

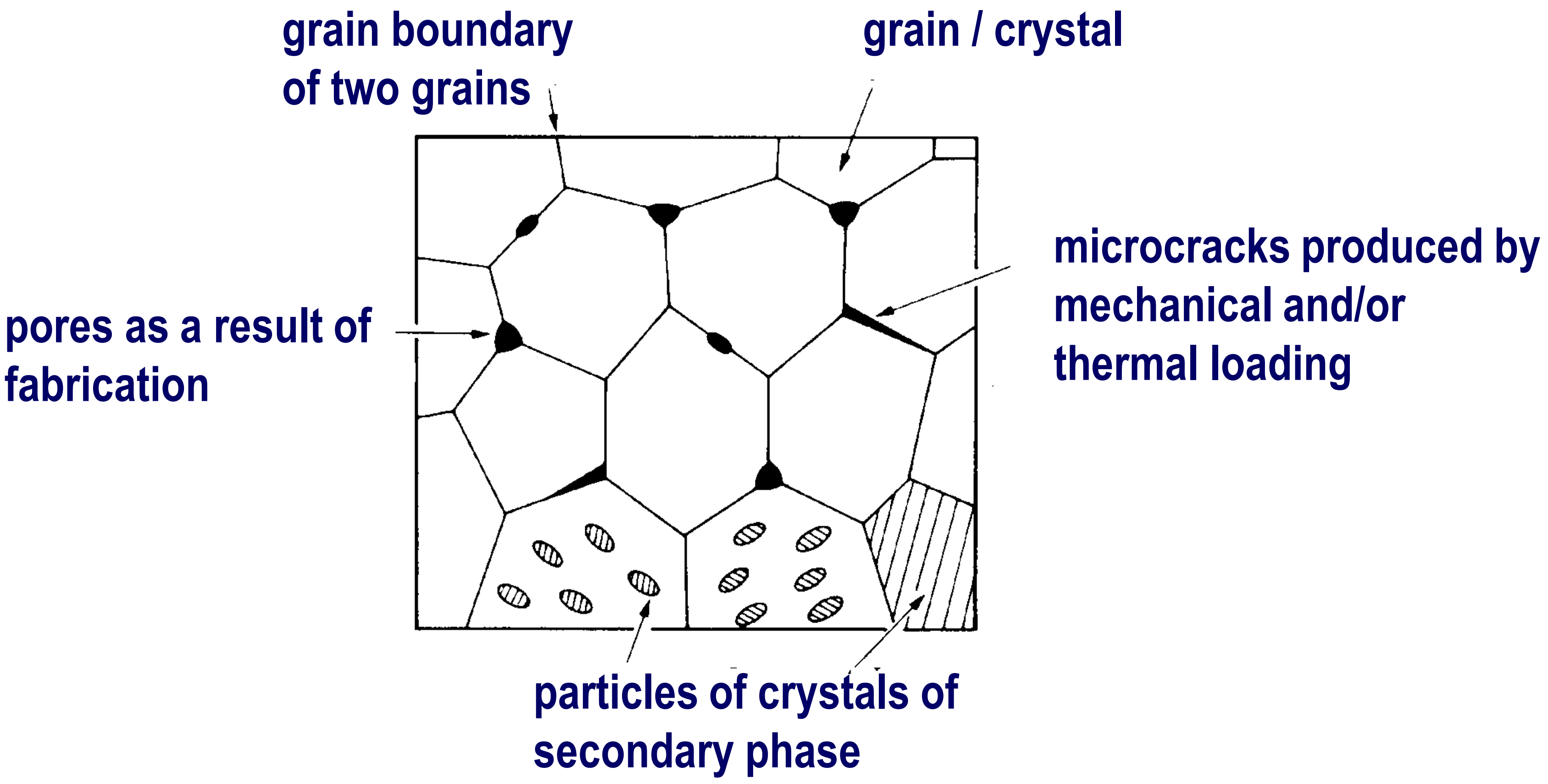
- **inherently strong hard material** - ion and covalent bonding
- high resistance to dislocation movement = impossibility of stress relaxation around structure imperfections (flaws, defects)

	Strength (tensile/bending) MPa	Compressive strength MPa	Elastic modulus GPa	Fracture Toughness $MPam^{0.5}$
Cast Iron	200 - 600	600 - 1500	200	10 - 25
Steel	600 - 1500	600 - 2000	200	20 - 200
Glass	20 - 60	200 - 400	70	0.6
Al2O3	200 - 400	1000 - 2000	380	2-4
SiC	100 - 200	500 - 1500	400	2-3
ZrO2	300 - 600	1000 - 2000	210	6-8

Origin of glass / ceramics brittleness

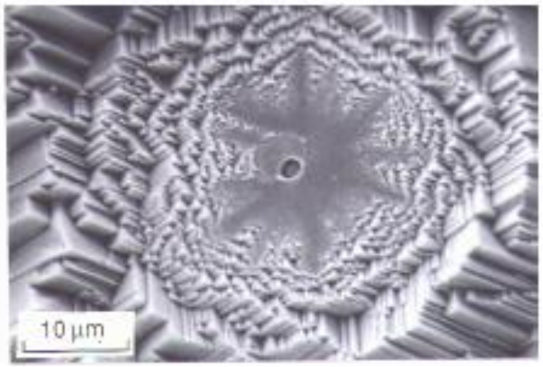
- **inherently strong hard material** - ion and covalent bonding

- high resistance to dislocation movement = impossibility of stress relaxation around structure imperfections (flaws, defects)

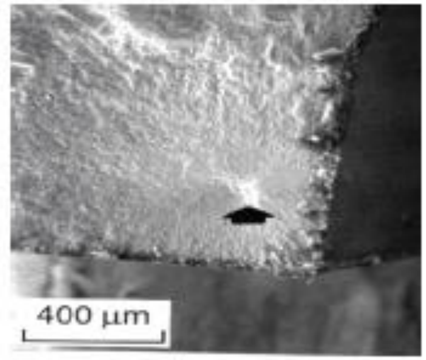


Origin of glass / ceramics brittleness

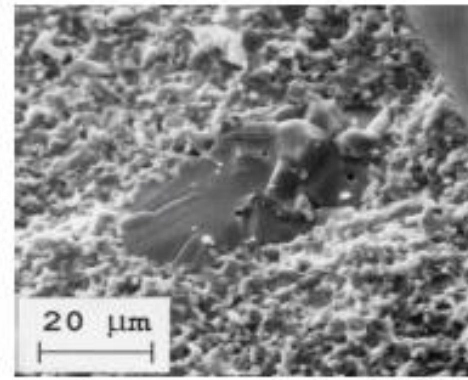
pores, agglomerates, inclusions / foreign particles, large grains, surface cracks, contact damage, thermal shock cracks



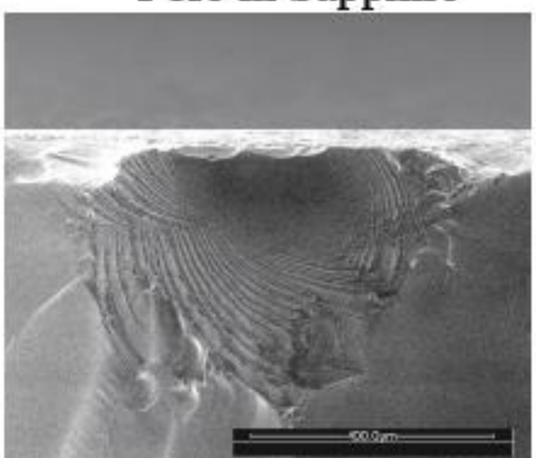
Pore in Sapphire

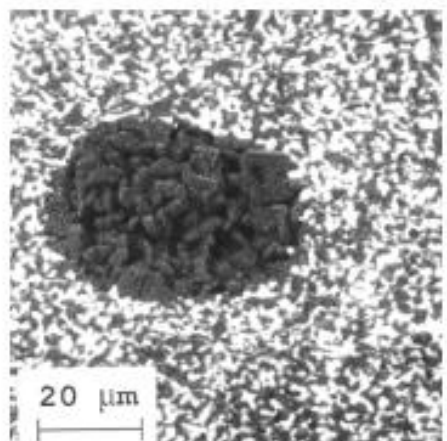


SiC inclusion in Si_3N_4

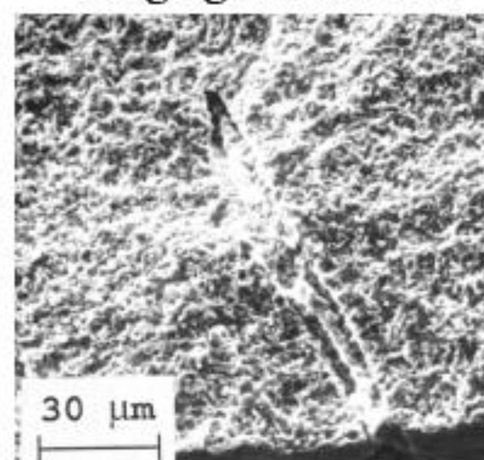


Large grain in ZTA


Surface damage in yttrium aluminum garnet



Agglomerate in ZrO_2 toughened Al_2O_3 (ZTA)



Pore (organic burn-out)

Origin of glass / ceramics brittleness

Allowable (maximum) flaw dimension

Strength

$\sigma_f \approx 200$ MPa

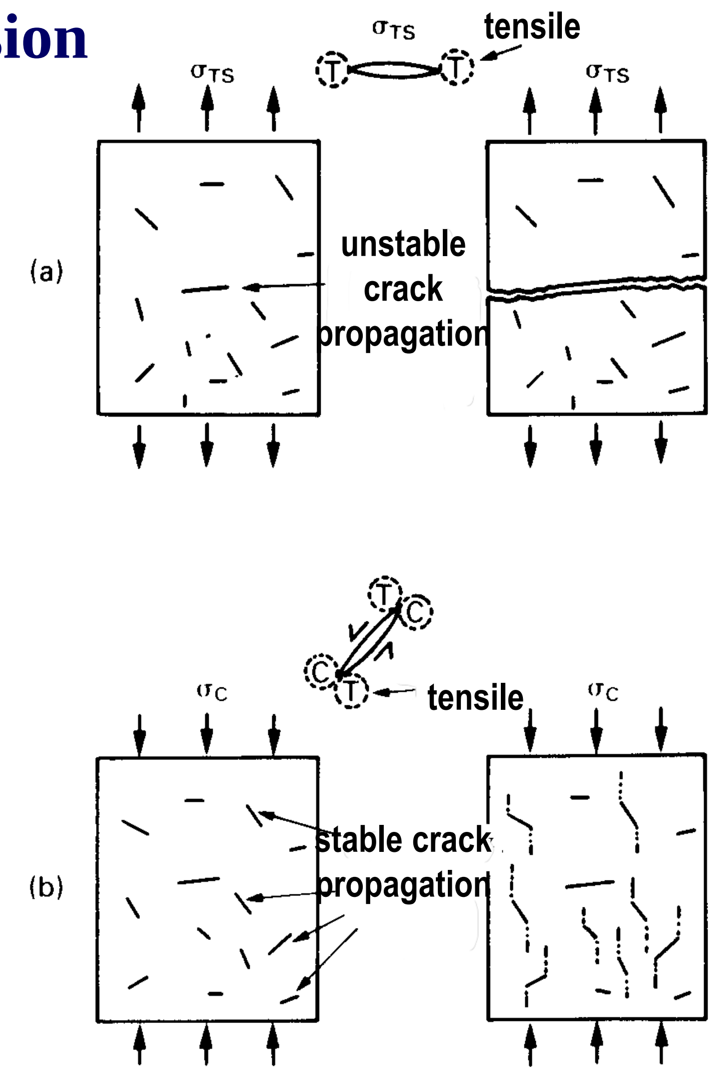
Fracture toughness

$K_{IC} \approx 2$ MPa.m$^{1/2}$

$$\sigma_f = R_m = \frac{K_{IC}}{\sqrt{\pi a}}$$

Crack dimension

$2a_{max} = 60$ µm



Origin of glass / ceramics brittleness

$$\sigma_f = R_m = \frac{K_{IC}}{\sqrt{\pi a}}$$

Strength of ceramics depends on its brittleness

How to increase the (fracture) strength of ceramics

1) **By decreasing present flaws and defects - a_{max}**

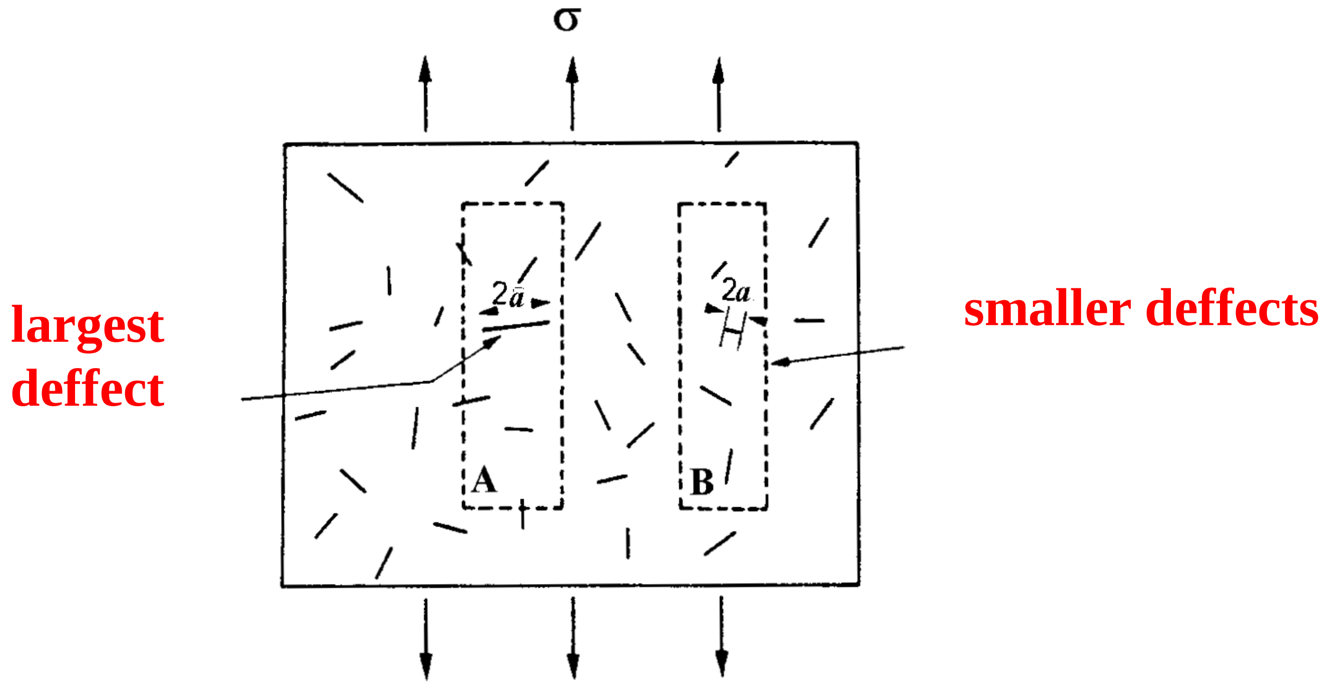
   (grain refinement, higher purity, precise fabrication, by diamond lapping)

2) **By increasing fracture toughness**

   (enhancement of crack resistance – corresponding materials design)

Statistical nature of brittle fracture

strength of test samples (of the same geometry) cut from the same plate



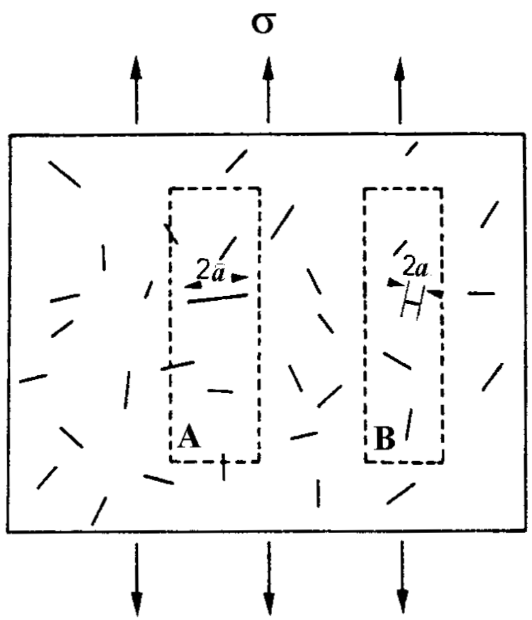
there is no certain (deterministic) tensile strength of the given ceramic sample…. only probability, that a given sample will have given strength (statistical size effect)

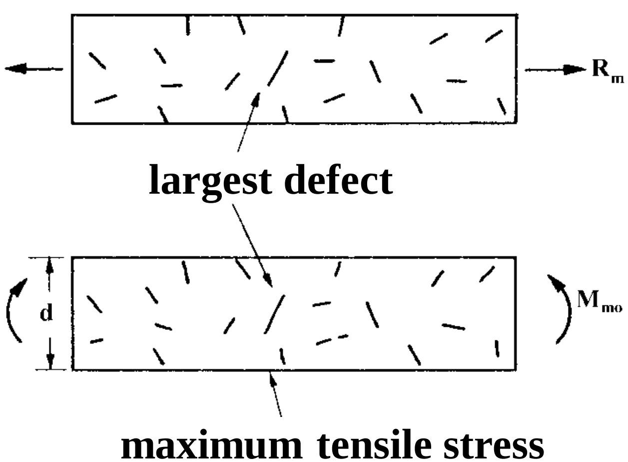
two nominally same samples A and B possesses different strength

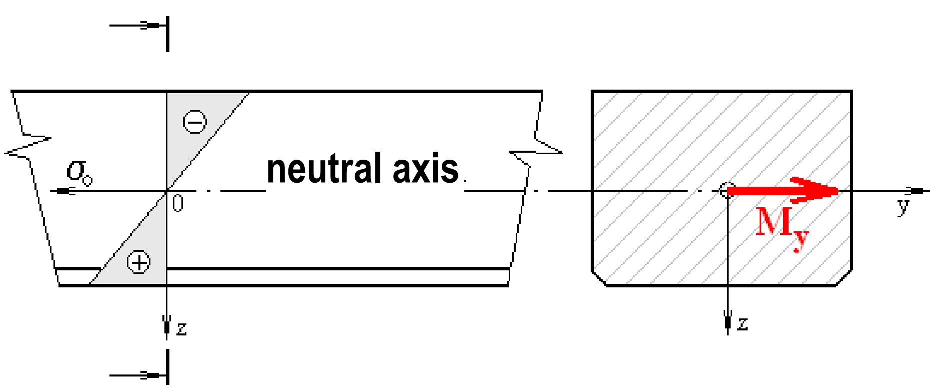
Statistical nature of brittle fracture

- two nominally same samples A a B have different strength
- bigger sample has lower strength (because of higher probability of largest defect presence)

- flexural strength is higher than tensile strength (about 1.7 x)





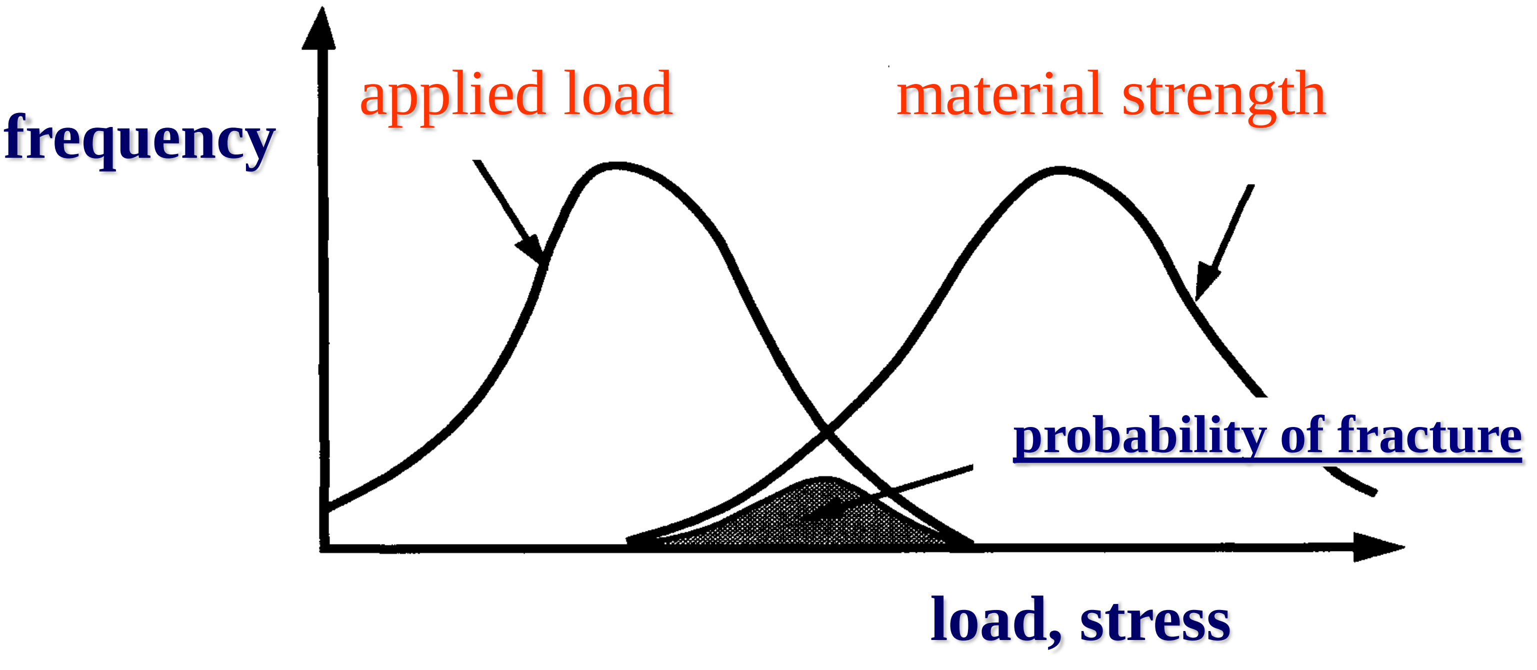


Statistical nature of brittle fracture

strength calculation of component design

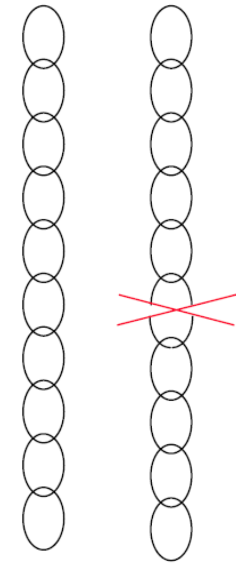
probability of fracture (survival)

chalk:	$P_f = 0.3$
cutting tool:	$P_f = 10^{-2}$
space component:	$P_f = 10^{-8}$



Statistical nature of brittle fracture

weakest link concept



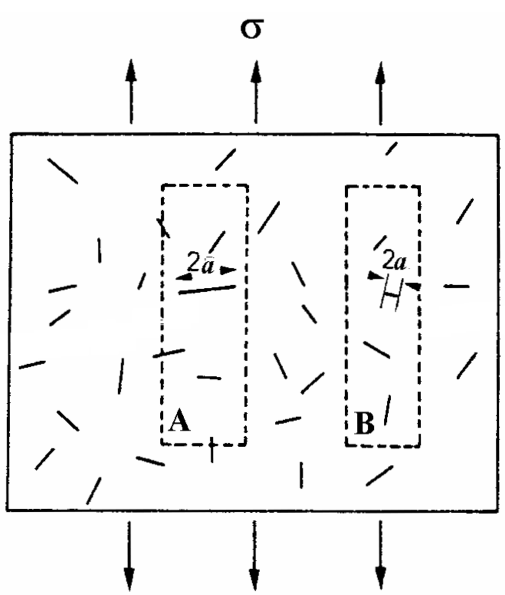
Weibull statistics

probability of sample survival, $P_S(V_0)$

(i.e. fracture will not occur)

ratio of identical samples, each having volume V_0, which survive applied stress σ, to total number of elementary volumes

$$P_s(V_0) = \exp\left\{-\left(\frac{\sigma}{\sigma_0}\right)^m\right\}$$



m – Weibull modulus … scatter

σ_0 – Weibull parameter … stress

Statistical nature of brittle fracture

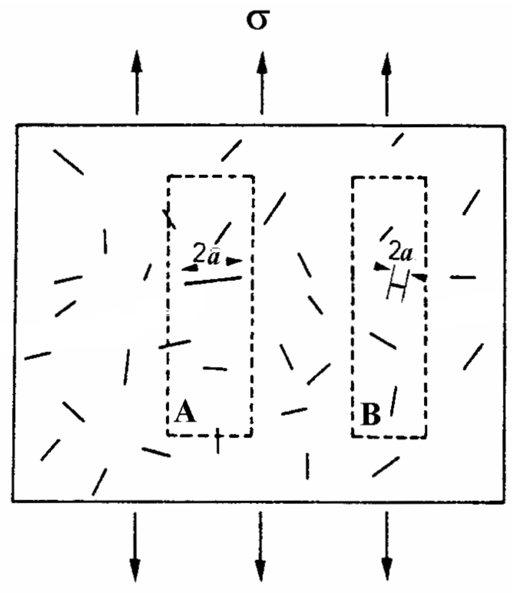
m – Weibull modulus

σ_0 – Weibull stress

probability of fracture $P_f(V_0)$

$$P_f(V_0) = 1 - P_s(V_0)$$

$$P_f(V_0) = 1 - \exp\left\{-\left(\frac{\sigma}{\sigma_0}\right)^m\right\}$$



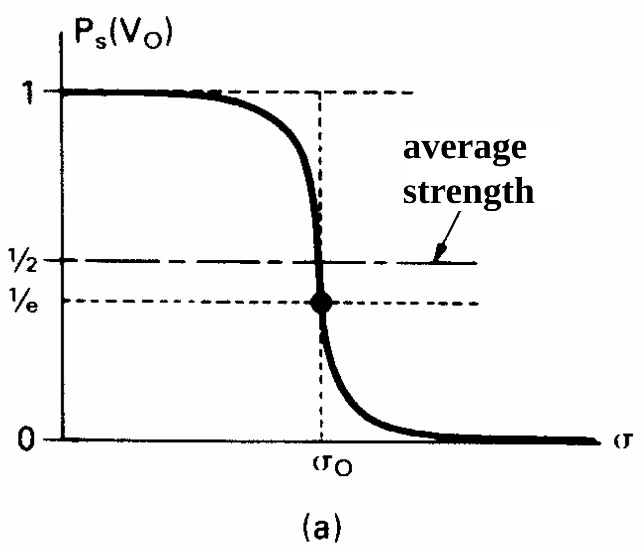
in inhomogeneous stress field

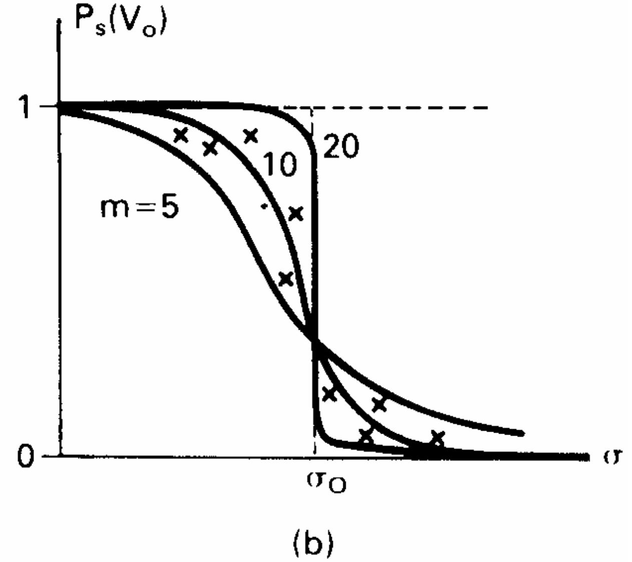
$$P_f = 1 - \exp\left[-\frac{1}{V_0}\int_V \left(\frac{\sigma}{\sigma_0}\right)^m dV\right]$$

$$P_s(V_0) = \exp\left\{-\left(\frac{\sigma}{\sigma_0}\right)^m\right\}$$

- when σ =0, all samples stay unbroken, i.e. $P_s(V_0) = 1$
- with increasing σ, probability of survival $P_s(V_0)$ decreases
- for $\sigma = \sigma_0$, we can get $P_s(V_0) = 1/e = 0.37$,

  i.e. at stress $\sigma = \sigma_0$ in total 37% of samples will stay unbroken and fracture probability equals to 63 % (Weibull stress definition)
- *m* – Weibull modulus – scatter parameter, how much strength is changing around σ_0 (*m* ≈ 5 brick, *m* ≈ 10 alumina)

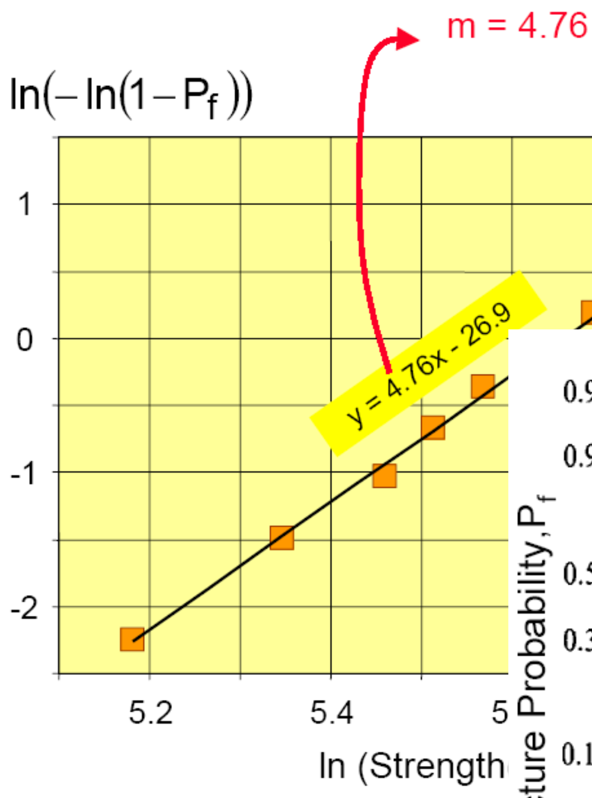


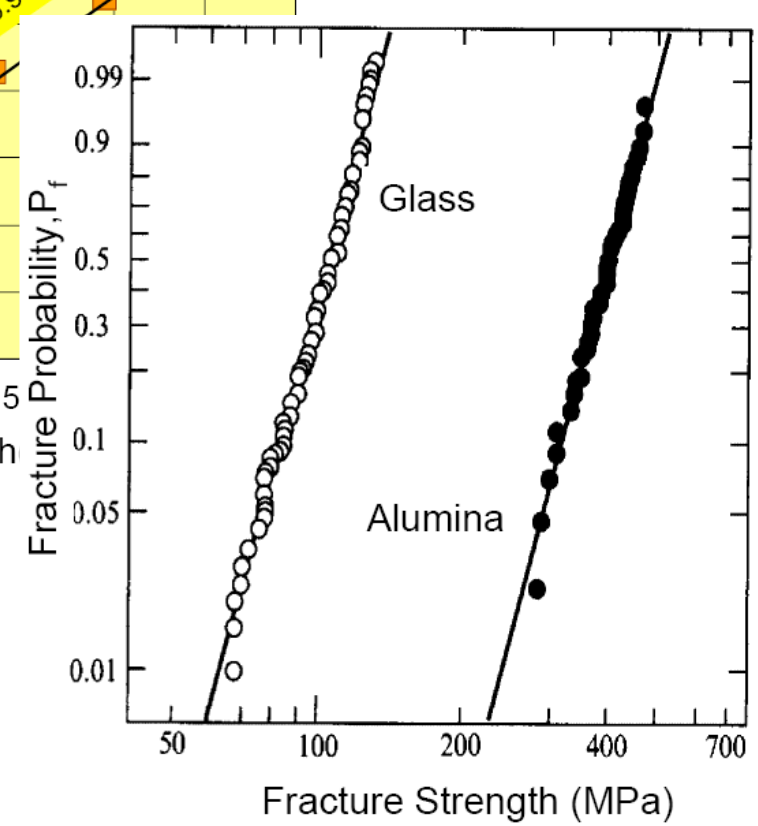


Statistical nature of brittle fracture

$$P_f(V_0) = 1 - \exp\left\{-\left(\frac{\sigma}{\sigma_0}\right)^m\right\}$$

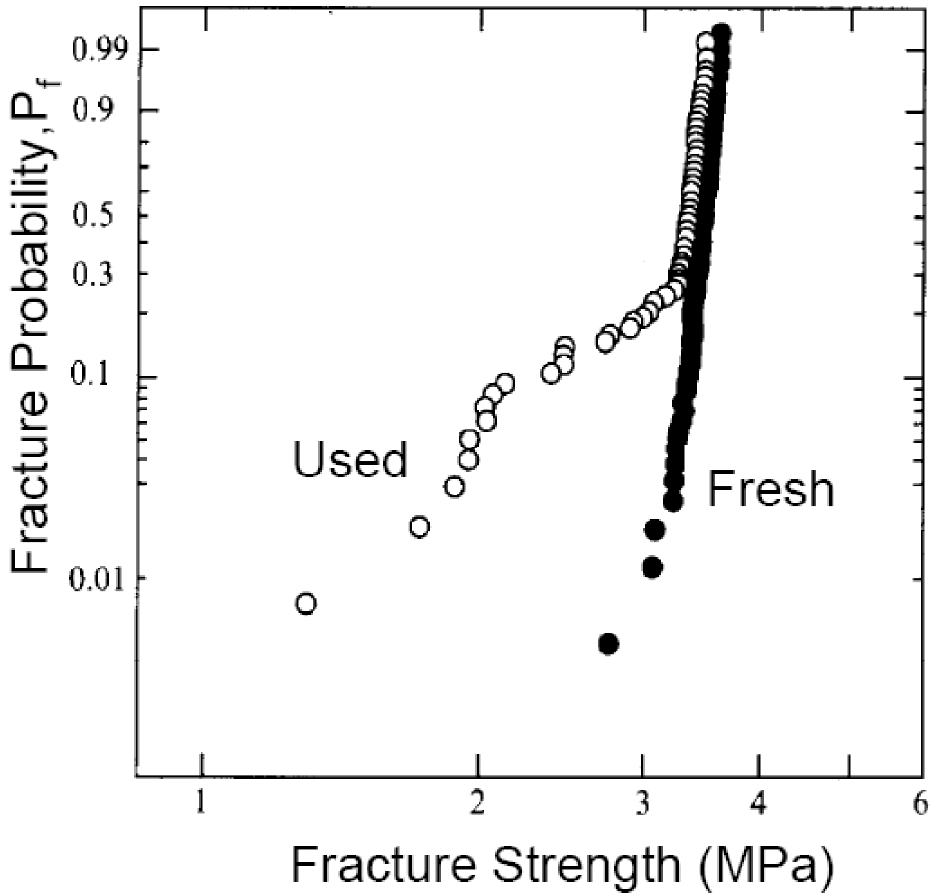
Strength (MPa)	Rank Order	Failure Prob.
178	1	0.1
210	2	0.2
235	3	0.3
248	4	0.4
262	5	0.5
276	6	0.6
296	7	0.7
318	8	0.8
345	9	0.9





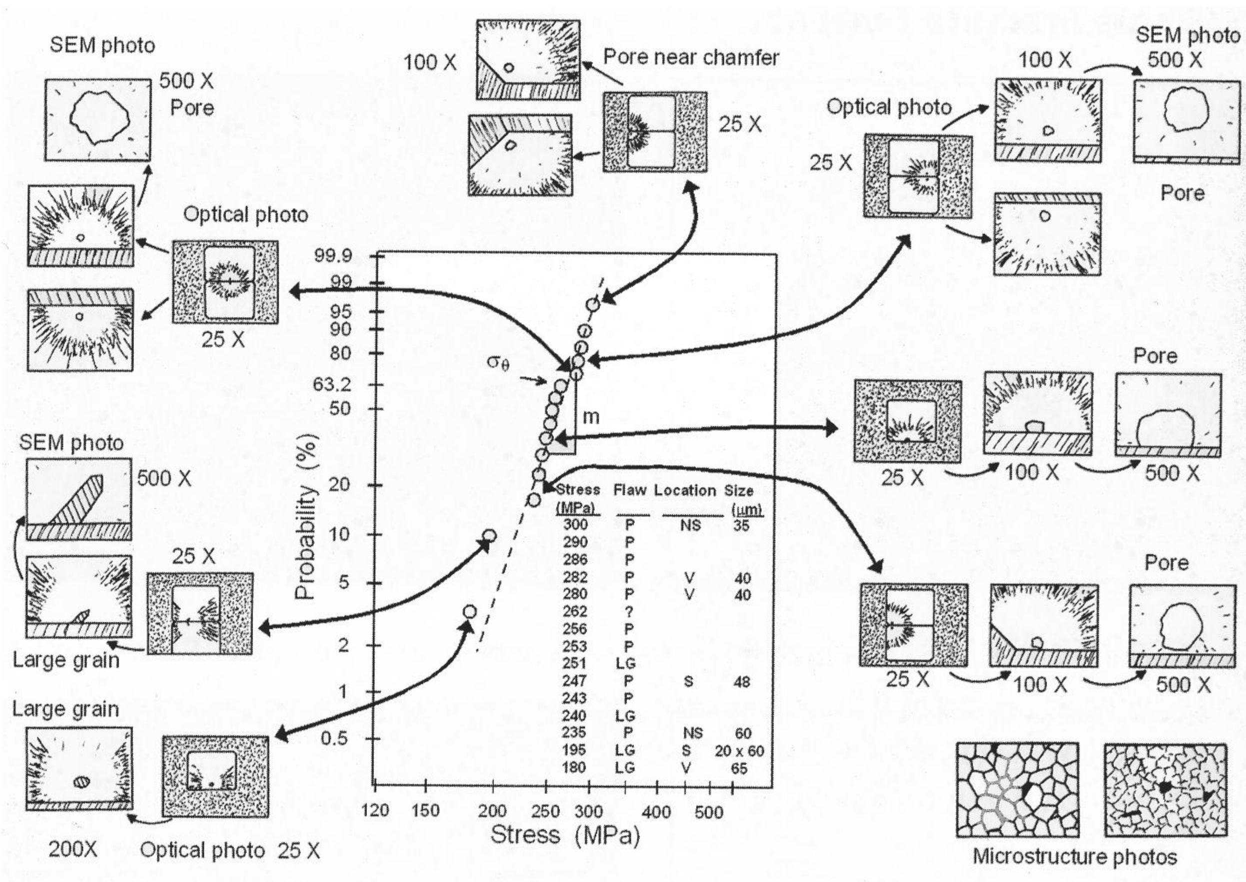
Statistical nature of brittle fracture

- Strength parameters: Weibull stress, Weibull modulus
- Comparison of different states of the same material (optimisation, degradation, change of micromechanism)
- High susceptibility to changes in microstructure
- Fractographically supported Weibull diagrams

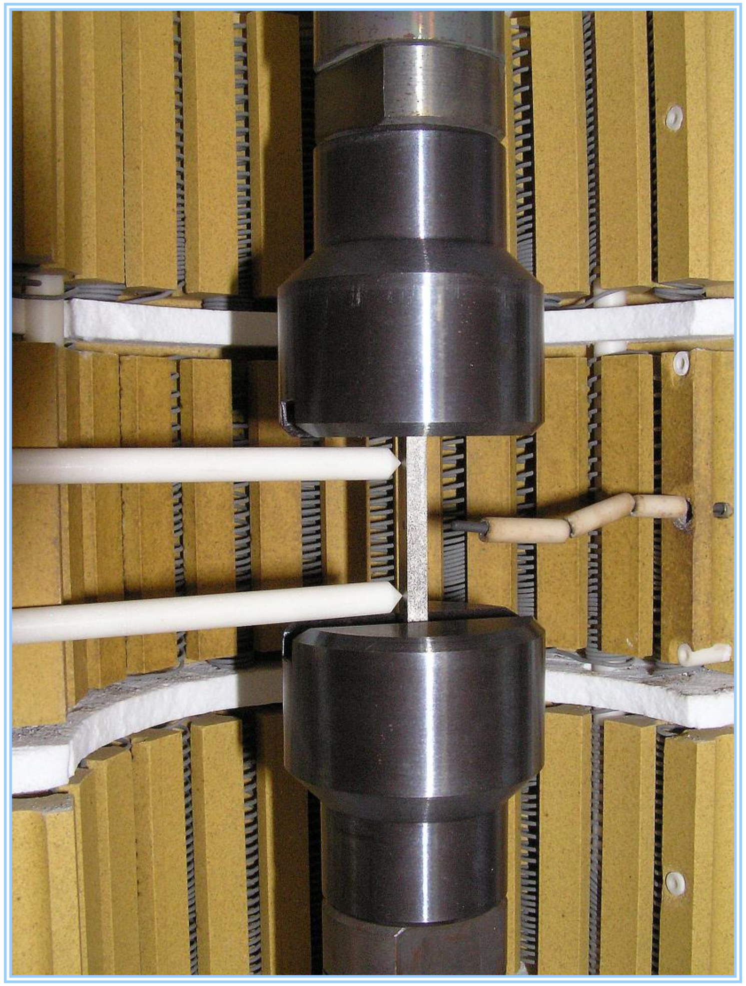


Statistical nature of brittle fracture

- Fractographically supported Weibull diagrams



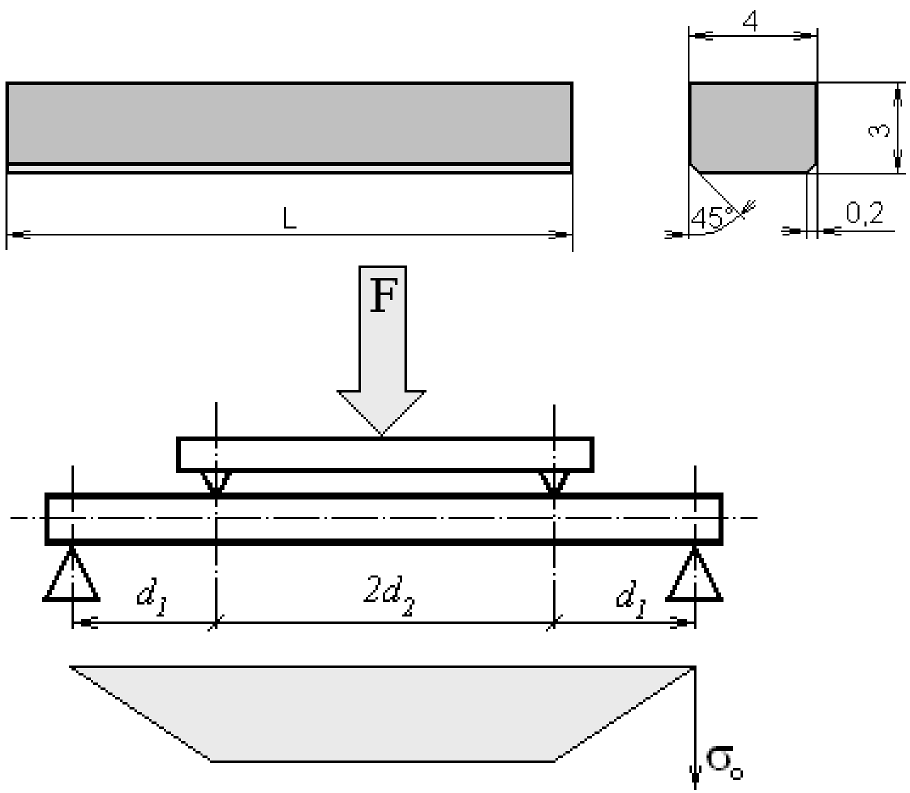
Tensile tests



- Specimen head loaded in compression

  (wedge backplates principle)

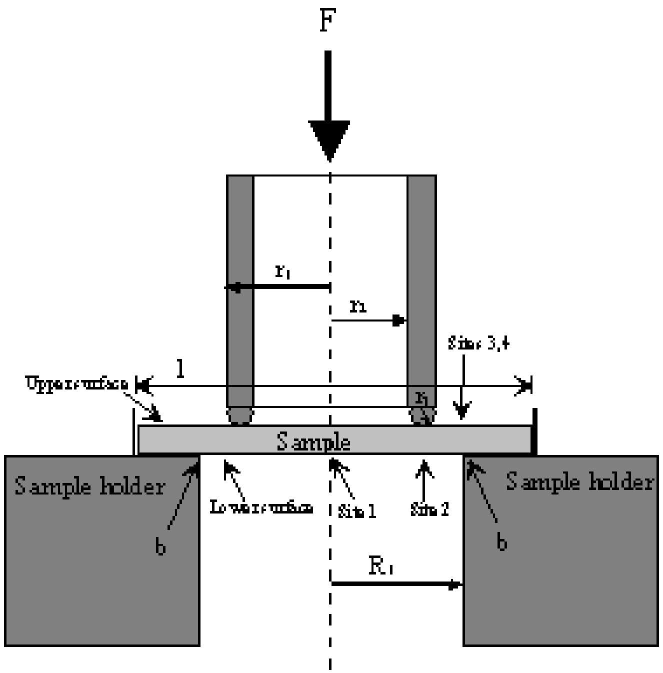
flexural tests

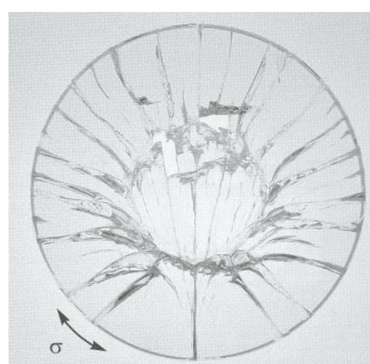


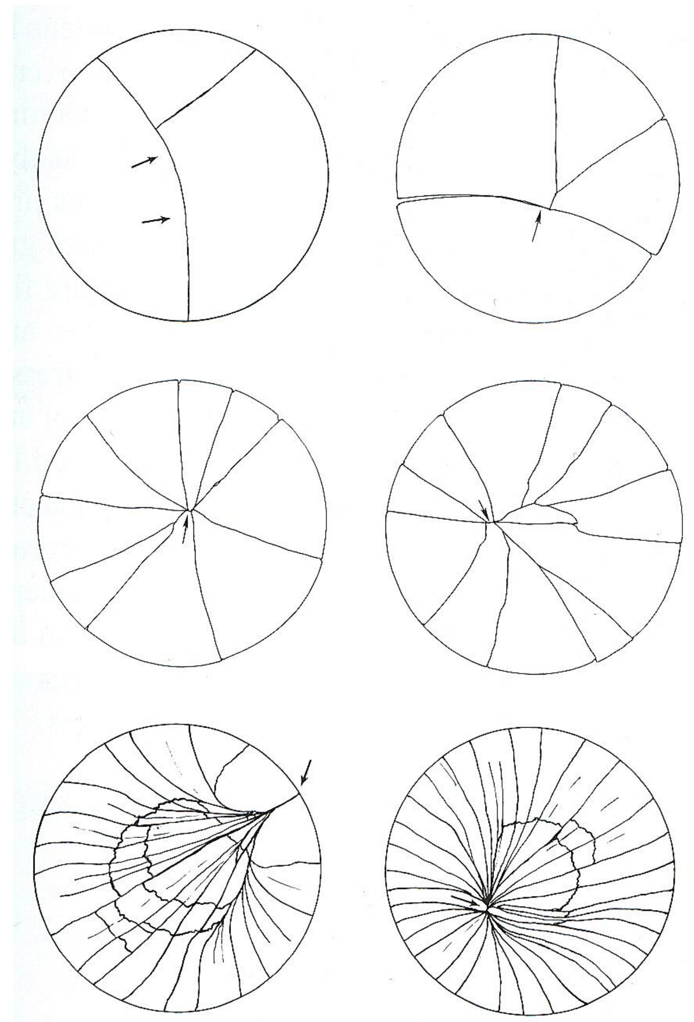
$$W_0 = h^2 b/6$$

$$\sigma_0 = \frac{F d_1}{2W_0} \qquad E = \frac{(F_2 - F_1)\,3 d_1 d_2^2}{(y_2 - y_1)\, b h^3}$$

Biaxial (ring on ring) tests

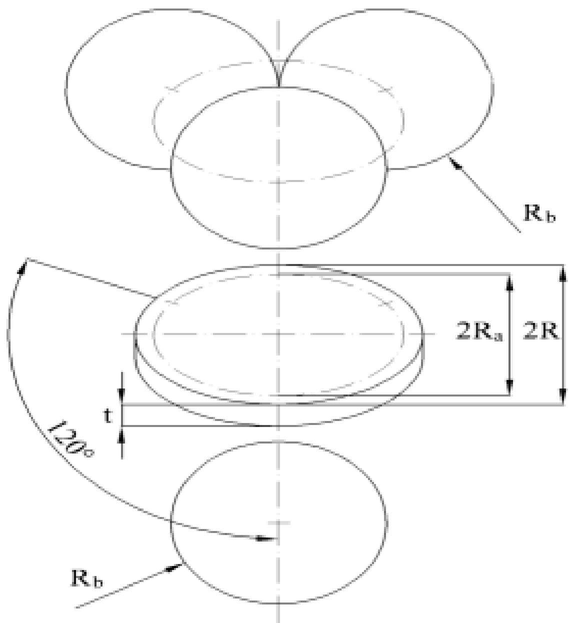






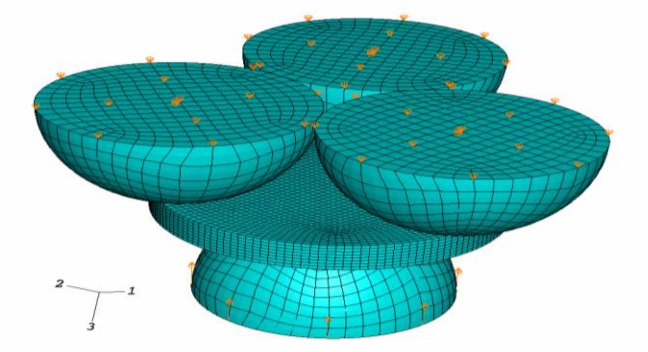
Biaxial (ball on three ball) test

Loading geometry

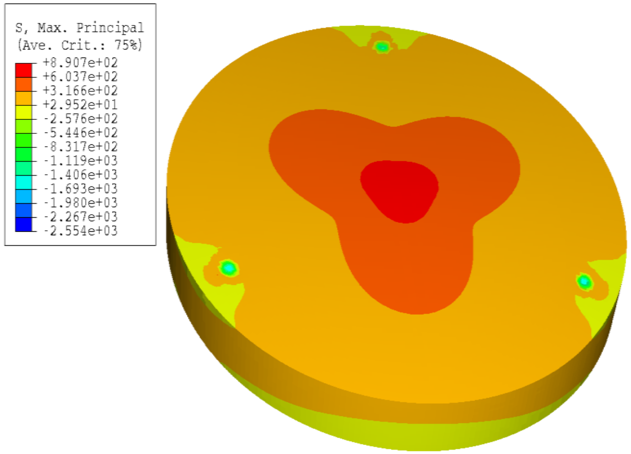


$$\sigma_{\max} = f \cdot \frac{F}{t^2}$$

3D MKP model



Principal stress distributions



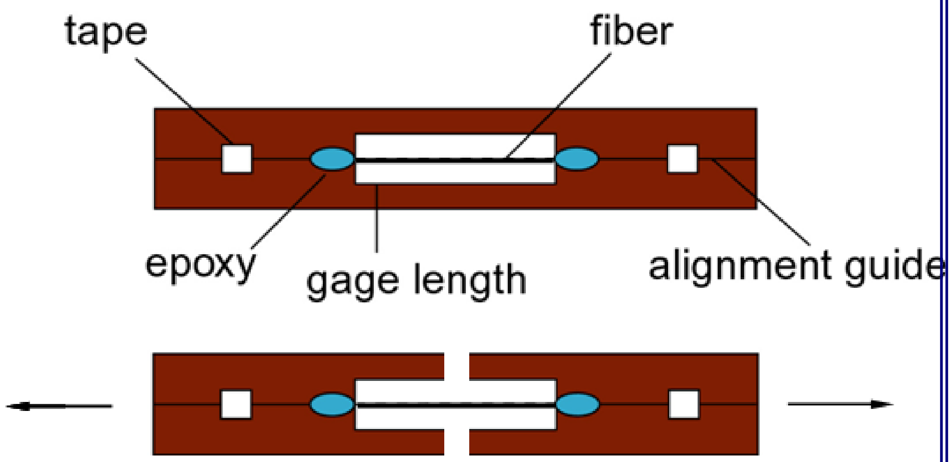
advantages:

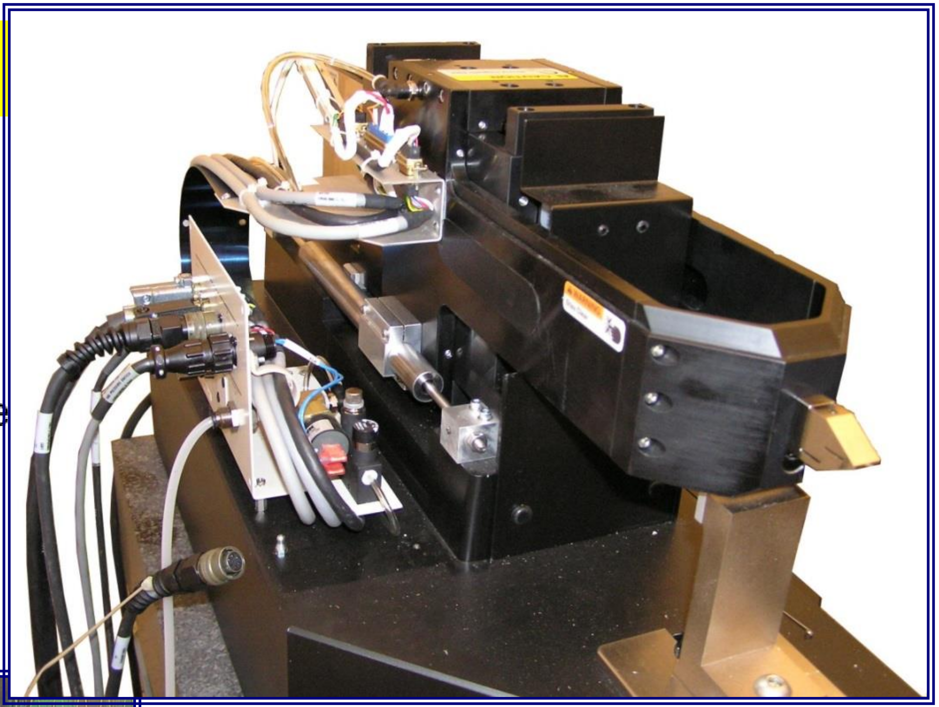
- simplicity
- applicability for small samples

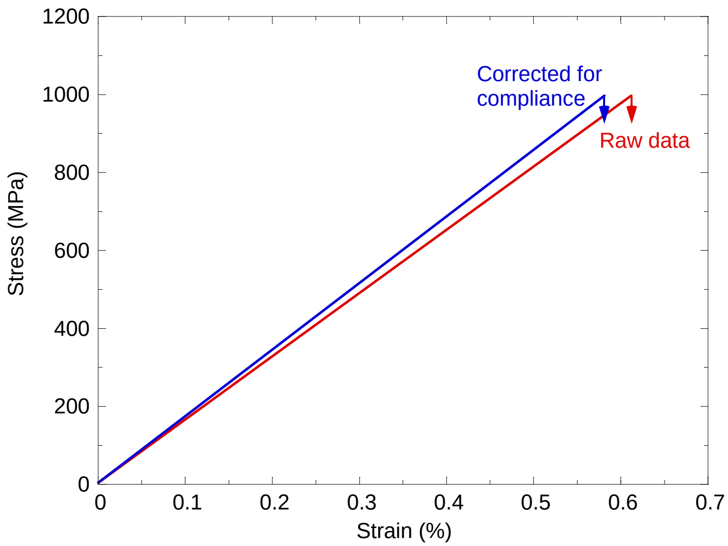
disadvantages:

- necessity to calculate calibration function
- poor applications (limited number of labs)

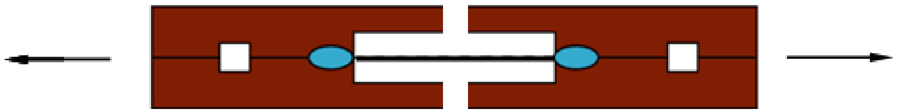
Tensile test of fibres

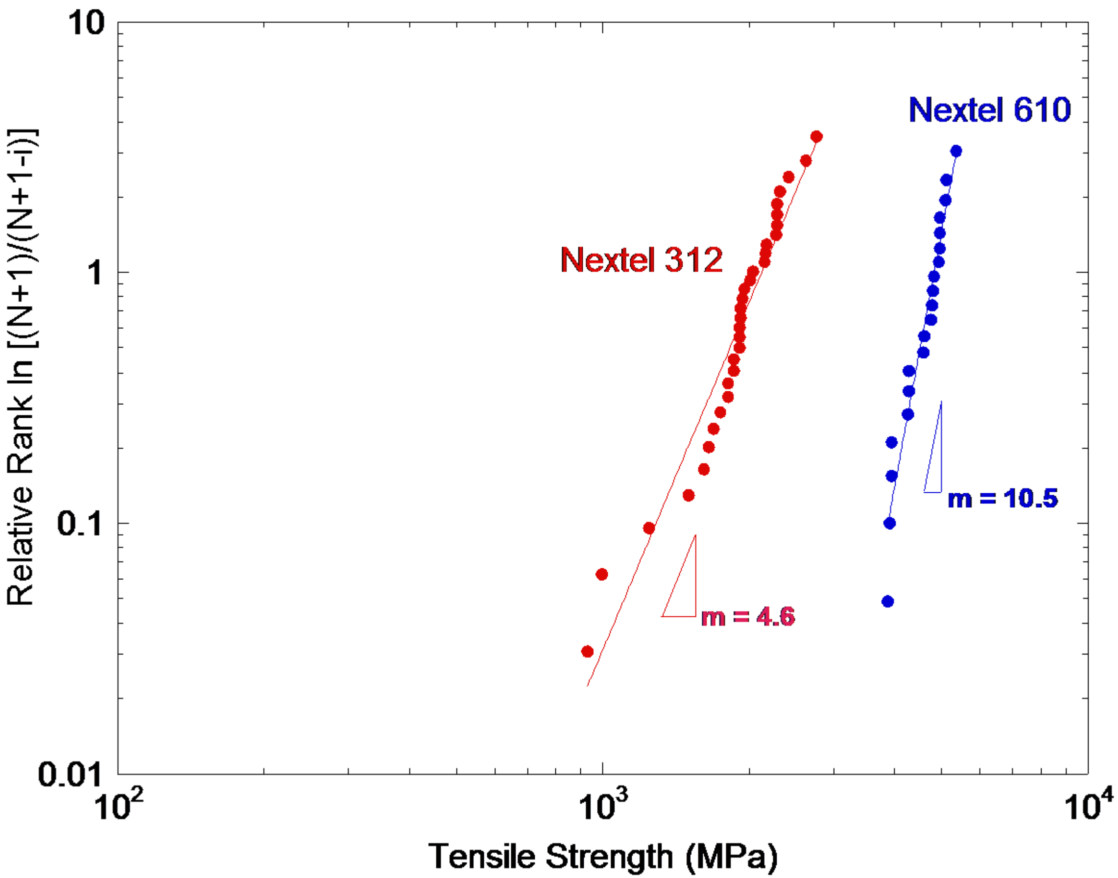


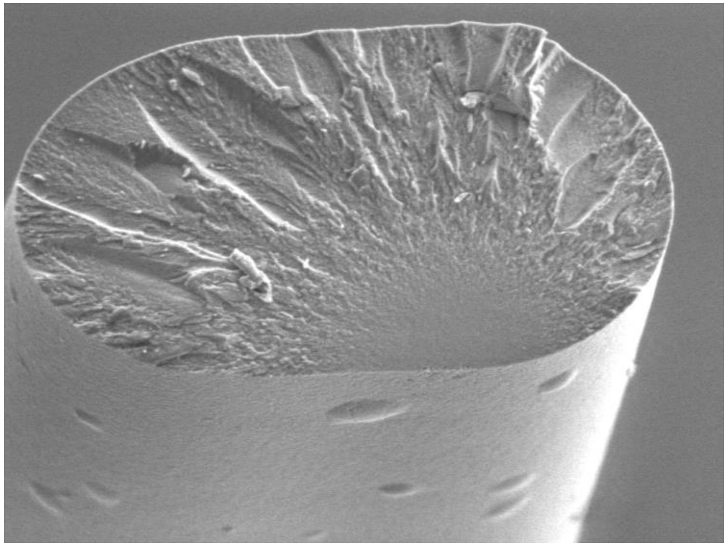


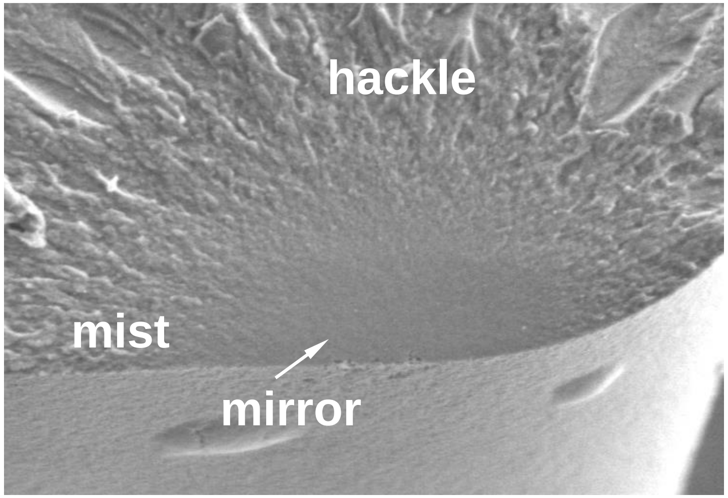


Tensile test of fibres

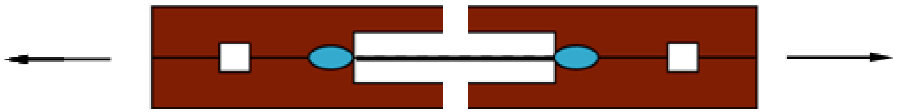


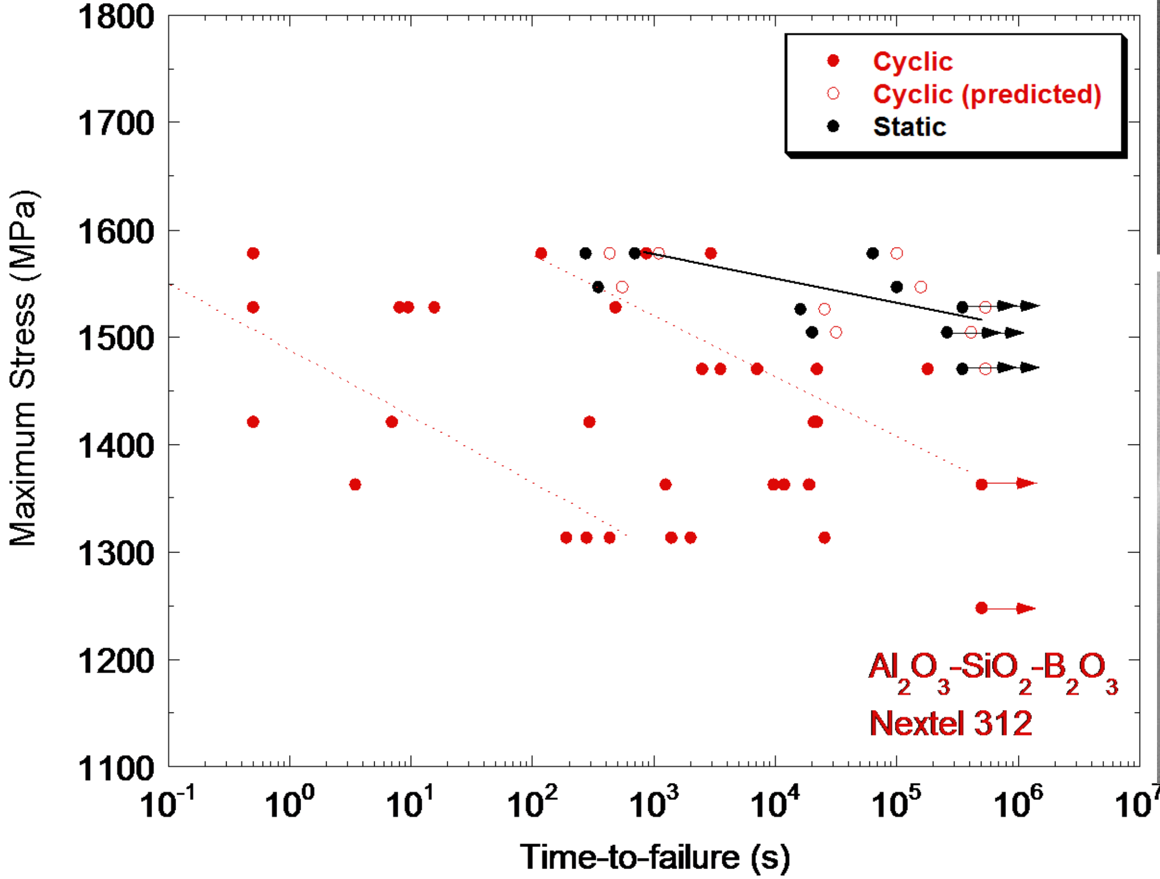


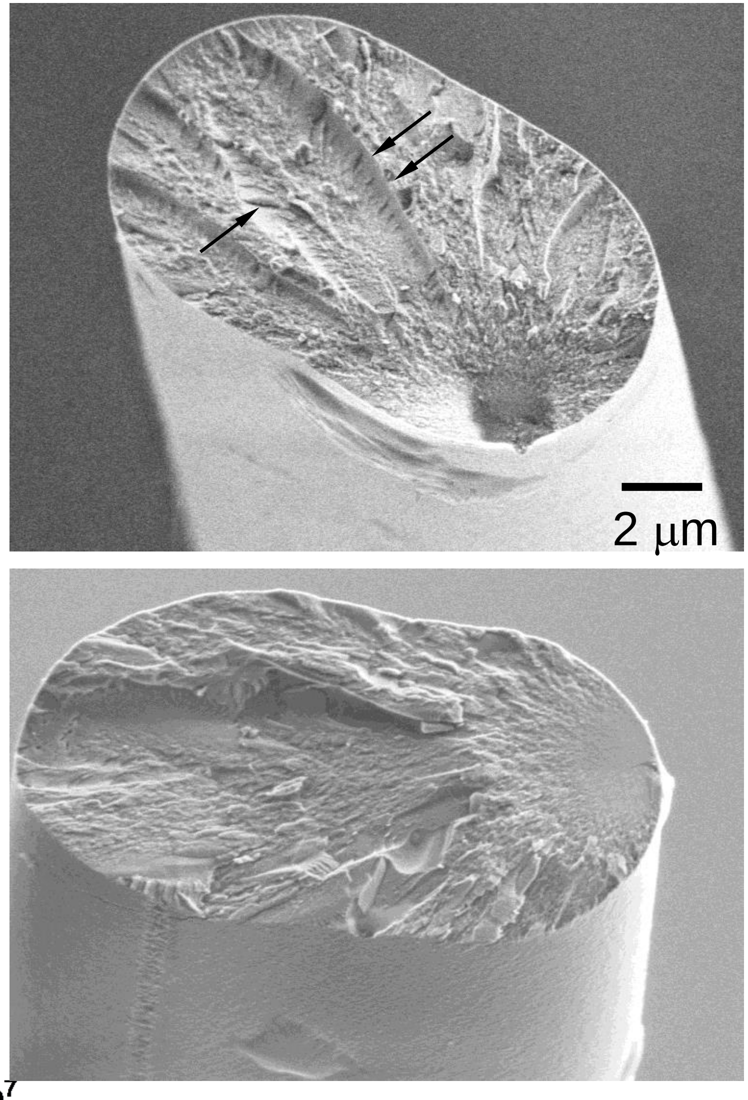




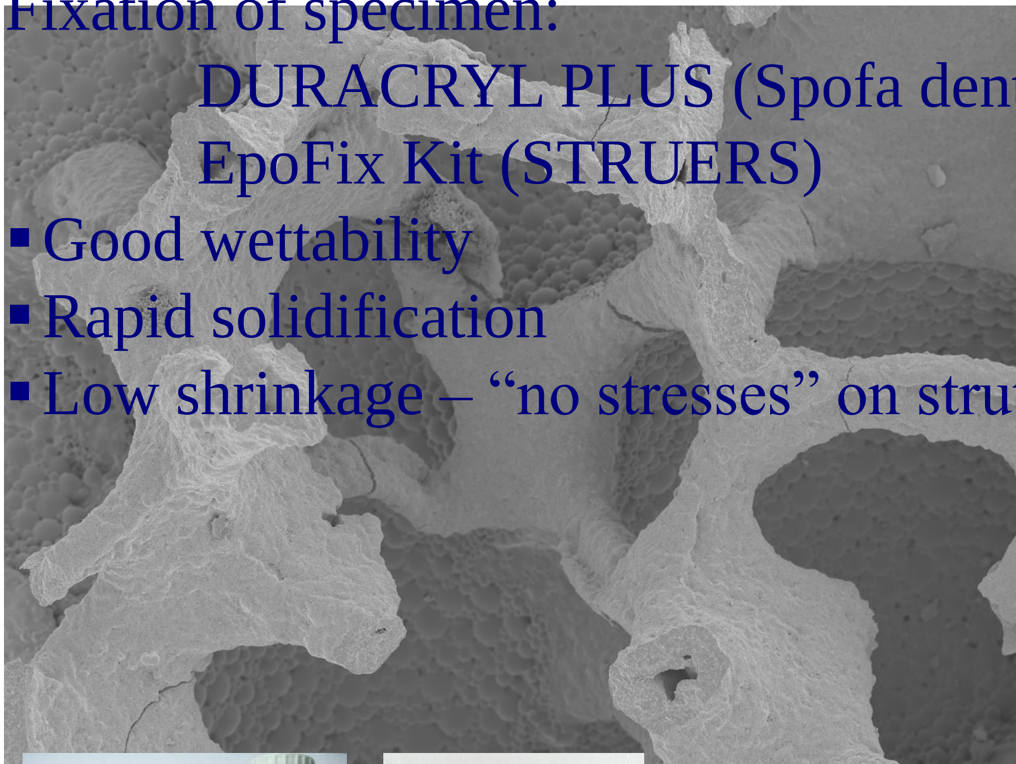
Fatigue tests of fibres







Mechanical response of foam ceramics

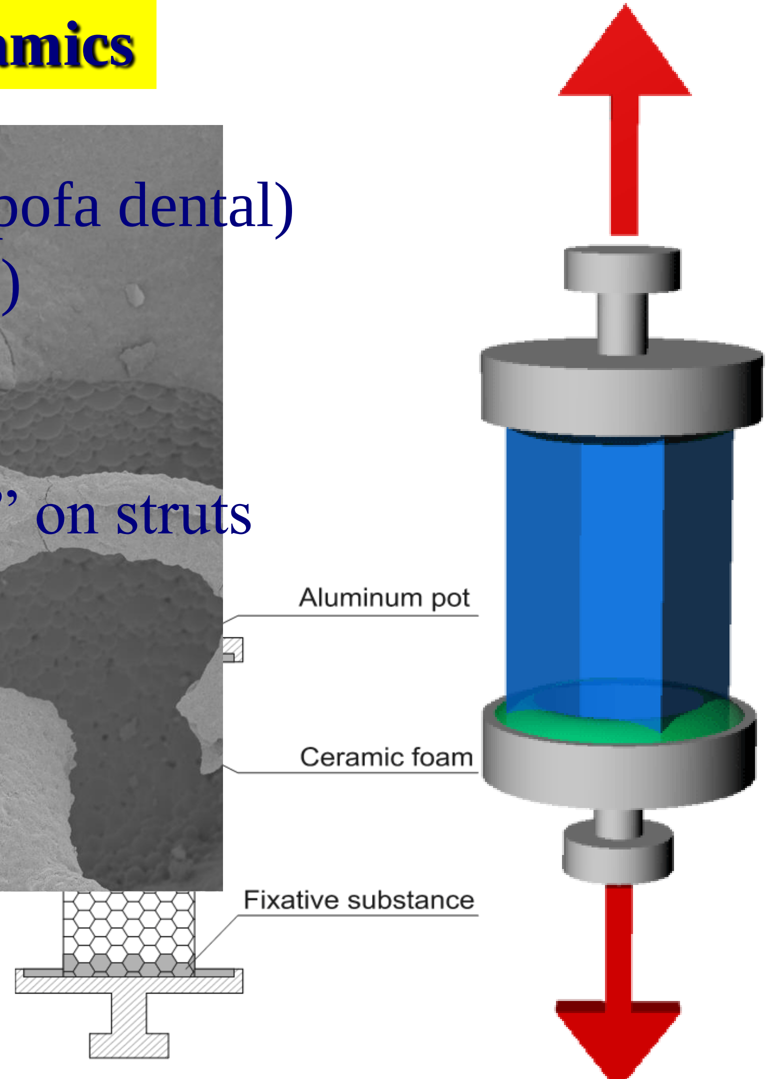


Fixation of specimen:

DURACRYL PLUS (Spofa dental)

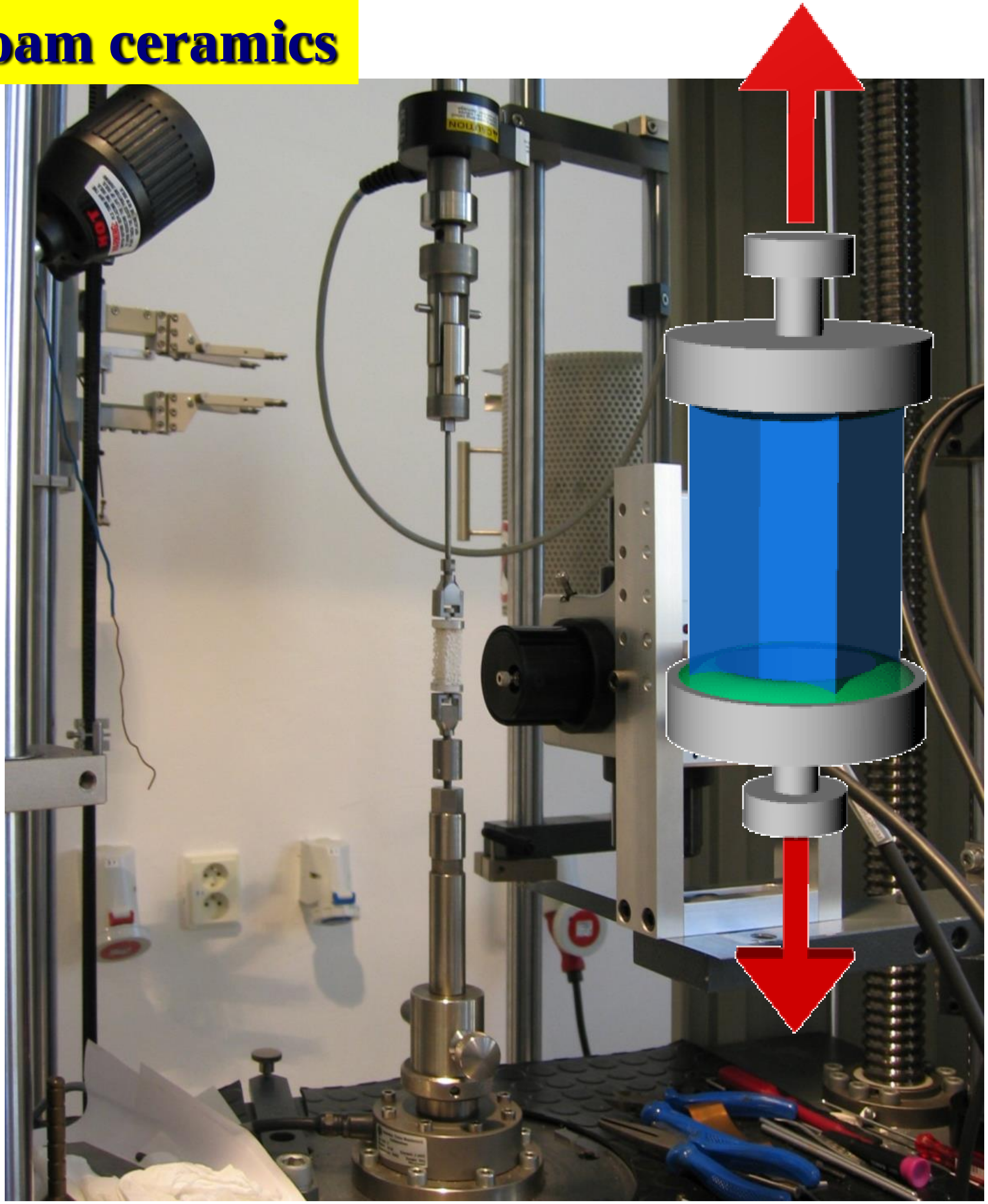
EpoFix Kit (STRUERS)

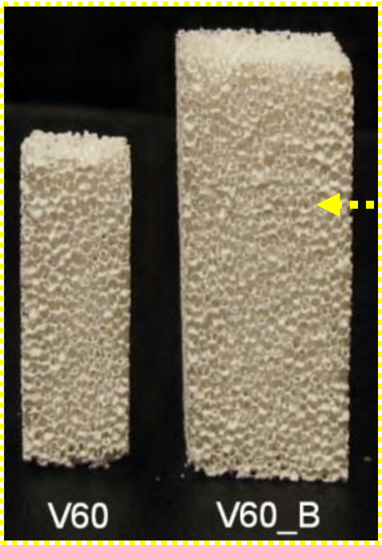
- Good wettability
- Rapid solidification
- Low shrinkage – “no stresses” on struts

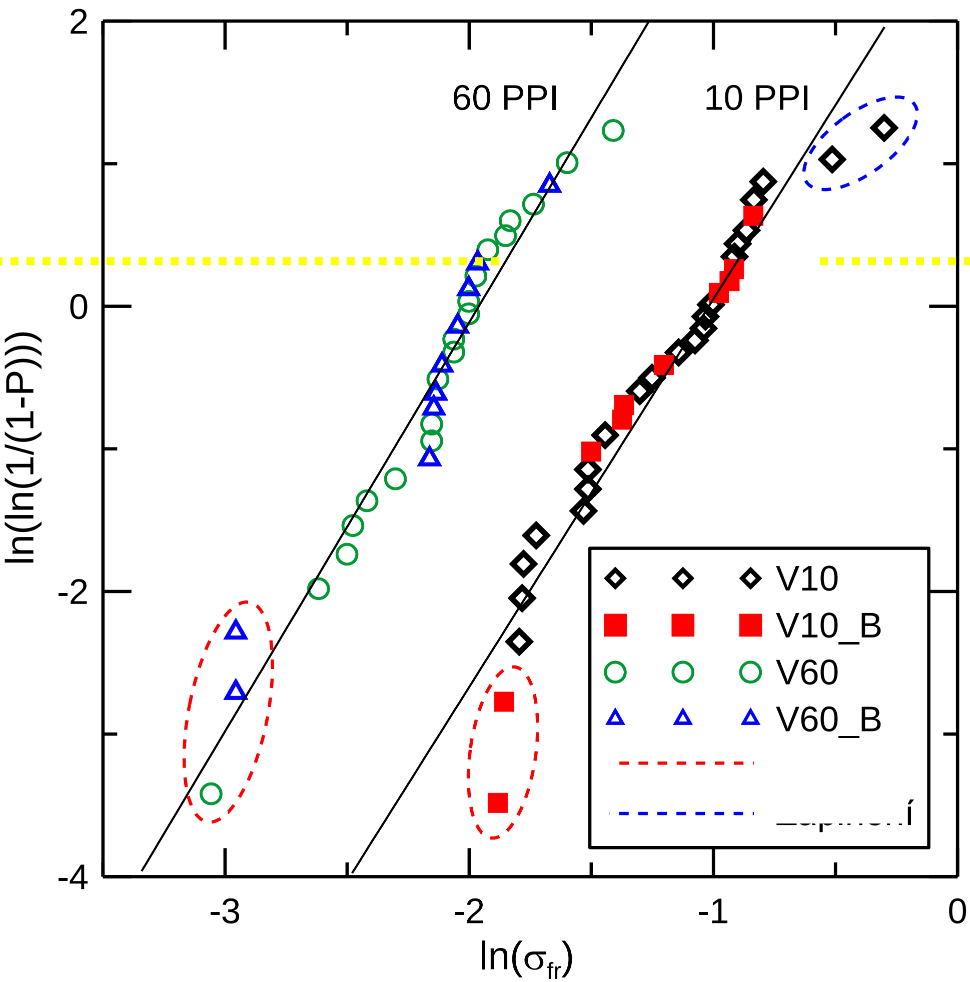


a)

Mechanical response of foam ceramics



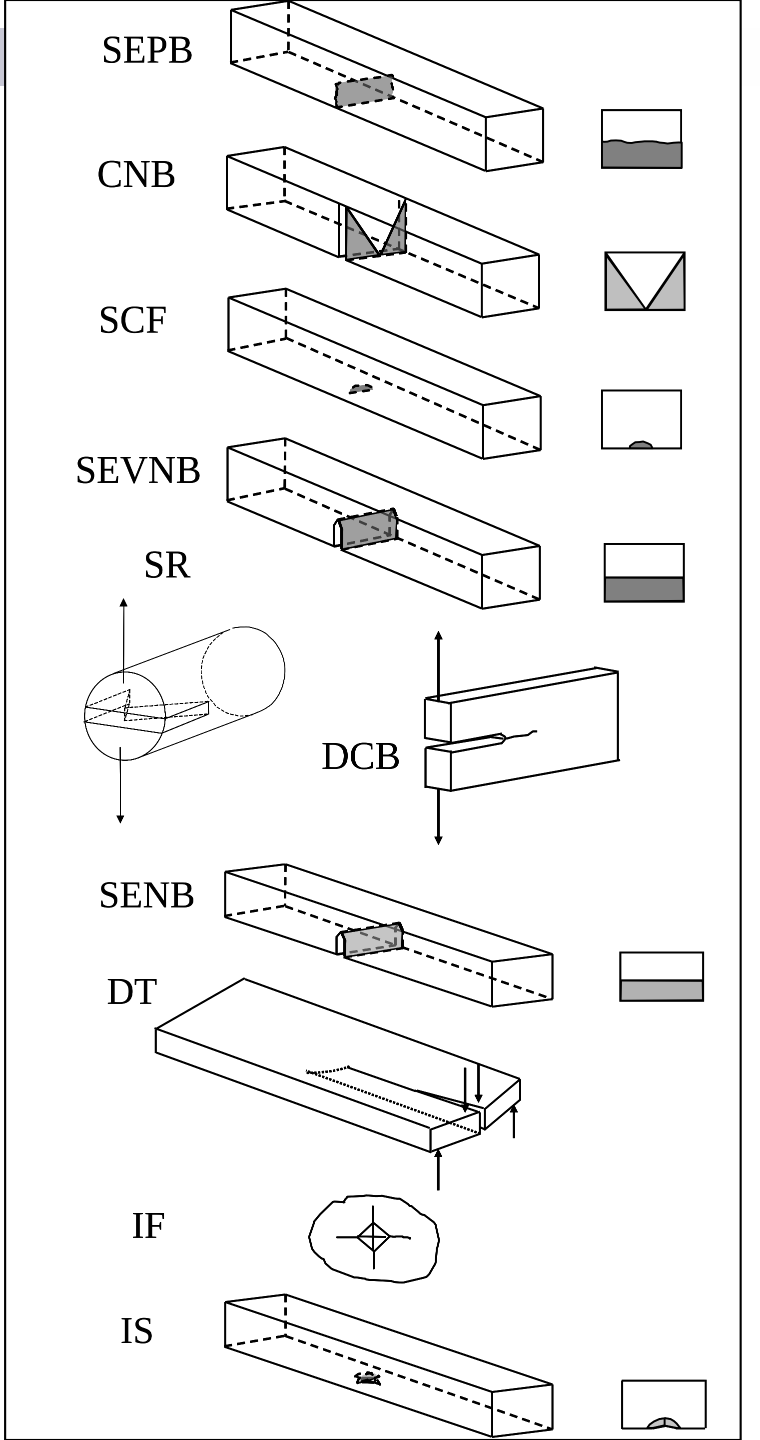

V60
V60_B


60 PPI
10 PPI
V10
V10_B
V60
V60_B
ln(ln(1/(1-P)))
ln(σ_fr)
2
0
-2
-4
-3
-2
-1
0


V10_B
V10

Fracture touhgness determination

- Several standardised methods:
  - SEPB (ASTM, JIS, ISO, CEN)
  - CNB (ASTM, ISO, CEN)
  - SCF (ASTM, ISO, CEN)
  - SEVNB (CEN, ISO (DIS)
  - SR (difficult – needs special machine)
- … and some methods which will not be standardised:
  - DCB (too expensive)
  - SENB (notch too blunt)
  - DT (uncertain calibrations – mixed mode)
  - IF (subjective measurement, residual stress)
  - IS (flaw shape and residual stress uncertainty)



Fracture touhgness determination

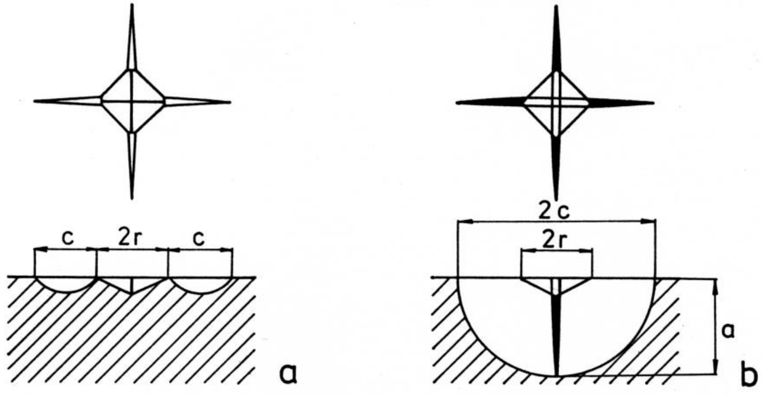
Indentation toughness – Vickers, Knoop

Flexural tests of bars with crack initiator

- sharp crack by cyclic loading
- surface crack by indentation
- surface crack by bridge method
- straight sharp notch (SENB)
- chevron notch (CVN)

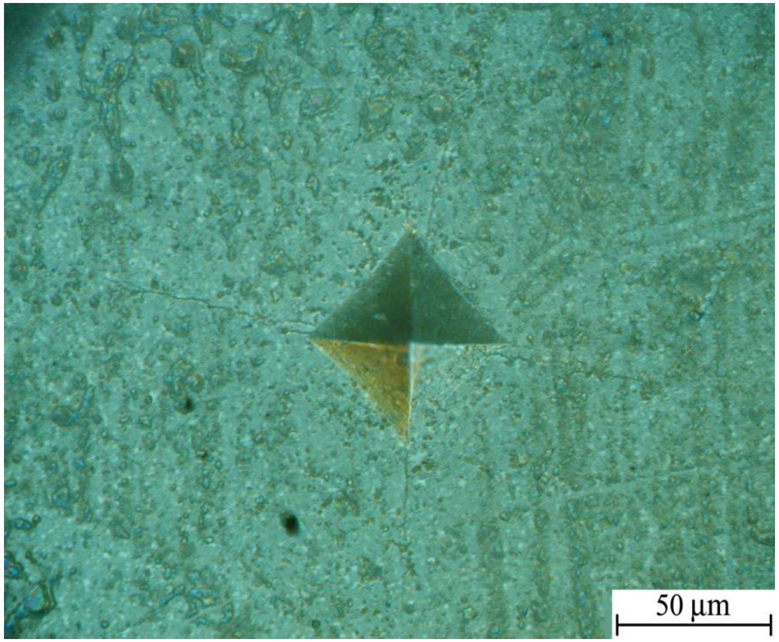
Eccentric tension of short rods and bars

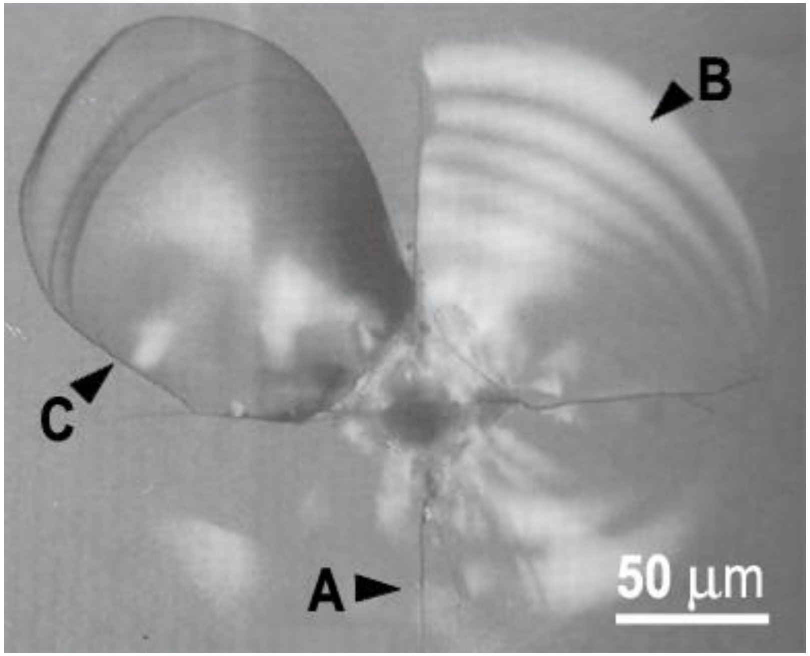
Indentation toughness



$$K_{IC} = 0{,}035\left(\frac{c}{r}\right)^{-1/2}\left(\frac{Ek}{HV}\right)^{2/5}\frac{HVr^{1/2}}{k}\left[MPam^{1/2}\right]$$

$$K_{IC} = 0{,}129\left(\frac{c}{r}\right)^{-3/2}\left(\frac{Ek}{HV}\right)^{2/5}\frac{HVr^{1/2}}{k}\left[MPam^{1/2}\right]$$

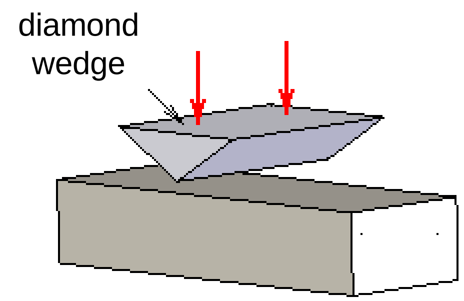


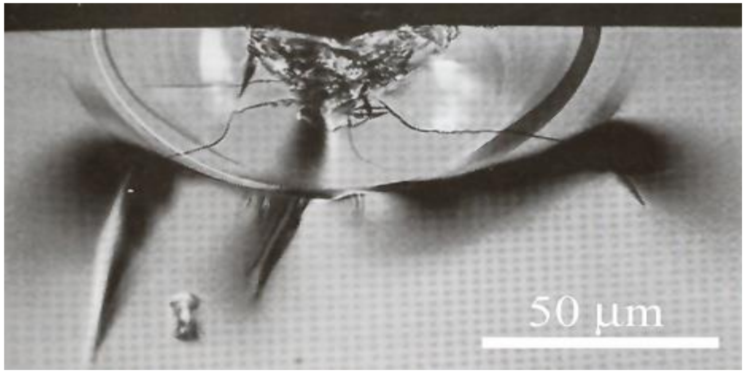


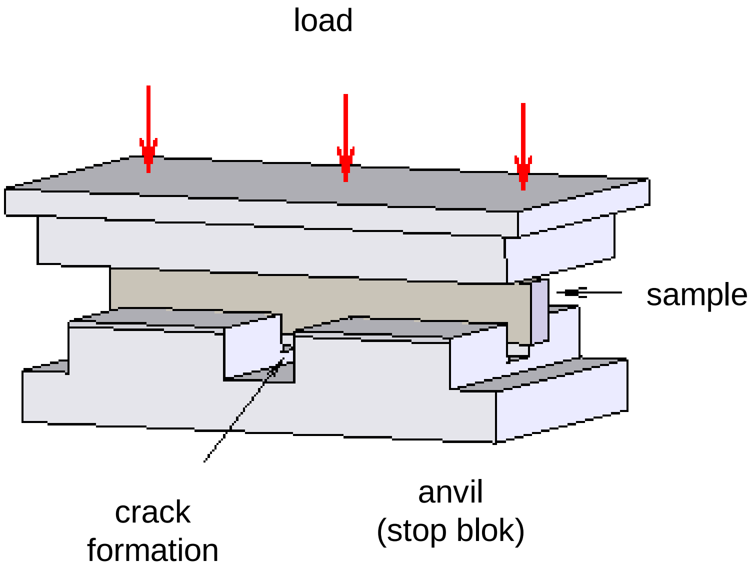
Flexural tests

How to prepare the crack starter

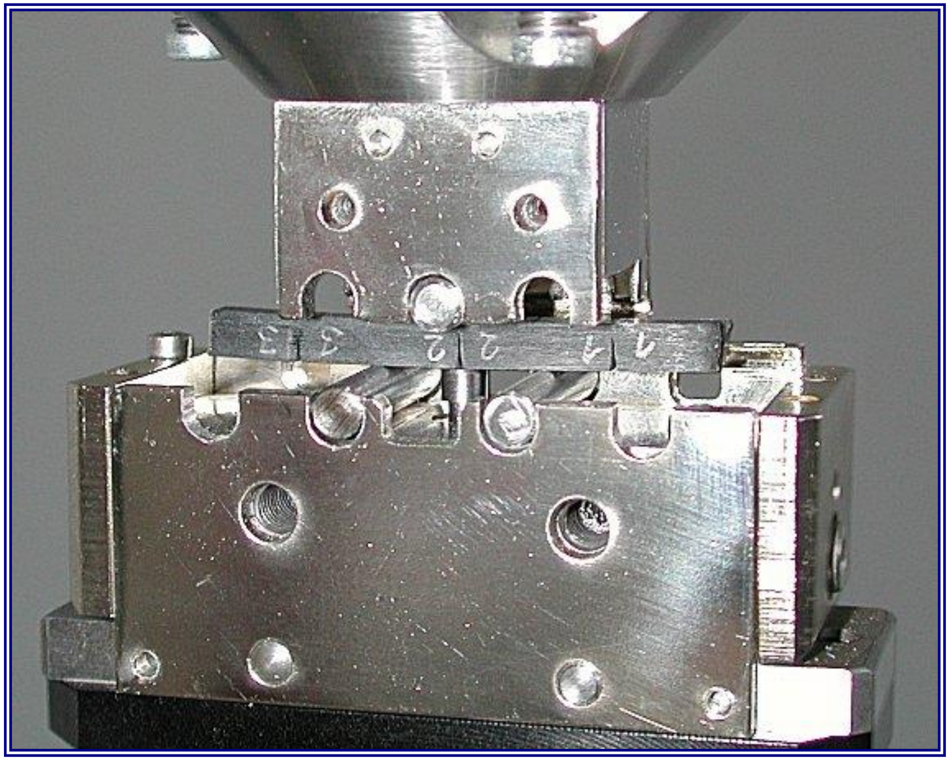
- wedge forced into the tensile surface
- indentation & flexural test (indentation strength)
- bridge technique for precracking (prescribed deflection)





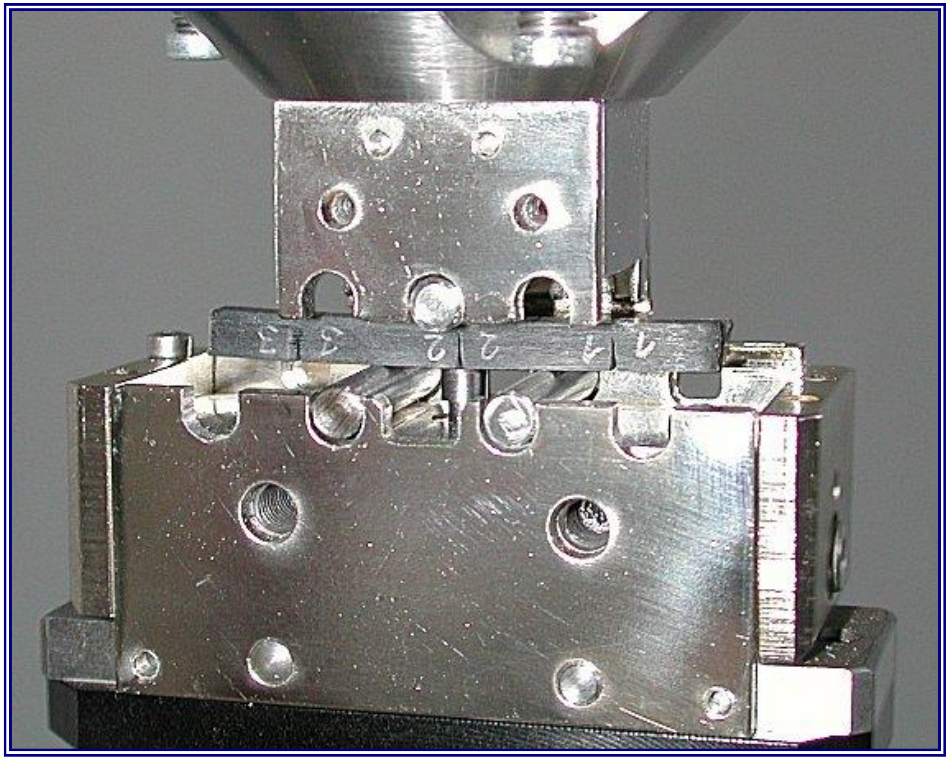


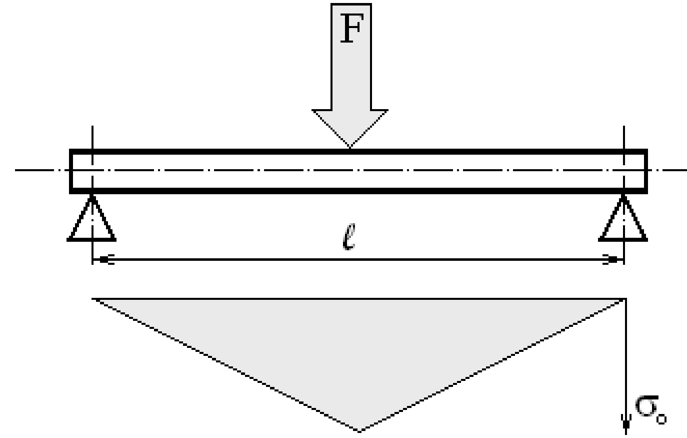
Flexural tests

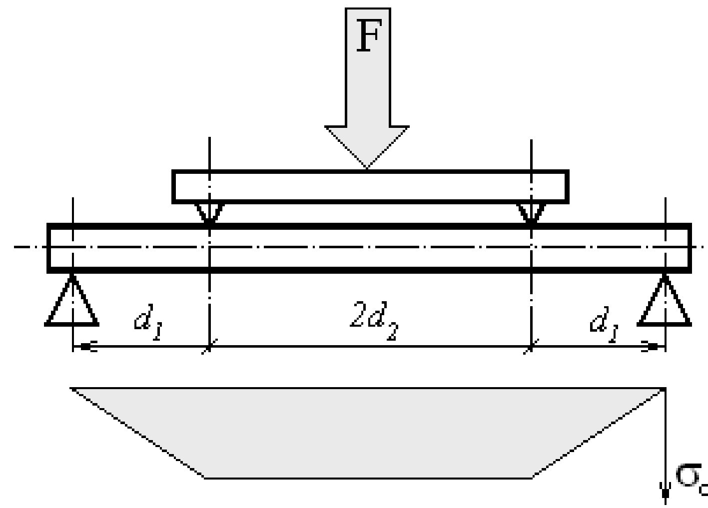


- 3 (4) point bending
- direct flexure measurements
- acoustic emission analysis
- applicable at high temperatures
- how to prepare the starting crack (and evaluate the crack length)

Flexural tests







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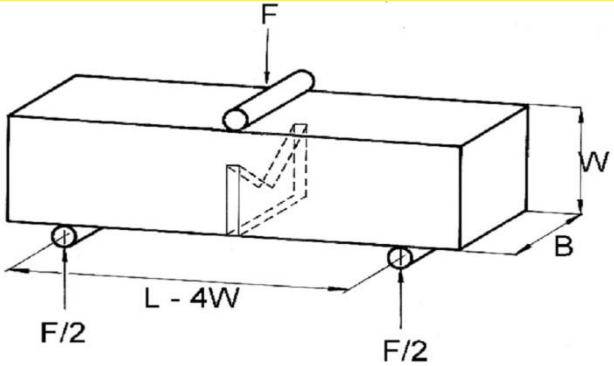
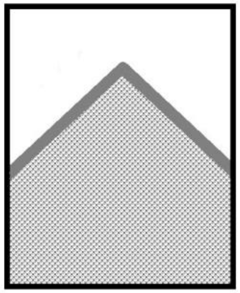
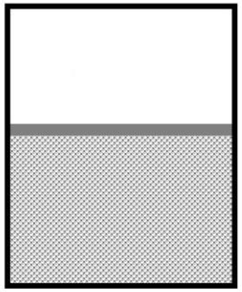
Flexural tests



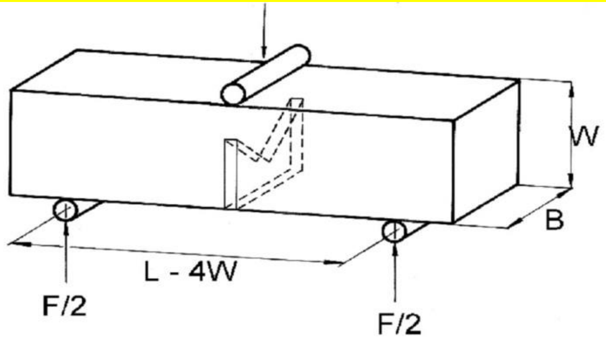
$$K_{Ic} = \frac{Y^*_{min} \, F_M}{B \, W^{1/2}}$$



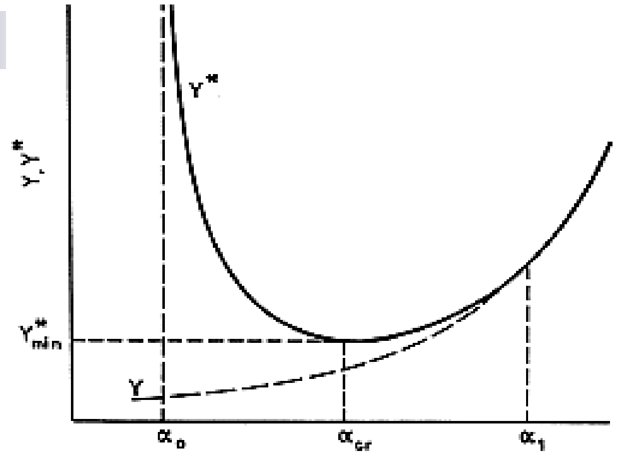
$$K_{Ic} = \frac{F_c \, Y}{B \, W^{1/2}}$$

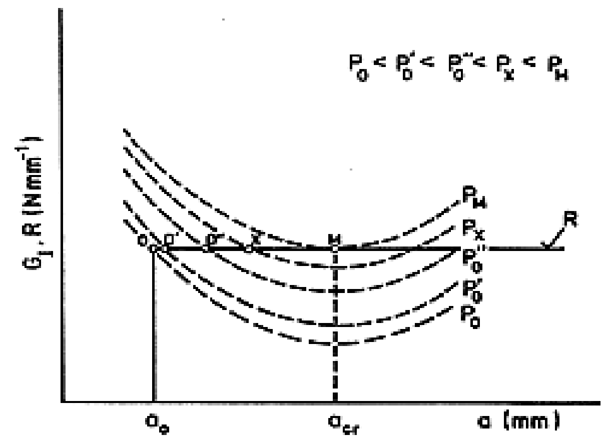


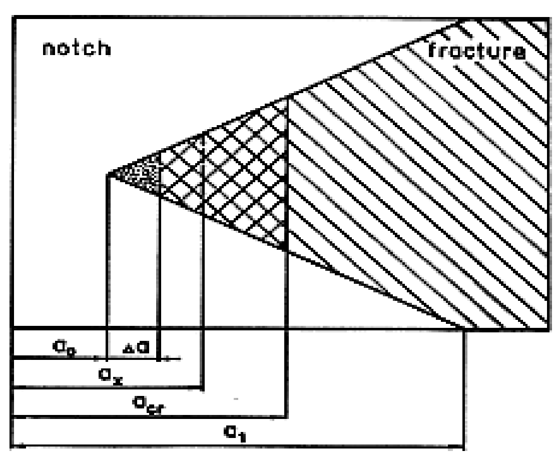
Flexural tests



$$K_{Ic} = \frac{Y^*_{min} F_M}{B W^{1/2}}$$







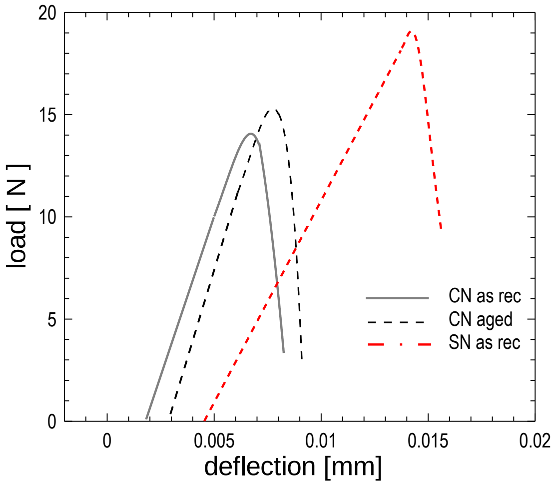
Excelent pre-requisities of the chevron notch for fracture toughness determination:

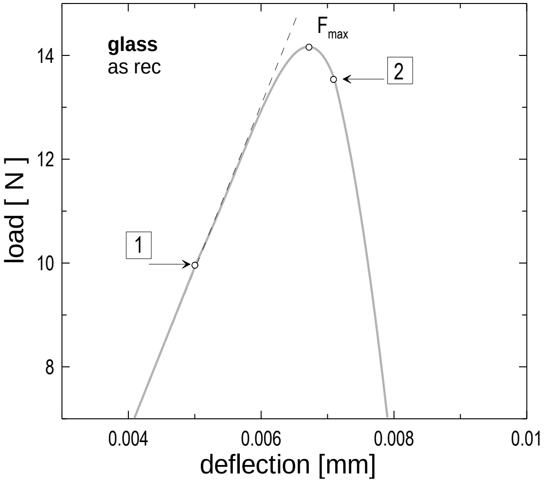
- it is not necesarry to prepare the crack and measure the crack length after the test
- there is a stable crack propagation, the crack is kept in this regime by increasing crack tip length (crack driving force is compensated by increasing crack tip length)
- crack trajectory (fracture plane) is kept in chevron notch plane

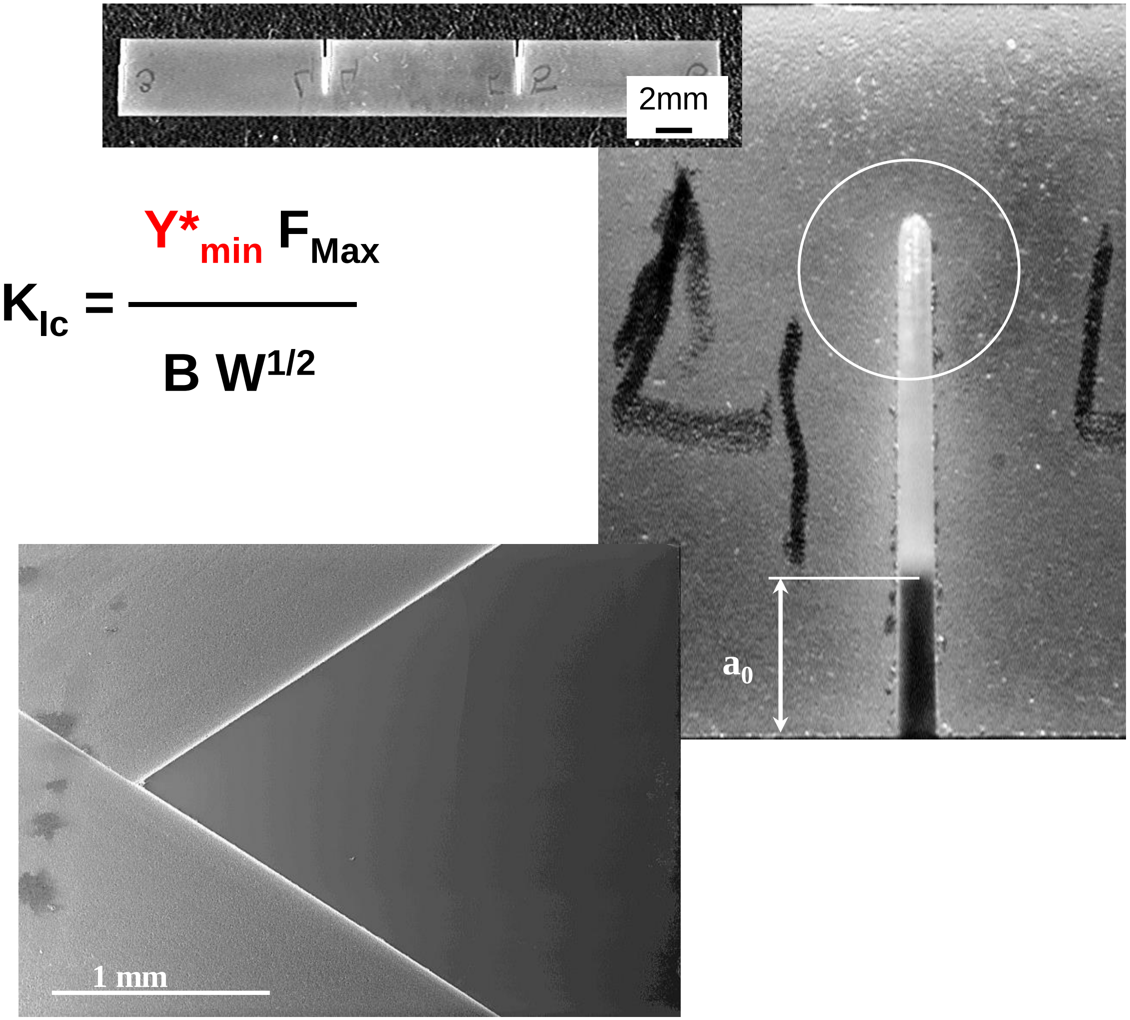
Flexural tests

Load-deflection traces

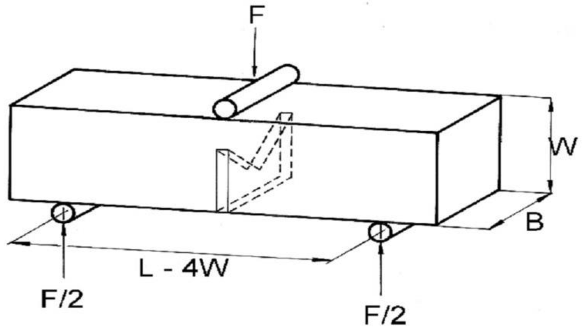
Specimen after test



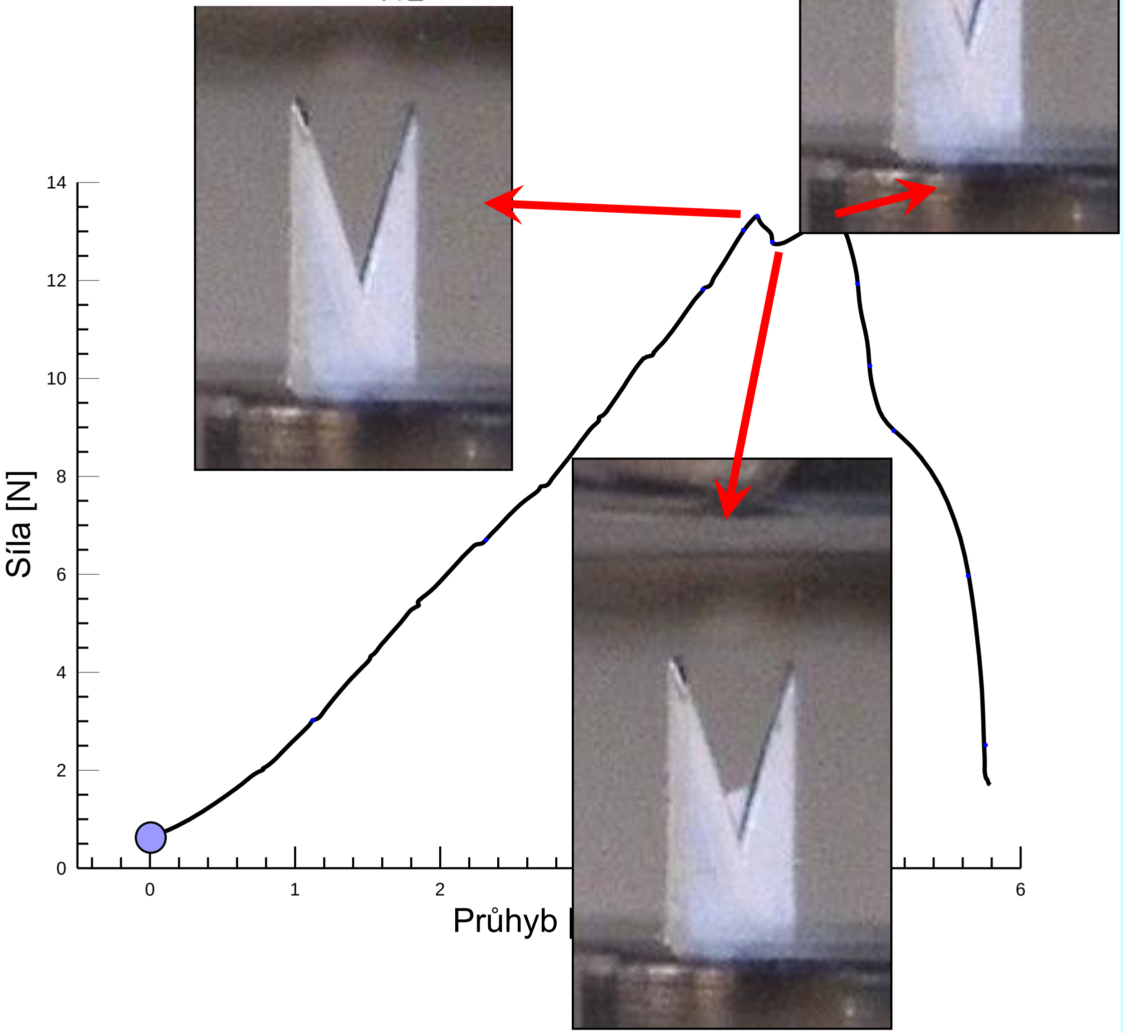




$$K_{Ic} = \frac{Y^*_{min}\, F_{Max}}{B\, W^{1/2}}$$



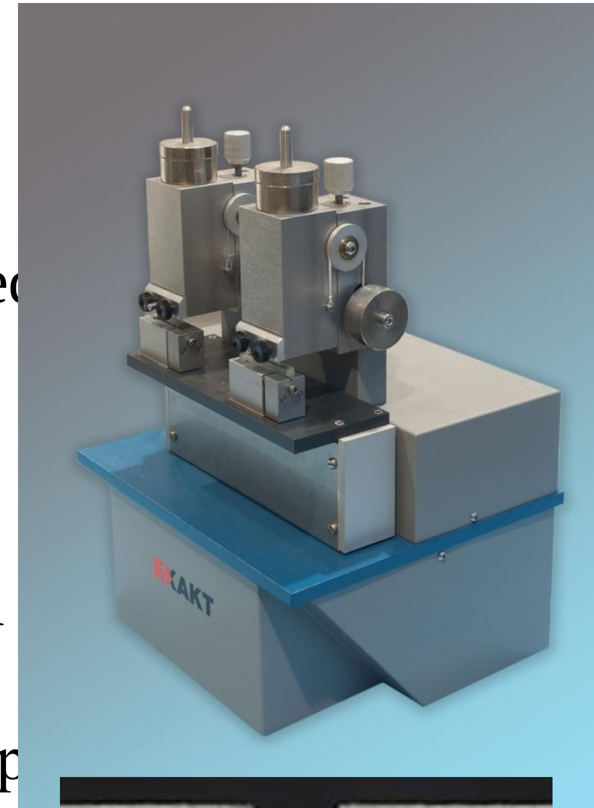
F
W
B
L - 4W
F/2
F/2

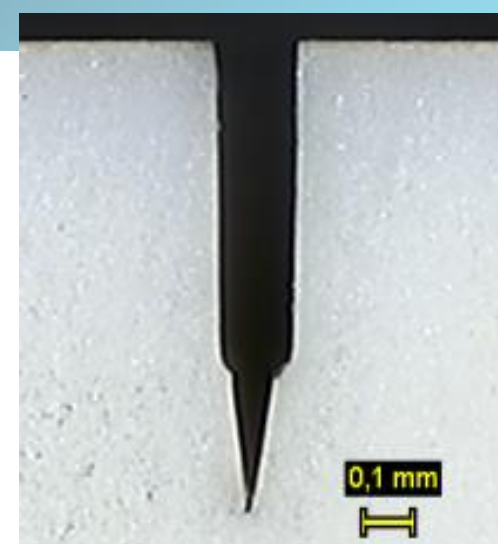


14
12
10
8
6
4
2
0
Síla [N]
0
1
2
6
Průhyb [

Flexural tests

- Straightforward to produce a very sharp-tipped notch using a small notching machine
- Does not require skill in pre-cracking
  (needed for the SEPB method)
- Straightforward measurement – always gets a result
- But… the notch root is still not a perfect sharp crack
  - Does not work well for toughened ceramics (Y-TZP, fiber reinforced etc.) !!!
  - Tends to give a small overestimate

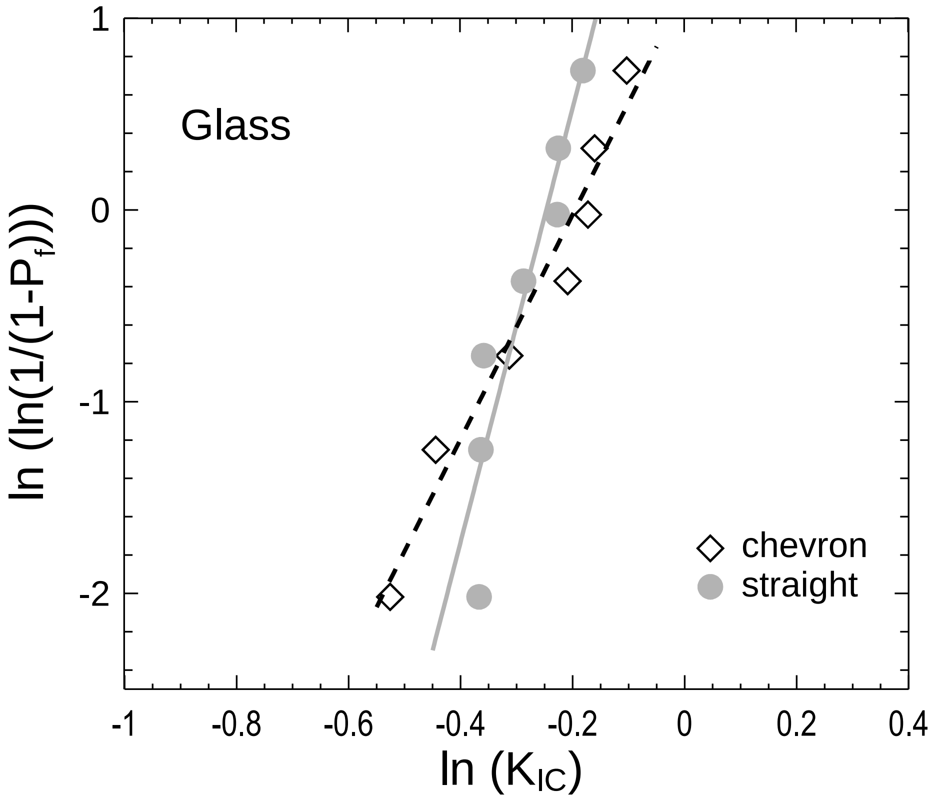


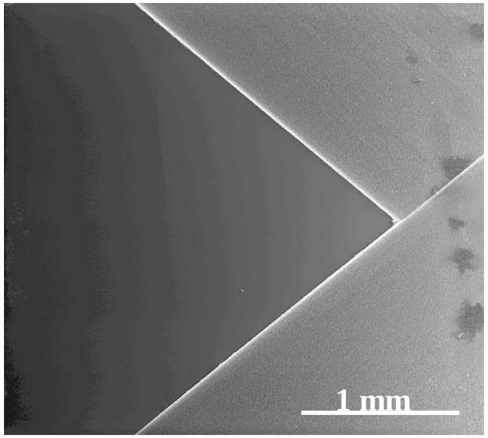


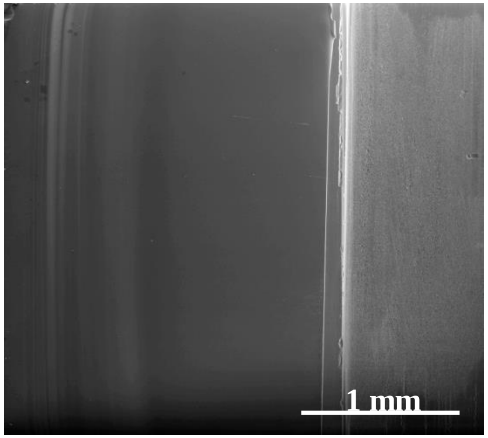
Flexural tests

Comparison of fracture toughness values

K_{IC}	for chevron notch	0.67 $MPam^{0,5}$
	for straight notch	0.69 $MPam^{0,5}$

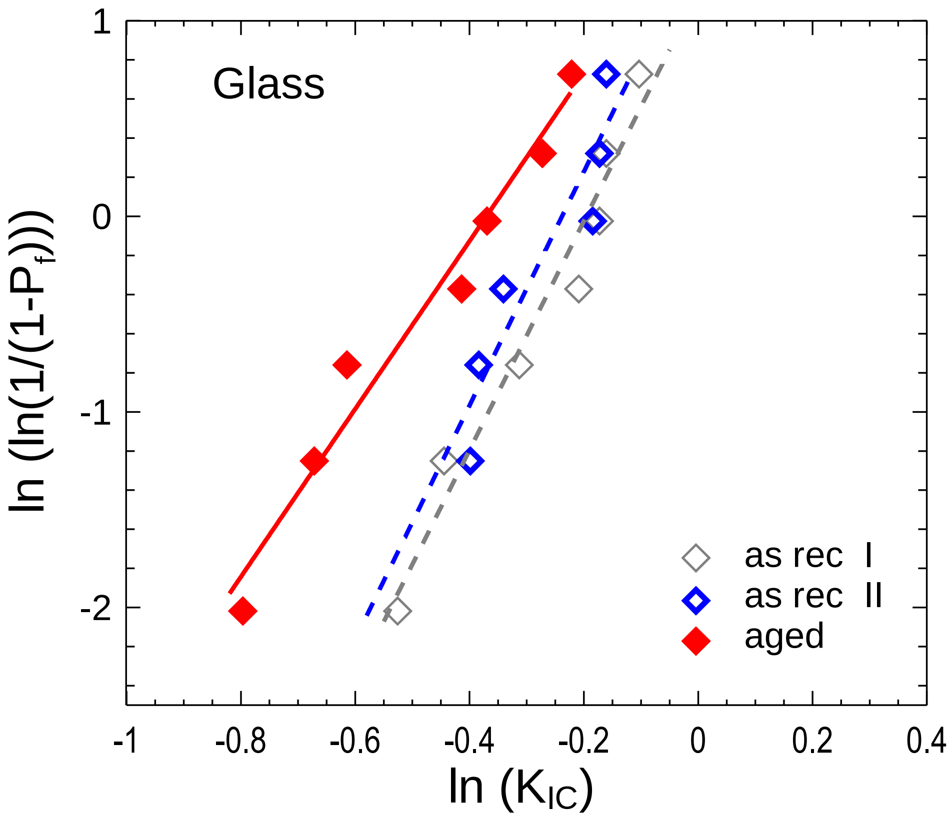






Flexural tests

Effect of ageing on fracture toughness of borosilicate glass



- **as rec II = as rec I after 2 years**
- **aged - at 550 ºC for 20 hrs**

Very subtle differences in fracture toughness values (0.64 and 0.58 MPam$^{1/2}$ before and after annealing) can be detected CN technique

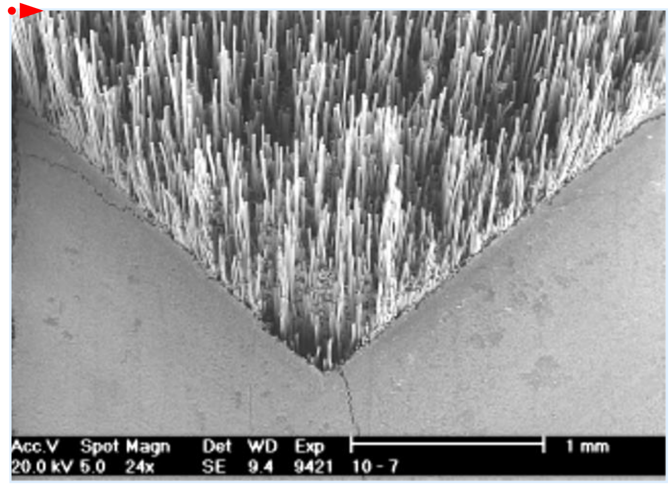
Flexural tests

- Chevron notched sp. technique

  - crack trajectory controlled by chevron notch

  - critical (fracture) condition reached for running crack

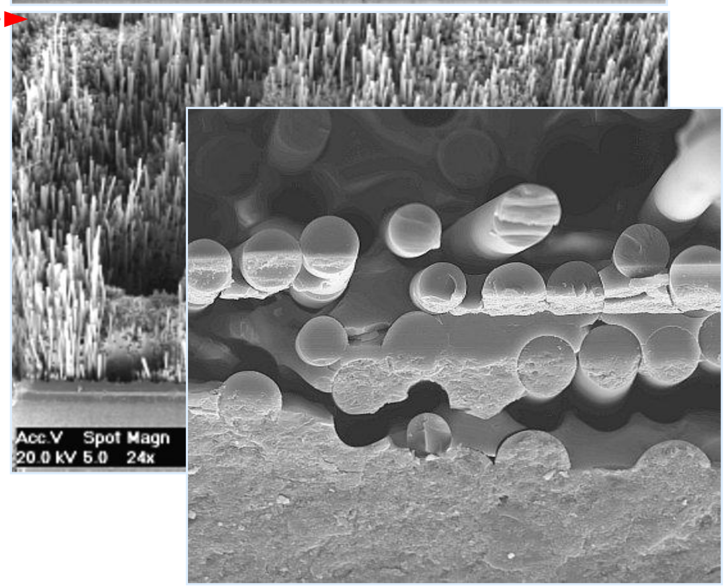
  - uniform fibre pull-out



- Straight notched sp. technique (simulating a sharp crack)

  - premature crack localisation along fibre (bundles) interface

  - in almost cases at the sharp notch root

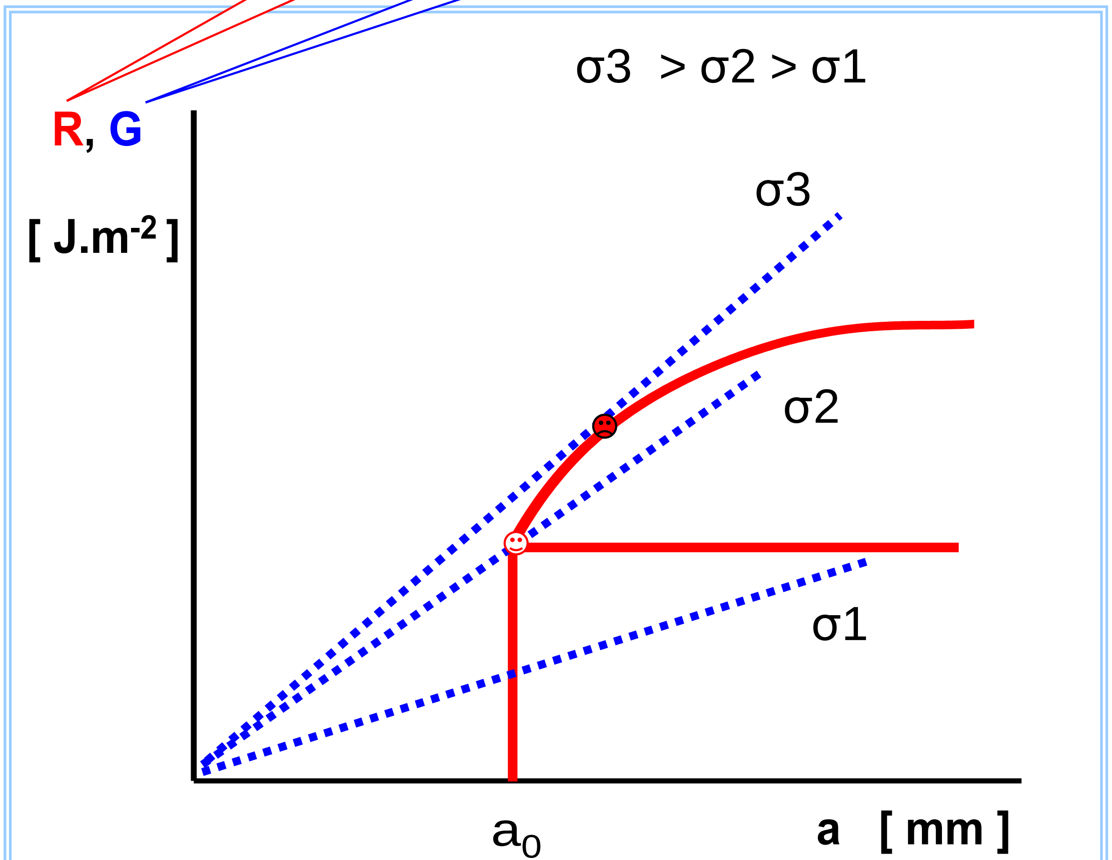


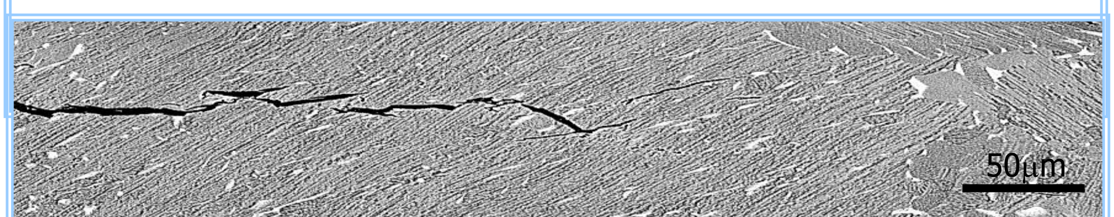
Energy needed for creation of new surfaces $R = dW_s/dA$

crack resistance

crack driving force

Work of applied load in dependence on surface increment $G = -(dW_{el}/dA)$



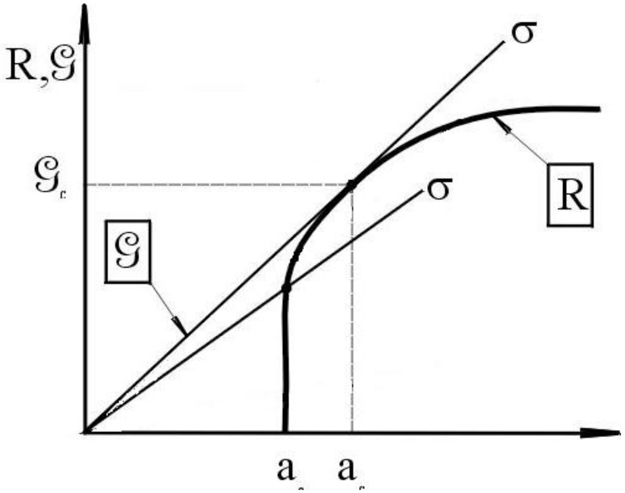


R - curve shape = material characteristics

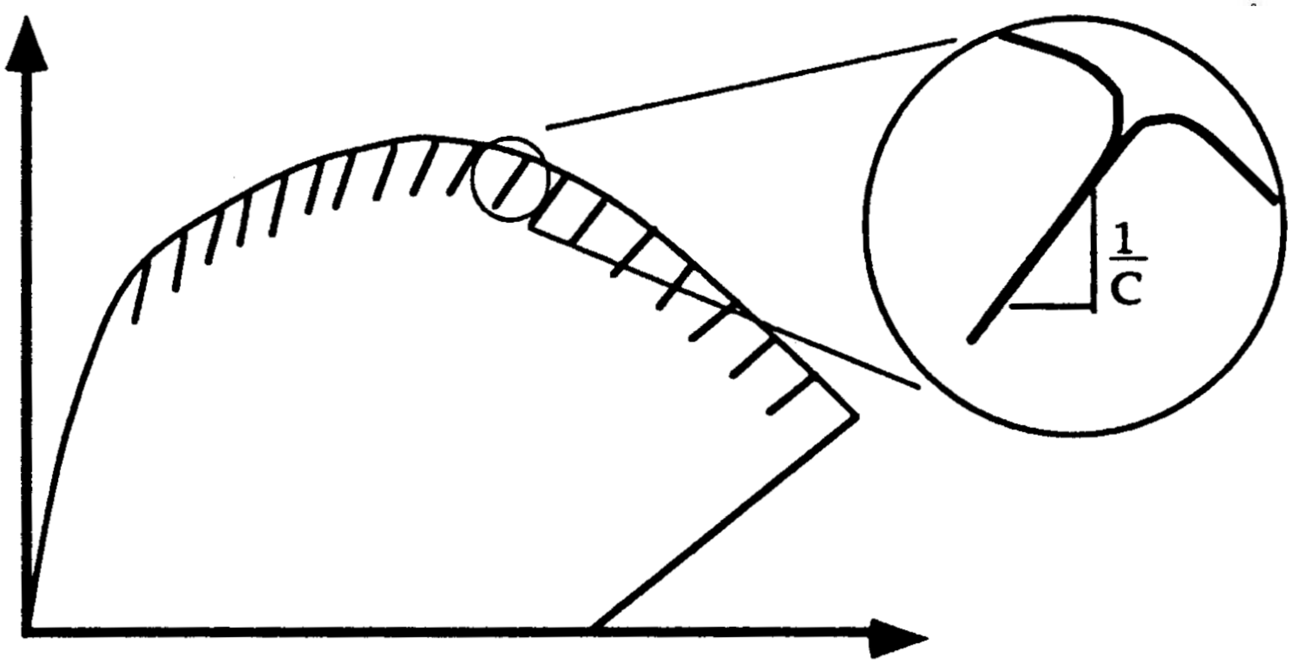
G_C, K_C – fracture toughness = material property quantifying the onset of unstable crack propagation

Flexural tests

R-curve determination



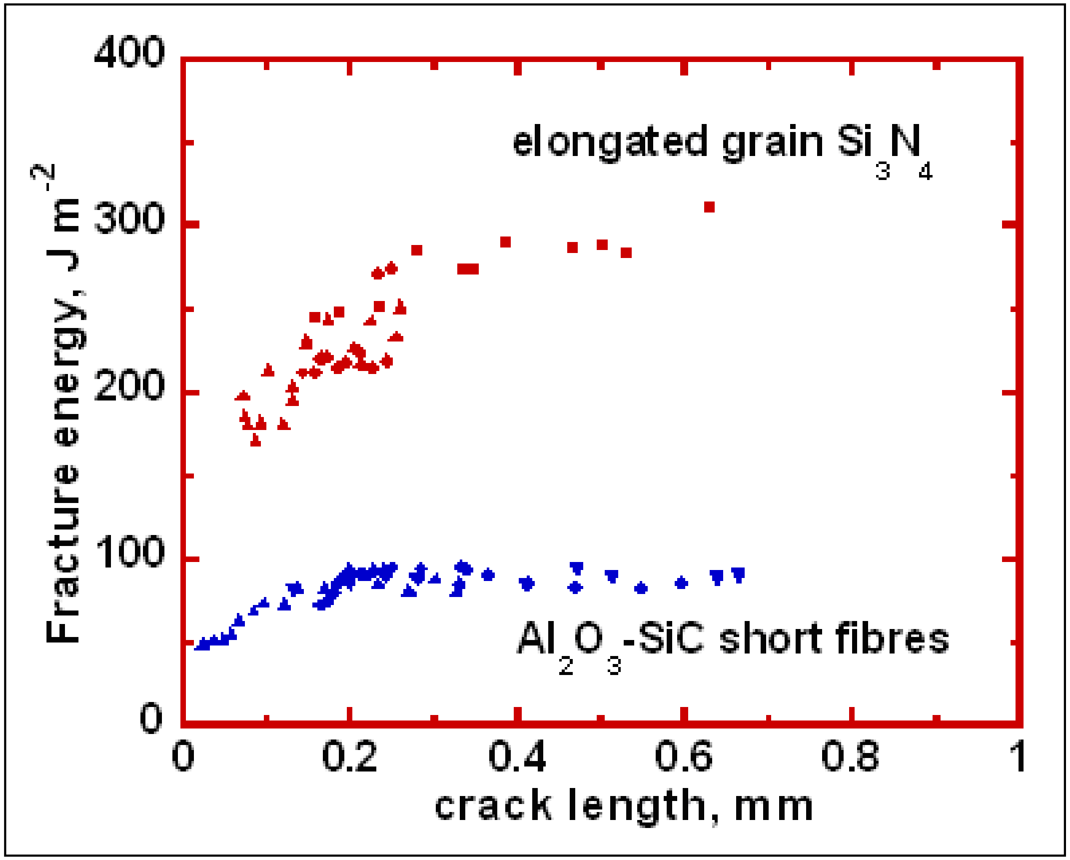
load



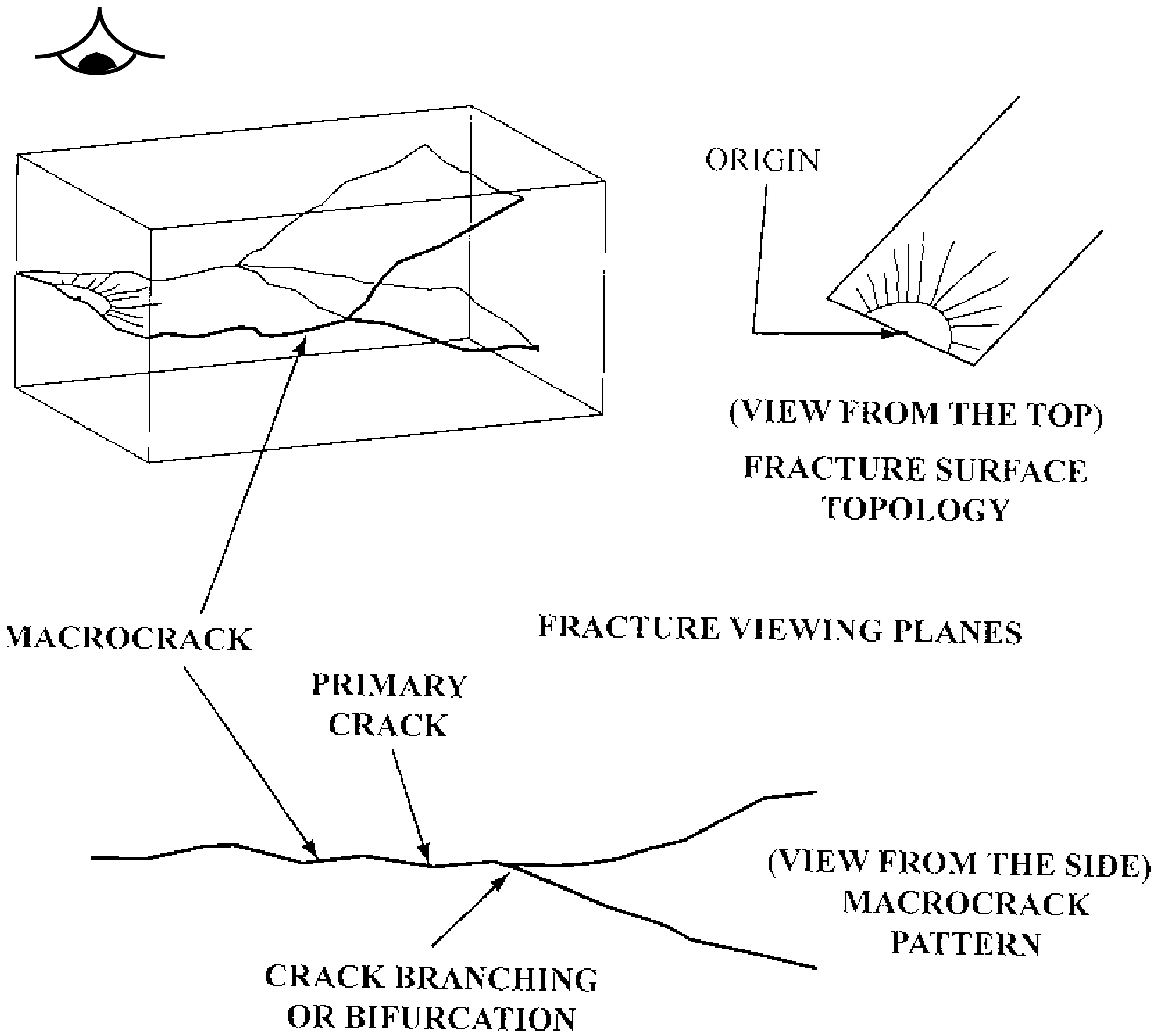
load displacement (deflection)

Flexural tests

r-curves



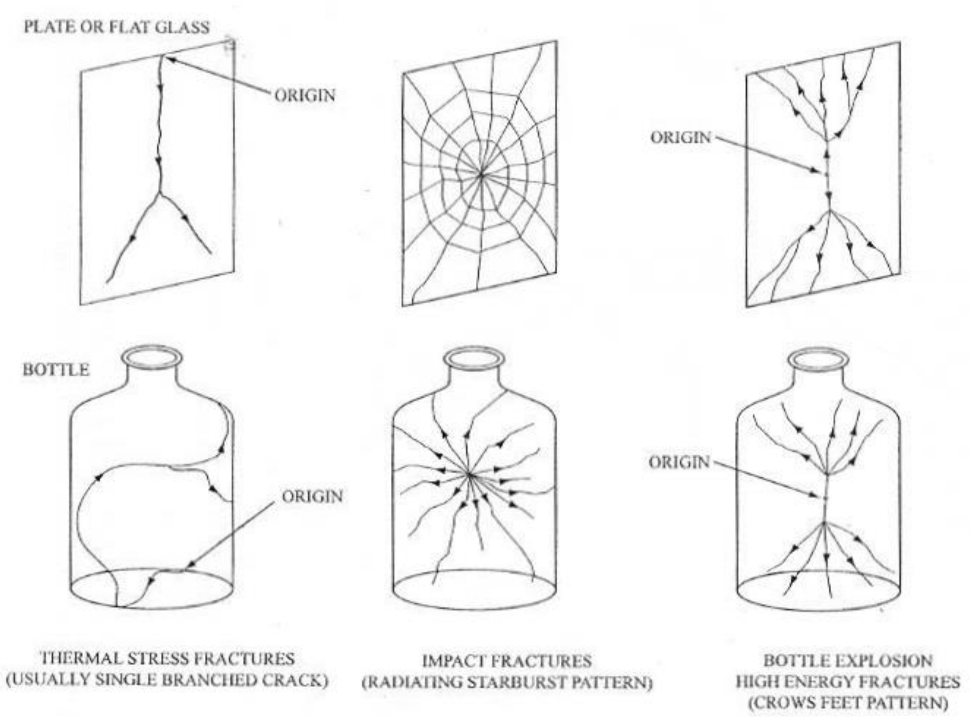
The fractography



Points to emphasize

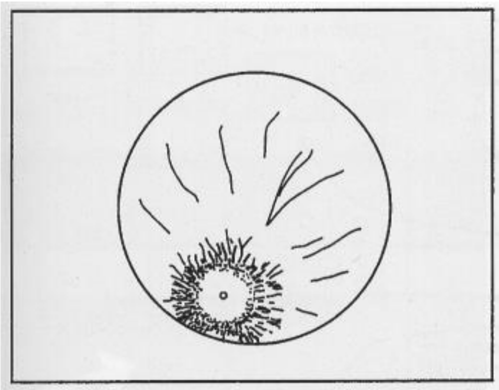
Macro-crack patterns

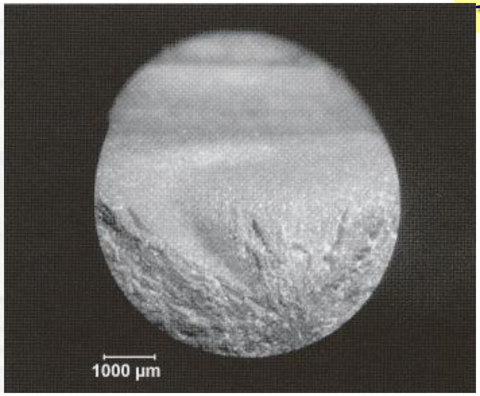
- Crack patterns are always consistent for similar kinds of fractures
- Thermal shocks, impact fractures and pressure fractures are each different
- Higher energy fractures creates more cracks (fragmetation)

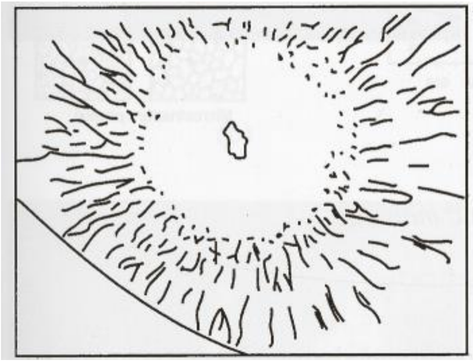


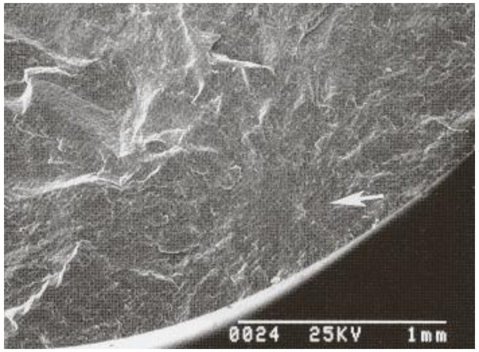
Micro-crack patterns

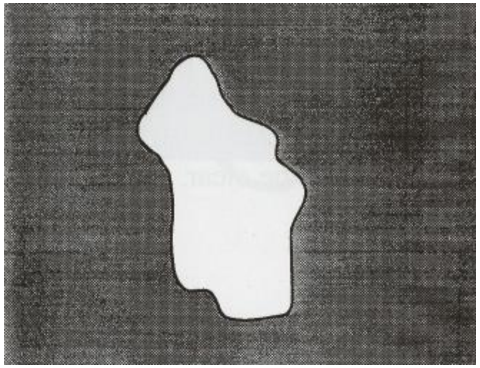
Fractographer's best friend

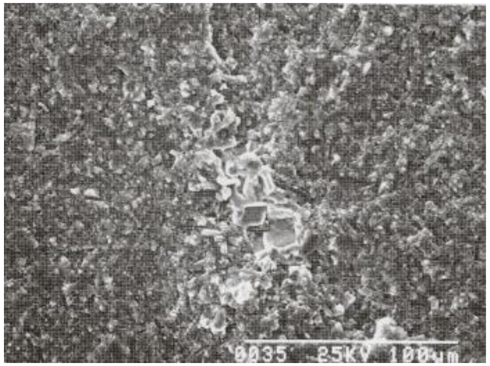


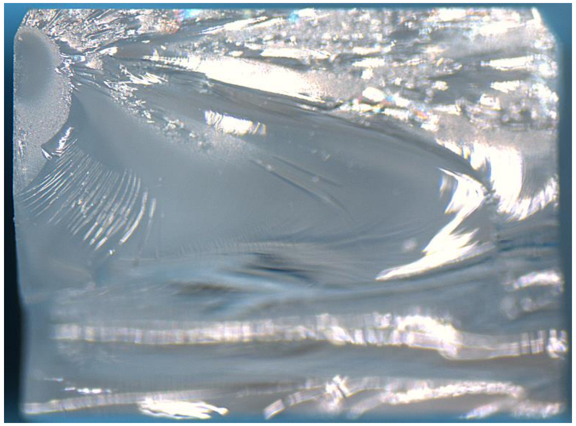


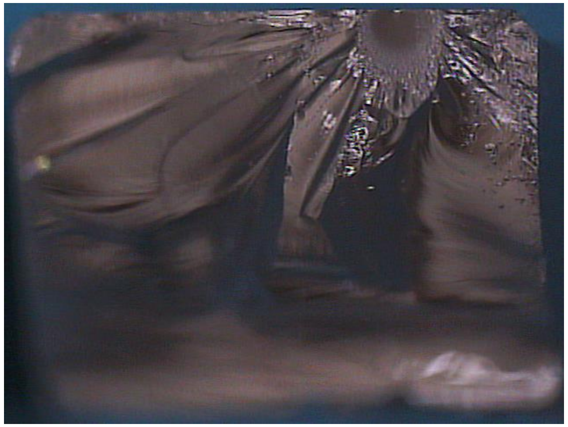




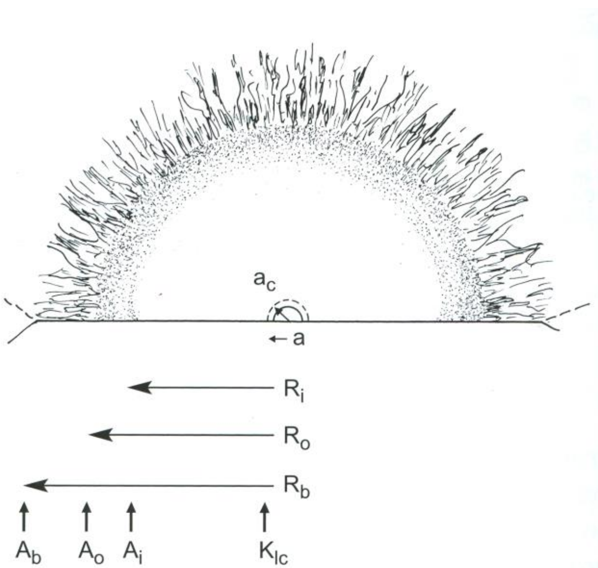


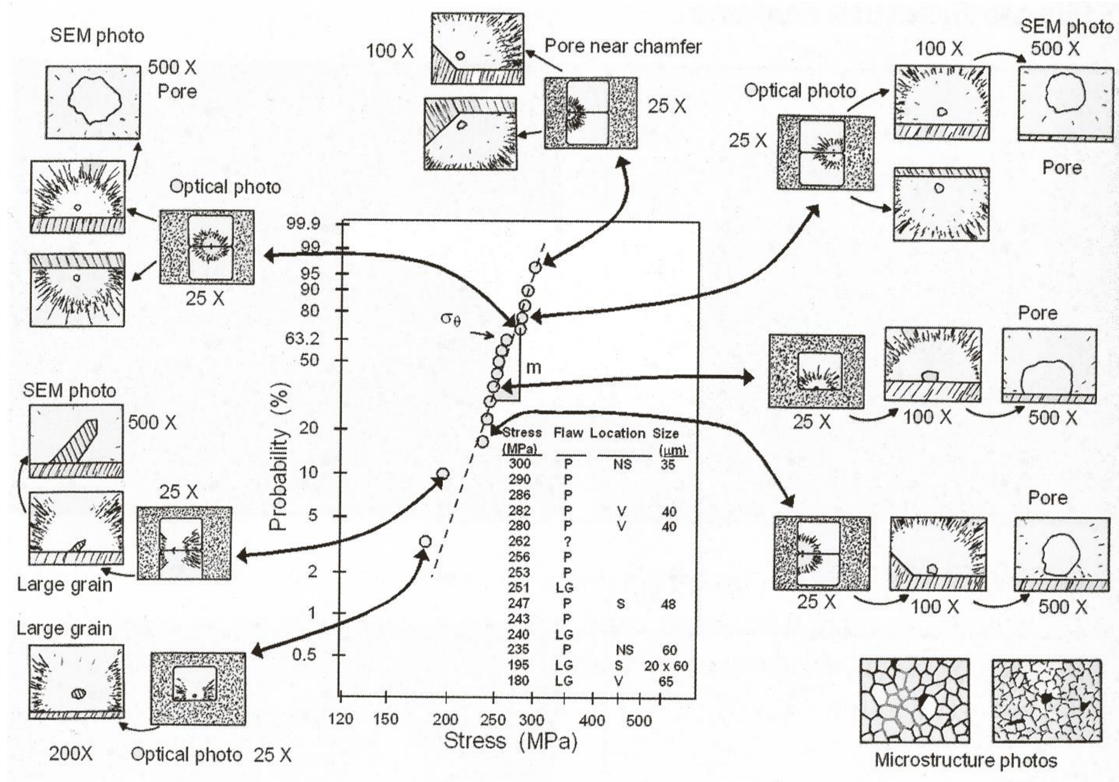






Micro-crack patterns





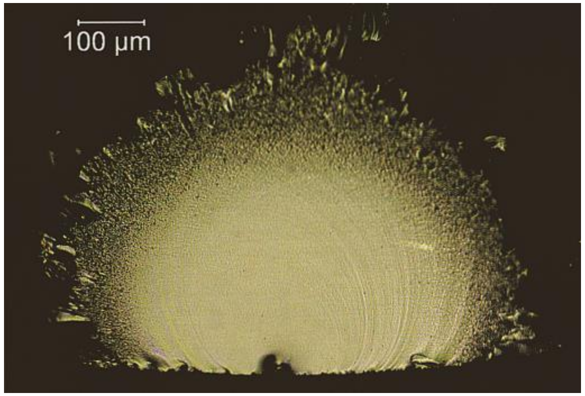
Stress (MPa)	Flaw	Location	Size (µm)
300	P	NS	35
290	P		
286	P		
282	P	V	40
280	P	V	40
262	?		
256	P		
253	P		
251	LG		
247	P	S	48
243	P		
240	LG		
235	P	NS	60
195	LG	S	20 x 60
180	LG	V	65

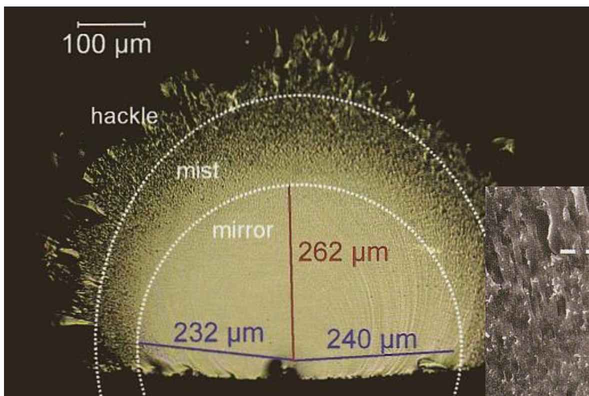
Ri - inner, mirror mist boundary

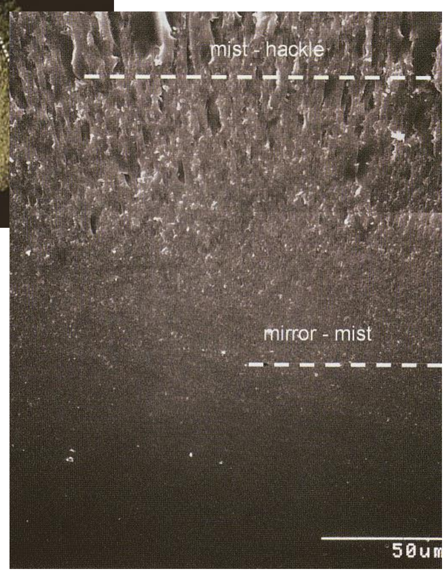
R0 - mirror-hackle boundary

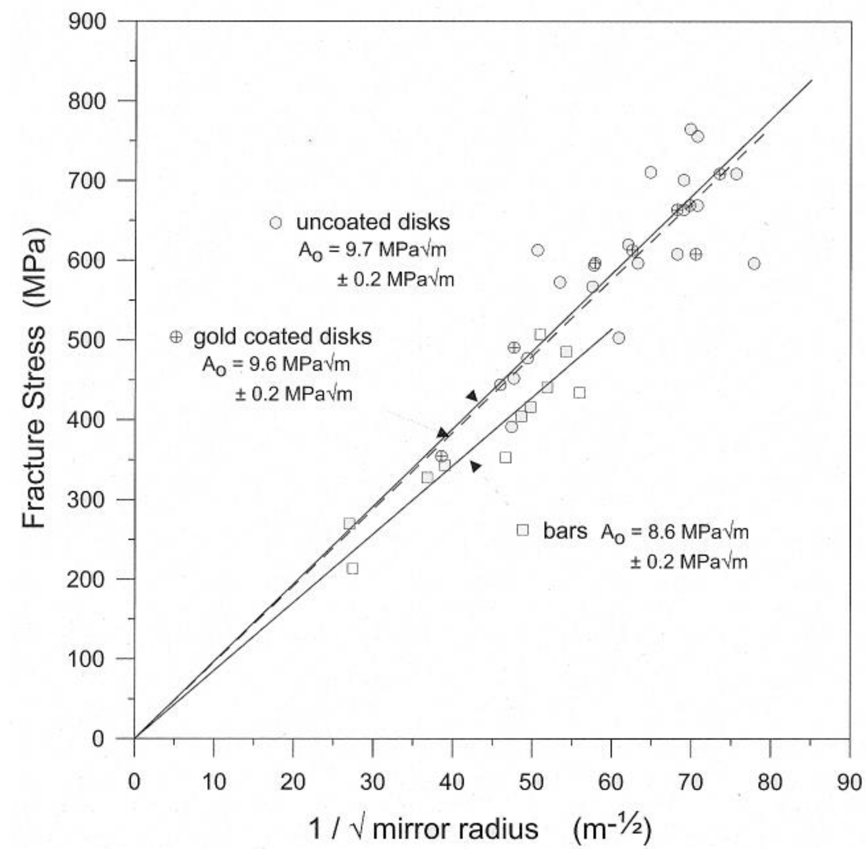
Rb - branching distance

Micro-crack patterns









$$A = \sigma_{*} R^{-1} \quad [MPam^{0.5}]$$

Origin of glass / ceramics brittleness

$$\sigma_f = R_m = \frac{K_{IC}}{\sqrt{\pi a}}$$

Strength of ceramics depends on its brittleness

How to increase the (fracture) strength of ceramics

1) **By decreasing present flaws and defects - a_{max}**

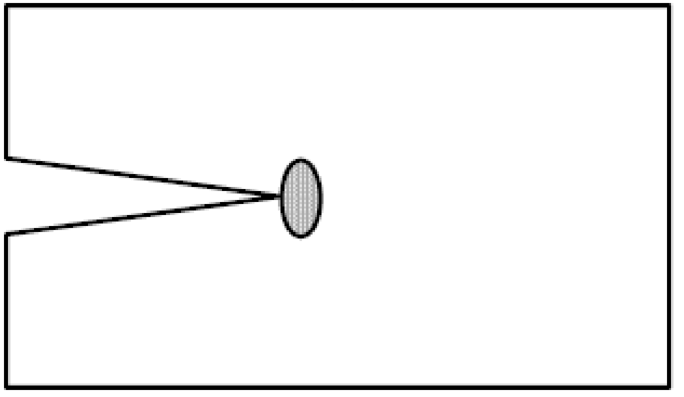
   (grain refinement, higher purity, precise fabrication, by diamond lapping)

2) **By increasing fracture toughness**

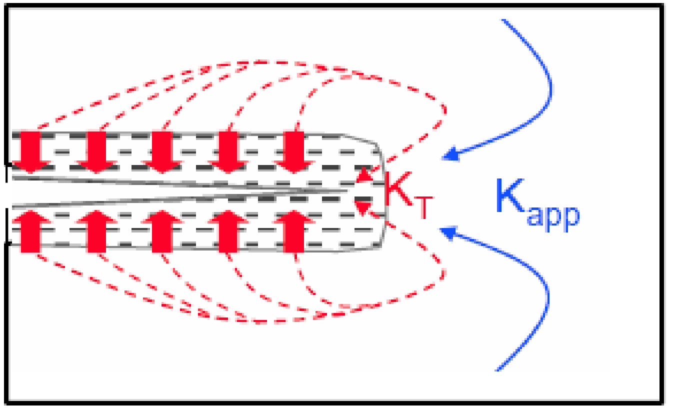
   (enhancement of crack resistance – corresponding materials design)

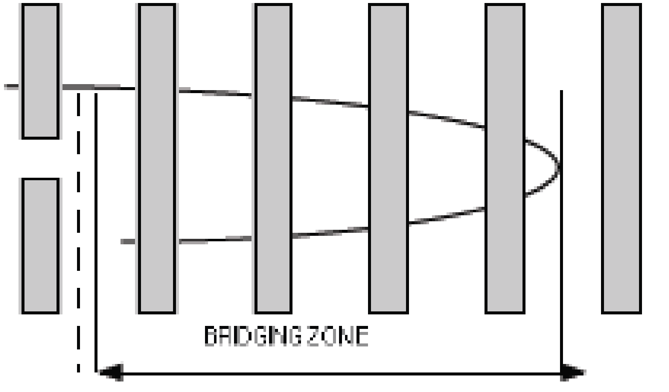
Change of the crack resistance curve

- Crack tip shielding



- Crack bridging

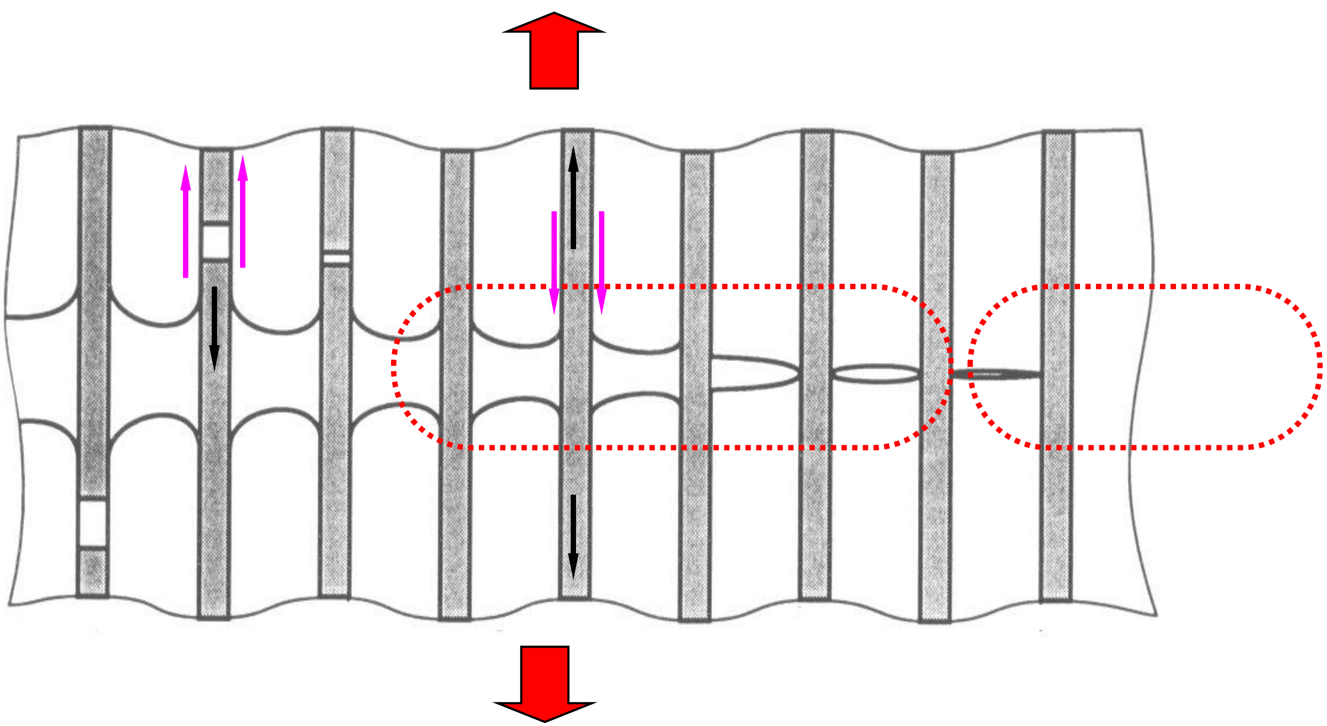




Crack bridging and pul-out

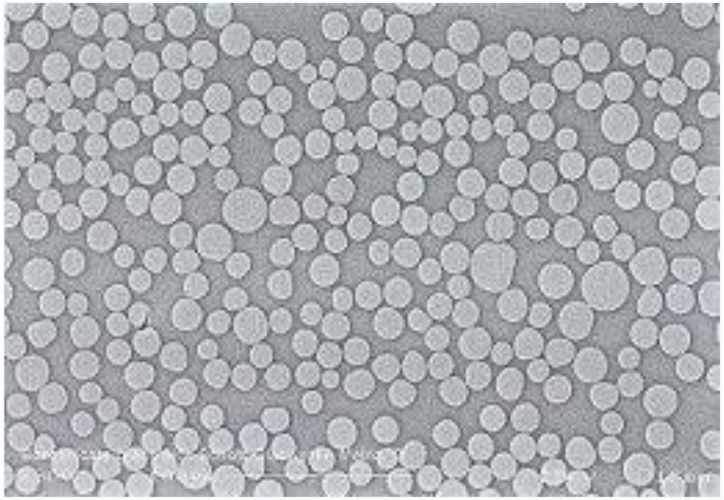
Synergy of basic toughening mechanisms:

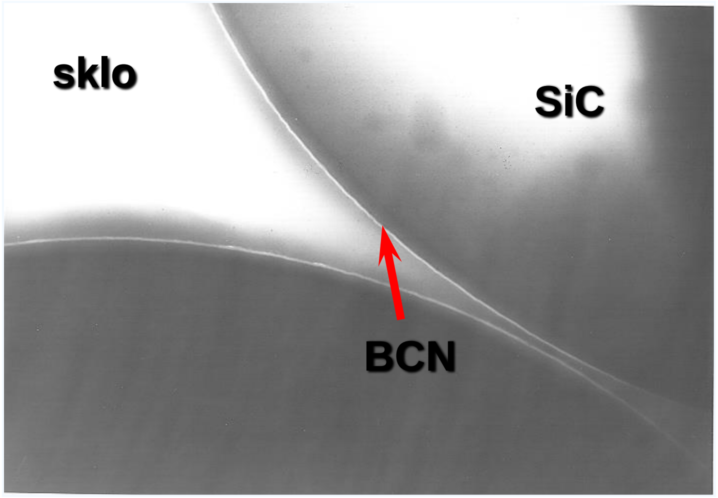
- load transmission in elastic region
- crack bridging
- friction et elastic deformation of the matrix
- friction and pul-out of fibre from matrix



Crack bridging and pul-out

	Youngs modulus [GPa]	Poisson. ratio	Thermal exp. [K^{-1}]	strength [MPa]	Fracture toughness [$MPam^{0.5}$]
glass matr. DURAN®	63	0,22	$3{,}25.10^{-6}$	60	**0,6**
fibre SiC Nicalon®	198	0,20	$3{,}0\ .10^{-6}$	2750	**?? (0,5)**
composite	118	0,21	$3{,}1\ .10^{-6}$	600-700	**~ 26**





fibre reinforced glass matrix