Variances in Testing Thermomechanical Properties of Engineered Refractories

DIFK

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IRS

· 3rd Iranian Refractory Symposium ·
· The Iran Refractory Society & Iran Ceramic Society ·
· Tehran, October 23-25, 2017 ·

① INTRODUCTION

▶ Overview

(i) Introduction

(ii) Definition of fracture

(iii) Thermomechanics

(iv) Innovative techniques

① INTRODUCTION

▶ Basics

- stresses and failure of ceramic components occur at room temperature due to a too high mechanical load

- heat also induces mechnical stresses but due to temperature changes in combination with the coefficient of thermal expansion of the material and therefore without the influence of external forces

- in application of refractories often mechanical as well as thermal induced stresses occur at the same time in a component causing damage

- in principle 3 kinds of thermal induced stresses are described :
  a) permanent (stationary) , b) latent, c) temporary stresses (non-steady-state)

① INTRODUCTION

▶ Basics

➡ Thermal induced stresses result out of :

- restraint of thermal expansion by construction demands
- stationary and/or instationary, inhomogeneous temperature distribution
- thermal mismatch caused by anisotropic behavior of material
- phase transformation (due to i.e. slag infiltration, temperature changes etc.)

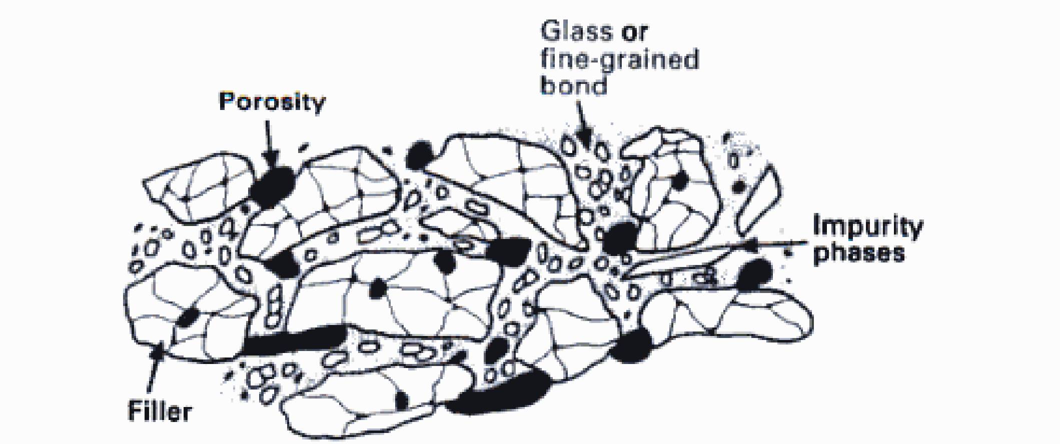
➡ in view of a high durability of refractory linings the thermal stresses caused by an inhomogeneous temperature distribution inside play the major role

➡ to describe the thermomechanical behavior of refractories it is important first to determine the fracture mechanic data of i.e. a material

② DEFINITION OF FRACTURE

▶ initial point

- ➡ almost all ceramic components exhibits defects resulting from production, processing and application conditions



source: W. E. Lee, W. Mark Rainforth

- ➡ the failure of crack infiltrated components or the crack propagation under load until final breakage are the topics of fracture mechanics

② DEFINITION OF FRACTURE

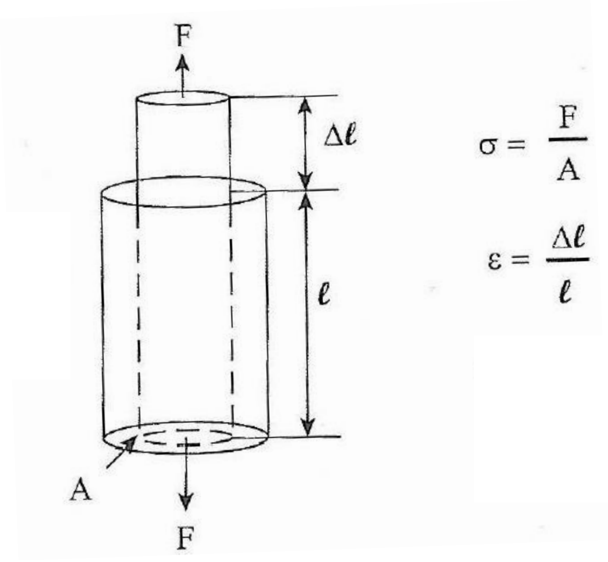
▶ YOUNG´s modulus

- ➡ the behavior of ceramic materials at room temperature can be stated as brittle elastic
- ➡ a proportional deformation **ε** occurs during application of stress **σ** and will result into HOOK´s law including a constant of proportionality **E**

<u>example:</u>

Test of tensile stress

$$\sigma = E \cdot \varepsilon_{reversible}$$



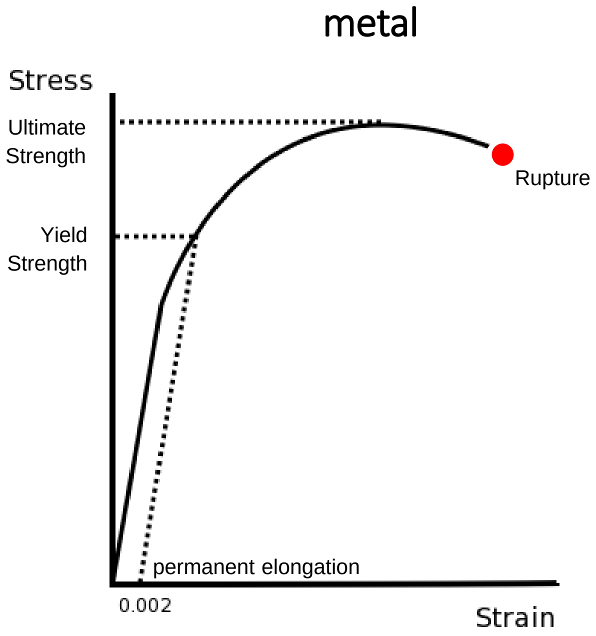
with:
F = force
A = cross sectional area
Δ l = elongation
l = original sample length

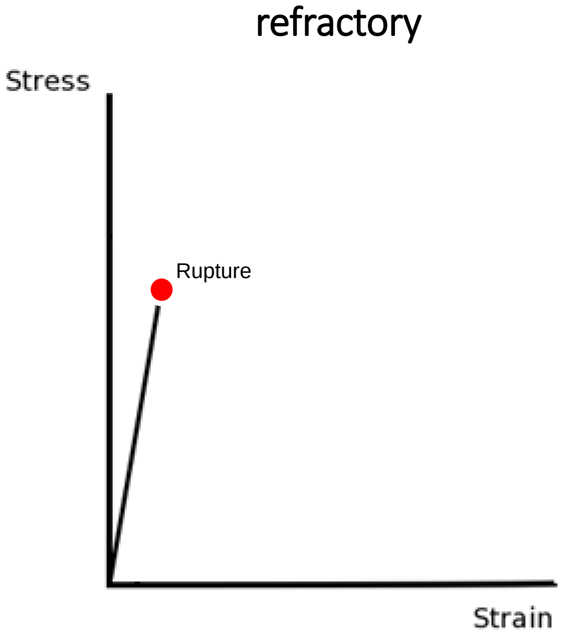
② DEFINITION OF FRACTURE

▶ YOUNG´s modulus

example:

strain stress diagrams





- ➡ due to a strong inhomogeneous microstructure (especially at high temperatures) refractories (caused by i.e. type of binding, pores, grain sizes, microcracks) exhibit elastic behavior only in a small area of deformation
- ➡ the YOUNG´s modulus can be measured by different modes (dynamic, static) with differing results

② DEFINITION OF FRACTURE

▶ YOUNG´s modulus

dynamic mode	static mode
■ resonance & ultrasonic methods	■ bending & compression tests
☐ easy to handle ☐ non-destructive method ☐ performance at low temperatures	☐ measures low values of YOUNG´s modulus (inelastic behavior under load is possible caused by appearance of micro-cracks or irreversible deformation) ☐ easy transferability to high temperature behavior is assigned

__example:__

typical values for refractories YOUNG´s modulus at room temperature in [GPa]

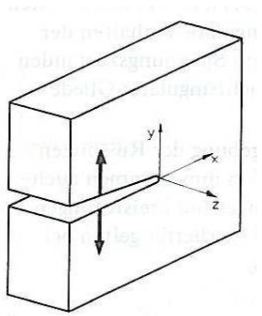
Magnesia brick	80
Magnesia Carbon brick	25
Magnesia Chrome brick	25
Corundum based LC castable	100

② DEFINITION OF FRACTURE

▶ crack characteristics

➡ at the location of a crack 3 different modes of cracks can be detected

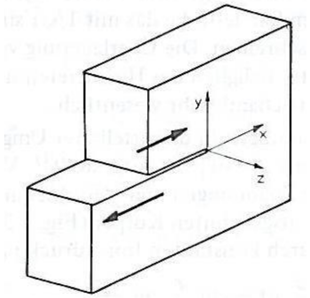
I



☐ strain perpendicular to the crack

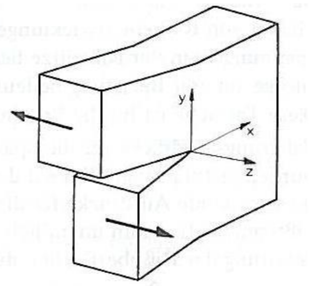
→ normal stressing → tensile loading

II



☐ asymmetrical movement of crack flanks in the direction of crack tip parallel to crack front → shear loading

III



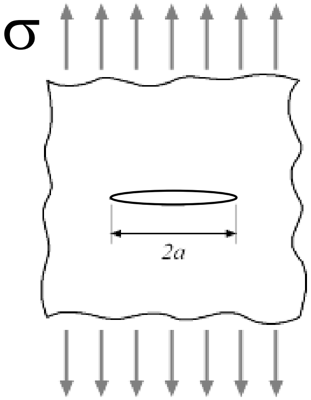
☐ tangential separation of crack flanks

→ Torsion

② DEFINITION OF FRACTURE

▶ crack characteristics

➡ if in a first approach materials were treated as linear elastic and the cracks mathematically can be described as sharp elliptical cut



elliptical crack inside a infinite panel treated by a tensile stress

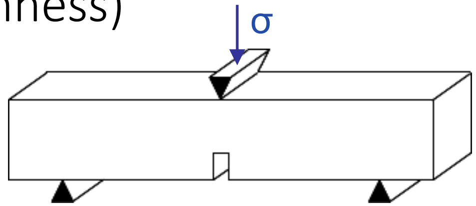
- ☐ cracks principally are weakest sites inside materials
- ☐ at a crack stresses are accumulated
- ☐ the state of stress at the crack tip is described by the stress intensity factor K_I (➞ K- concept)

$$K_I = \sigma \cdot \sqrt{a} \cdot Y = \sigma \cdot \sqrt{\pi \cdot a}$$

with:
σ = stress
a = crack length
Y = form factor

➡ the crack propagation occurs as the stress intensity factor K_I reaches the critical value K_{Ic} (➞ fracture toughness)

➡ the fracture toughness K_{Ic} can be described by notched beam tests



Notched beam test

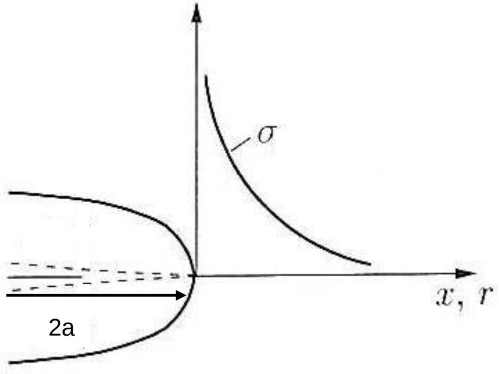
② DEFINITION OF FRACTURE

▶ crack characteristics

➡ there exist a defined amount of stored elastic energy (rel. to unit surface) corresponding to the stress state at the crack flank

$$G = \frac{K_I^2}{E} = \frac{\pi \cdot a \cdot \sigma^2}{E}$$

with:
σ = stress
a = crack length
E = YOUNG´s modulus
Y = form factor



so that both approaches – stress intensity vs. energy – can be replaced

➡ if G exceeds a critical value G_c (→ crack resistivity), the crack expands

➡ the stored elastic energy lead to the creation of a new fracture surface

➡ the crack resistivity G_c can be determined by notched beam tests

② DEFINITION OF FRACTURE

▶ measuring methods

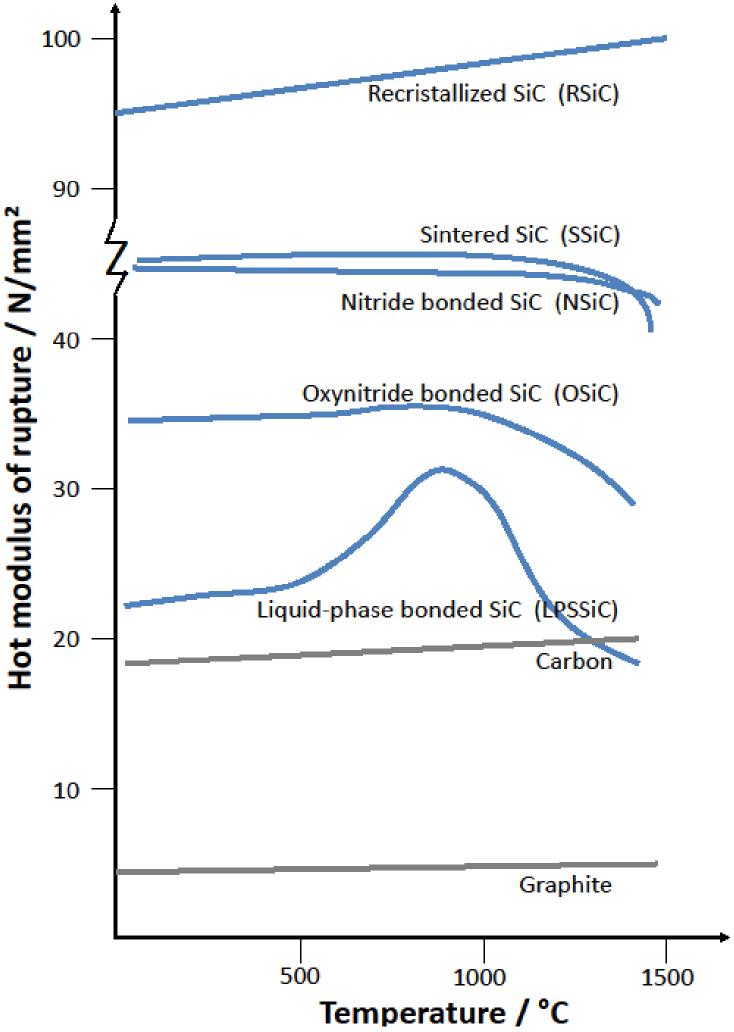
➡ thermophysical testing according to DIN EN 993-7

- ☐ setting of a defined operation temperature
- ☐ keeping the setup constantly at this temperature for 60 min.
- ☐ increasing slowly the force until fracture occurs
- ☐ arranging all temperature dependent fraction values in one diagram

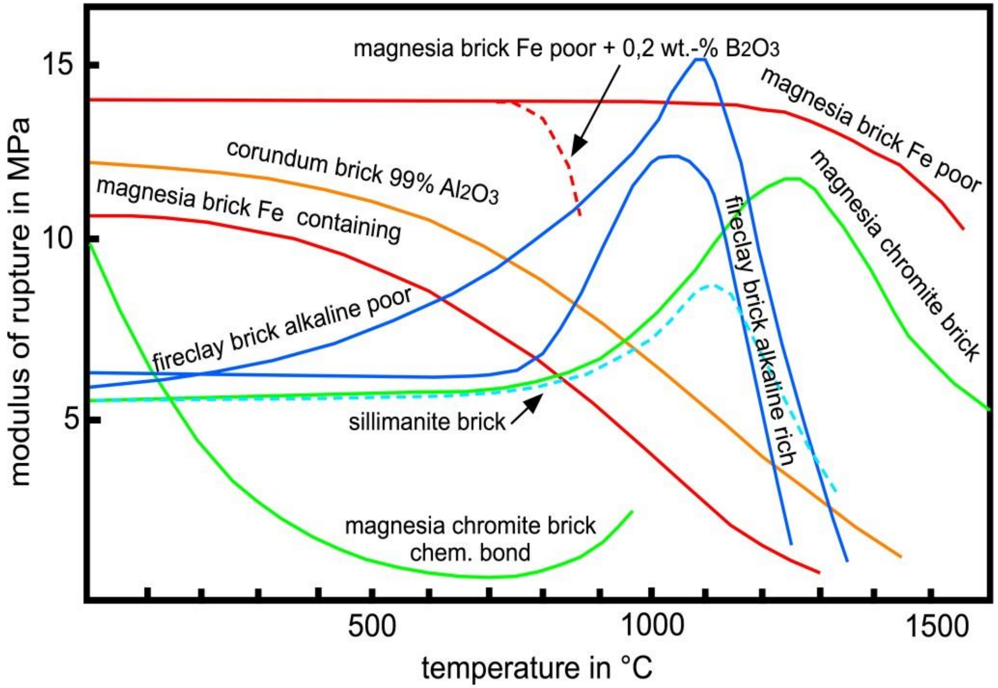
established standardized HMOR-Test equipment

② DEFINITION OF FRACTURE

▶ measuring methods



HMOR-test results of different candidate materials in the system Si – C



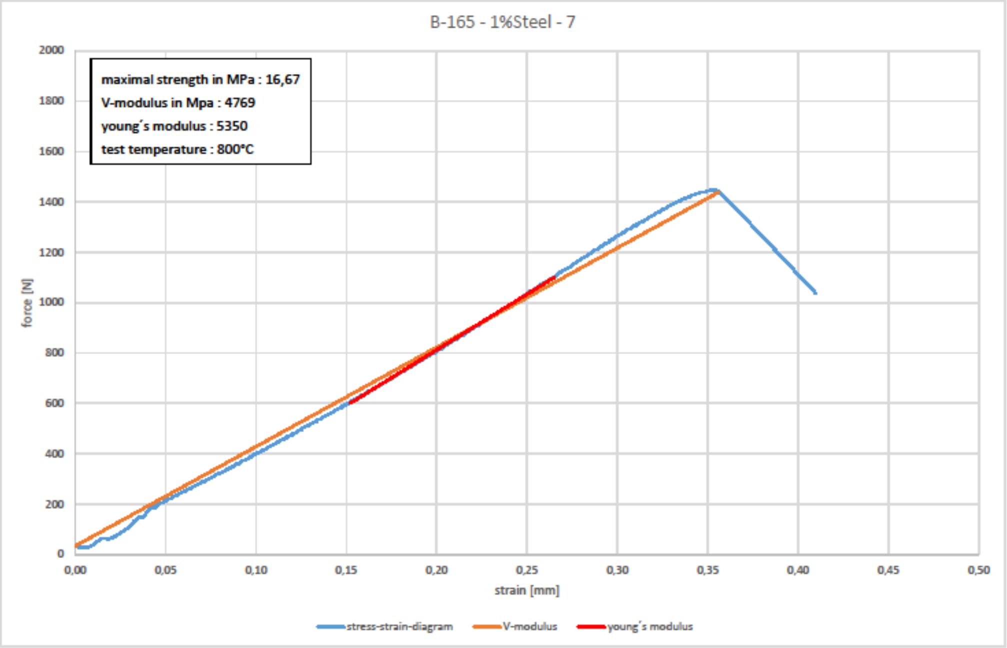
examples of HMOR-behavior of various refractories

② DEFINITION OF FRACTURE

► measuring methods

➡ also YOUNG´s modulus can be determined out of HMOR test (example: steel at test temperature of T = 800°C)

with:
Blue line = HMOR →
force (N) / strain (mm) diagram
Red line = slope of linear section of curve
l = distance of supports
b = width of sample
h = height of sample

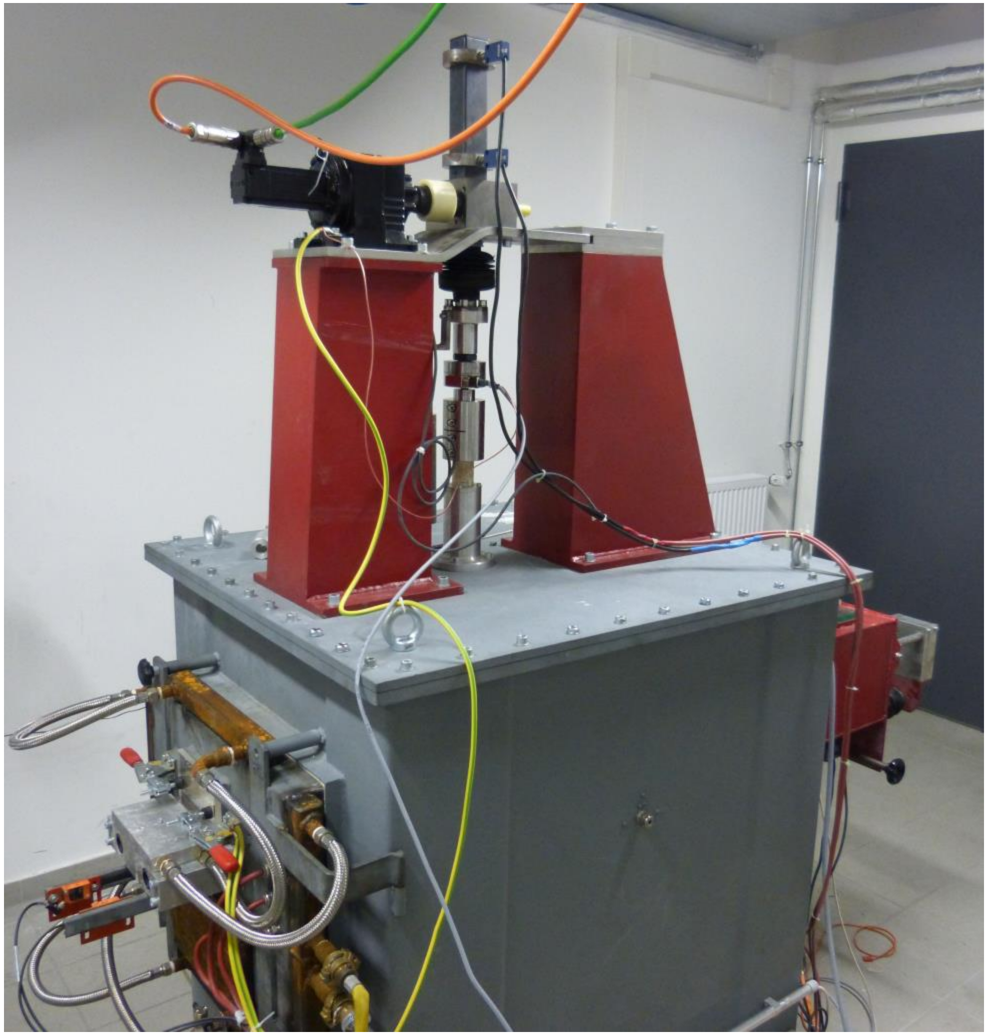


$$E = \frac{\Delta y_{linear\ section}}{\Delta x_{linear\ section}} \cdot \frac{l^3}{(4 \cdot b) \cdot h^3}$$

② DEFINITION OF FRACTURE

► measuring methods

- ➡ HMOR testing equipment to prevent O_2- attack and to state more precisely the measured data
  - → stress/strain-unit



Strongly optimized HMOR-Test equipment

② DEFINITION OF FRACTURE

▶ measuring methods

➡ thermophysical testing according to DIN 51064 with determination of compressive strength under load

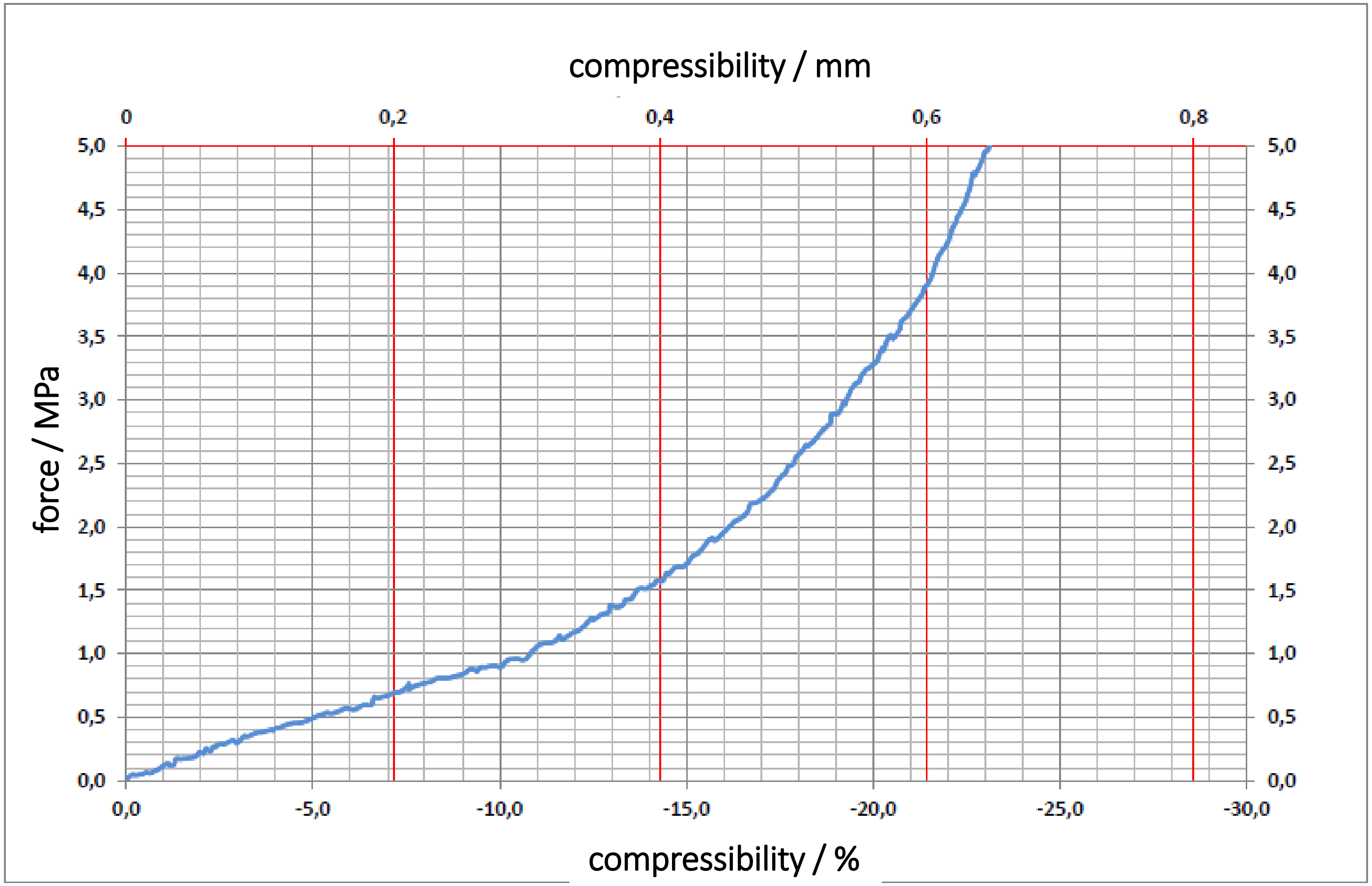
- ☐ setting of a defined constant operation force (N/mm²)
- ☐ increasing the temperature until sample is compressed about 0.3 mm → T_a
- ☐ increasing the temperature further until the sample is compressed about 10 mm from initial stage → T_e
- ☐ increasing temperature until fracture occurs → T_b

established standardized CSL-Test equipment

② DEFINITION OF FRACTURE

▶ measuring methods

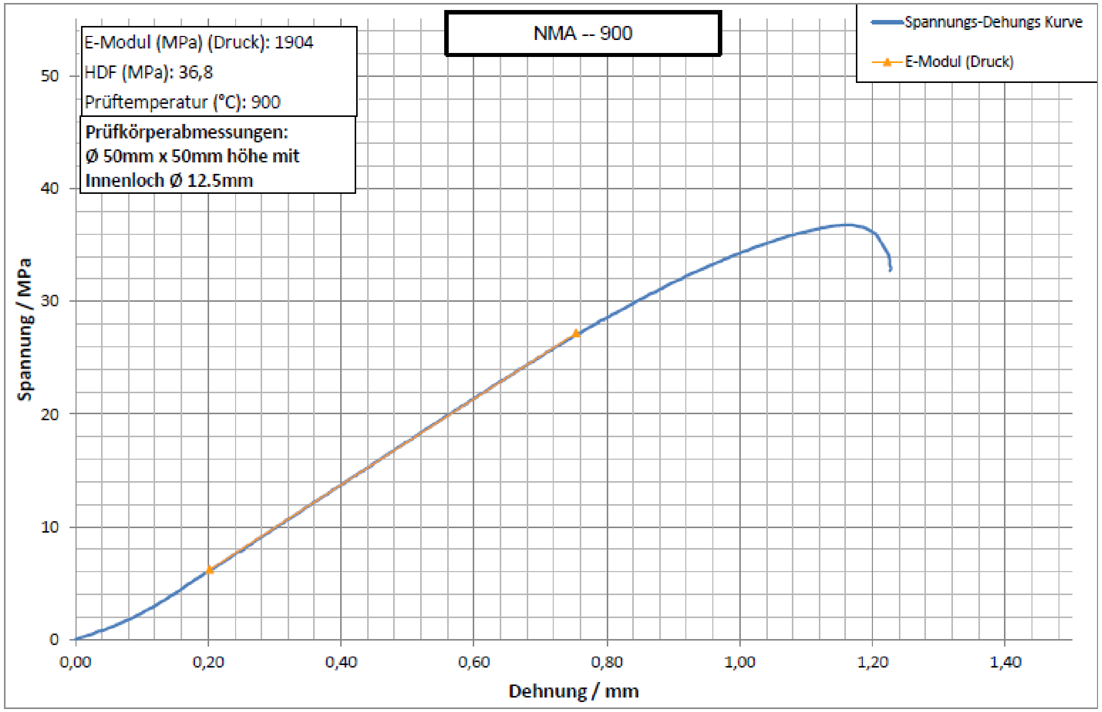
➡ the heating rates are 15 K/min at T ≤ 1000°C and 8 K/min at T ≥ 1000°C



② DEFINITION OF FRACTURE

▶ measuring methods

➡ also YOUNG´s modulus can be determined by using this method



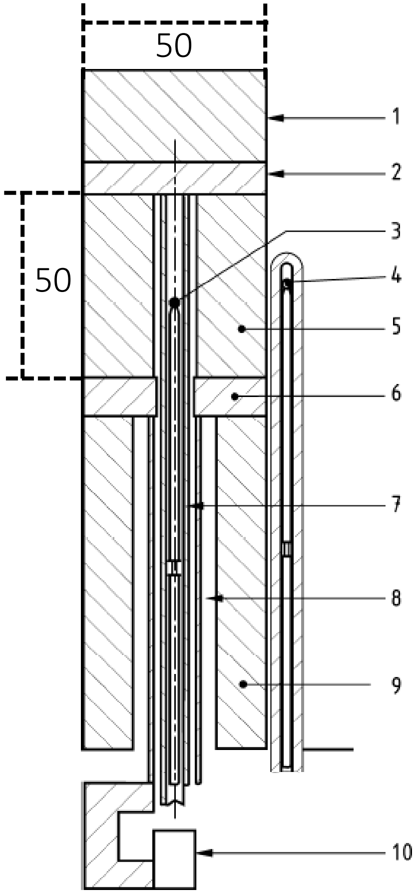
with:
Blue line = stress / elongation diagram
Red line = slope of linear section of curve
h = height of sample

$$E = \frac{\Delta y_{linear\ section}}{\Delta x_{linear\ section}} \cdot h_{sample}$$

② DEFINITION OF FRACTURE

► measuring methods

➡ thermophysical testing according to DIN EN ISO 1893 for measuring refractoriness under load (RuL) and creep under compression



with:
1 = movable pillar
2 = higher plate
3 = interior thermocouple
4 = control thermocouple
5 = sample
6 = lower plate
7 = interior Al_2O_3-tube
8 = outer Al_2O_3-tube
9 = fixed pillar
10 = measuring device

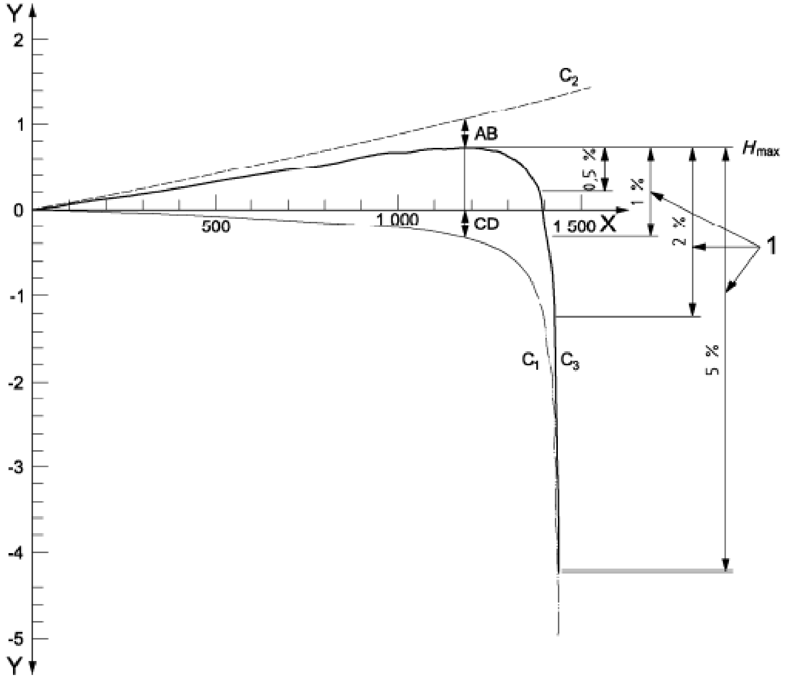
established standardized RuL-Test equipment

② DEFINITION OF FRACTURE

▶ measuring methods

➡ rules to determine characteristic temperatures for test samples for RuL

- ☐ setting of a defined constant pressure load for a cylindrical sample
- ☐ increasing temperature with a defined heating rate until a required deformation or compression occurs
- ☐ deformation related temperatures are recorded



with:

h_{max} = point of highest deformation with its characteristic temperature

$T_{0.5}$ = temperature at 0.5% compression of initial sample height

T_1 = temperature at 1.0% compression of initial sample height

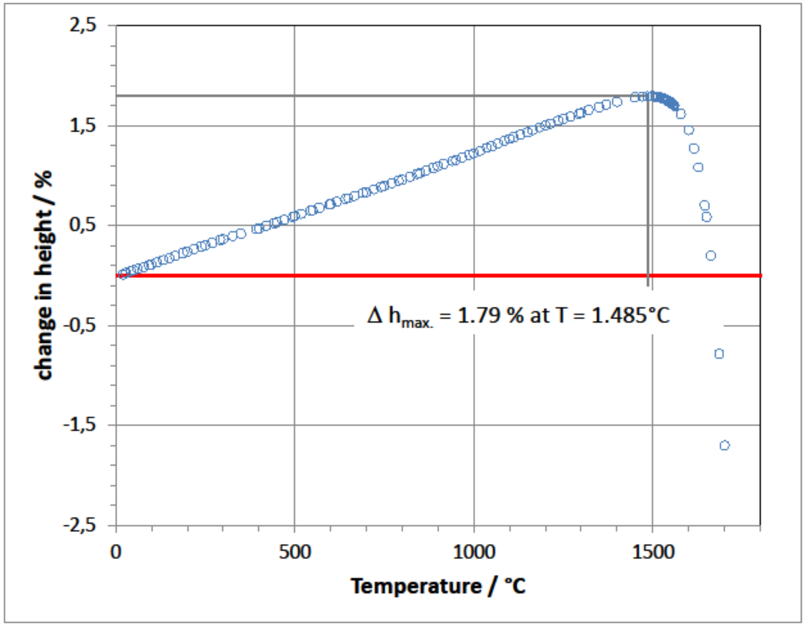
T_2 = temperature at 2.0% compression of initial sample height

T_5 = temperature at 0.5% compression of initial sample height

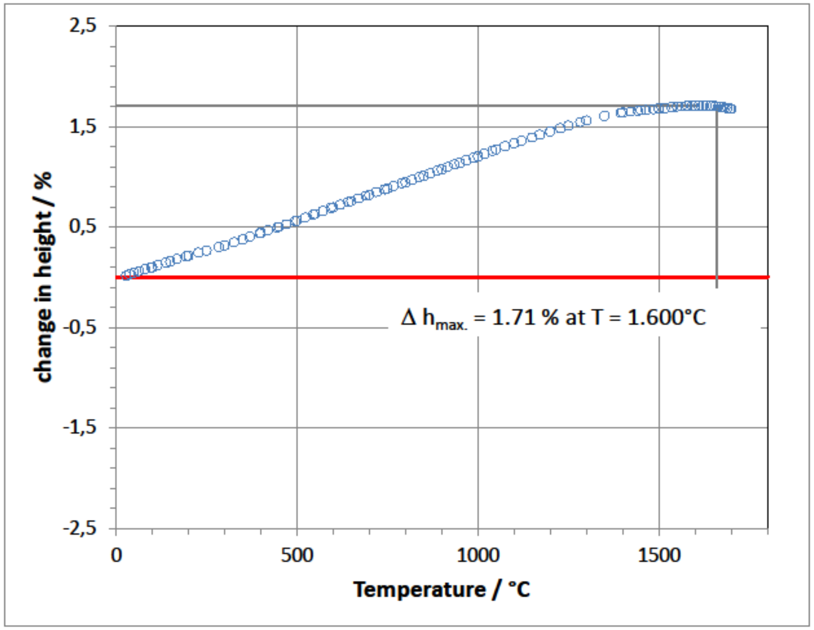
② DEFINITION OF FRACTURE

▶ measuring methods

➡ comparison of high temperature magnesia based construction materials without and with microstructural optimization



RuL of magnesia brick with a load of 0.2 MPa



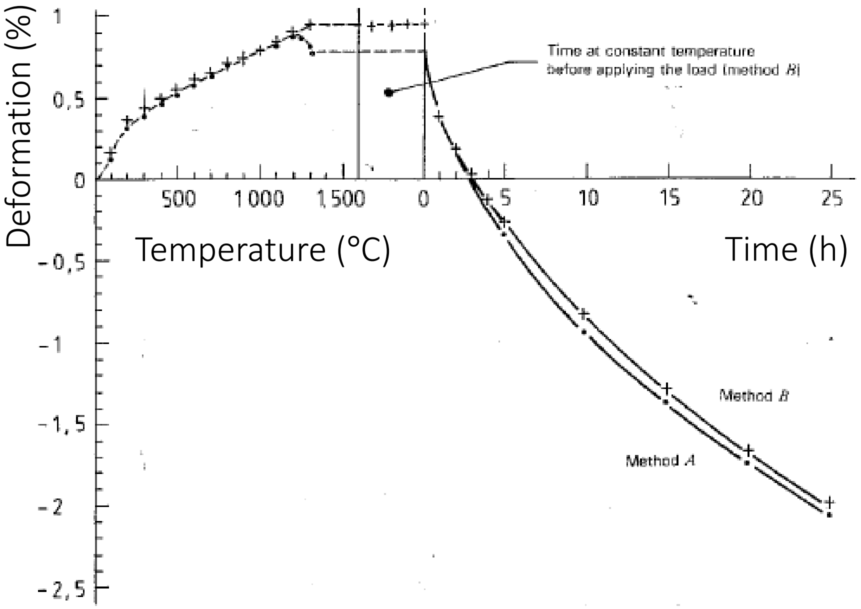
RuL of magnesia spinel brick with a load of 0.2 MPa

② DEFINITION OF FRACTURE

► measuring methods

➡ rules to determine characteristic values for creep under compression according to DIN EN 993-9

- ☐ maintaining a cylindrical sample for a period at a contant temperature
- ☐ applying a defined load to detect the deformation (relative to its initial height) in dependence of time
- ☐ a table show percentage change in height in intervals of 5 hours up to 25 h in total

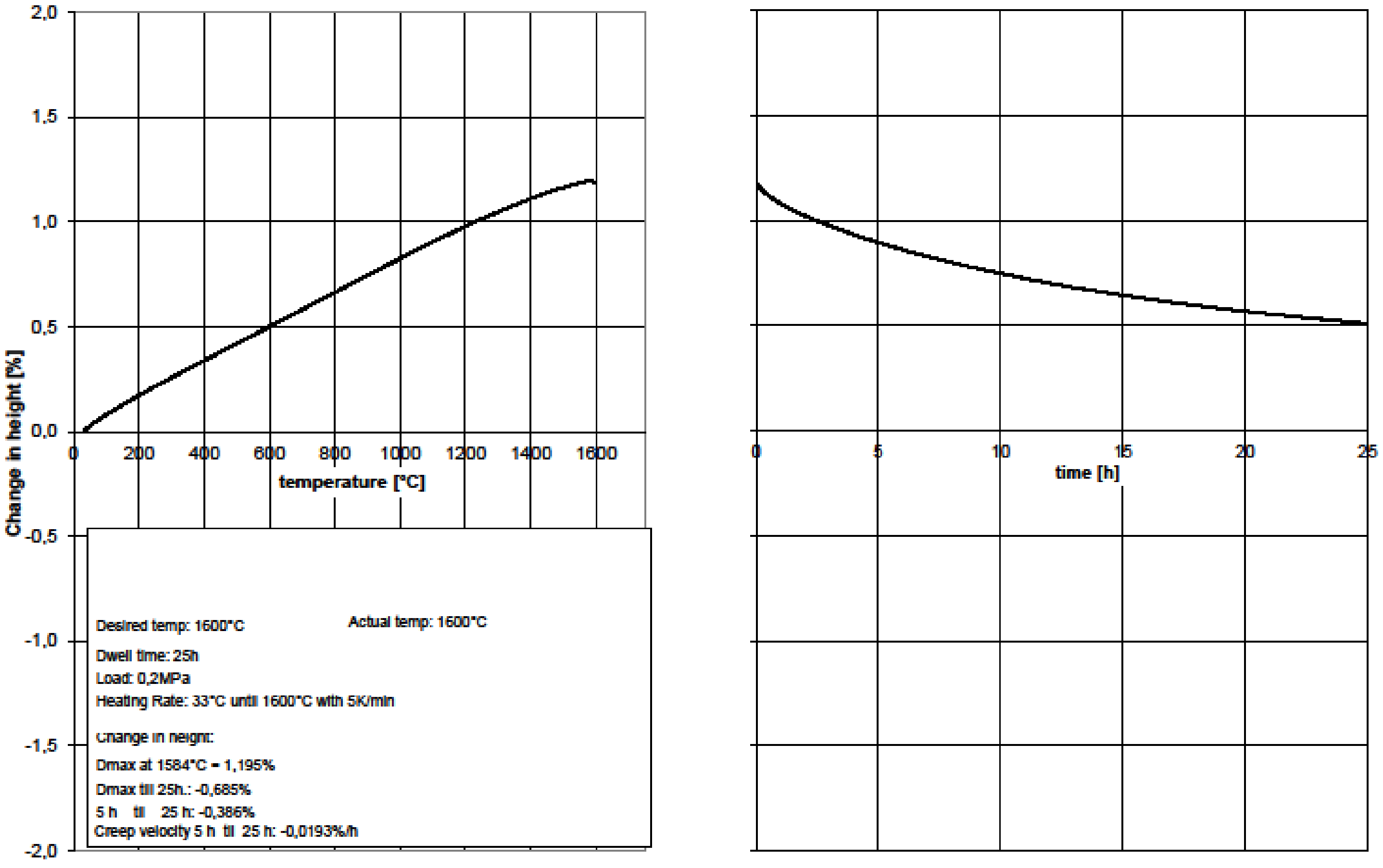


② DEFINITION OF FRACTURE

▶ measuring methods

<u>**example:**</u>

Test of Creep in compression according to DIN EN 993-9



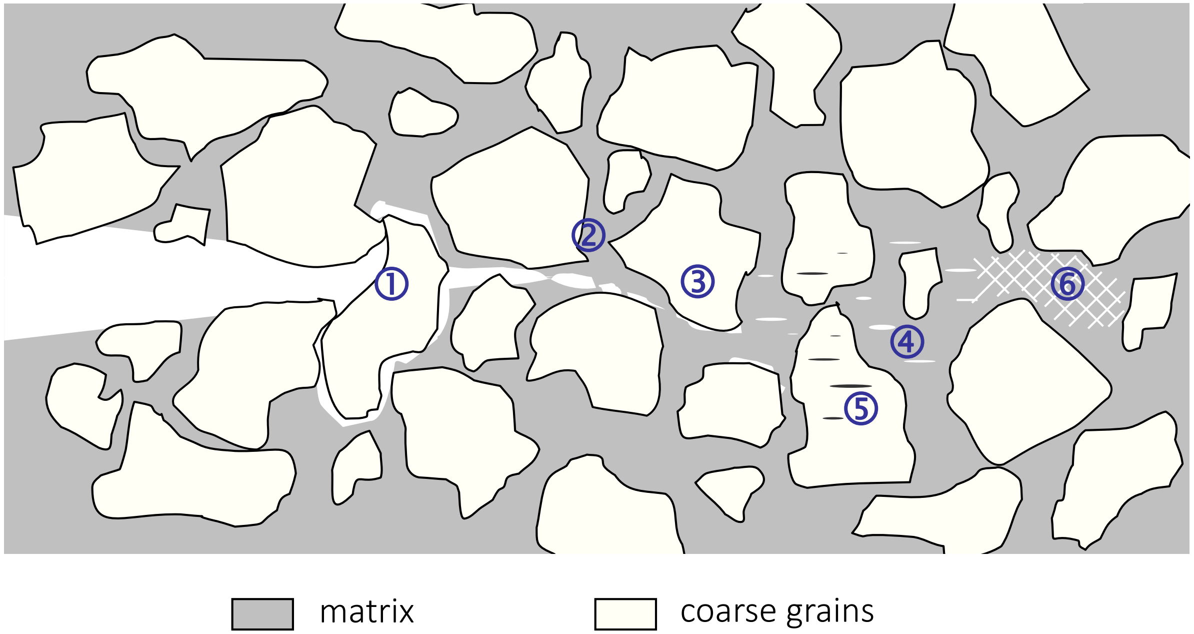
Desired temp: 1600°C
Actual temp: 1600°C
Dwell time: 25h
Load: 0,2MPa
Heating Rate: 33°C until 1600°C with 5K/min

Change in height:
Dmax at 1584°C = 1,195%
Dmax till 25h.: -0,685%
5 h till 25 h: -0,386%
Creep velocity 5 h till 25 h: -0,0193%/h

② DEFINITION OF FRACTURE

▶ non linear-elastic fracture mechanics

➡ in case of refractories (coarse grains ➝ heterogeneous materials) energy that is not needed for creation of new surfaces is transformed



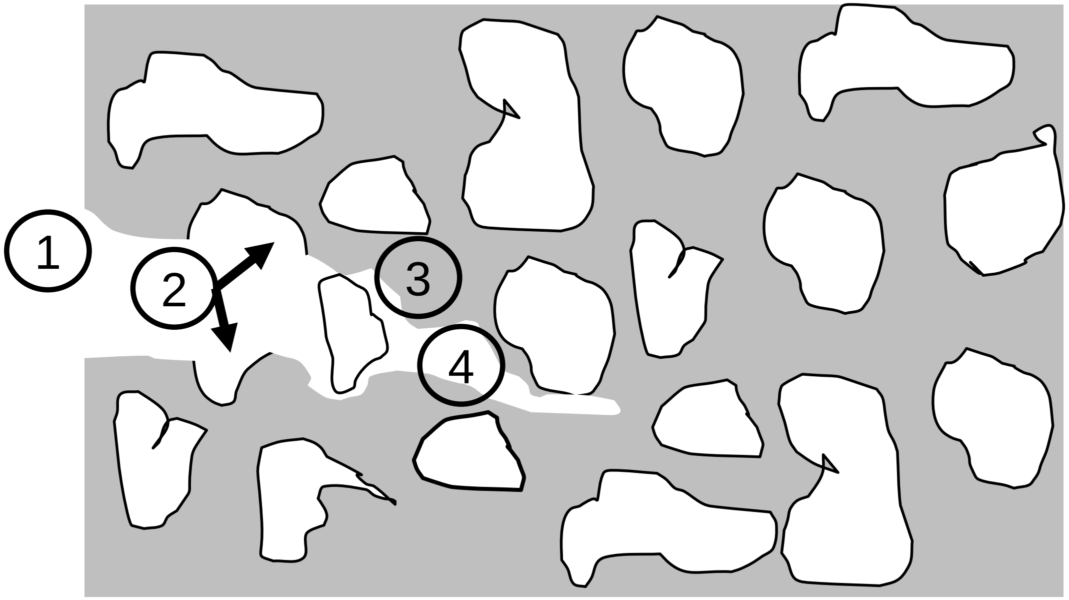
with:
1 = creation of grain bridges
2 = ductile materials areas
(especially at high temperatures)
3 = delamination
4 = creation of microcracks within the matrix
5 = creation of microcracks in grains
6 = plastic deformation

➡ the overall energy for creation und propagation of a crack is defined as specific fracture energy G_f

② DEFINITION OF FRACTURE

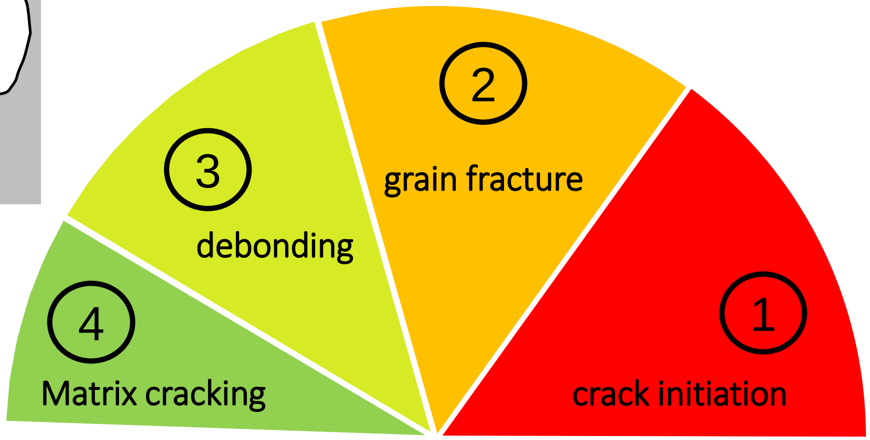
▶ non linear-elastic fracture mechanics

➡ energy consumption during cracking



▨ matrix ☐ coarse grains & aggregates

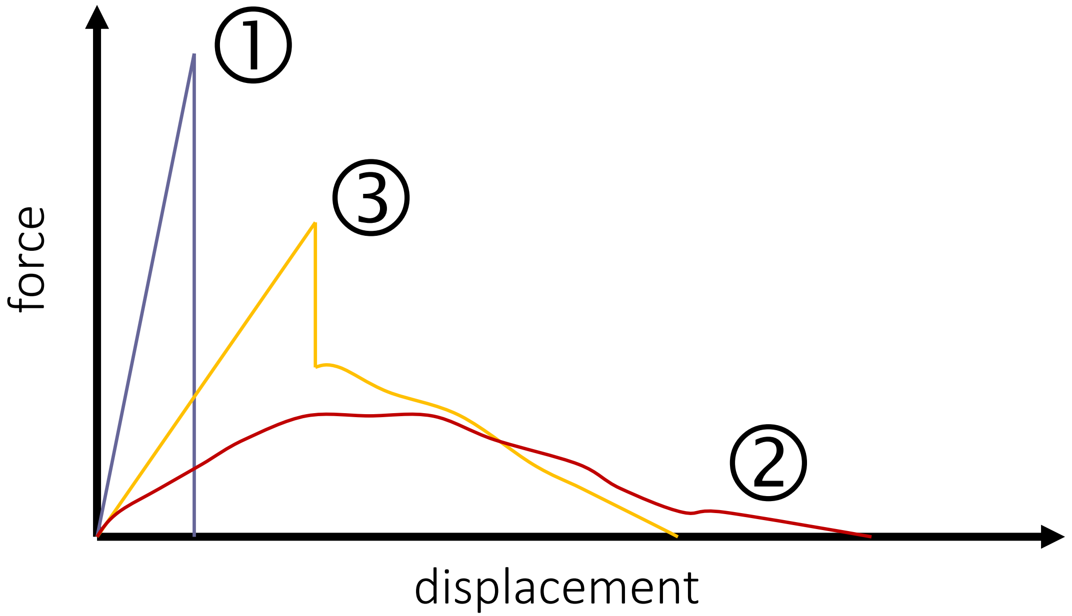
energy consumption distribution



② DEFINITION OF FRACTURE

▶ non linear-elastic fracture mechanics

- ➡ the specific fracture energy G_f can be demonstrated by an force (N) / displacement (mm) - diagram
- ➡ for differentation 3 cases of crack growth are used and also the subcritical crack growth is considered



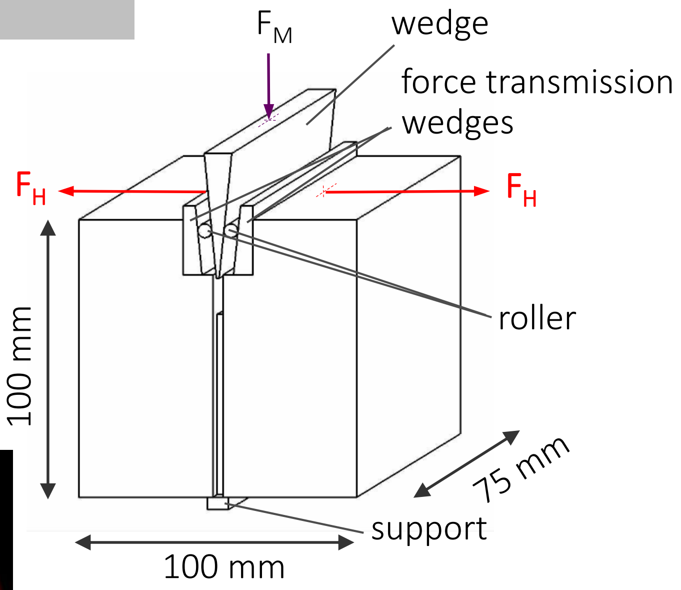
☐ ① unstable

☐ ② stable

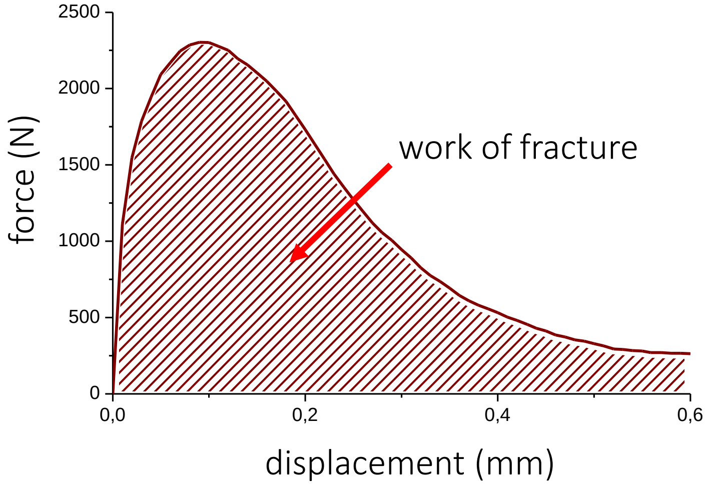
☐ ③ partially stable

② DEFINITION OF FRACTURE

▶ non linear-elastic fracture mechanics



- ➡ Wedge splitting test for detecting the specific fracture energy G_f



- ➡ specific fracture energy $$G_F = \frac{1}{2A}\int F_H d\delta$$

with:
F_H = horizontal force
A = plane of fracture
d = crack displacement

- ➡ notch bending tensile strength $$\sigma_{KZF} = \frac{F_{H\max}}{b \cdot h} + \frac{6 \cdot F_{H\max} \cdot y}{b \cdot h^2}$$

② DEFINITION OF FRACTURE

► non linear-elastic fracture mechanics

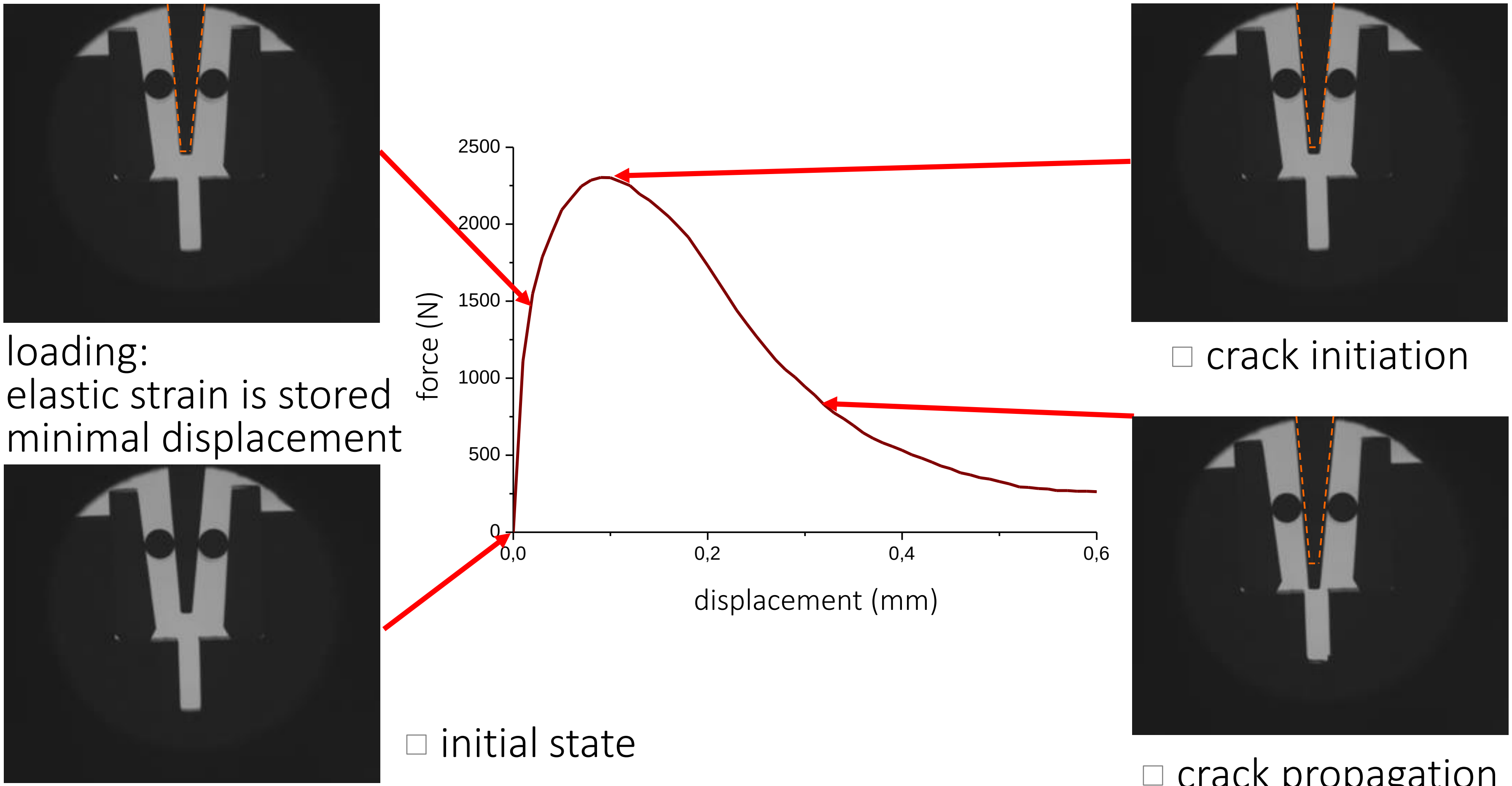
Furnace with controlled atmosphere up to T ≤ 1.700°C

Optical detection equipment with non-contact measurement method of the displacement

② DEFINITION OF FRACTURE

▶ non linear-elastic fracture mechanics

➡ Measuring result from i.e. a fireclay brick at T = 1.150°C



☐ loading:
elastic strain is stored
minimal displacement

☐ initial state

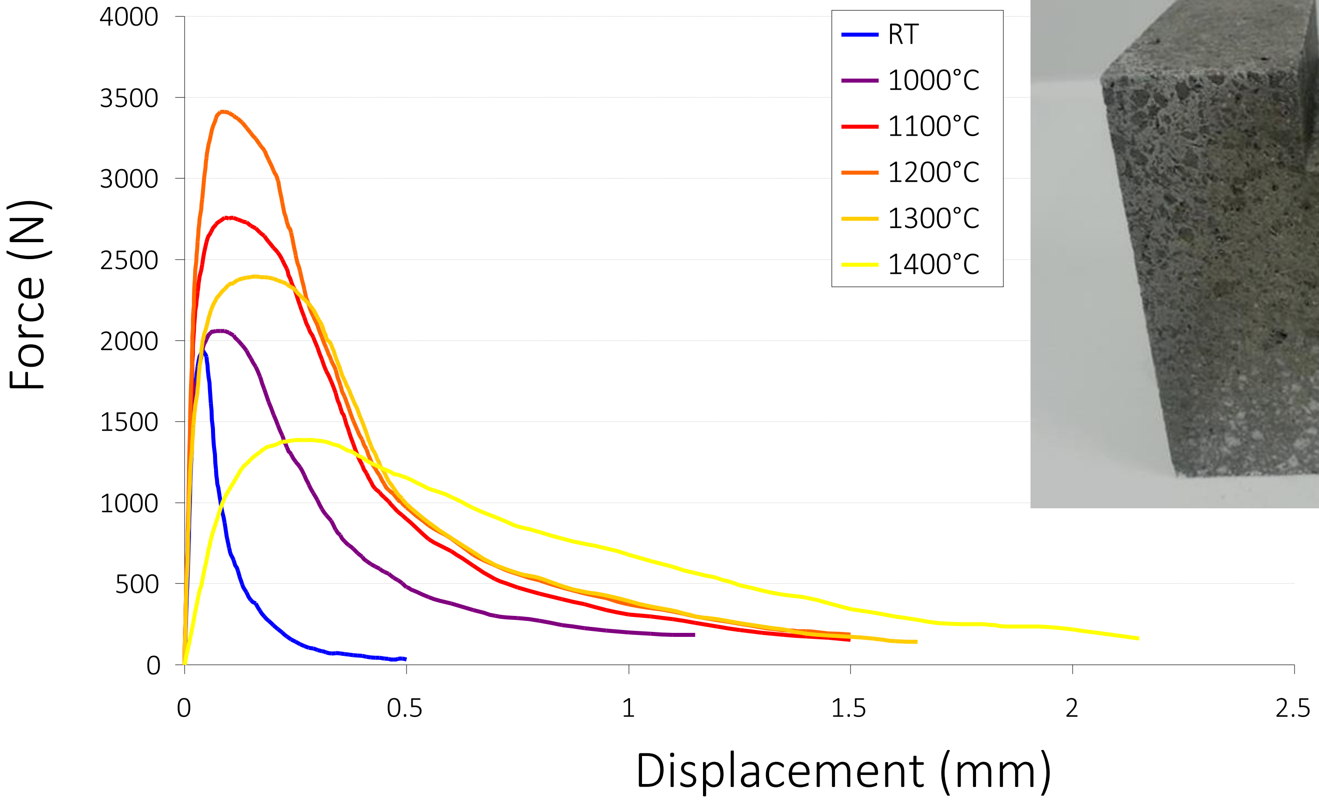
☐ crack initiation

☐ crack propagation

② DEFINITION OF FRACTURE

▶ non linear-elastic fracture mechanics

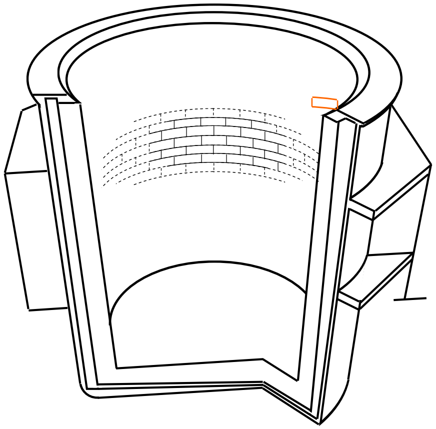
➡ Measuring result from i.e. a magnesia chrome bricks at various temperatures

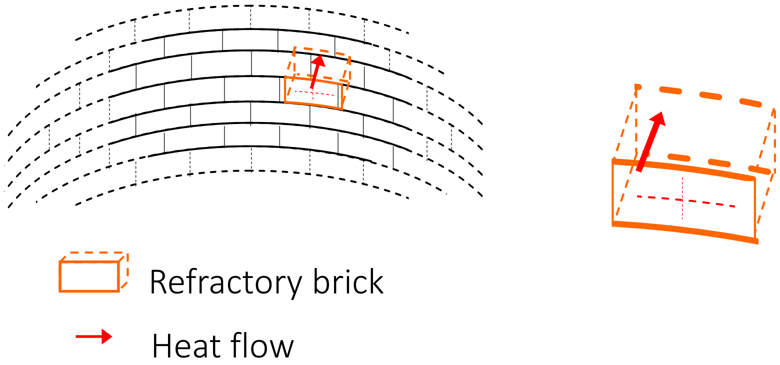


③ THERMOMECHANICS

▶ different basic definitions

➡ during application conditions the refractories are treated with stationary and/or non-stationary thermal loads that create thermal induced stresses





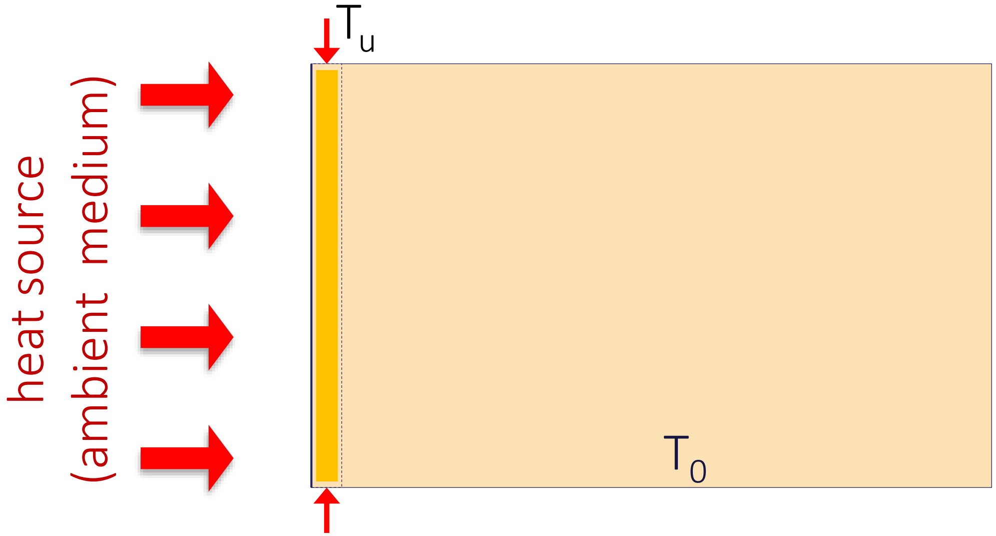
➡ the resistance of materials against harsh temperature changes or thermal stresses respectively is described as thermal shock resistance

③ THERMOMECHANICS

▶ thermal shock resistance parameter

- ➡ to evaluate the thermal shock behavior and the criteria for choosing the right materials under application conditions different concepts and models on the basis of fracture mechanics were developed

- ➡ there is a temperature diference $\Delta T = (T_U - T_0)$ with a critical value T_C a refractory material barely withstand before cracking



$$\varepsilon = \alpha \cdot (T_u - T_0) = \alpha \cdot \Delta T$$

with HOOK´s law : $\sigma = E \cdot \varepsilon$

$$\Rightarrow \quad \sigma = \frac{E \cdot \alpha \cdot \Delta T}{(1-\nu)}$$

③ THERMOMECHANICS

▶ thermal shock resistance parameter

➡ this critical temperature difference generates the resistance against crack initiation

$$\Delta T_c = \frac{\sigma_c \cdot (1-\nu)}{E \cdot \alpha} = R$$

with:
ν = POISSON´s ratio

➡ thermal fatigue resistance is influenced by temperature and coherent stresses and exressed in the form of the thermal stress parameter R

➡ there also exist damage resistance parameter with :

☐ resistance against crack propagation due to crack initiation (non-steady state crack growth)

$$R'''' = \frac{G_f \cdot E}{2 \cdot \sigma_c^2 \cdot (1-\nu)}$$

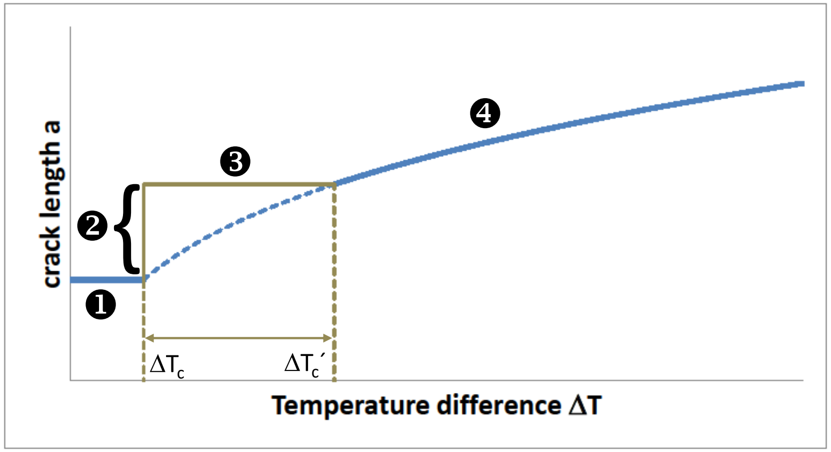
☐ resistance against crack propagation of large cracks (stationary crack growth)

$$R_{st} = \sqrt{\frac{G_f \cdot (1-\nu^2)}{2 \cdot \alpha^2 \cdot E}}$$

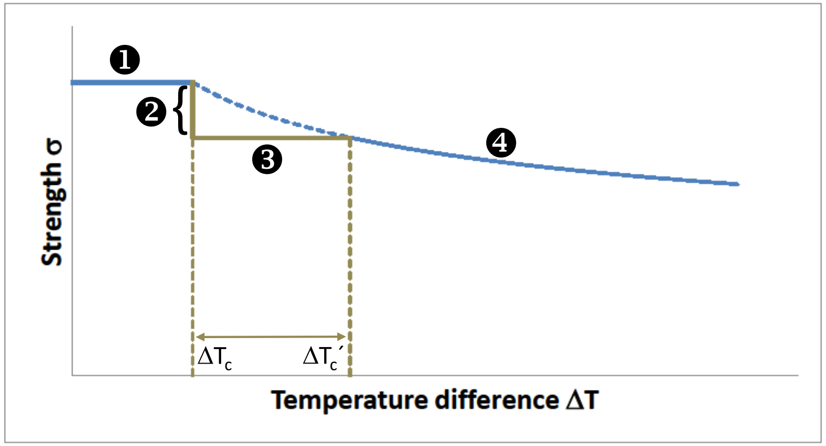
③ THERMOMECHANICS

▶ thermal shock resistance parameter

➡ it is possible to illustrate the characteristic thermal fatigue resistance parameters and their influence



$\Delta T_c \sim R$ and amplitude of ❷ $\sim 1/R''''$



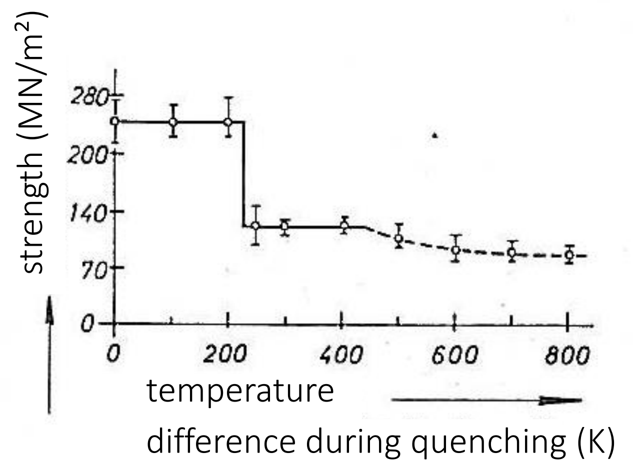
$\Delta T_c \sim R$ and amplitude of ❷ $\sim 1/R''''$

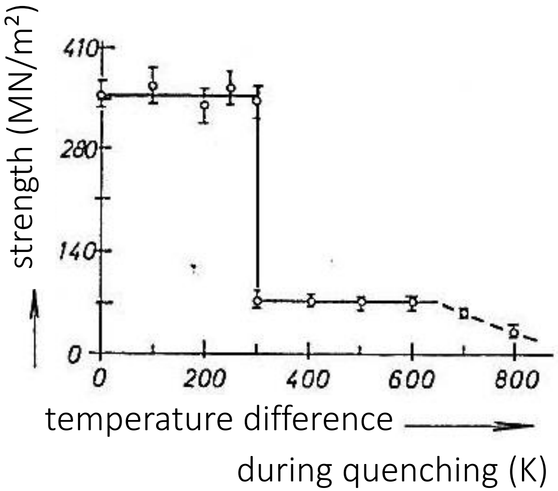
☐ ❶ no crack initiation	→ ❶ no change in strength
☐ ❷ kinetic crack formation	→ ❷ spontaneous decrease in strength
☐ ❸ no change in crack length	→ ❸ no change in strength
☐ ❹ quasi static crack propagation	→ ❹ continuous loss in strength

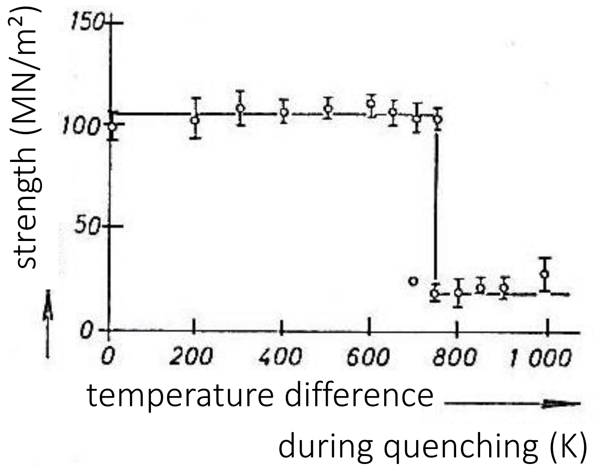
③ THERMOMECHANICS

▶ thermal shock resistance parameter

➡ strength behavior of refractories being quenched in water and resulting fatigue resistance parameter (after Hasselman)







example:

typical values for different refractories

Alumina A	Alumina B	mullite
☐ σ_C = 248 MPa	☐ σ_C = 345 MPa	☐ σ_C = 103 MPa
☐ R = 65	☐ R = 117	☐ R = 225
☐ R'''' = 0.89	☐ R'''' = 0.58	☐ R'''' = 0.19

③ THERMOMECHANICS

▶ thermal shock resistance parameter

➡ avoiding of fracture by complication of crack growth

- ☐ moderate ratio of critical fracture strength to YOUNG´s modulus
- ☐ high value of fracture energy G_F

➡ materials with good thermal shock behavior normally are characterized by :

- ☐ thermal expansion coefficient α : → chamotte bricks, cordierite, . . .
- ☐ specific fracture energy GF : preferably high → magnesia spinel bricks, carbon bonded refractories, . . .
- ☐ thermal conductivity λ : preferably high → carbon bonded refractories, . . .
- ☐ YOUNG´s modulus E : dependent on application
- ☐ strength σC : dependent on application

③ THERMOMECHANICS

▶ measuring methods

➡ standardized methods for measuring thermal shock resistance

- DIN EN 993-11
  - ☐ heating up to T = 950°C
  - ☐ quenching of samples with air up to 30 cycles
  - ☐ alternatively measuring of sonic velocity and/or measuring the bending strength after 5 quenching cycles
  - ☐ sample sizes : mode A – 114 x 64 x 54 mm³; mode B – 230 x 64 x 54 mm³

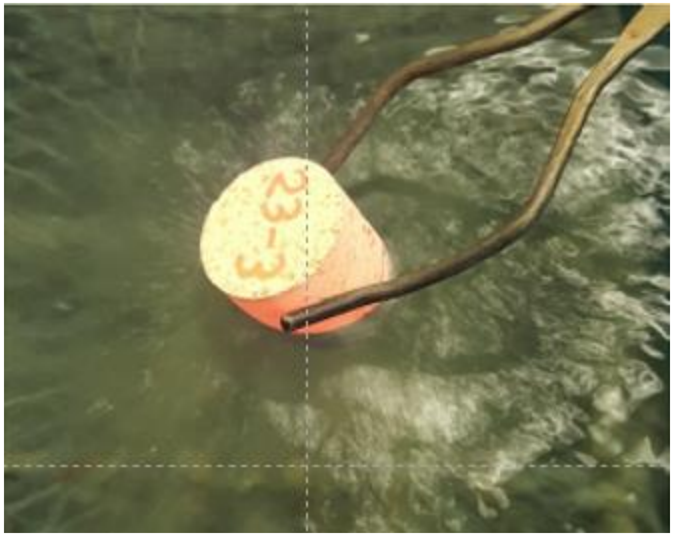
- ASTM C 1171-96
  - ☐ heating up to T = 1.200°C
  - ☐ quenching of samples with air up to 5 cycles
  - ☐ measuring of sonic velocity and/or measuring the bending strength
  - ☐ sample size : 152 x 50 x 50 mm³

③ THERMOMECHANICS

▶ measuring methods

➡ standardized methods for measuring thermal shock resistance

- DIN 51068
  - ☐ heating up to T = 950°C
  - ☐ quenching of samples with water up to 30 cycles
  - ☐ measuring of sonic velocity and/or measuring the bending strength
  - ☐ sample size : cylinder ∅ 50 mm x h = 50 mm

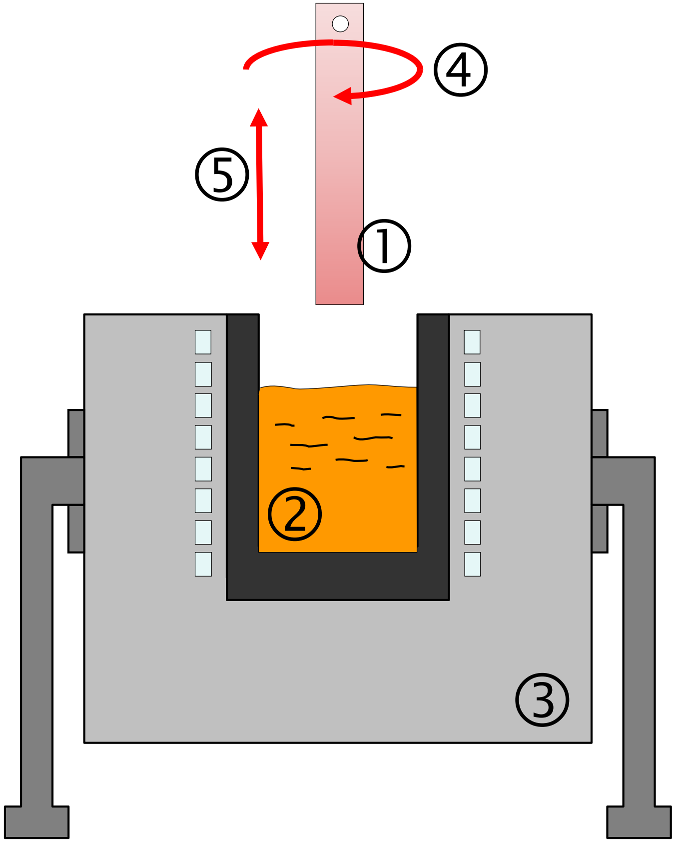


④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance

➡ not standardized technological orientated test procedure for refractories inside melts (metals, glas) and slags

➡ melt immersion test

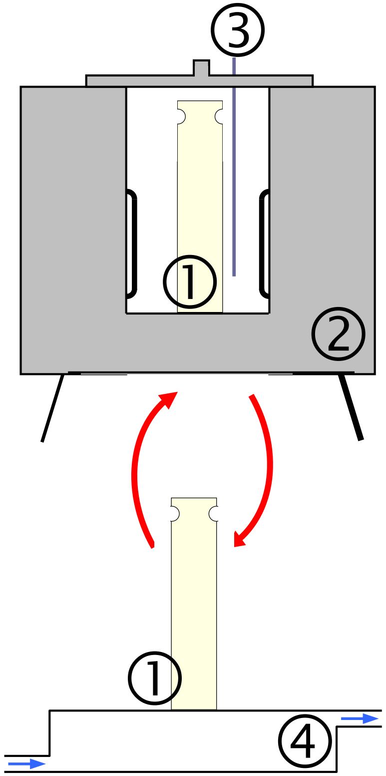


① sample
② metal melt
③ induction furnace
④ option for rotation
⑤ option for lifting

④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance

- ➡ not standardized technological orientated test procedure for refractories with a quasi unidirectional temperature gradient
- ➡ KOLTERMANN test at 1.350°C with on a copper-panel

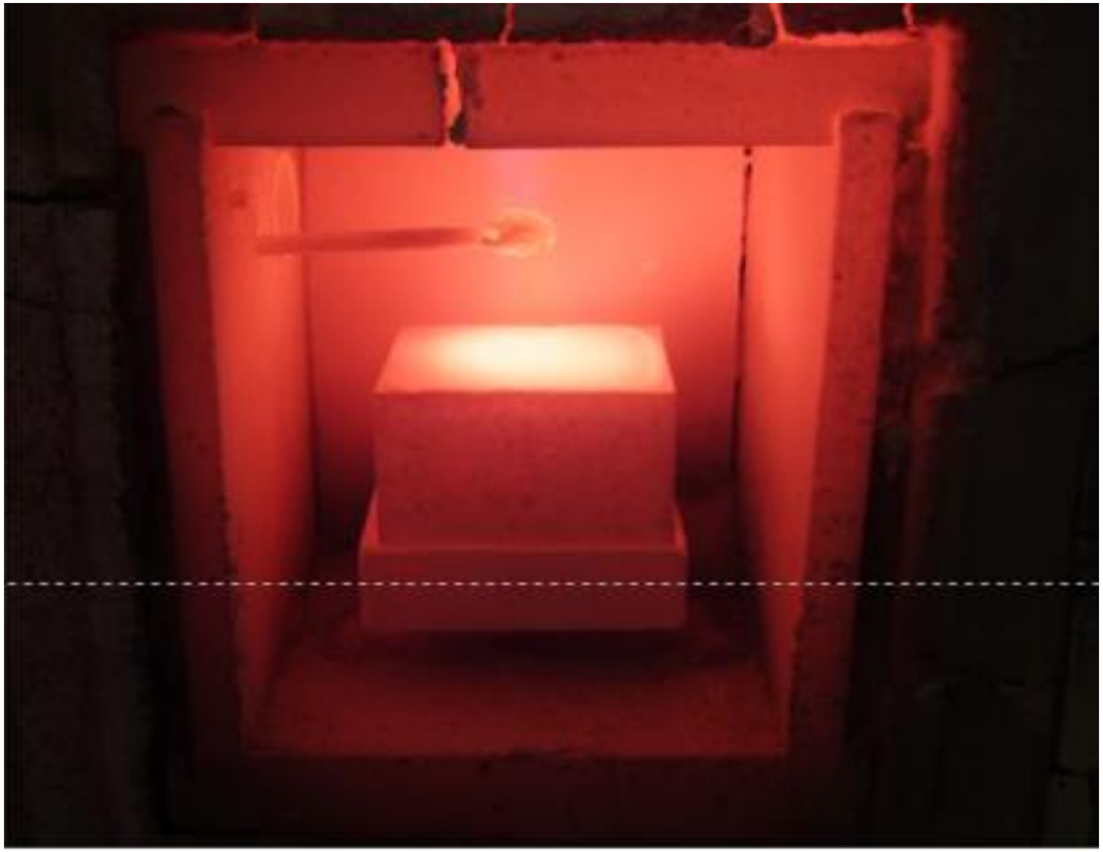


① Sample, ∅ 35 mm, h= 200 mm
② furnace
③ thermocouple
④ water cooled panel

④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance

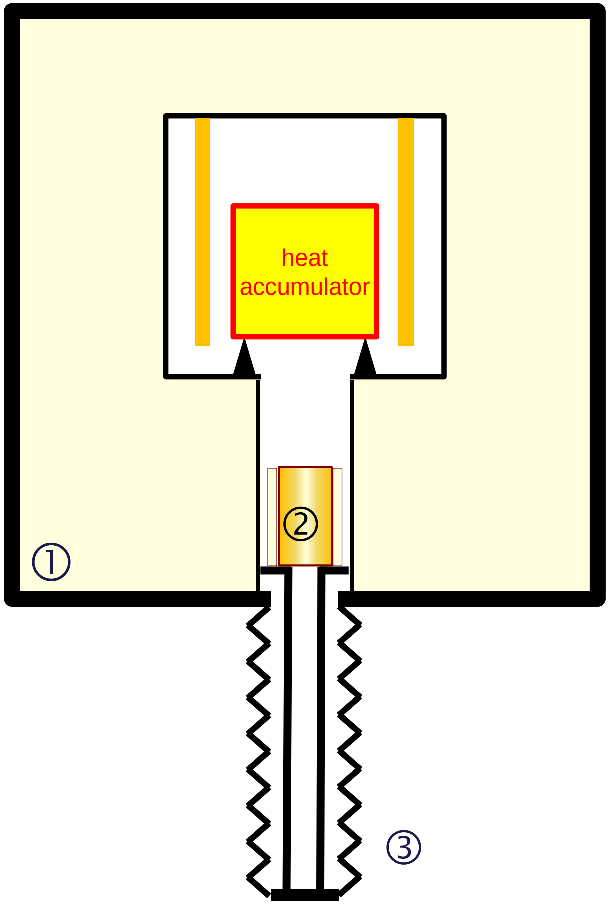
- ➡ not standardized technological orientated test procedure for refractories with cyclic loading at high temperatures
- ➡ with a burner as source
- ➡ with radiation as source



④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance

➡ not standardized application orientated test procedure for refractories with cyclic loading (dwell time : 15 min.) at high temperatures (T = 1.600°C)



① furnace
② sample
③ Lifting system

④ INNOVATIVE TECHNIQUES

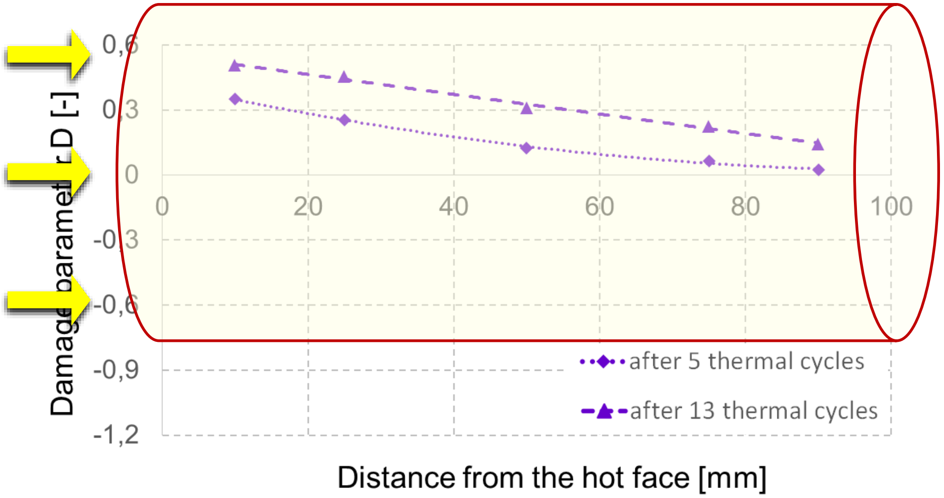
▶ thermal shock resistance

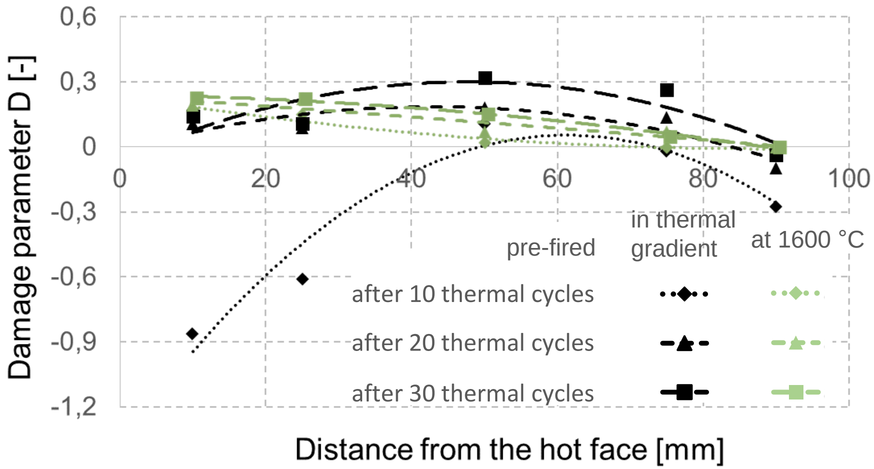
➡ not standardized application orientated test procedure for refractories with the damaging process

$$D = 1 - \left(\frac{v}{v_0}\right)^2$$

example:
Sample: K 99
- ∅ 50 mm, h = 100 mm

example:
Sample: MCA_MSO_MO
- ∅ 50 mm, h = 100 mm





④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance

➡ not standardized application orientated test procedure for refractories with the damaging process

$$D = 1 - \left(\frac{v}{v_0}\right)^2$$

example:

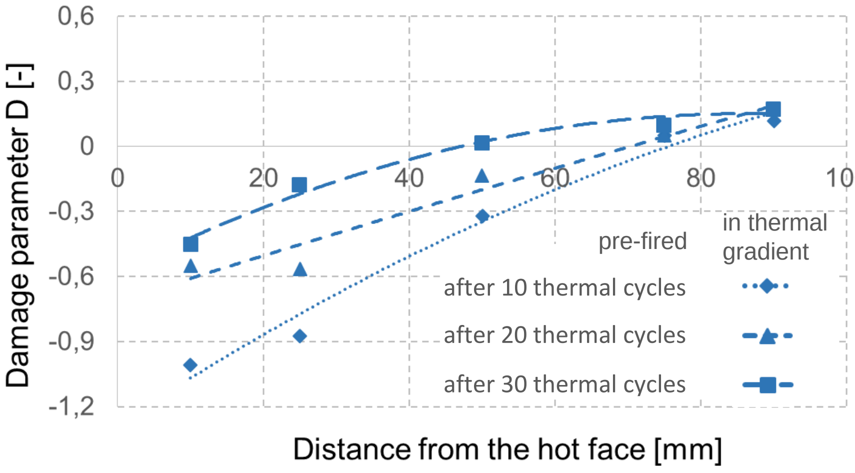
Sample: MCA_MS0,5_M4 (Nedmag)

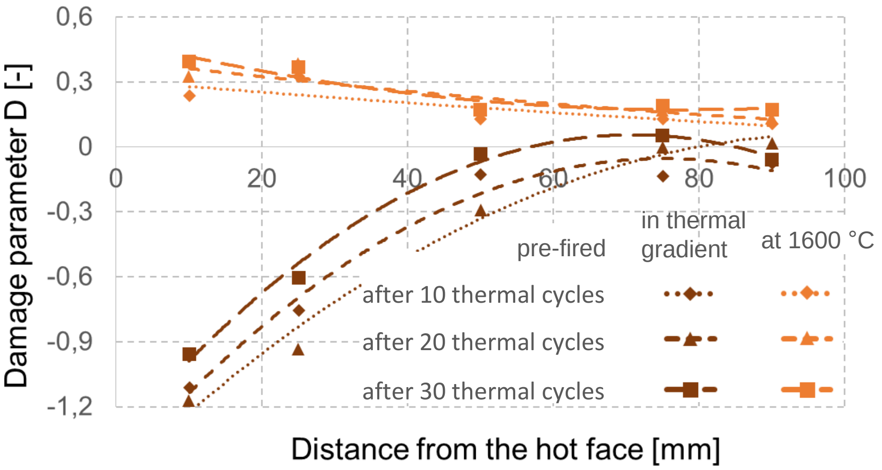
- ∅ 50 mm, h = 100 mm

example:

Sample: MCA_MSO_M4 (magnesite)

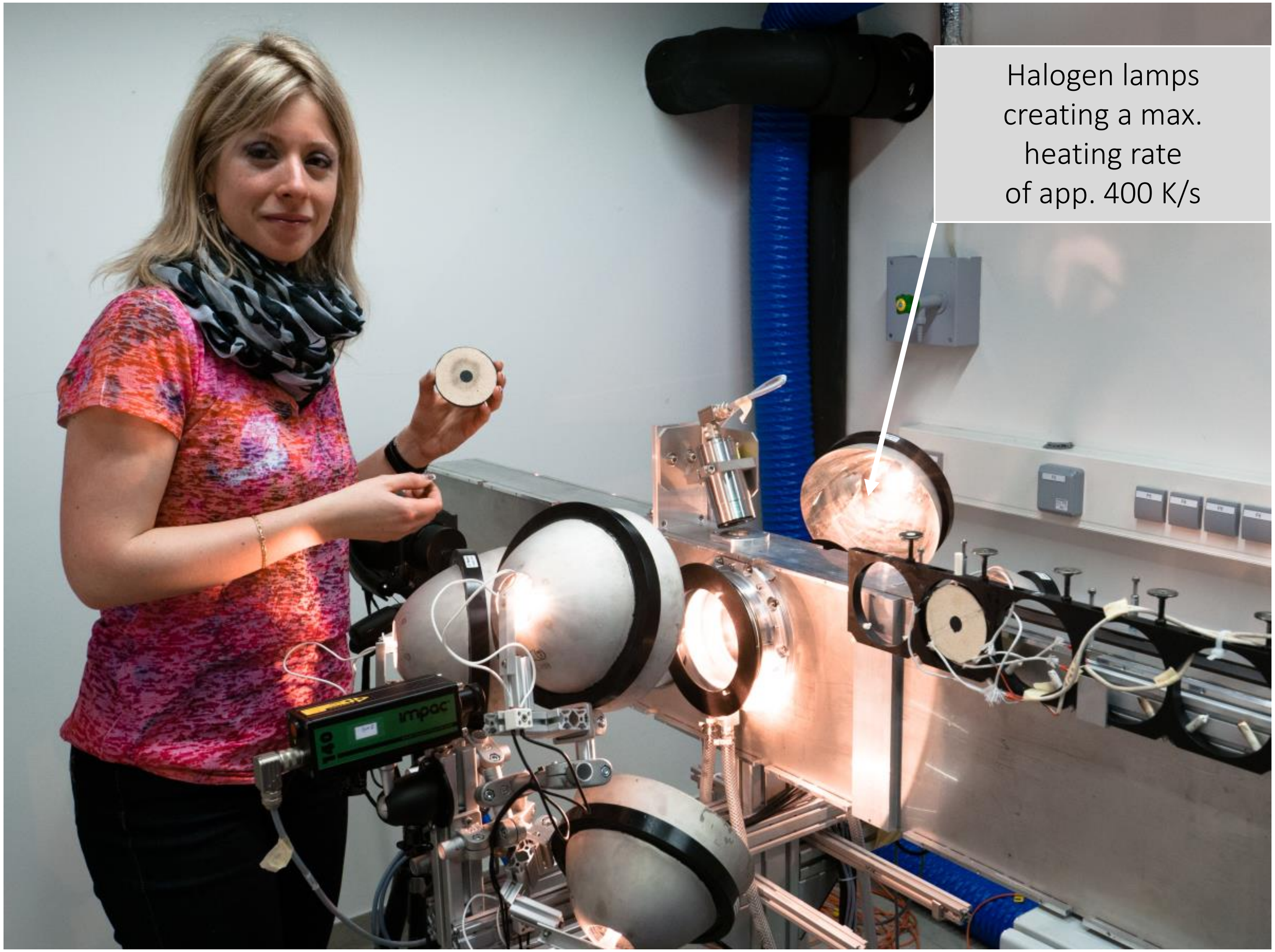
- ∅ 50 mm, h = 100 mm





④ INNOVATIVE TECHNIQUES

► thermal shock behavior

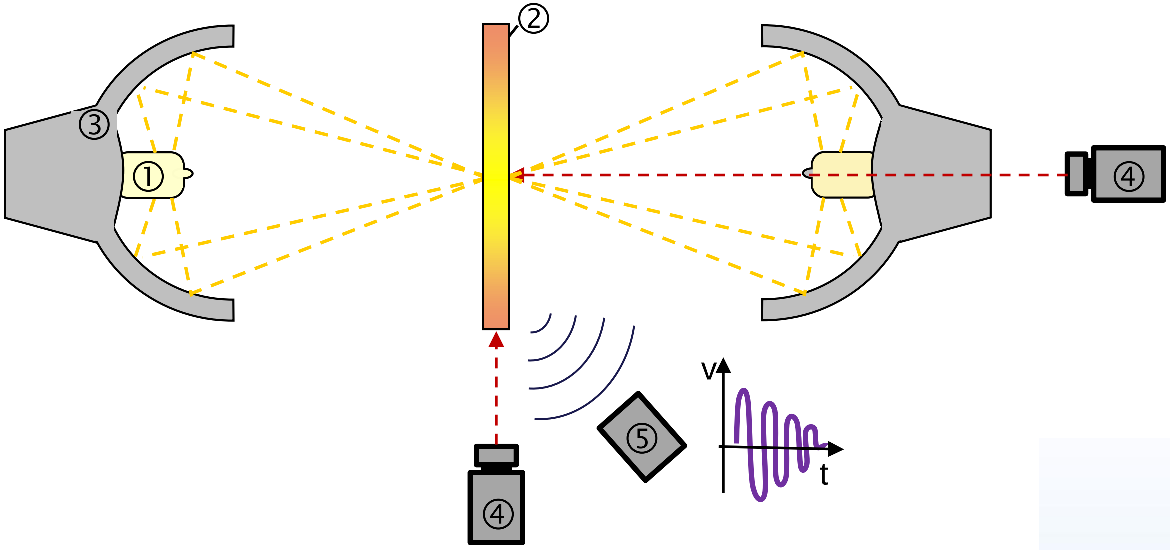


Halogen lamps creating a max. heating rate of app. 400 K/s

④ INNOVATIVE TECHNIQUES

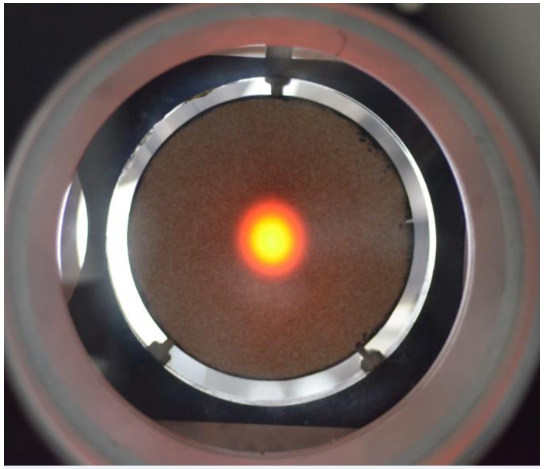
▶ thermal shock behavior

➡ Radiation based method

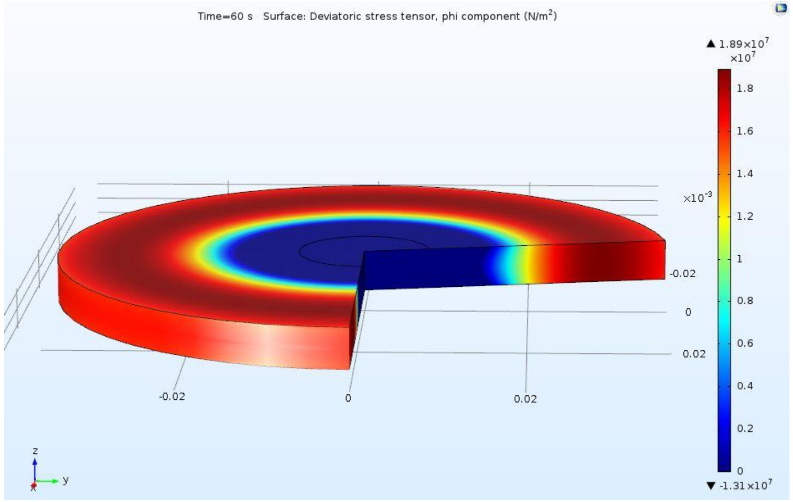


① Halogen bulb
② Sample
③ Reflector

④ Pyrometer
⑤ Acoustic emission sensor



T_{max} (centric) ≈ 1100 °C

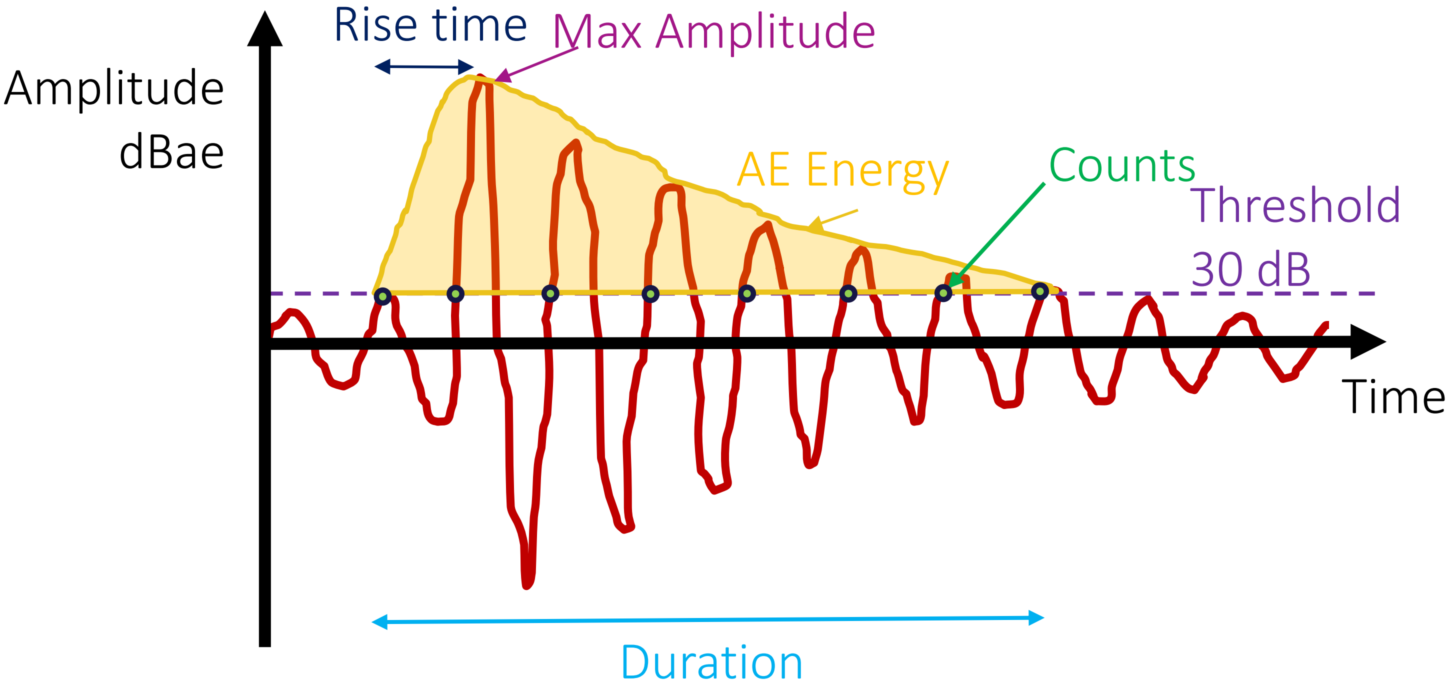


Stress distribution (circular direction)

④ INNOVATIVE TECHNIQUES

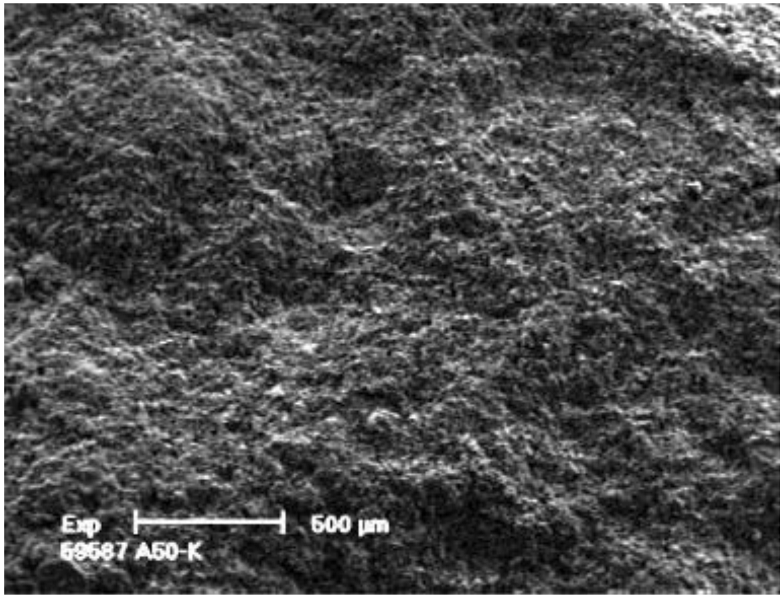
▶ thermal shock resistance

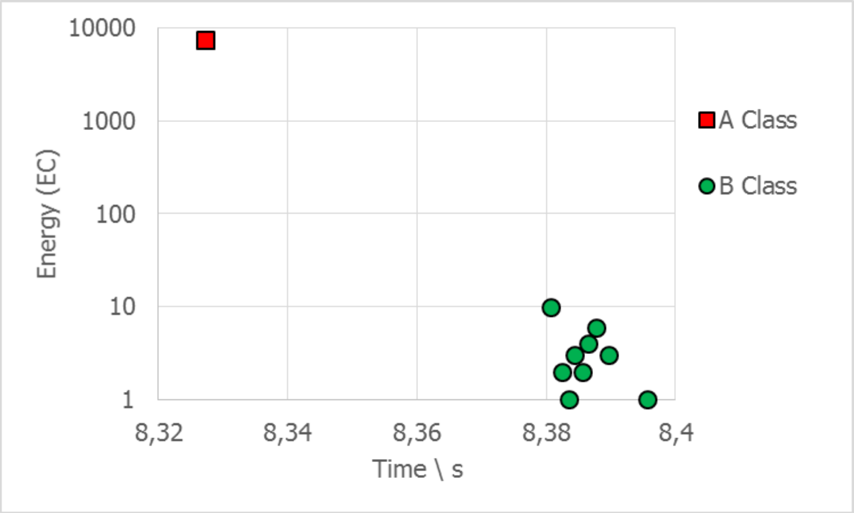
➡ not standardized application orientated test using acoustic emission detection for characterizing cracks and various energies



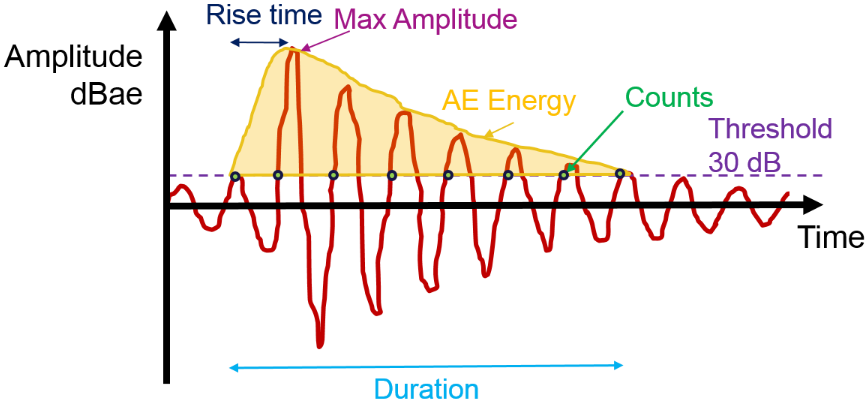
④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance



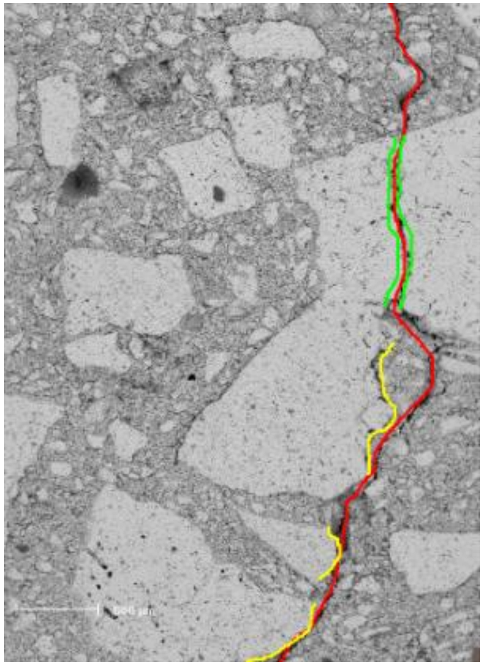


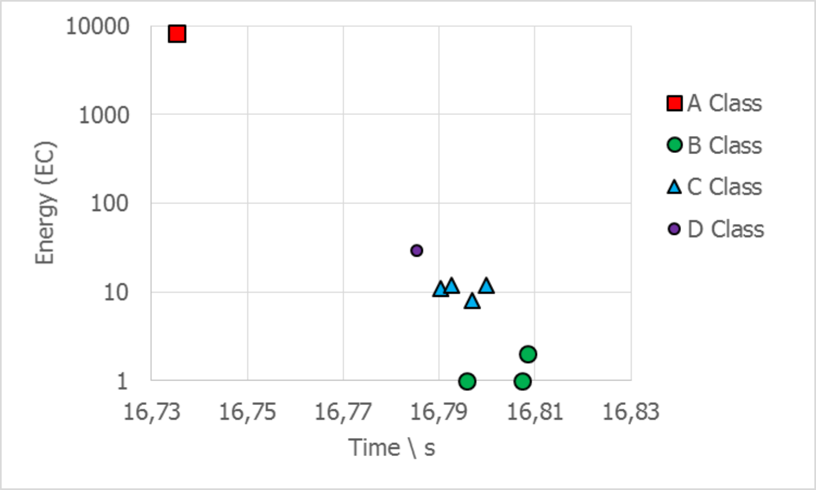
Crack initiation	✓
Matrix cracking	✓
Debonding	X
Grain fracture	X



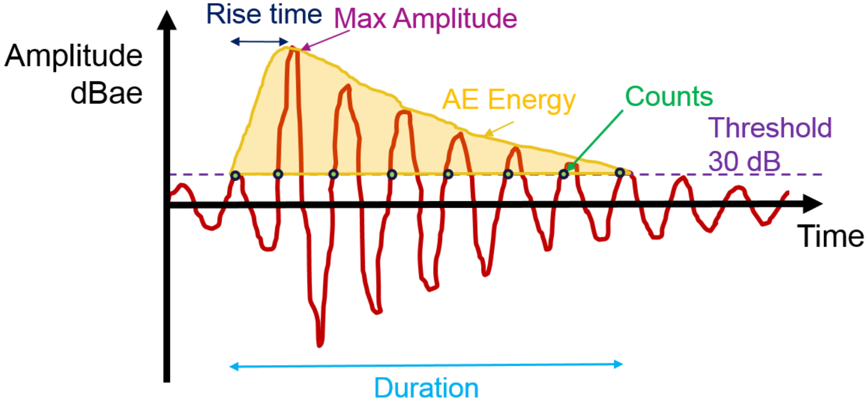
④ INNOVATIVE TECHNIQUES

▶ thermal shock resistance





Crack initiation	✓
Matrix cracking	✓
Debonding	✓
Grain fracture	✓



⑤ THANKS

▶ for your attention

➡ since Jan, 1st 2013 DIFK is in active service within ECREF

⑤ THANKS

► for your attention

Loriot