Sicherheit in Technik und Chemie

HELSMAC- Cambridge, UK

A FEW ASPECTS OF THE CURRENT UNDERSTANDING OF DPF MATERIALS THERMAL AND MECHANICAL PROPERTIES

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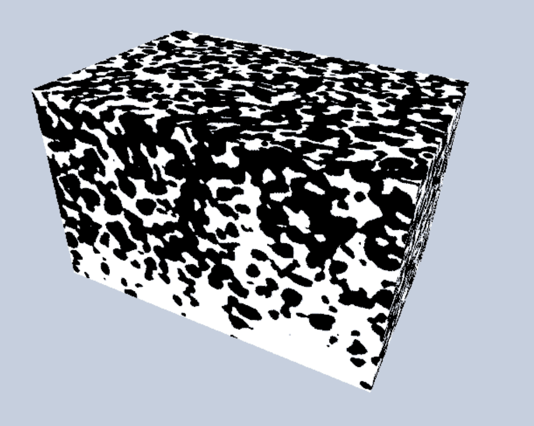

BAM guideline and mission

Safety in technology and chemistry

Pursuing our mission as a Federal institute for materials technology and chemical engineering, we ensure ongoing safety in technology and chemistry through

- research and development
- testing, analysis, approval and certification
- consultation, information and advice

within our objective of promoting German (and EU) industrial development.




Porous Ceramics in Industry

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Applications as

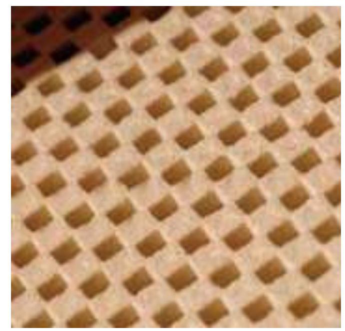
- Filters (mobile/ stationary)
- Membranes
- Substrates for catalyzers

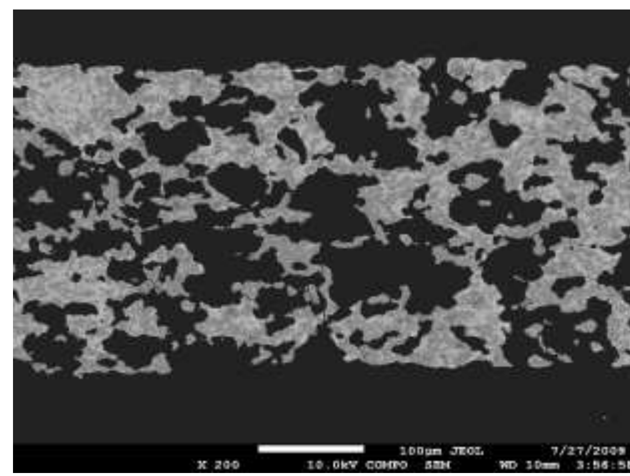
Typical materials

- Cordierite
- Silicon Carbide
- Aluminum Titanate
- β-Eucryptite

Typical Characteristics

- Low thermal expansion (not all)
- Microcracking (not all)
- High permeability
- High thermal shock resistance
- Strain tolerance

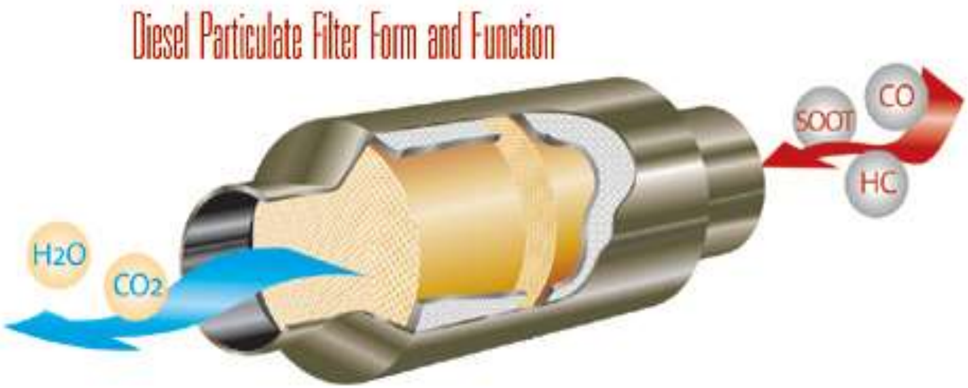


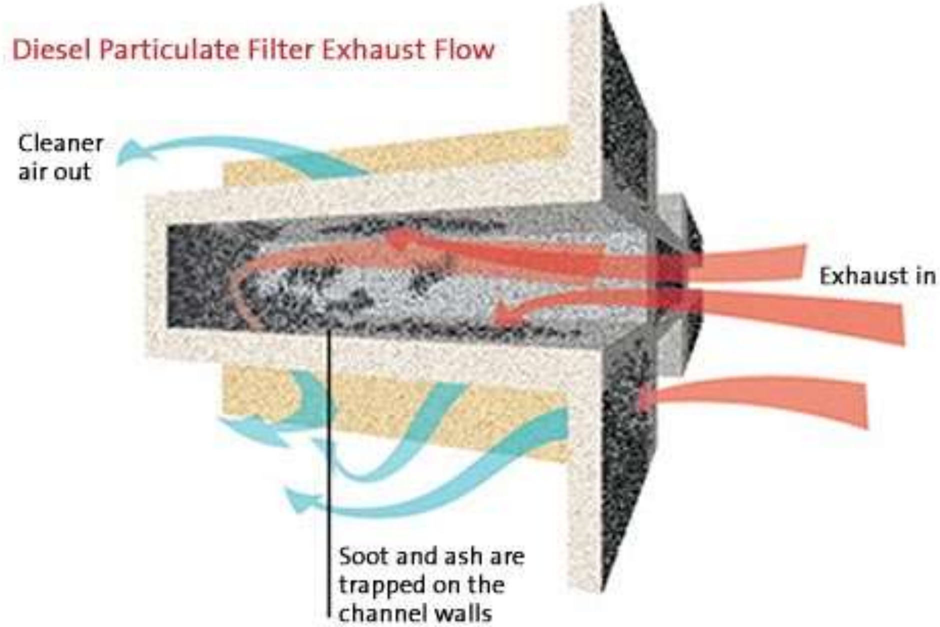


Porous Ceramic Filters

Main attributes

- Filtration efficiency
- Pressure drop
- Resistance to crack initiation
- Operation at high temperatures





Facts

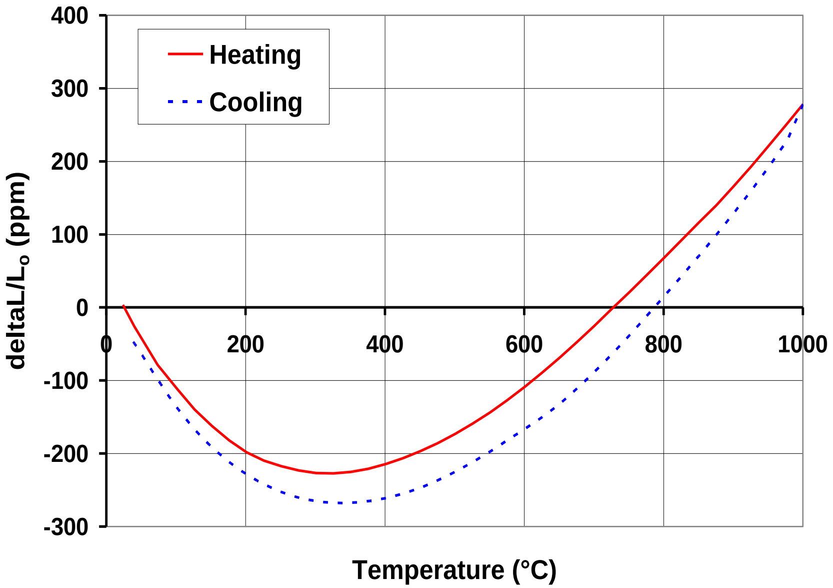
Microstructure

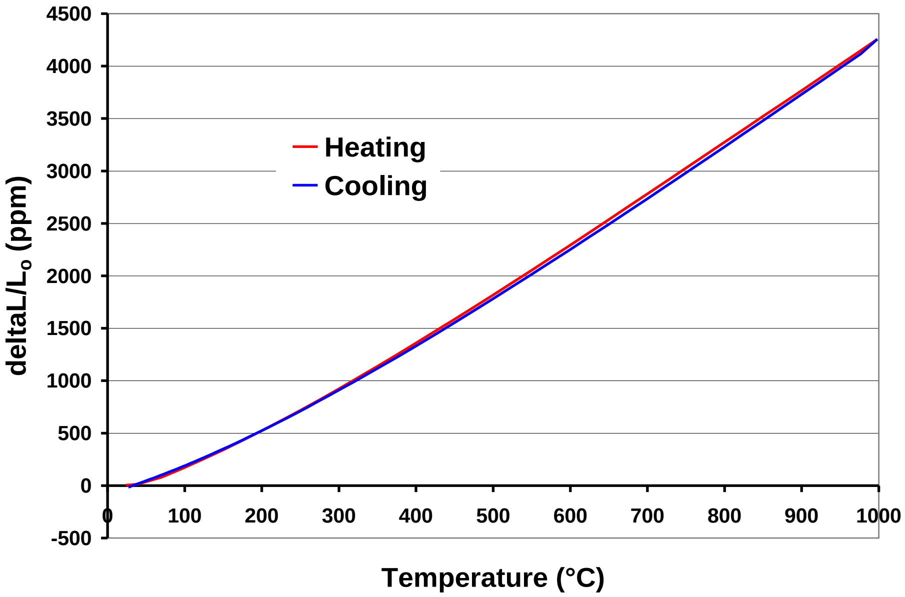
Mechanical behavior

Thermal behavior

DPF Ceramics:
Microstructure and Thermal Properties

Thermal expansion



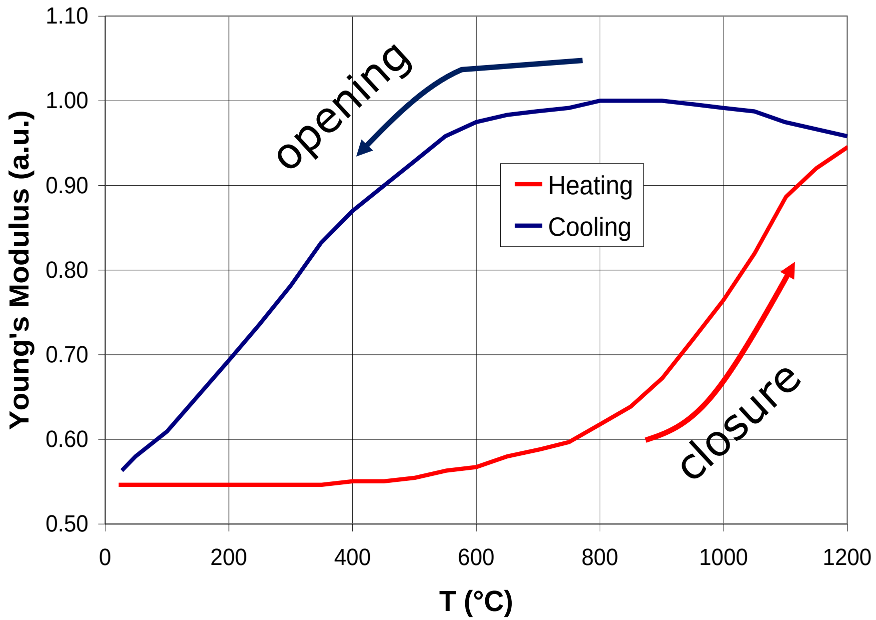


Cordierite

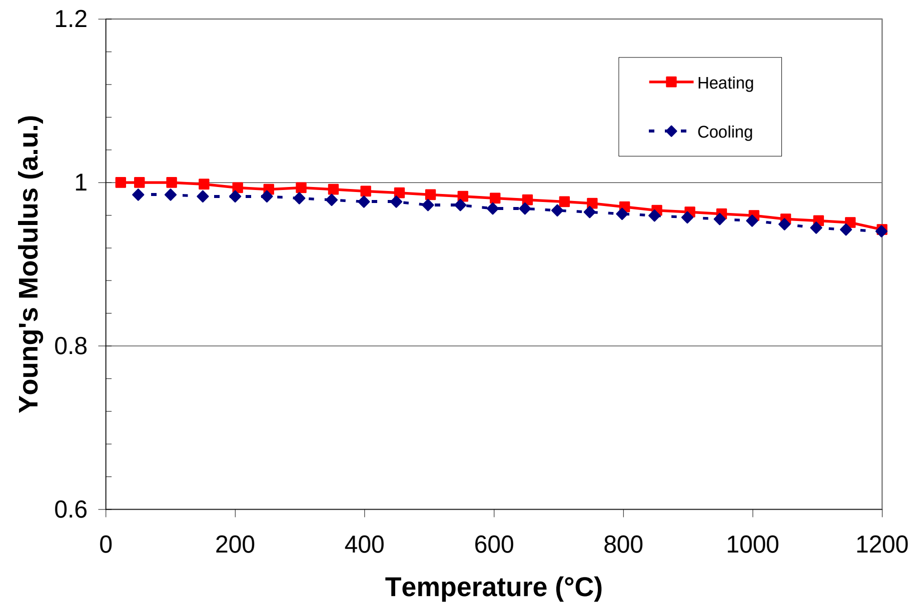
Silicon Carbide

DPF Ceramics:
Mechanical Properties

Young's Modulus



Cordierite



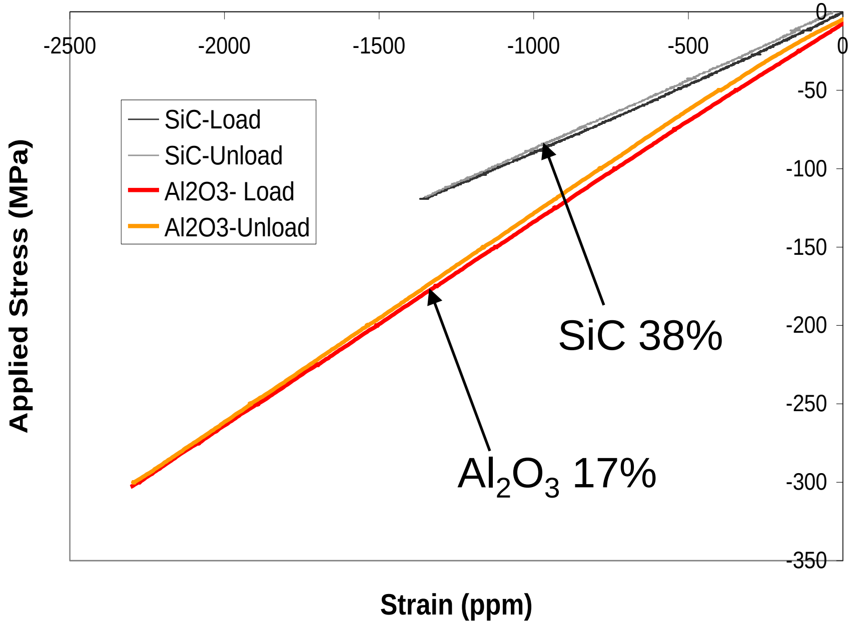
Silicon Carbide

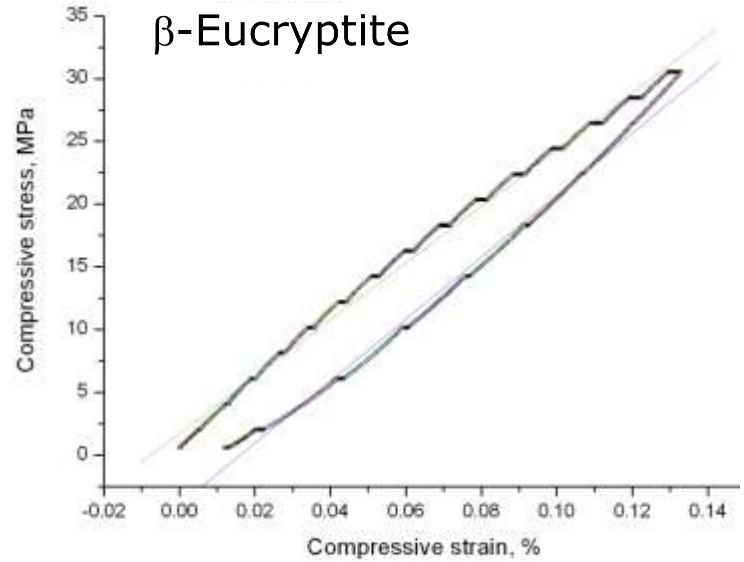
Microcracking often occurs because of lattice thermal expansion anisotropy (NB: low crystal symmetry ceramics)

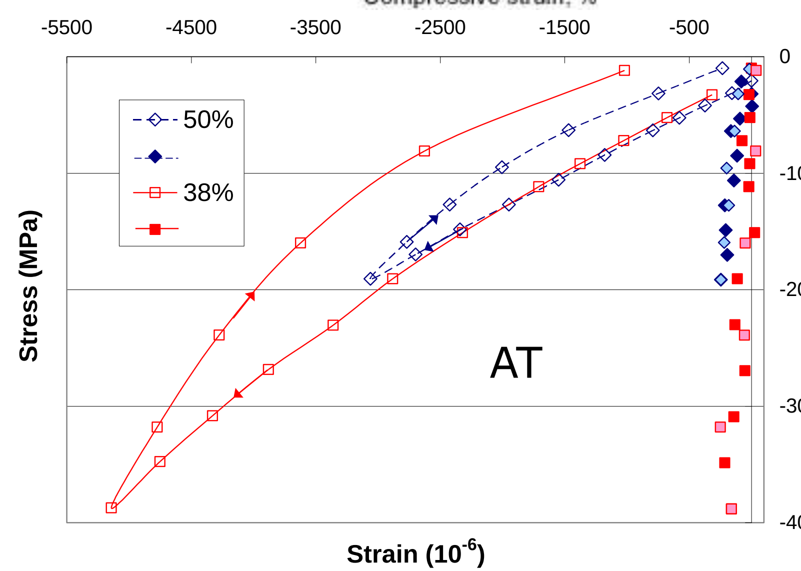
Hysteresis of dilation and Young's modulus *vs* T: thermal microcracking

Non-linear stress-strain behavior

Mechanical microcracking







Very visible in microcracked materials, present in non-microcracked

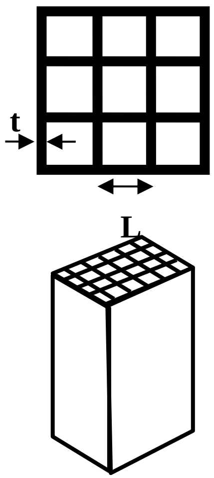
In detail

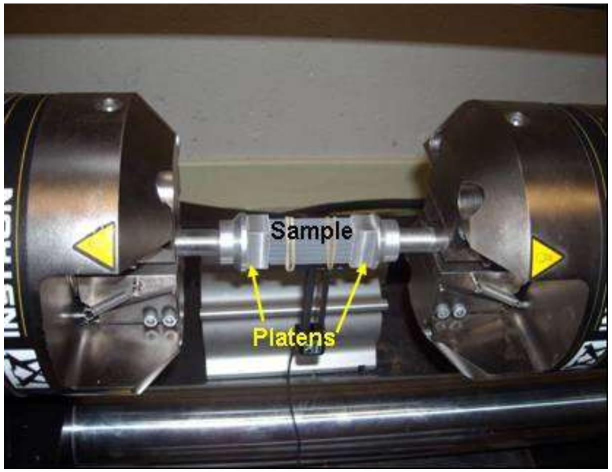
Mechanical behavior and its modeling

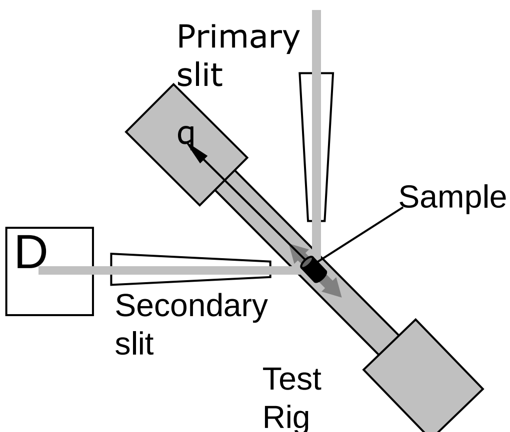
Thermal behavior and its modeling

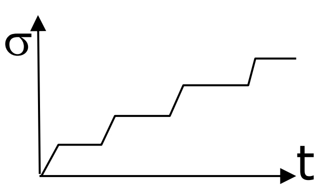
Microstructural aspects

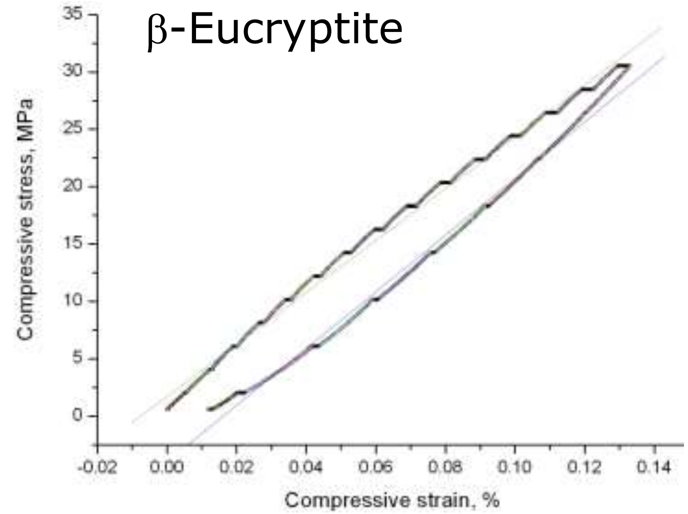
Stress-strain curves

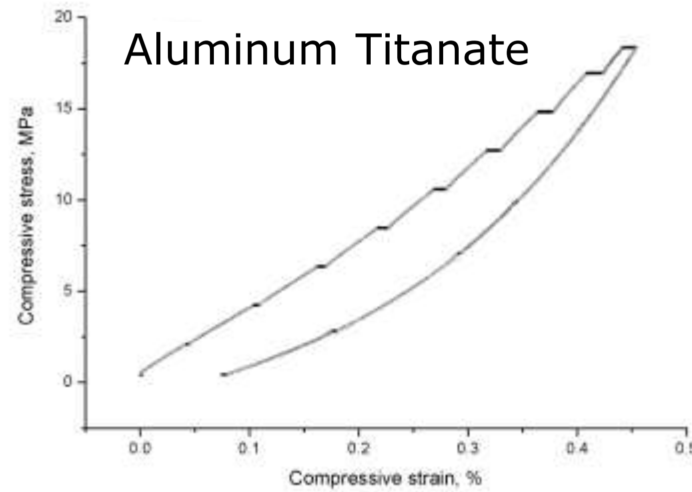




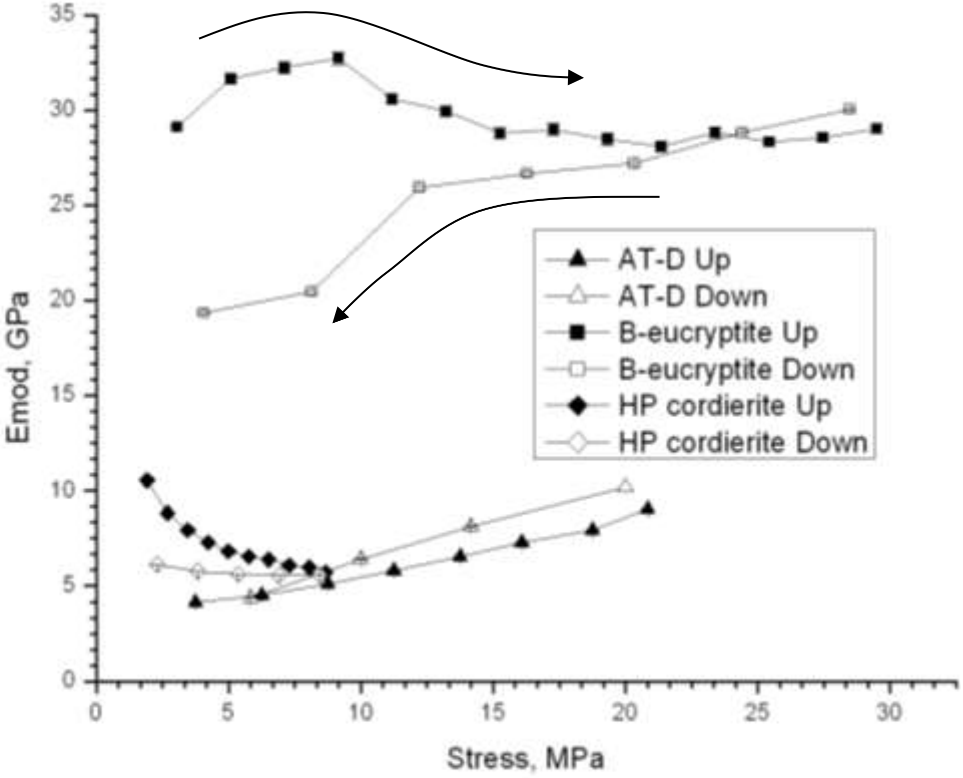






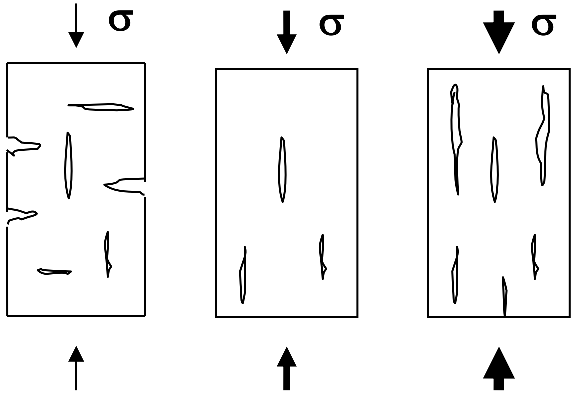


New constitutive laws for porous and microcracked ceramics



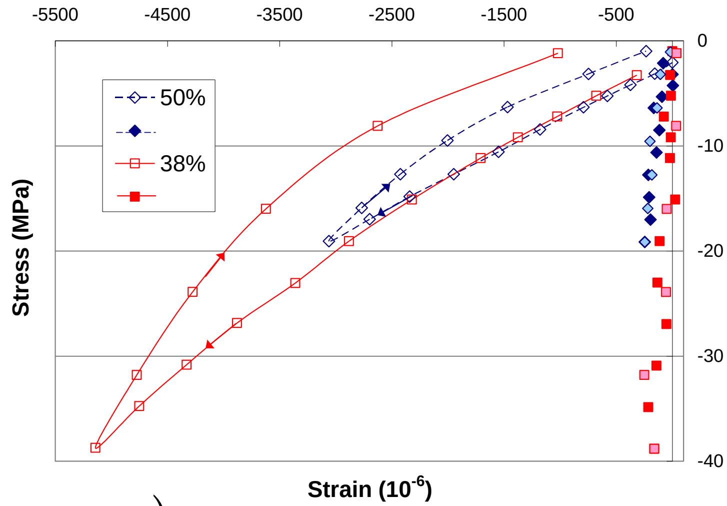
Emod vs load:
- increases continuously for AT
- decreases continuously for UHPC
- has two regimes for β-eucryptite

From μ-crack closure to opening



- Stress Modeling needs to take the dynamic behavior into account
- Operating conditions cannot be described by static properties
- Mechanical microcracks are irreversible and cannot be closed by load release: they are directional and larger

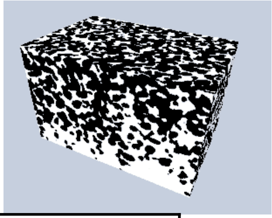
Modeling (AT)



Micromechanics:
Differential Scheme

$$\frac{E_0}{E_{eff}} = (1-p)^{-C_1} \cdot \exp\left(C_2 \rho^{open} + C_3 \rho^{sliding}\right)$$

From tomography

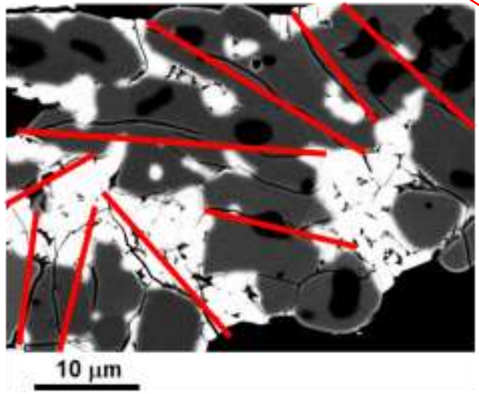


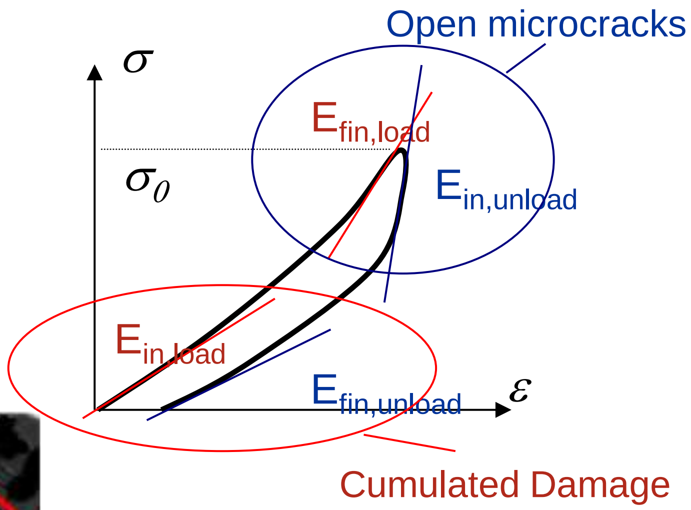
Density Parameter	Theory + Stress-Strain Curves	
	Porosity (%)	
	38	50
$\rho^{open,\ initial}$	1.44	1.17
$\rho^{open,\ peak}$	1.01	0.97
$\rho^{sliding,\ peak}$	1.08	0.83

$$\rho^{open,\ peak} + \rho^{sliding,\ peak} > \rho^{open,\ initial}$$

$$\rho = \frac{1}{V} \sum_i a^{(i)3}$$

a^i = microcrack radii





Mechanical Properties Summary

- **New relations** between macro and micro stress and strain for porous materials

- Microcracking induces a viscous behavior (stress-strain curves become time dependent)

- At least two parameters are needed to describe the *E vs. p*

- Differential model yields microcrack density (agreement with Eshelby approach on Emod vs T data): microcrack sliding is extremely important

- Diffraction Young's modulus rescales with porosity

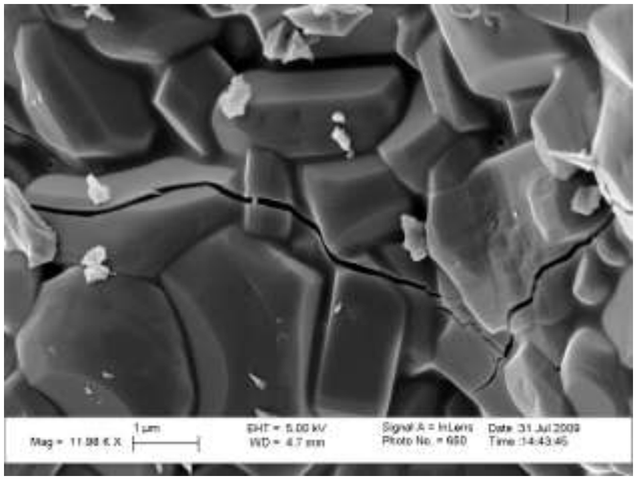
In detail

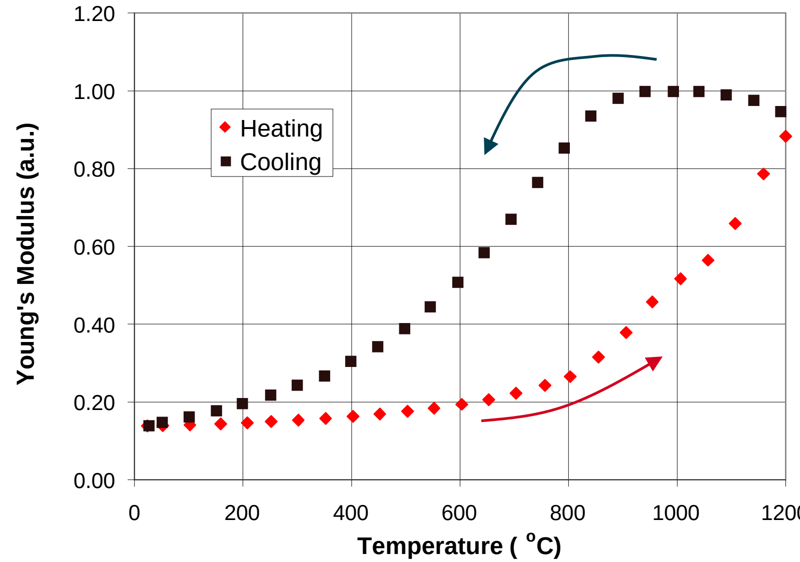
Mechanical behavior and its modeling

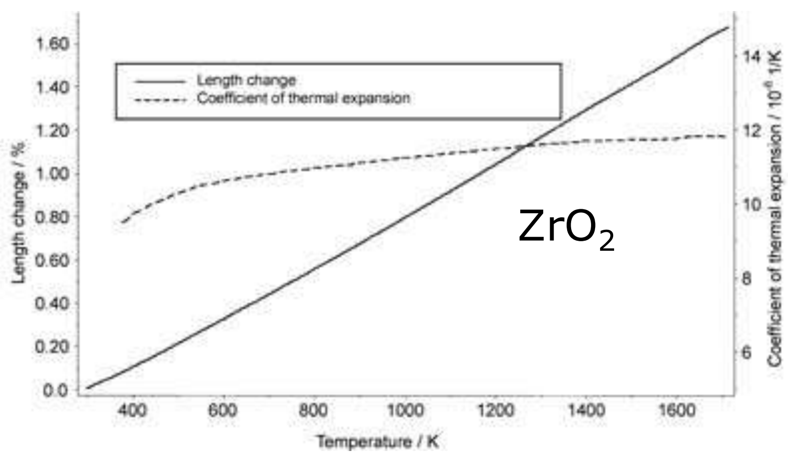
Thermal behavior and its modeling

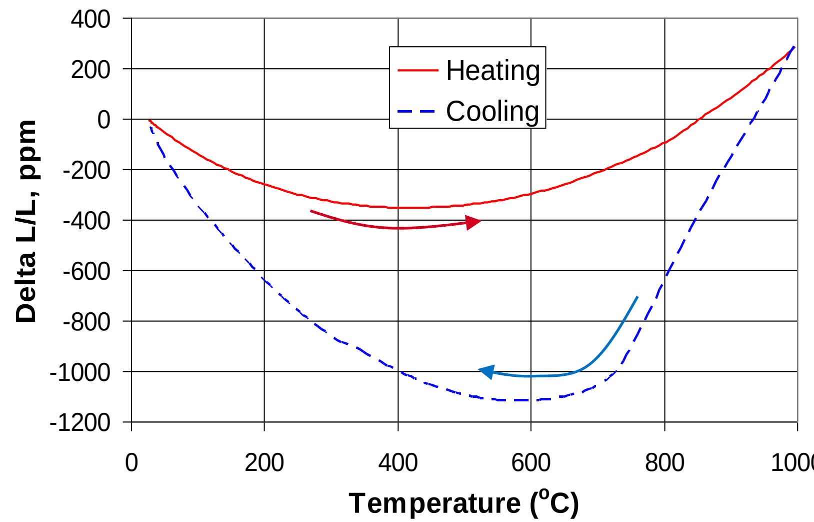
Microstructural aspects

Thermal expansion of Aluminum Titanate









Lattice expansion and Texture of AT

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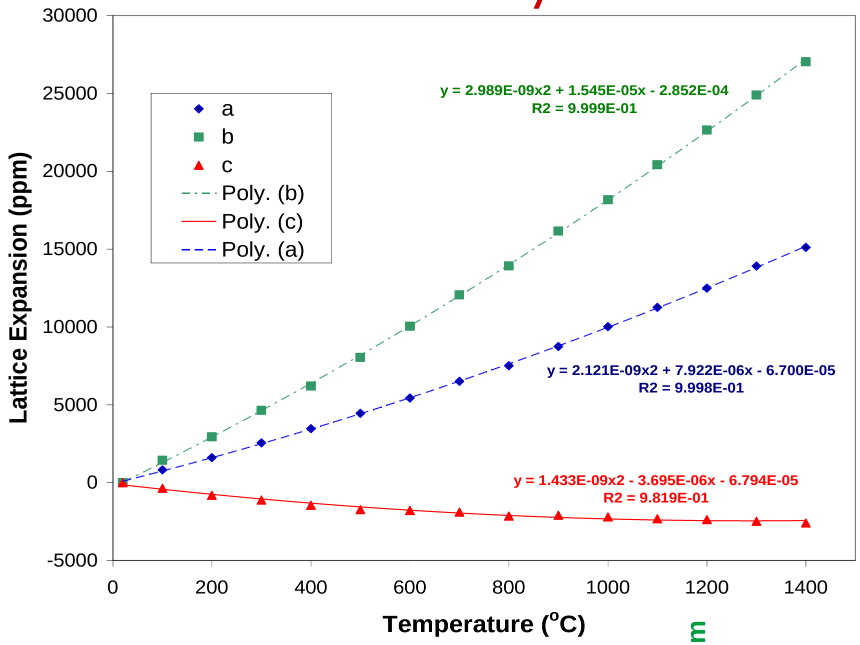
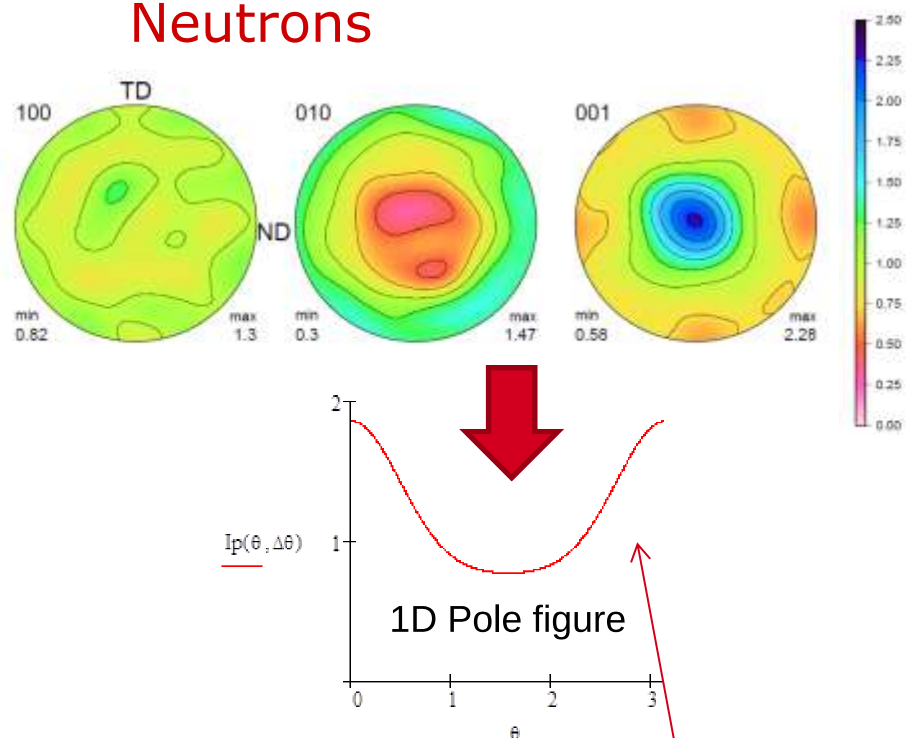
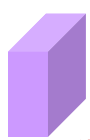
X-Rays



Neutrons



- Different a,b,c
- Negative c-axis



$$\varepsilon^{bulk} = \frac{\sum \varepsilon^{lattice}{}_i \cdot IF_i \cdot V_i \frac{E_i}{E_{Pi}+E_i}}{\sum IF_i \cdot V_i \frac{E_i}{E_{Pi}+E_i}}; \qquad \sum V_i \cdot \sigma_i = 0$$

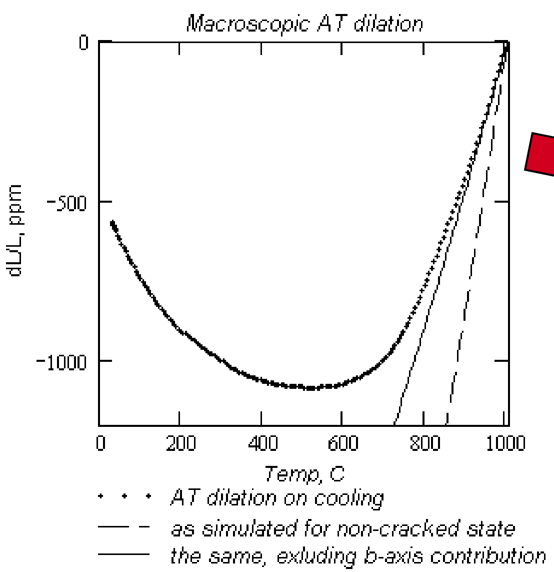
$$\sigma_i = \left(\varepsilon^{bulk} - \varepsilon^{lattice}{}_i\right) \cdot IF_i \cdot \frac{E_i \cdot E_{Pi}}{E_{Pi}+E_i} \cdot A$$

Integrity Factor model (Efremov, Phil Mag., 2013)

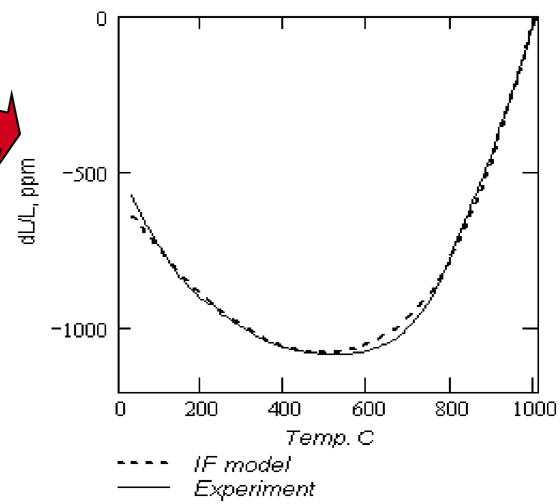
AT macroscopic axial CTE simulation

Modeling

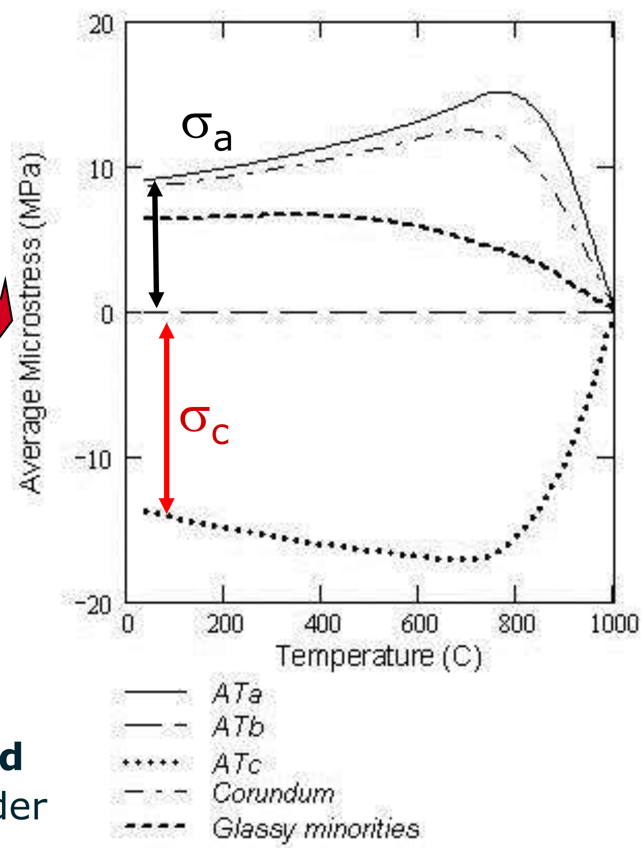
IF model- Matching the slope at HT



IF model- dilation curve



IF model- stress



- At all temperatures the ***c*-axis** remains attached to the body and is under **compression**
- Even at high temperatures the ***b*-axis** must be **disconnected**
- At RT, the **c-axis** is under **compression**, the **others** are under **tension**

Thermal Properties Summary

• **Mechanical and thermal microcracks** have different orientations

• Aluminum titanate behaves highly anisotropically: internal micro-stresses

• The **Integrity Factor Model** can rationalize the high-temperature behavior of Thermal Expansion and calculate stresses

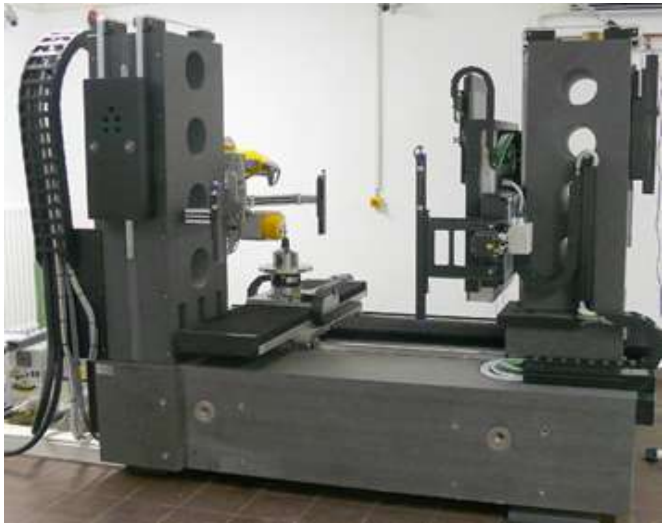
In detail

Mechanical behavior and its modeling

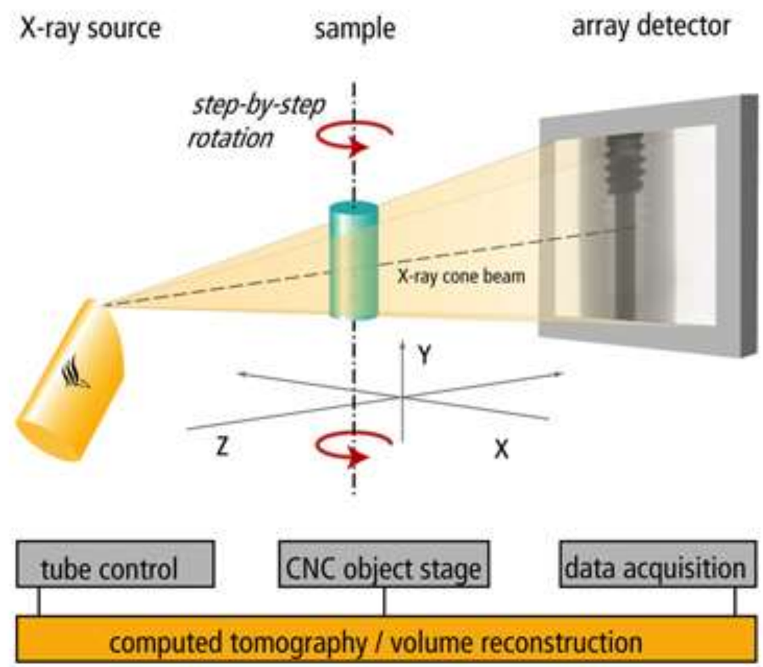
Thermal behavior and its modeling

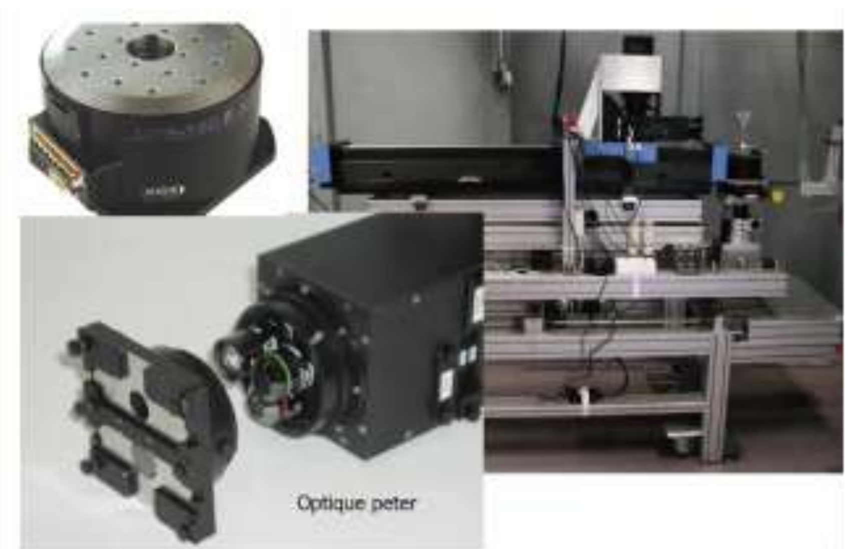
Microstructural aspects

Pore orientation by means of Computed Tomography



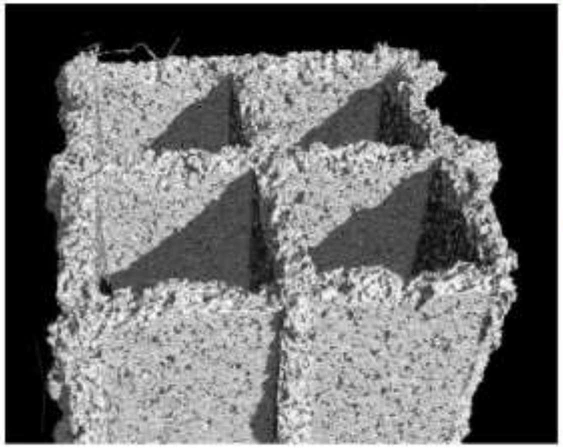
GE v|tome|x
Pixel size ~ 4 mm



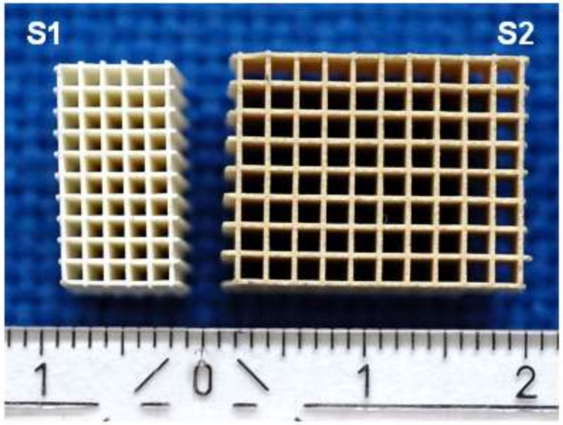


BAMLine, BESSY, Berlin, Germany
Pixel size ~ 0.4 mm

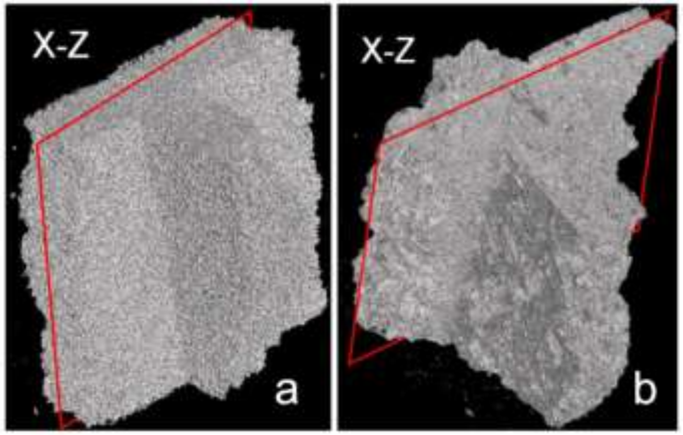
Results- CT reconstructions



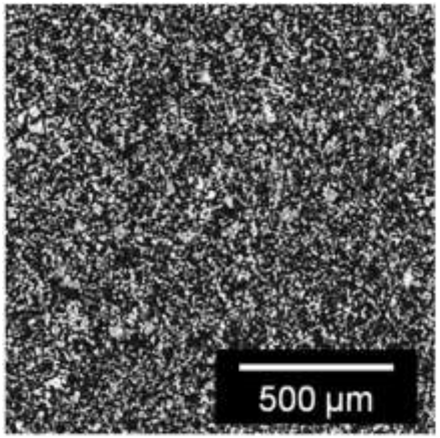
Laboratory CT Data



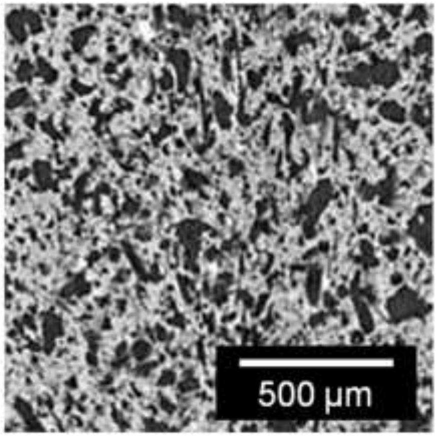
Cordierite samples



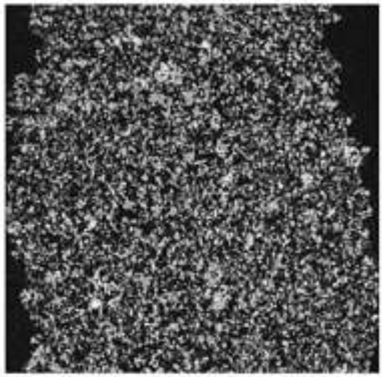
Synchrotron CT Data



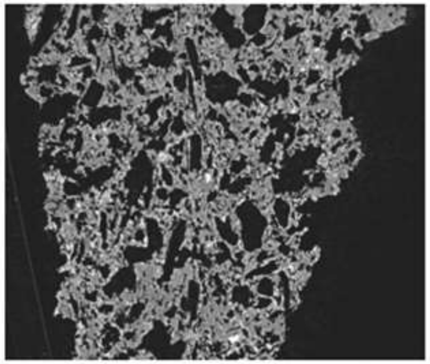
S1



S2

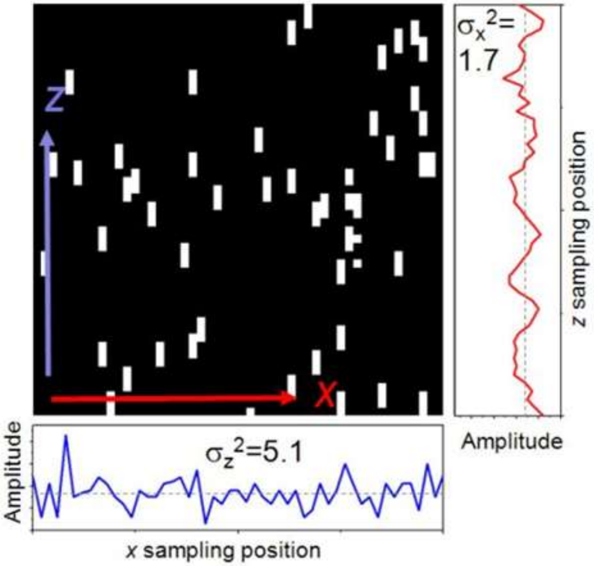


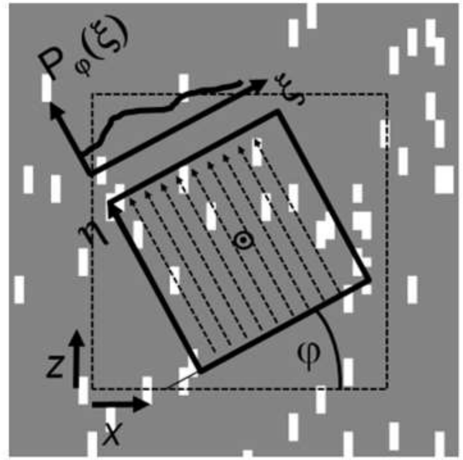
S1

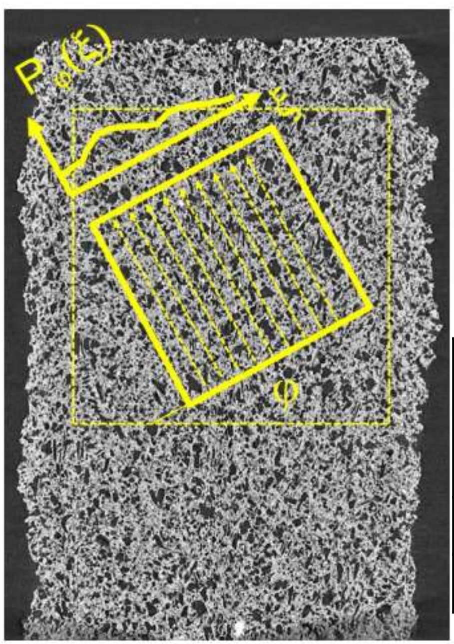


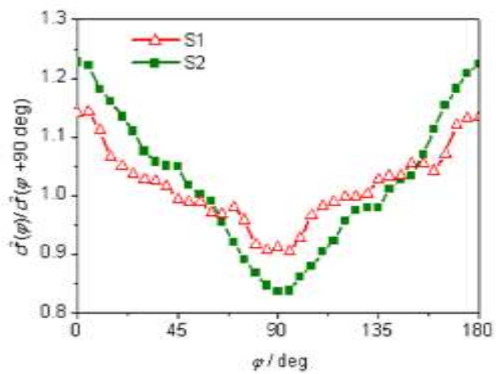
S2

CT Data Analysis: DIVA









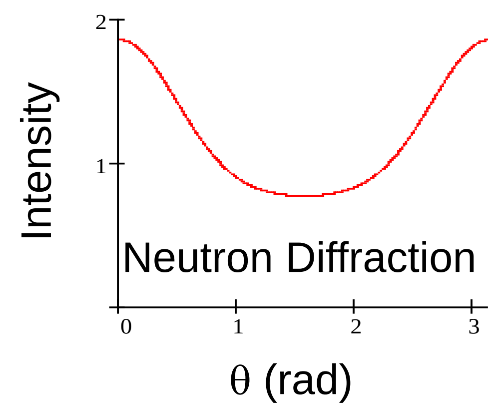
	X-ray Refraction (‖/⊥)	µ-CT DIVA (‖/⊥)	Synch. CT (‖/⊥)	Neutron Diffraction
S1	0.92	0.92	0.85	-
S2	0.86	0.84	0.74	0.77

- Calculation of the Variance of Gradients

$$\sigma_P^2(\varphi) = \frac{\sum_{\xi_i}^{n}\left(P_\varphi(\xi_i) - \langle P_\varphi \rangle\right)^2}{n}$$

$$P_\varphi(\xi) = \int_V |\nabla\mu(\mathbf{r})|\, d\eta$$

- Orientation parameter O_D by ratio of max to min variance



Morphology and crystal orientations coincide

Microstructure Summary

• **Morphological and crystallographic** orientations are the same in cordierite

• High resolution CT yields more complete information, yet not substantially different

• Integral information is as valuable as local one

Conclusions

- DPF (complex) materials need high resolution techniques at multi-scale levels
  - One technique (or even a few techniques) is not enough
  - Do not forget the MACRO scale
- Neutrons (and Synchrotrons) are very powerful tools, yet
  - ONLY in combination with others
  - *In-situ* is a MUST
- Modeling is necessary to capitalize experimental data

Questions = Interest