

Charakterisierung von Cordierite mit unterschiedlichen Methoden (X-ray, Neutronen)

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Kurzfassung

Poröse Keramiken haben ein breites Anwendungsgebiet als Filtermaterialien und Wärmeisolierungen, wozu sie vor allem wegen ihrer hervorragenden Abschreckbeständigkeit eingesetzt werden. Ihre Eignung beruht auf einer komplizierten Mikrostruktur, die zu anisotropen thermischen und mechanischen Eigenschaften, oft aber auch zu Mikrorissen führt.

Da die besonderen mikrostrukturellen Merkmale dieser Materialien auf verschiedenen Längenskalen angesiedelt sind, ist es schwierig, sie in einem universellen Modell zu erfassen. Um jedoch die Materialeigenschaften vorherzusagen, werden mikromechanische Modelle benutzt, die als Parameter Informationen über die Mikrorissdichte, die Anisotropie der thermischen Ausdehnung, der kristallographischen Textur sowie des mechanischen Verhaltens erfordern.

Am Beispiel von Wabenstrukturen aus Cordierit, die als Dieselpartikelfilter eingesetzt wird, zeigen wir, dass zu einer möglichst umfassenden und zerstörungsfreien Charakterisierung der genannten Größen der Einsatz von Röntgen- und Neutronentechniken unabdingbar ist. Es wird ausführlich diskutiert, wie mittels Röntgenrefraktion die Mikrorissdichte, mittels Röntgen-Computertomographie die 3D-Mikrostruktur, mittels Neutronen-Texturanalyse die Kristallitorientierung und mittels Neutronenbeugung das mikromechanische Verhalten bestimmt werden. Wir demonstrieren, dass die Kombination aus diesen Messergebnissen gemeinsam mit dem Einsatz eines analytischen Modells tiefere Einblicke in das Verhalten dieser Materialien gestatten.

Characterization of Cordierite DPFs by means of Synchrotron, Lab, and Neutron Techniques

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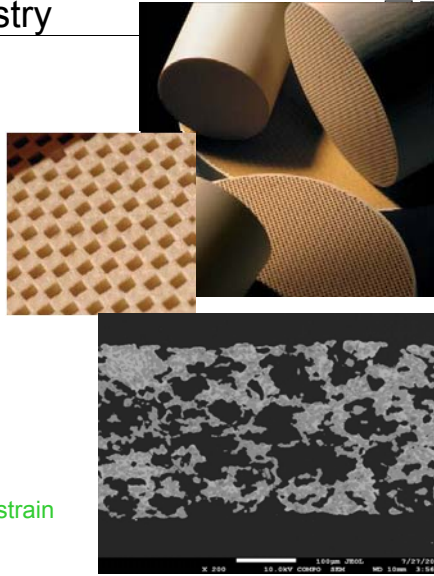
Lujan Center, LANL, Los Alamos, NM, USA

Outline

- Premise: Porous Ceramics and Applications
 - Rationale and Strategy
 - Microstructures
 - ...and models
 - Comparisons
 - Conclusions/ Lessons learnt
-

Porous Ceramics in Industry

- **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)

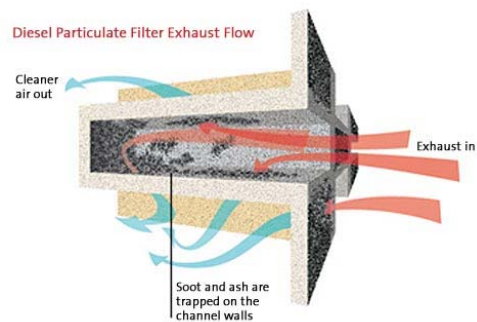
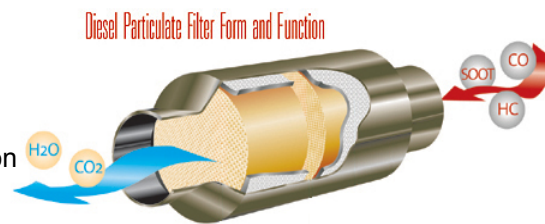
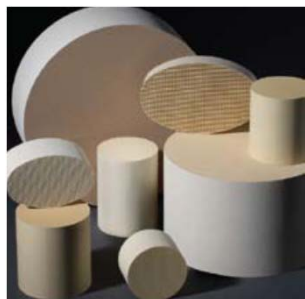


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Porous Ceramic Filters

Main attributes

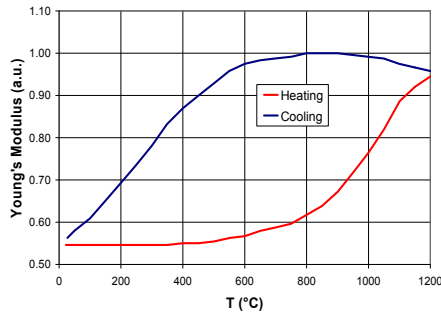
- Filtration efficiency
- Pressure drop
- Resistance to crack initiation
- Operation at high temperatures



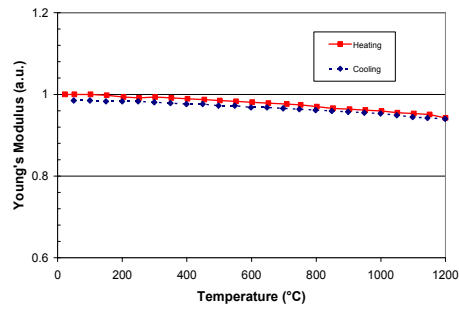
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Microcracking often occurs because of lattice thermal expansion anisotropy
NB: low crystal symmetry ceramics

Young's Modulus



Cordierite



Silicon Carbide

Hysteresis of both the dilation and the Young's modulus vs Temperature

Introduction

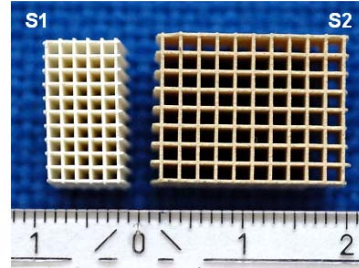
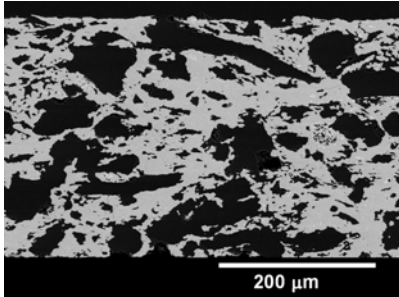
Rationale

- Macroscopic parameters do not fully explain the material and the component performance
- Microscopic features govern materials properties
- Link microscopic properties to macroscopic behavior/ material performance of porous (microcracked) ceramics

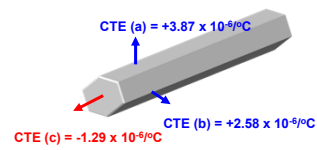
Strategy

- Problem solving approach (Fitting the method to the problem)
- Combination of Experiments and Calculations

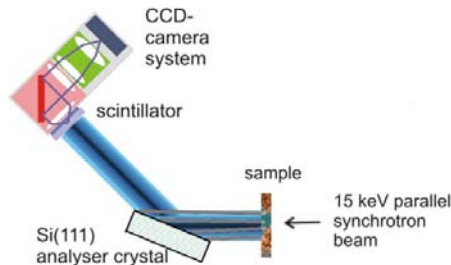
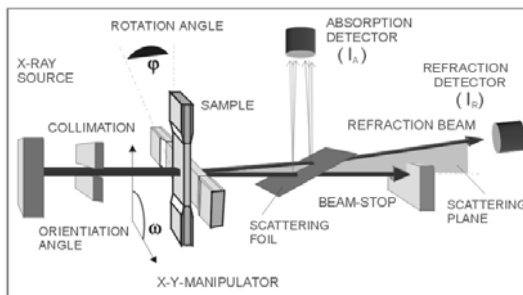
What?



Sample	Porosity (%)	Median Pore Diameter /μm	CTE (RT-1273K) 10 ⁻⁶ K ⁻¹
S1	62	10	1.15
S2	50	18	0.57



How? X-ray Refraction

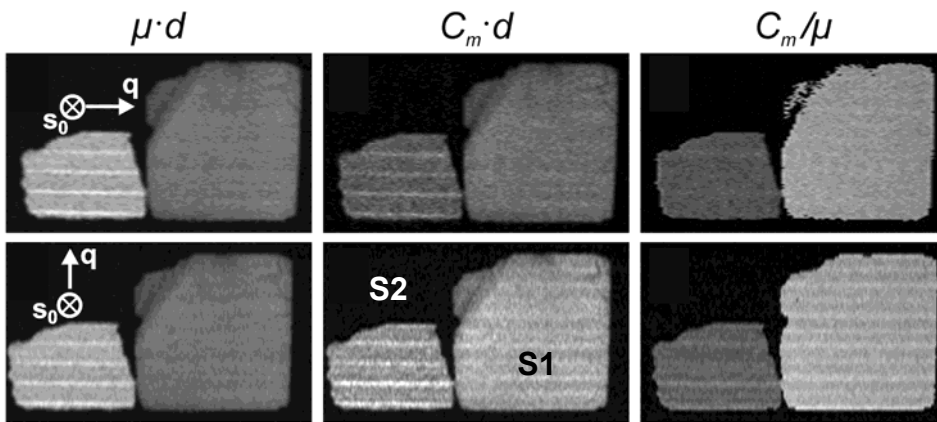


- Simultaneous measurement of absorption and refraction
- Sensitivity to specific surface ($C_m \propto \Sigma$):

$$C_m \cdot d = \frac{I_R / I_{R0}}{I_A / I_{A0}} - 1$$

- Fine step scanning or 2D mapping (lab or Synchrotron)
- Possible tomography by 360° sample rotation
- Coarse spatial resolution, but we do not need it !

Results- X-Ray Refraction



C_m/μ (\perp //)	S1	S2	C_m/μ	// extrusion	\perp extrusion
Laboratory	1.10	1.16	S1	2.02	2.2
Synchrotron	1.06	1.15	S2	1.10	1.28
			S1/S2	1.75	1.75

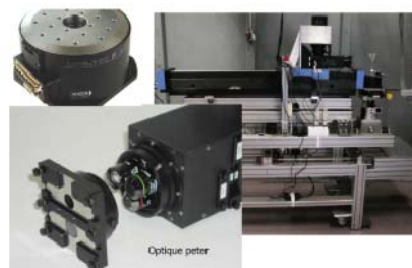
More C_m in \perp = surface oriented // to the extrusion axis

How? X-ray Computed Tomography

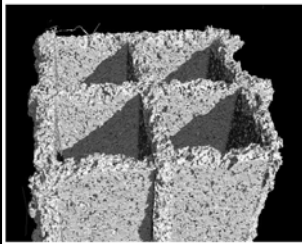


GE v|tome|x
Pixel size $\sim 4 \mu\text{m}$

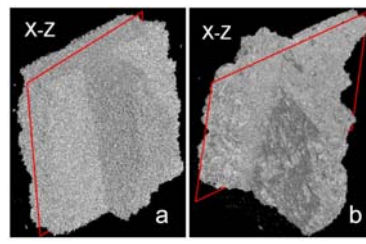
BAMLine, BESSY,
Berlin, Germany
Pixel size $\sim 0.4 \mu\text{m}$



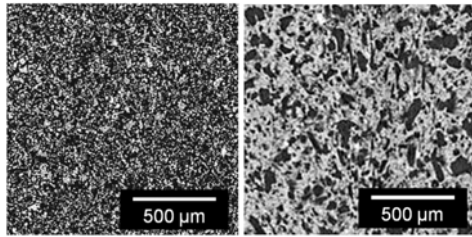
Results- CT



Laboratory CT Data

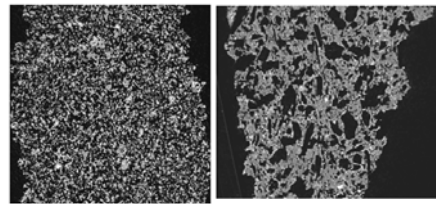


Synchrotron CT Data



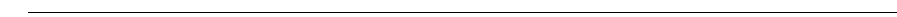
S1

S2



S1

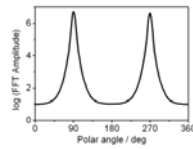
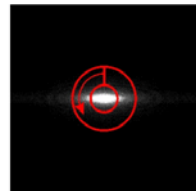
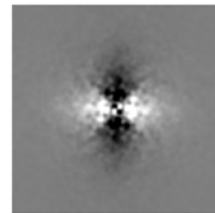
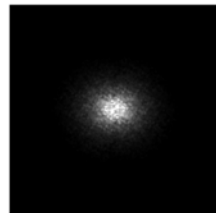
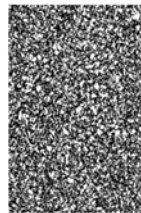
S2



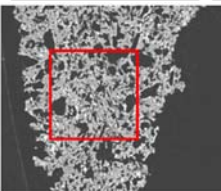
CT Data Analysis



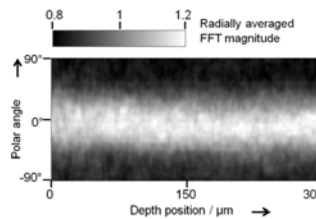
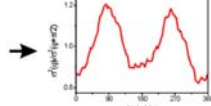
FFT Analysis:
The Orientation of objects is visible



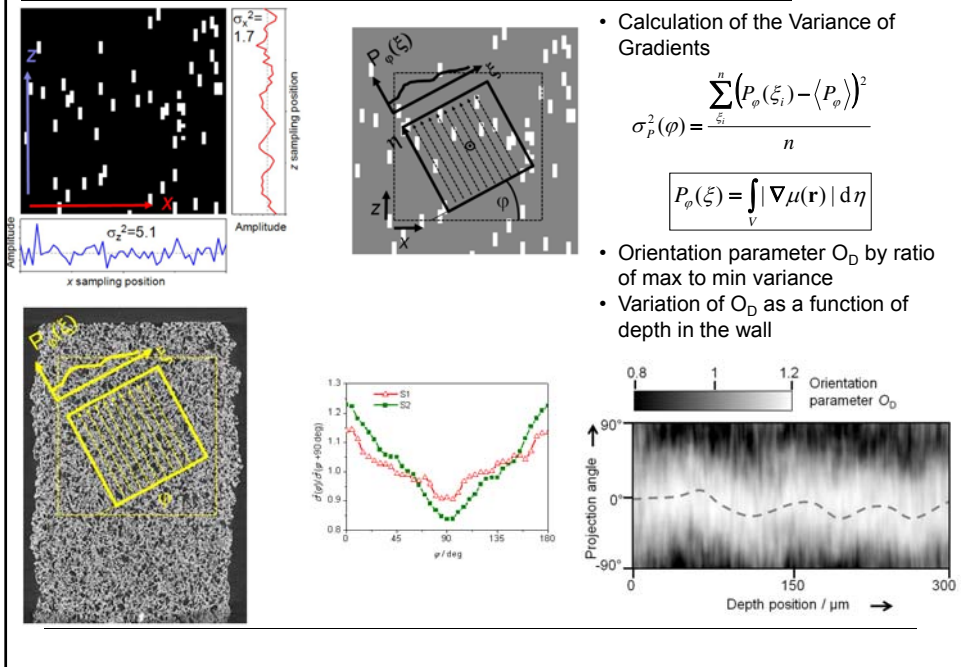
- FFT Periodograms:**
- Radial average of intensity
 - Orientation factor up to 1.2
 - No variation in wall depth



FFT



CT Data Analysis: DIVA



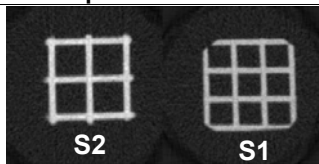
- Calculation of the Variance of Gradients

$$\sigma_p^2(\varphi) = \frac{\sum_{\xi} (P_{\varphi}(\xi_i) - \langle P_{\varphi} \rangle)^2}{n}$$

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

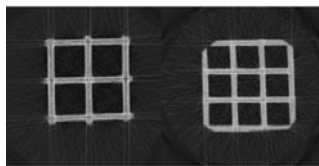
- Orientation parameter O_D by ratio of max to min variance
- Variation of O_D as a function of depth in the wall

Comparisons



Refraction

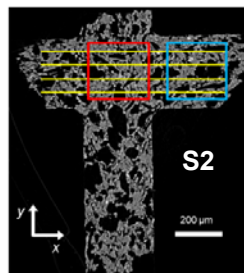
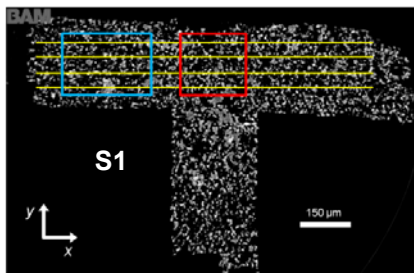
Different signal from inside the crosses than from the walls



Synchrotron CT

Quantification of the scattering power difference by means of DIVA

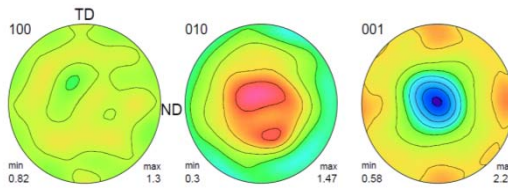
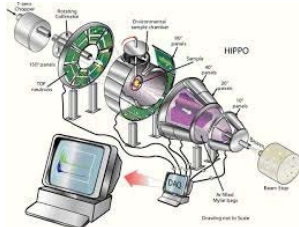
	walls	crosses	crosses / walls ratio
S1	0.85	0.77	0.91
S2	0.74	0.61	0.83



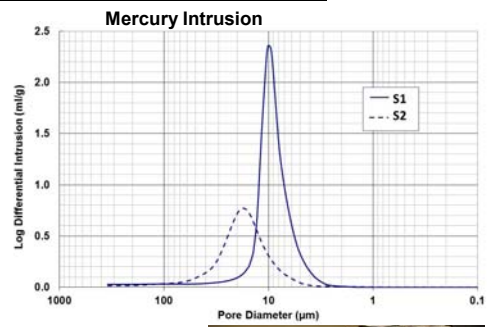
Comparisons



	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



Neutron Diffraction Texture Analysis



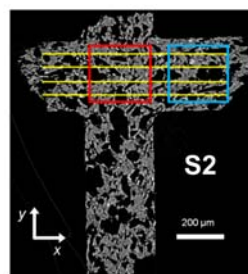
Comparisons



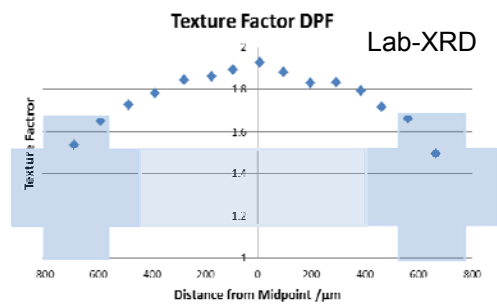
S1/S2	extrusion	⊥ extrusion
d_{50}	1/1.8	1/1.8
C_m/μ	1.75	1.75

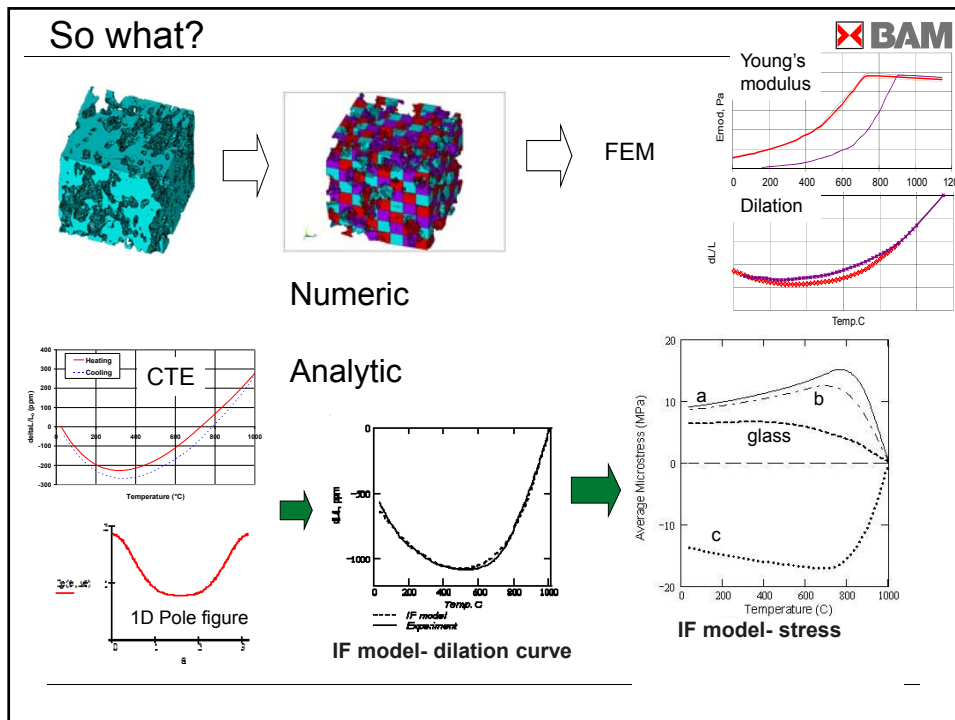


$$1/d \propto \Sigma \propto C_m/\mu$$



crosses / walls	XRD	CT
S1	-	0.91
S2	0.72	0.83





- ### Conclusions
- Complex materials (e.g., Porous Ceramics) need high resolution techniques at multi-scale levels
 - Microstructure is intimately linked to properties and performance
 - One technique (or even a few techniques) is not enough
 - Non-destructive Characterization and Quantitative Image Analysis Techniques (2D and 3D) are necessary
 - X-Ray refraction and CT are very powerful tools
 - Models need to be used to draw conclusions
- Questions = Interest