

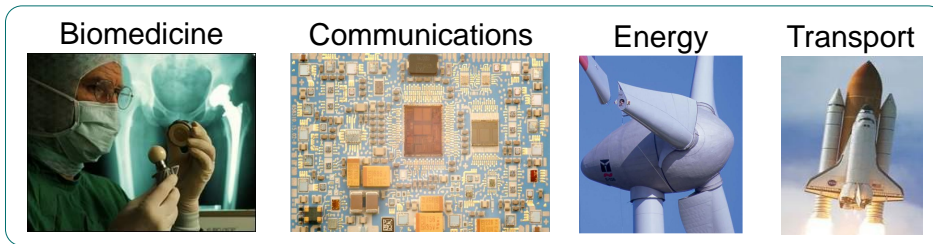
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Design and processing of ceramic materials

Bio-inspired layered ceramics with tailored microstructures

□ Ceramics for use in **structural** and **functional** systems



Example of failure

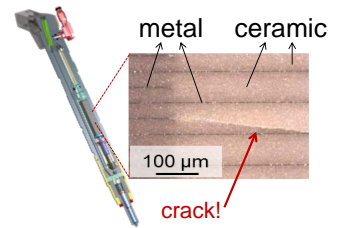


Fig. 1: Fuel injector for common rail systems

- **Problem:** Brittleness of ceramic parts – **unpredictable fracture**
- **Need** for tough and mechanically reliable ceramics

□ Vision → Build ceramics in a "**bio-inspired**" layered architecture

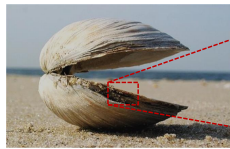


Fig. 2: Natural seashell



Fig. 3: Step-wise fracture of layered nacre

...from nature to the lab...

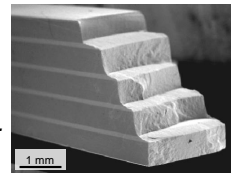


Fig. 4: Fracture of a layered ceramic

□ Design and characterization of layered ceramic composites

Tailored embedded "compressive stresses"

✓ Crack arrest (*high toughness*)
✓ Flaw tolerance (*high reliability*)

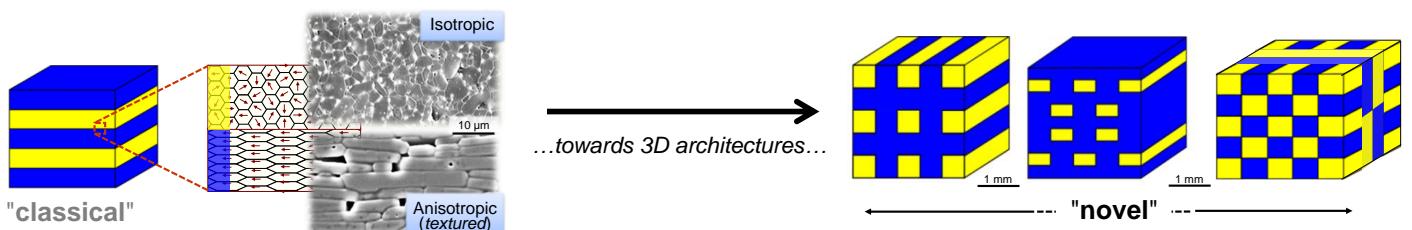
Complex distribution of "protective" layers

Novel "textured" microstructure

Aim: strong and tough ceramics

It is "hard" to be "tough"...
...it is "tough" to be "strong"

□ Novel processing of microstructures and architectures with enhanced properties



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Financial support:



References:

Open Ceram. 15 (2023)

J. Eur. Ceram. Soc. 37 (2017)

Design and processing of ceramic materials

Low temperature fabrication of ceramics by the cold sintering process

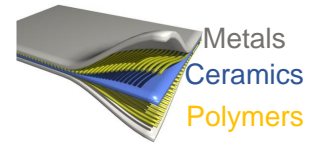
❑ **Problem statement:** Sintering of ceramics requires high temperatures $> 1000^{\circ}\text{C}$

➖ **High energy consumption & Carbon emissions**



➖ **Limited materials design and integration**

- Thermal stresses
- Decomposition/reaction between different phases

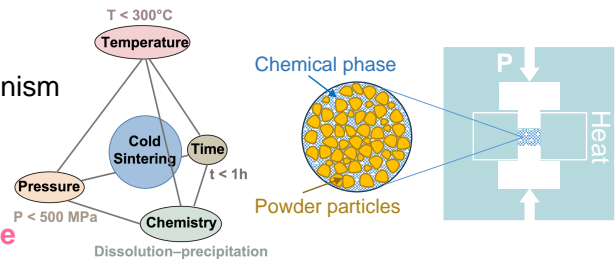


➔ *Need for alternative low-temperature sintering techniques*

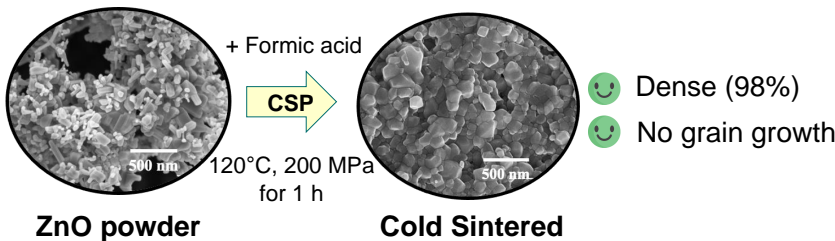
❑ **The Cold Sintering Process:** innovative technique that enables densification of ceramics at unprecedented low temperatures $< 300^{\circ}\text{C}$

The principle: Geologically inspired pressure-solution creep mechanism

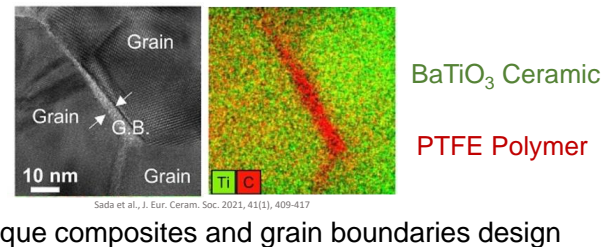
- 1) **Dissolution:** activated by the transient phase (**Chemistry**)
- 2) **Diffusion:** by chemical potential gradients induced by **Pressure**
- 3) **Precipitation:** due to liquid phase evaporation at low **Temperature**



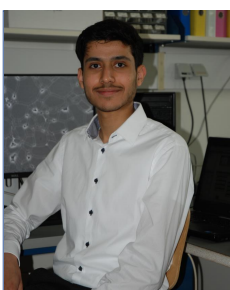
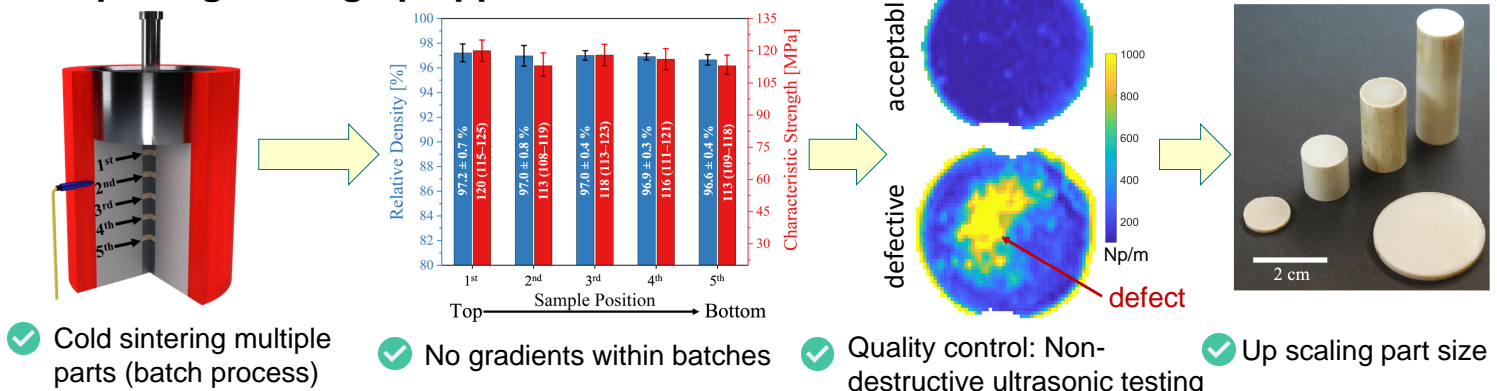
❑ **Densification by cold sintering**



❑ **Integration of ceramics/polymers/metals**



❑ **Exploring scaling up opportunities**



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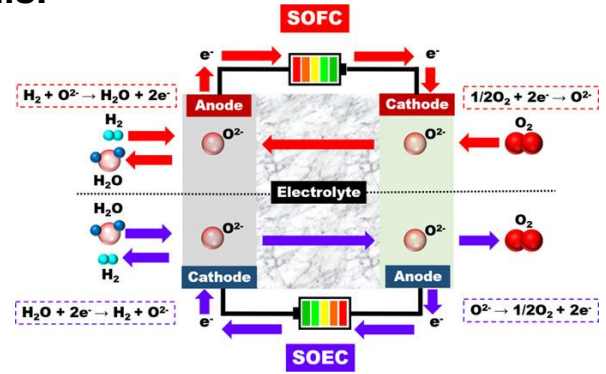
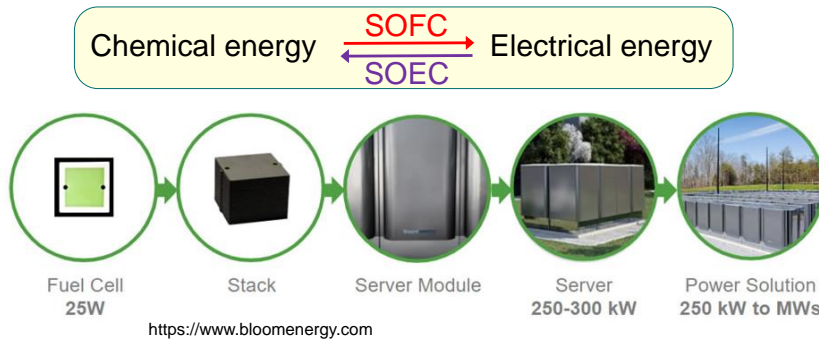
References:

- J. Eur. Ceram. Soc. 43 (2023)
- Ceram. Forum Int. 4 (2023)

Design and processing of ceramic materials

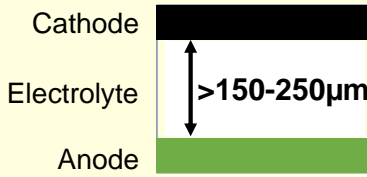
Fabrication of ceramic electrolytes for SOFC/SOEC through tape casting

Principle of Solid Oxide Fuel (Electrolyzer) Cells:



Problem definition

Electrolyte-supported SOFC/SOEC



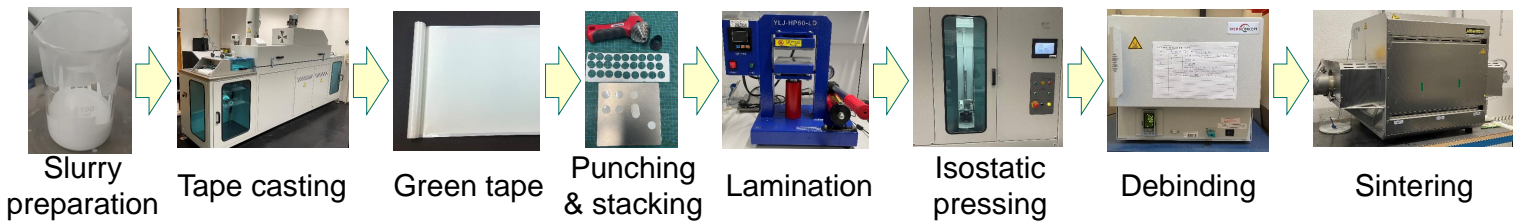
Challenges

- Higher ohmic resistance of thick electrolytes
 → “low cell performances”
- Thinner electrolytes
 → “less mechanical integrity”

Vision

Design and fabrication of **thin electrolytes with enhanced mechanical integrity and sufficient ionic conductivity** through tape casting

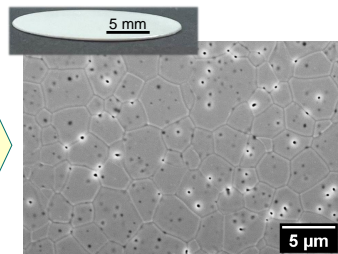
Processing of cubic zirconia (8YSZ) ceramic electrolyte through tape casting



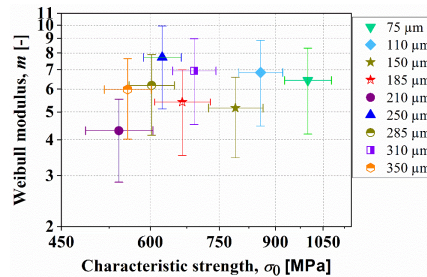
Results

Effect of **thickness** on the microstructure, strength and ionic conductivity of tape casted 8YSZ electrolytes

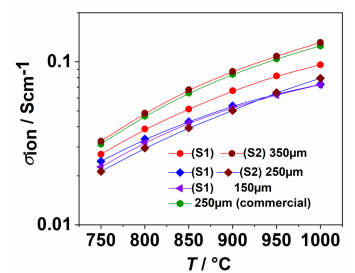
Microstructure



Mechanical strength



Ionic conductivity



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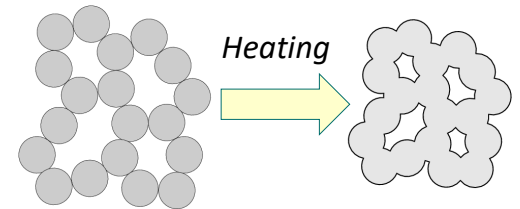
References:

Fuel Cells 20, 2020, No. 6, 644–649

Design and processing of ceramic materials

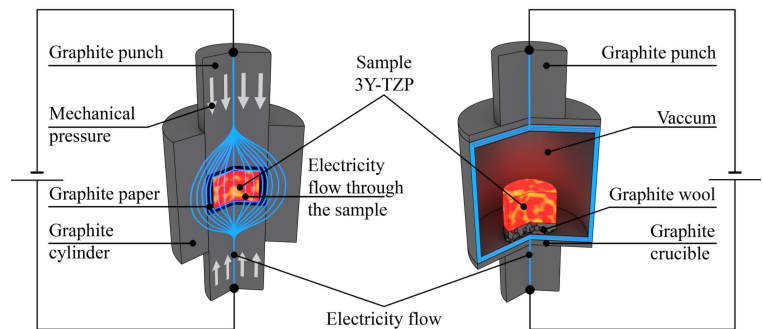
Rapid sintering by Spark Plasma Sintering

□ **Sintering** of **ceramic** powder is a process used in ceramic manufacturing to create solid ceramic materials from fine powders. It involves **heating** the **powder compact** at high temperatures, typically below the melting point of the primary material, but high enough to cause **atomic diffusion** and **densification** of the powder particles.

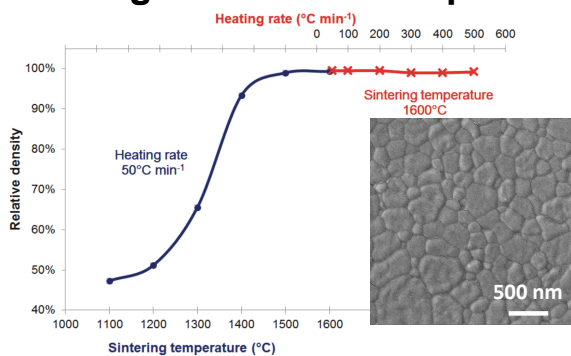


□ Spark Plasma Sintering technique

- Mechanical Pressure (30 – 100 MPa)
- Fast Heating/Cooling (100 – 1000 °C/min)
- Short dwell time (1 – 30 min)
- Direct Electric Heating (conductive samples)
- DC Electric Pulses/AC source



□ Sintering of 3Y-TZP nano powder

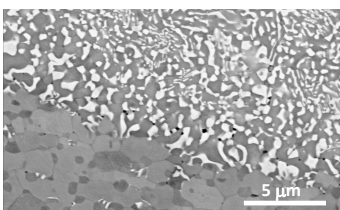


Densification of tetragonal zirconia stabilized with 3 mol% Y_2O_3 (3Y-TZP) by SPS.

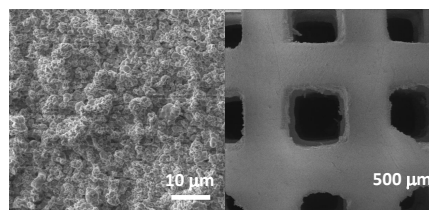
SPS conditions temperature/dwell time/pressure	% of theoretical density	Grain size (nm)	Standard deviation of grain size (nm)
1100°C/2 min/50 MPa	98.3	60	16
1200°C/2 min/50 MPa	99.6	126	49
1300°C/2 min/50 MPa	100.0	190	73
1400°C/2 min/50 MPa	100.0	264	111
1500°C/2 min/50 MPa	99.8	379	142
1600°C/2 min/50 MPa	100.0	642	226

□ Applications of SPS and rapid sintering

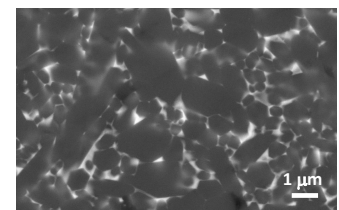
Complex high entropy carbides
prepared at 1700 °C/5 min/50 MPa



Pressure-less sintering of 3D printed
Hydroxyapatite scaffold



α -SiAlON prepared at
1750°C/3 min/50 MPa



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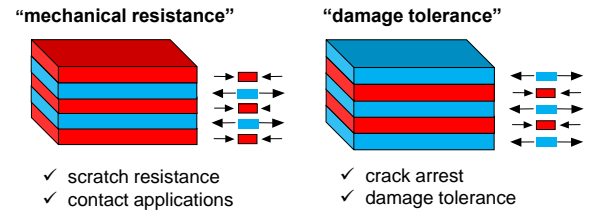


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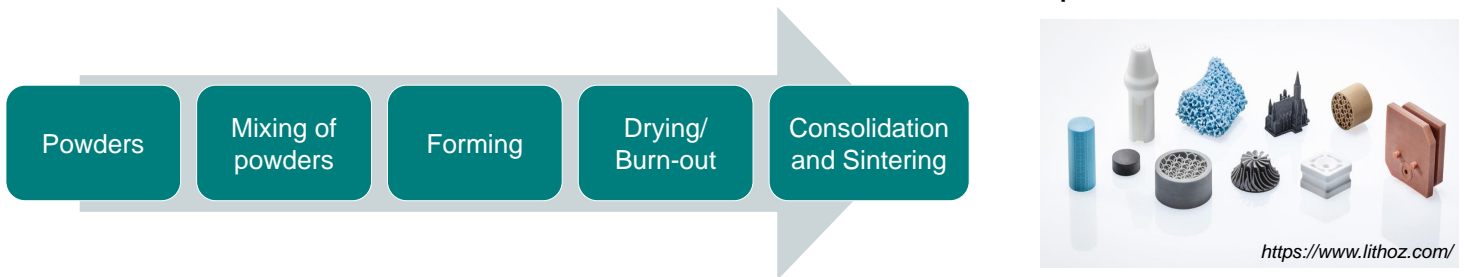
Design and processing of ceramic materials

Sintering of 3D additive manufactured ceramic multi-materials

❑ **Ceramic multi-materials** may enhance mechanical properties such as improved strength and/or damage tolerance through tailored residual stresses and architecture.



❑ **Additive manufacturing (AM)** is a way of forming ceramics with complex geometry. The combination of different materials in XY- and Z-direction is possible.



❑ **Sintering** is one of the most important steps in the processing of ceramics, whereby inorganic powders densify forming a microstructure, giving the material its properties.

Sintering within minutes
(instead of hours)

Setup for rapid sintering
A.-K. Hofer et al., Addit. Manuf. 59 (2022) 103141.

Rapid sintering vs. conventional sintering

2 μm 2 μm

With rapid sintering, fine grained microstructures can be realized.

Ceramics have **high hardness and strength**, but **brittle behavior**.

The goal is to increase the **damage tolerance** by using **multi-materials** with tailored microstructure.

❑ **Using different interface designs**, unique material combinations can be realized.

The **goal** of this work is to understand the **sintering of additive manufactured ceramic multi-materials** and to enable material combinations in complex shapes with specific mechanical properties.

Examples of multi-material designs

Sharp interface Interphase between layers Functional gradient

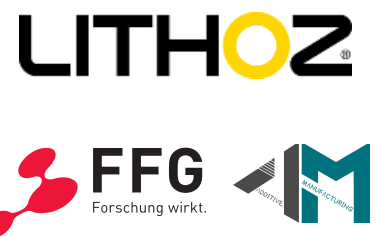


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Design and processing of ceramic materials

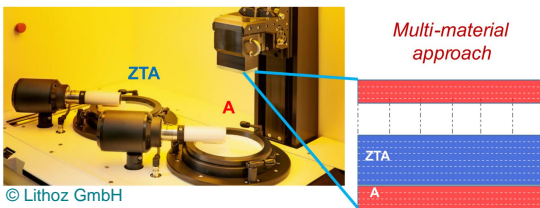
3D-printed multi-material components with improved mechanical properties

□ Motivation

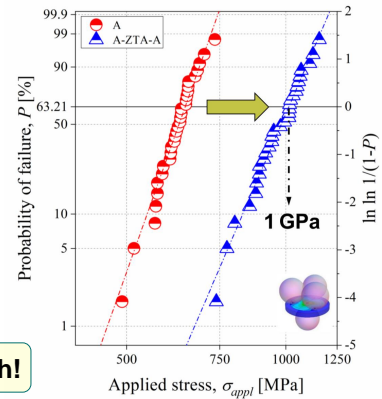
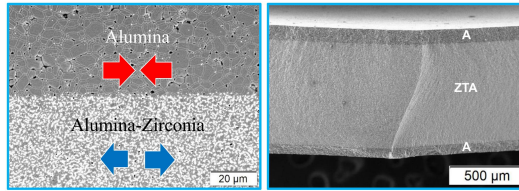
- **Additive manufacturing** may open new pathways for designing complex-shaped ceramic parts and components
- **Goal:** 3D-printed alumina-based ceramic components with superior mechanical properties
- **Strategy:** Multi-material design concepts for further enhancing the strength and the damage tolerance

□ Design concept – Mechanical resistance

Lithography-based Ceramic Manufacturing

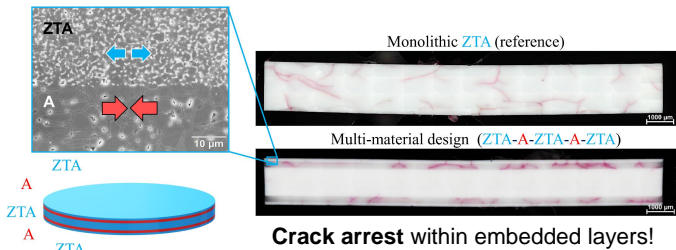


Mechanical characterization

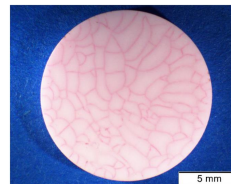


Surface compressive residual stresses (red arrows) for enhancing the mechanical strength!

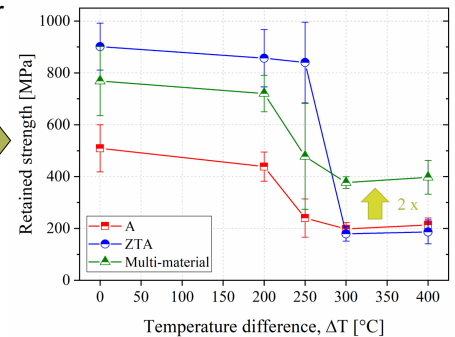
□ Design concept – Damage tolerance



Thermal shock behaviour



Enhanced thermal shock strength

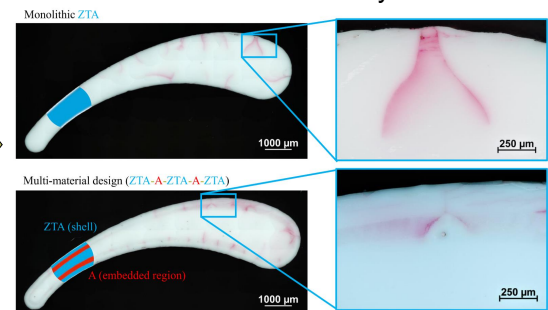
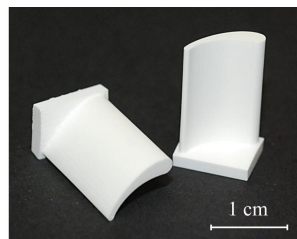
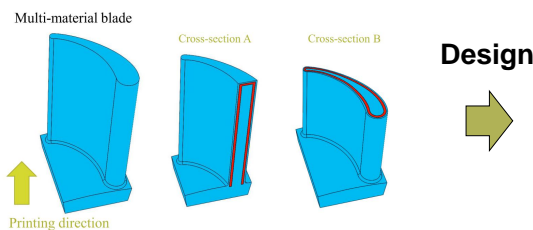


Embedding internal (protective) layers under compressive residual stresses (red arrows) for enhancing the damage tolerance!

□ Demonstrator component

3D-Printing

Thermal shock tests and analysis of cracks



Feasibility of 3D-printing components with embedded protective layers has been proven, which may open new application fields!



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References:

J. Eur. Ceram. Soc. 44 (2024)
Open Ceram. 5 (2021)

Characterization of ceramic materials

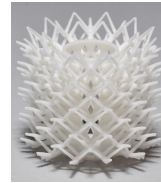
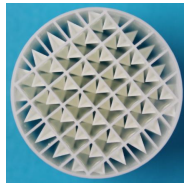
Strength testing of additively manufactured structures

□ Stereolithographic manufacturing opens new possibilities

Static mixer



Catalytic support structures

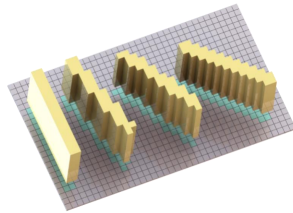


Heat exchangers

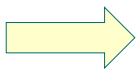


□ Challenges → Orientation-dependent surface structures

Aliasing effect of DLP-module



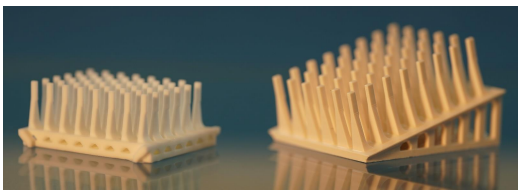
Rough surface due to layered manufacturing



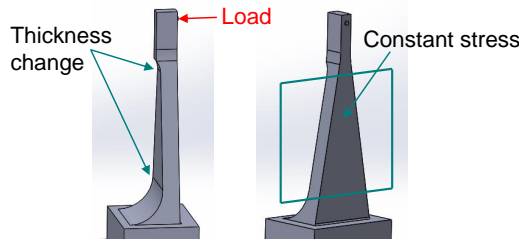
- **Problem:** Surface quality depends on surface orientation in relation to the building direction
- **Need for new testing methods** to reliably assess influence of surface orientation

□ Novel test specimen

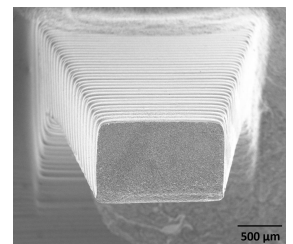
48 bending bars on a baseplate, different configurations (upright, inclined)



Region of constant stress on bending bars

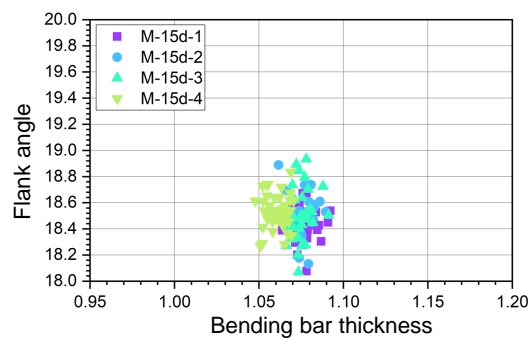
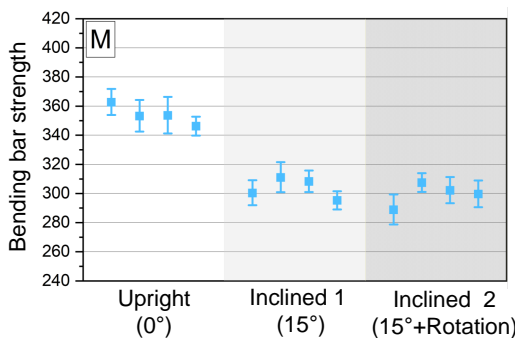


Fractured specimen with surface structures



□ Results

Strength depends significantly on surface orientation



Exceptional dimensional reproducibility



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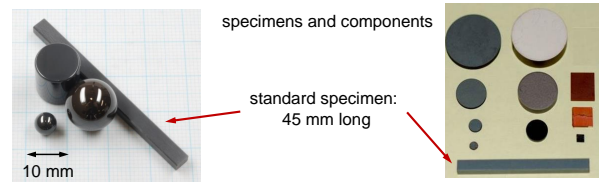
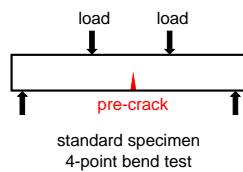


Characterization of ceramic materials

Fracture toughness tests for components

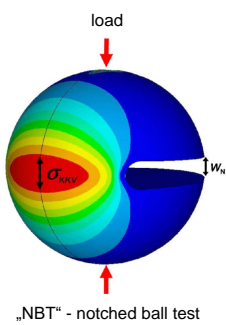
- Fracture Toughness is a material property that tells us how difficult it is to propagate an existing crack in a component. It is measured by breaking specimens with **artificial pre-cracks**.

- Problem:** Many parts are too small to make standard specimens.

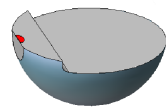


- Solution:** New tests which use the parts directly have to be developed. The Finite-Element-Method has to be used to evaluate results.

Bearing balls – NBT-KIc

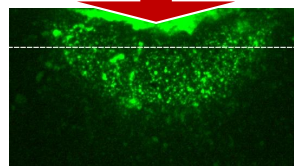


„NBT“ - notched ball test

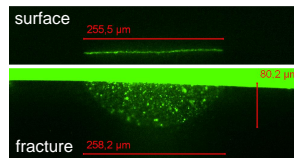


NBT-test: ball with artificial pre-crack

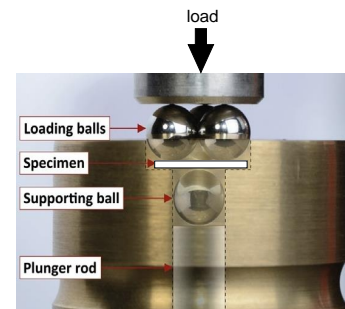
Precracking with Knoop indenter



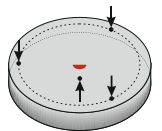
pre-crack



Discs and Plates – B3B-KIc

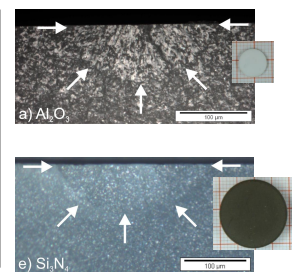
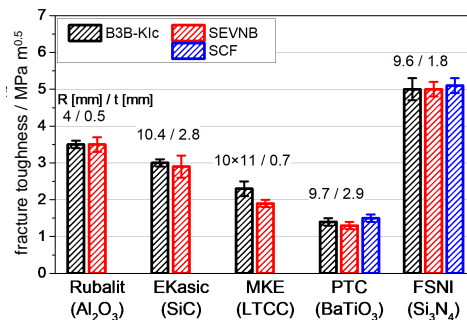
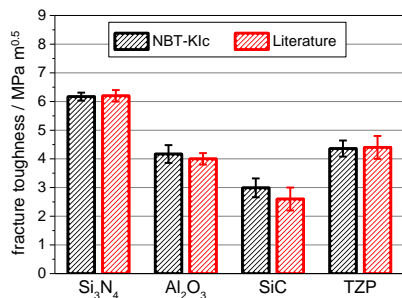
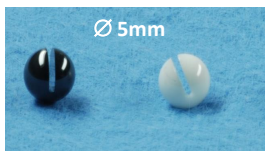


„B3B“ - ball-on-3-balls test



B3B test: disc with artificial pre-crack

- Verification:** Correct values can be measured with new methods.



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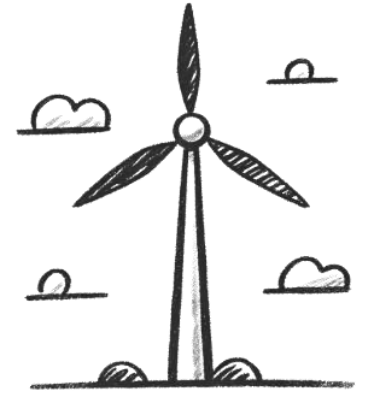
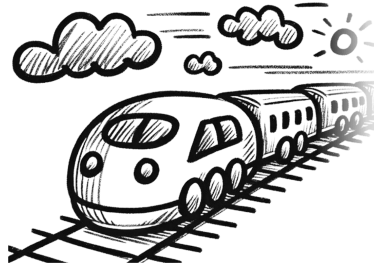
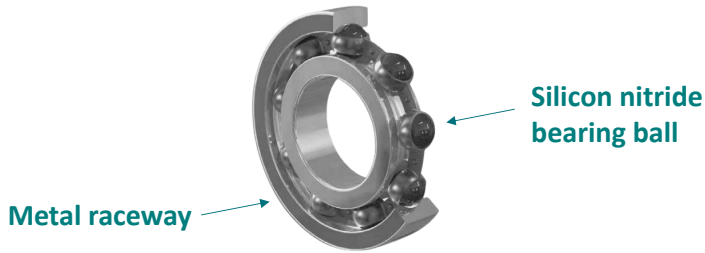
References:

S. Strobl, T. Lube, O. Schöpl: *JECs* 34 (2014), 1881-1892
S. Strobl, P. Supancic, T. Lube, R. Danzer: *JECs* 32 (2012), 1163-1173
T. Lube, S. Rasche, T.G.T. Nindhia: *JaCerS* 99 [1] (2016), 249-256

Characterization of ceramic materials

Testing of bearing balls

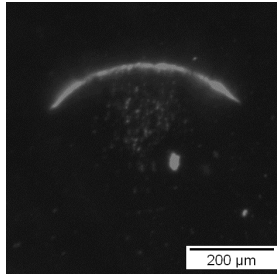
Application of **hybrid bearings**



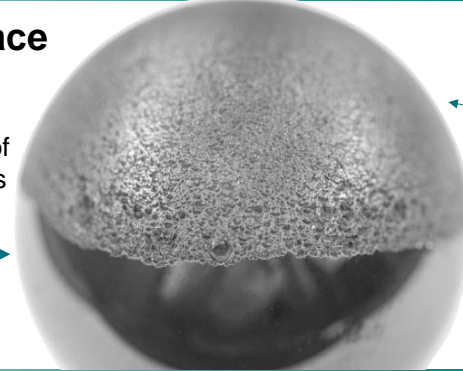
Fracture of the balls originates at the **surface**

Initial contact crack

Cause:
blunt & elastic contact of bearing balls bumping against each other



Further loading of the mounted balls leads to



Spalled surface

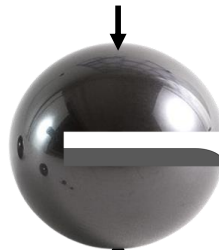
Intact surface

Surface sensitive testing



Insight on plastic behaviour & surface stresses

Vickers hardness & Toughness



Insight on surface defects

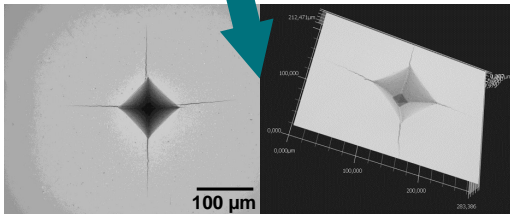
Strength



Insight on elastic behaviour

Critical load

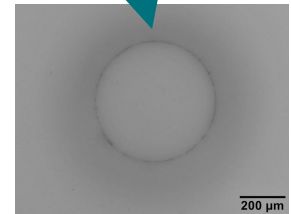
Fractographic analysis



Determination of wear resistance



Determination of defect size



Determination of contact damage



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Characterization of ceramic materials

Fractography and Failure Analysis

□ Fractography: Identification of fracture origins

Fracture features (stress field at the moment of failure, crack branching patterns, hackle lines,...)

Background (processing, microstructure, service conditions,.....)

Fracture origin

Theoretical approach:

$$K \geq K_{IC}$$

$$\sigma = \frac{M}{\sqrt{R}}$$

$$K_{Ic} = \sigma Y \sqrt{\pi a}$$

$$a_c = \frac{1}{\pi} \left(\frac{K_{Ic}}{\sigma Y} \right)^2$$

Practical use:

1. Locating the fracture origin: stereo microscope

2. Precise identification: SEM-EDX

EDX: Si, Pores, EDX: Fe, Cr, Ni

Most used methods: microscopy (magnifying lens, stereo microscope, SEM, EDX,...), Thermography, RAMAN-spectroscopy, Fracture mechanics, FE-analysis,...

□ Contact cracks in Si₃N₄-rollers for rolling of superalloy wires

Sketch of a pair of ceramic rollers during application

Failure during application

track **cracks**

FE-Analysis

Cross section (Hertzian cracks caused by contact damage leads to failure)

Roller track

□ Failure analysis on a ZrO₂-ball bearing raceway used in a mixer

Side A **deposits** **Free from debris** **Wear debris**

Side B **Wear debris** **Free from debris**

Assumption: Unbalanced bearing ⇒ stress induced phase transformation ⇒ flaking off of surface layers

Proof: RAMAN-spectroscopy (inner race): phase transformation from tetragonal to monoclinic phase

Volume change leads to failure

ZrO₂-particle

Side A

Raman Intensity

Wavenumber [cm⁻¹]

□ Failure analysis of electroceramic components by HALT test

Thermography: top side

Thermography: cross-section

Light microscope: cross-section

FIB Analysis of hot spot: micro crack filled with Ag

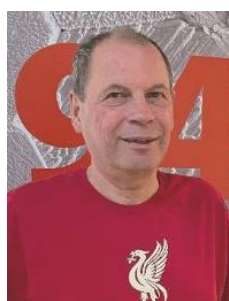
hot spot

hot spot

hot spot



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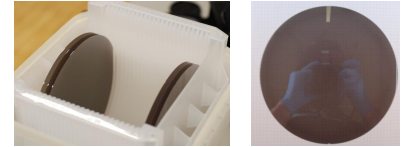
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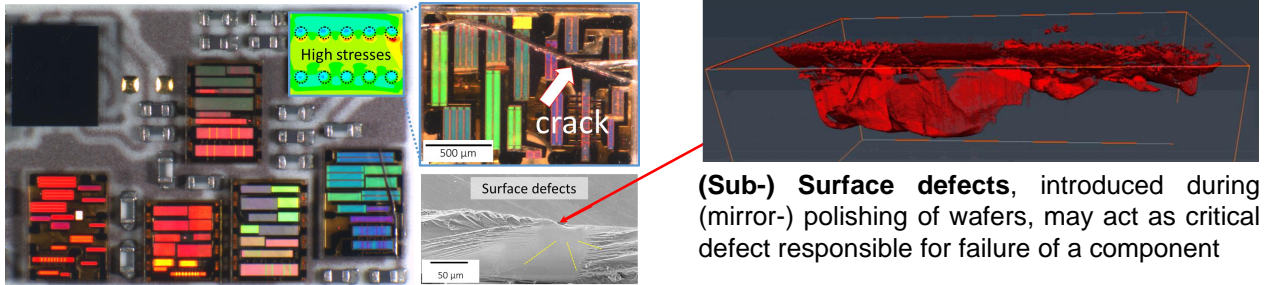
Characterization of ceramic materials

Single crystalline materials for microelectronic applications

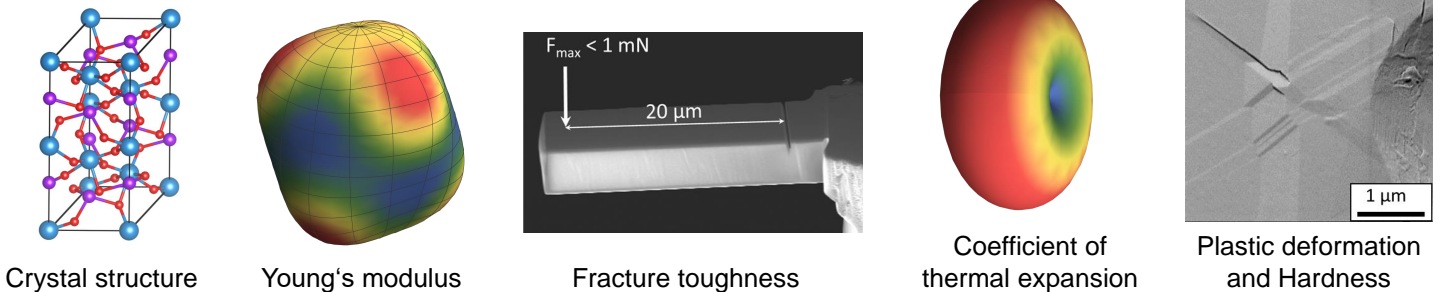
□ **Single crystals** are widely employed in **microelectronics** in form of wafers, which act as the basis for manufacturing of semiconductor, optoelectronic or radio frequency devices



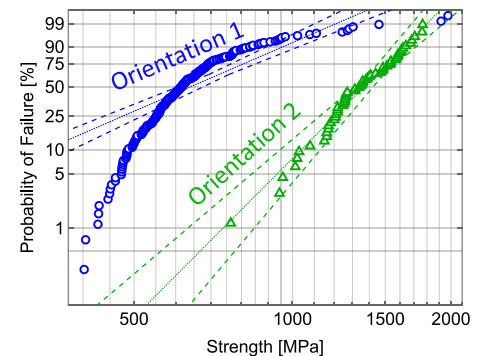
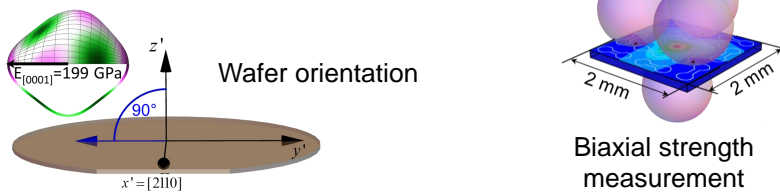
□ **Challenges** → The thereby used wide range of material classes with **distinct thermo-mechanical properties** may lead to **cracks** in those often **brittle** substrates.



□ **Requirements** → Deep understanding of crystal **orientation dependent** contributions to failure of single crystalline components:



□ **Goal** → Wafer orientations and surface conditioning for **enhanced mechanical reliability** while maintaining functionality



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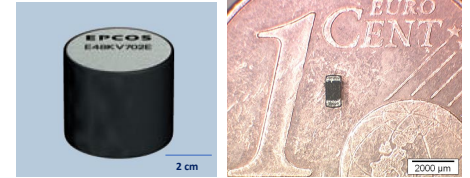
References:

Materials & Design 213 (2022)
Acta Materialia 150 (2018), 373 – 80
Materials & Design 153 (2018), 221-31
J. Am. Ceram. Soc. 101 (2018), 5705-16
J. Eur. Ceram. Soc. 37 (2017), 4397 – 406

Characterization of ceramic materials

Microscale investigations of grain boundaries in varistor ceramics

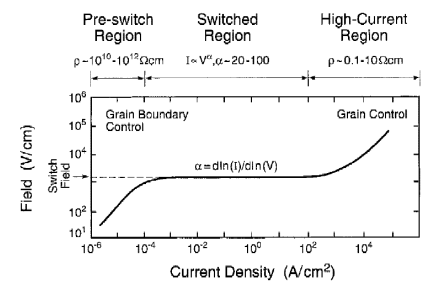
□ **Varistor components** are used as overvoltage protection elements for almost all electric and electronic circuits. Their exceptional electrical resistance characteristics is based on Double-Schottky barriers at grain boundaries in doped zinc-oxide ceramics.



Typical commercial varistor components for high and low voltage applications

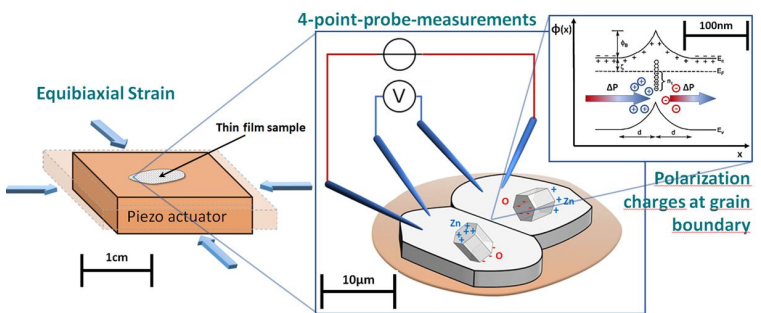
□ **Challenges:** Tuning the macroscopic current-voltage characteristics and increase the ampacity (i.e. the ability to withstand strong current pulses).

□ **Scientific goal:** microscopic characterisation at grain boundary level and modelling the relationship to macroscopic properties to get a deeper understanding of the functional microstructure-property relationship.



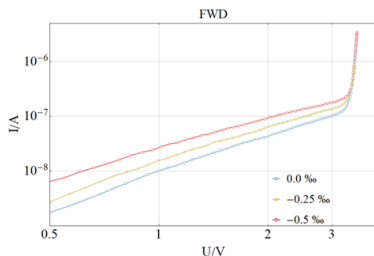
Typical current-voltage characteristics of varistor ceramics

□ **Key experiment:** Micro-4-point probe measurement of single grain boundaries of a varistor sample mounted on a piezoelectric stage for strain control (piezotronic effect).

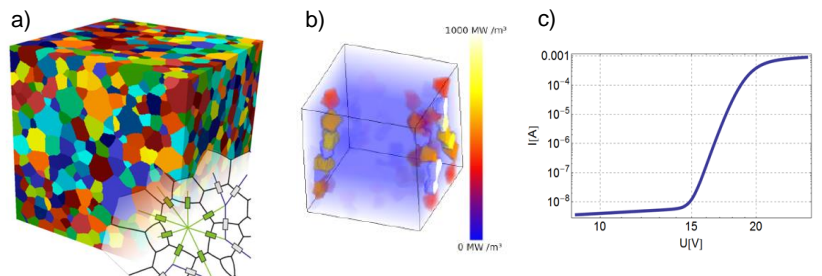


Measurement of the current-voltage characteristics of single varistor grain boundaries with respect to an applied mechanical stress.

Example for a strain dependent current-voltage characteristics of a single grain boundary



□ **Model approach:** Multiscale model, based on a realistic microstructure, measured current-voltage behaviour of single grain boundaries and micro-residual stress distribution (piezotronic effect), solving large non-linear network equations to get the macroscopic electrical characteristic of the material.



A modelled microstructure (a) is used to extract an equivalent network. Summing up local current paths (b) leads to the global current-voltage characteristics (c)



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References:

- T. Bilovits et al., Open Ceramics, 6:100125 (2021)
- M. Hofstätter et al., J. Eur. Ceram. Soc. 33 (2013)
- N. Raidl et al., Adv. Eng. Mater. 19 [4] (2017)
- M. Hofstätter et al., BHM, 158, 206-210 (2013)

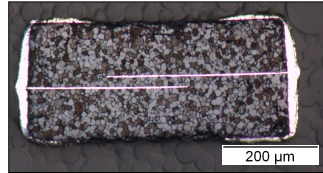
Characterization of ceramic materials

Lock-in Infrared Microscopy (LIIM)

Characterization of microelectronic ceramic components

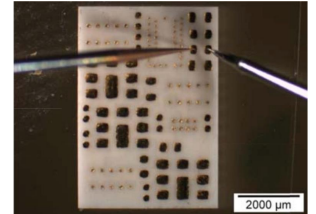
Multilayer varistor:

Zinc oxide-based (voltage dependent VARIABLE RESISTOR) used for surge and overvoltage protection of devices [1]



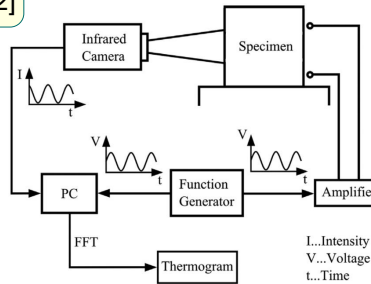
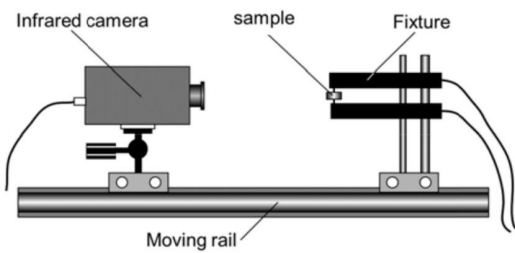
LTCC module:

Hybrid integrated circuit based on LTCC (low temperature co-fired ceramics) [2]



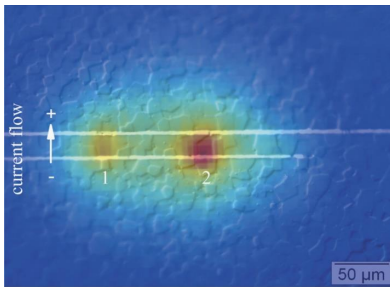
Method

Schematic of an IR camera mounted on a rail track in front of the sample [2]



A sinusoidal voltage is applied to the specimen. An infrared camera records the resulting periodic heating. The thermogram is evaluated by Fourier Transformation [1]

Thermograms

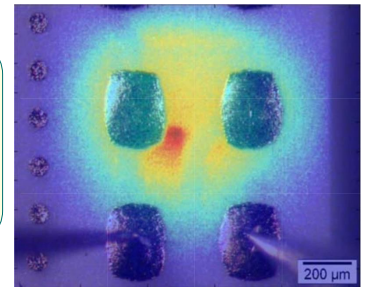


Varistor thermogram

Two distinct areas with current paths between electrodes are detected by the Lock-in system [3]

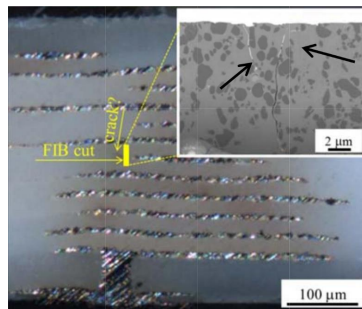
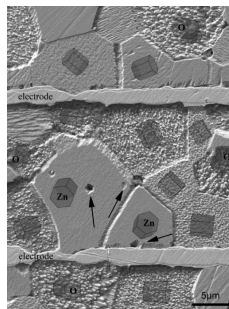
Hot spot in LTCC module

Heat generation caused by leakage current in the LTCC module is detected by the Lock-in system [2]



Results

Lock-in IR microscopy can be used as a tool for microscale current detection to reveal the device dominating junctions in electroceramic varistors [3]



After hot spot detection a crosssection of the sample reveals the source of error current: cracks partially filled with electrode material [2]

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References:

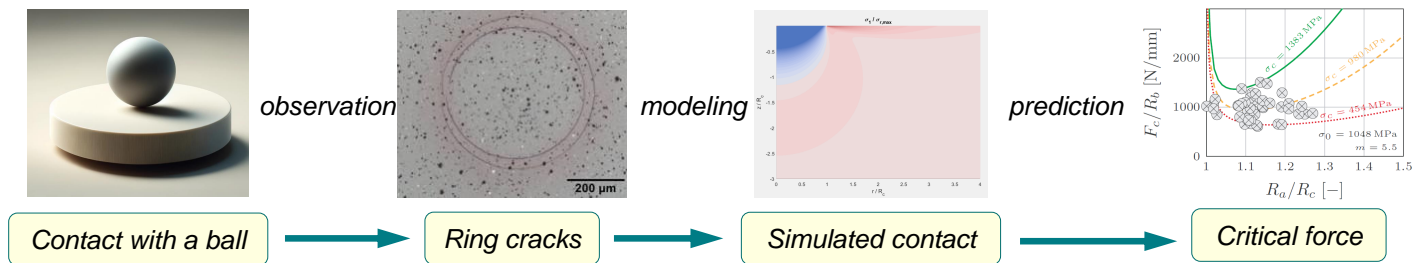
- [1] N.Raidl et. al. *Adv. Eng. Mater.* 19 [4] (2017)
- [2] S.Röhrig et. al. *J. Ceram. Sci. Tech.* 6 (2015)
- [3] M.Hofstätter et. al. *Microscopy and Microanalysis* 21 (2015)

Modelling of fracture in ceramics

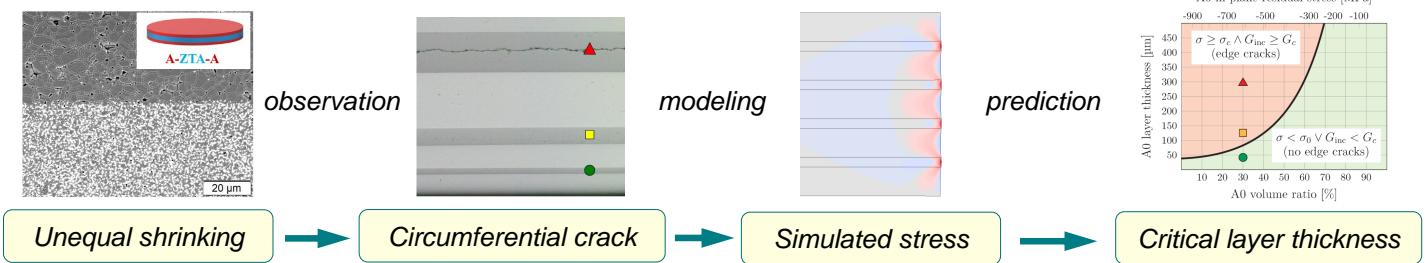
Predicting conditions that cause initiation of cracks in ceramics

- ❑ **Motivation:** observation of cracking 😞 → computational modelling 🖥️
→ prediction of critical conditions 💡 → crackless design ✅
- ❑ **Finite fracture mechanics** is an approach for modelling crack initiation:
 - ❑ **Stress** must overcome the tensile **strength** and simultaneously
 - ❑ **Energy** release rate must overcome the **fracture toughness**

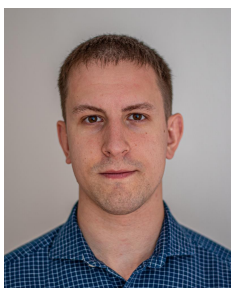
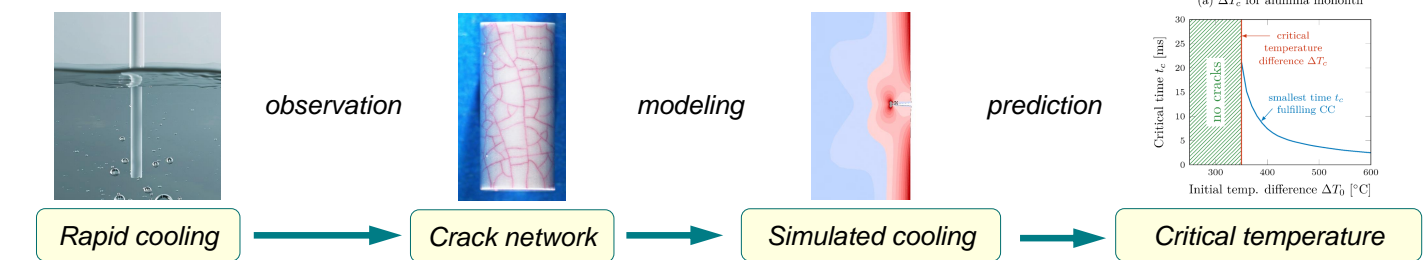
❑ Indentation cracking due to contact with hard objects



❑ Residual stress cracking due to an unequal material dilatation



❑ Thermal shock cracking due to a rapid temperature change



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Papšík, Ševeček,
Hofer, Králeva,
Kreith & Bermejo.
JES 43 (2023)

Papšík, Ševeček,
Martin & Bermejo.
JACS 106 (2023)

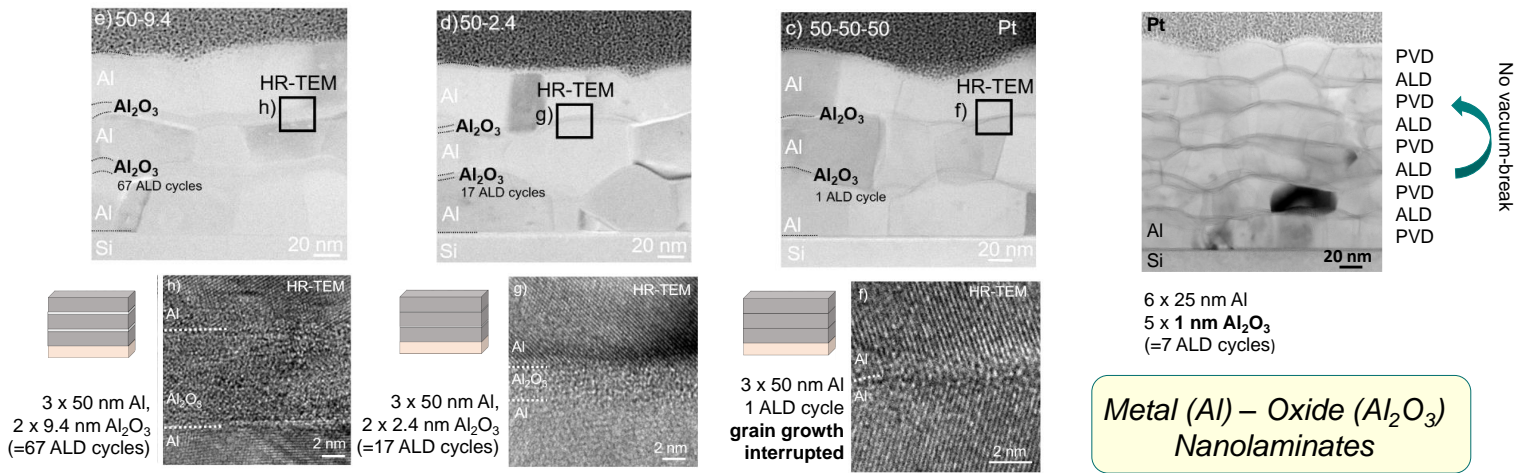
Design & characterization of surfaces & interfaces

Deformation behavior of Metal-Oxide nanolaminates

Combining Atomic Layer (ALD) and Physical Vapor Deposition (PVD) opens new possibilities for coating design with sub-nm precision

Variation of oxide layer thickness

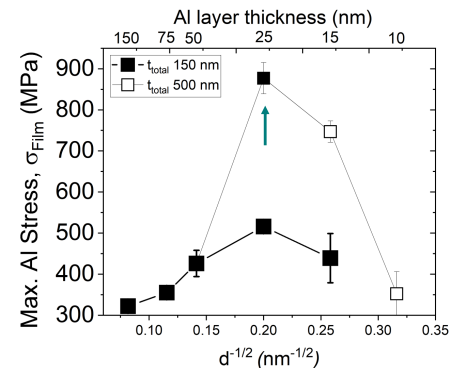
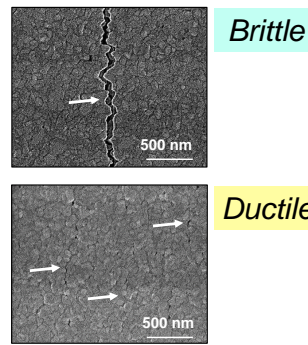
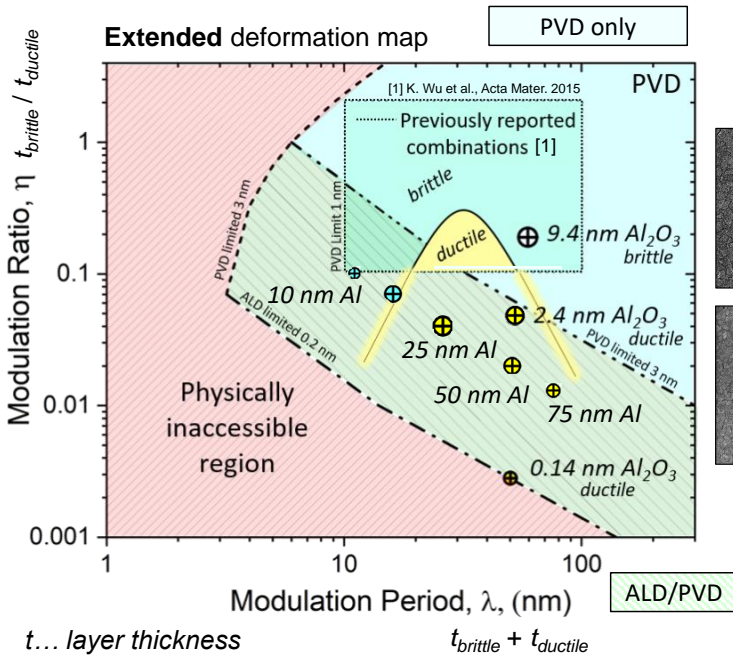
Variation of metal layer thickness



Deformation behavior on flexible polymer substrates

Challenge: Crack formation

Benefit: Strengthening mechanisms



Majority of nanolaminates exhibit ductile crack pattern

Maximum strength at 25 nm Al layer thickness



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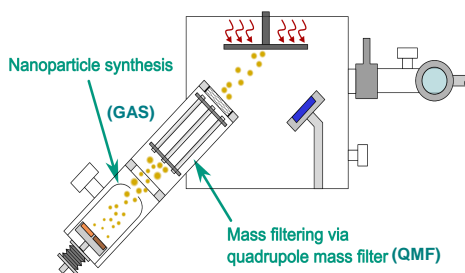


B. Putz et al. / Advanced Engineering Materials, 2022
 B. Putz et al. / Materials and Design, 2023

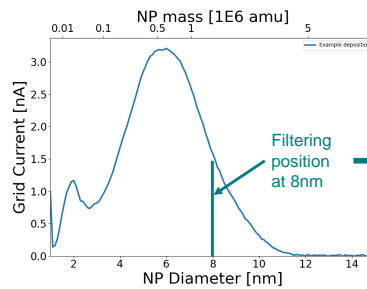
Design and characterization of interfaces and surfaces

Deposition and characterization of Cu nanoparticles for CO₂-reduction

□ **Nanoparticles (NPs)** have interesting properties for catalysis due to their high surface-to-volume ratio. **Gas Aggregation Sources (GAS)** in magnetron sputtering setups are a versatile technique for their synthesis.

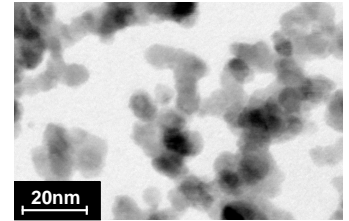


Schematic of NP deposition setup



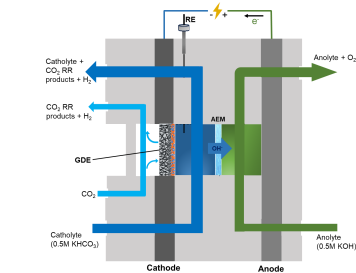
Typical signal obtained from QMF (in-situ)

TEM

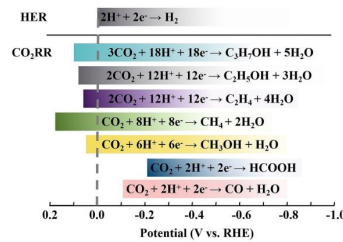


TEM-micrograph of Cu nanoparticles

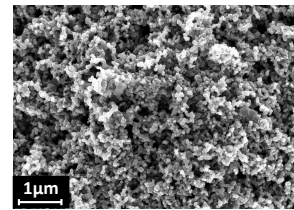
□ **Gas Diffusion Electrodes (GDEs)** are integral parts of the **Flow Cell**. GDEs consist of a **porous carbon support** and the **catalyst**, where **surface CO₂** is reduced to diverse Carbon products. **Copper NPs** are of special interest, since CO binds well to it, enabling reaction to products like methane, ethane or methanol.



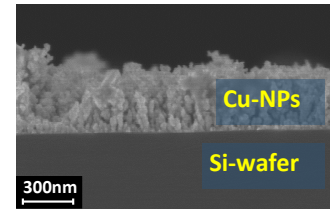
Schematic of a flow cell: GDE (left); counter electrode (right)



Possible products in CO₂ reduction reaction [1]



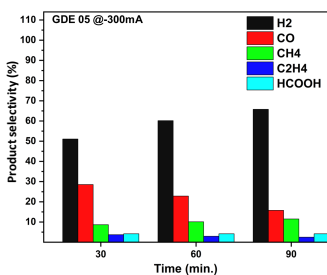
SEM-micrograph of the porous carbon support of the GDE surface to allow gas flow.



SEM cross-section of Cu nanoparticle thin film catalyst deposited on a Si wafer.

□ Results and challenges

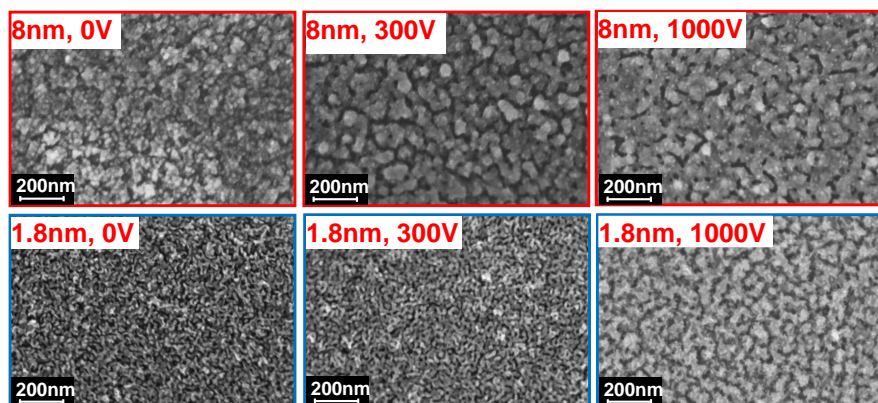
Catalytic tests with produced Cu catalysts



- Promising catalytic activity
- Variety of products found
- Issue with stability of catalysts

Study of the change of morphology with different substrate biases

SEM top-view images of Cu nanoparticle thin films (1.8 nm and 8 nm particle diameter) on Si.



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References: Project Nr. 896659 AMAGADE

[1] Chen, X., et al.(2023), ChemPlusChem 88(1)
<https://doi.org/10.1002/cplu.202200370>