Controlled microcrack steering into toughened regions – What microelectronics can learn from nature?

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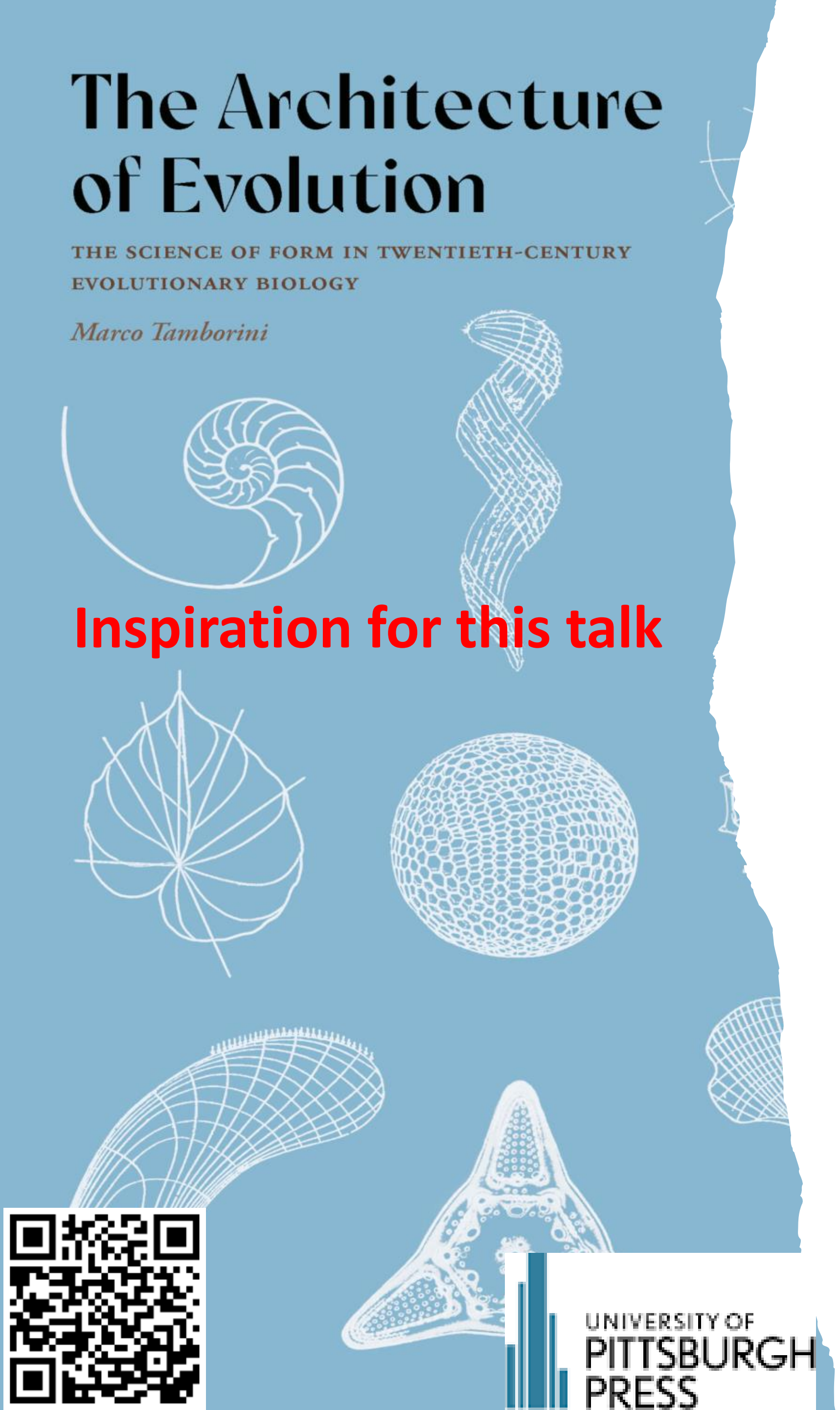
IRSP 2023, Bad Schandau, 25 April 2023

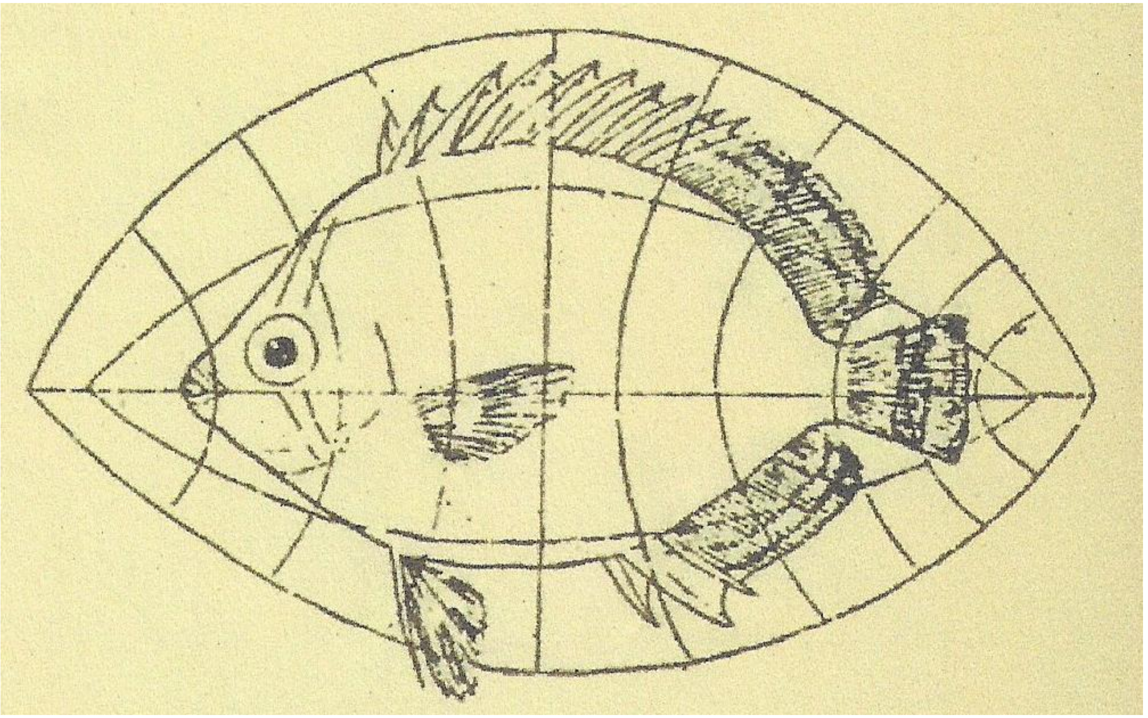


Inspiration for this talk

The Intersection of Biology and Technology

- A continuous cycle of blending biology and technology is emerging, blurring the lines between the two
- This trend has led to the creation of interdisciplinary research clusters dedicated to studying the enigmatic structures of form variation
- These books explore the encounters between the study of biological and technical forms, their production and the implications of these intersections.

Theoretical basis for the Science of Form
Biologists: The Architectural Approach to morphology



- **Form emerged from organizational principles.**
- **Materiality is only one aspect of understanding the essence of form, and the focus was rather on the notion of arrangement.**
- **The study of the functional organization of organic form requires a focus on structure and a technical vocabulary to describe form adaptation.**

M. Tamborini, The Architecture of Evolution (2022)

Impact of modern morphological research on engineering and materials science

Johann-Gerhard Helmcke: Explanation of form development and structure formation in diatoms.

- Small changes in physicochemical force relations could cause substantial variations in shell patterns.
- Forces could cause different kinds of morphogenesis.

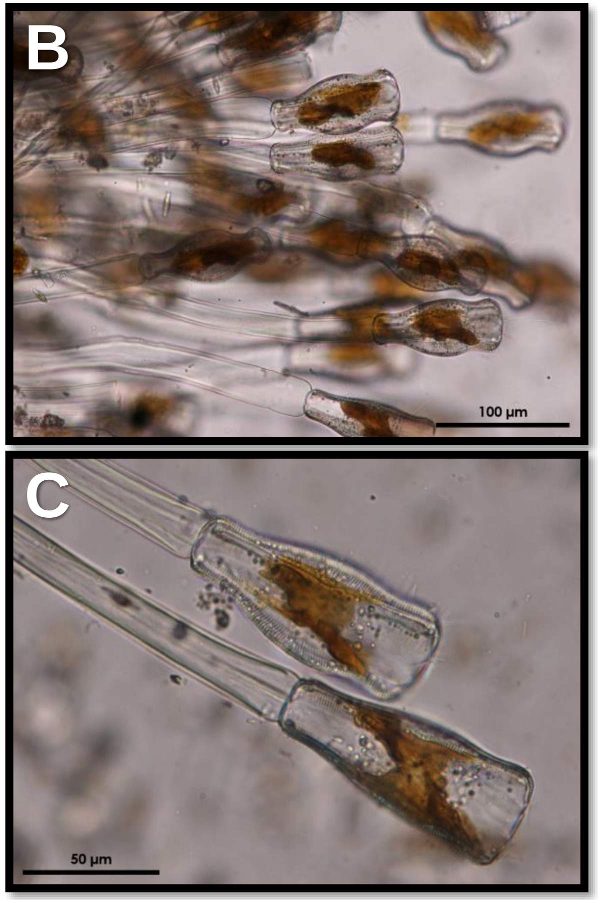
Microscopy (particularly electron microscopy and X-ray microscopy) allows to see similarities between organic morphogenesis and the creation of technical and architectural forms.

- Many structural elements formed on diatom shells are also well-known in the field of engineering and material science.
- Comparing natural diatom structures with recent developments in architectural lightweight constructions led to an explanation of both according to the same building principles.

J. G. Helmcke, **Bild der Wissenschaft (1968)**

Biomineralization ➔ Hierarchical structures
Example: *Didymosphenia geminata* diatom frustules *(Dunajec river)*

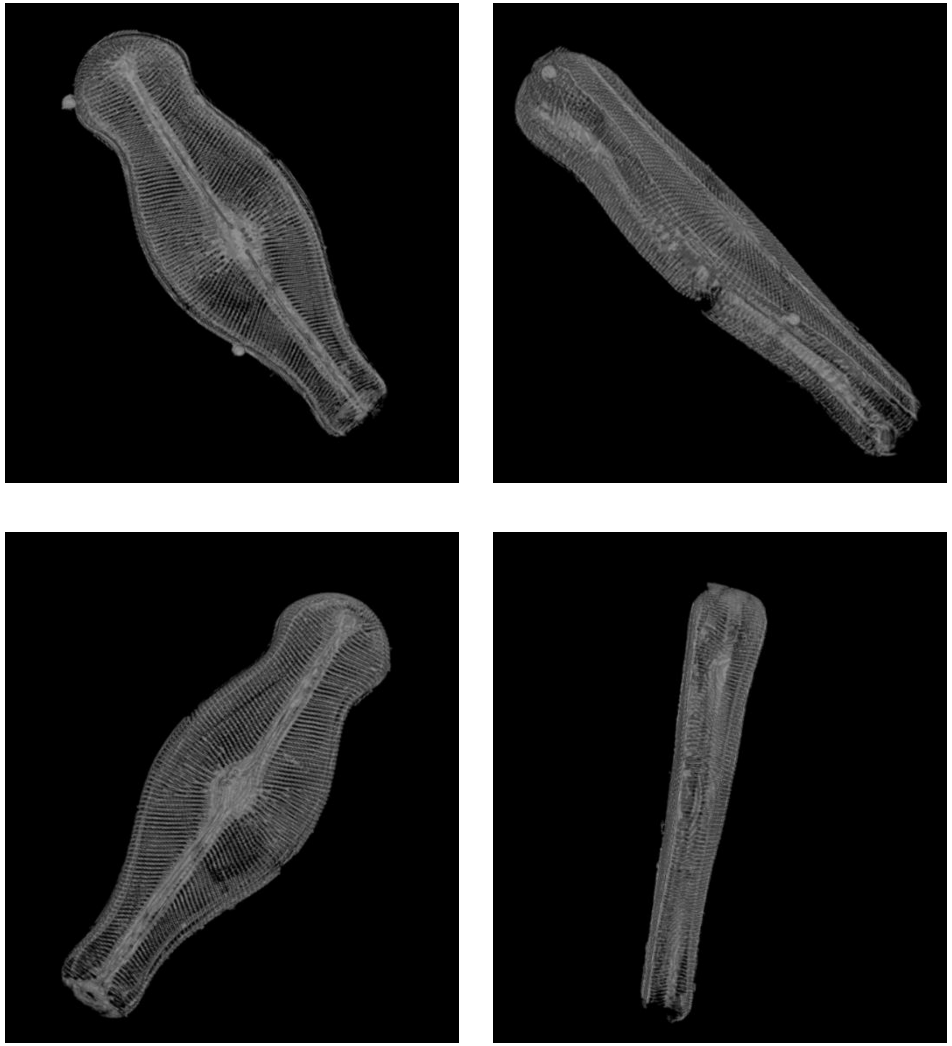
Didymosphenia geminata: **A:** macroscopic photo – material in natural habitat (rock), **B, C:** LM images of cells with stalks after sampling *(PhD thesis of I. Zgłobicka)*

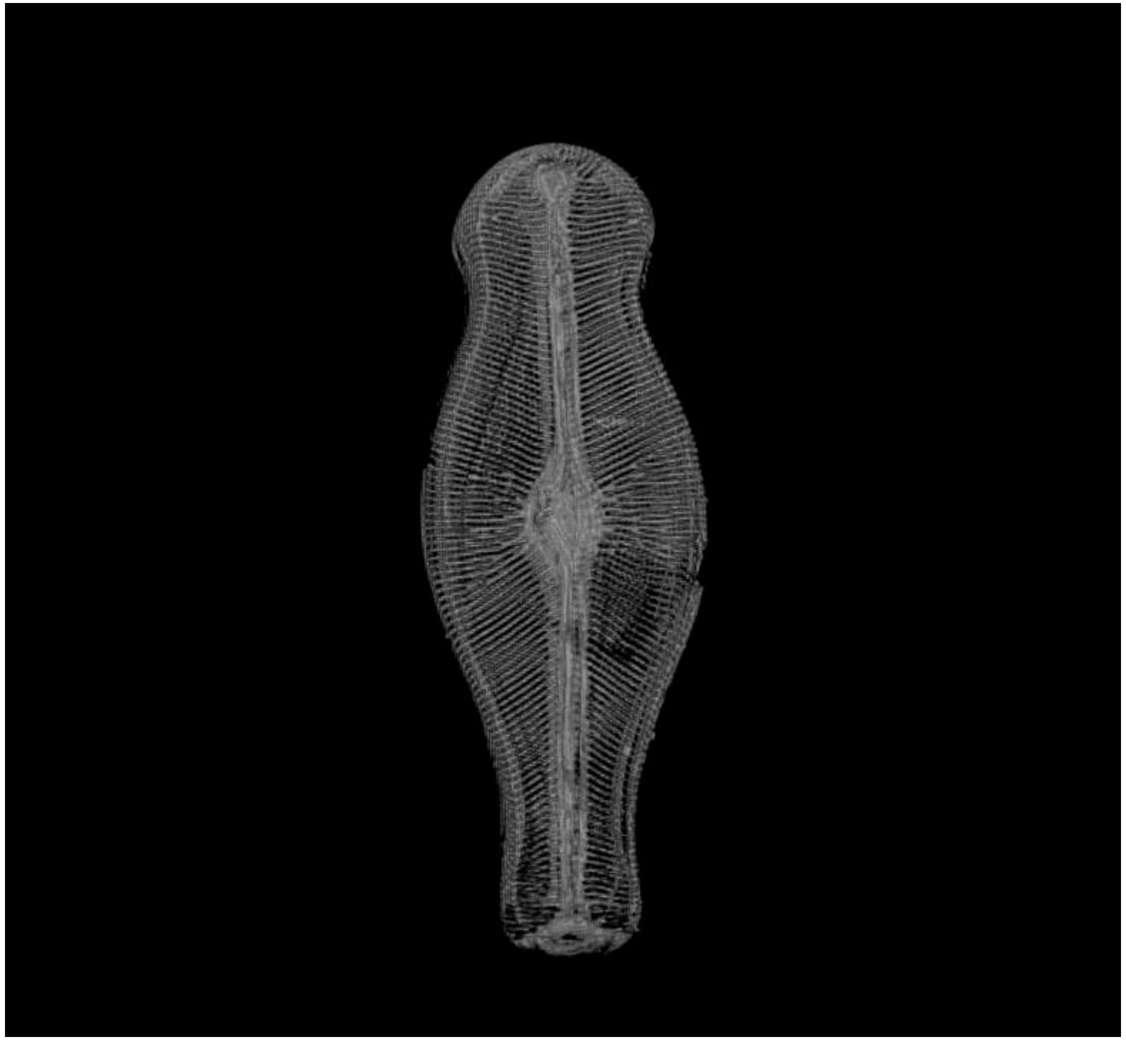


The frustule (cell wall): mainly of amorphous bio silica

I. Zglobicka et al., **Scientific Reports 7, 9086 (2017)**

3D imaging of the morphology of a *Didymosphenia geminata* diatom frustule based on nano-XCT data





I. Zglobicka et al., Scientific Reports 7, 9086 (2017)

Biomimetics
- What can we learn from nature?
- Which role miniaturized tests and high-resolution 3D imaging can play?

- **Natural materials (e.g. teeth, mollusk shells) are characterized by a unique combination of stiffness, strength and toughness ➔ outperform engineering materials.**

Why?

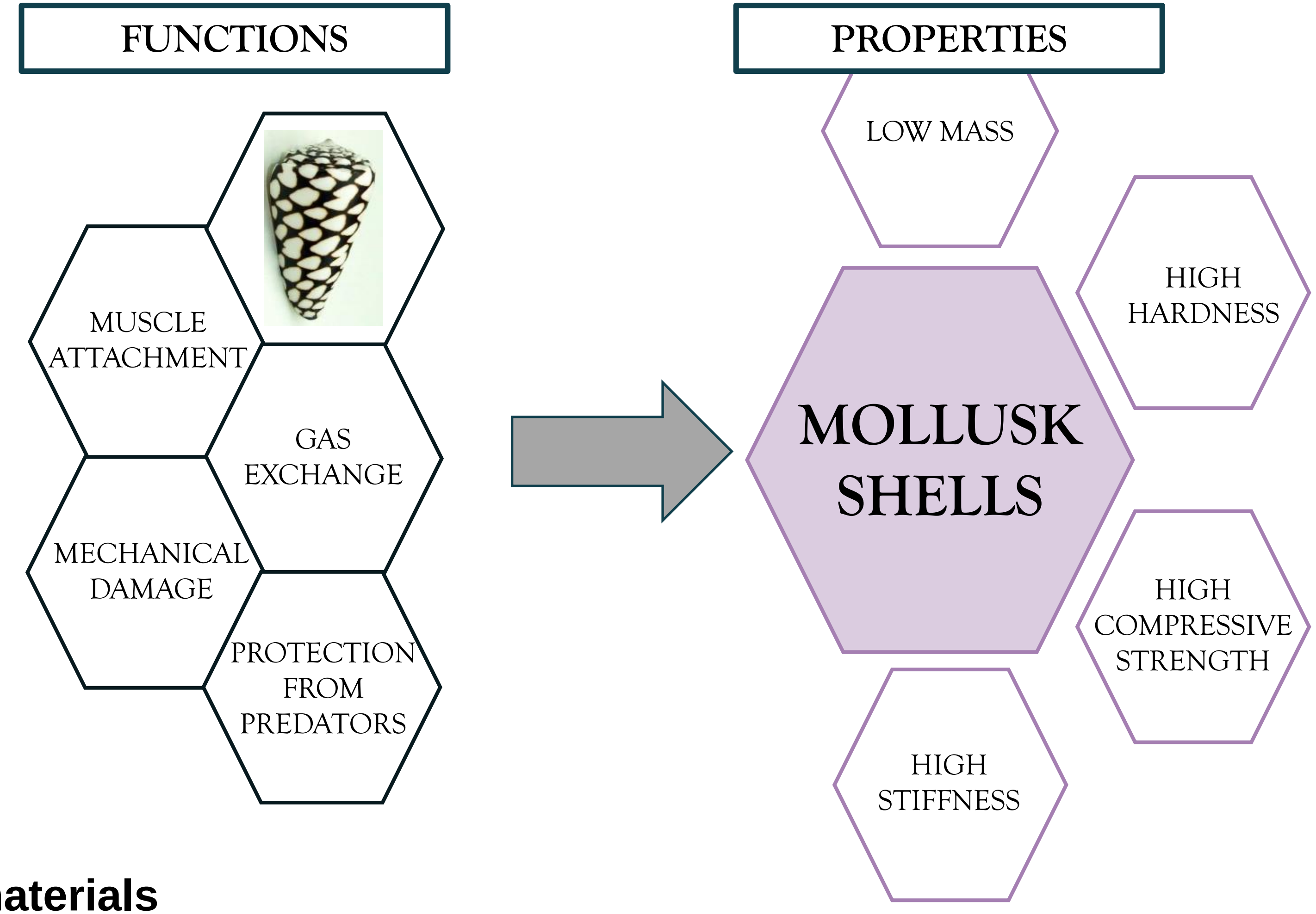
- **Biological objects have been „designed“ in a long-time evolution process.**
- **Microstructure of hierarchically structured biocomposites is taylored according to their functionality (which stress ? which environmental conditions ?).**

➔ We have to understand the hierarchical design (architecture) of the materials and the local mechanical properties ➔ determination of the local critical energy release rate of crack propagation ➔ experiment: micro-mechanical test and simultaneous crack imaging.

➔ Conclusions for engineering materials with high damage tolerance.

Design of mechanically robust structures in nature
Example: *Pinctada Margaritifera,* bivalve (Mollusk Shells, French Polynesia)

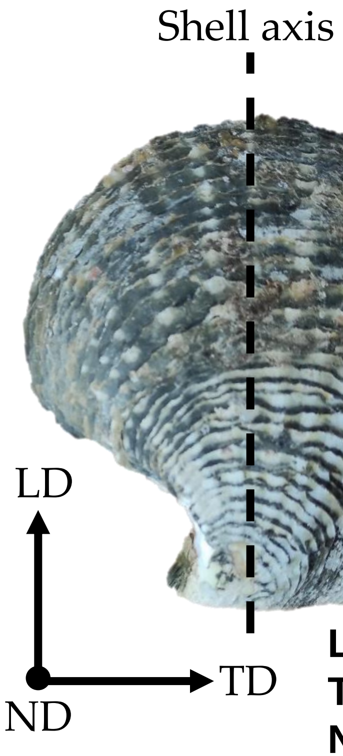
Shells: Calcareous exoskeleton formig part of marine and freshwater organisms

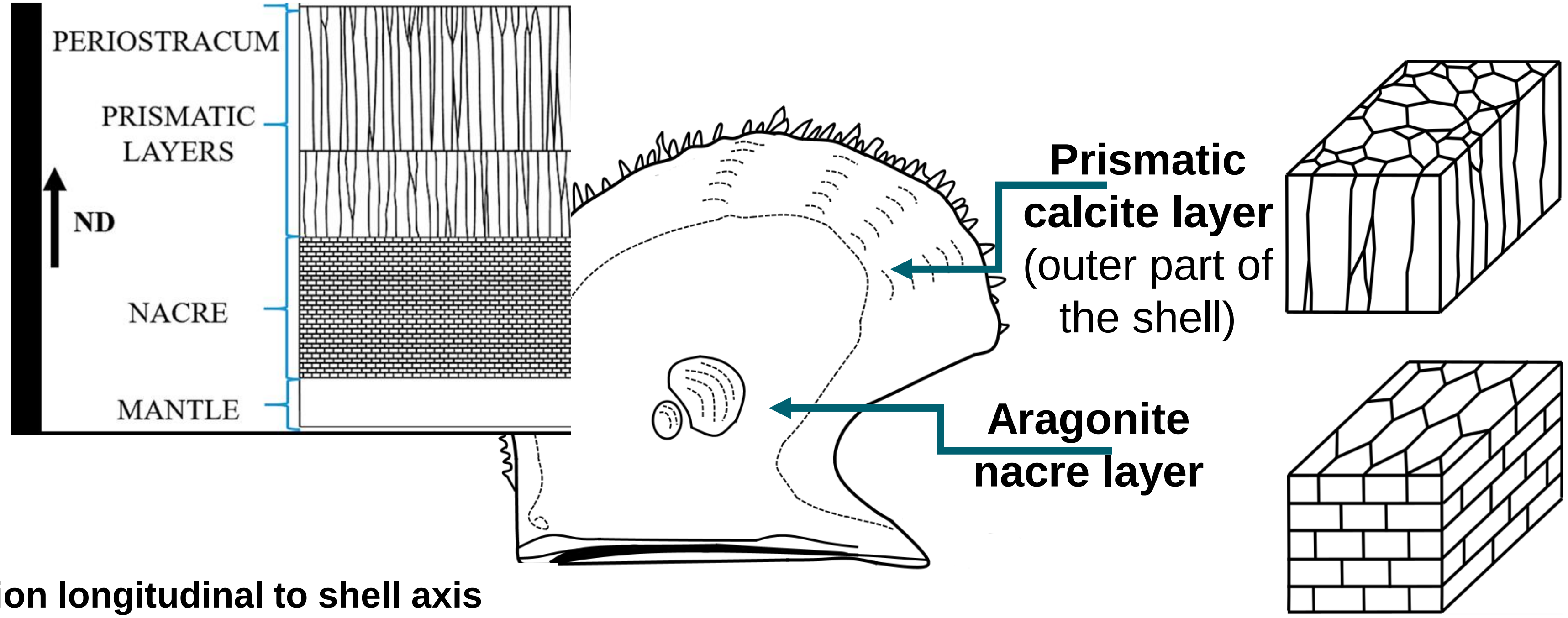


Motivation: Design of bio-inspired materials

X.W. Li et al., Journal of the Mechanical Behavior of Biomedical Materials 74 (2017) 54-71

Design of mechanically robust structures in nature
Example: *Pinctada Margaritifera,* bivalve (Mollusk Shells, French Polynesia)



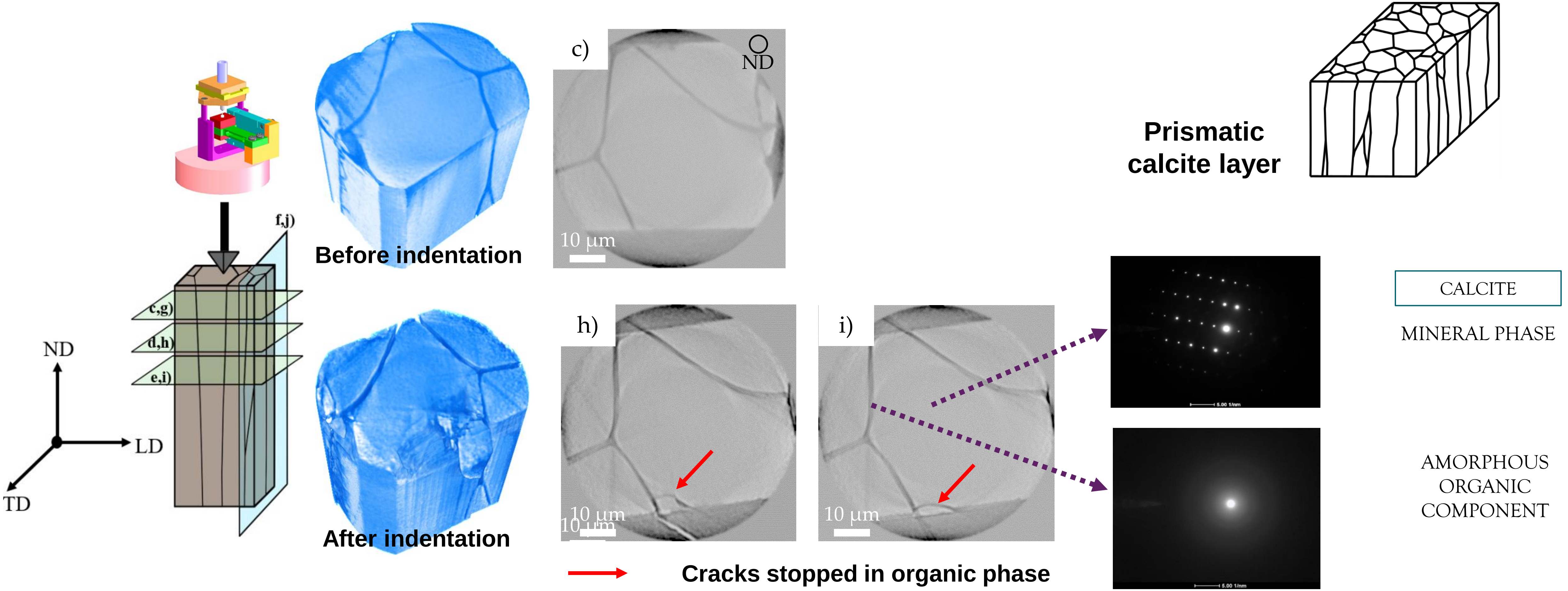


LD – direction longitudinal to shell axis
TD – direction transverse to shell axis
ND – direction normal to shell axis

Study of the anisotropy of the mechanical properties of the prismatic columnar calcite layer:
***In-situ* indentation experiment in the X-ray microscope**

X.W. Li et al., Journal of the Mechanical Behavior of Biomedical Materials 74 (2017) 54-71

In-situ indentation study of Mullusk Shell material in the nano-XCT tool *(Pinctada Margaritifera)*



Cracks formed by indentation propagate in the calcite prism and are stopped in the organic phase

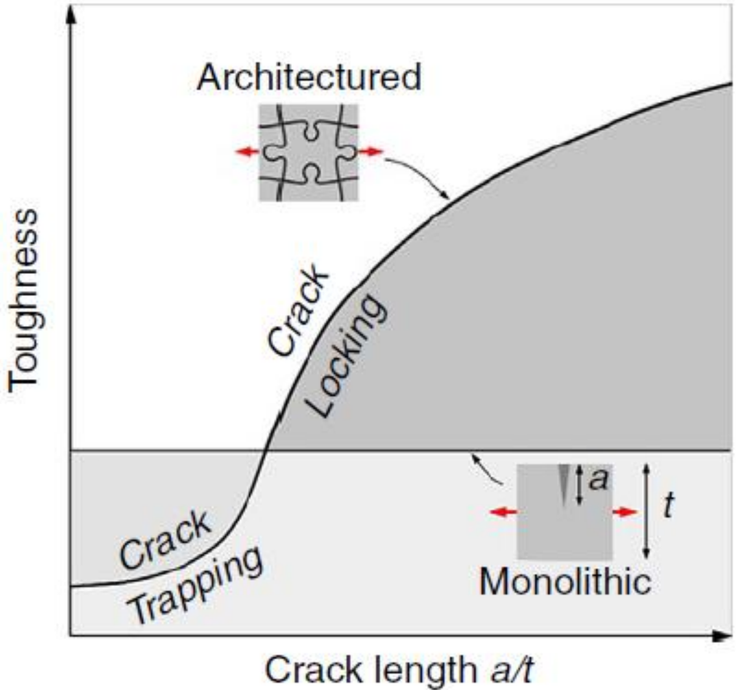
M. Strag et al., J. Nanomaterials 10, 634 (2020)

Biomimetics
- What can we learn from nature? – 3D design of bio-inspired materials!
- Which role miniaturized tests and high-resolution 3D imaging can play? – Understanding of fracture mechanics in small dimensions!

Natural materials are able to steer propagating cracks into toughened regions:

Specific hierarchical architectures designed for the „natural use case“ (considering the external load)

- **High-strength and high-stiffness building blocks (mineral phase)**
- **Ductile components (amorphous organic phase) and respective „weak“ interfaces.**



Toughened bio-inspired „architectured“ materials:

Phase 1 –
Crack propagation along weak interfaces requires low energy.

Phase 2 –
Steering of cracks into regions with high fracture toughness, trapping cracks in stable configurations.

M. Mirkhalaf et al, Nature Comm. 2014

Modern fracture mechanics in small dimensions: Controlled microcrack steering into toughened regions of BEoL stacks

Study of microcrack evolution in 3D-structured systems and materials (BEoL stack) requires monitoring of force and displacements at the micro- and nano-scale

➔

Combination of

- (miniaturized) mechanical tests
- nondestructive high-resolution imaging of the BEoL stack

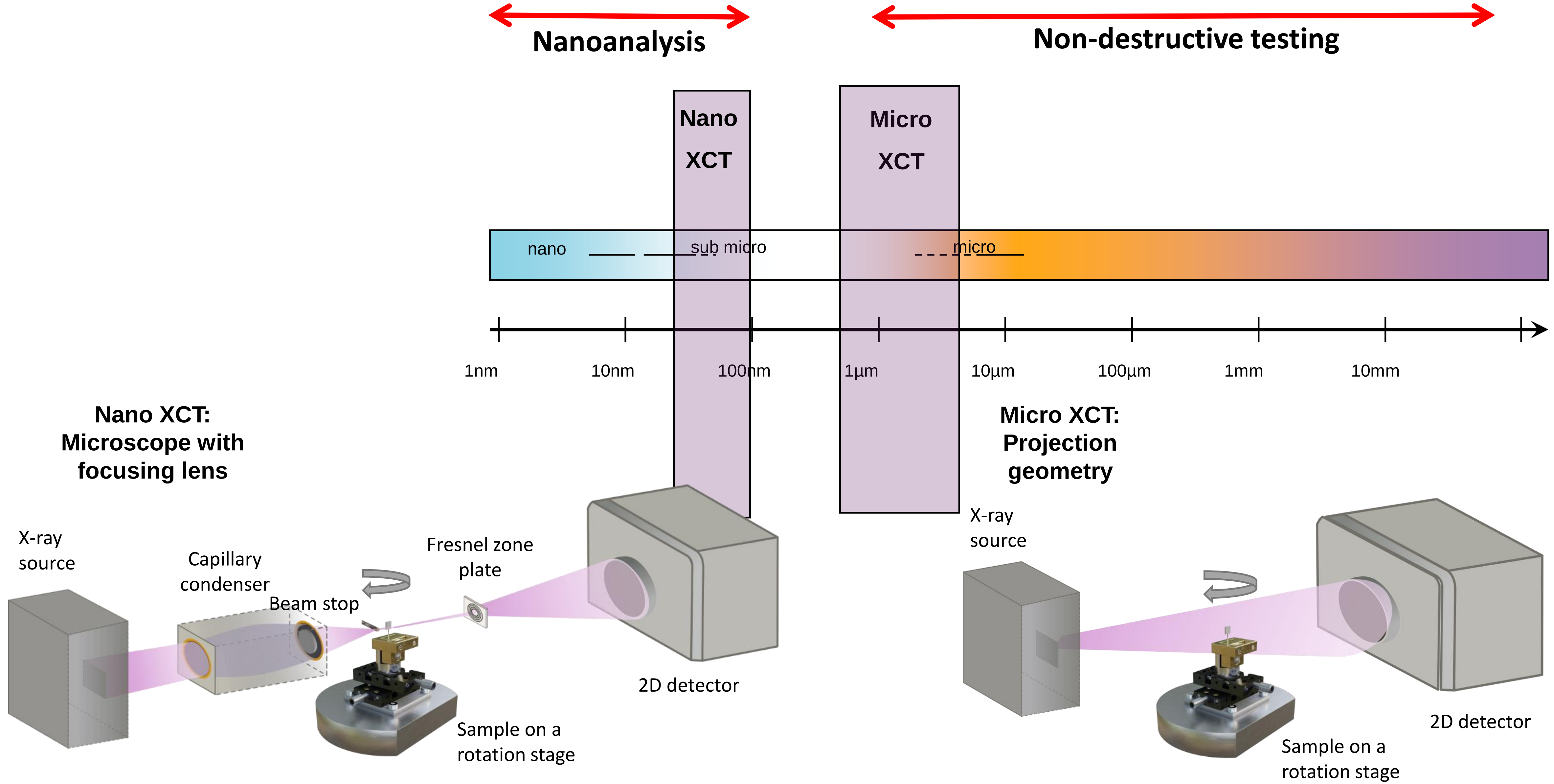
Unique solution for 3D imaging of microcrack evolution – sub-100nm resolution - while a force is applied:

***In-situ* micro double cantilever beam (micro-DCB) test in a laboratory nano-XCT tool**

➔

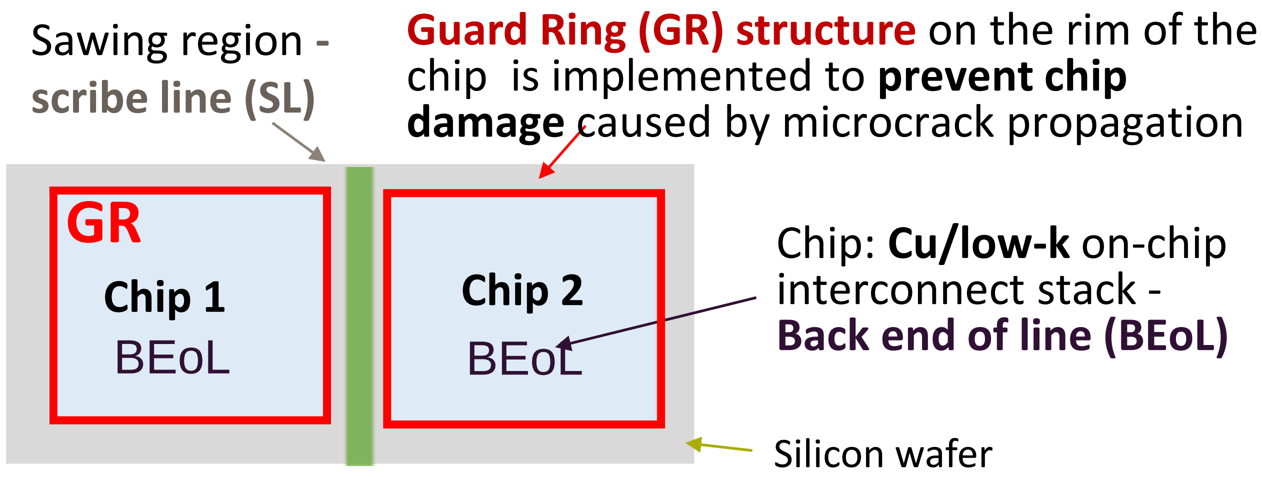
- Controlled steering of microcracks into regions with high fracture toughness

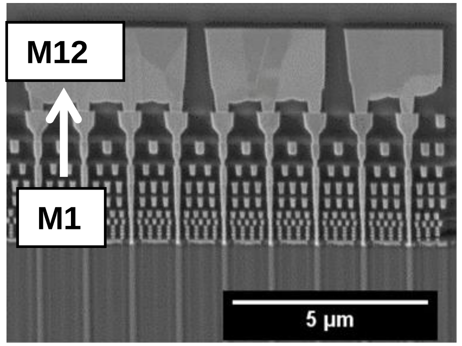
Why lens-based X-ray microscopy? Resolution !



Motivation: Need of mechanically robust microchips and chiplets

- **Wafer dicing ➔ Microchips with <u>microcracks</u> at the periphery**
- **Microcrack growth and eventually catastrophic failure have to be avoided**





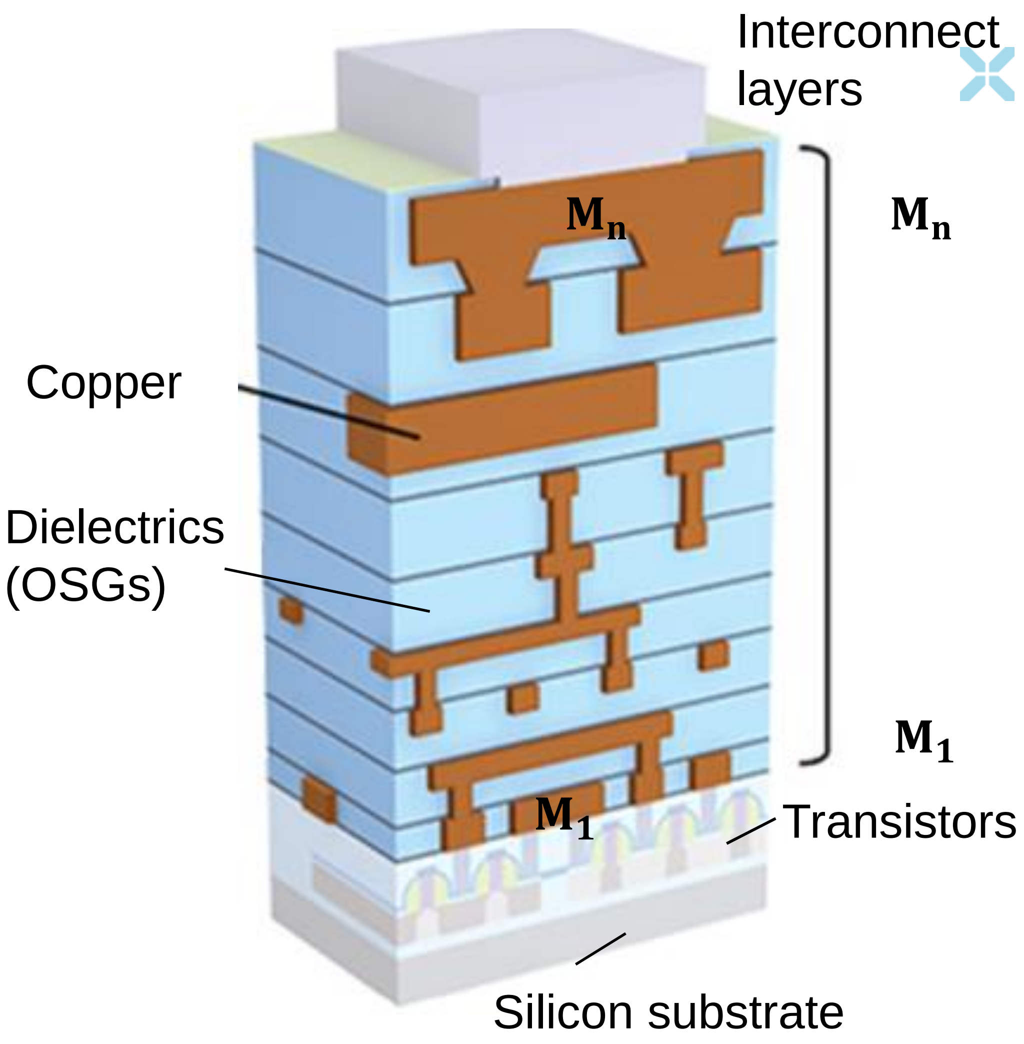
SEM image of a cross-section with GR structure of a microprocessor chip with 12 copper layers (M1 to M12)

State-of-the-art solution:
<u>metal guard ring structure</u> to prevent microchip damage caused by microcrack propagation

Future microelectronics - Technology trends

Microcrack propagation is pronounced by

- **geometrical shrinking of metal interconnects**
- **novel manufacturing technologies and integration schemes**
- **new materials for interconnect stacks and packaging**



Mechanical properties of BEoL stacks

Mechanical properties of (ultra-)low-k materials in the BEoL stack are critical

POR: CVD porous organosilicate glass (OSG) thin films

Hierarchically structured multilayer Cu interconnects

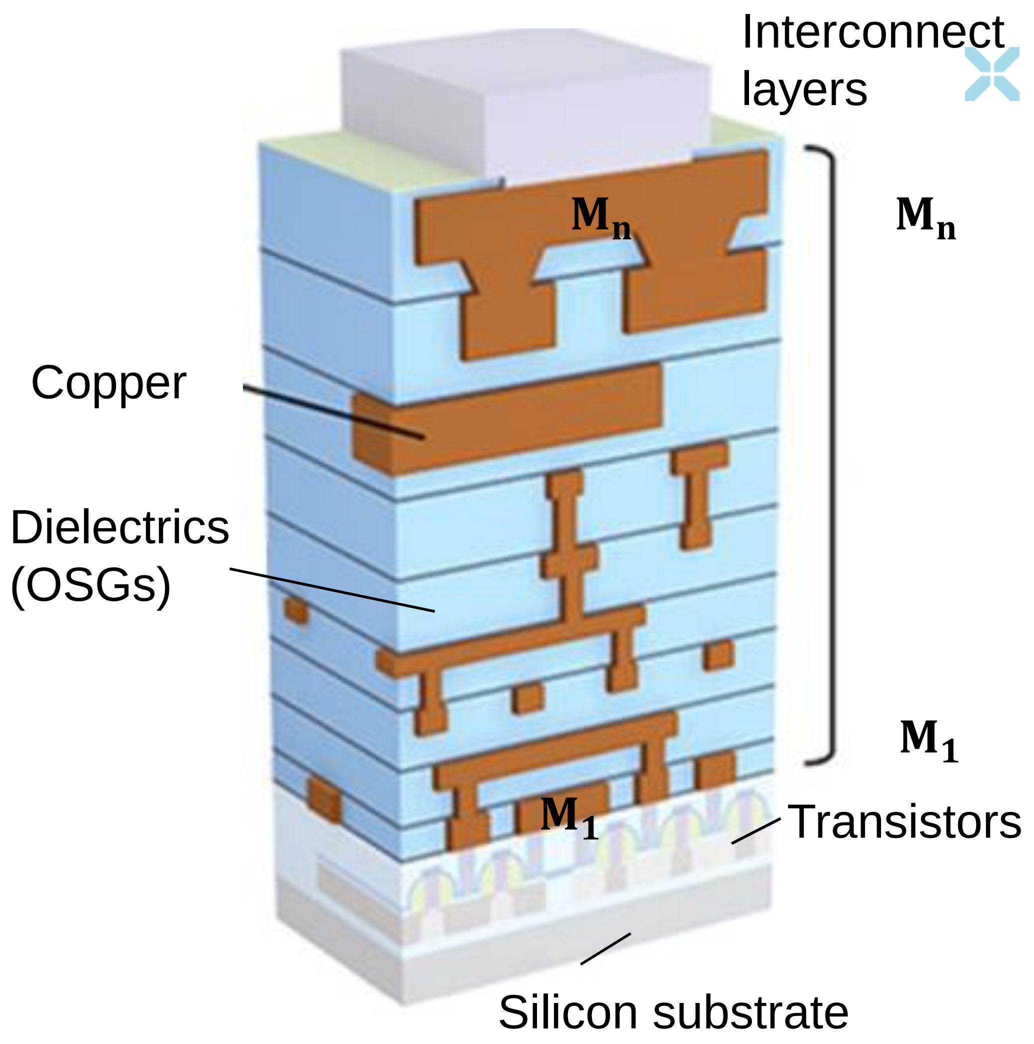
POR: Electrochemically deposited Cu structures, from several 10 nm to several μm

How metallic structures can mitigate the risk of microcrack propagation ?

<u>Design</u>: Effect of size of Cu structures on the critical energy release rate G_c for crack propagation in low-k dielectrics

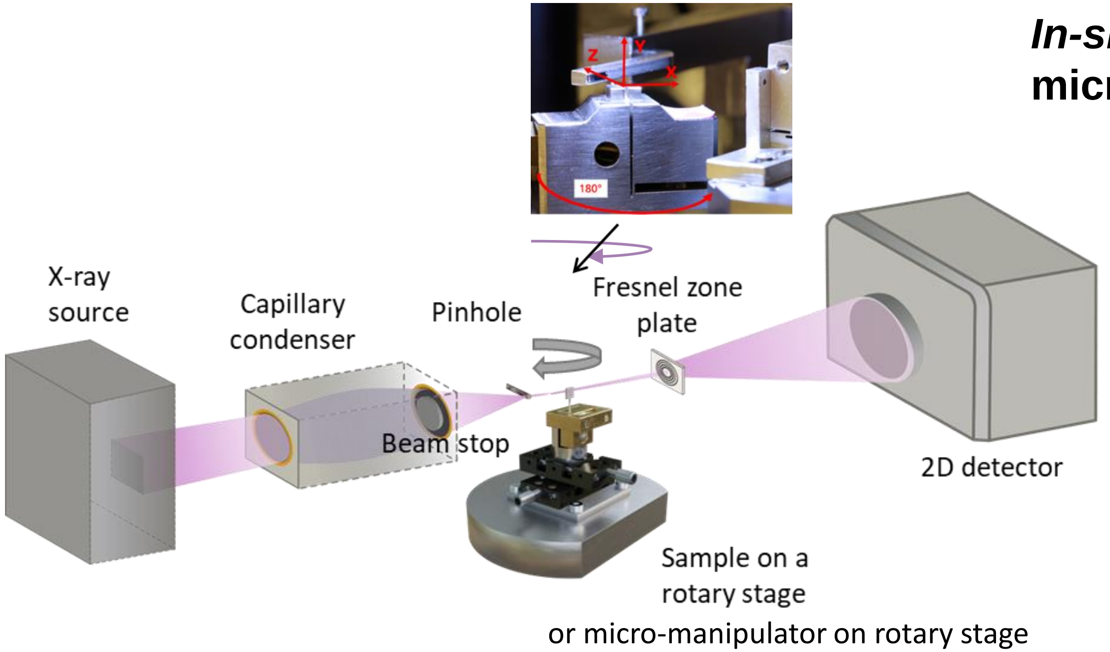
➔

Is a controlled microcrack steering into regions with high fracture toughness possible?



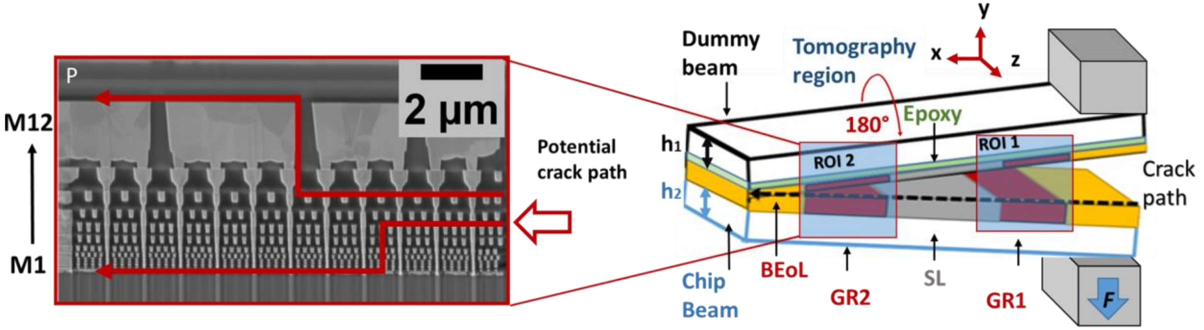
Micro Double Cantilever Beam test (micro DCB) in the X-ray microscope

***In-situ* crack evolution study of guard ring structure: micro-DCB tester (study under load)**



Laboratory X-ray microscope (8 keV): 50 nm spatial resolution (sample thickness 50 µm)

Typical "sandwich" specimen (chip and dummy) dimension: 50 µm × 50 µm × 1000 µm



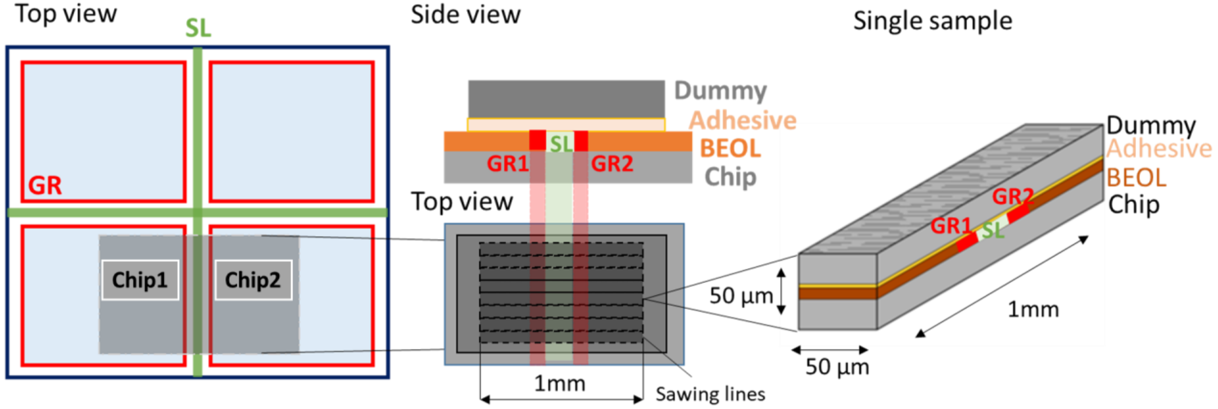
ROI GR: guard ring structure M1 – M12

GR - guard ring
ROI - region of interest
SL- scribe line
BEoL – Back end of line

Scheme of the sample geometry*

*Rotated by 90° compare to set-up

Process of Mechanical sample preparation

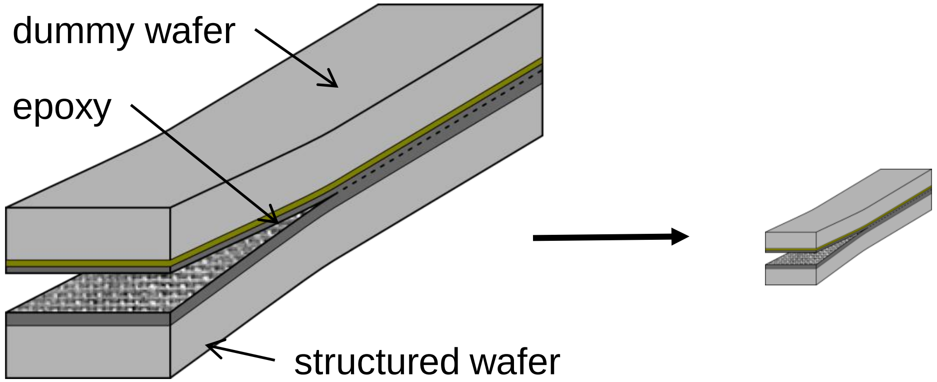


S. Niese, PhD Thesis, BTU Cottbus 2015, K. Kutukova, PhD Thesis, BTU Cottbus 2023
K. Kutukova et al., Mater. Today Comm. 2018, K. Kutukova et al., Appl. Phys. Lett. 2018

Why miniaturized double cantilever beam test? – X-ray transmission, fracture mode !

Miniaturization

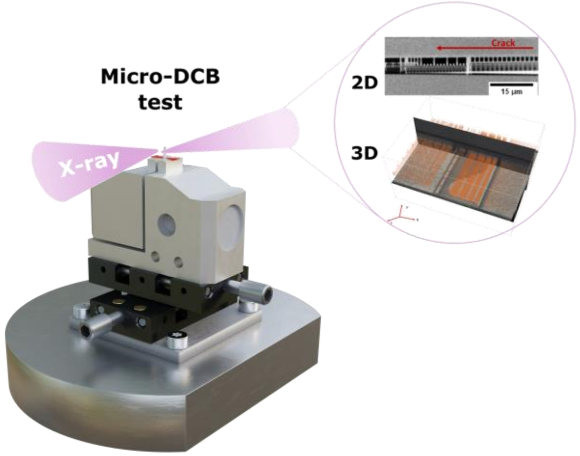
- Limited space in the X-ray microscope
- Sample thickness ~ 50 µm ("X-ray transparent" sample @ 8 keV)

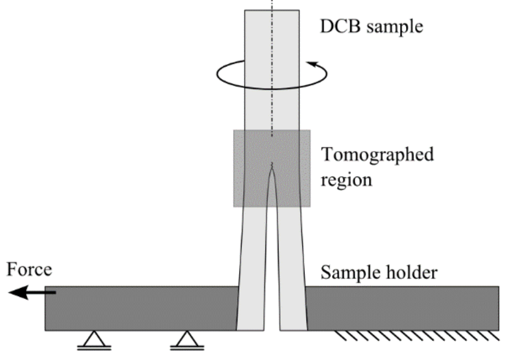


Standard DCB test:
(*ex-situ*) fracture mechanics
➔ G_c

Miniaturized DCB:
in-situ 3D crack evolution using X-ray microscopy

- **Displacement-controlled tester ➔ Critical energy release rate (Gc) investigation in patterned multilayer stacks by measuring crack length and crack opening**
- **Adaption of the standard DCB test to the more complex micro-DCB geometry**





- Description of fracture modes at nano-scale
- Fixed feature position (e.g. crack) during sample rotation (tomography)
- Reasonable load/displacement range
- Stable operation and repeatability of the experiment

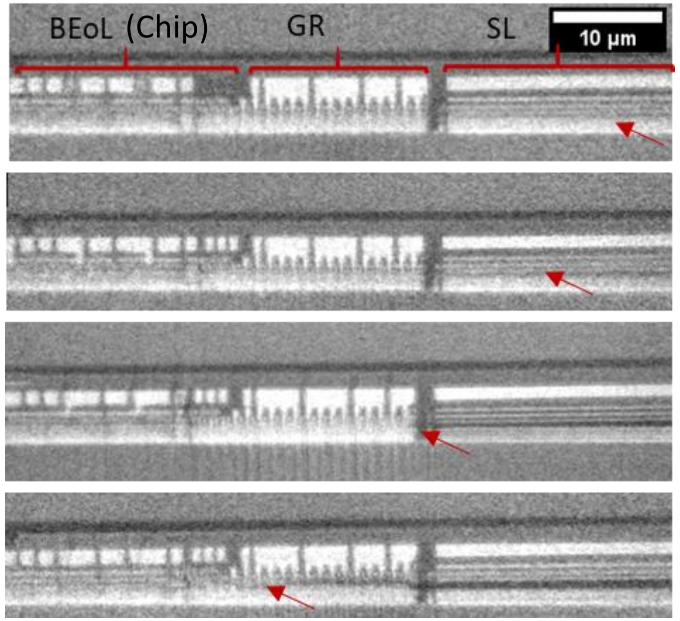
K. Kutukova et al., Mater. Today Comm. 2018
K. Kutukova et al., Appl. Phys Lett. 2018

In-situ micro-DCB test in the nano-XCT tool: 3D

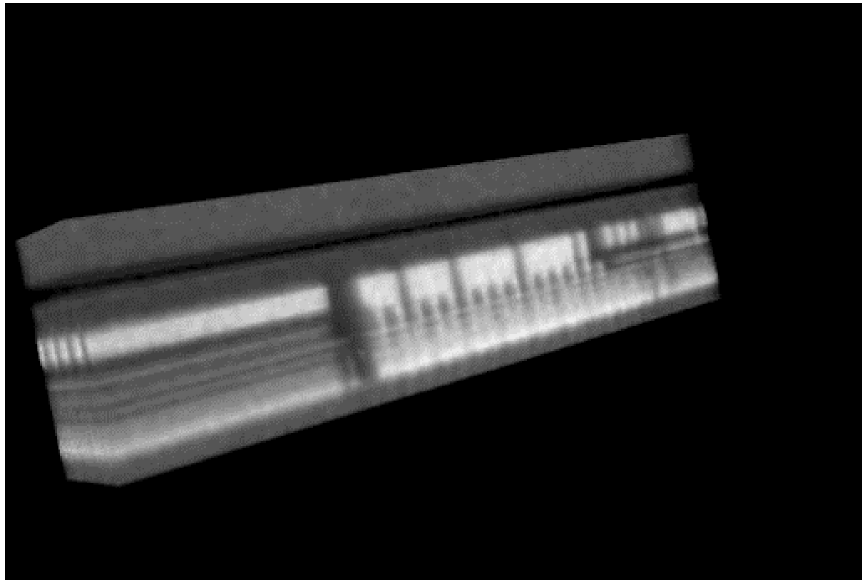
Crack propagation in on-chip interconnect stacks and GR structure of microchip

→ **Crack path localization in 3D**

Virtual cross-sections during micro-DCB experiment at certain loading state



3D data during or after micro-DCB test
→ detailed crack path investigation



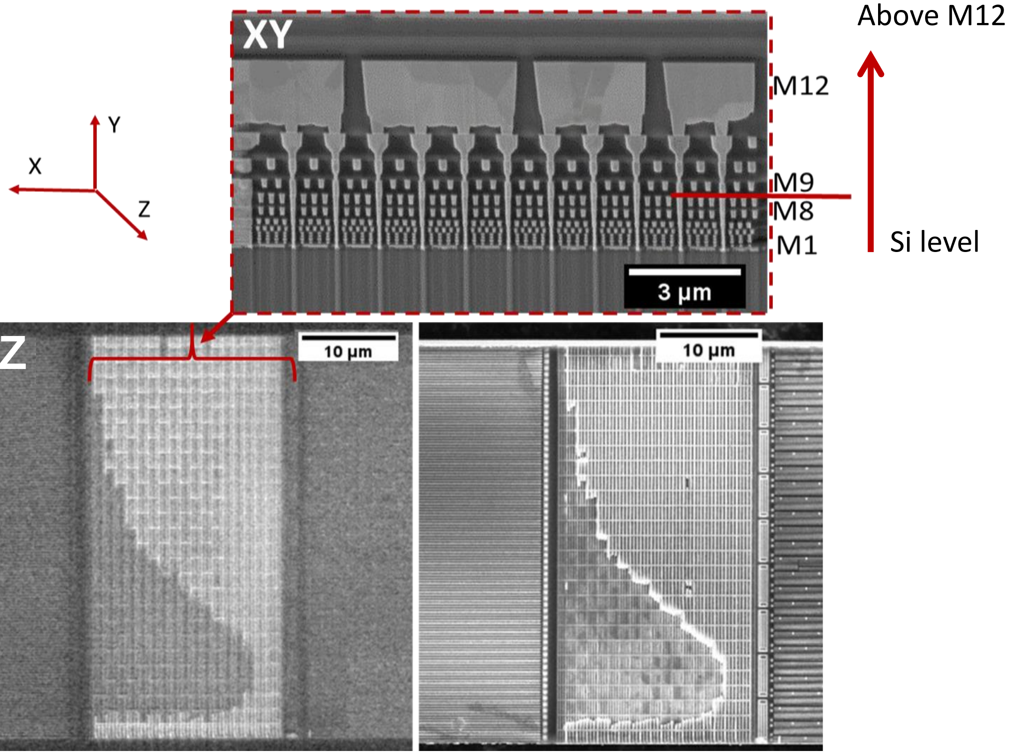
3D reconstructed data at different loading steps

K. Kutukova et al., Appl. Phys. Lett. 2018

In-situ micro-DCB test in the nano-XCT tool

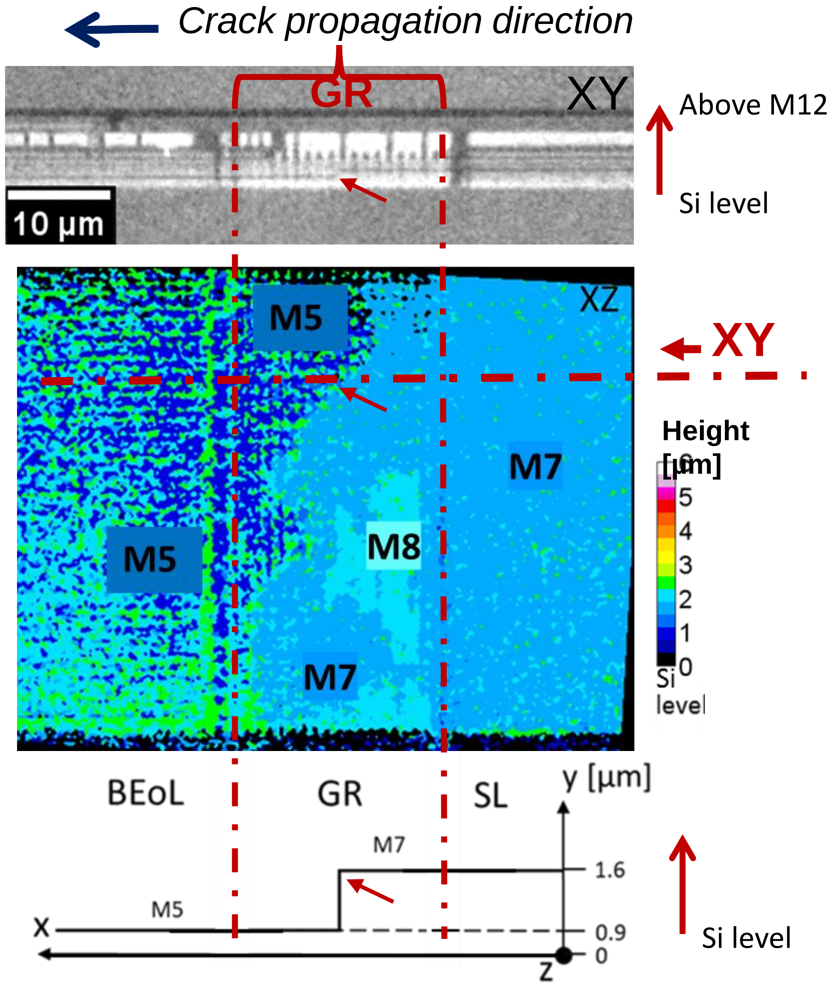
Visualization of the crack path in 3D interconnect systems

Metallization layers
FIB cross-section/SEM image



Virtual horizontal cross-section based on 3D data; M8

SEM image; M8



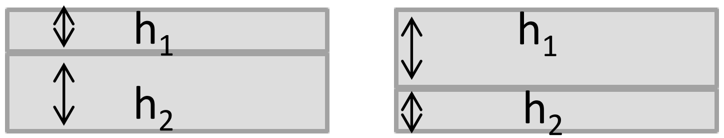
K. Kutukova et al., Appl. Phys. Lett. 2018, K. Kutukova et al., Materials & Design 2022

Crack steering - Fracture modes

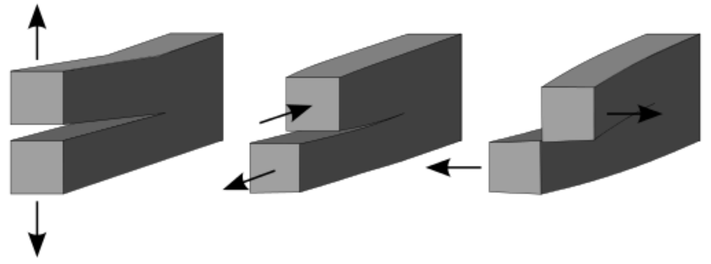
DCB test with tailored goemetry: Tuning fracture mode mixity
by the ratio e = dummy beam thickness / chip beam thickness

$$e = \frac{h_1}{h_2}$$

e – beam ratio
h_1 – beam height 1 (Dummy)
h_2 – beam height 2 (Chip)

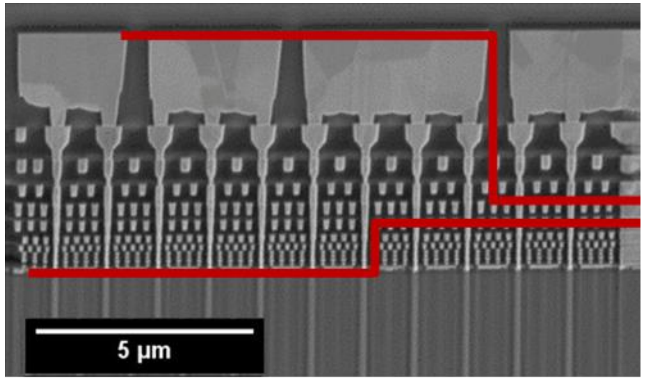


- **Fracture mode and tests:**
  - Mode I (SDCB test)
  - Mixed mode (ADCB)
- **Micro-DCB test**
  - e = 1 → Mode I
  - e ≠ 1 → Mode mix



e affects microcrack pathway

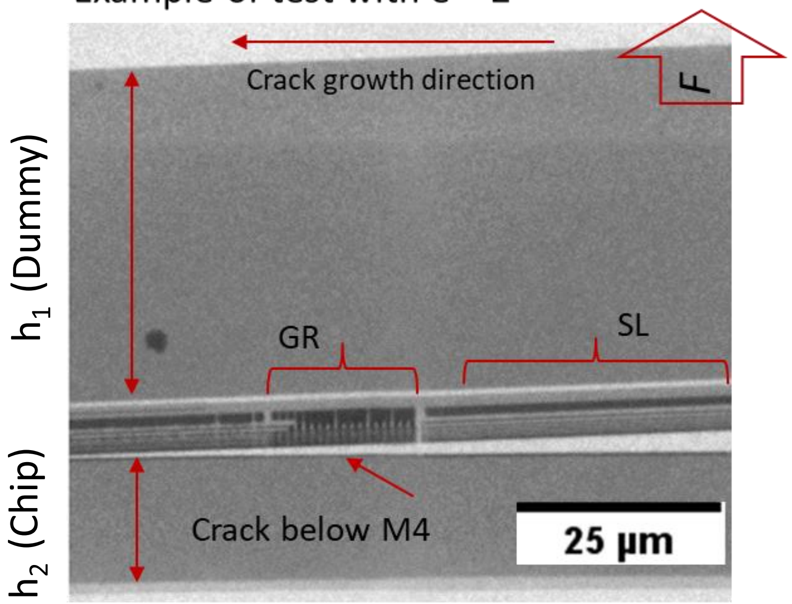
Mode mixity to steer the crack in GR



Potential crack path

Demonstrates possibility to steer cracks into regions with higher toughness

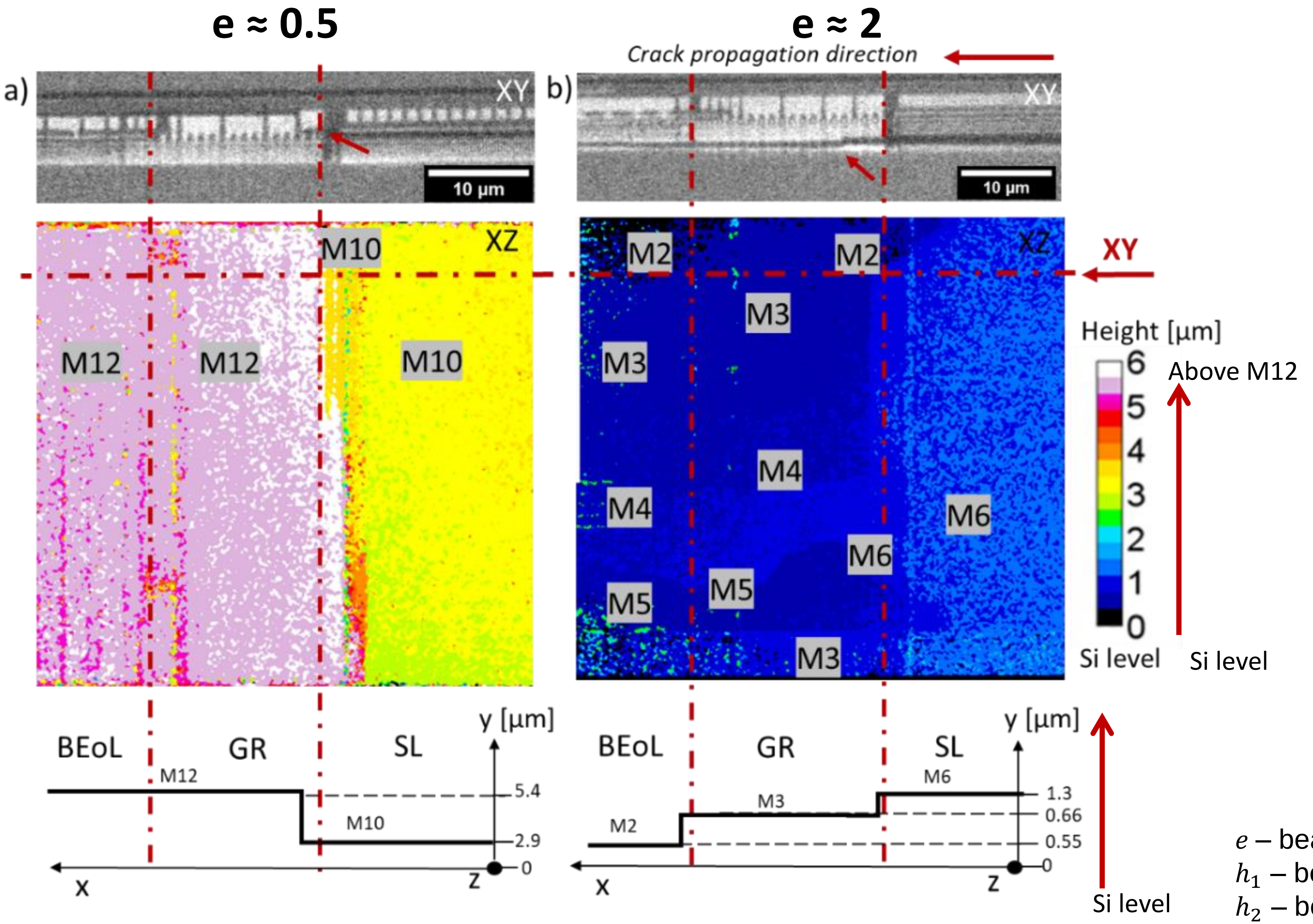
Example of test with e = 2

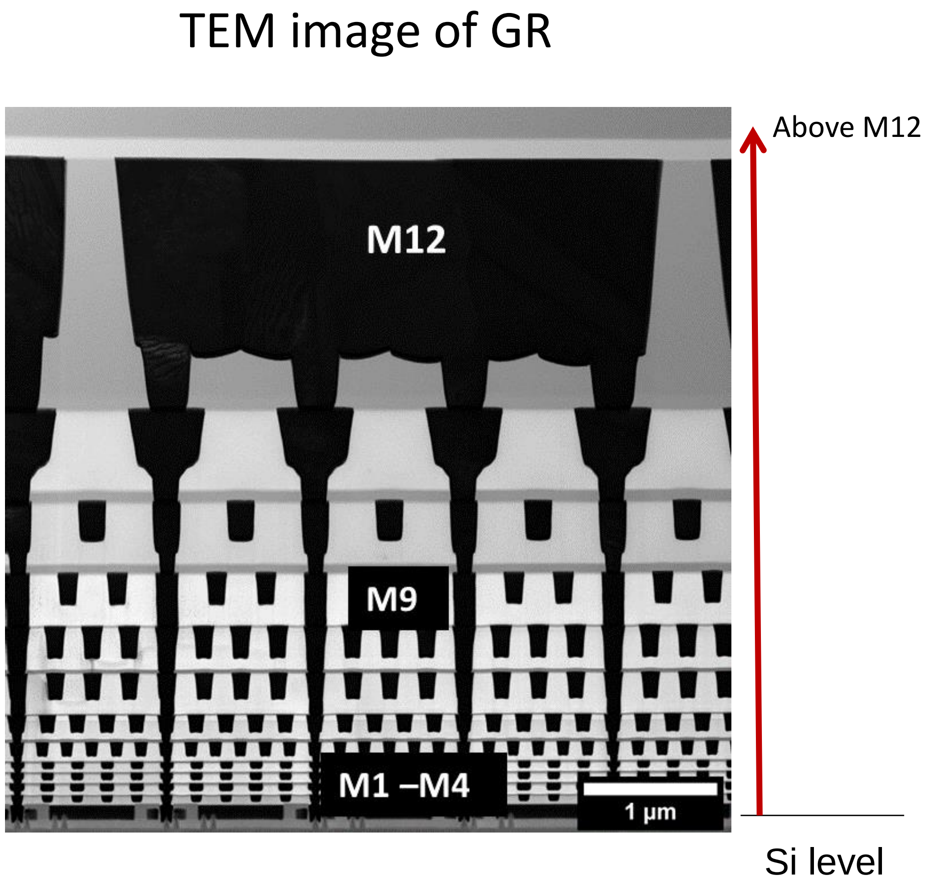


E. Zschech, M. R. Elizalde, in "More-than-Moore Devices and Integration for Semiconductors" (eds. F. Iacopi, F. Balestra) Springer 2023

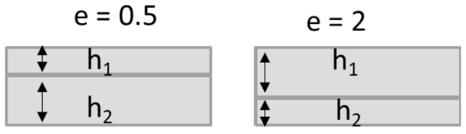
Controlled steering of microcracks

Image analysis for crack localization based on the 3D data set





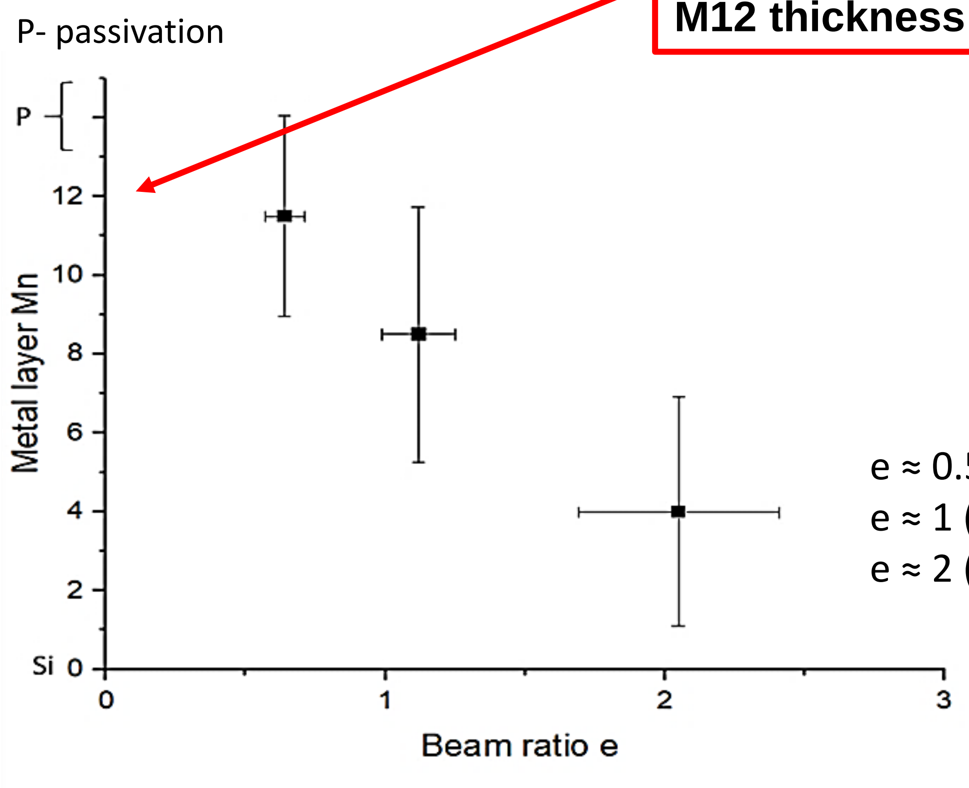
e – beam ratio
h_1 – beam height 1 (Dummy)
h_2 – beam height 2 (Chip)



K. Kutukova et al., Materials & Design 2022

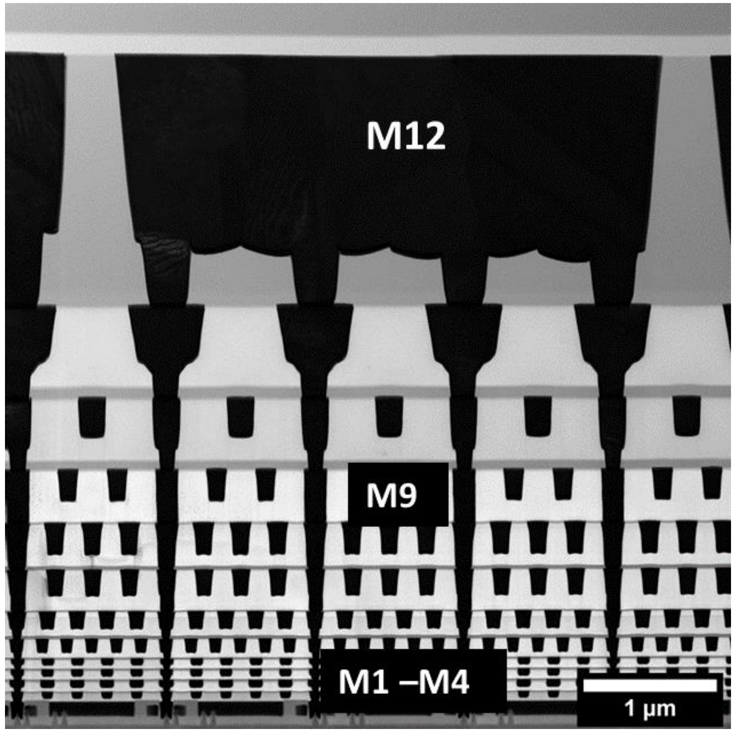
Controlled steering of microcracks – Dependence on micro-DCB beam ratio

Relatively high fracture toughness:
M12 thickness ~ 2 µm

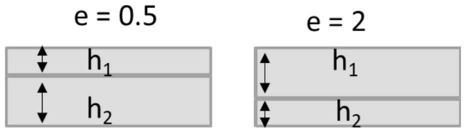


e ≈ 0.5 (7 samples)
e ≈ 1 (9 samples)
e ≈ 2 (12 samples)

TEM image of GR



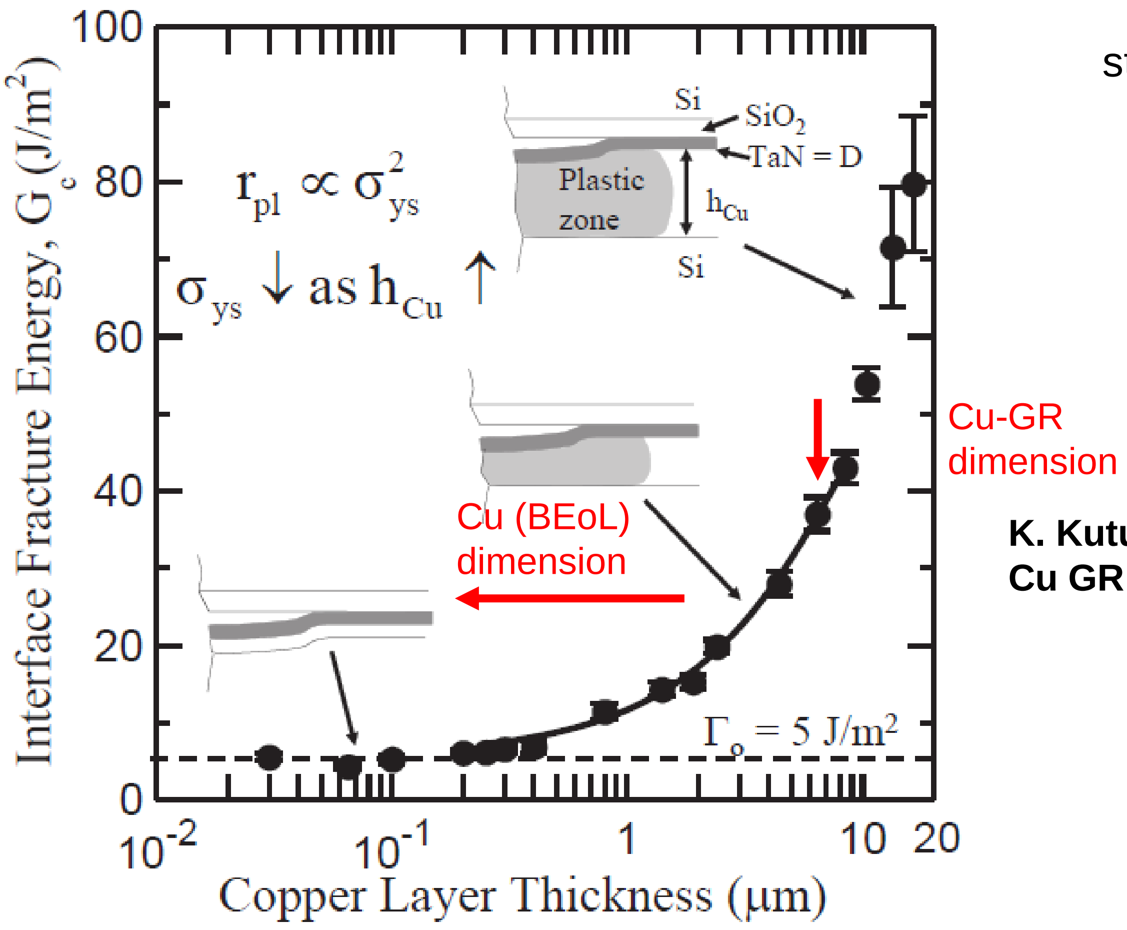
e – beam ratio
h_1 – beam height 1 (Dummy)
h_2 – beam height 2 (Chip)



Altered beam ratios allow to tailor mode mixity ➔
Experimental approach for controlled microcrack steering; preferably to regions of high metallization levels with higher G_c (➔ e < 1)

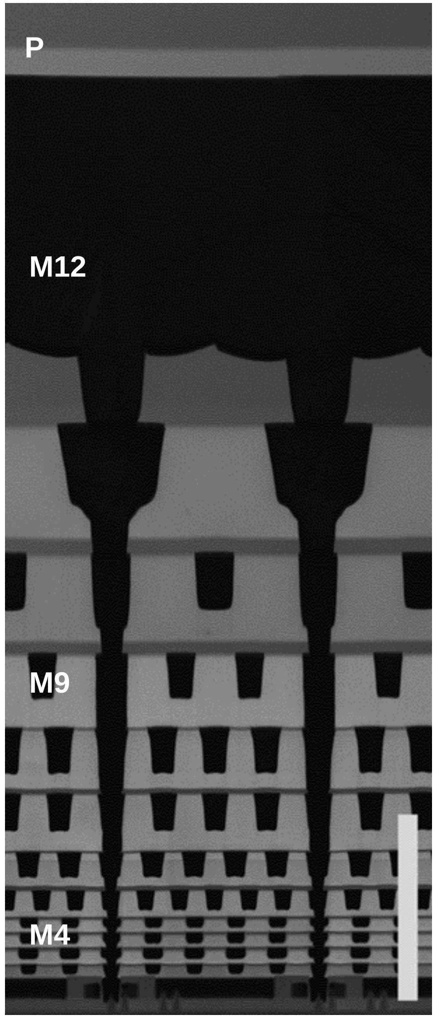
K. Kutukova et al., Materials & Design 2022

The effectiveness of the guard ring structure to stop micro-cracks depends on materials and design



TEM image of a part of a GR structure with 12 metallization layers and a post passivation layer P

K. Kutukova et al., IRSP 2023:
Cu GR: G_c increased to > 30 J/m²



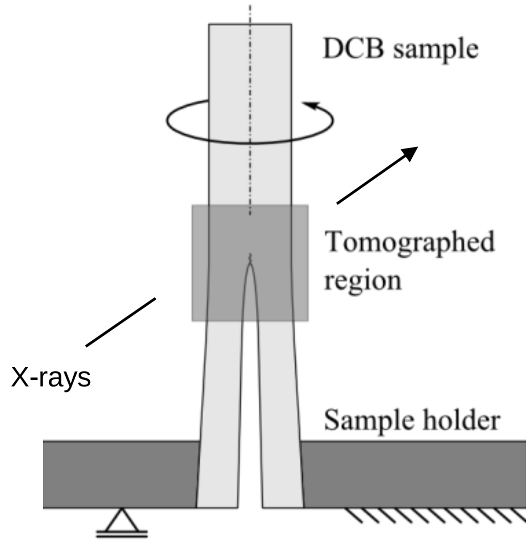
Scale bar 1 micron

Cu structures > 1 μm: Increase of fracture toughness / G_c !

M. Lane, R. Dauskardt, J. Mater. Res. 2000
K. Kutukova et al., Materials & Design 2022

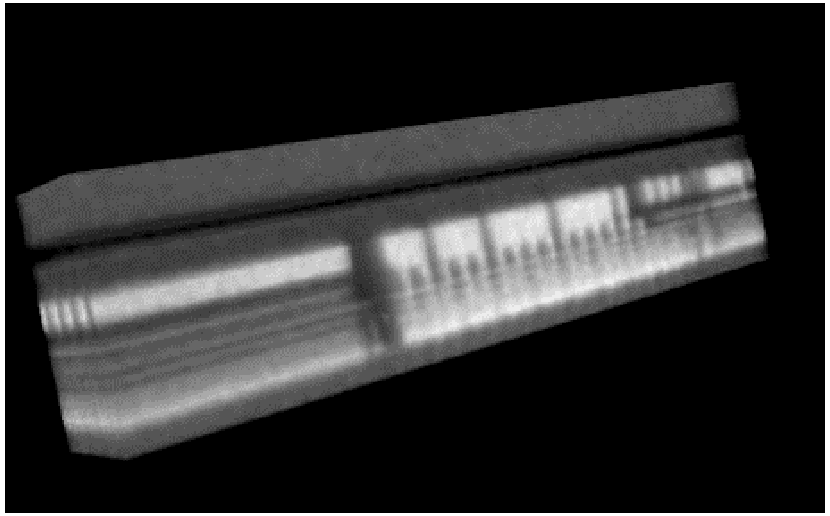
Summary

High-resolution 3D imaging of microcrack evolution while a mechanical force is applied:
<u>Displacement-controlled micro-DCB test in a laboratory nano-XCT tool.</u>



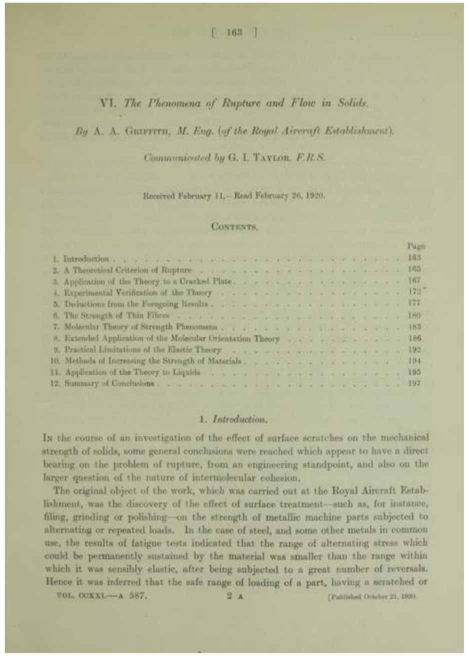
Results:

- **Experimentally demonstrated the controlled microcrack steering by tuning the fracture mode mixity locally at the crack tip.**
- **The data provide valuable input for the design of guard ring structures → Risk mitigation!**



3D data during or after micro-DCB experiment → detailed crack path investigation

100 years of Griffith‘s Theorem – What will be the next?



[163]

VI. *The Phenomena of Rupture and Flow in Solids.*

By A. A. Griffith, *M. Eng.* (*of the Royal Aircraft Establishment*).

Communicated by G. I. Taylor, *F.R.S.*

Received February 11,—Read February 26, 1920.

Contents.

1. *Introduction.*

In the course of an investigation of the effect of surface scratches on the mechanical strength of solids, some general conclusions were reached which appear to have a direct bearing on the problem of rupture, from an engineering standpoint, and also on the larger question of the nature of intermolecular cohesion.

The original object of the work, which was carried out at the Royal Aircraft Establishment, was the discovery of the effect of surface treatment—such as, for instance, filing, grinding or polishing—on the strength of metallic machine parts subjected to alternating or repeated loads. In the case of steel, and some other metals in common use, the results of fatigue tests indicated that the range of alternating stress which could be permanently sustained by the material was smaller than the range within which it was sensibly elastic, after being subjected to a great number of reversals. Hence it was inferred that the safe range of loading of a part, having a scratched or

VOL. CCXXI.—A 587. 2 A [Published October 21, 1920.

A, A. Griffith (1893-1963),
<u>The phenomena of rupture and flow in solids</u>.
1921 … the ground-braking paper of the fundamentals of fracture mechanics.

In this paper about the criterium of cracking in brittle materials, Griffith proposed his theory, described his experiments, speculated about molecular basis and size effects.

Fracture mechanics in small dimensions

Microcracks limit the mechanical robustness of materials and systems

- **Design of new structural and functional materials has to include their mechanical robustness**
- **Knowledge of fundamental mechanisms of materials ageing and device degradation needed**
- **Particularly understanding crack evolution on multi-scale, including micro- and nanoscale**

➔ Fracture mechanics in small dimensions has become an important area of fundamental research.

Need to understand toughening mechanisms in constrained materials at small scale:

- **Multi-scale modeling and multi-scale materials characterization (including nano-scale)**
- ***(in-situ)* micromechanical experiments and simultaneous high-resolution 3D imaging**

Thank you !

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