

# Micromechanical characterization of SiC-SiC fiber composite for accident tolerant fuel cladding applications

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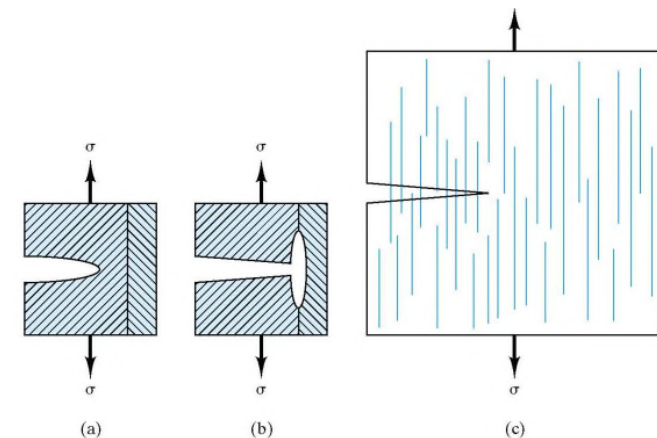
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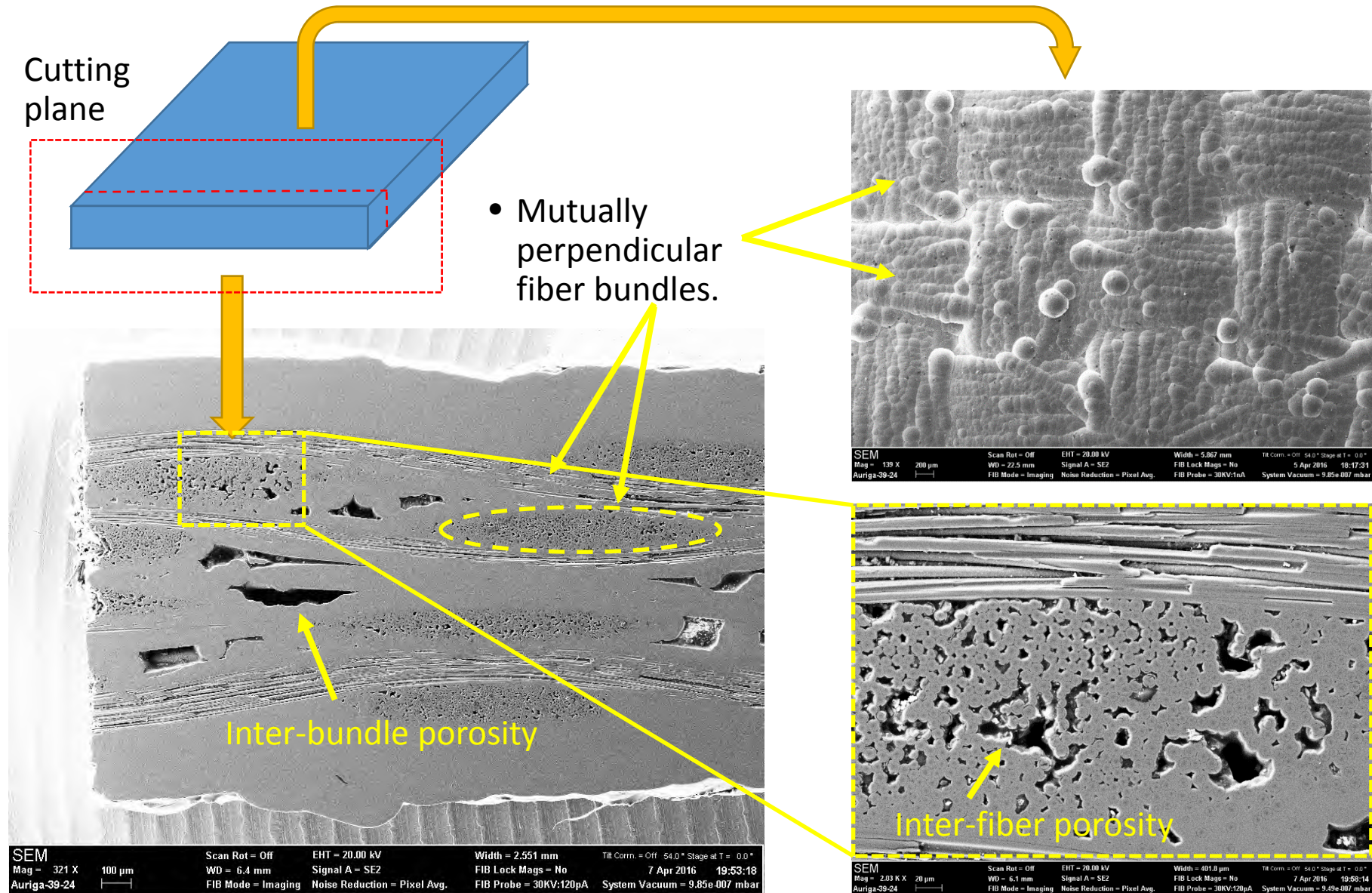
# Background

- Fuel cladding → zirconium alloys (“zircaloy”).
- **Problem** → loss-of-coolant accidents (LOCAs):
  - Zirconium reacts with water steam → oxidizes, producing hydrogen.
  - Danger of explosion of the hydrogen-oxygen mixture.
- Concept of **accident-tolerant fuel (ATF)** → SiC as a cladding material.
  - High-temperature strength.
  - Stability under irradiation.
  - **Reduced oxidation under accident conditions.**
- SiC → ceramics → brittle → use in the form of a composite.
- Improved toughness by introduction of interphases:
  - SiC fibres – *commercially available, Tyranno (Ube Industries)*.
  - Coated with pyrolytic carbon, weaved into a fabric-like structure – *General Atomics*.
  - SiC matrix grown on fibres by **chemical vapour infiltration (CVI)** method – *General Atomics*.



- US Department of Energy Nuclear Energy University Programs (NEUP):  
*Developing a macro-scale SiC-cladding behaviour model based on localized mechanical and thermal property evaluation on pre- and post-irradiation SiC-SiC composites.*
- Goal – develop a **macroscopic** final element model based on **microscopic** properties.
- Measurements of local properties → matrix, fibers and interphases → correlated with microstructure.
- **Micromechanical study:**
  - Microcantilever testing;
  - Nanoindentation;
  - Fiber push-out.
- **Microstructural study:**
  - Scanning electron microscopy (SEM);
  - Transmission electron microscopy (TEM);
  - Energy-dispersive X-ray spectroscopy (EDX);
  - Electron backscatter diffraction (EBSD);
  - Transmission Kikuchi diffraction (TKD);
  - Selected area diffraction (SAED).

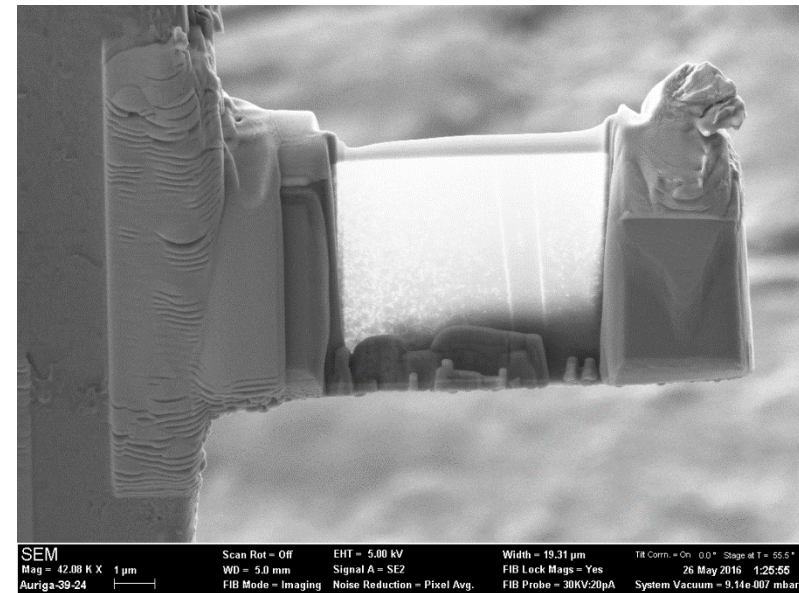
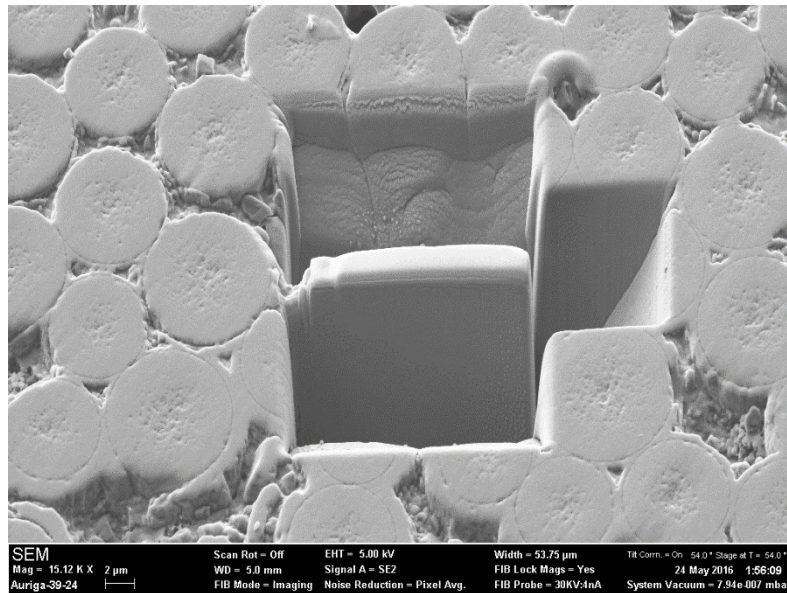
# Microstructure





# Microstructure

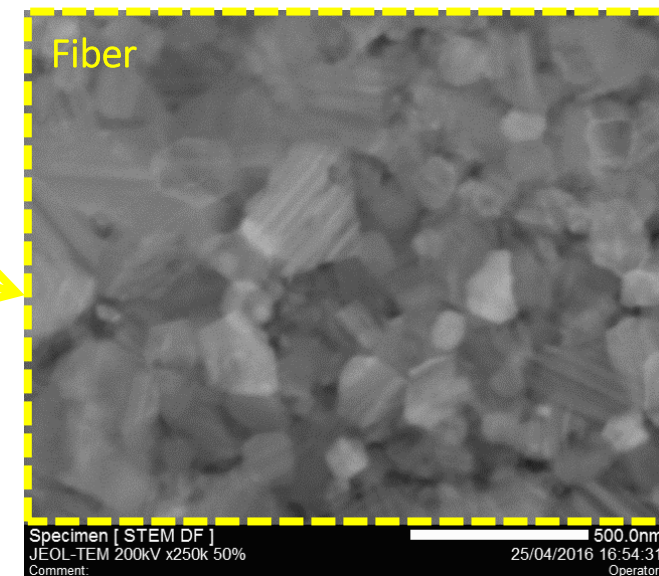
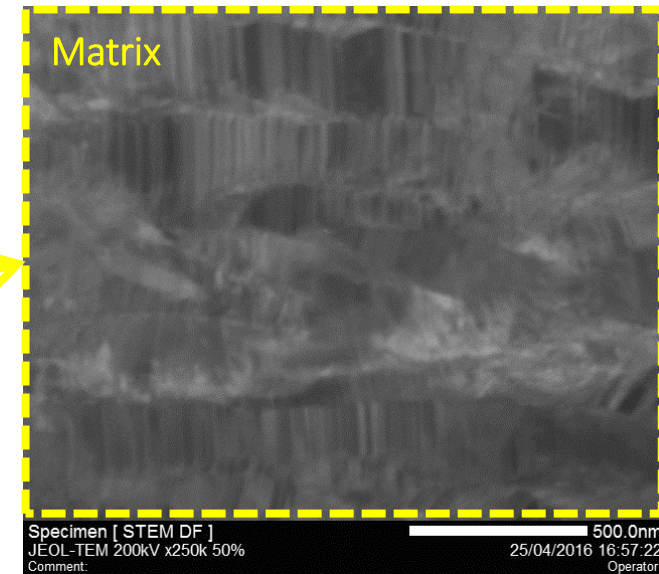
- Microstructure studied by TEM.
- FIB lift-out samples.



- Study of local microstructure  $\rightarrow$  interphases, fibers, matrix.

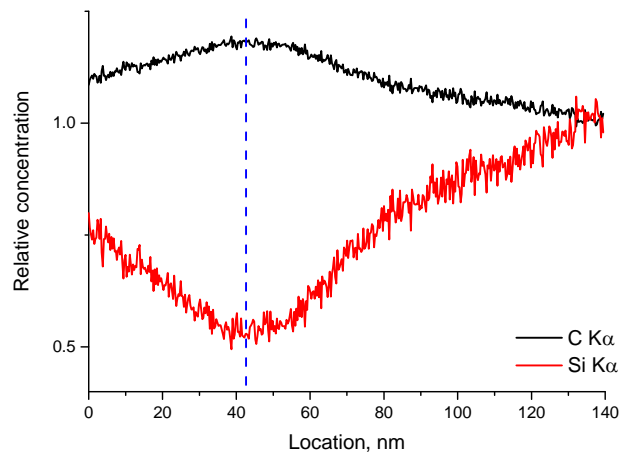
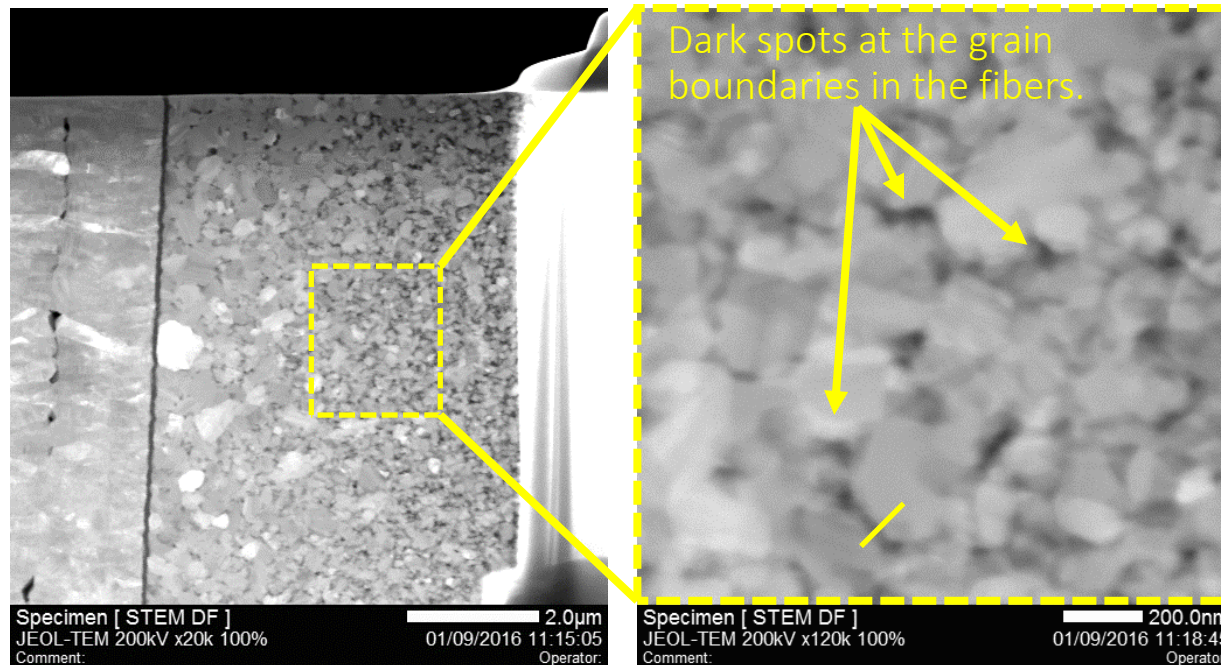
# Microstructure

- Microstructure studied by TEM.
- FIB lift-out samples.



- Elongated grains in the matrix → radial growth.
- Equiaxed grains in the fiber.
- Submicron-size porosity between the fibers.

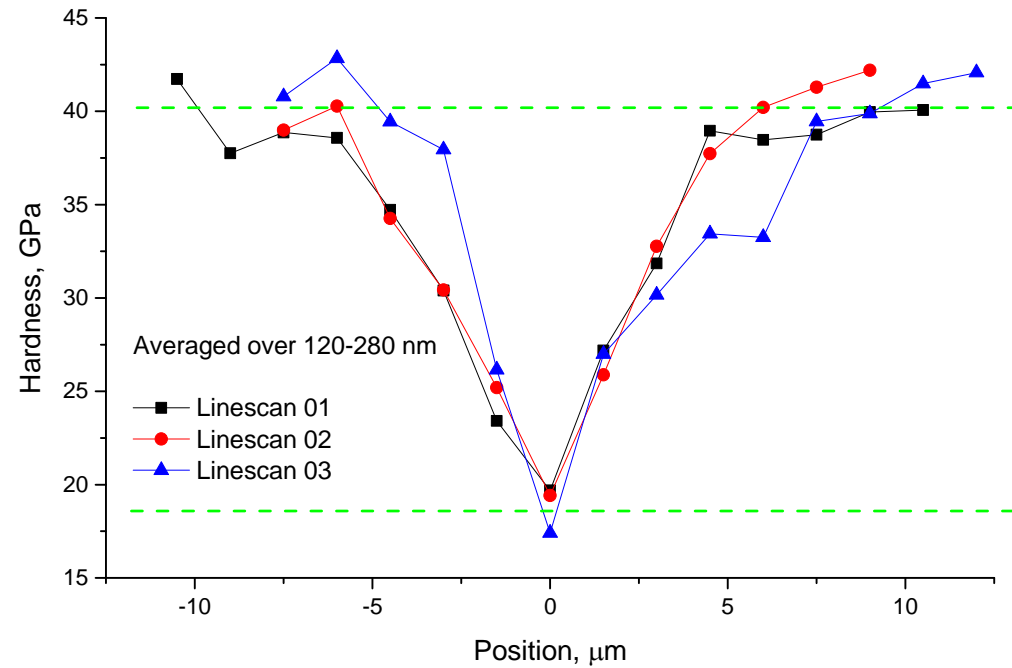
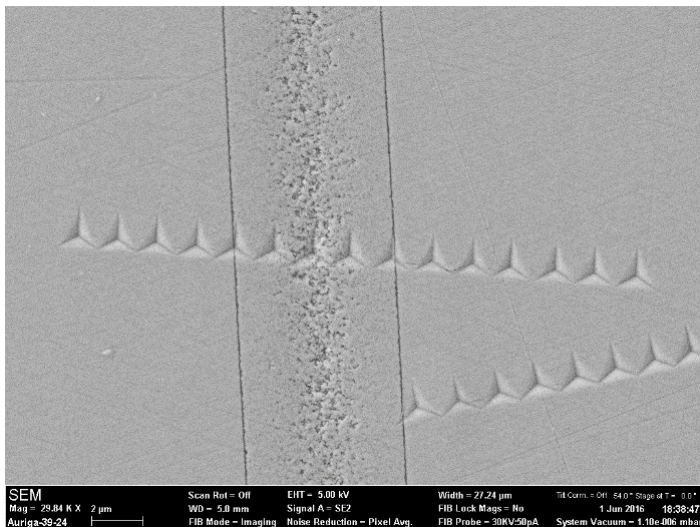
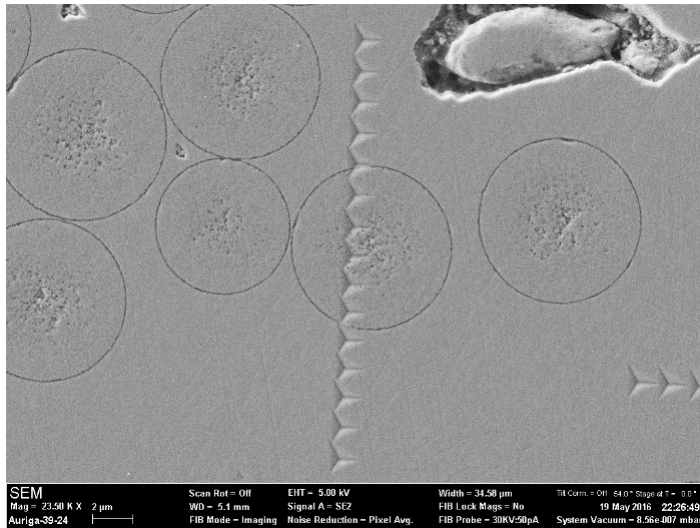
# Microstructure



- Dark areas at the grain boundaries in the fiber:
- *Depleted of Si.*
- *Enriched in C.*
- Probably graphite particles decorating the grains within the fibre material.



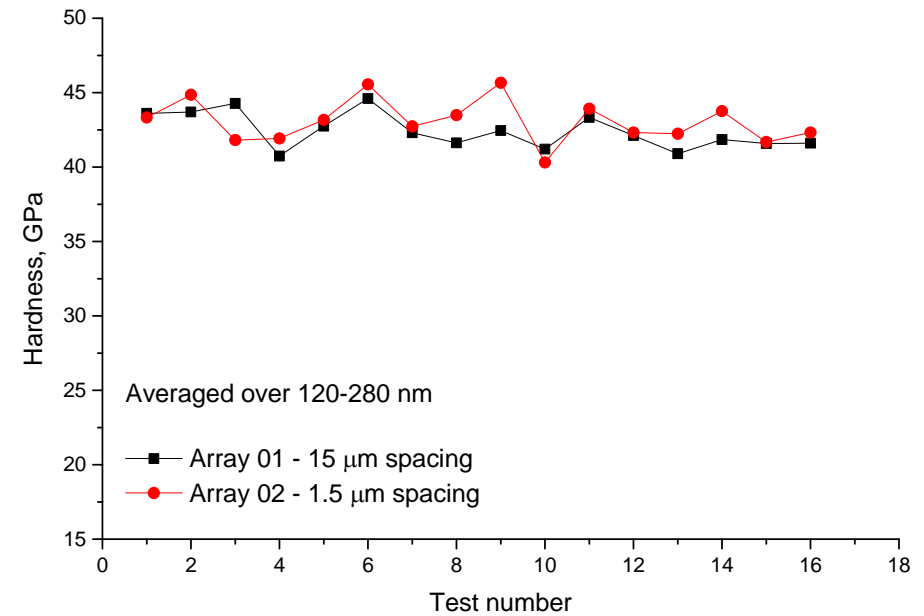
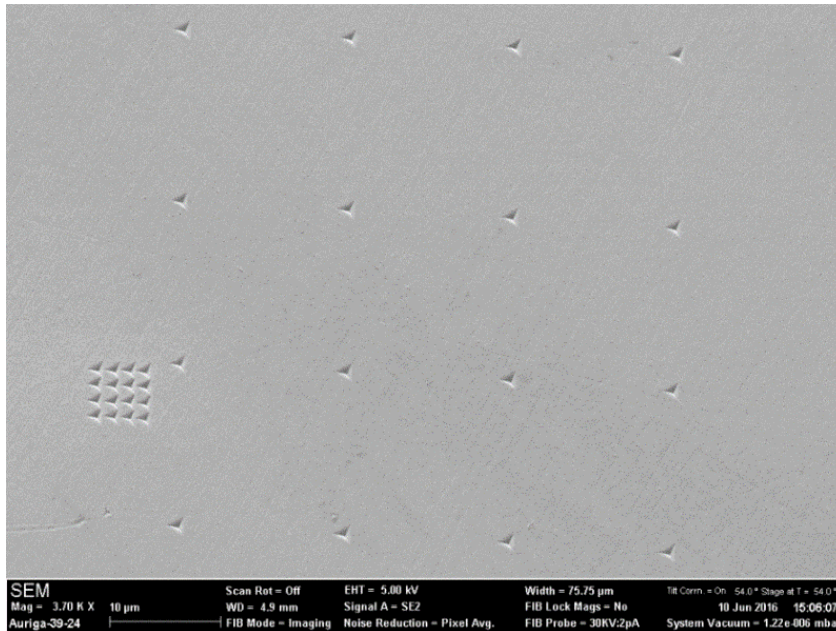
# Nanoindentation



- Non-uniform hardness within the fiber.
- Correlated with the presence of excess C.
- Higher C content  $\rightarrow$  lower hardness.

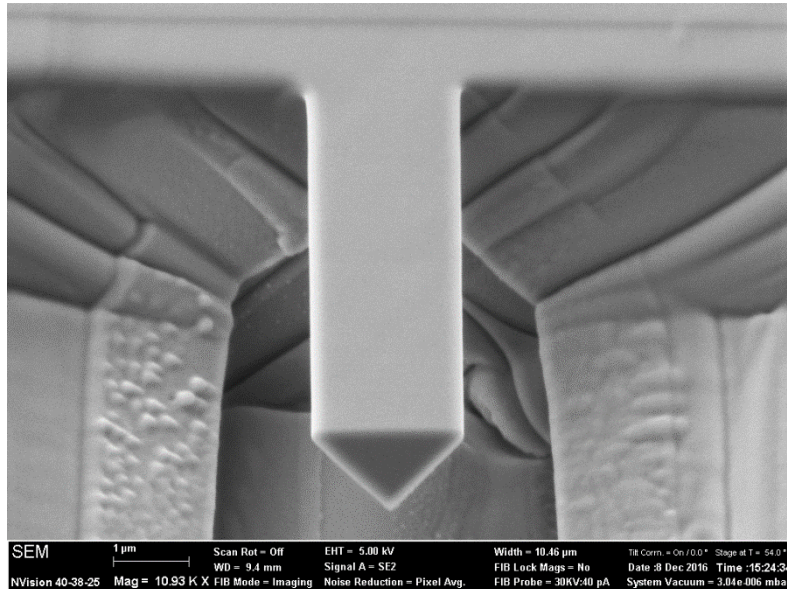


# Nanoindentation

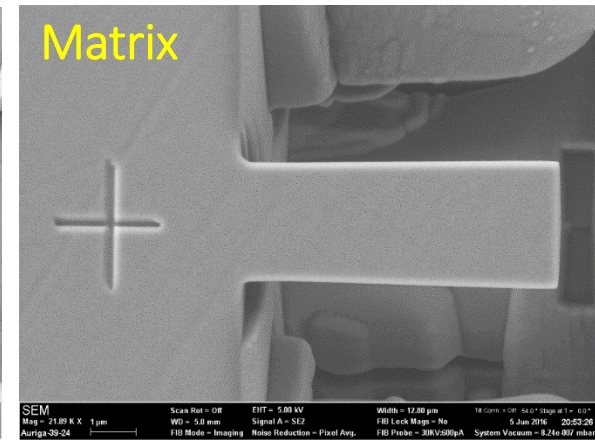
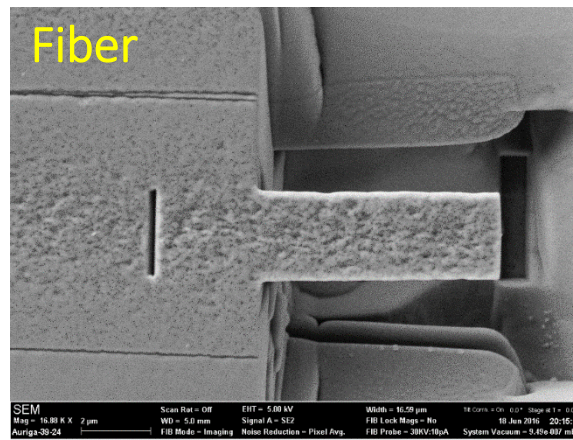
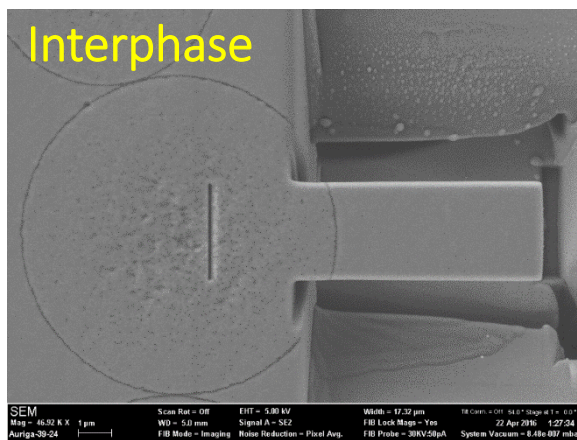


- No difference in hardness values regardless of inter-indent distance.
- Very constrained plastic zone around indents.

# Microcantilever testing

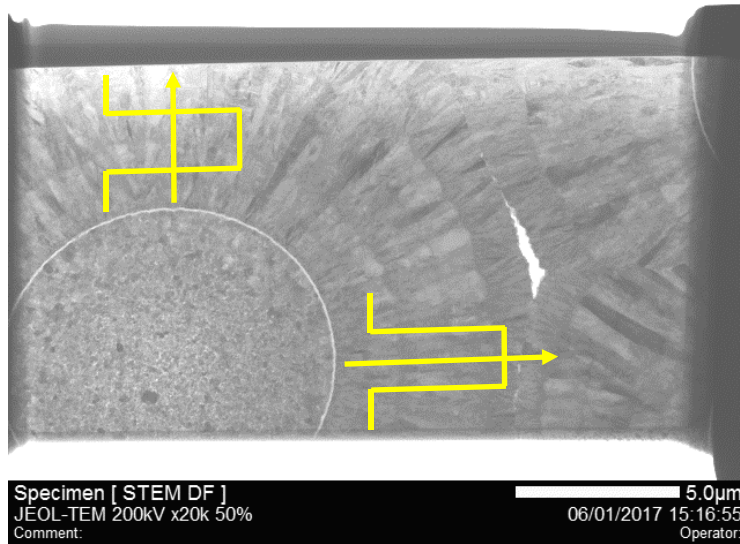


- FIB-machined cantilevers.
- Triangular cross-section.
- Load applied with nanoindenter.
- Cantilevers at the interphase.
- Cantilevers in the fibers.
- Cantilevers in the matrix.

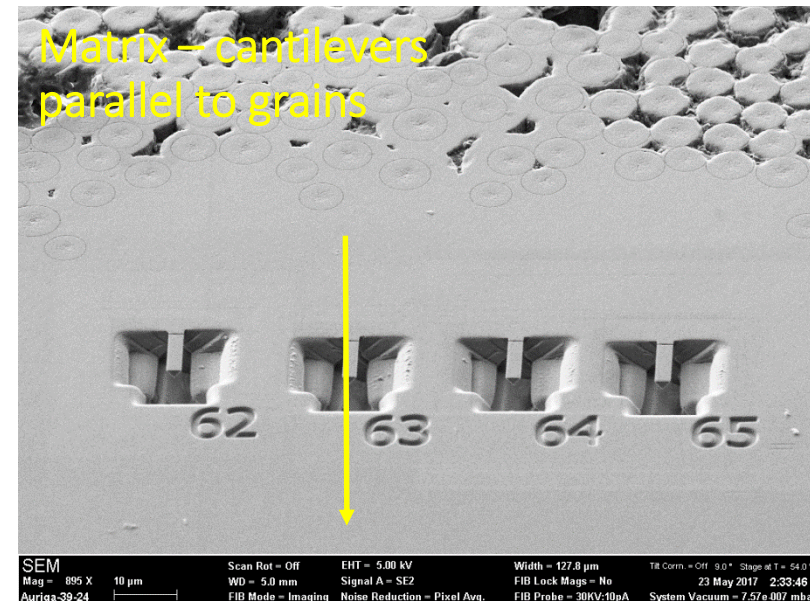
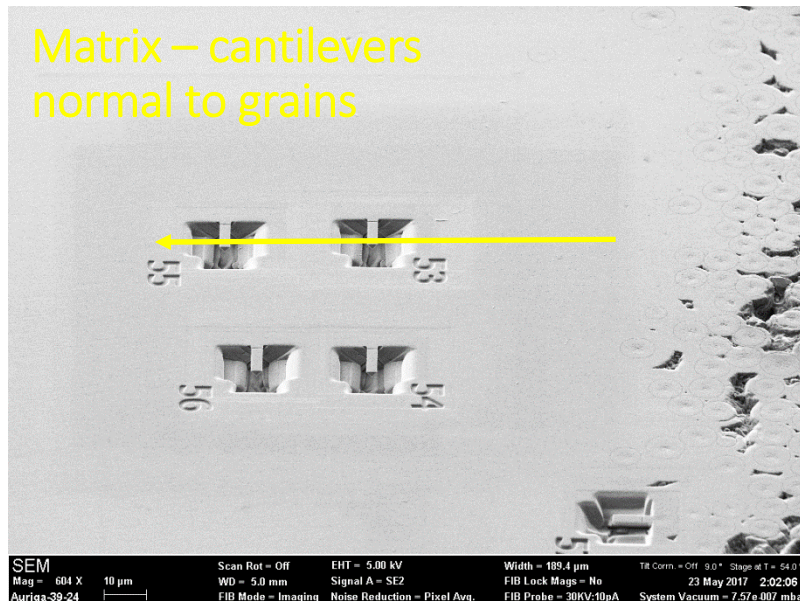




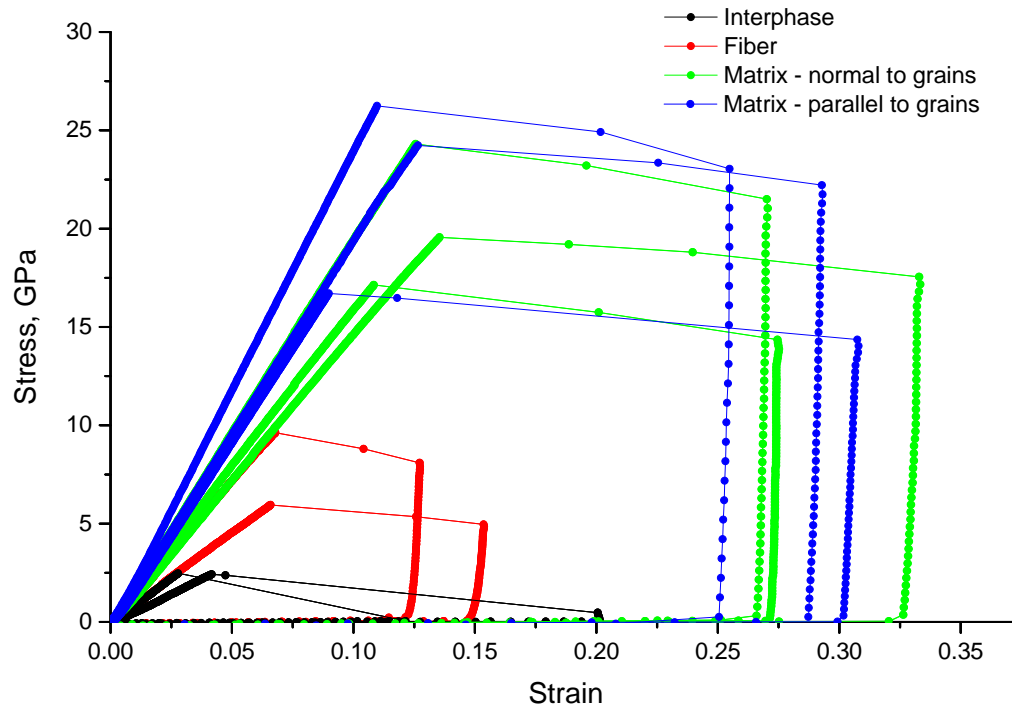
# Microcantilever testing



- Elongated grains in the matrix.
- Cantilevers in the matrix can be oriented parallel or perpendicular to the direction of grain growth.



# Microcantilever testing

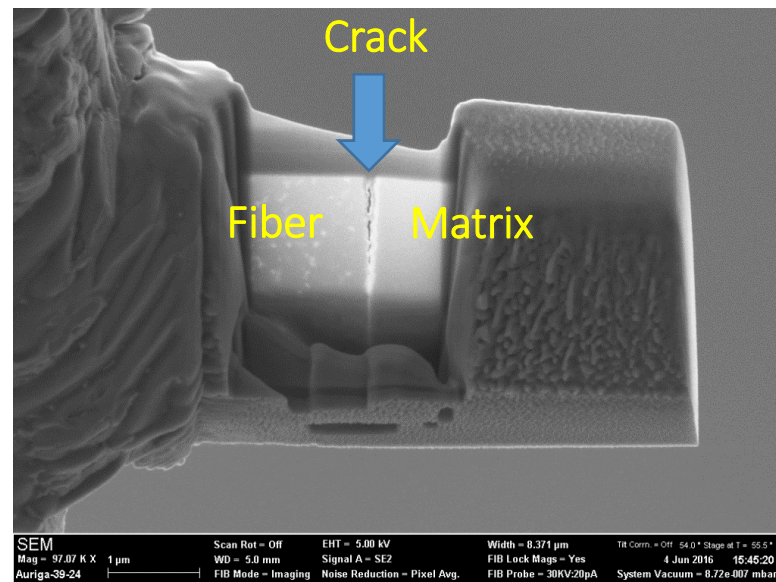
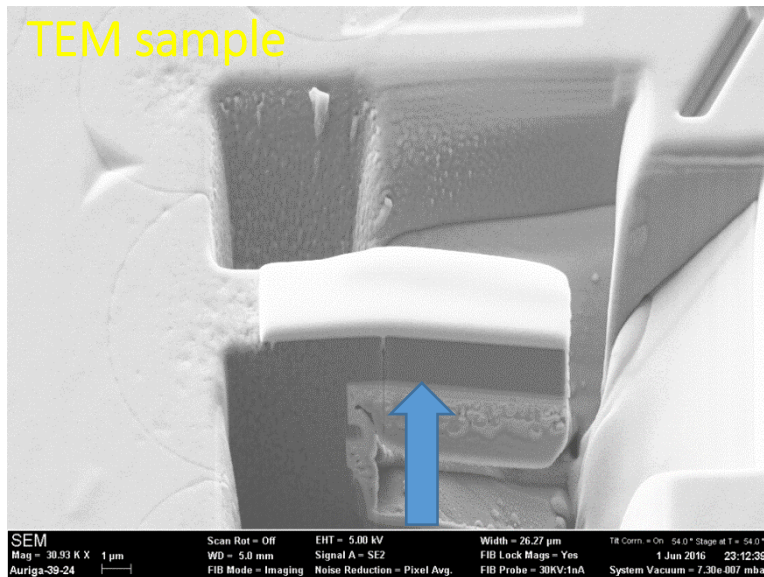
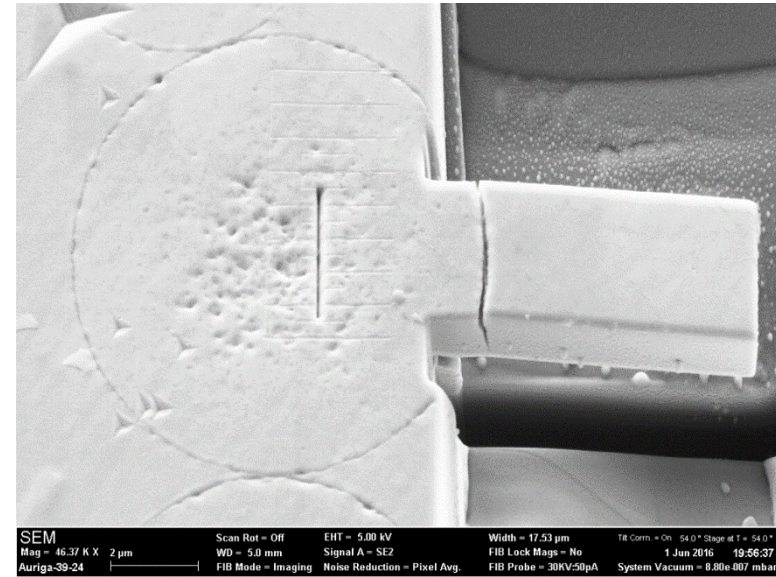
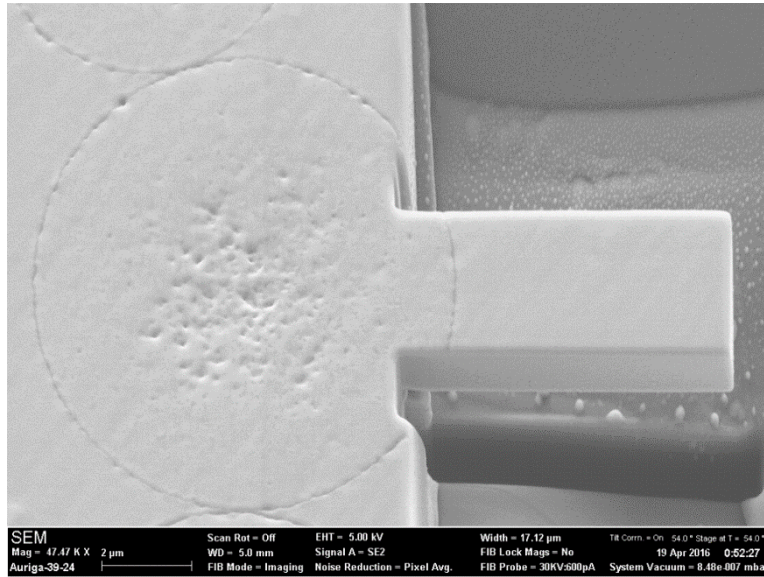


- Load-displacement curves measured.
- Converted to stress-strain using simple beam theory.
- Interphase:  
Fracture stress – 2.3 GPa;  
Strain at fracture – 3.5%;
- Fiber:  
Fracture stress – 8 GPa;  
Strain at fracture – 6.7%;
- Matrix:  
Fracture stress – 21 GPa;  
Strain at fracture – 13%.

- Interphases are weak spots.
- Fibers intermediate → weaker than matrix due to excess C?
- Matrix the strongest → no systematic difference for different orientations.

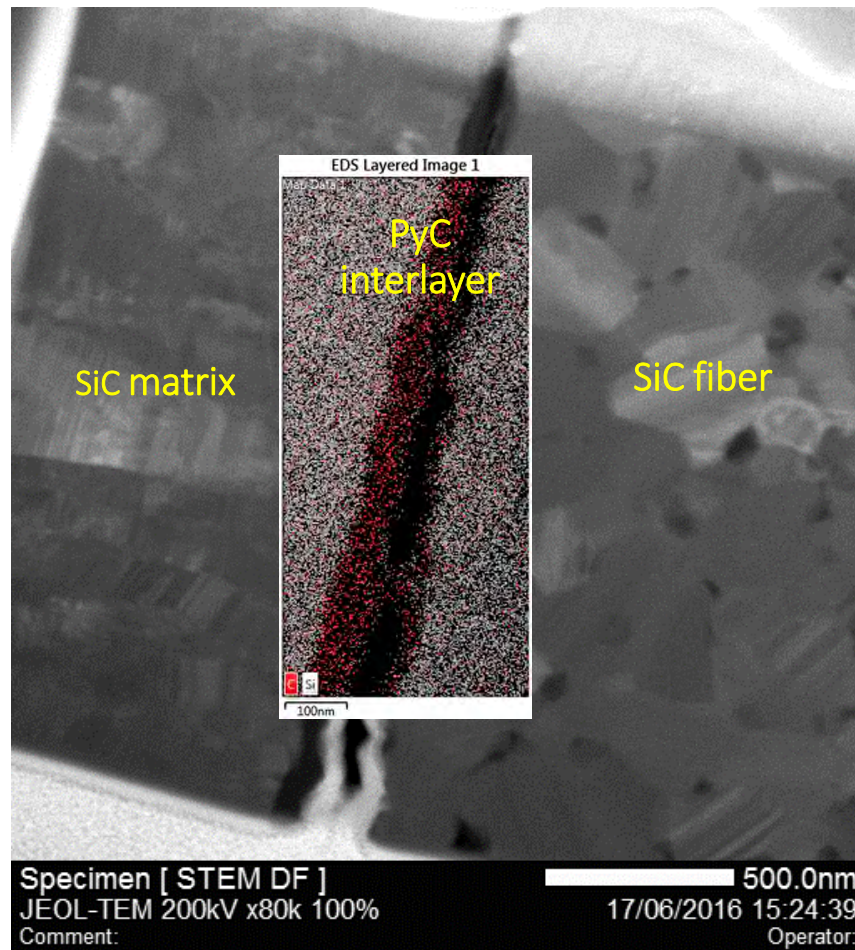


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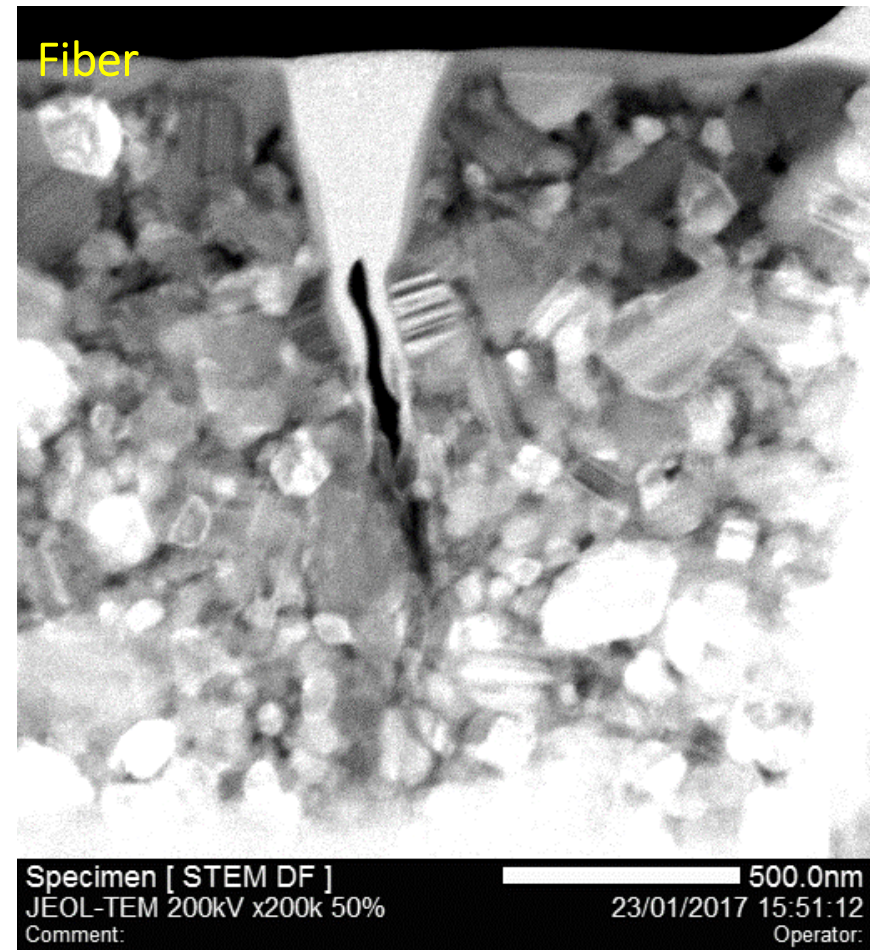




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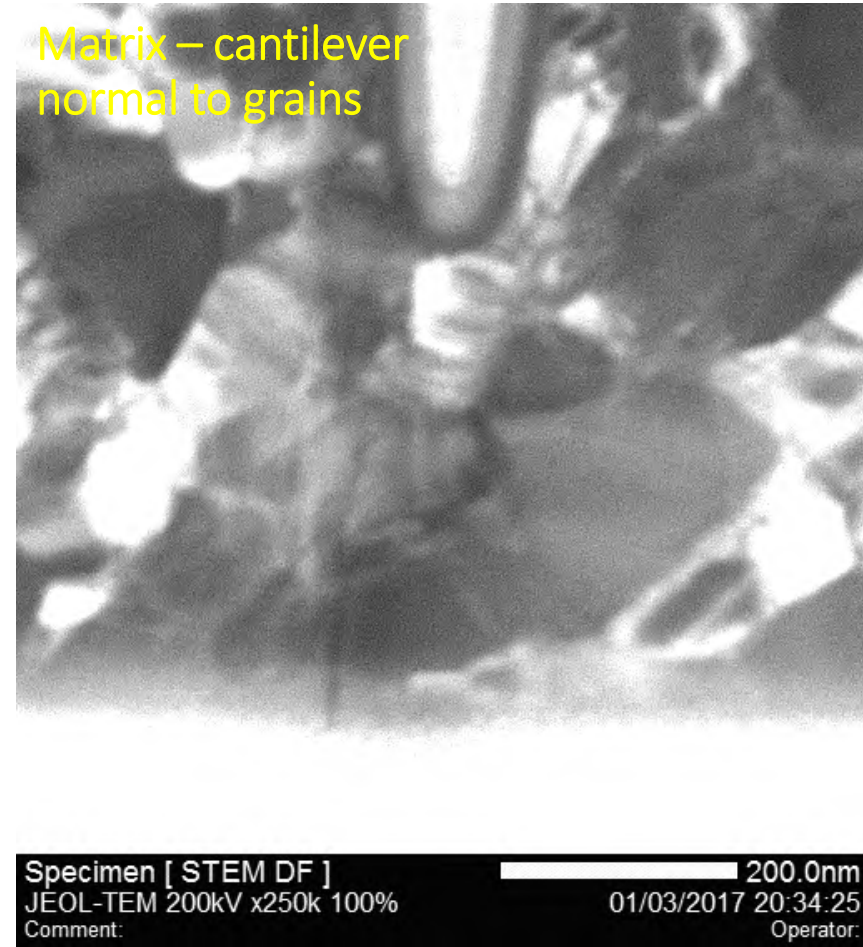
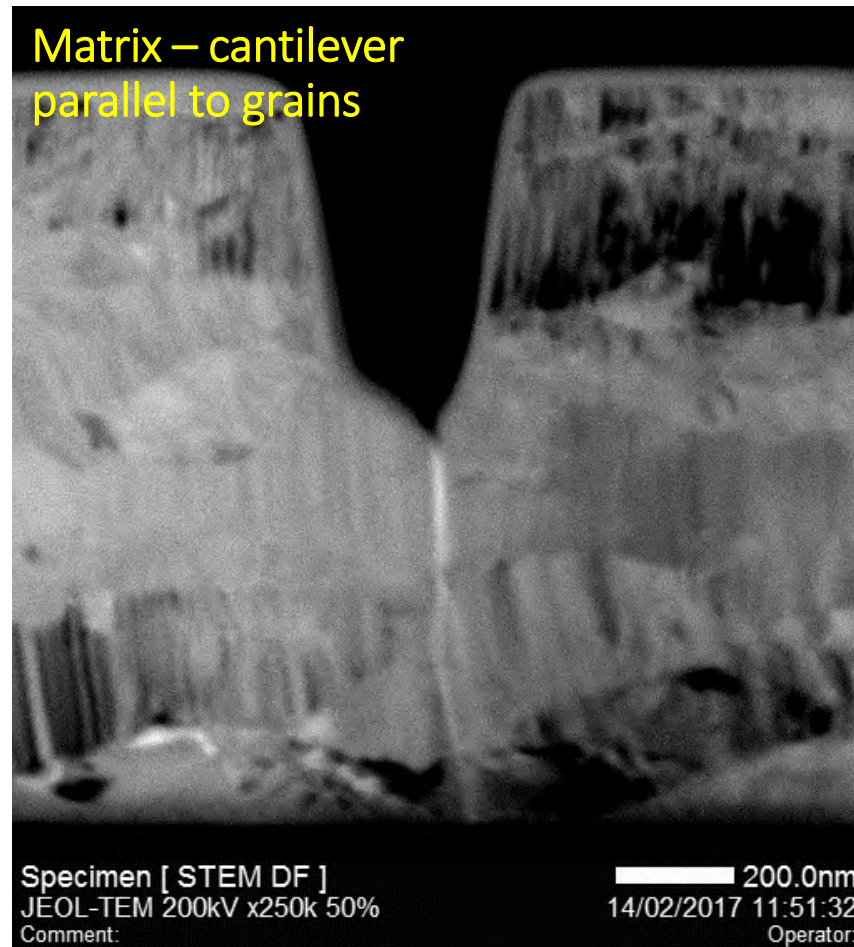


Fracture close to fiber-interlayer boundary.



Transgranular and intergranular fracture in the fiber.

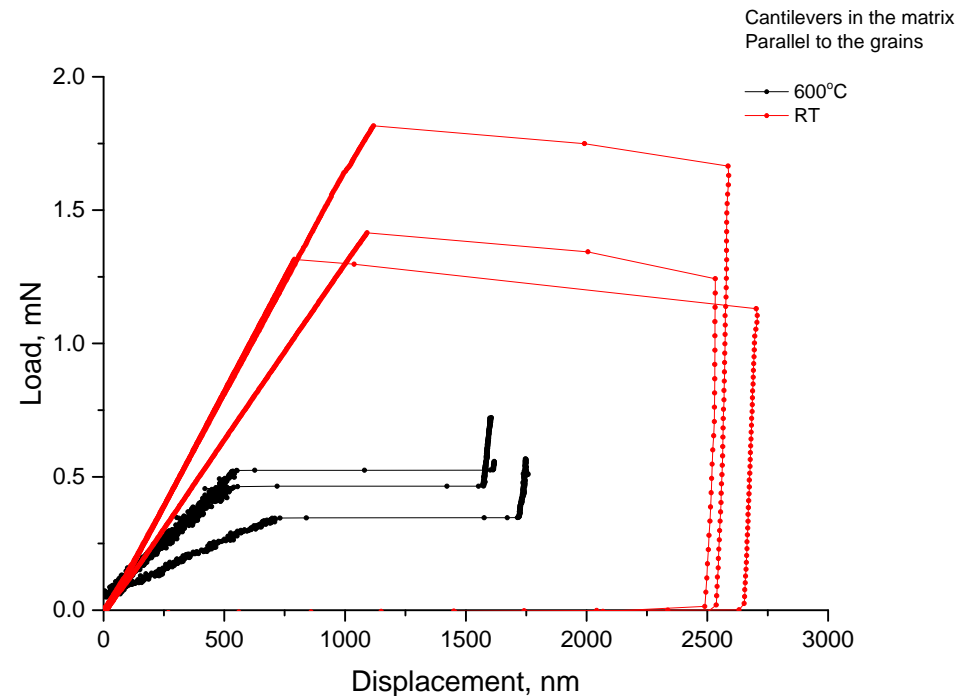
# Microcantilever testing



Transgranular fracture in the matrix.

# Preliminary high-temperature data

- Hot nanoindenter – vacuum tests up to 700°C (possible extension to 900°C).



- At 600°C – decrease of the matrix fracture load by a factor of ~3 compared to RT.
- Systematic study of temperature dependence, for fibers and interphases, underway.



# Summary and outlook

- Complex microstructure:
  - Matrix material – highly elongated grains, multi-level hierarchical structure.
  - Fiber material – symmetrical grains, with carbon decorating grain boundaries.
  - Growth of matrix creates submicron-sized porosity.
- Micromechanical testing:
  - Cantilever fracture – weak interphases, strong matrix, intermediate fiber.
  - Fracture close to fiber-interlayer boundary.
  - Nanoindentation – fibers softer than matrix, correlates with the presence of carbon.
- Plans:
  - Micromechanical testing at elevated temperature – hot nanoindenter.
  - Development of push-out testing.
  - Orientation mapping.
  - Micromechanical testing on irradiated samples (UC Berkeley).