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

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Background

- Fuel cladding \(\rightarrow\) zirconium alloys ("zircaloy").

**Problem** \(\rightarrow\) loss-of-coolant accidents (LOCAs):
  - Zirconium reacts with water steam \(\rightarrow\) oxidizes, producing hydrogen.
  - Danger of explosion of the hydrogen-oxygen mixture.

- Concept of **accident-tolerant fuel (ATF)** \(\rightarrow\) SiC as a cladding material.
  - High-temperature strength.
  - Stability under irradiation.
  - *Reduced oxidation under accident conditions.*

- SiC \(\rightarrow\) ceramics \(\rightarrow\) brittle \(\rightarrow\) 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*.
Background

• 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

- Mutually perpendicular fiber bundles.

Cutting plane

Inter-bundle porosity

Inter-fiber porosity
Microstructure

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

- Study of local microstructure ➔ interphases, fibers, matrix.
• 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.
Dark spots at the grain boundaries in the fibers.

- 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.
Microcantilever testing

TEM sample

Crack

Fiber

Matrix
Microcantilever testing

Fracture close to fiber-interlayer boundary. Transgranular and intergranular fracture in the fiber.
Microcantilever testing

Matrix – cantilever parallel to grains

Matrix – cantilever normal to grains

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).