



DE LA RECHERCHE À L'INDUSTRIE

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# HRTEM INVESTIGATION OF LOCAL DAMAGE MECHANISMS AT FIBER/MATRIX INTERFACE OF SiC/SiC COMPOSITES

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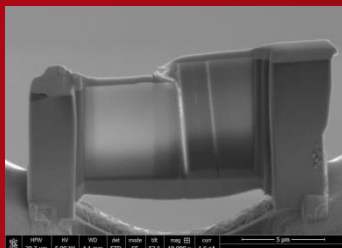
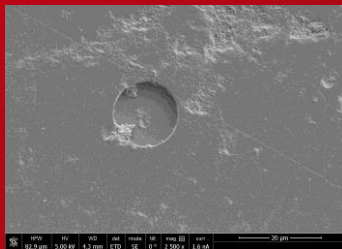
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HTCMC-9 | H2 Symposium

**SiC/SiC and C/C composite Materials  
Interfaces and Interphases in CMC's**

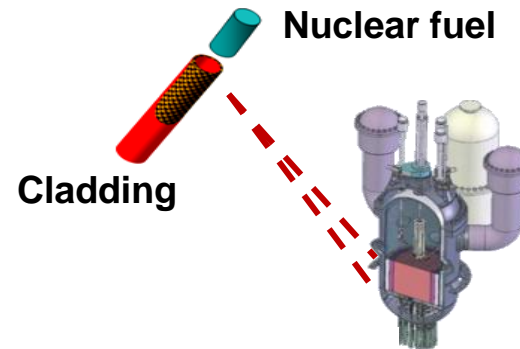
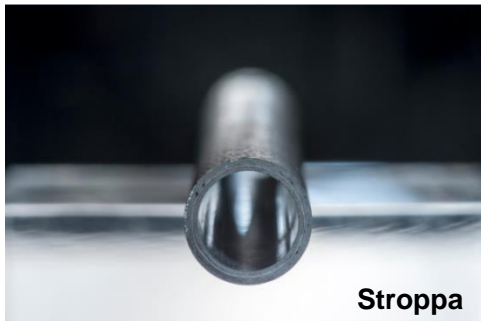
28 JUNE 2016



- Development of innovative materials for nuclear power plants



**Ceramic matrix composite : An alternative to metallic alloys**



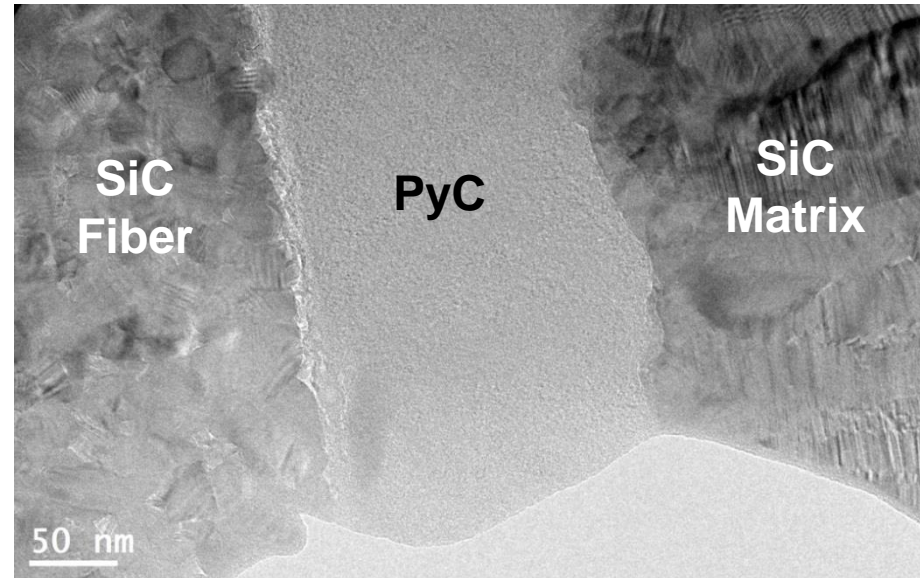
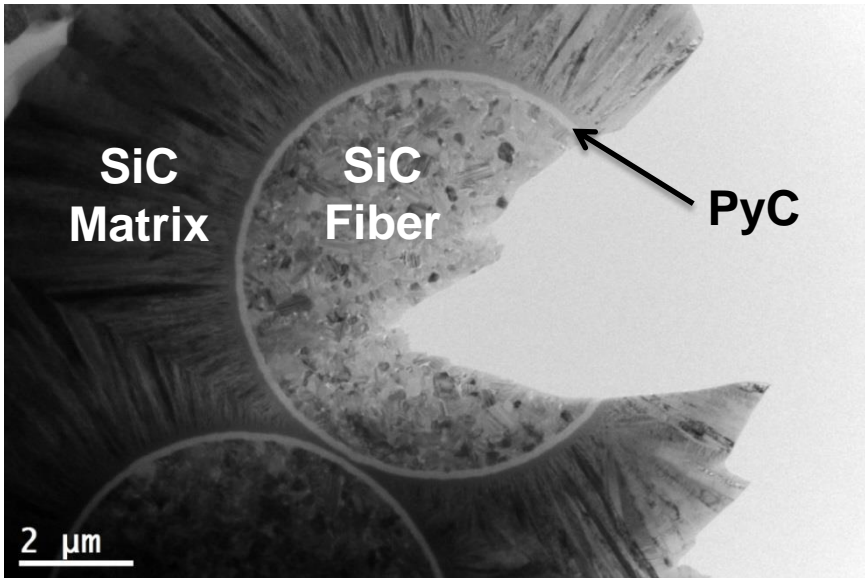
- Advantages of SiC/SiC composites for nuclear applications :
  - Refractoriness
  - Radiation tolerance and neutron transparency
  - Good microstructural stability under high neutron irradiation energy

**Improvement of SiC/SiC composites properties to use them in high temperature applications (nuclear, aeronautics, aerospace industries) :  
Thermal conductivity + Damage tolerance**

1. Studied materials : SiC/SiC composites
2. Evaluation of interfacial properties
3. Study of local damage mechanisms
4. Additional investigations

## ■ Composition :

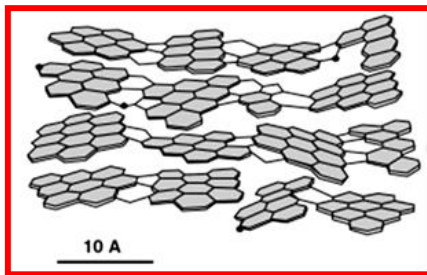
- Reinforcement : SiC fibers
- Matrix : SiC
- Interphase : Pyrocarbon (PyC)



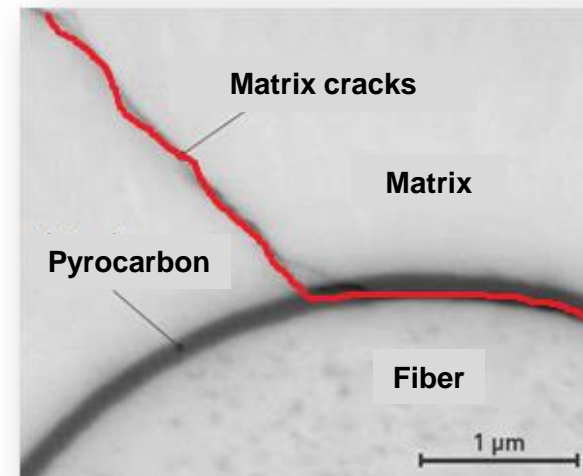
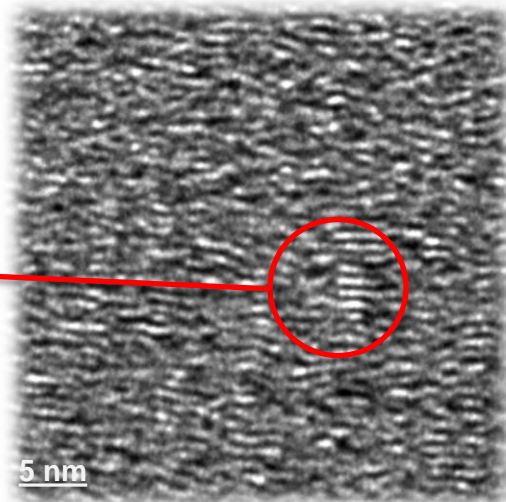
## ■ SiC :

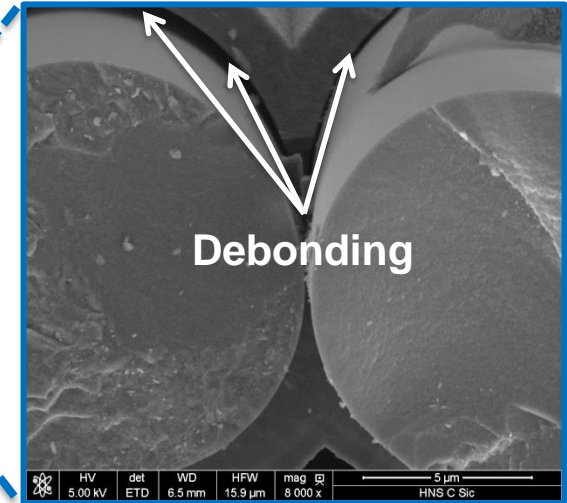
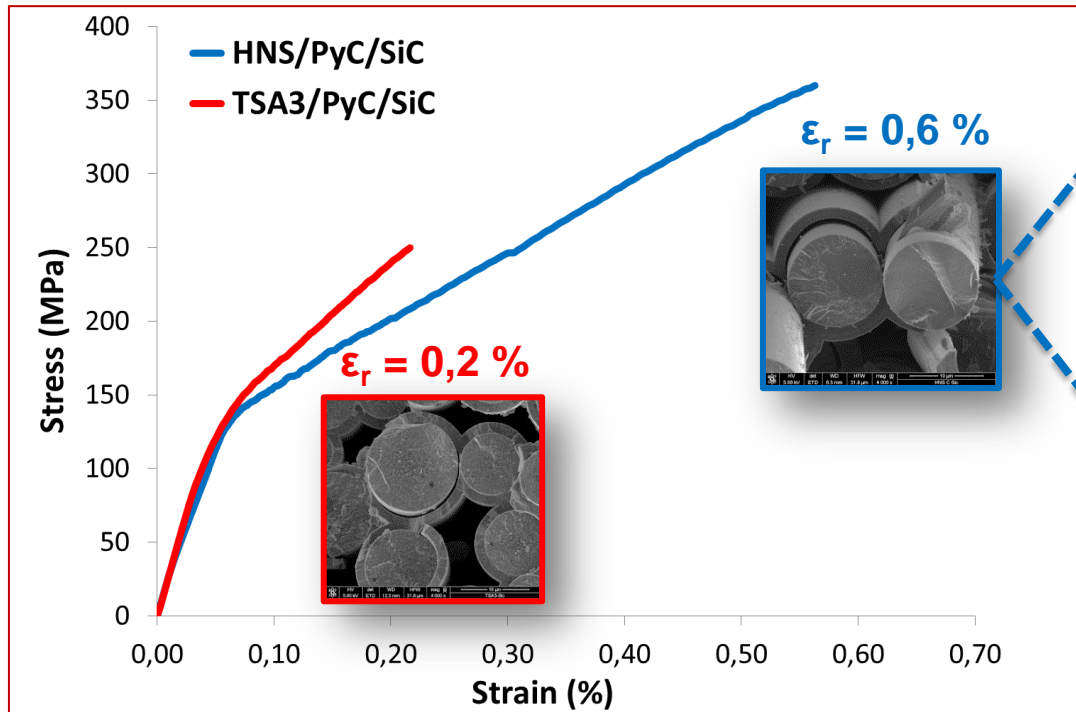
- Good thermal stability ( $T > 2000^{\circ}\text{C}$  and  $\alpha = 2-5 \cdot 10^{-6} \text{ K}^{-1}$ )
- Hardness ( $E = 350-450 \text{ GPa}$ )
- Brittleness ( $K_{Ic} = 2-3 \text{ MPa}\cdot\text{m}^{1/2}$ )

- SiC fibers : Hi-Nicalon S fibers (HNS) and Tyranno SA3 fibers (TSA3)
    - C/Si ratio  $\approx 1$
    - $\alpha \approx 4,5 \cdot 10^{-6} \text{ K}^{-1}$  at  $600^\circ\text{C}$
    - $E \approx 310 \text{ GPa}$  and  $\varepsilon_r \approx 0,70 \%$
- Similar properties**
- **Differences between the two fibers :** thermal conductivity
  - **Highly anisotropic pyrocarbon :** Parallel graphene sheets with random orientations in the (002) lattice plane of the carbon



A.Oberlin, 1984





**HNS/PyC/SiC composites**  
Fair damage tolerance

**TSA3/PyC/SiC composites**  
Poor damage tolerance

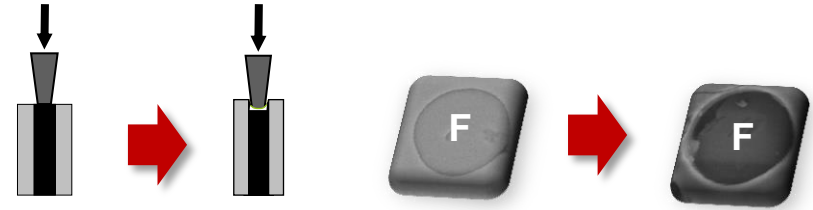
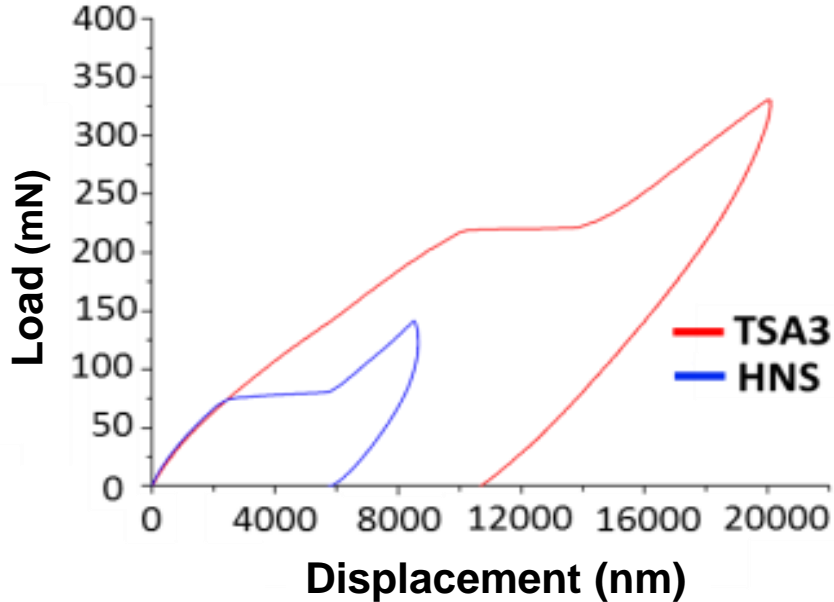


**Opposite mechanical behavior  
and fiber/matrix coupling mode**

- **Observation of the local damage mechanisms at fiber/matrix interface**
- **Understanding the phenomena affecting the interfacial properties of SiC/SiC composites**



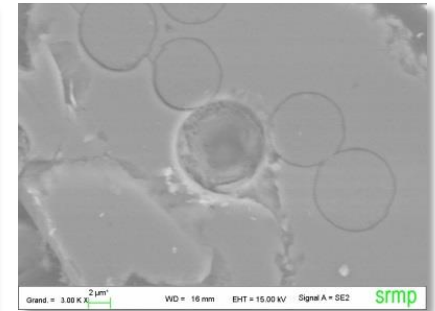
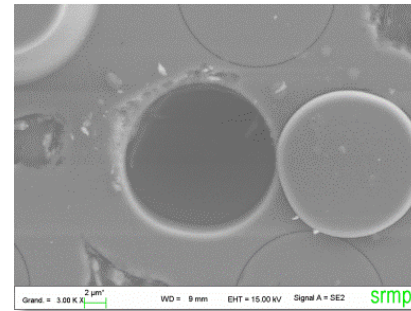
Push-out tests



Minicomposites

HNS/PyC/SiC

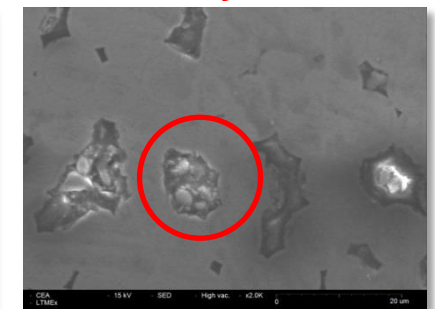
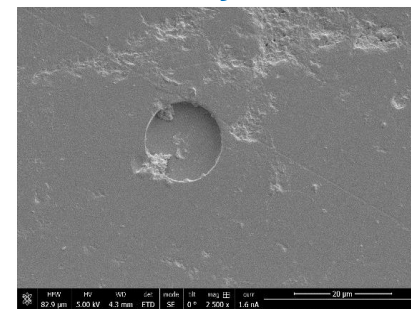
TSA3/PyC/SiC



Composites

HNS/PyC/SiC

TSA3/PyC/SiC



Decoherence normal stress :

$\sigma_d$  (HNS/PyC/SiC) = **770** MPa

$\sigma_d$  (TSA3/PyC/SiC) = **1370** MPa

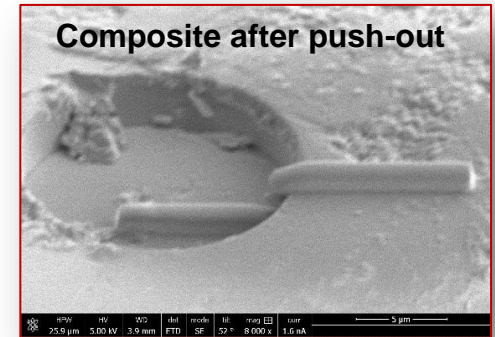
Interfacial shear stress :

$\tau_s$  (HNS/PyC/SiC) = **35** MPa

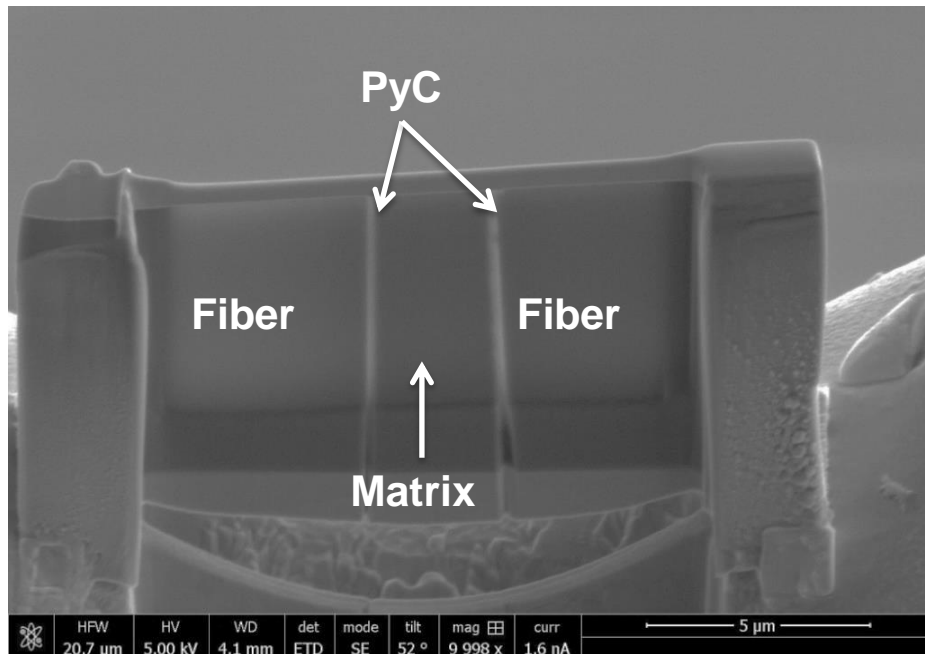
$\tau_s$  (TSA3/PyC/SiC) = **210** MPa

**TSA3/PyC/SiC samples :  
Strong fiber/matrix coupling**

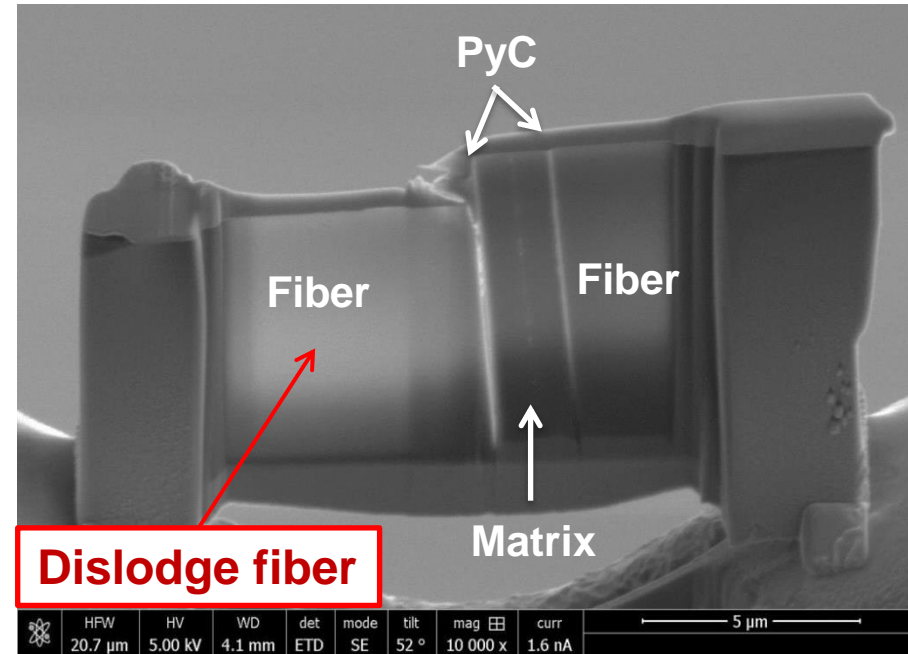
- Sample preparation : FIB (Focused Ion Beam)
  - Extraction of indented fibers after push-out test
  - Study of PyC microstructural organisation around a dislodge fiber



Before push-out



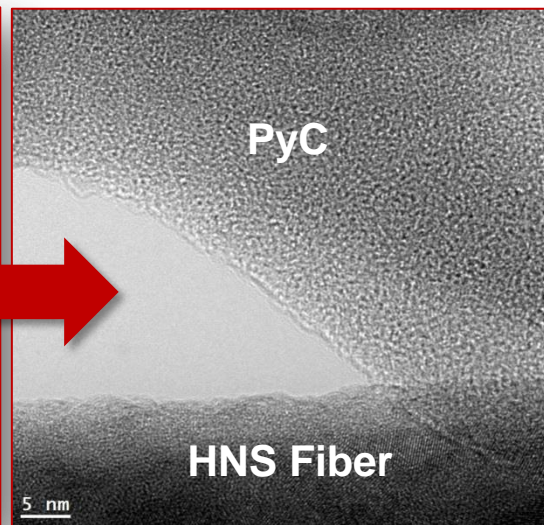
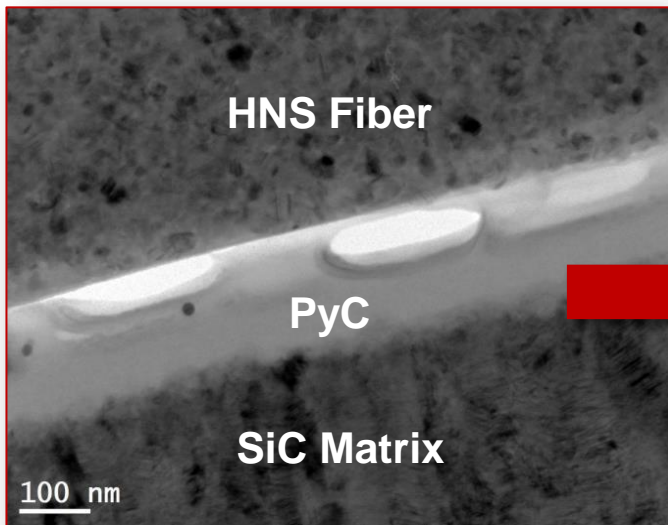
After push-out



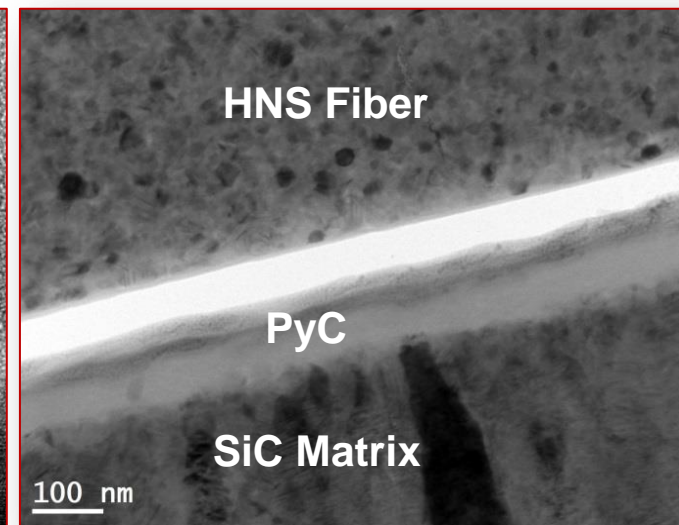


- HRTEM observations : **HNS/PyC/SiC**
  - Crack deflection occurs near the fiber/PyC region
  - Several local damage mechanisms are observed at the interface
  - The F/M debonding leads to the damage tolerance response of HNS/PyC/SiC

**Bridging**



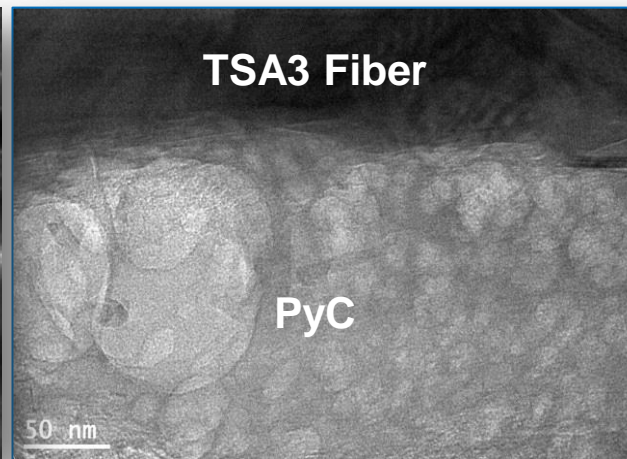
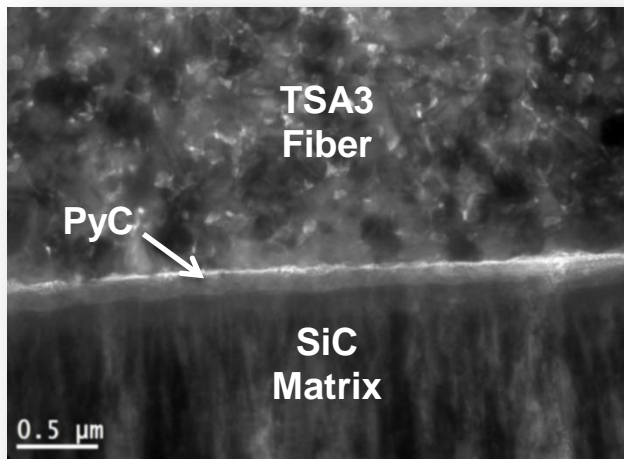
**Delamination**



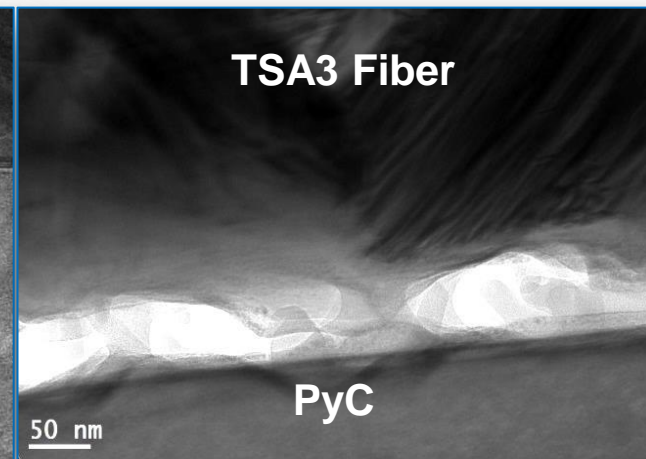
## ■ HRTEM observations : TSA3/PyC/SiC

- Different local damage mechanisms are observed
- A strong interfacial shear stress is required to lead to the break of the F/M bonds and to the fiber sliding
- Strong fiber/matrix coupling involves the brittle behavior of TSA3/PyC/SiC

### Damaged PyC

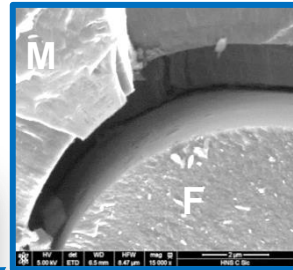


### Bridging

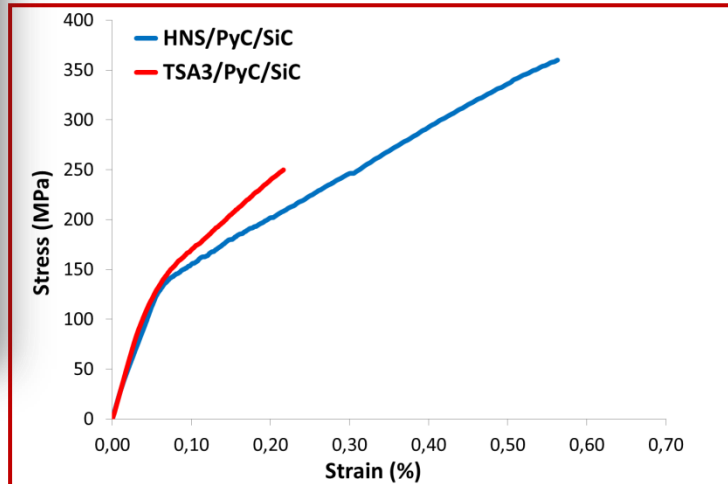
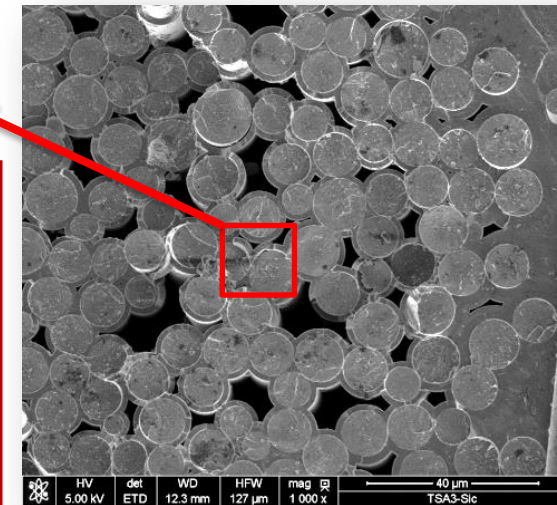
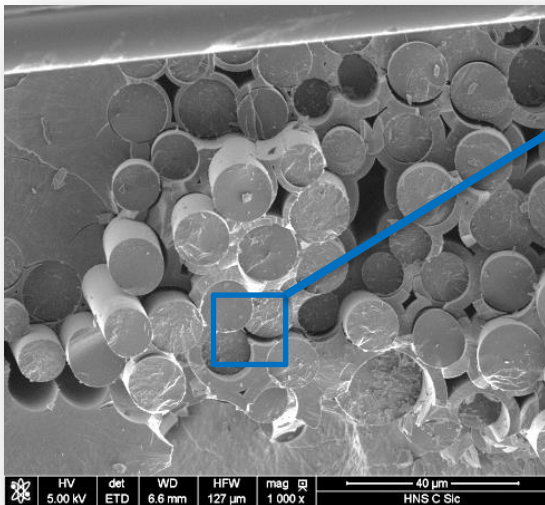
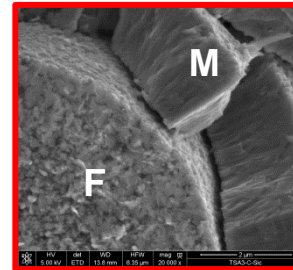


- Studies of fiber/matrix debonding and local damage mechanisms after tensile tests on SiC/SiC composites
- Confirmation of the results obtained by push-out tests

**HNS/PyC/SiC  
composites**

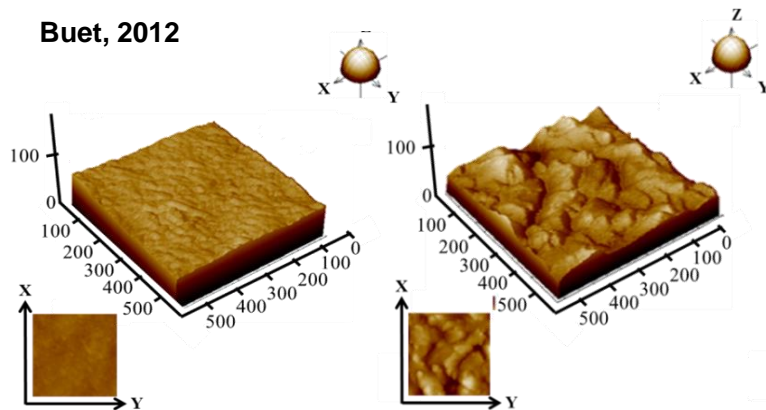


**TSA3/PyC/SiC  
composites**



- Study :
  - Comparison of two SiC/SiC composites
  - Identification of the impact of SiC fibers surface on the F/M bonding
  
- Difference between SiC/SiC composites :
  - Local damage mechanisms
  - Surface characteristics of SiC fibers

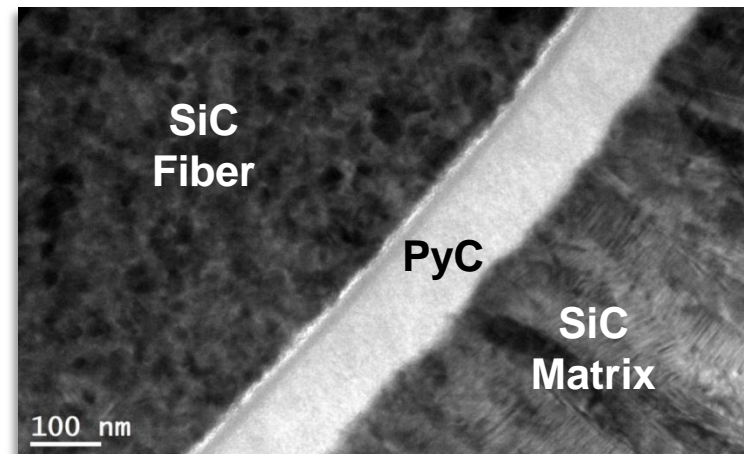
### Surface roughness



**HNS Fiber**

**TSA3 Fiber**

### Surface chemistry





- Two compatible SiC fibers for nuclear applications :
  - **HNS fibers reinforced composites** → Pseudo-ductile mechanical behavior
  - **TSA3 fibers reinforced composites** → Brittle mechanical behavior + better thermal conductivity + cost
  
- HRTEM investigation of local damage mechanisms at fiber/matrix interface of SiC/SiC composites after push-out test :
  - HNS/PyC/SiC : Good F/M coupling + Delamination + Bridging
  - TSA3/PyC/SiC : Too strong F/M coupling + Damaged PyC + Bridging
  
- Differences in mechanical behavior and in the bonding mode of the PyC to the fiber surface → Key role of the characteristics of SiC fibers surface

 **Modification of the F/M bonding and SiC fibers surface characteristics**  
**Improvement of the mechanical behavior of TSA3/PyC/SiC**



# Thank you for your attention

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