



SiC / SiC Lifetime Limiting Mechanisms

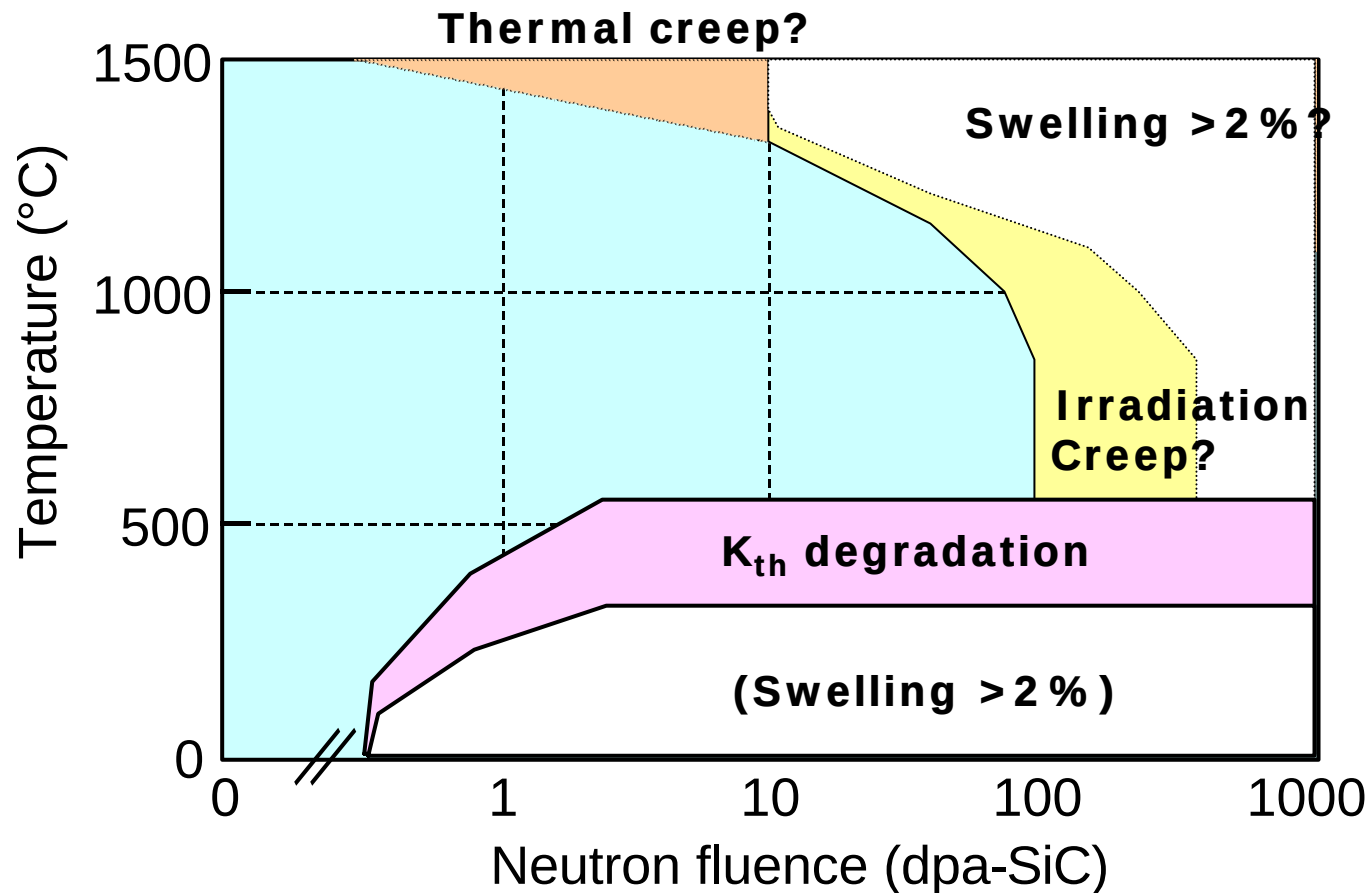
Y.Katoh, A.Kohyama and H.Kishimoto

IAE Kyoto University

T.Shibayama and H.Takahashi

CARET, Hokkaido University

Very Preliminary 'Design Window' – Do Not Refer!



- Issues have not yet well identified. Potential issues not indicated include: He-effect on strength and fracture toughness, fatigue, oxidation at very low P_{O_2} and P_{H_2O} , microstructural and microchemical instability @high-T.
- Irradiation database is definitely lacking. Data on older generation materials are not applicable.

Potential Lifetime Limiting Issues

■ 'Cold' issues:

- ◆ Fatigue
- ◆ Thermal creep
- ◆ Environmental degradation

■ Neutron-related issues

- ◆ Swelling
- ◆ Irradiation creep
- ◆ Strength/toughness degradation due to irradiation
- ◆
- ◆

Fatigue Lifetime

- ◆ The fatigue limit at room temperature and inert environment is much higher than the proportional limit, while that in an oxidation environment coincides with the proportional limit.
- ◆ Fatigue behavior depends on fiber architecture. The matrix cracking initiates typically on the large inter-bundle pores at a crossover point of 0/90 fiber bundles in plain-weave composites.

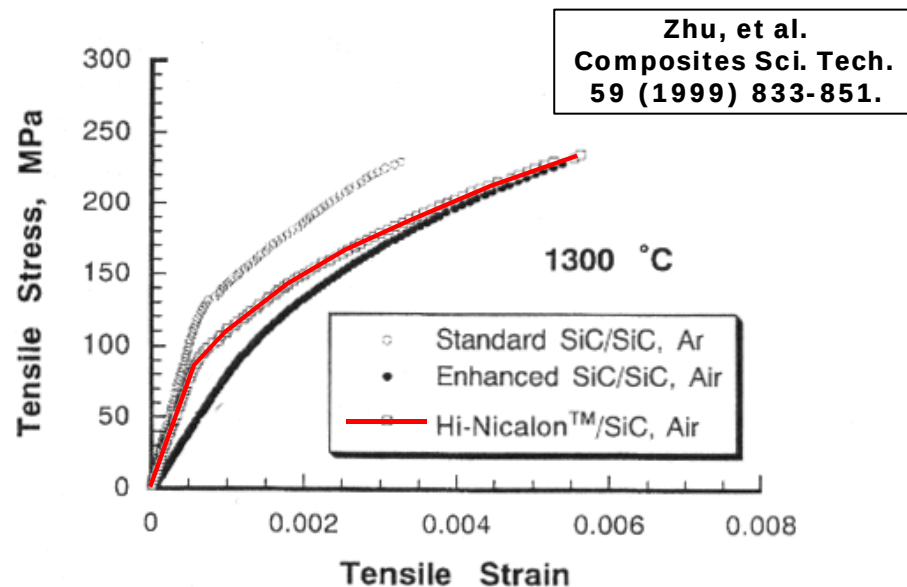


Fig. 4. Tensile stress versus strain in Hi-Nicalon™/SiC in air, enhanced SiC/SiC in air and standard SiC/SiC composites in argon at 1300°C.

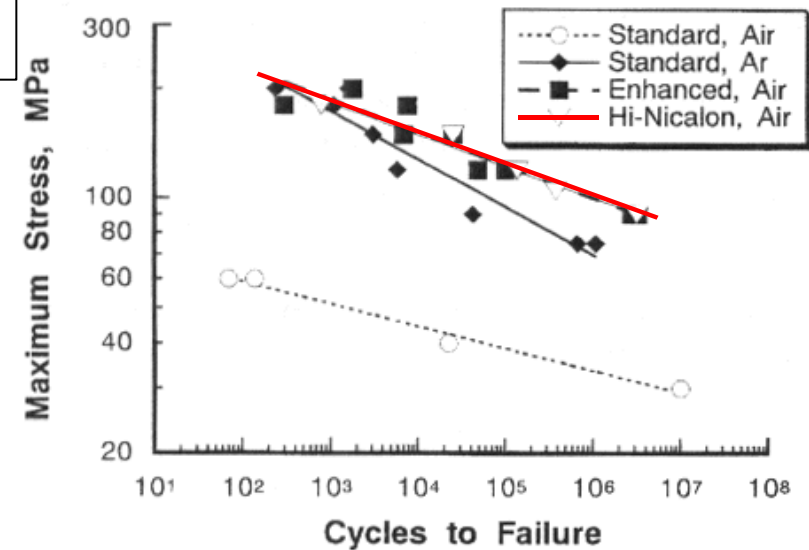


Fig. 9. Maximum stress versus cycles to failure of Hi-Nicalon™/SiC in air, enhanced SiC/SiC in air, and standard SiC/SiC in air and argon at 1300°C.

Fatigue Models for Composites

- ◆ Linear strength degradation theory (Broutman and Sahu):

$$s_{res} = s_U - (s_U - s_{max}) \cdot \frac{N}{N_f}$$

N_f = fatigue life at $s = s_{max}$

- ◆ Non-linear residual strength degradation theory (Yang and Liu):

$$s_{res}^c = s_U^c - s_0^c \cdot K \cdot (s_{max} - s_{min})^d \cdot N$$

c = damage development parameter

K, d = S - N parameters where $K \cdot (s_{max} - s_{min})^d \cdot N_f = 1$

- ◆ Global strain theory (O'Brien)

Combines residual strength degradation and modulus degradation

- ◆ Residual stiffness theory (Liu and Lessard)

$$\frac{E}{E_0} = 1 - KN^{2/(2+b)} s_{max}^{2b/(2+b)} \left(1 - \frac{s_{max}}{s_U} \right)$$

K, b = constants

- Applicability of the existing models needs to be studied.

On the Existing Data on Fatigue

- ◆ Existing fatigue data are mostly on Nicalon-CG and Hi-Nicalon composites. Large elastic modulus mismatch in those composites does not exist in advanced fiber composites.
- ◆ ‘Commercial grade argon’ environment in which most fatigue tests at around 1000°C have been carried out provides ‘passive oxidation’ condition for SiC, where silica glass coating decelerates crack propagation and interphase erosion.
- ◆ Cracking behavior largely depends on reinforcement architecture. Data on unidirectional and 2D composites may not be relevant to 3D composites.
- ◆ Wearing behavior of the F-M interface may substantially be influenced by the initial surface roughness of the fibers. Highly heat-resistant fibers are high temperature-treated and therefore have enhanced surface roughness.

Fatigue-induced Cracking in Plain-Weave SiC/SiC

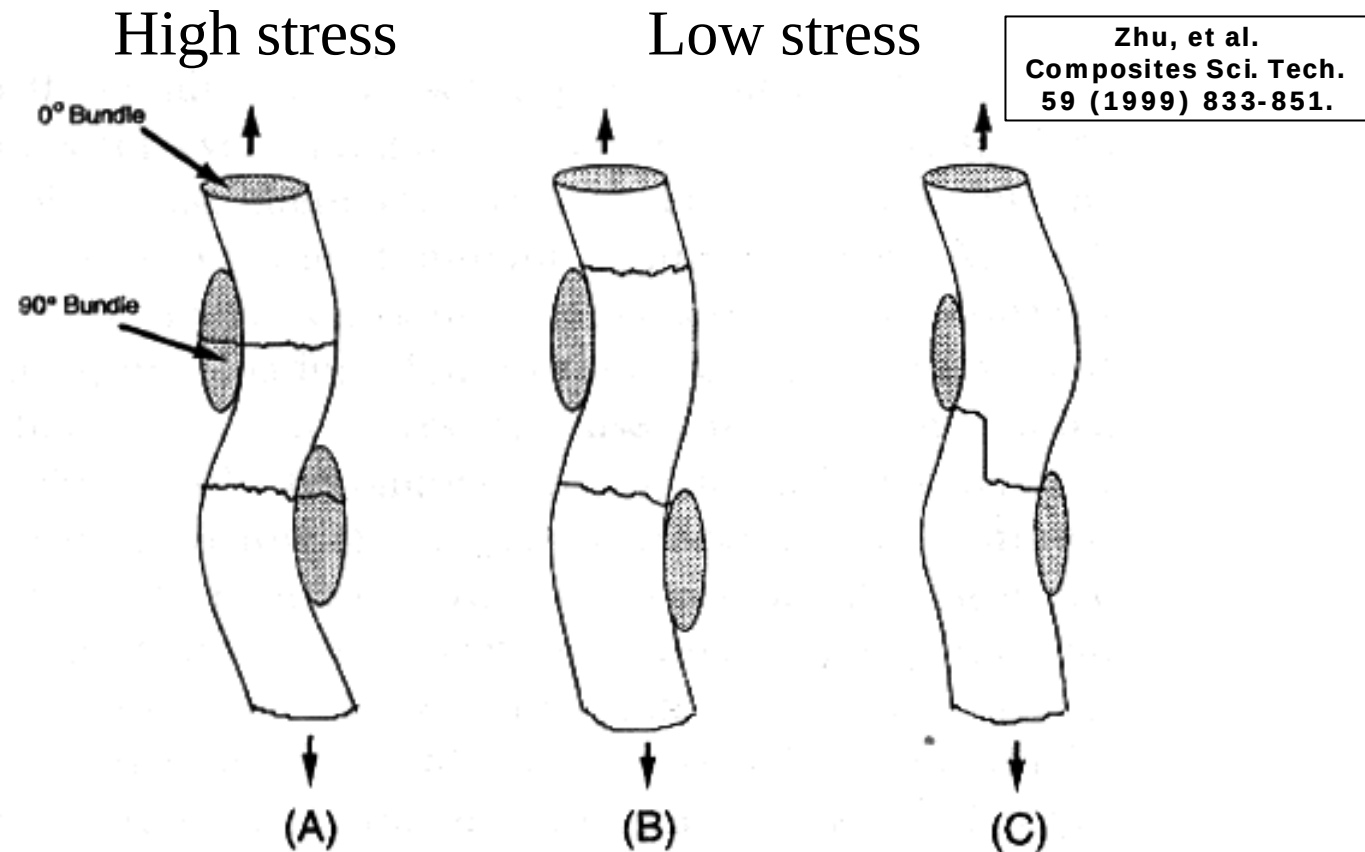


Fig. 7. Schematic diagram of fracture modes of 0° bundles.

- ◆ Inter-bundle pores and fiber waviness are affecting the low stress fatigue fracture of plain-weave composites.

Fatigue-induced F-M Interface Wearing

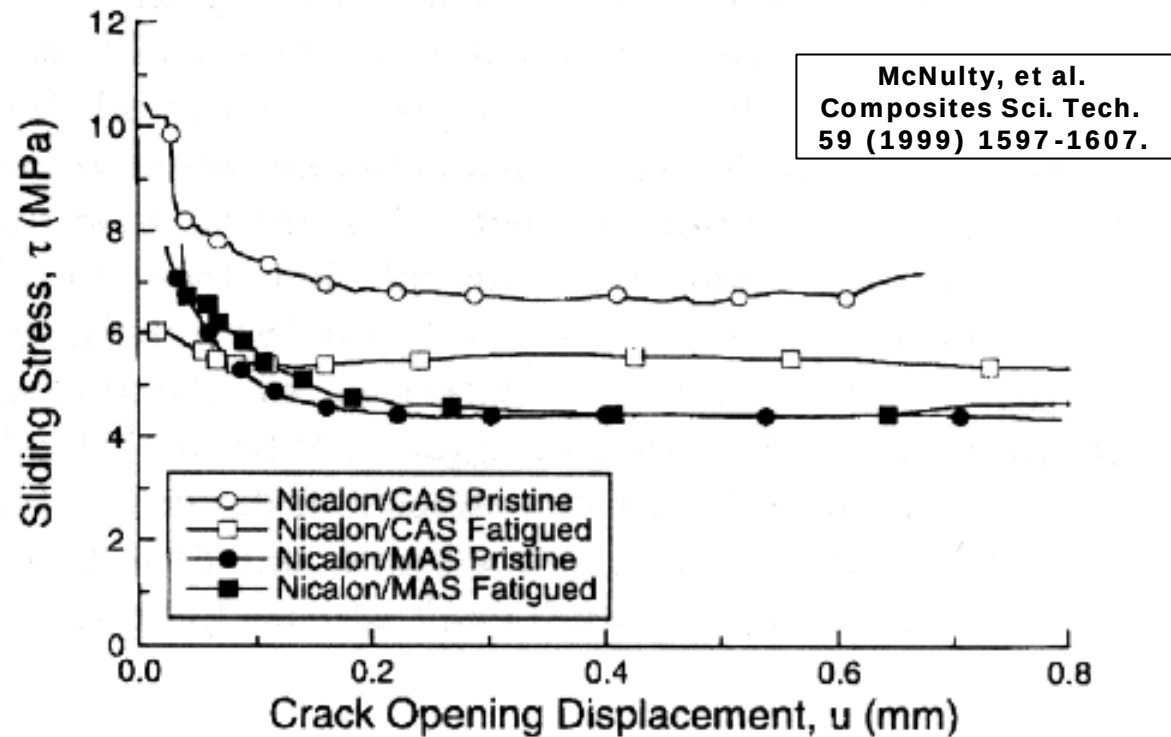
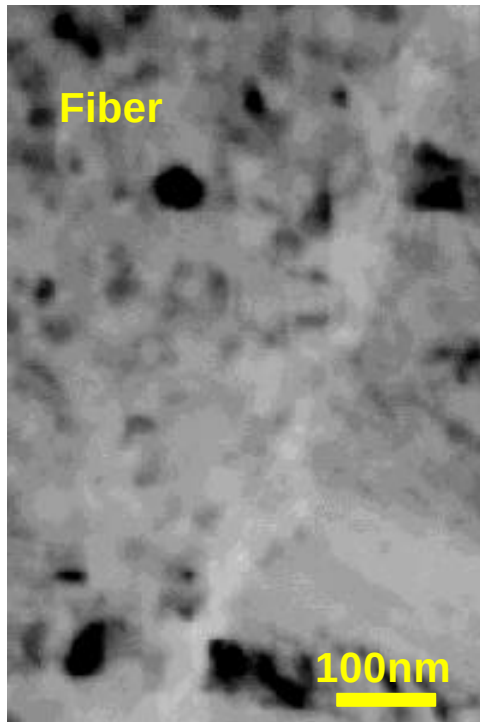


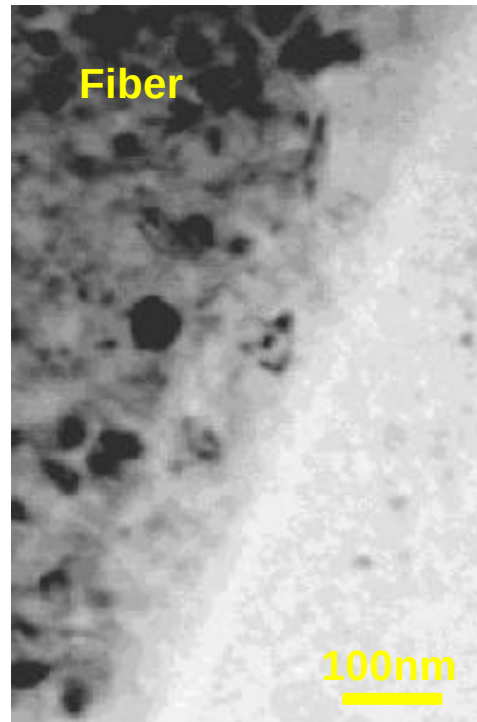
Fig. 12. Results of fiber pull-out experiments. For clarity, only the data for the fatigued materials are shown in (a) and (b).

Surface Roughness of Advanced SiC Fibers

Hi-Nicalon



Hi-Nicalon Type-S



Tyranno-SA(1997)



Thermal Creep of NL/CVI-SiC

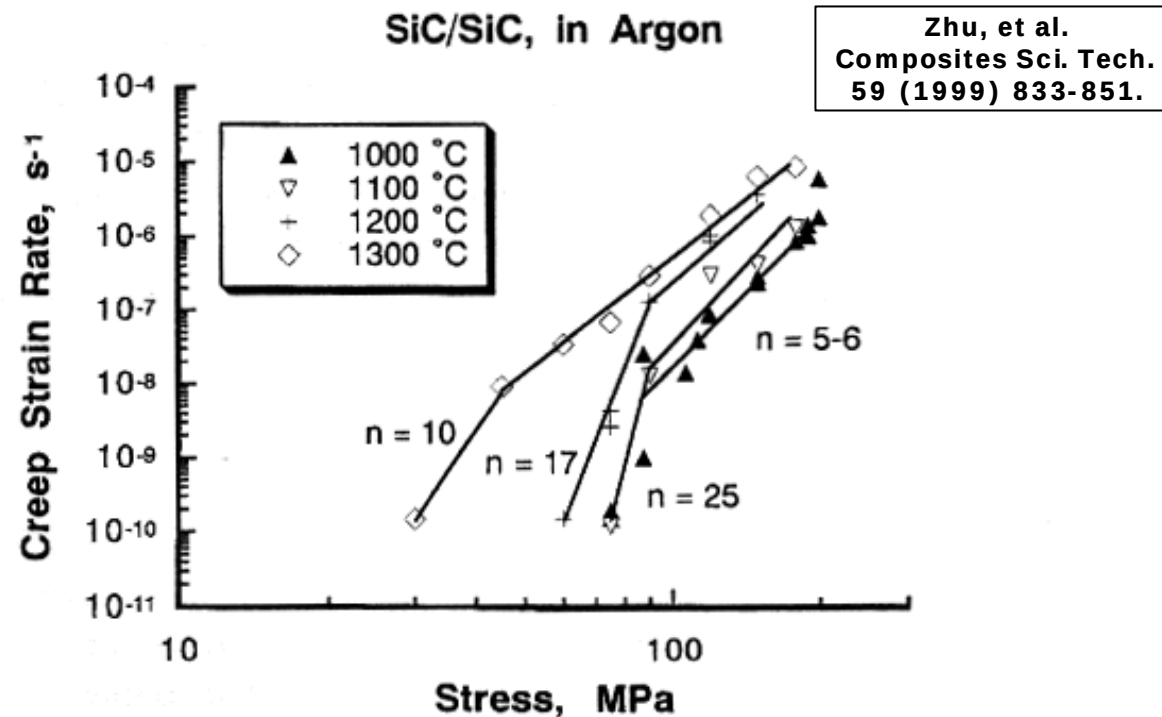


Fig. 13. Tensile minimum creep strain rate versus stress in argon at 1000, 1100, 1200 and 1300°C.

- ◆ Creep deformation of CVI-SiC matrix is controlling the creep rate of Nicalon-CG-reinforced composites.
- ◆ Advanced fibers may alter the creep mechanism.
- ◆ Reduced matrix porosity will improve the apparent creep rates.

Creep Rate – Rupture Time Correlation

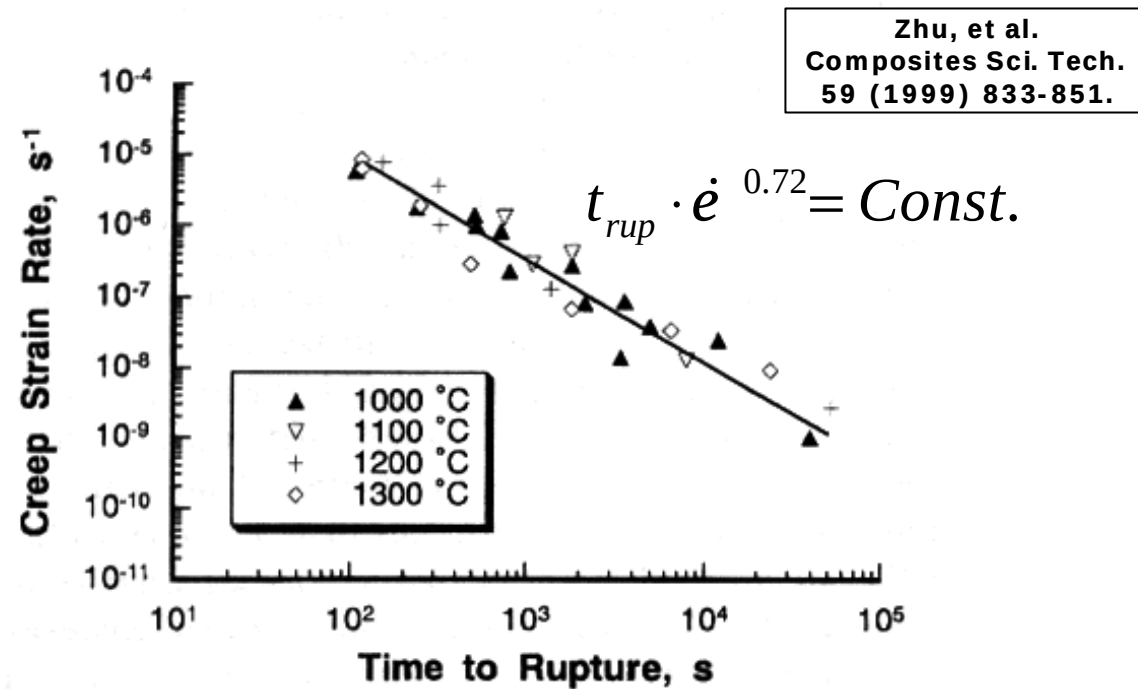


Fig. 16. Tensile minimum creep strain rate versus time to rupture at different stresses in argon at 1000, 1100, 1200 and 1300°C.

- ◆ The steady-state creep strain rate versus time to rupture falls into the Monkman-Grant relationship.

A Larson-Miller Plot

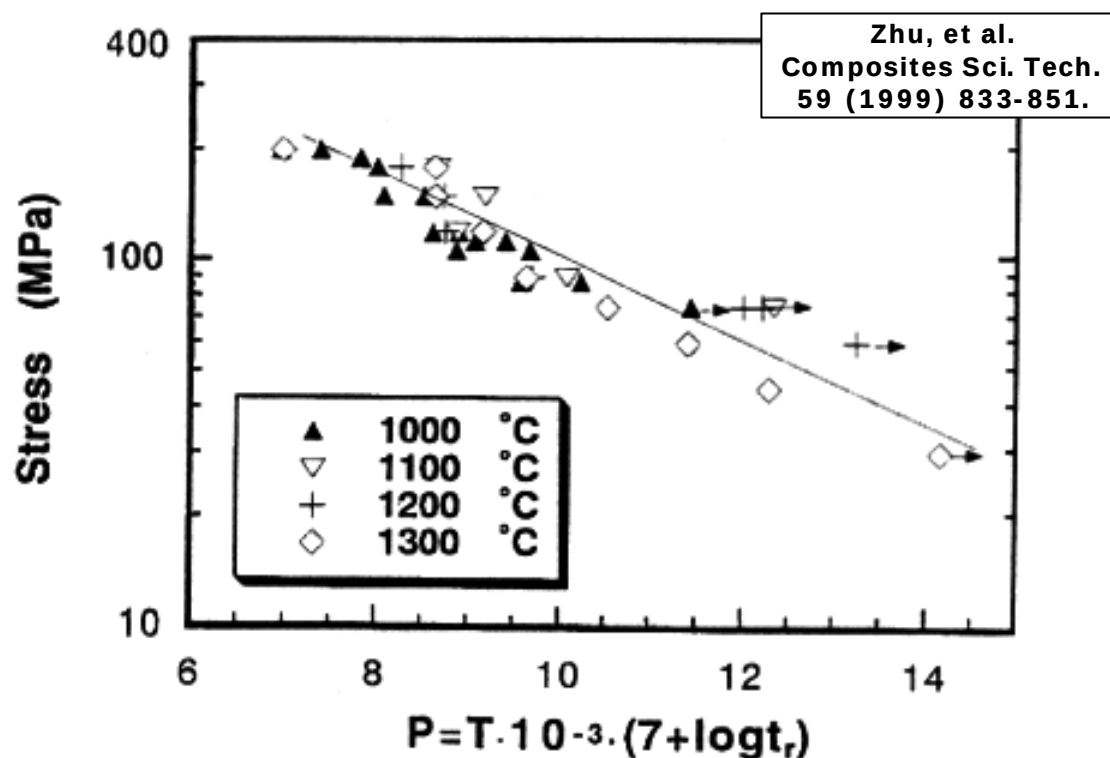
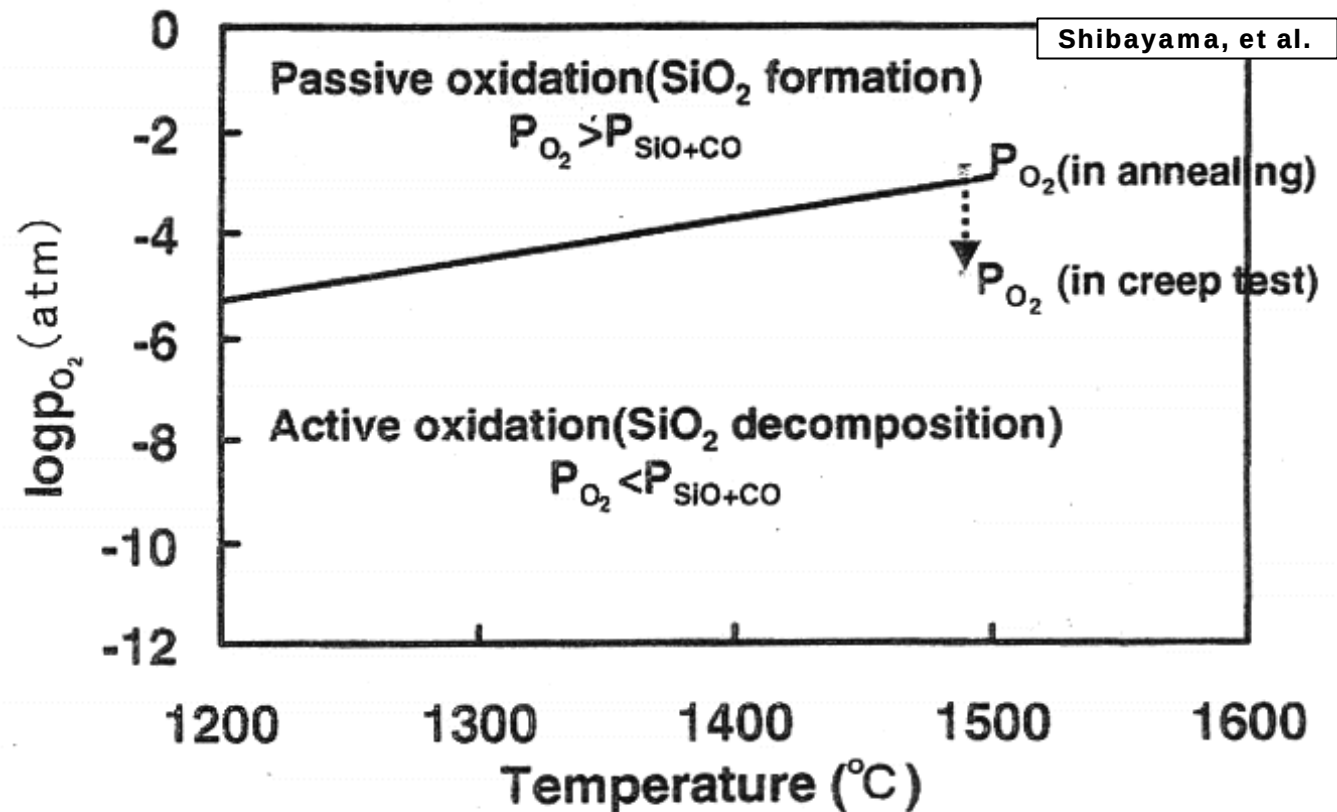


Fig. 17. Tensile stress versus Larson-Miller parameter at different stresses in argon at 1000, 1100, 1200 and 1300°C.

- ◆ Larson-Miller approach may be applicable to stress-rupture time prediction.

Oxidation Behavior of SiC

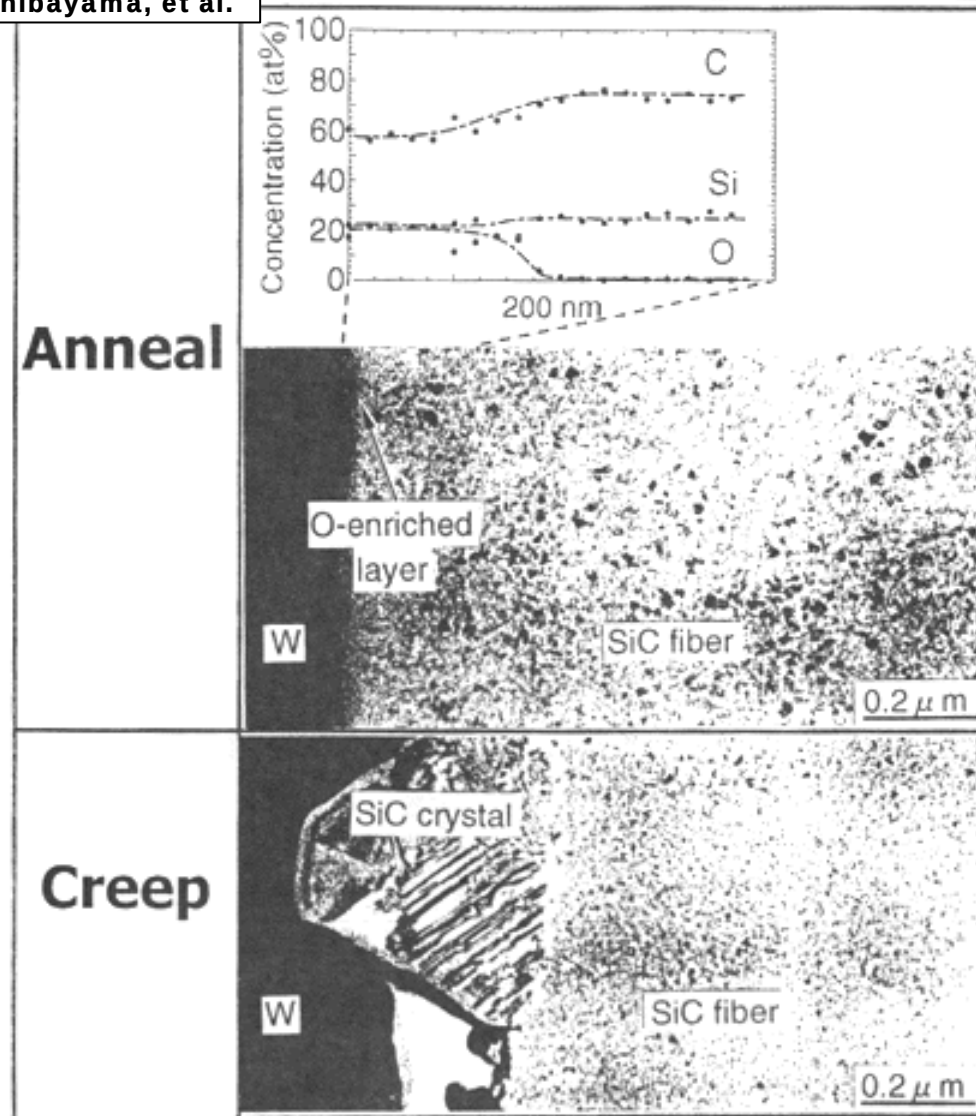


* Earl A. Gulbransen and Sven A. Jansson: Oxidation of Metals. 4 pp.181-201 (1972).

- ◆ Commercial Ar: $\text{O}_2 < 10\text{ppm}$ (normal grade), $\text{O}_2 < 1\text{ppm}$ (high purity),
 $\text{O}_2 < 0.2\text{ppm}$ (semiconductor process grade)

Effect of Tensile Stress on Erosion Behavior

Shibayama, et al.

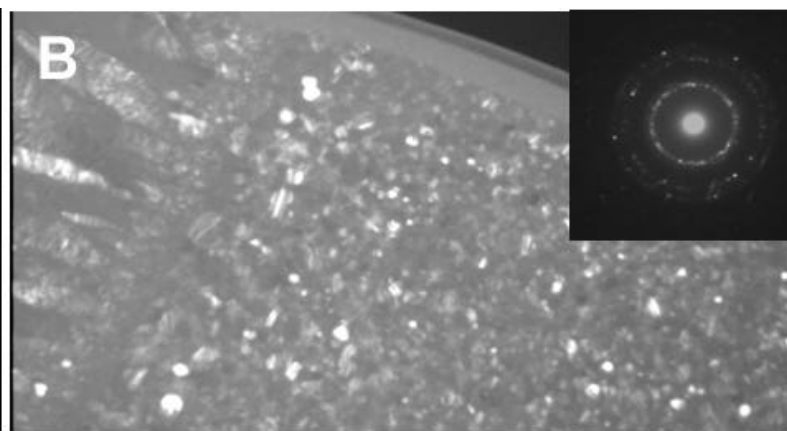
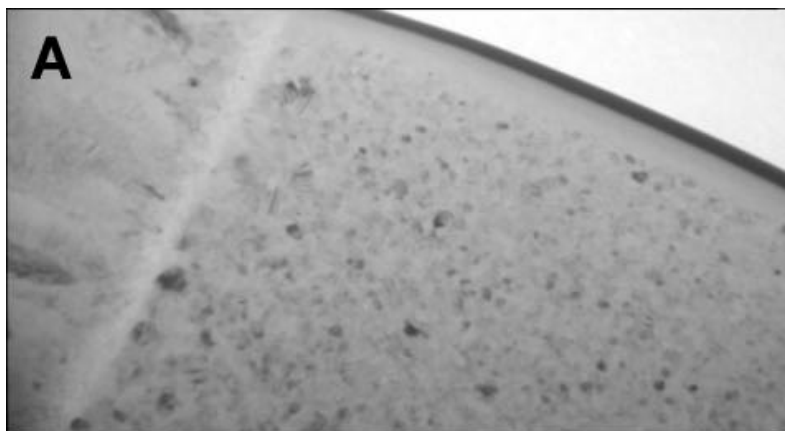


- ◆ Erosion behavior of Hi-Nicalon fiber under tensile stress is dissimilar to that in stress-free state.
- ◆ Creep and fatigue behavior in nominally 'passive oxidation' environment may be complex.
- ◆ Advanced fibers are expected to exhibit improved stability.

TEM images and SAD patterns for Hi-NL Type-S composite

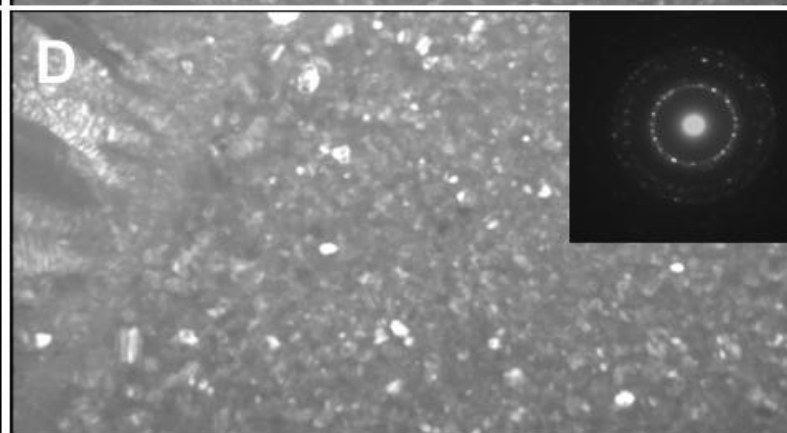
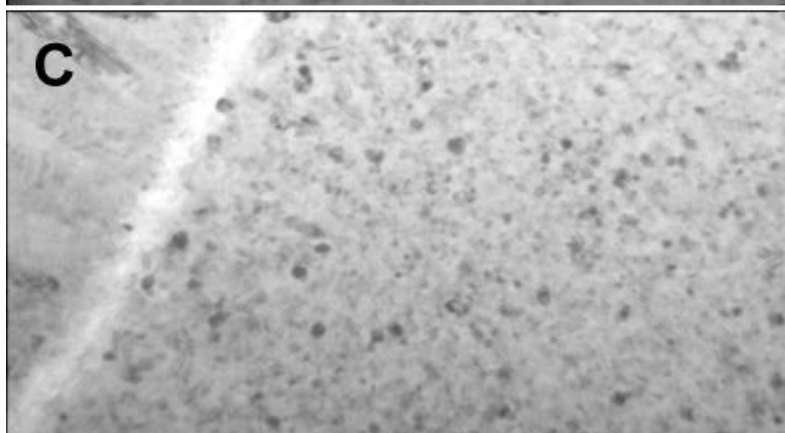
Single-beam

10dpa
873K
 1×10^{-3} dpa/s



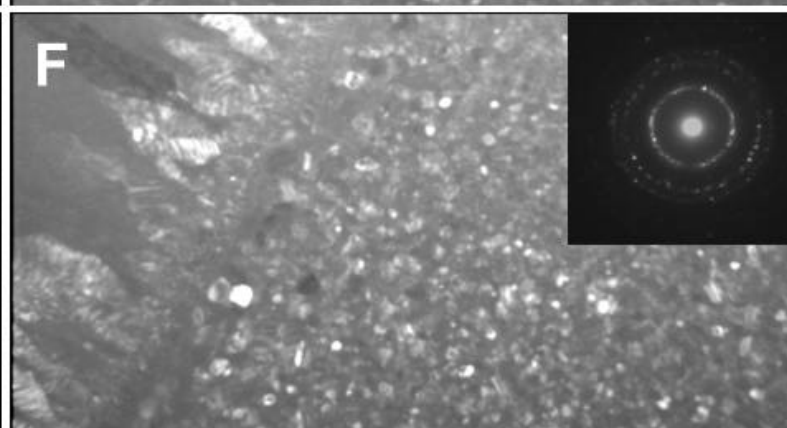
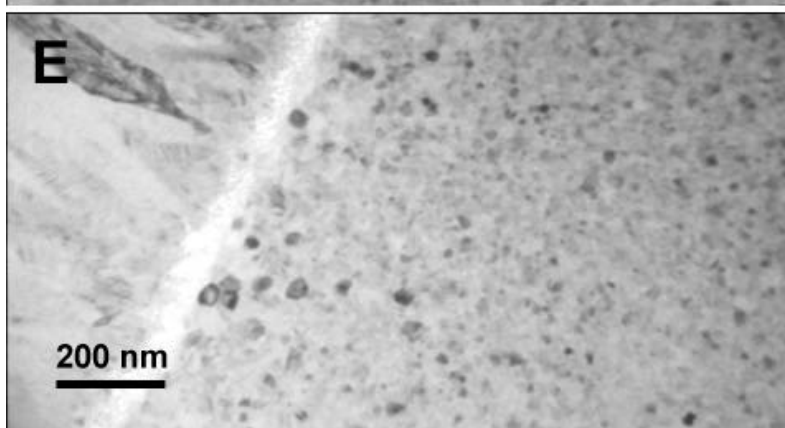
Dual-beam

10dpa
873K
 1×10^{-3} dpa/s
60appm-
He/dpa

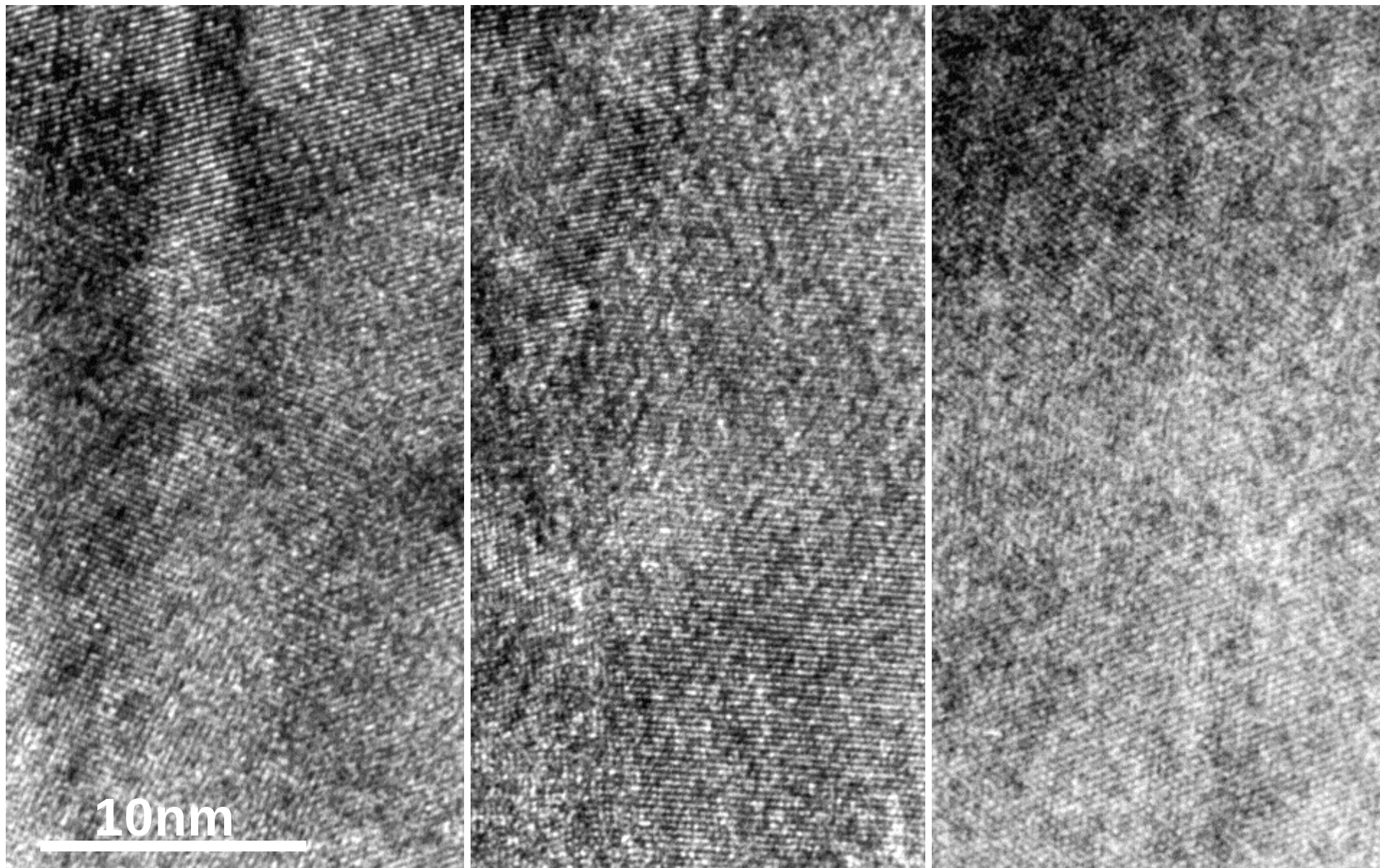


Unirradiated

Dark field
images from
SiC $\langle 111 \rangle$
diffraction
rings.



As-CVI'ed and Ion-Irradiated Hi-Nicalon® Type-S

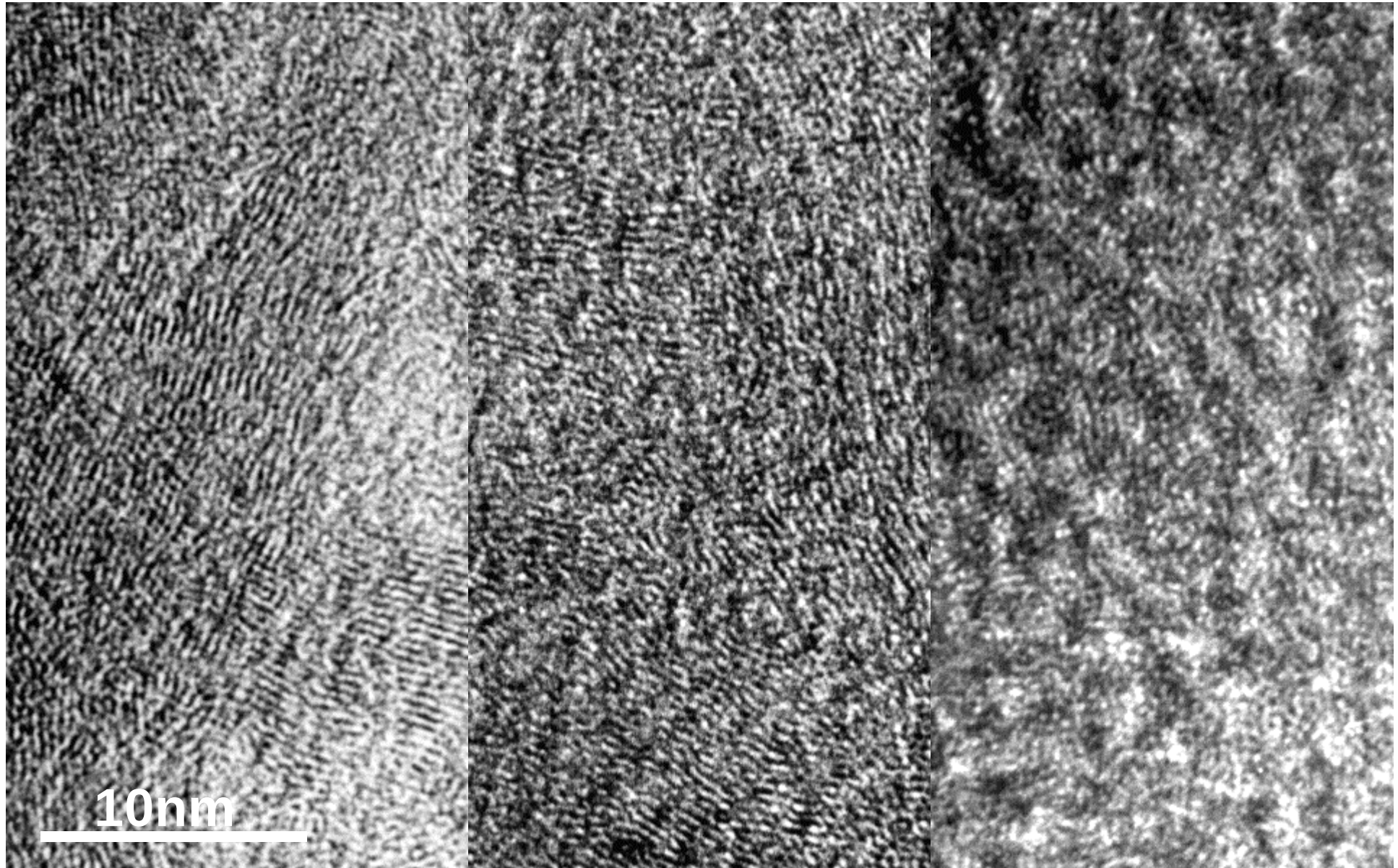


Unirradiated

Single-beam irradiated

Dual-beam irradiated

As-CVI'ed and Ion-Irradiated Pyrolytic Carbon Interphase



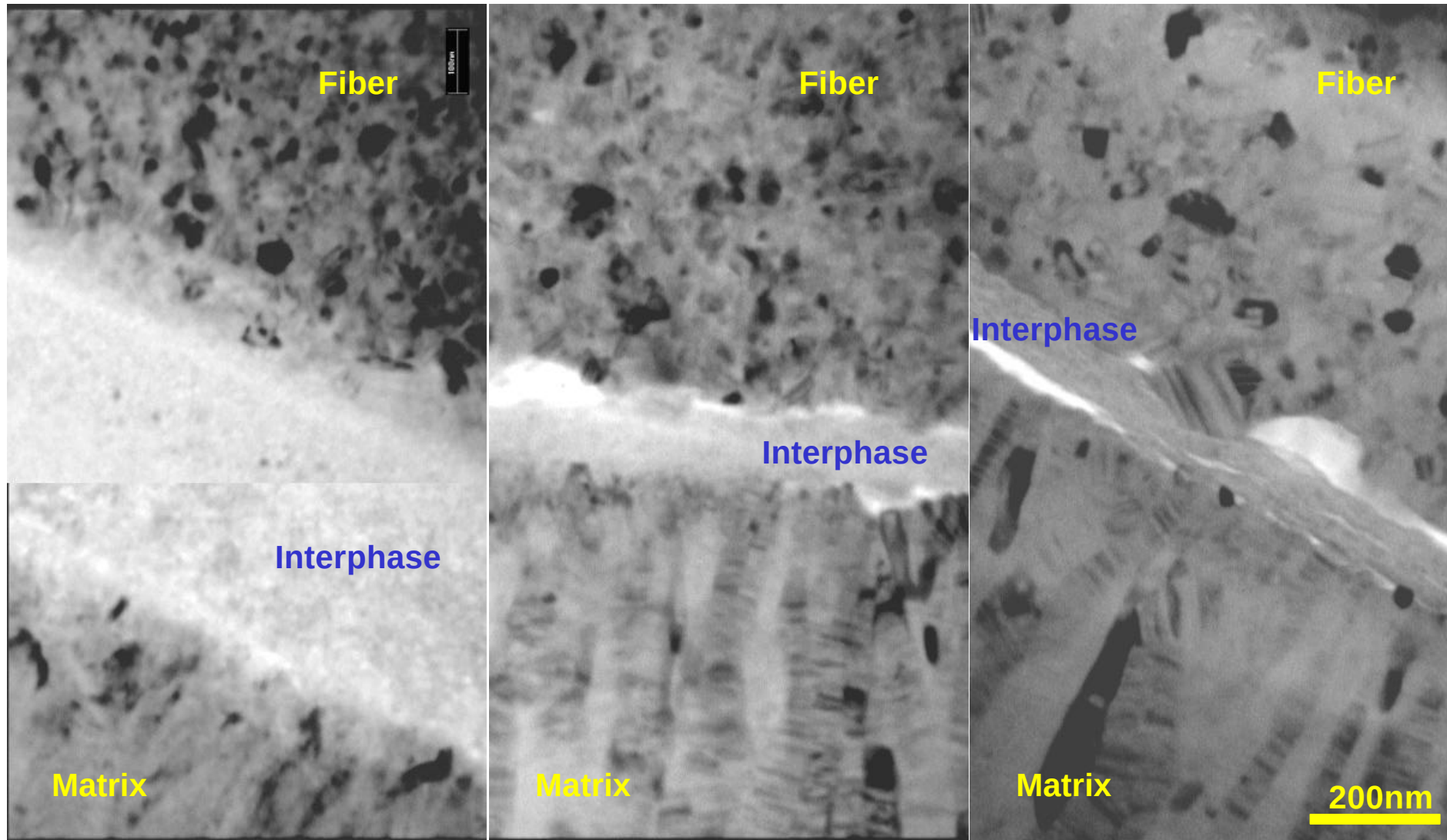
Unirradiated

Single-beam irradiated

Dual-beam irradiated

Structural Instability of SiC at F-M Interfaces

Hi-Nicalon Type-S / PyC / CVI-SiC Composite



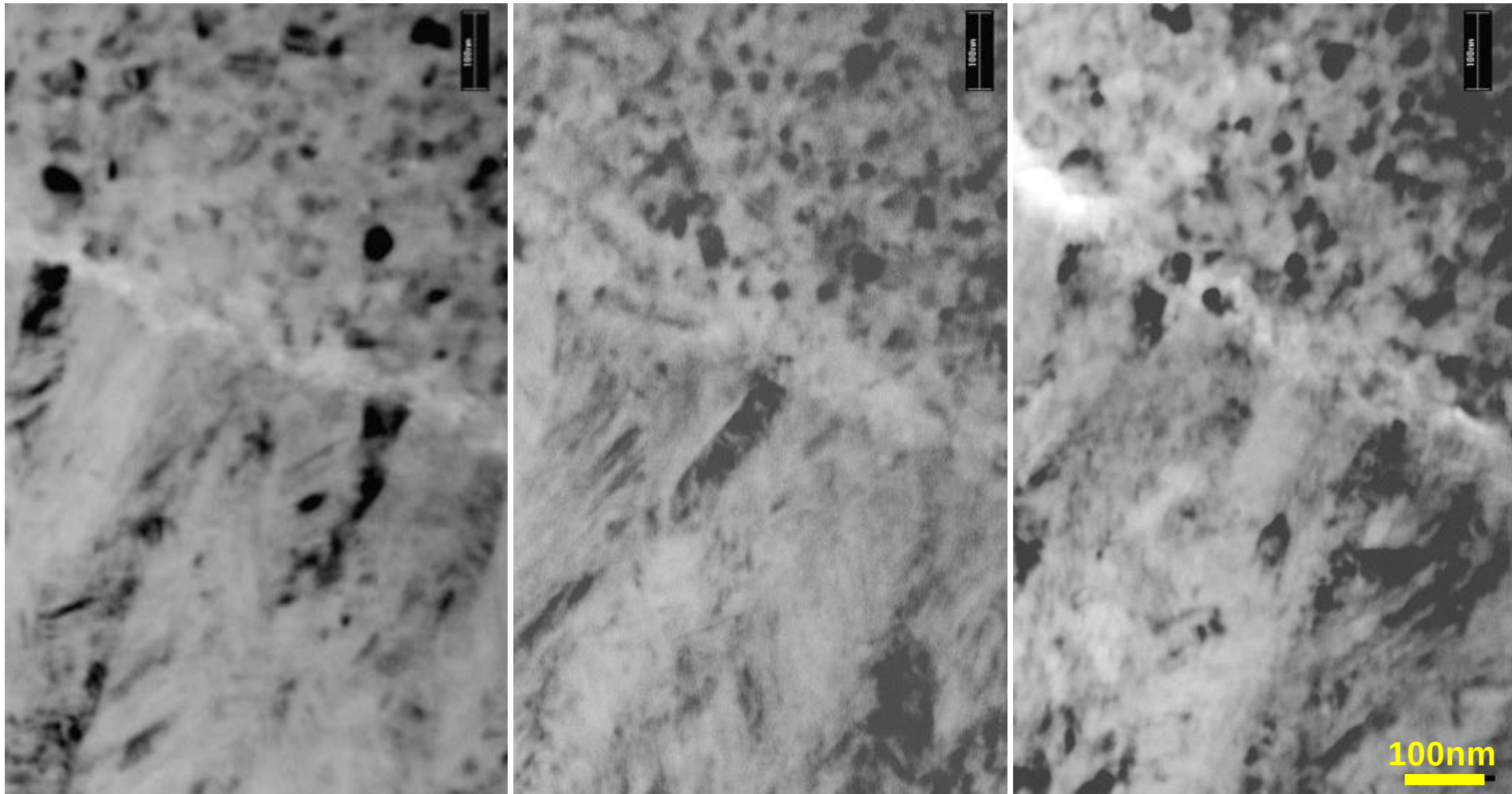
Annealed at 1473K for 3600s

Annealed at 1673K for 3600s

Annealed at 1873K for 3600s

Structural Instability of Hi-NL at Interfaces

Annealed at T for 3600s following dual-ion irradiation to 10dpa at 873K



T = 1073K

T = 1273K

T = 1473K

Low Temperature Swelling

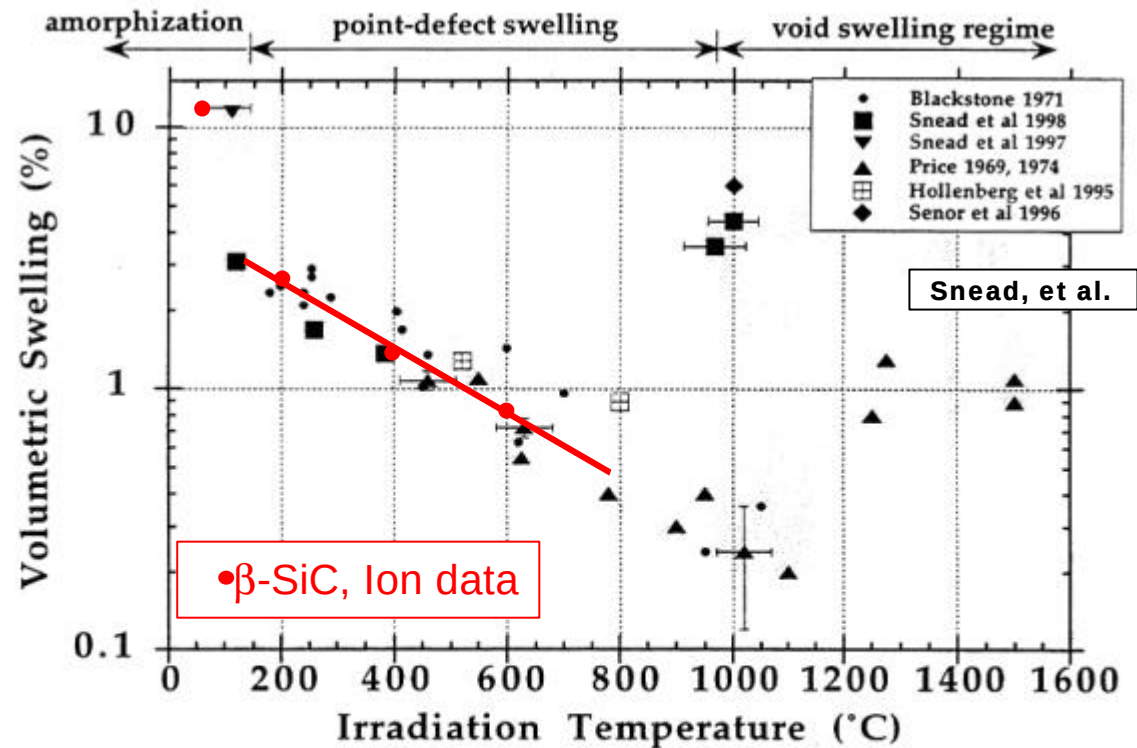
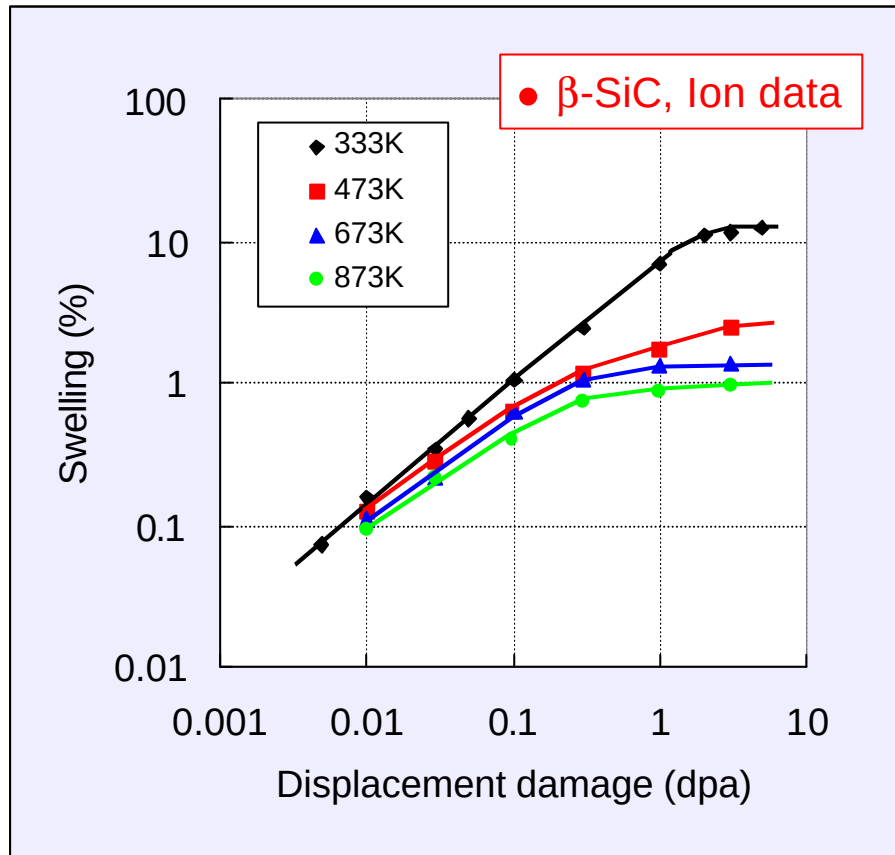


Fig. 3. Radiation induced swelling in bulk SiC as a function of irradiation temperature [6,14,26-29].

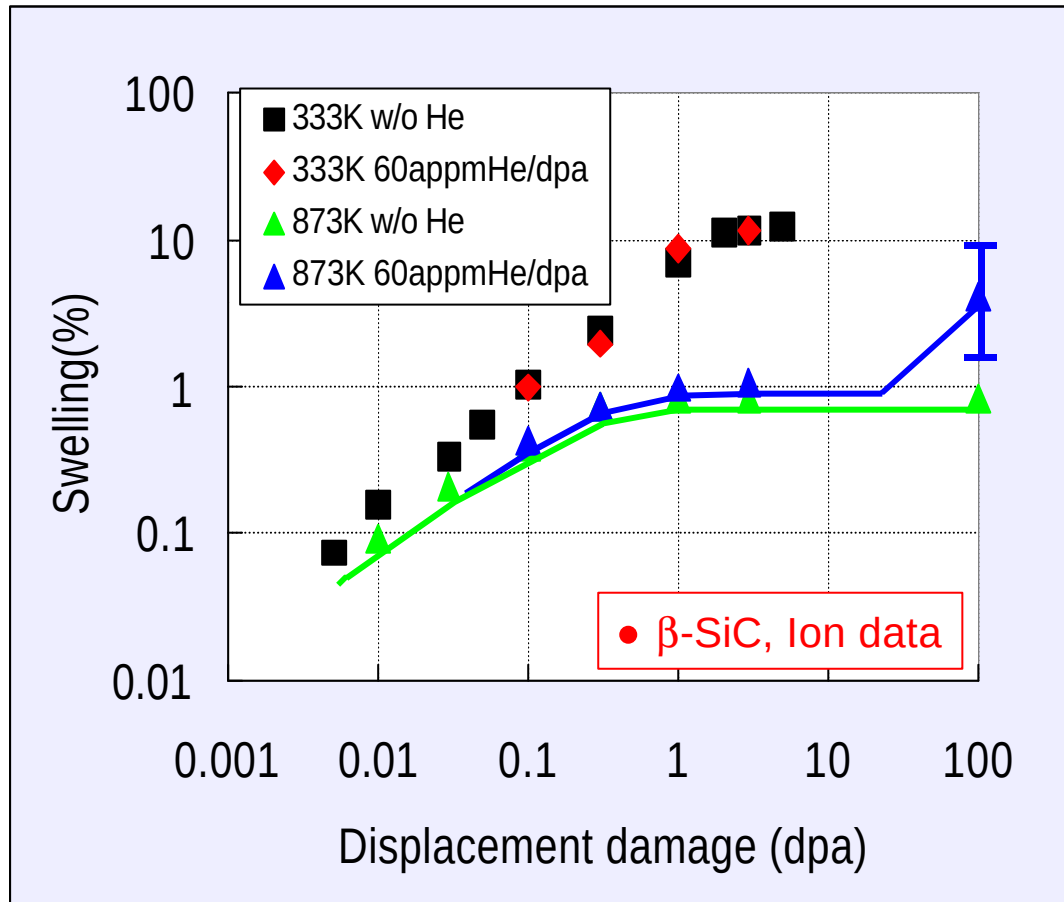
- ◆ Low temperature swelling seems to have been well characterized.
- ◆ Neutron data may be affected by uncertainty and transient effect of irradiation temperature. Ion irradiation experiments will confirm it under accurate temperature control.

Low Temperature Swelling



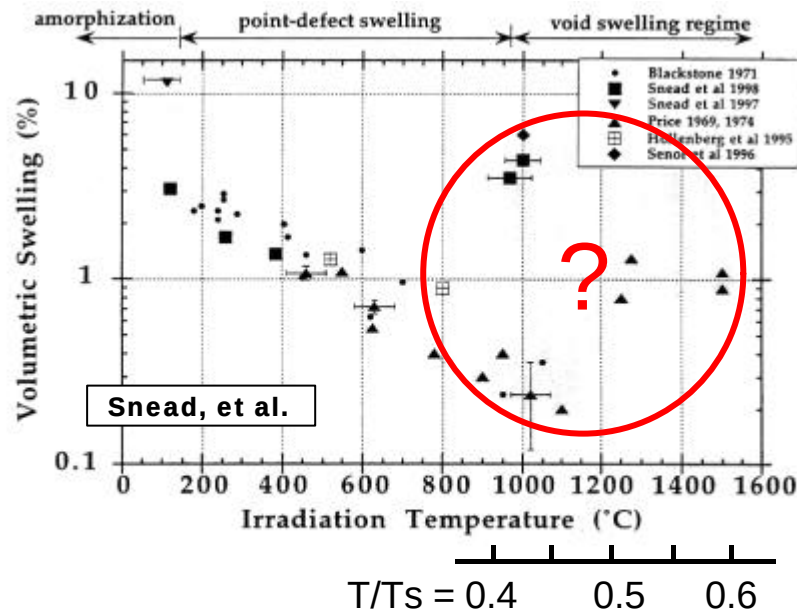
- ◆ Low temperature swelling due to point defect accumulation may exceed 1% at temperatures below 673K.
- ◆ Low temperature swelling saturates in helium free conditions. Saturation behavior is highly predictable.

Helium Effect on Low Temperature Swelling

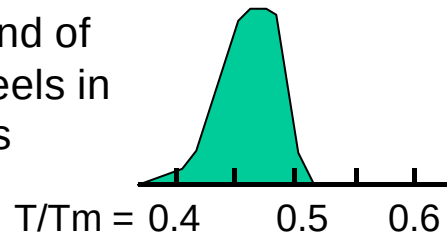


- ◆ Helium accumulation alters the state of point defects (and clusters) once saturated by lower doses at low temperatures.

High Temperature Swelling



Swelling trend of
stainless steels in
fast reactors



- ◆ We know very little about the high temperature swelling behavior of SiC.
- ◆ Transmutant helium will play the key role.
- ◆ Dual-beam ion irradiation experiment is needed to explore the synergistic effect of high-fluence irradiation and helium production.

Helium Effects on Mechanical Properties

- Helium accumulation in crystal grains:
 - ◆ Effect of swelling
- Helium precipitation in grain boundaries
 - ◆ Fracture energy degradation?
- Helium agglomeration at F-M I/F:
 - ◆ F-M debonding
 - ◆ Enhanced amorphization of carbon interphase

Irradiation Effects on F-M Interface Integrity

■ Amorphization of carbon

- ◆ Anisotropic swelling of oriented graphitic carbon
- ◆ Isotropic swelling of glassy carbon
- ◆ Strength degradation of interphase itself
- ◆ Bonding strength degradation

■ SiC crystal growth at boundaries due to chemical instability

- ◆ Debonding at C/SiC boundaries
- ◆ Sliding stress increase

■ Helium effect

- ◆ Potential helium bubble precipitation
- ◆ Other unknown effects?

Summary

- Limited amount of data on fatigue, thermal creep and erosion behavior are available for older generation SiC/SiC.
- Neutron-related lifetime limiting issues are not sufficiently identified for SiC/SiC composites for fusion blanket applications.
- These properties of ‘fusion grade’ SiC/SiC need to be characterized. It is expected that the performances of fusion grade SiC/SiC largely exceeds those of conventional materials.