Materials & Design Issues for Joining SiC Composites for Fusion Energy

C.A. Lewinsohn and R.H. Jones
Pacific Northwest National Laboratory
Richland, WA, USA.

M. Singh
NASA Glenn Research Center
Cleveland, OH, USA.

H. Serizawa
JWRI Osaka University
Osaka University

T. Hinoki, Y. Katoh, and A. Kohyama
IAE, Kyoto University
Kyoto, Japan

T. Shibayama
CARET, Hokkaido University
Sapporo, Japan

D. Carnahan
Busek Co., Inc
Natick, MA, USA
Primary goals for the use of SiC/SiC in fusion energy

- Low residual radioactivity to minimize:
  - risk to workers
  - contamination in the event of accidents
  - environmental impact of waste disposal
- Mechanical reliability
- Microstructural stability during irradiation of:
  - fibers
  - interphases
  - matrix
- Minimal gaseous transmutation
- Hermetic behavior
Critical materials requirements for joining SiC/SiC for fusion energy

- Mechanical properties
- Thermal expansion match
- Radiation/Chemical stability
- Thermal conductivity
- Time-dependent properties
- Hermeticity
Design requirements for joining SiC/SiC for fusion energy

- Thermo-mechanical stress state
  - Low shear stresses: in-plane and through thickness
  - Minimal thermal gradients
  - Principal stresses below matrix cracking stress (?)

- Field assembly
  - Compatible with processing and machining techniques
  - Assemblage under ambient conditions
  - Practical for 1 m scale components
  - Hermeticity
Illustrative joint designs for SiC/SiC
Some critical issues in joint durability

For a given joint design:
- Strength of joint material vs. matrix
- Stability of matrix and fibers
- Radiation effects on joint materials
- Thermal cycling effects
- Differential creep effects
Physical parameters required for materials R&D

- **Stress-state**
  - Principal stresses, bending moments, shear stresses
  - Temporal behavior (fatigue, TMF)

- **Temperature**
  - Gradients
  - Temporal behavior

- **Chemical environment**
  - Oxygen content
  - He pressure
  - Coolant composition, temperature, pressure

- **Neutron flux**

- **Plasma-surface interactions**
  - Particle momentum
  - Particle elastic properties
Candidate joint materials for use in Fusion Energy Systems

- Melt infiltrated and reaction-formed silicon carbide.
- Preceramic-polymer derived silicon carbide.
- Low-activation, high-temperature glasses.
- In-situ reinforced silicides.
- High-temperature brazes.
Material studied

- **Reaction Formed Silicon Carbide**: silicon carbide formed by reaction of a carbonaceous structure with molten silicon or silicon alloys. Fabricated using the ARCPJoinT process developed at NASA Glenn Research Center.

- **Reaction Bonded Silicon Carbide**: particulate silicon carbide bonded by silicon carbide formed by reaction of carbon powders with molten silicon. Fabricated by BUSEK Co., Inc., Natick, MA.
Material studied

Substrates:

- *Hexoloy SA*: monolithic, sintered, alpha SiC. Approximate grain size 2-3 μm.
- *Hi-Nicalon reinforced, CVI silicon carbide*: 40 vol. % fibers, 0/90° plain-weave.
Reaction-formed silicon carbide is a promising joint material.
Interfacial reactions must be studied

Long-term compatibility between joint material and composites must be investigated
Effects of microstructural evolution

- Interface reactions may lead to brittle phases or composite damage
- Time dependent properties may occur due to chemical diffusion:
  - Thermal Expansion
  - Elastic Moduli
  - Shear Strength
  - Stress distribution
Microscopy may be used to examine interfacial reactions

Untreated joint appears micro-crystalline

Hexoloy (SiC)

Joint material
HRTEM used to determine phase distribution

*In practice, joints must be made in the field.*

Joining conditions: 1250-1425 °C
5-10 min
no external pressure

SiC   C   Interface   Hexoloy SA

(Selected area diffraction patterns [SADP])

Control of spatial phase distribution may allow functional grading of thermomechanical properties.
Mechanical Testing

**Maximum Tensile Stress**
- 4-pt bending

**In-plane Shear Stress**
- Double-notch-shear in compression
Mechanical Testing

Butt-joined flexural test specimen

45° Butt-joined flexural test specimen

Double-notch-shear specimen

Offset sandwich specimen

not drawn to scale.
Mechanical Testing

Through Thickness Shear Stress
Asymmetric 4-pt bending

Upper Fixture

Lower Fixture

specimen

roller/loading point

x-y y
Results: Flexural Strength

* Four-point bend strengths on the order of 200-300 MPa are commonly reported
Results: Flexural Strength

Bond strengths for composite substrates were in the range of those for monolithic substrates.
**Results: Flexural Strength**

Additional heat treatment improves flexural strength.

- 25°C untreated
- 25°C 1100°C 100 h, Ar
- 1100°C untreated
Results: Through-thickness shear strength

*The value of the Through-Thickness Shear Strength was similar for composites joined at cut surfaces or surfaces coated with CVD-SiC.*
## Results

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Joint Material</th>
<th>Test Method</th>
<th>Test Temp. (K)</th>
<th>Joint Thickness (µm)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RBSC</td>
<td>RFSC</td>
<td>4PBS</td>
<td>298</td>
<td>10</td>
<td>210 ± 6</td>
</tr>
<tr>
<td>Hexoloy SA</td>
<td>RFSC</td>
<td>4PBS</td>
<td>298</td>
<td>45-50</td>
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<td>RFSC</td>
<td>4PBS</td>
<td>298</td>
<td>52</td>
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<tr>
<td>Hexoloy SA</td>
<td>RBSC</td>
<td>4PBS</td>
<td>298</td>
<td>130</td>
<td>85 ± 10</td>
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<tr>
<td>SiC_f/SiC_m</td>
<td>RFSC</td>
<td>A4PB</td>
<td>298</td>
<td>115</td>
<td>28 ± 7</td>
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<tr>
<td>SiC_f/SiC_m</td>
<td>RFSC</td>
<td>4PBS</td>
<td>298</td>
<td>115</td>
<td>78 ± 8</td>
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<tr>
<td>SiC_f/SiC_m</td>
<td>RFSC</td>
<td>4PBS</td>
<td>298</td>
<td>125</td>
<td>65 ± 5</td>
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<tr>
<td>SiC_f/SiC_m</td>
<td>RFSC</td>
<td>4PBS</td>
<td>1073</td>
<td>125</td>
<td>66 ± 9</td>
</tr>
<tr>
<td>SiC_f/SiC_m</td>
<td>RFSC</td>
<td>4PBS</td>
<td>1473</td>
<td>125</td>
<td>59 ± 7</td>
</tr>
</tbody>
</table>

RBSC = reaction-bonded silicon carbide
RFSC = reaction-formed silicon carbide
4PBS = 1/4, four-point bend strength
A4PB = assymmetrical, four-point bend strength
Summary

Materials issues: radiation stability, physical properties, hermeticity.
Design issues: thermomechanical stresses, field assembly.
Critical issues: time-dependent properties, radiation effects, thermal cycling, differential creep.
Materials studied: reaction-formed and reaction-bonded SiC.

Results:
- Flexural strengths around 200 MPa were obtained.
- Flexural strengths were independent of substrate material.
- Additional heat treatment improved the flexural strengths.
- Through-thickness shear strengths of joint materials were lower than the tensile strengths, but were not dependent on the surface treatment.
Future work

* Optimize joint processing conditions and thickness for mechanical properties.

* Study effects of thermal exposure and irradiation on microstructure and properties.

* Evaluate mechanical test methodology for irradiation studies.

* Investigate stress distribution in realistic joint geometries via FEM modeling.