Robust integration and assembly technologies are critical for the successful implementation of advanced metallic, ceramic, carbon-carbon, and ceramic matrix composite components in a wide variety of aerospace, space exploration, and ground based systems. Typically, the operating temperature of these components varies from few hundred to few thousand Kelvin with different working times (few minutes to years). The wide ranging system performance requirements necessitate the use of different integration technologies which includes adhesive bonding, low temperature soldering, active metal brazing, diffusion bonding, ARCJoinT, and ultra high temperature joining technologies. In this presentation, a number of joining examples and test results will be provided related to the adhesive bonding and active metal brazing of titanium to C/C composites, diffusion bonding of silicon carbide to silicon carbide using titanium interlayer, titanium and hastelloy brazing to silicon carbide matrix composites, and ARCJoinT joining of SiC ceramics and SiC matrix composites. Various issues in the joining of metal-ceramic systems including thermal expansion mismatch and resulting residual stresses generated during joining will be discussed. In addition, joint design and testing issues for a wide variety of joints will be presented.
Robust Joining and Integration Technologies for Advanced Metallic, Ceramic, and Composite Systems

M. Singh, Tarah Shpargel, Gregory N. Morscher*, Michael H. Halbig**
QSS Group, Inc.
*Ohio Aerospace Institute
**US Army Vehicle Technology Directorate
NASA Glenn Research Center
Cleveland, OH 44135

Rajiv Asthana
Technology Department
University of Wisconsin-Stout
Menomonie, WI 54751
Outline

• Need for Joining and Integration Technologies
• Technical Issues and Challenges
  • Joint Design and Analysis
  • Thermomechanical Issues
  • Testing and Evaluation
• Active Metal Brazing (Metals to Composites)
  • Microstructural Analysis
  • Mechanical Behavior
• Diffusion Bonding (Ceramic-Ceramic)
  • Joint Microstructure
  • Interfacial and Mechanical Characterization
• Reactive Joining Using ARCJoinT (Ceramic-Ceramic, Composites-Composites)
  • Joint Microstructure and Interfacial Analysis
  • Thermomechanical Performance
• Applications
• Summary and Conclusions
Need for Joining and Integration Technologies

- Joining and integration technologies are key to development and utilization of advanced ceramics and composites in aerospace and ground based applications.

  - **Aerospace Systems**
    - *Aerospace and Space Propulsion Components (Combustor Liners, Exhaust Nozzles, Nozzle Ramps, Turbopump Blisks)*
    - *Thermal management systems (Radiators, recuperators), optical components, and dimensionally stable space structures*

  - **Ground Based Systems**
    - *Nuclear Industries, Land Based Power Generation, Process Industries, Heat Exchangers, Recuperators, Microelectronic Industries (Diffusion Furniture, Boats)*
    - The development of robust joining and assembly capability will allow the application of advanced ceramics and composites technology in a timely manner.
Technical and Performance Requirements for Joined Structures

- Typically for the high temperature aerospace and ground based applications (ceramic and composite-based systems):
  - Use temperature $> 1200 \, ^\circ\text{C}$ (*joint properties comparable to base materials*).
  - Good thermomechanical properties (strength and oxidation resistance)
  - Low CTE mismatch to minimize residual stresses and good thermal shock resistance
  - Leak tight joints

- In ceramic-metal systems, joint performance is limited by the temperature capability of metallic component in the system (braze/bond layer, metallic substrate). These systems have operational capability around 700-800°C.

- Practical, reliable, and affordable technique adaptable to in-field installation, service, and repair.
Robust Joining and Attachment Technologies

Design Considerations

Supporting Technologies

Analysis

Full-Scale Tests

Component Tests

Subcomponent Tests

Element Tests

Coupon Tests

Opportunities to Utilize Building-Block Approach to Design and Manufacturing of Large Ceramic and Composite Structures
Bonding of Metals to Ceramics and Composites
Using Metallic Interlayers

Ceramics/Composite Systems:
- SiC, Si3N4
- YSZ, Alumina
- C/C Composites
- C/SiC, SiC/SiC

Metallic Systems:
- Titanium
- Inconel and Other Ni-Base Superalloys
- Kovar
- Stainless Steels

Active Metal Brazing
Soldering

Interlayer Systems:
- Active Metal Brazes (Ag, Cu, and Pd based)
- Metallic Glass Ribbons
- Solders (Zinc based)

Technical Issues:
- Melting range / behavior
- Wetting characteristics
- Flux or atmosphere compatibility
- Compositional compatibility
- Cost & availability
Melting Temperature Range of Typical Braze and Solder Systems

<table>
<thead>
<tr>
<th>SOLDERING</th>
<th>BRAZING</th>
<th>HIGH TEMPERATURE BRAZING</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Cadmium</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Nickel</td>
</tr>
<tr>
<td>Lead / Tin</td>
<td>Aluminum</td>
<td>Silver</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Gold</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Platinum &amp; Palladium &amp; Silicides</td>
</tr>
</tbody>
</table>

Some Braze / Solder melting temperature ranges

Temp °F 200 400 600 800 1000 1200 1400 1600 1800 2000 2200 2400 2600

From *Principles of Brazing*, ASM International (2005)
Nuclear Electric Propulsion Technologies are Critical for Space Exploration Missions

NEP Enables:
- Outer Planet Orbiters (rather than Flybys)
- Multiple Targets on Single Mission
- High Power, Long Duration In-Situ Science
- High Data Rate Communications
Joining and Assembly Needs in Heat Rejection System

- Radiator Face Sheets
  - C/C Composites
  - CFRP Composites

- Saddle Materials
  - Foams
  - Composites (2D,3D)

- Bonding/Assembly
  - Active Metal Brazing
  - Adhesives

- Mechanical Attachments

- Thermal Control Coatings and Treatments

- Heat Pipes and Related Technologies

- Testing and Analysis
- Lifetime Testing
- Property Database
- Performance database
Brazed and Adhesive Bonded Sub-elements for Heat Rejection System

Brazed Radiator Sub-elements

Adhesive Bonded Radiator Sub-element
Thermal Expansion Mismatch Issues are Critical in Brazing of Metal-Composite System

Innovative joint design concepts, new braze materials, and robust brazing technology development are needed to avoid deleterious effects of thermal expansion mismatch.
Active Metal Brazing

- Ti tubes and plates brazed to P120 CVI C/C composite (Goodrich)
- Several braze/solder compositions compared (processing Temp):
  - TiCuSil (910 C) foil and paste
  - CuSil-ABA (820 C) foil and paste
  - CuSin-1ABA foil (810 C)
  - Incusil foil (725 C)
  - S-Bond solder (~ 400 C)
- Two tests have proved successful:
  - Butt Strap Tension (BST)
  - Tube-Plate Tensile Test

- Require good wetting, bonding and spreading properties
- Desire minimal residual stress induced cracking in C/C
Microstructure of Brazed Ti and C-C Composites using TiCuSil and CuSil ABA Paste

Titanium Plate

Ti- Tube

Ti- Plate

C/C

10 μm

P120

CuSil ABA

100 μm

Ti
Microstructure of Joint Interface in Ti and C-C Composites Brazed using CuSin ABA Foil

Composition:
1) 98% Ti, 1%Cu, 0.5% Ag, 0.5% Sn
2) 61%Ti, 36%Cu, 2%Ag, 2%Sn
3) 37% Ti, 59%Cu, 2%Ag, 2%Sn
4) 28% Ti, 47%Cu, 25% Ag
5) 3%Ti, 84%Cu, 13%Ag,
6) 1%Ti, 3%Cu, 96%Ag
7) 100%C
Mechanical Testing of Brazed/Soldered Joints

Factors to consider:
- Braze composition, Processing variables
- Bonded area, Location of failure
- Architecture effects
**Tube Tensile Test Data for Brazed Joints**

- **TiCuSil Foil**: 34.2 N
- **TiCuSil Paste**: 41.1 N
- **Cusil-ABA Foil**: 18.7 N
- **Cusil-ABA Paste**: 49.7 N
- **Cusin-1ABA Foil**: 13.5 N
- **Incusil Foil**: 8.2 N

Legend:
- **Best spreading and largest bonded area**
Failure Behavior of Ti Tube - C/C Composite Joints

Tube and C/C plate fracture surfaces for CuSil-ABA paste braze material showing the bonded area of the outer ply of C/C brazed to the Ti tube (left) and C/C plate (right).

Tube and C/C plate fracture surfaces for Incusil ABA foil braze material showing the distinct bonded areas of the outer ply of C/C brazed to the Ti tube (left) and C/C plate (right).
Butt Strap Tensile (BST) Test Data

No thermal-induced cracks in C/C

Thermal-induced cracks in C/C

Shear Strength, MPa

TiCuSil ABA Foil
TiCuSil ABA Paste
CuSil ABA Foil
CuSil ABA Paste
S Bond Solder
C/C to C/C w/CuSilABA Paste
Thermally-Induced Cracking in C/C Controls Shear Strength of Brazed Joints

For braze materials where there was strong bonding between the braze and the C/C and failure occurred in the outer-ply of the C/C

\[ \Delta \alpha = \alpha (\text{Ti}) - \alpha (\text{C/C}) \]

\[ \Delta T = T (\text{liquidus} \sim \text{processing}) - 25^\circ \text{C} \]

<table>
<thead>
<tr>
<th>Joint Material</th>
<th>Proc. Temp., C</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-Bond</td>
<td>~ 400</td>
</tr>
<tr>
<td>CuSil ABA</td>
<td>830</td>
</tr>
<tr>
<td>TiCuSil</td>
<td>910</td>
</tr>
</tbody>
</table>
Active Metal Brazing of C/C Face Sheet/Poco Foam/Titanium System

CuSil-ABA Braze Alloy

Brazed Joints

Poco Foam

C/C

150um

Poco Foam

Ti

150um
Locations of Potential Joint Failure in C/C Face Sheet/Poco Foam Saddle/Titanium

In addition the geometry of joining surfaces will affect strength of joint and influence spreading of joint material: flat to flat, flat to tube, curved surfaces...

Therefore, knowing the location of joint failure is critical
  • Weakest link requiring further improvement
  • Affects interpretation of results (material or test-dependent property)

Key factor: Bonded area dictated by braze composition and applied pressure, C/C constituent composition, fiber orientation, geometry of joined surface
Diffusion Bonding of Silicon Carbide Using Metallic Interlayers
Multi-Point Lean Direct Injector

(Left) Multi-Point Lean Direct Injector accelerates fuel-air mixing and has small recirculation zones with short residence time that reduces NOx emission.

(Center) 3-inch square metal MP-LDI with 45 injectors.

(Right) Detail of fuel and airflow.

Lean Direct Injector Fabricated by Laminates

SiC laminates can be used to create intricate and interlaced passages to speed up fuel-air mixing to allow lean-burning, ultra-low emissions.

Key Technologies:
- Bonding of SiC to SiC
- Brazing of SiC to Metallic (Kovar) Fuel Tubes

Benefits of Laminated Plates
- Passages of any shape can be created to allow for multiple fuel circuits
- Provides thermal protection of the fuel to prevent choking
- Low cost fabrication of modules with complicated internal geometries through chemical etching
Microcracking may be due to the formation of two detrimental phases:

- Phase B Ti$_5$Si$_3$C$_x$ – Ti$_5$Si$_3$ if highly anisotropic in its thermal expansion where CTE(c)/CTE(a) = 2.72 (Schneibel et al).

- Phase E – Ti$_3$Al has low ductility at low temperatures. Al can be in the range of 23-35 atm % (Djanarthany et al).

Both phases can contribute to thermal stresses and microcracking during cool down.
Microprobe of CVD SiC Reaction Bonded Using PVD Ti Conditions: 1250 °C, 31 MPa, 2 hr, vacuum, 5 °C/min

The undesirable phases of Ti₅Si₃ and Ti₃Al were not formed.
No microcracks are observed.
Identity/source of the black phase or voids still needs to be determined.
Initial Strength Tests on Diffusion Bonded CVD SiC with a PVD Ti Interlayer

Initial pull test tensile strengths:
> 23.62 MPa (3.43 ksi)*
> 28.38 MPa (4.12 ksi)*
* failure in the adhesive

The injector application requires a strength of about 3.45-6.89 MPa (0.5 - 1.0 ksi).
The new 1” sample design (partially coated disks) will allow for stresses of 62 MPa (9 ksi) to be applied (due to a large adhesive/pull area compared to the diffusion bond area).
Joining of Advanced Ceramics and Composites
- Monolithic SiC Ceramics
- Fiber Reinforced Composites
Joining of Ceramic Components Using Affordable, Robust Ceramic Joining Technology (ARCJoinT)

Apply Carbonaceous Mixture to Joint Areas

*Cure at 110-120°C for 10 to 20 minutes*

Apply Silicon or Silicon-Alloy (paste, tape, or slurry)

*Heat at 1250-1425°C for 10 to 15 minutes*

Affordable and Robust Ceramic Joints with Tailorable Properties

1999 R&D 100 Award
2000 NorTech Innovation Award

**Advantages**
- Joint interlayer properties are compatible with parent materials.
- Processing temperature around 1200-1450°C.
- No external pressure or high temperature tooling is required.
- Localized heating sources can be utilized.
- Adaptable to in-field installation, service, and repair.
ARCJoinT is Currently Being Used to Join and Repair a Wide Variety of Ceramic and Composite Materials

**SiC-Based Ceramics**
- Reaction Bonded SiC
- Sintered SiC
- CVD SiC, Porous SiC

**SiC/SiC Composites**
- Melt Infiltrated SiC/SiC
- CVI SiC/SiC Composites
- PIP SiC/SiC Composites

**C/SiC Composites**
- Melt Infiltrated C/SiC
- CVI C/ SiC Composites
- PIP C/ SiC Composites

**C/C Composites**
- CVI C/C Composites
- Resin Derived C/C
- C-C/ SiC with MI

- Composites with Different Fiber Architectures and Shapes
- Ceramics with Different Shapes and Sizes
Technical Challenges in Design and Selection of Joints in Advanced Ceramics and Composites

Typical Ceramic Joints will have Combination of Stresses Under Operating Conditions

(a) Compression; (b) Tension; (c) Shear; (d) Peel; (e) Cleavage

Different Types of Shear Tests
Technical Challenges in Joining of Ceramic Matrix Composites

• **Joint Design**
  - High elastic modulus of ceramic joint materials provides significant challenges to joint design and characterization.
  - Understanding of stress state in the joints.

• **Materials Related Issues**
  - Optimization of in-plane tensile properties of CMCs by engineering the fiber/matrix interface is accomplished at the expense of interlaminar properties. Weak interfaces complicate joint properties and performance
    - Composition and microstructure
    - Bonding and adhesion
    - Testing and data analysis
  - High elastic modulus ceramic joint materials.

• **Life Time Testing for Specific Applications**
  - Time dependent thermomechanical properties of joints.
  - Environmental effects on joint properties.
Microstructure of As-Fabricated Joints in Monolithic SiC Ceramics

Sintered SiC (Hexoloy-SA)

CVD-SiC

Ecoceramics
African Bubinga
TEM Analysis of Reaction Formed Joints

SINTERED SiC

RB-SiC

SiC

JOINT

500 nm

60 μm

1 μm
Fracture Strength Distribution of Joined SiC (Hexoloy-SA) at Different Temperatures

- 25 C: 270 MPa, m = 7.7
- 1200 C: 302 MPa, m = 6.2
- 1350 C: 296 MPa, m = 13.7
- As-rec.: 395 MPa, m = 7.1

*Characteristic stress, probability of failure = 0.632
Flexural Strengths of Joined CVD SiC Ceramics

Average data for five specimens

No. of specimens unknown
Effect of Surface Roughness on the Shear Strength of Joined CVI C/SiC Composites

CVI C/SiC Composites

Joints with As-Fabricated Surfaces

Joints with As-Fabricated/Machined Surfaces

Joints with Machined Surfaces
Specimen Geometry and Test Fixture Used for Compression Double-Notched Shear Tests

ASTM C 1292-95a (RT) and ASTM C 1425-99 (HT)

Specimen Dimensions
- Specimen length (L): 30 mm (±0.10 mm)
- Distance between notches (h): 6 mm (±0.10 mm)
- Specimen width (W): 15 mm (±0.10 mm)
- Notch width (d): 0.50 mm (±0.05 mm)
- Specimen thickness (t): (adjustable)
Typical Stress-Strain Behavior Obtained During the Compression Double-Notched Shear Tests

![Graph showing stress-strain behavior at different temperatures](image)

- 1200C - a
- RT - b
- 800C - a
- 1350C - a
Compression Double Notch Shear Strength of Joined CVI SiC Composites at Different Temperatures

- Shear strength of joints increases with temperature and is higher than the CVI SiC composite substrate.
- No apparent influence of surface condition on the shear strength of joints.
Examples of Components Joined Using ARCJoinT

- Joined SiC Tubes for Wafer Fabrication System
- Carbon-Carbon Composite Valves for Race Car Engines
- Joined C/SiC Composites Attachment for Sensors
Summary and Conclusions

- Joining and assembly technologies are critically needed for the robust design and manufacturing of components.
- Braze/Solder effectiveness is dictated by several issues: wetting, spreading, bonding, and thermal mismatch.
- Thermal expansion mismatch between C-C/Braze/Titanium and interlaminar properties of C/C composites play a key role in mechanical behavior of joint.
  - CuSil ABA paste was most successful even though not the lowest temperature braze
  - S-Bond Solder had best shear strengths due to low processing temperature
- The purity control of metallic interlayer is also very critical for good joints.
- ARCJoinT process has been used to make several types of joints in SiC, C/SiC, and SiC/SiC composites. Joints in monolithic ceramics (CVD and Sintered SiC) show ~75% of the strength compared to bulk materials.
- In C/SiC composites, whether the joined surfaces are as-received (rough) or machined (smooth) has no effect on the shear strength of the joint. Furthermore, the shear strength of all joints exceeds that of the as-received C/SiC at elevated temperatures up to 1350 C.
- High elastic modulus of ceramic joints and weak interfaces in composite materials provide significant challenges to joint design and are critical to joint properties and performance.
- A combination of tensile, shear, and subcomponent testing of joints coupled with fracture mechanics based design and analysis is needed to generate useful engineering design data.