Ceramic Integration Technologies for Aerospace and Energy Systems: Technical Challenges and Opportunities

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Abstract

Ceramic integration technology has been recognized as an enabling technology for the implementation of advanced ceramic systems in a number of high-temperature applications in aerospace, power generation, nuclear, chemical, and electronic industries. Various ceramic integration technologies (joining, brazing, attachments, repair, etc.) play a role in fabrication and manufacturing of large and complex shaped parts of various functionalities. However, the development of robust and reliable integrated systems with optimum performance requires the understanding of many thermochemical and thermomechanical factors, particularly for high temperature applications. In this presentation, various challenges and opportunities in design, fabrication, and testing of integrated similar (ceramic-ceramic) and dissimilar (ceramic-metal) material systems will be discussed. Experimental results for bonding and integration of SiC based LDI fuel injector, high conductivity C/C composite based heat rejection system, solid oxide fuel cells system, ultra high temperature ceramics for leading edges, and ceramic composites for thermostructural applications will be presented. Potential opportunities and need for the development of innovative design philosophies, approaches, and integrated system testing under simulated application conditions will also be discussed.

Ceramic Integration Technologies for Aerospace and Energy Systems

Technical Challenges and Opportunities

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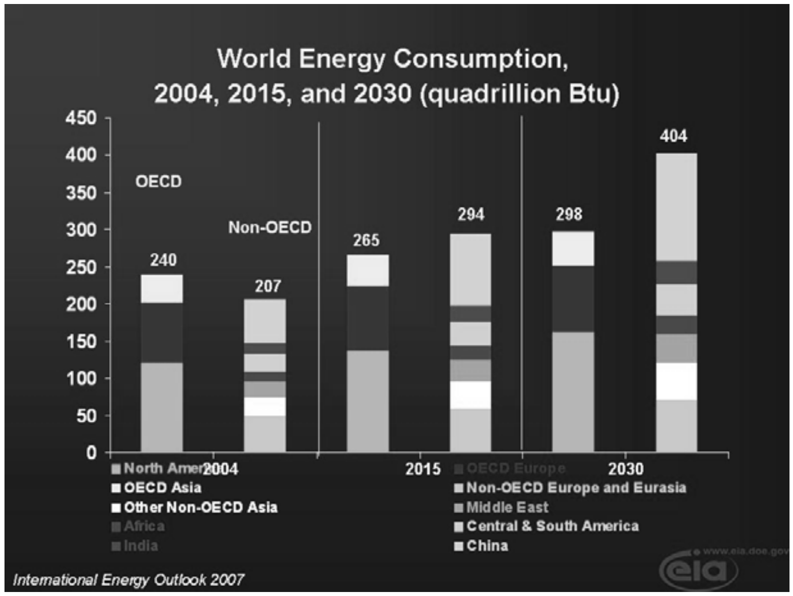
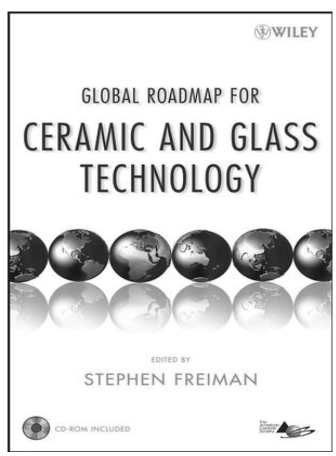
Overview

- **Introduction and Background**
  - ***Global Energy Issues and Role of Ceramics***
- **Technical Challenges in Integration**
  - ***Ceramic-Metal Systems***
  - ***Ceramic-Ceramic Systems***
- **Ceramic Integration Technologies**
  - ***High Temperature Systems:*** *Thermostructural components*
  - ***Energy Efficiency:*** *MEMS-LDI Fuel Injector, Thermal Management (Heat Exchangers and Recuperators, etc.)*
  - ***Alternative Energy:*** *SOFC Systems*
- **Concluding Remarks**
- **Acknowledgments**



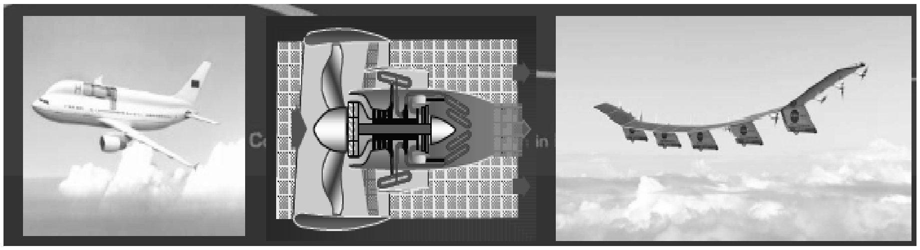
Critical Role of Advanced Ceramic Technologies in Energy and Aerospace Applications

- **Energy Production**
  - Fuel Cells, Thermoelectrics, Photovoltaics
  - Nuclear, Wind, Biomass
- **Energy Storage and Distribution**
  - Batteries, Capacitors, Hydrogen Storage Materials
  - High Temperature Superconductors
- **Energy Conservation and Efficiency**
  - Ceramic Components (Gas Turbines, Heat Exchangers, etc.), Coatings, Bearings



Stephen Freiman, Mrityunjay Singh, Gary Fischman, et al, ACerS-Wiley (2007).

Vision of Future Aircraft and Engine Configurations
[Broichhausen (2005), Szodruch (2005)]



Hydrogen **Intercooler/Recuperator** **Solar/Fuel Cells**

Lower & Slower

Technical Challenges in Integration of Ceramic-Metal *vs* Ceramic-Ceramic Systems

Ceramic-Metal System

- **Flow and wettability**

Ceramic-Ceramic System

- **Reaction and diffusion**

Common Issues

- Roughness
- Residual stress (ΔCTE)
- Multi-axial stress state
- Joint design
- Joint stability in service

Ceramic-Metal System

- **Metal – *forgiving***
- **Elastic-plastic system**
- **Lower use temperatures**
- **Less aggressive environment**

Ceramic-Ceramic System

- **Ceramic – *unforgiving***
- **Elastic-elastic system**
- **Higher use temperatures**
- **More aggressive environment**

Ceramic Integration Technologies

- Joining/Bonding
- Attachments, Fasteners/Rivets
- Repair

Affordable, Robust Ceramic Joining Technology (ARCJoinT)

Apply Carbonaceous Mixture to Joint Areas

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

⇩

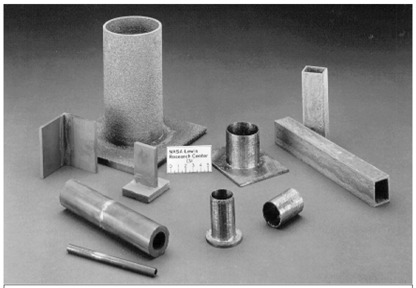
Apply Silicon or Silicon-Alloy (paste, tape, or slurry)

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.

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ARCJoinT can be Used to Join 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

ARCJoinT

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***

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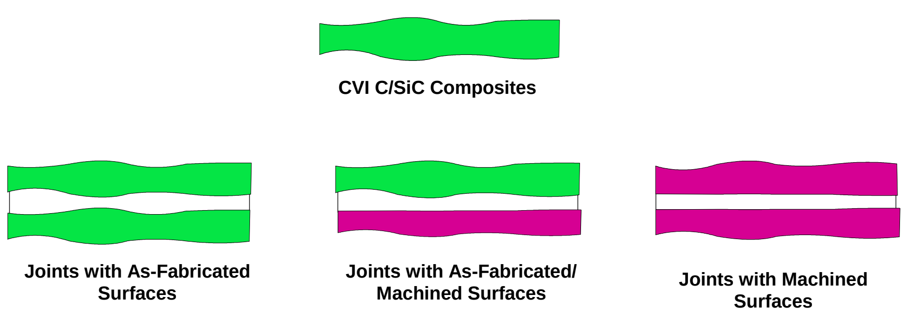
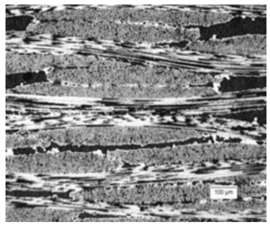
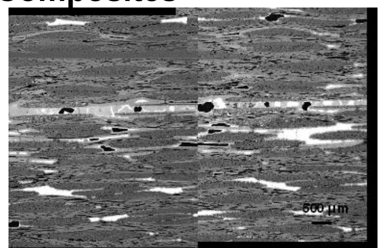
Effect of Surface Roughness on the Shear Strength of Joined CVI C/SiC Composites



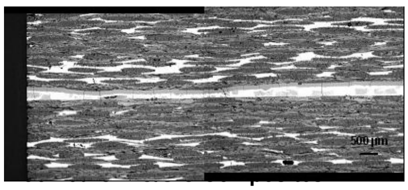
Microstructure of As-Fabricated and Joined CVI C/SiC Composites



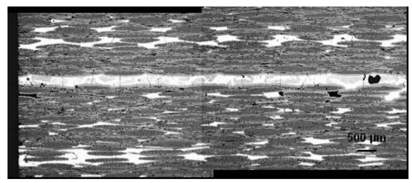
CVI C/SiC Composites (as fabricated)



Joined CVI C/SiC Composites (both surfaces machined)

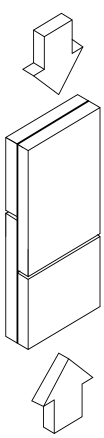


(one surface machined and one surfaces as received)



Joined CVI C/SiC Composites (both surfaces as received)

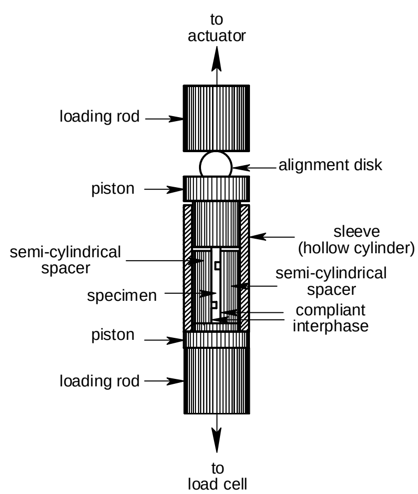
Specimen Geometry and Test Fixture Used for Compression Double-Notched Shear Tests



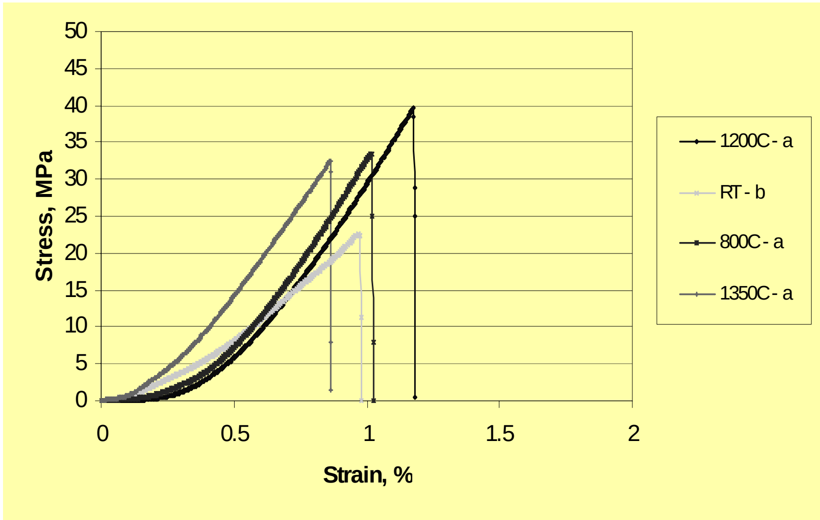
ASTM C 1292-95a (RT) and ASTM C 1425-99 (HT)

Specimen Dimensions

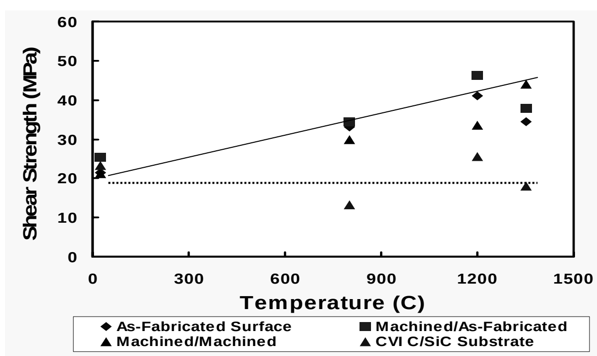
- **Specimen length (L) : 30 mm *(±0.10 mm)***
- **Distance between notches (h) : 6 mm *(±0.10 mm)***
- **Specimen width (W) : 15 mm *(±0.10 mm)***
- **Notch width (d) : 0.50 mm *(±0.05 mm)***
- **Specimen thickness (t) : (*adjustable)***



Typical Stress-Strain Behavior Obtained During the Compression Double-Notched Shear Tests



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.

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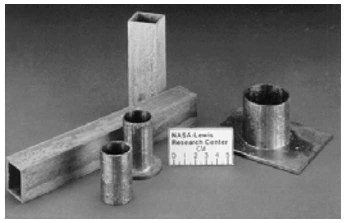
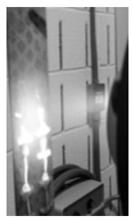
Integration of Advanced CMC-CMC Systems

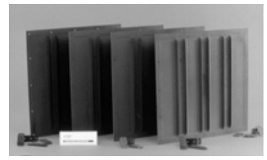
High Temperature CMC Joining

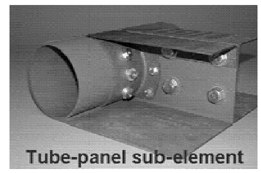


C-C Engine Valve **Attachments for Sensors**

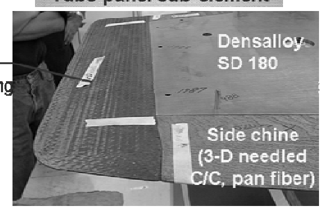


CMC Control Surfaces





Hyper-X Program Leading Edge



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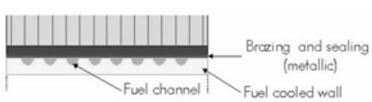
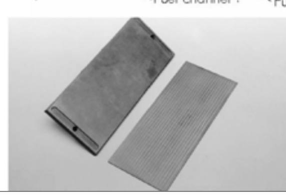
Integration of CMC-Metal and CMC-CMC System

LEA CMC Cooled Combustion Chamber

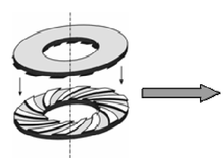


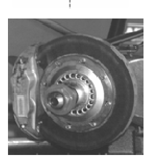


Development at Snecma and Onera through PWR & AFRL, A3CP program-tested at AFRL, Onera, and other test facilities

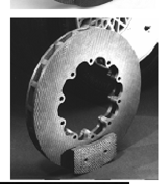
Joined C-C/SiC Intake Flap for HYTEX-German Hypersonic Technology Program (DLR)

Joining of CMCs for Ground Based Applications

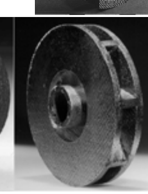




Prof. Dr. Walter Krenkel, Germany



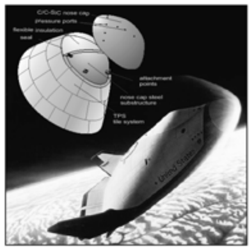
Heat Exchanger Substructure

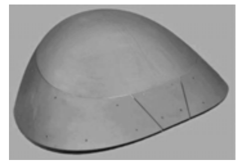


Radial Pump Wheel

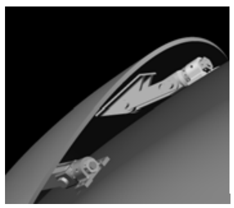
C-C/SiC Composites

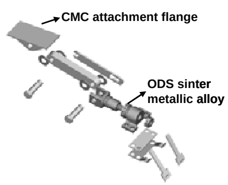
Ceramic Integration Technologies:
Attachment Systems/ Fasteners

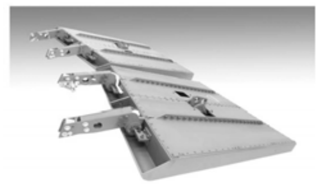




C/SiC Nosecap and Skirts
(DLR/Astrium and MT)





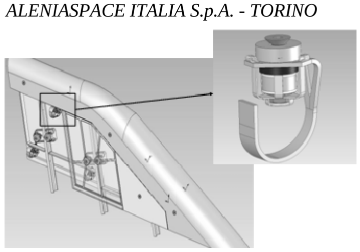


C/SiC body Flaps (MT Aerospace)

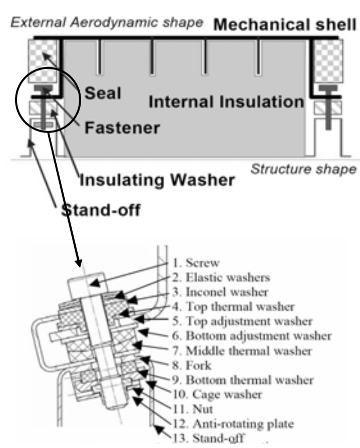
NASA-DLR-MT Aerospace (X-38 Program)

Ceramic Integration Technologies:
Attachment Systems/Fasteners

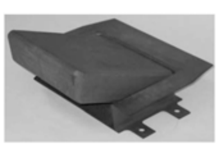
New CMC Attachment Design Concepts being developed at *THALES ALENIASPACE ITALIA S.p.A. - TORINO*

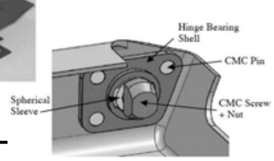


CMC Shingle TPS Attachment (Snecma)

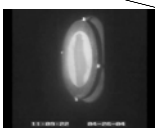


EXPERT CMC Open Flaps and Attachments developed at ***MT Aerospace***

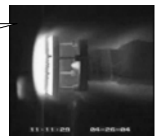




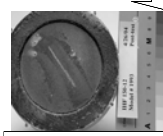
Glenn Refractory Adhesive for Bonding and Exterior Repair
(GRABER)



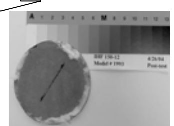
Arc Jet Testing
Front View



Arc Jet Testing
Side View



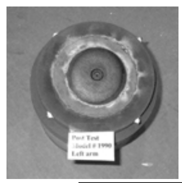
Post Test- Front Side



Post Test- Back Side

Multiuse Capability/Versatility of GRABER

- Repair of cracks, gouges, small holes, and missing surface coatings
- Edge sealant/adhesive for Plug concept
- Gap filler for T-seals and other areas
- Sealing the edges, gaps, attachment areas for flexible ceramic/metallic wrap concepts for large area damage repair
- Prepregs made with various ceramic fabrics are useful for various high temperature applications in aerospace and ground based systems.



- **Analogue RCC Plug Sealed with GRABER 5A Crack Sealant**
- **Survived the ArcJet Testing at JSC**

- **2005 R&D 100 Award**
- **Northern Ohio Live Magazine- Awards of Achievement, S&T Category- Runner Up**

Bonding and Integration of MEMS-LDI Fuel Injector

Objective: Develop Technology for a SiC Smart Integrated Multi-Point Lean Direct Injector (SiC SIMPL-DI)

- Operability at all engine operating conditions
- Reduce NOx emissions by 90% over 1996 ICAO standard
- Allow for integration of high frequency fuel actuators and sensors

Possible Injector Approaches

1. Lean Pre-Mixed Pre-Evaporated (LPP)

Advantages - Produces the most uniform temperature distribution and lowest possible NOx emissions

Disadvantages - Cannot be used in high pressure ratio aircraft due to auto-ignition and flashback

2. Lean Direct Injector (LDI)

Advantages - Does not have the problems of LPP (auto-ignition and flashback)

- Provides extremely rapid mixing of the fuel and air before combustion occurs

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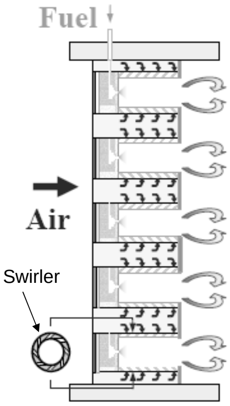
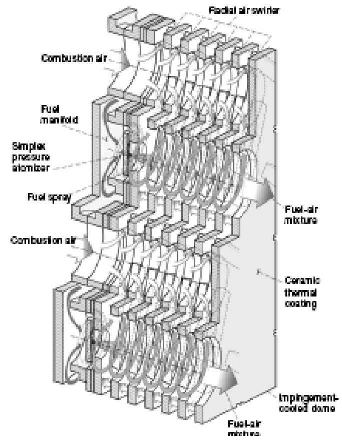
Multi-Point Lean Direct Injector





(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.

From Robert Tacina, et al., “A Low Lean Direct Injection, Multi-Point Integrated Module Combustor Concept for Advanced Aircraft Gas Turbines,” NASA/TM-2002-211347, April 2002.

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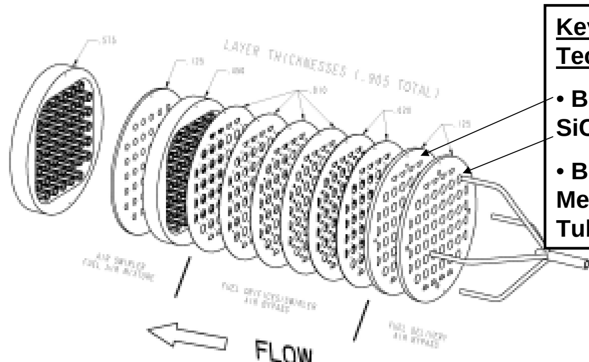
Lean Direct Injector Fabricated by Bonding of SiC Laminates

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 Enabling 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**

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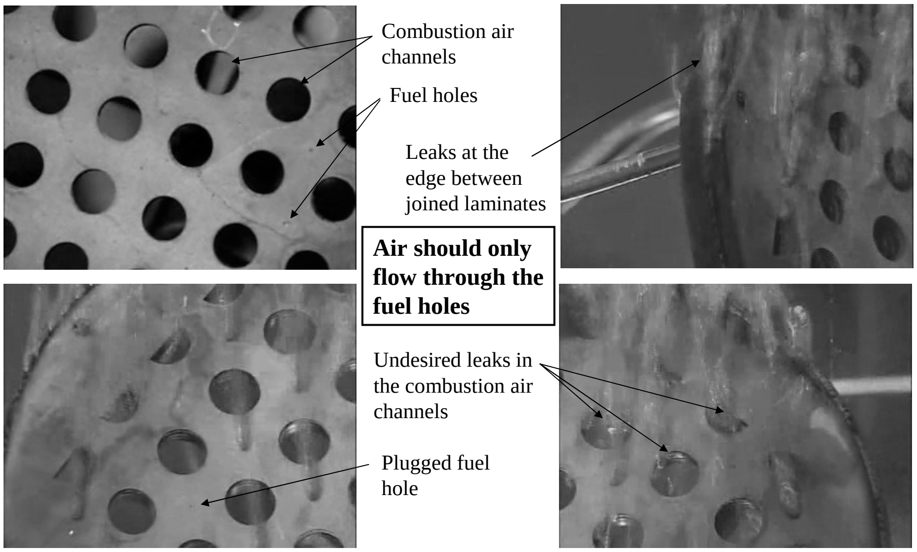
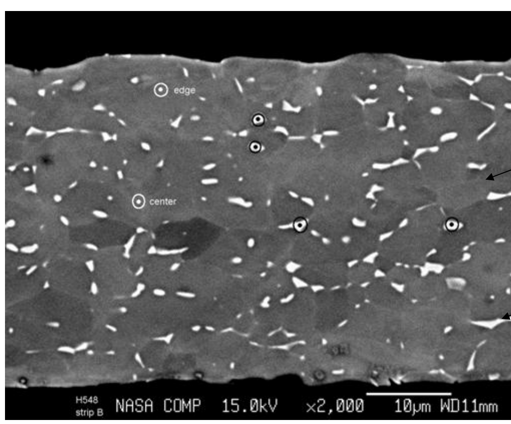
Leak Test of SiC Laminates Joined with Silicate Glass



Microprobe Analysis of Alloyed Ti Foil



Ti-6Al-4V (weight %)

Grey phase – Alpha alloy

White phase – Beta alloy

Microprobe from the cross-section of alloyed Ti foil (averages taken from several points near the edge and at the center of the foil)

	Phase	Al	Fe	Ti	V	Total
Atomic Ratio	Grey Phase	10.196	0.042	86.774	2.988	100.000
Weight (%)	Grey Phase	5.999	0.051	90.632	3.318	100.000
Atomic Ratio	White Phase	4.841	1.850	76.507	16.803	100.000
Weight (%)	White Phase	2.748	2.172	77.084	17.997	100.000

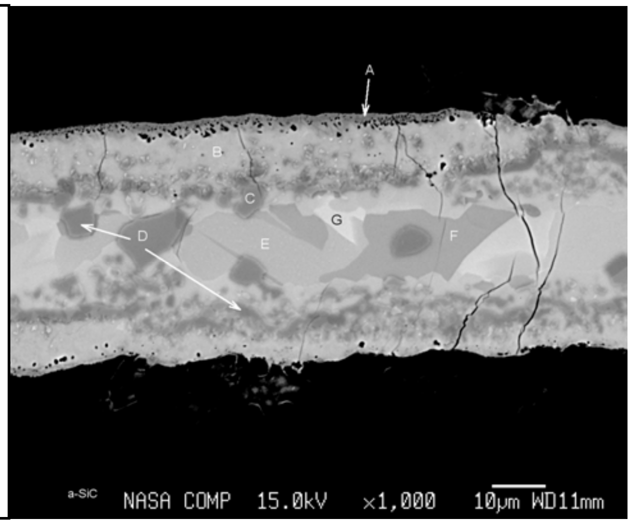
Microprobe of α-SiC Bonded Using Ti Foil

Conditions: 1250 ˚C, 24 MPa, 2 hr, vacuum, 5˚C/min

Microcracking may be due to the formation of two detrimental phases:

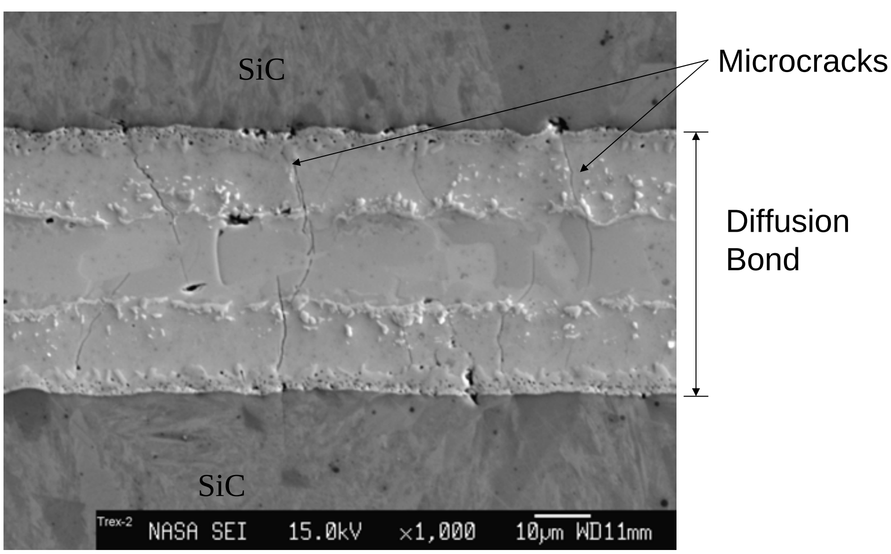
- Phase B $Ti_5Si_3C_X$ – Ti_5Si_3 is highly anisotropic in its thermal expansion where CTE(c)/CTE(a) = 2.72 (Schneibel et al).
- Phase E – Ti_3Al has low ductility at low temperatures. Al can be in the range of 23-35 atm % (Djanarthany et al).

Both phases can contribute to thermal stresses and microcracking during cool down.



M. Singh and M.C. Halbig, Key Engineering Materials Vol. 352 (2007) pp. 201-206

Secondary Electron Image of the Diffusion Bond - Alloyed Ti Foil and Trex CVD SiC

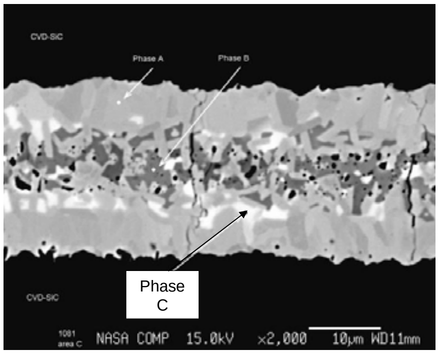


Diffusion Bonding of CVD-SiC Using PVD Ti Interlayer

20 Micron Ti Interlayer

Microcracking is still present due to the presence of $Ti_5Si_3C_X$.

Naka et al suggest that this is an intermediate phase.



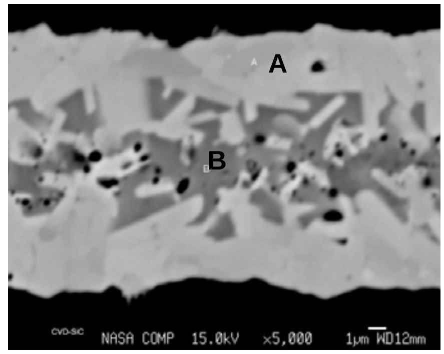
Phases in bond with the 20 μ Ti Interlayer – Atomic Ratios

Phase	Ti	Si	C
Phase A	56.426	17.792	25.757
Phase B	35.794	62.621	1.570
Phase C	58.767	33.891	7.140

10 Micron Ti Interlayer

No microcracking or phase of $Ti_5Si_3C_X$ is present.

Thin interlayers of pure Ti down-selected as the preferred interlayer.

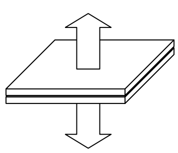


Phases in bond with the 10 μ Ti Interlayer – Atomic Ratios

Phase	Ti	Si	C
SiC	0.011	54.096	45.890
Phase A	56.621	18.690	24.686
Phase B	35.752	61.217	3.028

High Strength of Bonds Greatly Exceeds the Application Requirements

1" x 1" Bonded Substrates



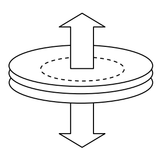
Pull test tensile strengths:

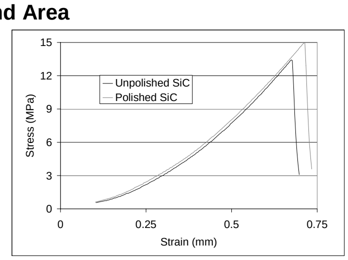
> 23.6 MPa (3.4 ksi)*

> 28.4 MPa (4.1 ksi)*

* failure in the adhesive to the test fixture

1" Diameter Discs with a 0.65" Diameter Bond Area





Pull test tensile strengths:

13.4 MPa (1.9 ksi)

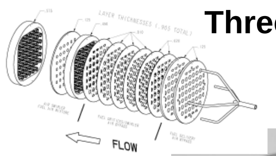
15.0 MPa (2.2 ksi)

Slightly higher strength from the highly polished SiC suggests that a smoother surface contributes to stronger bonds or less flawed SiC.

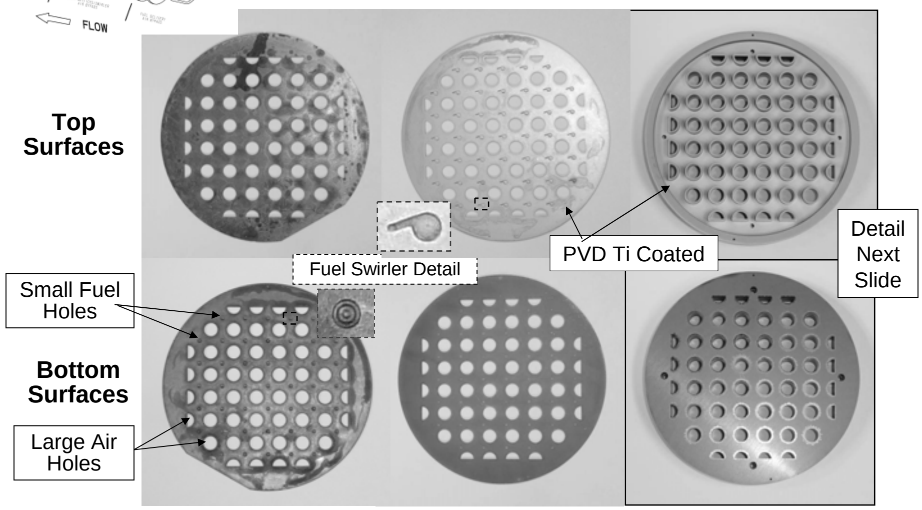
Failures are primarily in the SiC substrate rather than in the bond area.

The injector application requires a strength of about 3.45-6.89 MPa (0.5 - 1.0 ksi).

Three Part 10 cm (4”) Diameter SiC Injector

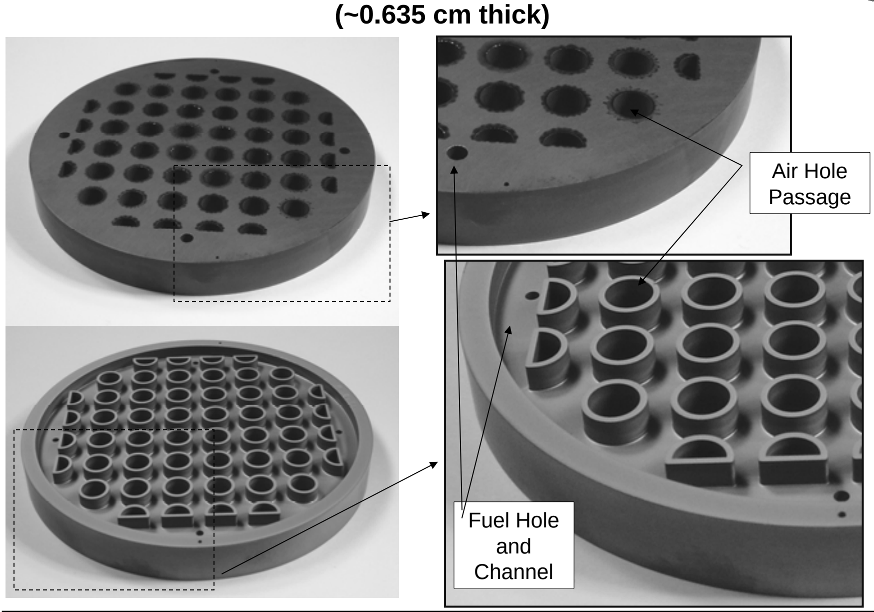


Stacking Sequence
Top to Bottom

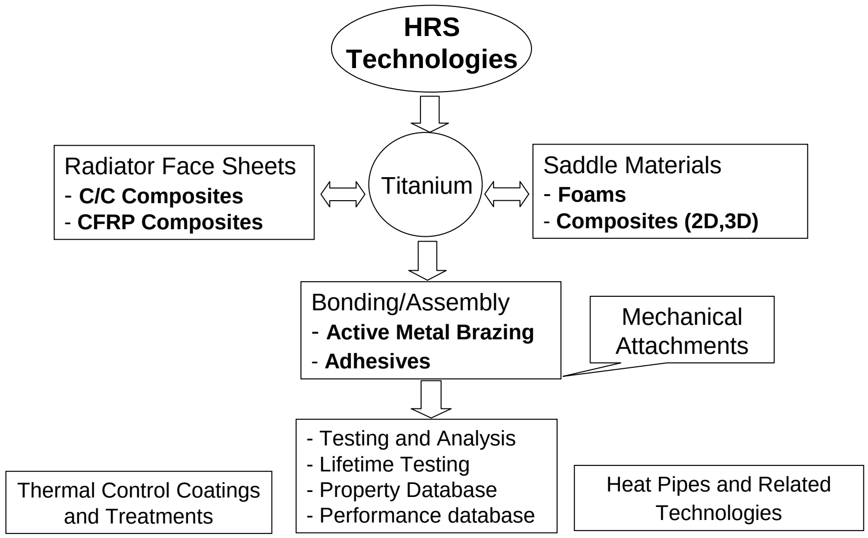


Detail of the Thickest Injector Substrate

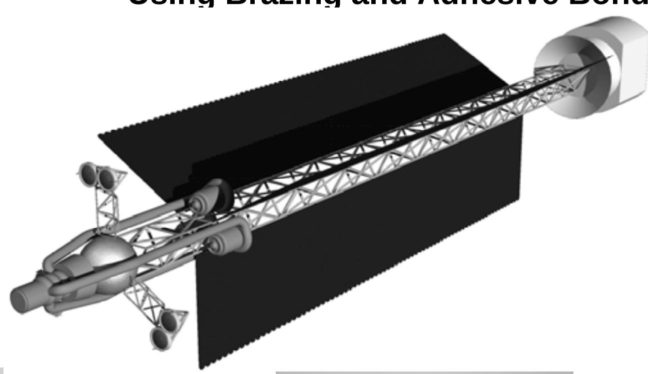
(~0.635 cm thick)

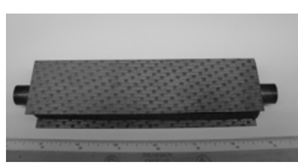


Integration Needs in Heat Rejection System

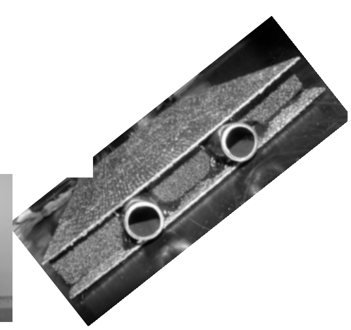


Integration of Sub-elements for Heat Rejection System Using Brazing and Adhesive Bonding



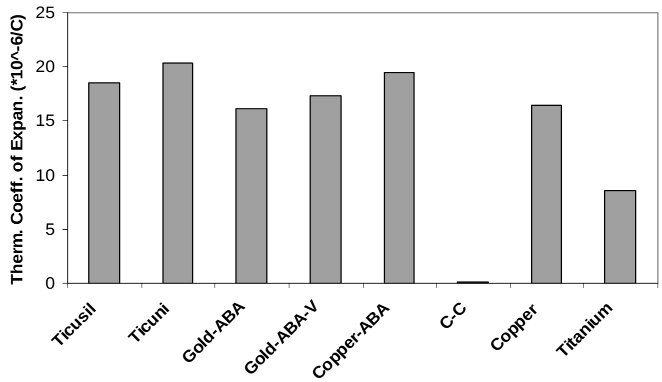


Brazed Radiator Sub-elements



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.

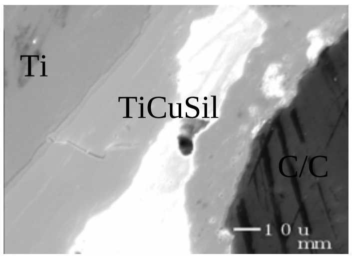
Active Metal Brazing

- **Ti tubes and plates brazed to P120 CVI C/C composite**
- **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

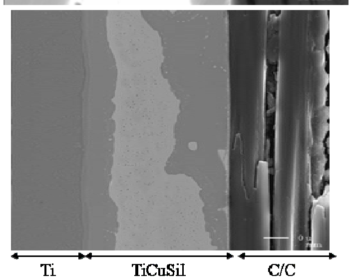
• ***Require*** ***good wetting, bonding and spreading properties***
• ***Desire*** ***minimal residual stress induced cracking in C/C***

Microstructure of Brazed Ti and C-C Composites using TiCuSil and CuSil ABA Paste

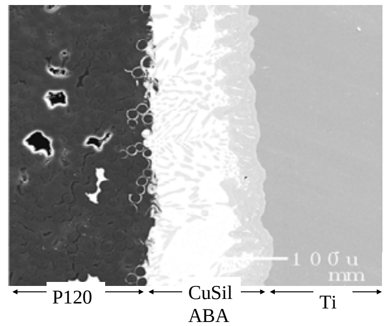
Ti- Tube



Ti- Plate

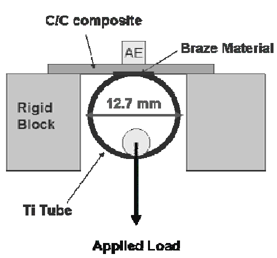


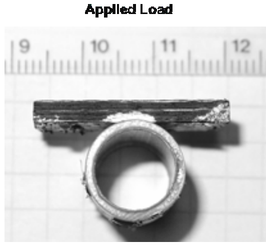
Titanium Plate



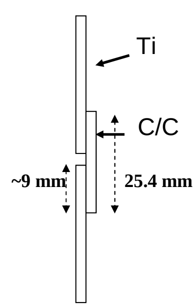
Mechanical Testing of Brazed/Soldered Joints

Tube Tensile Test



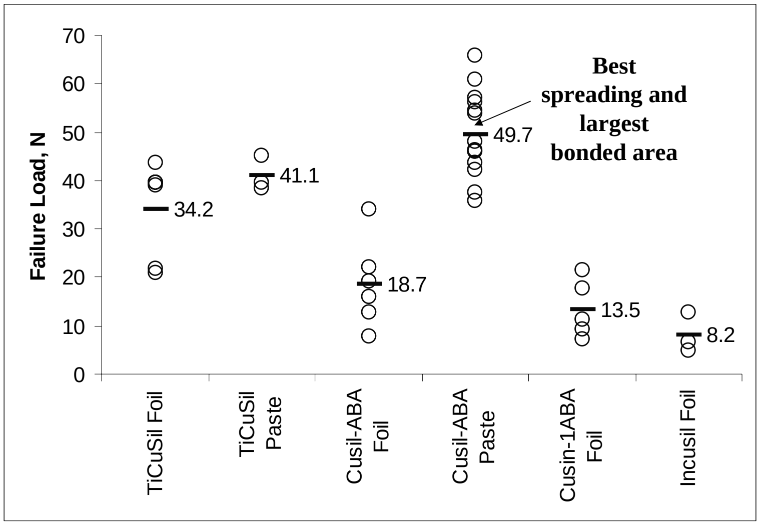


Butt Strap Tensile Test

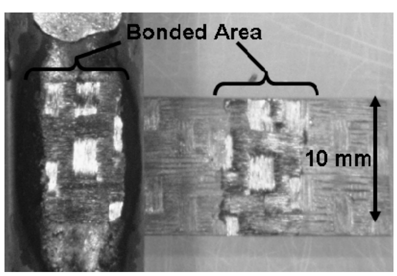


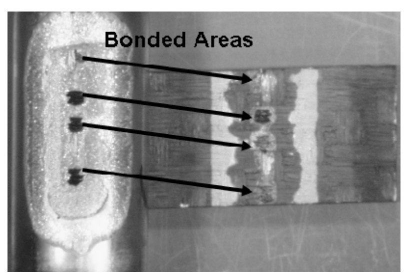
Factors to consider:
-Braze composition, Processing variables
-Bonded area, Location of failure
-Architecture effects

Tube Tensile Test Data for Brazed Joints



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).

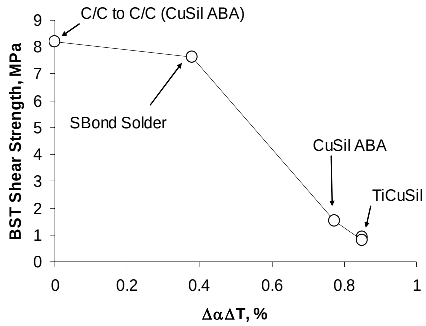
Tube and C/C plate fracture surfaces for Incusil ABA foil braze material showing the distinct bonded areas of the outer ply of C/C brazed to the Ti tube (left) and C/C plate (right).

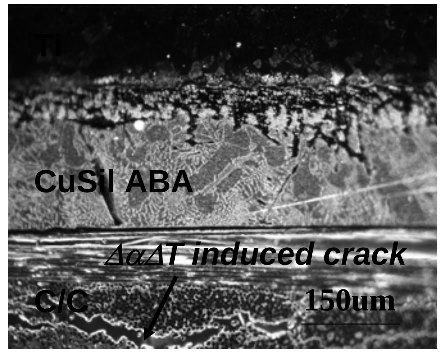
Test data on a wide variety of brazes reported in Mater. Sci. Engg. A, 412 (2005) 123-128 and Mater. Sci. Engg. A., 418 (2006) 19-24.

National Aeronautics and Space Administration

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





$\Delta\alpha = \alpha$ (Ti) – α (C/C)

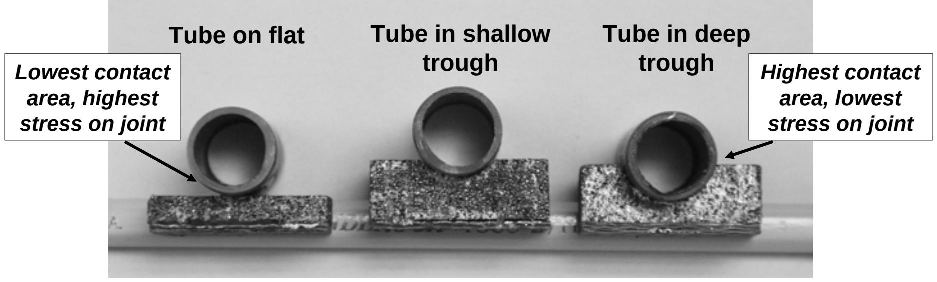
ΔT = T (liquidus ~ processing) – 25ºC

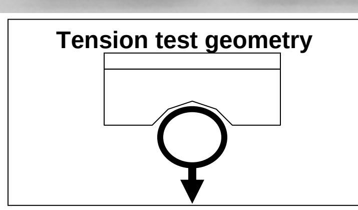
Joint Material	Proc. Temp., C
S-Bond	~ 400
CuSil ABA	830
TiCuSil	910

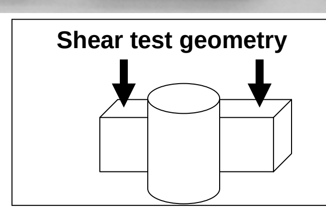
National Aeronautics and Space Administration

Brazed Structures with Woven K1100 and P120 C/C Facesheet/Foam/Titanium Tube

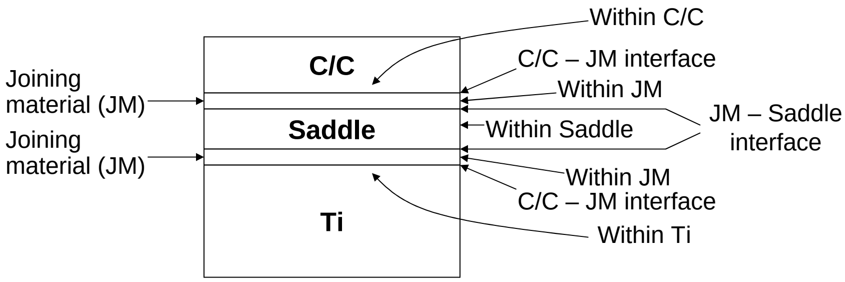
Specimens with different tube-foam contact areas were fabricated in order to vary braze contact area and stress applied to joints







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…

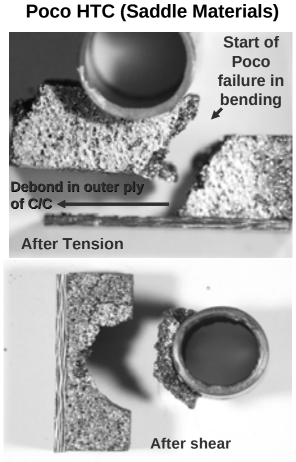
Therefore, knowing the location of joint failure is critical

- **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

Brazed Structures with Woven K1100 and P120 C/C Facesheet/Foam/Titanium Tube

- **Excellent bonding of CuSil-ABA Braze to Poco HTC, C-C Composite, and Ti Tube**
- **Failure always occurred in Poco HTC (Saddle Materials)**



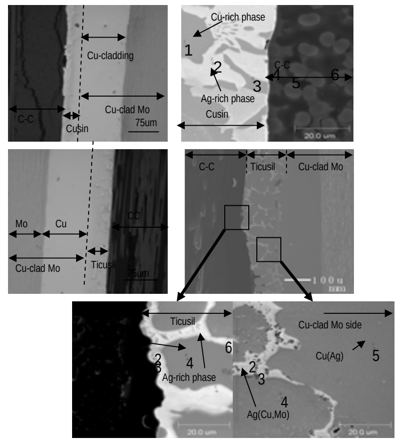
Observations:

- The Poco HTC (carbon foam saddle material) is the weak link in the sandwich structures and not the brazed joint.
- Failure in tension and shear always occurs in the foam regardless of (1) P120 or K1100 woven face sheet materials or (2) whether the Ti tube was brazed to a curved Poco surface to maximize bond area or a flat Poco surface to maximize stress in the joint.
  – Maximum *shear stresses* subjected to braze *exceeded 12 MPa* based on load applied and approximate braze area.
  – Maximum *tensile stresses* subjected to braze *exceeded 7 MPa* based on load applied and approximate braze area.

M. Singh, G. Morscher, R. Asthana, T. Shpargel, Mater. Sci. Eng., A 2007 in press.

C-C Composite/Cu-Clad-Mo Joints for Thermal Management Applications

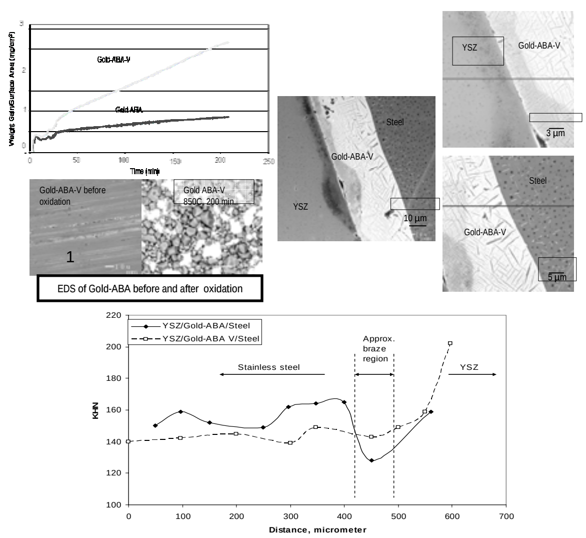
- C-C composites were brazed to Cu-clad Mo using four ABA's (Cu-ABA, Cusin, Ticusni and Ticusil).
- Good metallurgical bonding at joints, with some dissolution, diffusion, and solute redistribution.
- Ti preferentially segregated at the C-C/braze interface. Cu-ABA joints displayed the largest Ti concentrations at joint.
- Microhardness gradients exist at joint (peak HK is 300-350 in Cusin-1 ABA and Ticusil, and 200-250 HK in Cu-ABA and Ticuni).
- C-C/Cu-clad Mo systems are useful for thermal management applications.



M. Singh, R. Asthana, T. Shpargel, Mater. Sci. Eng., A 452-453, 2007, 699-704

Integration of YSZ/Steel for SOFC Applications

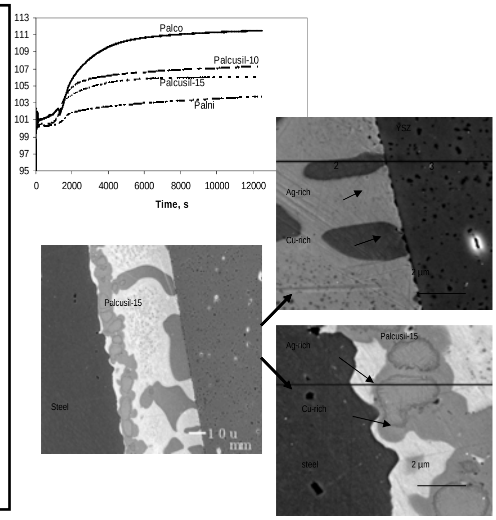
- **Gold-ABA and Gold ABA-V exhibit linear oxidation kinetics at 850 C. Gold-ABA-V shows faster oxidation kinetics than Gold-ABA.**
- **Ti in Gold-ABA and V in Gold-ABA V caused discoloration (darkening) of YSZ.**
- **The darkening is caused by Ti and V that act as oxygen getters and form oxygen-deficient YSZ. No reaction layers formed at joint.**
- **The oxygen-deficient YSZ is better wet by gold than stoichiometric YSZ.**



M. Singh, T. Shpargel, R. Asthana, Int. J. Appl. Ceram. Tech. 4(2), 2007, 119-133.

Integration of YSZ/Steel for SOFC Applications

- Pd-base brazes (Palco and Palni) and Ag-base brazes (Palcusil-10 and Palcusil-15) were characterized for oxidation at 750ºC.
- Structural changes accompany oxidation which is fastest for Palco, slowest for Palni, and intermediate for Palcusil-10 and Palcusil-15.
- All brazes were effective in joining yttria stabilized zirconia (YSZ) to stainless steel for solid oxide fuel cell (SOFC).
- Dissolution of YSZ and steel in braze, and braze constituents in YSZ and steel led to diffusion and metallurgically sound joints.
- Knoop hardness (HK) profiles are similar for all brazes, and exhibit a sharp discontinuity at the YSZ/braze interface.

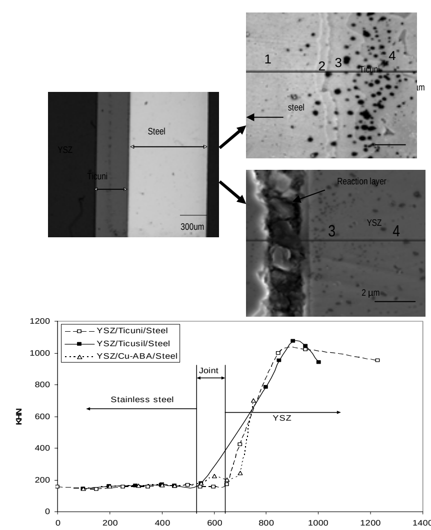


M. Singh, T. Shpargel, R. Asthana, Mater. Sci. Eng. A (2007 in press)

National Aeronautics and Space Administration

Integration of YSZ/Steel for SOFC Applications

- Active braze alloys, Cu-ABA, Ticuni and Ticusil, were characterized for oxidation at 750-850ºC.
- Oxidation is fastest for Ticusil, slowest for Cu-ABA, and intermediate for Ticuni.
- Brazes were used for joining yttria-stabilized-zirconia (YSZ) to stainless steel for Solid Oxide Fuel Cell (SOFC) applications.
- Interdiffusion, compositional changes, and reaction layer formation led to high-integrity joints.



M. Singh, T. Shpargel, R. Asthana, J. Mater. Sci (2007 in press)

Concluding Remarks

- Ceramic integration technologies are critically needed for the successful development and applications of ceramic components in a wide variety of energy and aerospace applications.
- Significant efforts are needed in developing joint design methodologies, understanding the size effects, and thermomechanical performance of integrated systems in service environments.
- Global efforts on standardization of integrated ceramic testing are required. In addition, development of life prediction models for integrated components is also needed.
- There have been a number of short term design, development, and evaluation efforts in various parts of the world. However, a concerted and long term sustained effort is needed to make the significant progress in this area.

Acknowledgements

- **Dr. Gregory N. Morscher, Ohio Aerospace Institute**
- **Mr. Michael H. Halbig, US Army VTD**
- **Prof. Rajiv Asthana, University of Wisconsin-Stout**
- **Ms. Tarah Shpargel and Mr. Ron Phillips, ASRC Corp.**
- **Mr. Ray Babuder, Case Western Reserve University**