Advanced ceramic bonding and integration technologies play a critical role in the fabrication and application of silicon carbide based components for a number of aerospace and ground based applications. One such application is a lean direct injector for a turbine engine to achieve low NOx emissions. Ceramic to ceramic diffusion bonding and ceramic to metal brazing technologies are being developed for this injector application. For the diffusion bonding, titanium interlayers (PVD and foils) were used to aid in the joining of silicon carbide (SiC) substrates. The influence of such variables as surface finish, interlayer thickness (10, 20, and 50 microns), processing time and temperature, and cooling rates were investigated. Microprobe analysis was used to identify the phases in the bonded region. For bonds that were not fully reacted an intermediate phase, Ti5Si3Cx, formed that is thermally incompatible in its thermal expansion and caused thermal stresses and cracking during the processing cool-down. Thinner titanium interlayers and/or longer processing times resulted in stable and compatible phases that did not contribute to microcracking and resulted in an optimized microstructure. Tensile tests on the joined materials resulted in strengths of 13-28 MPa depending on the SiC substrate material. Non-destructive evaluation using ultrasonic immersion showed well formed bonds. For the joining technology of brazing Kovar fuel tubes to silicon carbide, preliminary development of the joining approach has begun. Various technical issues and requirements for the injector application are addressed.
Bonding and Integration Technologies for Silicon Carbide Based Injector Components

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Outline

• **Application** – Micro-Electro-Mechanical Systems Lean Direct Injector (MEMS LDI) for Advanced Aircraft Gas Turbines

• **Joint Processing** - Diffusion Bonding With a Titanium Interlayer

• **Characterization** – SEM, Microprobe, NDE, Pull Tests

• **Near Term Plans** – Subcomponent Fabrication

• **Brazing** – Kovar to SiC

• **Summary and Conclusions**
Motivation

Improve Ceramic to Ceramic and Ceramic to Metal Joining Methods
- allow for the fabrication of complex shapes and structures.
- allow for easier integration of ceramics components into engine systems.

*Ceramics can be either monolithics or fiber reinforced composites*.

Examples of Component Applications Benefiting from Joining Technology
– components for aeronautic and ground based engine applications (i.e. ceramic turbine vanes, blades, injectors, rotors, combustor liners, valves, and heat shields) and fusion reactor components, optical space components, and bi-layer armor.

Benefits – simplified component fabrication, enabling technology, increased operating temperatures, improved efficiency, and reduced cooling requirements.
Joining Approaches - Mechanical Fasteners

A wide variety of ceramic nuts, bolts, and washers can be made out of alumina or zirconia.

Issues with Mechanical Fasteners

- Difficult and costly to machine fastener parts
- Articles to be joined must have holes machined
- Limited material options for commercially available mechanical fasteners
- Must be chemically and thermally compatible with the articles to be fastened
- High mechanical and thermal loads on the fastener
- Slots require tight tolerances and must be airtight
Joining Approaches— Chemical Joining

- **Fusion Welding** – mating surface regions of components are melted and mixed before solidifying to form a permanent bond.

- **Brazing** – liquid metal flows into a narrow gap between the mating surfaces and solidifies to form a permanent bond.

- **Glazing and Glass Sealing** – uses a fluid glass to bond mating surfaces in processes analogous to brazing or fusion welding.

- **Adhesive Bonding** – component gaps are filled by fluid organic compounds that polymerize to form rigid bonding interlayers.

- **Diffusion Bonding** – mating surfaces are pressed together and heated to cause bonding by interdiffusion of the components.

- **High Temperature Reactive Joining** - reactive formation of high temperature capable joints (ARCJoinT).

Focus of Current Efforts on Diffusion Bonding Using a Ti Interlayer
Joining Approaches

Examples of Complex Shapes and Components

Examples of SiC ceramics and composite sub-elements joined using ARCJoinT (Jay Singh, NASA GRC)

Nascimento et al, Cerâmica vol.49 no.312
Fabrication of Lean Direct Injector Components by Diffusion 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
Leak Test of SiC Laminates Joined with Silicate Glass

- Combustion air channels
- Fuel holes
- Leaks at the edge between joined laminates

Air should only flow through the fuel holes

- Undesired leaks in the combustion air channels
- Plugged fuel hole
Previous Approach of Joining SiC With a Silicate Glass Layer

Disadvantages of Joining Silicon Carbide with a Silicate Glass Layer
- Difficult to achieve a uniform layer
- Relatively low strength
- Glass flows and fills in holes and edges where it is not desired
- Glass joints were not leak-free

Leak Test Movie
Current Approach of Joining SiC With a Ti Layer

Advantages of Diffusion Bonding Using a Ti Layer

- Uniform Ti layers can be applied
- Ti can be applied by different methods (foil, PVD and other coating approaches)
- High strength and leak-free bonds
- Good high temperature stability

The objective is to develop joining technology that has the following capabilities necessary for the injector application:

- Joining of relatively large geometries (i.e. 4” diameter discs)
- Leak-free at an internal pressure of 200 psi (1.38 MPa)
- Stability and strength retention at 800°F (427°C)
SiC-Ti-SiC Diffusion Bond Processing Matrix
- first processing matrix investigated

SiC and Ti Material Combinations:
1. 1.75” diameter α-SiC (CRYSTAR from Saint-Gobain) discs joined with a 38 micron alloyed Ti foil
2. 1.75” diameter CVD SiC (TREX Enterprises) discs joined with a 38 micron alloyed Ti foil
3. 1” x 2” CVD SiC (Rohm & Hass) coupons joined with ~10 micron PVD Ti coating on one of the surfaces
4. 1” x 2” CVD SiC (Rohm & Hass) coupons joined with a 38 micron alloyed Ti foil
5. 1” x 2” CVD SiC (Rohm & Hass) coupons joined with ~10 micron PVD Ti coating on both of the surfaces (20 micron total PVD Ti)

<table>
<thead>
<tr>
<th>Condition</th>
<th>Temp. (°C)</th>
<th>Pressure* (MPa)</th>
<th>Time (hr)</th>
<th>Atmosphere</th>
<th>Cooling Rate (°C/min)</th>
<th>Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>A (materials 1, 2, and 3)</td>
<td>1250</td>
<td>24, 24, 31</td>
<td>2</td>
<td>vacuum</td>
<td>5</td>
<td>Microscopy &amp; Microprobe</td>
</tr>
<tr>
<td>B (materials 1 and 3)</td>
<td>1300</td>
<td>24, 31</td>
<td>2</td>
<td>vacuum</td>
<td>2</td>
<td>Microscopy</td>
</tr>
<tr>
<td>C (materials 1 and 3)</td>
<td>1250</td>
<td>50</td>
<td>2</td>
<td>vacuum</td>
<td>2</td>
<td>Microscopy</td>
</tr>
<tr>
<td>D (materials 1, 4 and 5)</td>
<td>1250</td>
<td>24, 31</td>
<td>2</td>
<td>vacuum</td>
<td>2</td>
<td>Microscopy &amp; Microprobe</td>
</tr>
</tbody>
</table>

*at minimum clamping pressure for the hot press
Alloyed Ti Foil

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

<table>
<thead>
<tr>
<th>Phase</th>
<th>Al</th>
<th>Fe</th>
<th>Ti</th>
<th>V</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic Ratio</td>
<td>Grey Phase</td>
<td>10.196</td>
<td>0.042</td>
<td>86.774</td>
<td>2.988</td>
</tr>
<tr>
<td>Weight (%)</td>
<td>Grey Phase</td>
<td>5.999</td>
<td>0.051</td>
<td>90.632</td>
<td>3.318</td>
</tr>
<tr>
<td>Atomic Ratio</td>
<td>White Phase</td>
<td>4.841</td>
<td>1.850</td>
<td>76.507</td>
<td>16.803</td>
</tr>
<tr>
<td>Weight (%)</td>
<td>White Phase</td>
<td>2.748</td>
<td>2.172</td>
<td>77.084</td>
<td>17.997</td>
</tr>
</tbody>
</table>

Ti-6Al-4V (weight %)

Grey phase – Alpha alloy
White phase – Beta alloy
Secondary Electron Image of the Diffusion Bond
Alloyed Ti Foil and Trex CVD SiC

Microcracks
Diffusion Bond

SiC
Secondary Electron Image of the Diffusion Bond
Alloyed Ti Foil and Alpha-SiC
Microprobe of α-SiC Reaction Bonded Using Ti Foil
Conditions: 1250 °C, 24 MPa, 2 hr, vacuum, 5 °C/min

Microcracking may be due to thermal stresses during cooling down from processing:

Phase B Ti$_5$Si$_3$C$_X$ (Ti$_5$Si$_3$) is highly anisotropic in its thermal expansion where CTE(c)/CTE(a) = 2.72 (Schneibbel et al).

Phase E is a beta Ti alloy phase and phase F is an alpha Ti alloy phase.

### Alpha-SiC and alloyed Ti foil - Atomic Ratios

<table>
<thead>
<tr>
<th>Phase</th>
<th>Al</th>
<th>V</th>
<th>Cr</th>
<th>Si</th>
<th>C</th>
<th>Ti</th>
<th>Fe</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase A</td>
<td>0.020</td>
<td>0.970</td>
<td>0.023</td>
<td>7.662</td>
<td>34.787</td>
<td>56.531</td>
<td>0.007</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase B</td>
<td>0.076</td>
<td>0.923</td>
<td>0.020</td>
<td>34.151</td>
<td>6.502</td>
<td>58.220</td>
<td>0.108</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase C</td>
<td>0.223</td>
<td>0.760</td>
<td>0.019</td>
<td>6.015</td>
<td>32.269</td>
<td>60.690</td>
<td>0.025</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase D</td>
<td>21.862</td>
<td>2.363</td>
<td>0.050</td>
<td>0.432</td>
<td>10.394</td>
<td>64.865</td>
<td>0.036</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase E</td>
<td>27.102</td>
<td>21.318</td>
<td>0.611</td>
<td>0.473</td>
<td>1.901</td>
<td>46.456</td>
<td>2.141</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase F</td>
<td>25.975</td>
<td>5.962</td>
<td>0.145</td>
<td>1.186</td>
<td>1.898</td>
<td>64.666</td>
<td>0.168</td>
<td>100.000</td>
</tr>
</tbody>
</table>
Central core of diffusion bond has concentrated alpha and beta Ti alloy phases. The alpha phase has an anisotropic thermal expansion which is 20% greater along the c-axis (Boyer, Welsch, and Colling). Also, the beta phase has a thermal expansion that is 6x higher in the temperature range of 600-1000°C (5.8x10⁻⁵/°C) compared to the thermal expansion below 600°C (9.2x10⁻⁶/°C) (Elmer et al).
Microprobe of TREX CVD SiC Reaction Bonded Using Ti Foil
Conditions: 1250 °C, 24 MPa, 2 hr, vacuum, 5 °C/min

Microcracking is observed and the following phases are likely contributors:

Phase B: Ti₅Si₃Cₓ (Ti₅Si₃)
Phase E: beta Ti alloy phase
(microprobe analysis may have missed the presence of an alpha Ti alloy phase)

TREX SiC and alloyed Ti foil – Atomic Ratios

<table>
<thead>
<tr>
<th>Phase</th>
<th>Al</th>
<th>V</th>
<th>Cr</th>
<th>Si</th>
<th>C</th>
<th>Ti</th>
<th>Fe</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase A</td>
<td>0.101</td>
<td>0.578</td>
<td>0.026</td>
<td>18.733</td>
<td>24.317</td>
<td>56.243</td>
<td>0.004</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase B</td>
<td>0.384</td>
<td>1.220</td>
<td>0.025</td>
<td>34.639</td>
<td>6.904</td>
<td>56.794</td>
<td>0.034</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase C</td>
<td>0.604</td>
<td>0.715</td>
<td>0.023</td>
<td>3.148</td>
<td>35.366</td>
<td>60.124</td>
<td>0.020</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase D</td>
<td>19.887</td>
<td>1.424</td>
<td>0.026</td>
<td>0.537</td>
<td>13.719</td>
<td>64.381</td>
<td>0.025</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase E</td>
<td>26.941</td>
<td>14.935</td>
<td>0.396</td>
<td>0.588</td>
<td>0.747</td>
<td>55.684</td>
<td>0.710</td>
<td>100.000</td>
</tr>
</tbody>
</table>
Microprobe of Rohm & Haas SiC Reaction Bonded Using Ti Foil

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

Microcracking is again observed and the following phases are likely contributors:

Phase B: Ti$_5$Si$_3$C$_x$ (Ti$_5$Si$_3$)
Phase E: alpha Ti alloy phase
Phase F: beta Ti alloy phase

<table>
<thead>
<tr>
<th>Phase</th>
<th>Al</th>
<th>V</th>
<th>Cr</th>
<th>Si</th>
<th>C</th>
<th>Ti</th>
<th>Fe</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase A</td>
<td>0.070</td>
<td>0.848</td>
<td>0.030</td>
<td>17.085</td>
<td>25.802</td>
<td>56.164</td>
<td>0.001</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase B</td>
<td>0.303</td>
<td>0.961</td>
<td>0.018</td>
<td>33.766</td>
<td>6.675</td>
<td>58.212</td>
<td>0.064</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase C</td>
<td>0.293</td>
<td>0.661</td>
<td>0.010</td>
<td>5.234</td>
<td>35.671</td>
<td>58.122</td>
<td>0.010</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase D</td>
<td>19.002</td>
<td>1.392</td>
<td>0.039</td>
<td>0.557</td>
<td>12.707</td>
<td>66.288</td>
<td>0.014</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase E</td>
<td>23.244</td>
<td>7.649</td>
<td>0.213</td>
<td>0.758</td>
<td>3.970</td>
<td>64.104</td>
<td>0.062</td>
<td>100.000</td>
</tr>
<tr>
<td>Phase F</td>
<td>19.833</td>
<td>20.784</td>
<td>0.634</td>
<td>0.514</td>
<td>4.622</td>
<td>52.302</td>
<td>1.313</td>
<td>100.000</td>
</tr>
</tbody>
</table>
Summary of Diffusion Bonds Formed With Alloyed Ti Foil Interlayers Between Different SiC Substrates

Microcracks formed regardless of the substrate and variations in the processing: higher temperatures, higher pressures, slower cooling rate.

Microcracking is very likely due to thermal stresses during cooling down from processing.

The CTE issues of the alpha and beta Ti alloy phases can be avoided if pure Ti is used.

The anisotropic Ti$_5$Si$_3$C$_x$ phase could possibly be avoided with different processing conditions.
Diffusion Bonds from Using PVD Ti as the Interlayer

20 Micron Ti Interlayer

10 Micron Ti Interlayer
**Diffusion Bonds from Using PVD Ti as the Interlayer**

**20 Micron Ti Interlayer**
Microcracking is still present due to the presence of $\text{Ti}_5\text{Si}_3\text{C}_x$.
Naka et al suggest that this is an intermediate phase.

**10 Micron Ti Interlayer**
No microcracking or phase of $\text{Ti}_5\text{Si}_3\text{C}_x$ is present.

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

---

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

<table>
<thead>
<tr>
<th>Phase</th>
<th>Ti</th>
<th>Si</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase A</td>
<td>56.426</td>
<td>17.792</td>
<td>25.757</td>
</tr>
<tr>
<td>Phase B</td>
<td>35.794</td>
<td>62.621</td>
<td>1.570</td>
</tr>
<tr>
<td>Phase C</td>
<td>58.767</td>
<td>33.891</td>
<td>7.140</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>Phase</th>
<th>Ti</th>
<th>Si</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>0.011</td>
<td>54.096</td>
<td>45.890</td>
</tr>
<tr>
<td>Phase A</td>
<td>56.621</td>
<td>18.690</td>
<td>24.686</td>
</tr>
<tr>
<td>Phase B</td>
<td>35.752</td>
<td>61.217</td>
<td>3.028</td>
</tr>
</tbody>
</table>
Microprobe of CVD SiC Reaction Bonded Using PVD Ti
Conditions: 1250 °C, 31 MPa, 2 hr, vacuum, 5 °C/min

The use of a high purity, thin Ti interlayer contributed to a less complex diffusion bond.
An isothermal section of the Ti-Si-C phase diagram at 1200°C. The diffusion path is denoted by the dotted line. $\text{SiC}/\text{Ti}_3\text{SiC}_2/\text{Ti}_5\text{Si}_3/\text{two-phase Ti}_5\text{Si}_3$ plus $\text{TiC}_{1-y}/\text{Ti}_5\text{Si}_3/\text{Ti}$

Effect of Ti Foil Thickness and Processing Time on the Resulting Diffusion Bonds
- second processing matrix investigated

The following conditions were investigated:
– 10 micron foil at 2 hr
– 20 micron foil at 1, 2, and 4 hr
– 50 micron foil at 1, 2, 4, 8 and 16 hr

* all other conditions are the same as before: 1250 °C, 30 MPa applied pressure, vacuum, 2 °C /min cool down
SiC Substrates Joined with a 10 µm Ti Foil Interlayer for 2 hr

Similar results to the diffusion bond formed with a 10 micron thick PVD Ti coating
- no microcracks
SiC Substrates Joined with a 25 μm Ti Foil Interlayer

An intermediate phase is observed after 2 hr but not after 4 hr.
- no microcracks
SiC Substrates Joined with a 50 μm Ti Foil Interlayer

Over time, the formation of microcracks and an intermediate phase are observed followed by void formation and a lessening of the intermediate phase.
Close-up View of PVD Ti Coatings on SiC Discs

On Less Polished SiC

The coating is more dense on the highly polished SiC.

On Highly Polished SiC
NDE: Pulsed Thermography Overview

Light pulse uniformly heats sample surface

Thermal energy diffuses into the sample and is uniformly emitted from surface

Increase IR radiation at the surface due to the presence of a subsurface defect
Pulsed Thermography Results of Bonded SiC Derivative Images

- Presence of bonding was detected in the pulsed thermography images from front and back of the SiC disks.
- Results regarding bond quality are inconclusive due to surface conditions (i.e. reflections).

**Thermal Parameters:**
- 160x128 portion of focal plane array
- Capture rate: 409 Hz
- 223 frames (0.5 s)
NDE: Immersion Ultrasonic Testing Basics

- A high frequency ultrasonic pulse (U) enters specimen.
- Some of the signal is reflected back at the bond interface (R) while the remaining energy is transmitted (T) and eventually reflected at the back surface of the sample.
- The amount of energy reflected at the interface depends on the acoustic impedance (z) mismatch between the materials at the interface.
- Well bonded, similar materials will result in very little or no reflection at the interface while a disbond or air gap will cause more of the energy at the interface to reflect back.
Non-Destructive Evaluation (NDE) Method of Ultrasonic Immersion Shows Very Good Bonding of 1” Discs

Clay on backside of sample to verify that ultrasound reached backwall

0.65” Diameter PVD Ti Coating

0.65” Diameter PVD Ti Coating

Ti bonded region

Clay on backside of sample to verify that ultrasound reached backwall

Ti bonded region

Clay on backside of sample to verify that ultrasound reached backwall
High Strength of Bonds Greatly Exceed the Application Requirements

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 suggest 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).
Fracture Surfaces from the Pull Tests of the 1.0” Joined Discs with 0.65” Diameter Bond Region

Failure was in the SiC substrate rather than the bond. The bond was pulled out intact from the failing SiC substrate.

Failure was primarily in the SiC as failure started in one substrate crossed through the bond region and continued in the other SiC substrate.
Three Part 10 cm (4”) Diameter SiC Injector

Stacking Sequence
Top to Bottom

Top Surfaces

Small Fuel Holes

Bottom Surfaces

Large Air Holes

Fuel Swirler Detail

PVD Ti Coated

Detail
Next Slide
Detail of the Thickest Injector Substrate
(~0.635 cm thick)
Kovar Tube to SiC Brazing

B1 Copper-ABA(L) (Cu-92.75, Si-3.0, Al-2.0, Ti-2.25)

B2 MBF 20 Alloy - AWS BNi2/AMS 4777 (Cr-7, Fe-3, Si-4.5, C-0.006, B-3.2, Ni-balance)
Kovar Tube to SiC Substrate Joining with Ticusil Paste

- Sectioned perpendicular to the tube.
- Good reactivity.
- However there was difficulty filling the curved gap with paste.
Kovar Tube to SiC Substrate Joining with Ticusil Paste

- Sectioned parallel to the tube.
- Better reactivity and filling than the previous sample.
Future Studies

- Future efforts will further evaluate this bonding method to determine if it is fully capable of meeting the needs of the proposed injector application – uniform, leak-free bonds with stability and strength retention at temperatures up to 800°F.

- Develop and characterize Kovar to SiC joining.

- Apply joining techniques to component fabrication and support in-house component testing.
Summary and Conclusions

- Diffusion bonds fabricated with the alloyed Ti foil as the interlayer formed five to seven phases in the bond.
  - Microcracks due to the formation of thermally anisotropic phases.

- Diffusion bonds fabricated with the 10 micron PVD Ti coating gave better diffusion bonds than the alloyed Ti foil.
  - Fewer and preferred phases were formed which resulted in bonds without microcracks.

- Processing with thinner interlayers and/or for longer durations gave better diffusion bonds.

- NDE method of ultrasonic immersion shows good, uniform bonding.

- Strengths are well above the application requirements.
Acknowledgements

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  • Richard. E. Martin and Laura M. Cosgriff of Cleveland State University for conducting NDE