DIFFUSION BONDING OF SILICON CARBIDE FOR A MICRO-ELECTRO-MECHANICAL SYSTEMS LEAN DIRECT INJECTOR

Michael C. Halbig, Army Research Laboratory, Vehicle Technology Directorate, Cleveland, OH
Mrityunjay Singh and Tarah P. Shpargel, QSS Group, Inc., Cleveland, OH
James D. Kiser, NASA Glenn Research Center, Cleveland, OH

Robust approaches for joining silicon carbide (SiC) to silicon carbide sub-elements have been developed for a micro-electro-mechanical systems lean direct injector (MEMS LDI) application. The objective is to join SiC sub-elements to form a leak-free injector that has complex internal passages for the flow and mixing of fuel and air.

Previous bonding technology relied upon silicate glass interlayers that were not uniform or leak free. In a newly developed joining approach, titanium foils and physically vapor deposited titanium coatings were used to form diffusion bonds between SiC materials during hot pressing. Microscopy results show the formation of well adhered diffusion bonds. Initial tests show that the bond strength is much higher than required for the component system. Benefits of the joining technology are fabrication of leak free joints with high temperature and mechanical capability.

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Michael C. Halbig[1], Mrityunjay Singh[2], Tarah P. Shpargel[2],
and J. Douglas D. Kiser[3]

1 - U.S. Army Research Laboratory, Vehicle Technology Directorate, Cleveland, Ohio
2 - QSS Group, Inc., Cleveland, Ohio
3 - NASA Glenn Research Center, Cleveland, Ohio

VEHICLE TECHNOLOGY DIRECTORATE

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    - Dr. Robert Okojie of NASA GRC for providing PVD Ti Coated CVD SiC.

    - Laura Cosgriff of Cleveland State University at NASA GRC for conducting NDE.

Outline

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

2. **Previous Joining Approach** – Joining Of Silicon Carbide Ceramics With Silicate Glass Layers

3. **Current Joining Approach** – Diffusion Bonding With a Titanium Layer

    A. Titanium Foils

    B. PVD Titanium Coatings

4. **Joint and Sub-Element Tests and Demonstrations**

5. **Summary and Conclusions**

Injector Program Objective and Approach

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

Two Possible Injector Approaches

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

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

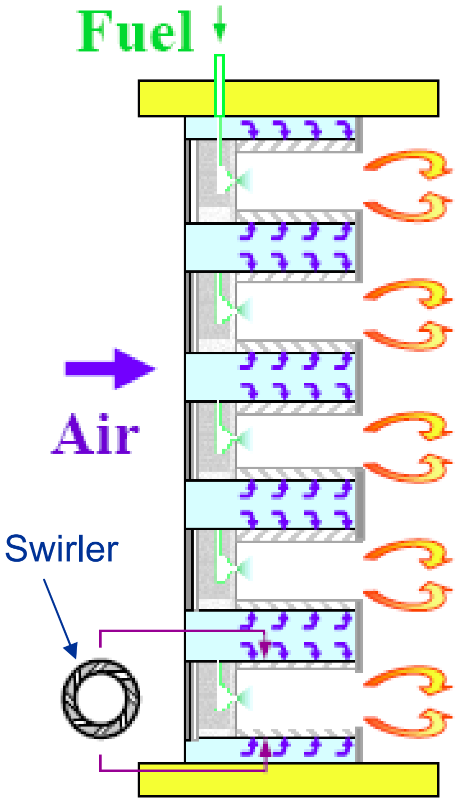
Disadvantages - Cannot be used in high pressure ratio aircraft due to auto-ignition and flashback

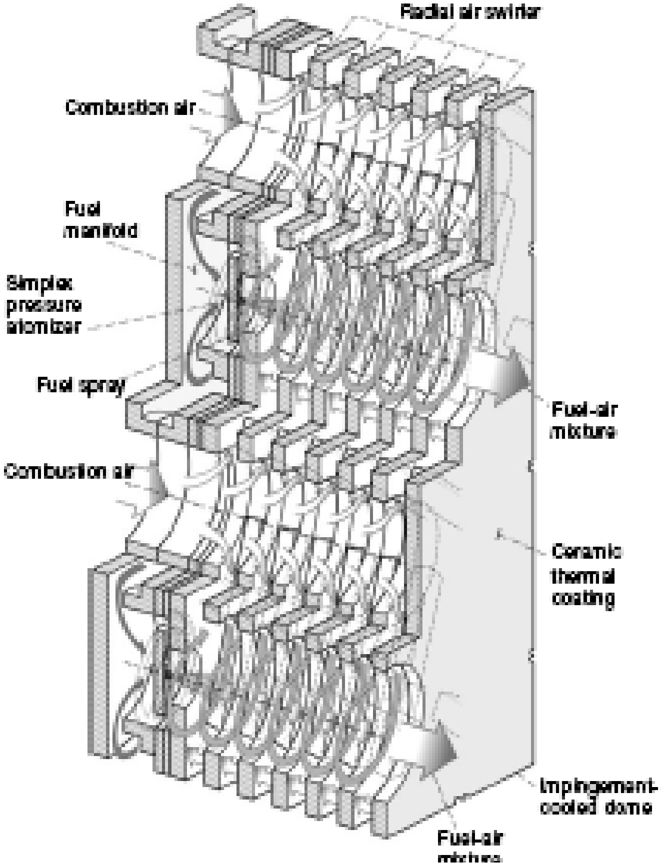
2. Lean Direct Injector (LDI)

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

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

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.

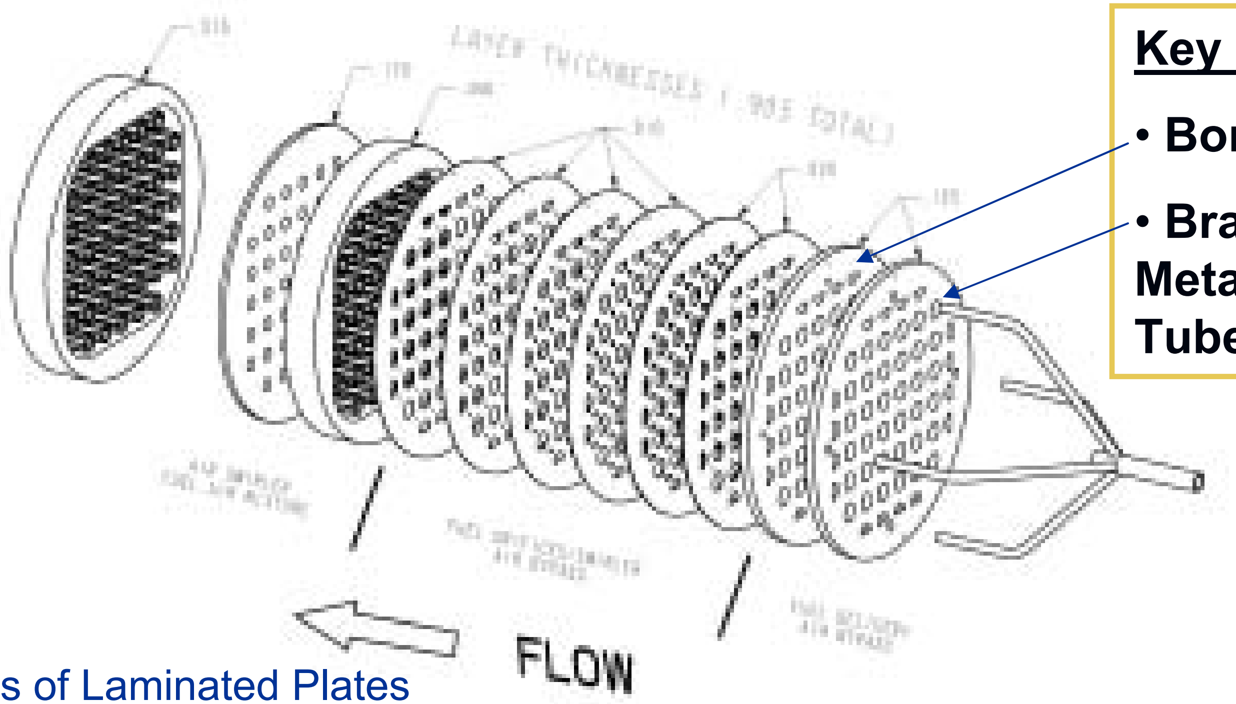
(Center) 3-inch square metal MP-LDI with 45 injectors.

(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.

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.



<u>Key Technologies:</u>

- **Bonding of SiC to SiC**
- **Brazing of SiC to Metallic (Kovar) Fuel Tubes**

<u>Benefits of Laminated Plates</u>

- 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

Previous Approach of Joining SiC With a Silicate Glass Layer

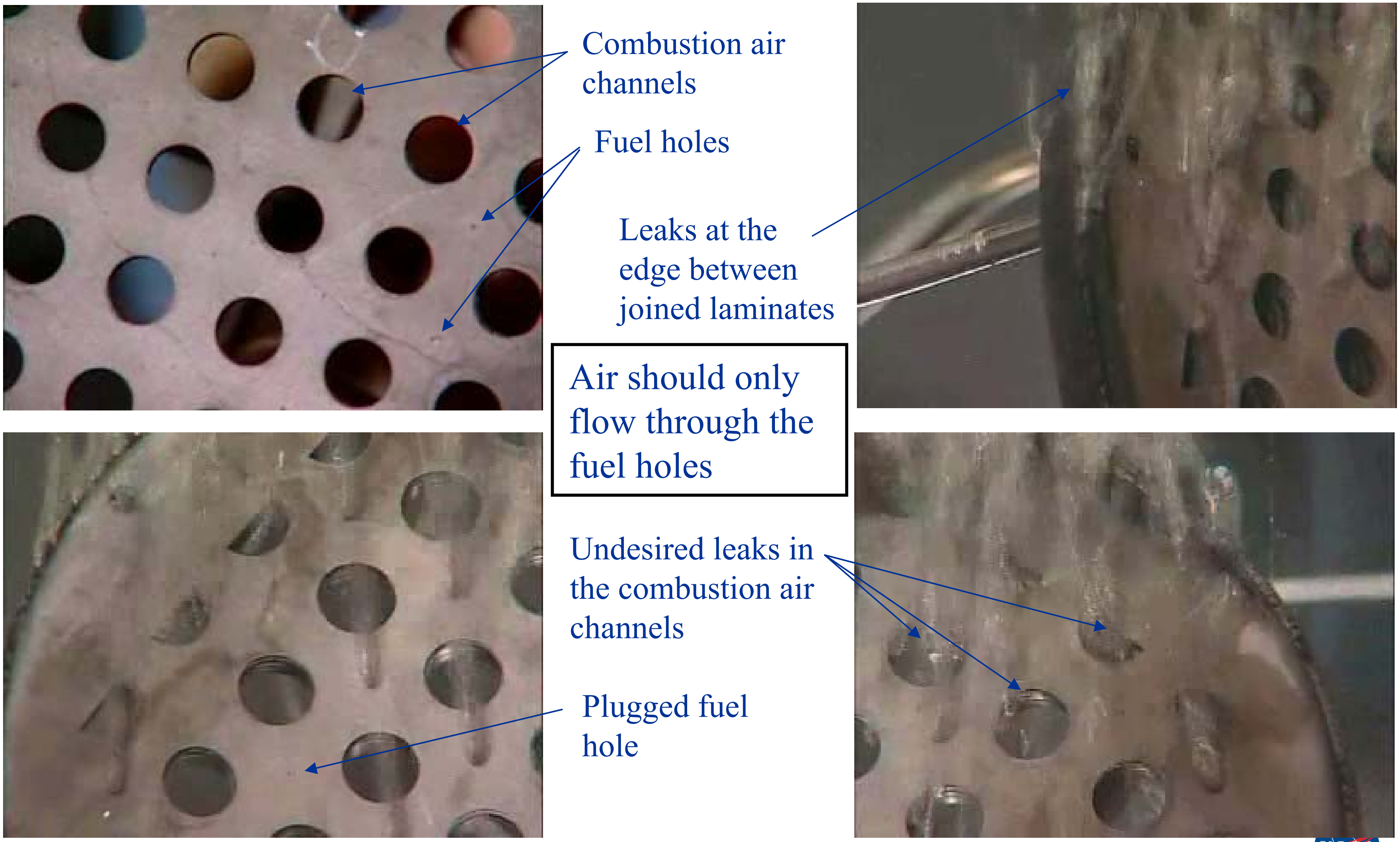
Leak Test
Movie

Movie Courtesy of
Chip Redding at
NASA GRC

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 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

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:

- Joining of relatively large geometries (i.e. 4” diameter disks)

- 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

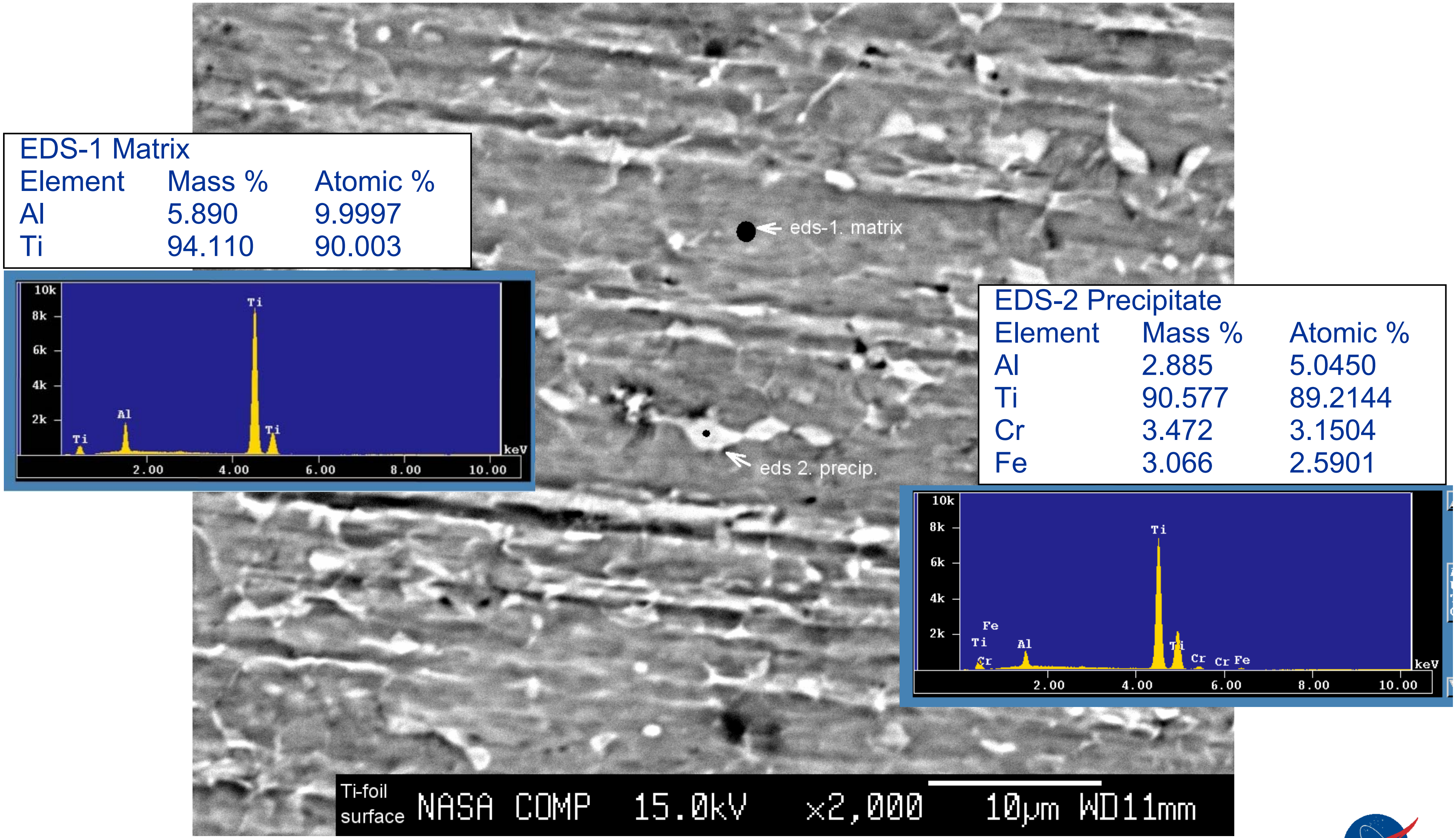
SiC and Ti Material Combinations:

1. 1.75” diameter α-SiC (CRYSTAR from Saint-Gobain) disks joined with a 1.5 mil (38 micron) foil
2. 1.75” diameter CVD SiC (TREX Enterprises) disks joined with a 1.5 mil (38 micron) 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 1.5 mil (38 micron) foil
5. 1” x 2” CVD SiC (Rohm & Hass) coupons joined with ~10 micron PVD Ti coating on both of the surfaces

Condition	**Temp. (˚C)**	**Pressure* (MPa)**	**Time (hr)**	**Atmosphere**	**Cooling Rate (˚C/min)**	**Status**
A (materials 1, 2, and 3)	1250	24, 24, 31	2	vacuum	5	Microsopy & Microprobe
B (materials 1 and 3)	1300	24, 31	2	vacuum	2	Microscopy
C (materials 1 and 3)	1250	50	2	vacuum	2	Microscopy
D (materials 1, 4 and 5)	1250	24, 31	2	vacuum	2	Microscopy

*at the minimum clamping pressure for the hot press (except for processing at 50 MPa)

Electron Micro Probe Analysis of the “Titanium” Foil



EDS-1 Matrix

Element	Mass %	Atomic %
Al	5.890	9.9997
Ti	94.110	90.003

EDS-2 Precipitate

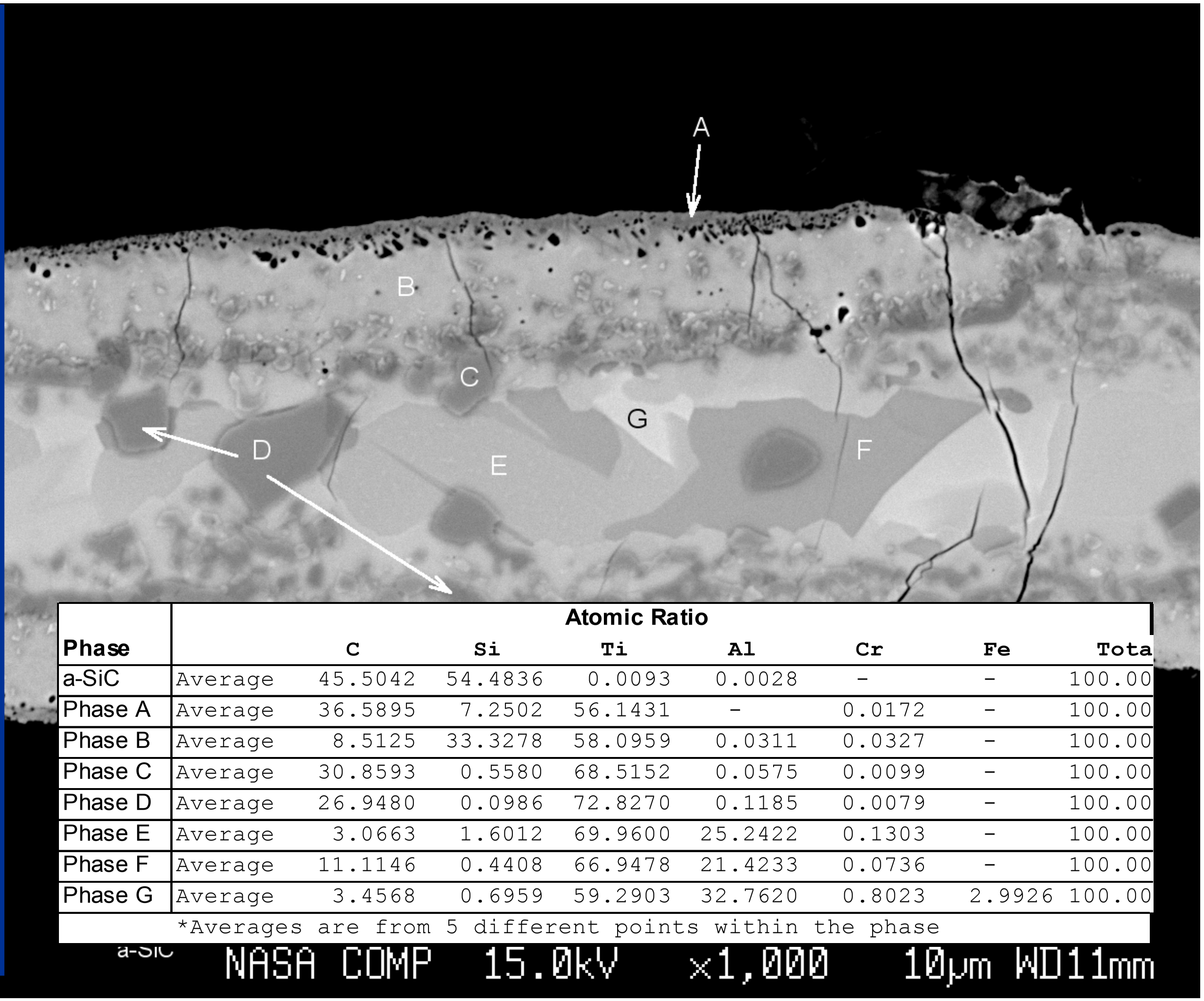
Element	Mass %	Atomic %
Al	2.885	5.0450
Ti	90.577	89.2144
Cr	3.472	3.1504
Fe	3.066	2.5901

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 the formation of two detrimental phases:

- Phase B $Ti_5Si_3C_X$ – Ti_5Si_3 if 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.



Phase		Atomic Ratio						
		C	Si	Ti	Al	Cr	Fe	Tota
a-SiC	Average	45.5042	54.4836	0.0093	0.0028	-	-	100.00
Phase A	Average	36.5895	7.2502	56.1431	-	0.0172	-	100.00
Phase B	Average	8.5125	33.3278	58.0959	0.0311	0.0327	-	100.00
Phase C	Average	30.8593	0.5580	68.5152	0.0575	0.0099	-	100.00
Phase D	Average	26.9480	0.0986	72.8270	0.1185	0.0079	-	100.00
Phase E	Average	3.0663	1.6012	69.9600	25.2422	0.1303	-	100.00
Phase F	Average	11.1146	0.4408	66.9478	21.4233	0.0736	-	100.00
Phase G	Average	3.4568	0.6959	59.2903	32.7620	0.8023	2.9926	100.00

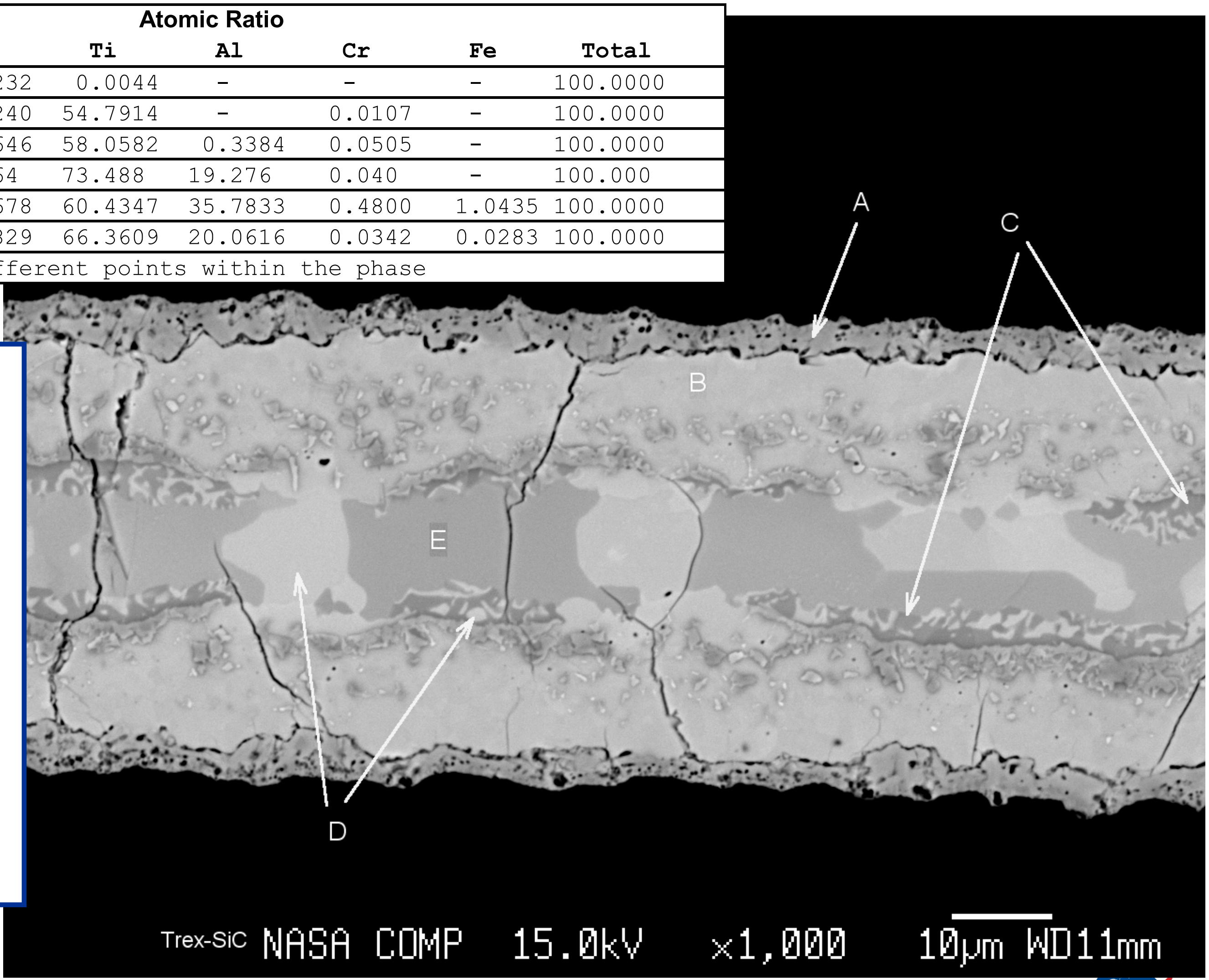
*Averages are from 5 different points within the phase

Microprobe of TREX CVD SiC Reaction Bonded Using Ti Foil
Conditions: 1250 ˚C, 24 MPa, 2 hr, vacuum, 5 ˚C/min

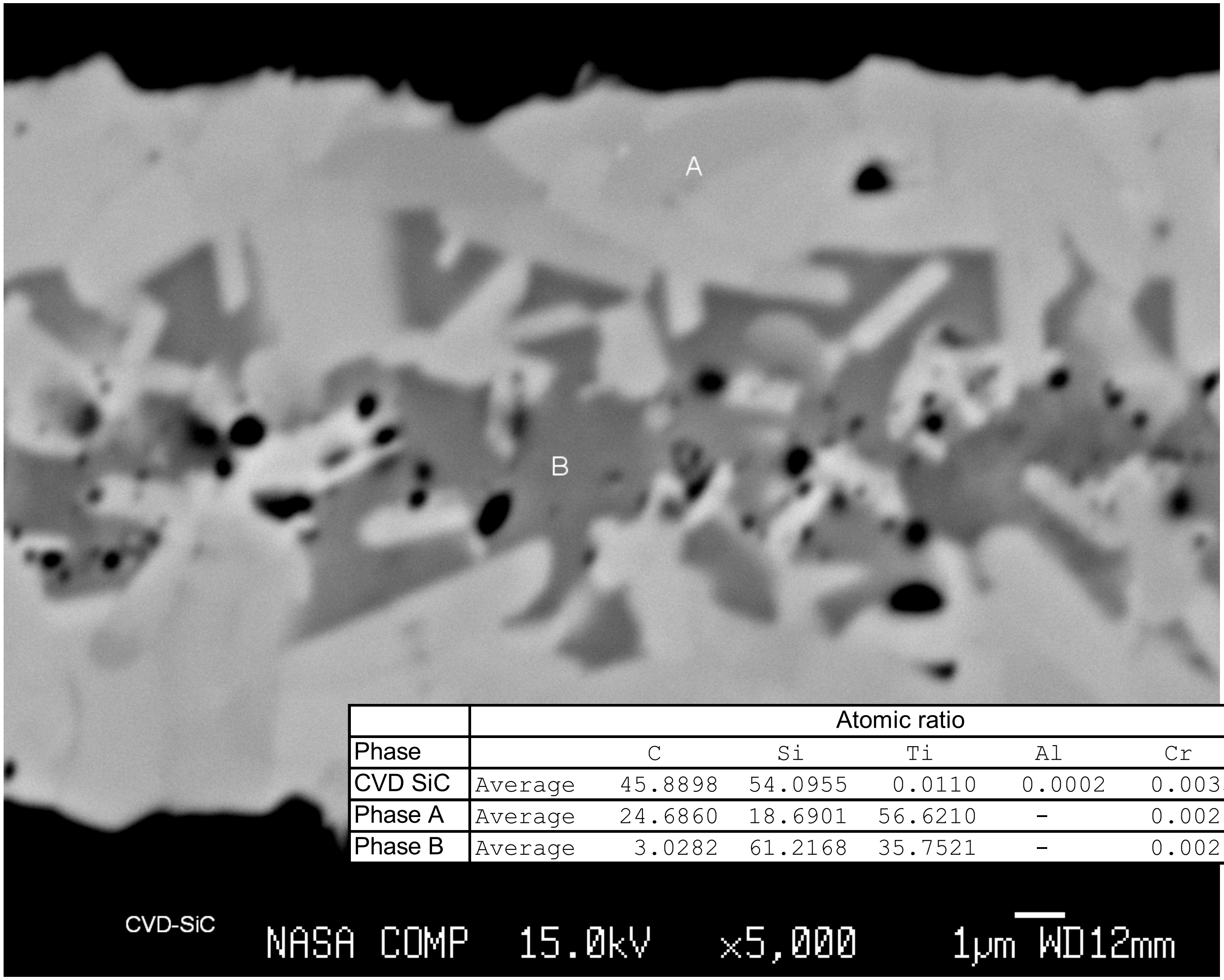
		Atomic Ratio						
Phase		**C**	**Si**	**Ti**	**Al**	**Cr**	**Fe**	**Total**
CVD SiC	Average	45.0724	54.9232	0.0044	-	-	-	100.0000
Phase A	Average	27.6739	17.5240	54.7914	-	0.0107	-	100.0000
Phase B	Average	7.3882	34.1646	58.0582	0.3384	0.0505	-	100.0000
Phase C	Average	6.432	0.764	73.488	19.276	0.040	-	100.000
Phase D	Average	1.1908	1.0678	60.4347	35.7833	0.4800	1.0435	100.0000
Phase E	Average	12.9321	0.5829	66.3609	20.0616	0.0342	0.0283	100.0000
	*Averages are from 5 different points within the phase							

The same detrimental phases of Ti_5Si_3 (B) and Ti_3Al (D) are formed which can contribute to microcracking during cool down.

Note how cracks appear to originate in Phase B or in the core, however they are absent from outer phase (Phase A)



Microprobe of CVD SiC Reaction Bonded Using PVD Ti Conditions: 1250 ˚C, 31 MPa, 2 hr, vacuum, 5 ˚C/min

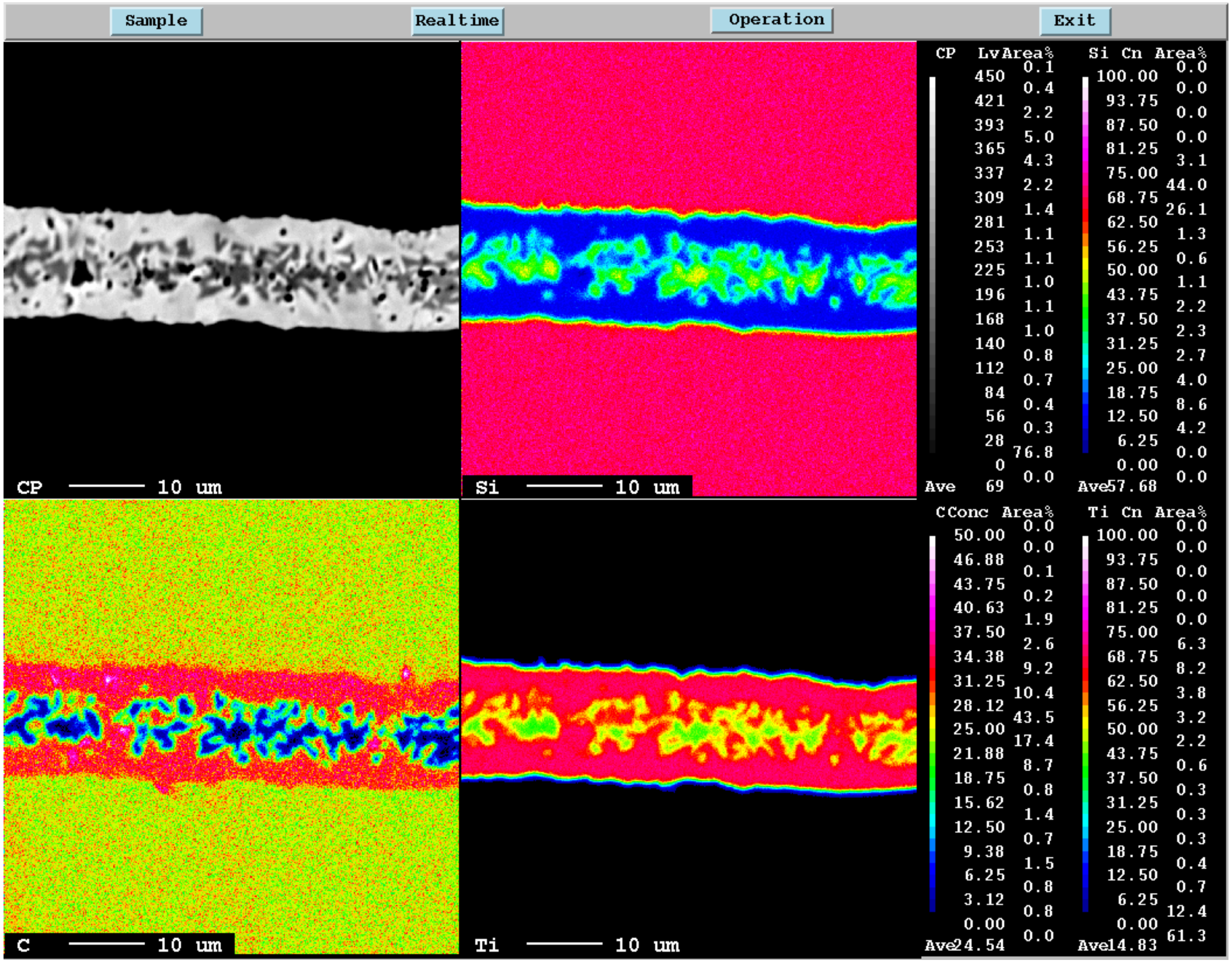


Phase	Atomic ratio						
		C	Si	Ti	Al	Cr	Total
CVD SiC	Average	45.8898	54.0955	0.0110	0.0002	0.0035	100.0000
Phase A	Average	24.6860	18.6901	56.6210	-	0.0029	100.0000
Phase B	Average	3.0282	61.2168	35.7521	-	0.0029	100.0000

The undesirable phases of Ti_5Si_3 and Ti_3Al were not formed.

Identity/source of the black phase or voids still needs to be determined.

Microprobe of CVD SiC Reaction Bonded Using PVD Ti Conditions: 1250 ˚C, 31 MPa, 2 hr, vacuum, 5 ˚C/min

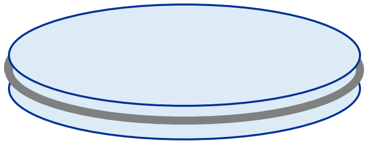


```
CP   LvArea%      Si  Cn  Area%
         0.1                  0.0
     450         100.00
         0.4                  0.0
     421          93.75
         2.2                  0.0
     393          87.50
         5.0                  0.0
     365          81.25
         4.3                  3.1
     337          75.00
         2.2                 44.0
     309          68.75
         1.4                 26.1
     281          62.50
         1.1                  1.3
     253          56.25
         1.1                  0.6
     225          50.00
         1.0                  1.1
     196          43.75
         1.1                  2.2
     168          37.50
         1.0                  2.3
     140          31.25
         0.8                  2.7
     112          25.00
         0.7                  4.0
      84          18.75
         0.4                  8.6
      56          12.50
         0.3                  4.2
      28           6.25
        76.8                  0.0
       0           0.00
         0.0                  0.0
Ave   69      Ave57.68
```

```
CConc  Area%      Ti  Cn  Area%
         0.0                  0.0
   50.00         100.00
         0.0                  0.0
   46.88          93.75
         0.1                  0.0
   43.75          87.50
         0.2                  0.0
   40.63          81.25
         1.9                  0.0
   37.50          75.00
         2.6                  6.3
   34.38          68.75
         9.2                  8.2
   31.25          62.50
        10.4                  3.8
   28.12          56.25
        43.5                  3.2
   25.00          50.00
        17.4                  2.2
   21.88          43.75
         8.7                  0.6
   18.75          37.50
         0.8                  0.3
   15.62          31.25
         1.4                  0.3
   12.50          25.00
         0.7                  0.3
    9.38          18.75
         1.5                  0.4
    6.25          12.50
         0.8                  0.7
    3.12           6.25
         0.8                 12.4
    0.00           0.00
         0.0                 61.3
Ave24.54      Ave14.83
```

The use of a high purity Ti interlayer contributed to a less complex diffusion bond.

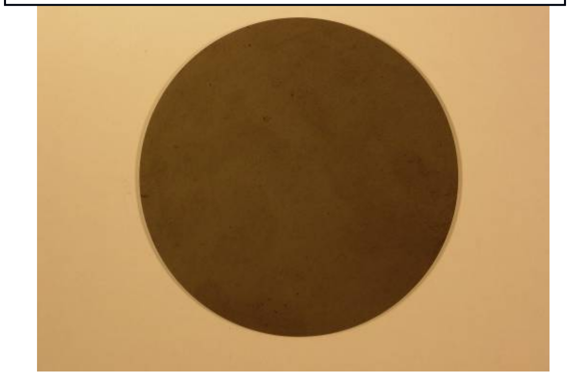
Sub-Element Diffusion Bonding/Demonstrations: Joining 4” Disks, 200 psi (1.38 MPa), and 800˚F (427˚C)



Demonstrate the Joining of 4” diameter disks

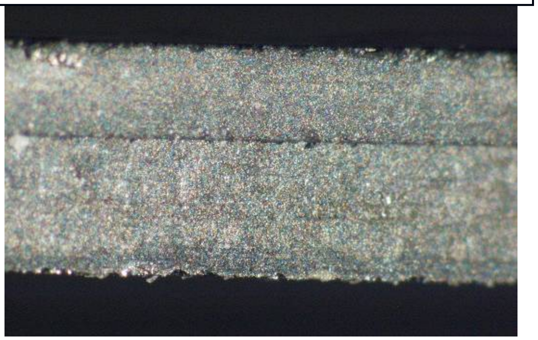
Both substrates before joining.

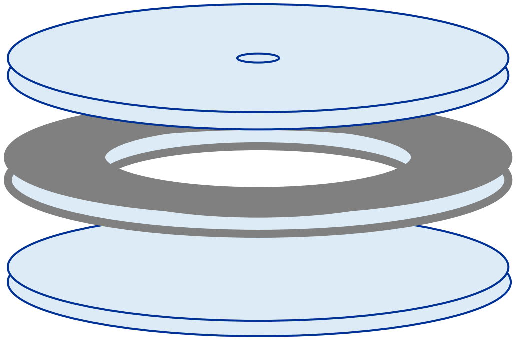
Surface after joining.



As-Fabricated edge of joined disks.

Overall thickness is 0.049”

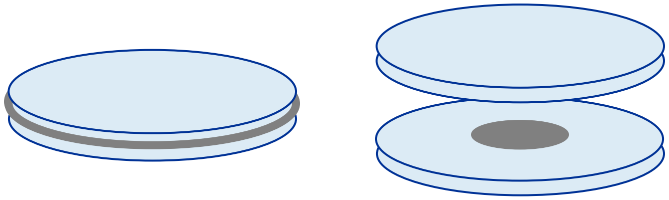




Leak/Pressure tests of joined disks (1.75” OD and 1.25” ID).

Demonstrate pressure of 200 psi.

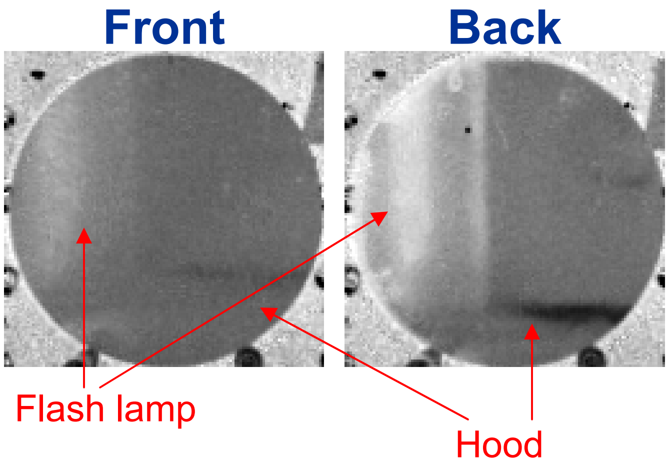
Full Surface Coating or Partial



Demonstrate the strength of joined 1” diameter disks at R.T. and 800˚F.

NDE of 4” Bonded CVD SiC-Titanium Disk Using Flash Thermography

Thermal Derivative Images



Lens to sample distance: ~30”

- Both sides of disk were reflective, making interrogation difficult
- Reflections of system parts are shown in the thermal images (left)
- Results are inconclusive

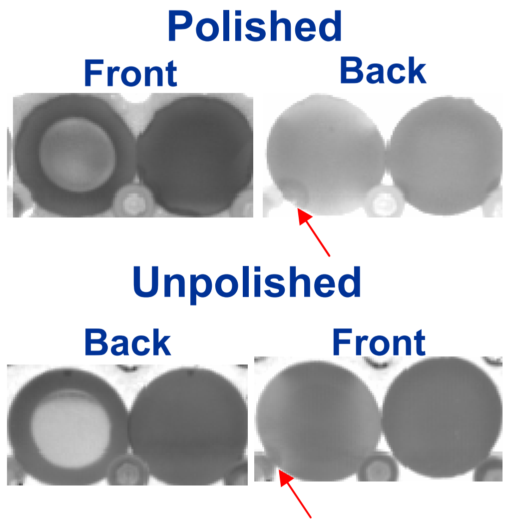
NDE of 1” Diameter Polished and Unpolished Disks

Coated center
3/4” diameter

The front and back of 4 disks were evaluated using thermography

2 polished: 1 with, 1 without coating

2 unpolished: 1 with, 1 without coating

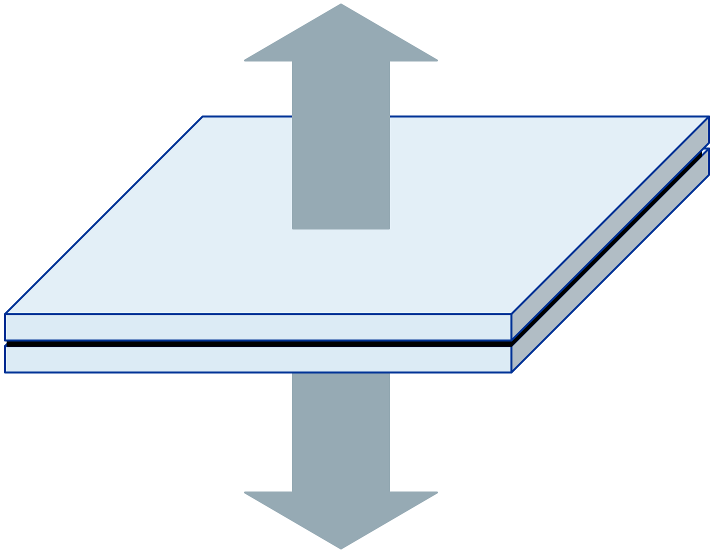


Lens to sample distance: ~17.5”

- Both sides of disks were reflective, making interrogation difficult
- Reflections of system parts (pins) are shown in the thermal images (left)
- Results are inconclusive

Once the disks are bonded, NDE may more clearly show distinct regions that are bonded and not bonded (i.e. central area with the coating and the outer ring without the coating)

Initial Strength Tests on Diffusion Bonded CVD SiC with a PVD Ti Interlayer



Initial pull test tensile strengths:

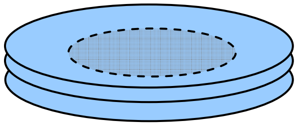
> 23.62 MPa (3.43 ksi)*

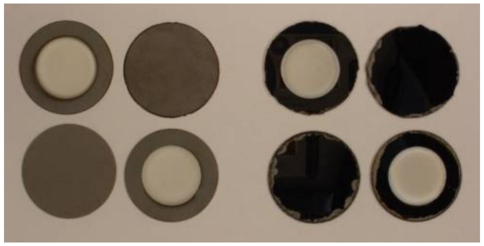
> 28.38 MPa (4.12 ksi)*

* 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).





Summary and Conclusions

- A robust method of bonding SiC to SiC has been developed and optimized.
- Diffusion bonds fabricated with the alloyed Ti foil as the interlayer formed microcracks due to the formation of thermally anisotropic and low ductility phases.
- Diffusion bonds fabricated with the PVD Ti coating gave better diffusion bonds than the alloyed Ti foils
  - Bonds were uniform with no delaminations.
  - Preferred phases were formed which resulted in bonds without microcracks.
- The currently planned sub-element tests 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.