Diffusion Bonding of Silicon Carbide for MEMS-LDI Applications

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

A robust joining approach is critically needed for a Micro-Electro-Mechanical Systems-Lean Direct Injector (MEMS-LDI) application which requires leak free joints with high temperature mechanical capability. Diffusion bonding is well suited for the MEMS-LDI application. Diffusion bonds were fabricated using titanium interlayers between silicon carbide substrates during hot pressing. The interlayers consisted of either alloyed titanium foil or physically vapor deposited (PVD) titanium coatings. Microscopy shows that well adhered, crack free diffusion bonds are formed under optimal conditions. Under less than optimal conditions, microcracks are present in the bond layer due to the formation of intermetallic phases. Electron microprobe analysis was used to identify the reaction formed phases in the diffusion bond. Various compatibility issues among the phases in the interlayer and substrate are discussed. Also, the effects of temperature, pressure, time, silicon carbide substrate type, and type of titanium interlayer and thickness on the microstructure and composition of joints are discussed.

DIFFUSION BONDING OF SILICON CARBIDE FOR MEMS-LDI APPLICATIONS

Michael C. Halbig[1], Mrityunjay Singh[2], Tarah P. Shpargel[3], and J. Douglas Kiser[4]

1 - U.S. Army Research Laboratory, Vehicle Technology Directorate, Cleveland, Ohio
2 - Ohio Aerospace Institute, Cleveland, OH
3 –ASRC Aerospace Corporation, Cleveland, Ohio
4 - NASA Glenn Research Center, Cleveland, Ohio

VEHICLE TECHNOLOGY DIRECTORATE

Ohio Aerospace Institute

ASRC AEROSPACE CORPORATION

31st International Conference & Exposition on Advanced Ceramics and Composites,
Daytona Beach, Florida, January 21-26, 2007.

Acknowledgements

- This effort was supported by the NASA Glenn Research Center under the Subsonics and Supersonics Foundation Research Programs.
- The authors would like to thank the following:
  - Dr. Dan L. Bulzan and Robert R. Tacina at NASA GRC for their support and for providing the injector design and requirements.
  - James Smith of QSS Group, Inc. at NASA GRC for conducting electron microprobe work.
  - Dr. Robert Okojie of NASA GRC for providing PVD Ti Coated CVD SiC.

Outline

- **Application** – Micro-Electro-Mechanical Systems Lean Direct Injector (MEMS LDI) for Advanced Aircraft Gas Turbines
- **Previous Joining Approach** – Joining Of Silicon Carbide Ceramics With Silicate Glass Layers
- **Current Joining Approach** – Diffusion Bonding With a Titanium Layer
- **Near Term Plans** – Subcomponent Fabrication
- **Summary and Conclusions**

Potential Applications for SiC Bonding

SiC Ceramic Matrix Composite (CMC) to SiC CMC

- Aerospace and Ground Based Engine Applications
- Hot Structure Fusion Reactor Applications

Monolithic SiC to Monolithic SiC

- Heating Elements
- Diffusion Furniture in Microelectronic Industry
- Optical Components in Space Applications
- Ceramic Armor
- **Smart Integrated Multi-Point Lean Direct Injector**

Injector Program Objective and Approach

Objective

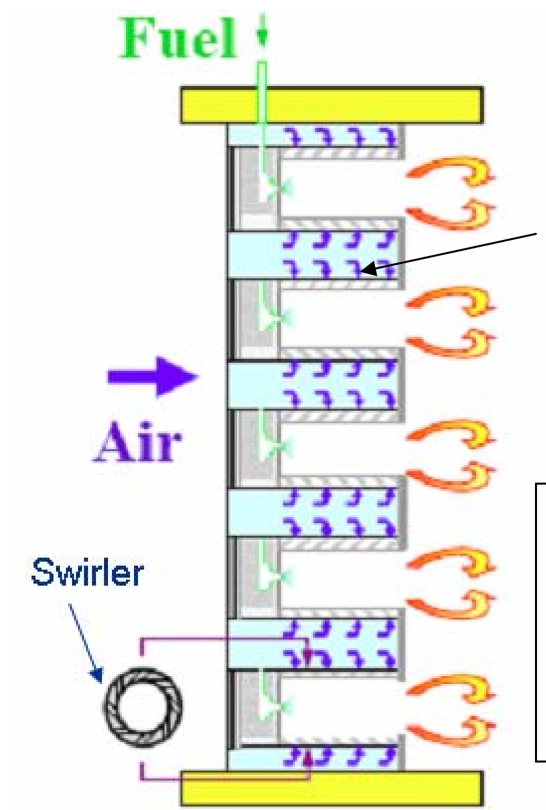
Develop technology for a SiC Smart Integrated Multi-Point Lean Direct Injector (SiC SIMPL-DI) that is operable at all engine operating conditions and has reduced NOx emissions.

Advantages of a Lean Direct Injector (LDI) Approach

- Does not have the problems of auto-ignition and flashback that other approaches may have.

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

Multi-Point Lean Direct Injector



Air channels and the swirler exits on the side are built up from laminates.

The complex channels can only be fabricated by stacking and joining laminates with different hole patterns.

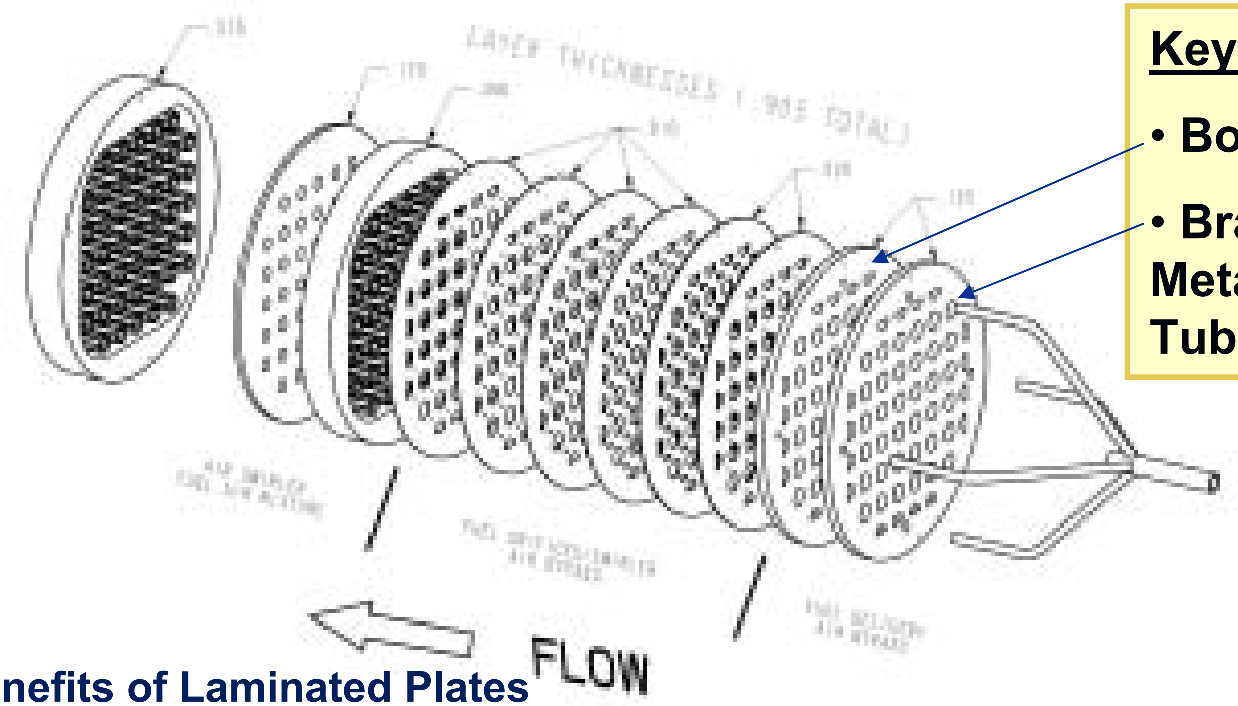
(Left) 3-inch square metal MP-LDI with 45 injectors.

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

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



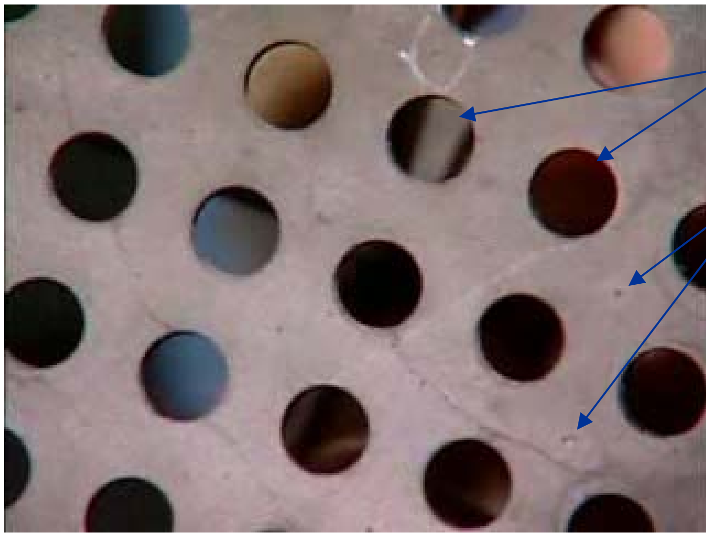
Key Technologies:

- **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

Leak Test of SiC Laminates Joined with Silicate Glass



Combustion air channels

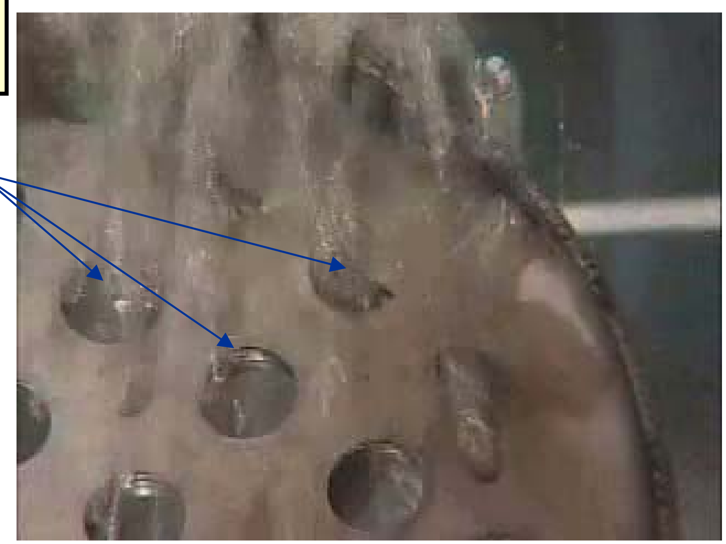
Fuel holes

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

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

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

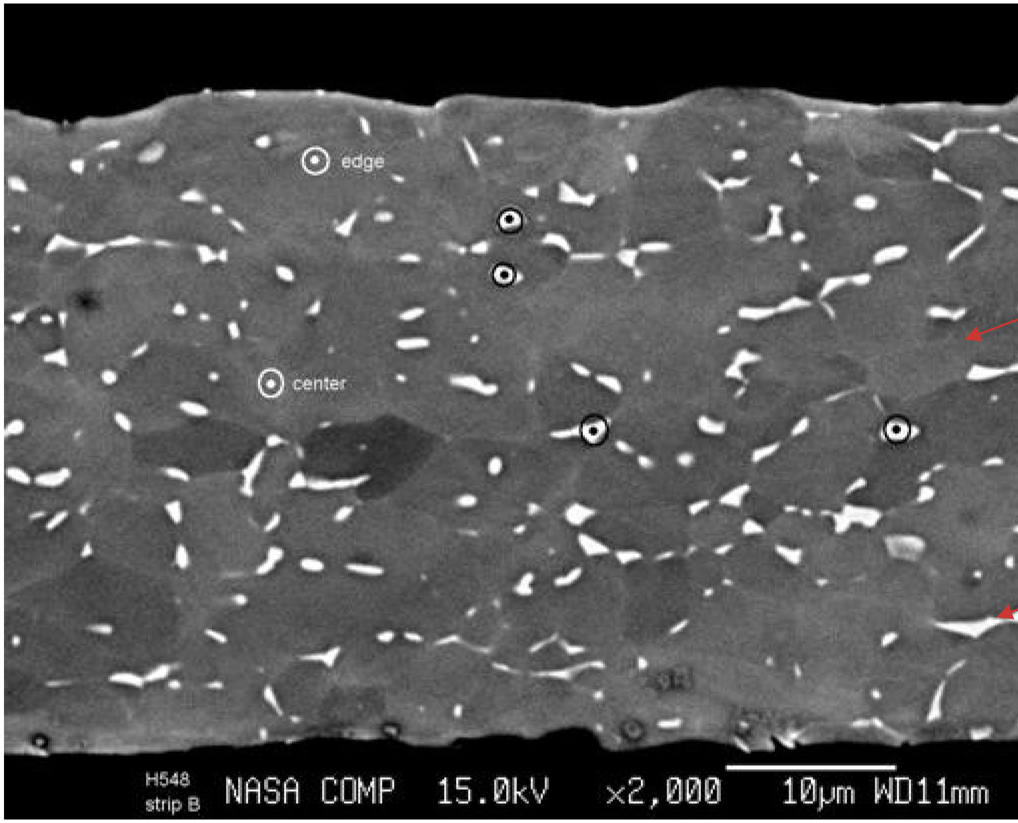
SiC and Ti Material Combinations:

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)

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

*at minimun clamping pressure for the hot press

Alloyed Ti Foil



Ti-6Al-4V (weight %)

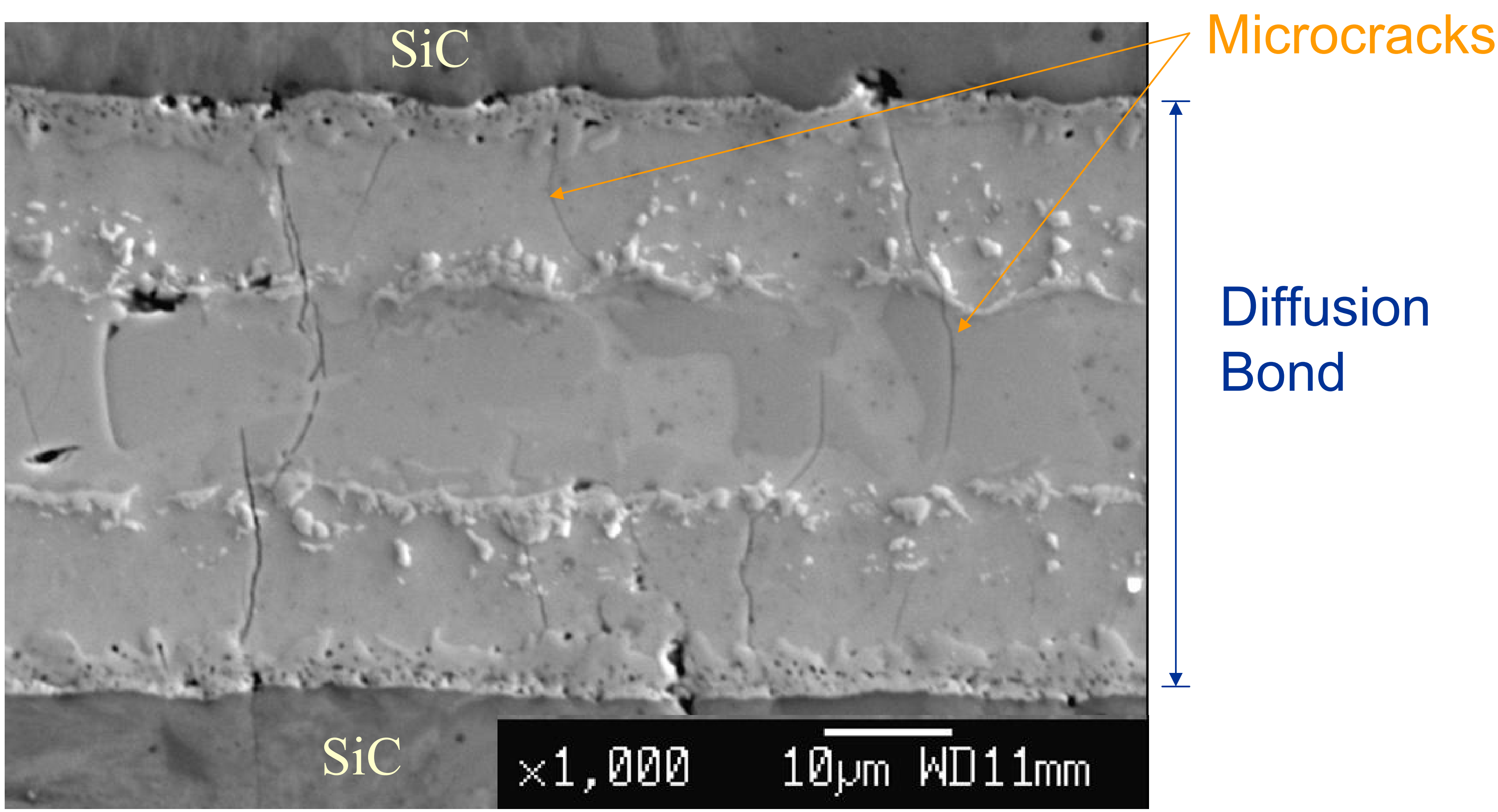
Grey phase – Alpha alloy

White phase – Beta alloy

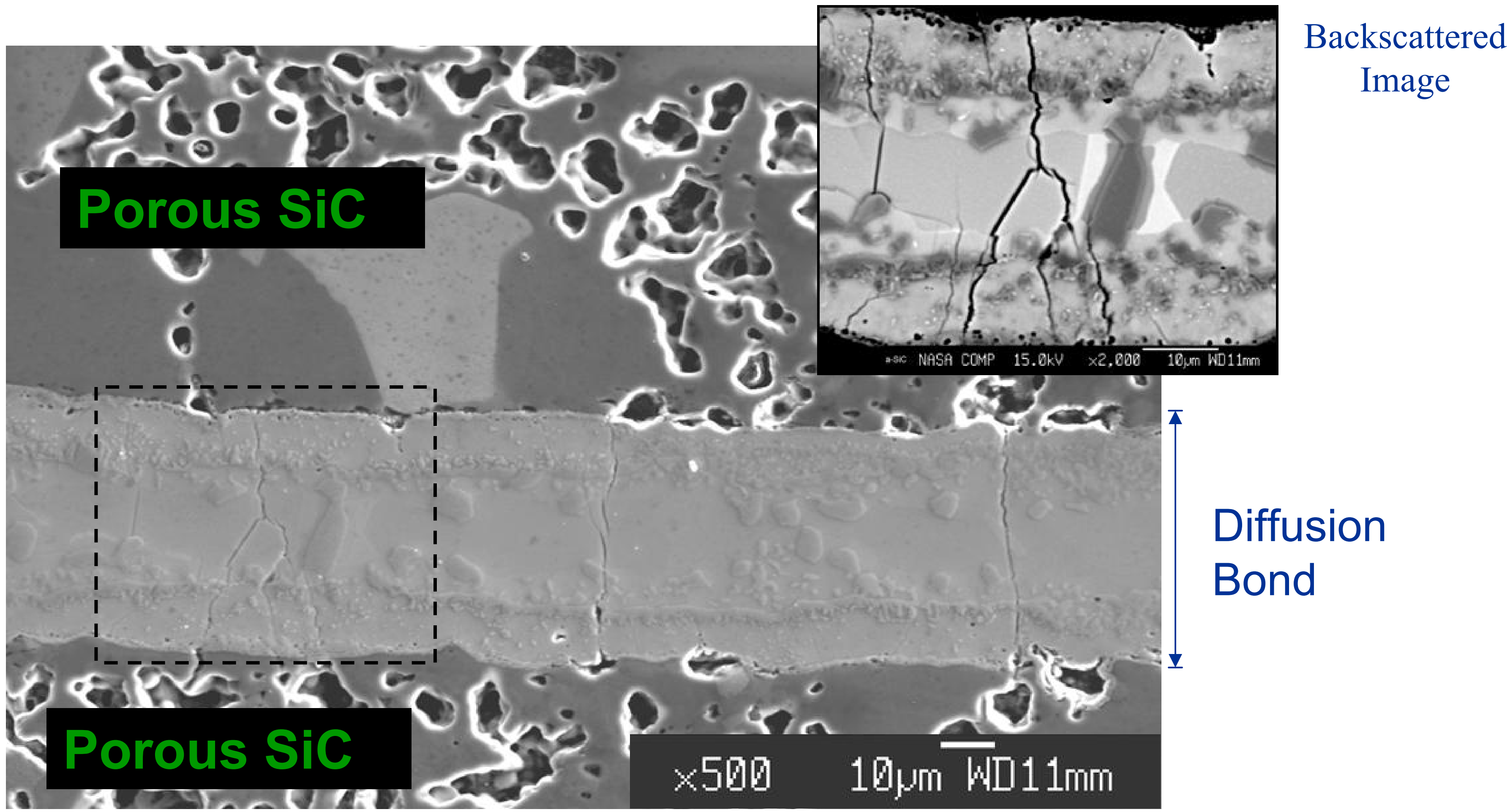
Microprobe from the cross-section of alloyed Ti foil (averages taken from several points near the edge and at the center of the foil)

	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

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

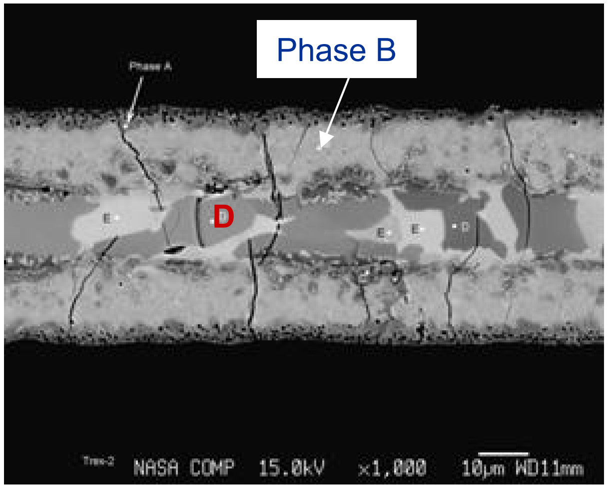


Secondary Electron Image of the Diffusion Bond - Alloyed Ti Foil and Alpha-SiC

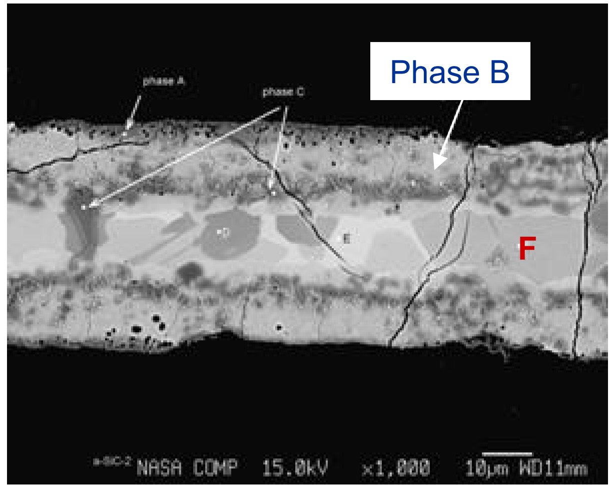


Bonding with the Alloyed Ti Foil Between Different SiC Substrates

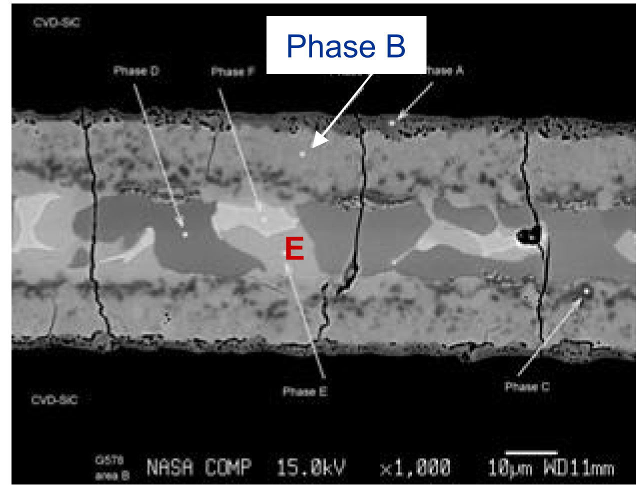
TREX CVD SiC



CRYSTAR α-SiC



Rohm & Haas CVD SiC

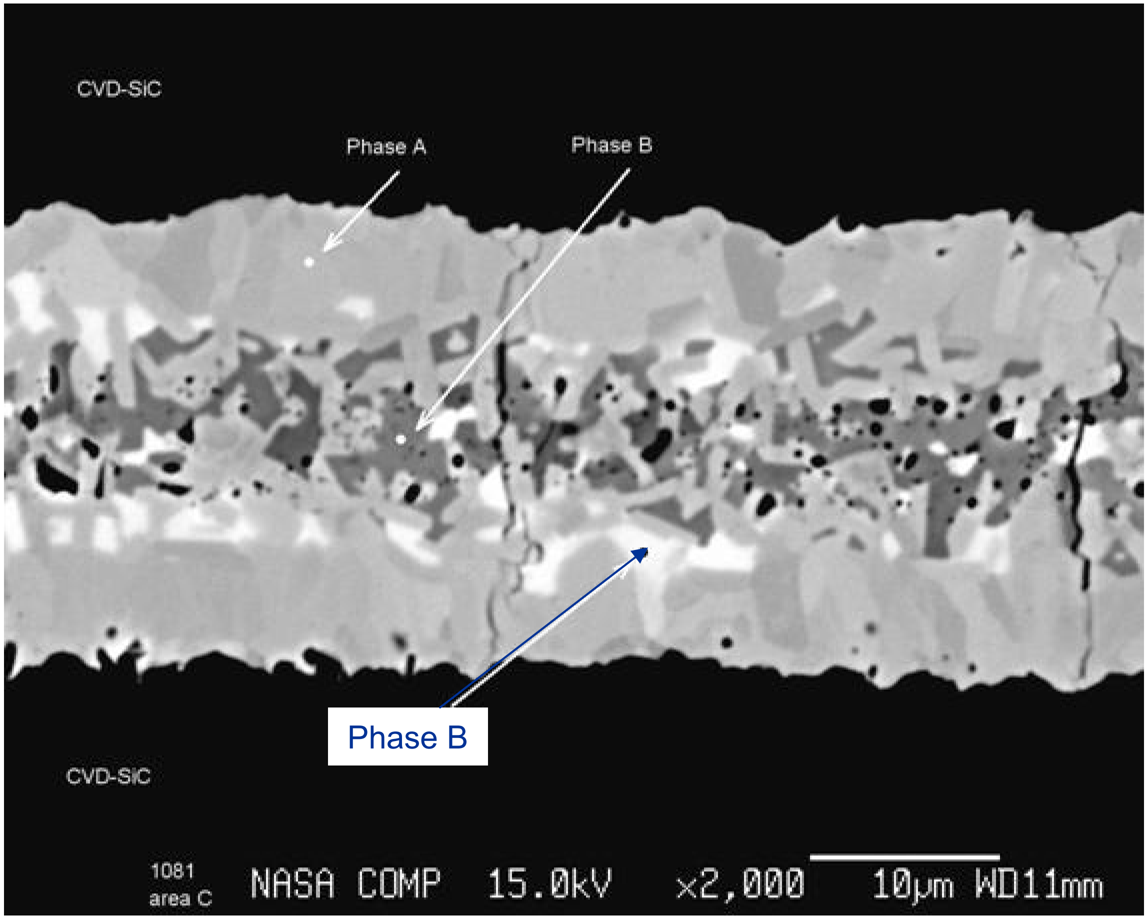


Microcracks formed regardless of the substrate and variations in the processing: higher temperatures, higher pressures, slower cooling rate.

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

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

Microprobe of CVD SiC Reaction Bonded Using 20 Micron PVD Ti
Conditions: 1250 ˚C, 31 MPa, 2 hr, vacuum, 2 ˚C/min



Simpler and fewer reaction formed phases present, 3 phases versus 5-6 with the alloyed Ti foil.

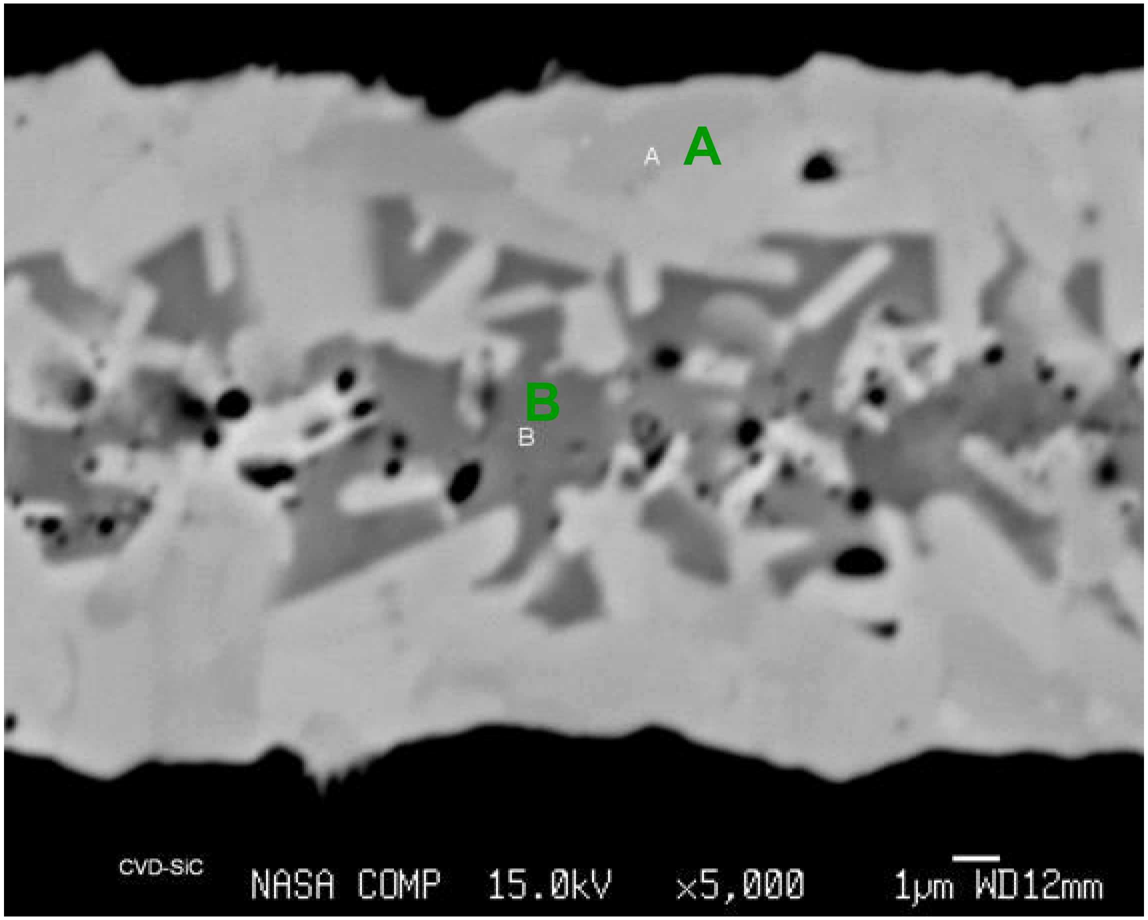
However, there is still microcracking due to the presence of $Ti_5Si_3C_X$ (Ti_5Si_3).

Naka et al suggest that this is an intermediate phase that will not be present when the full diffusion/reaction process is completed.

Rohm & Haas CVD SiC and 20 micron PVD Ti Interlayer – Atomic Ratios

Phase	Al	Fe	Ti	Si	C	Cr	Total
Phase A	0.011	0.001	56.426	17.792	25.757	0.014	100.000
Phase B	0.007	0.005	35.794	62.621	1.570	0.003	100.000
Phase C	0.027	0.153	58.767	33.891	7.140	0.023	100.000

Microprobe of CVD SiC Reaction Bonded Using 10 Micron PVD Ti
Conditions: 1250 ˚C, 31 MPa, 2 hr, vacuum, 5 ˚C/min



Thinner PVD Ti interlayer (10 micron)

- no microcracking

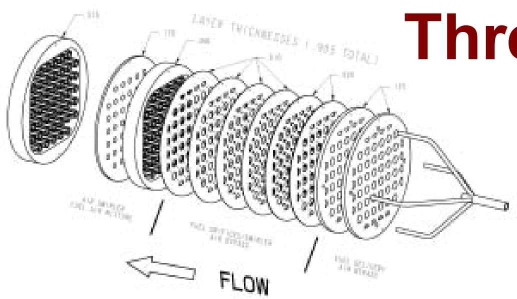
- no phase of $Ti_5Si_3C_X$ (Ti_5Si_3).

Thin interlayers of pure Ti down-selected as the preferred interlayer (i.e. 10 micron PVD Ti coatings and Ti foil)

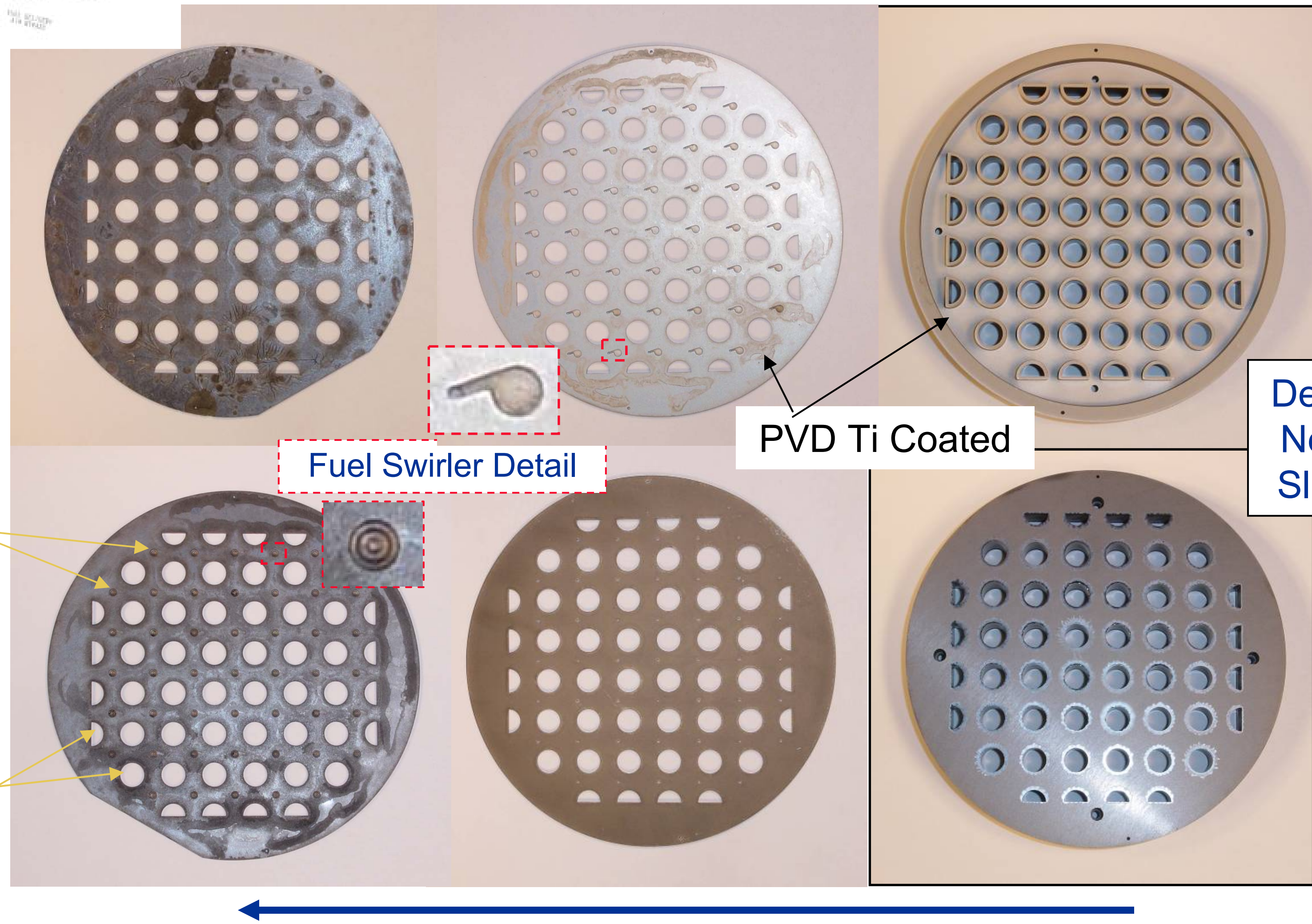
Rohm & Haas CVD-SiC and 10 micron PVD Ti Interlayer - Atomic Ratios

Phase	C	Si	Ti	Al	Cr	Total
SiC	45.890	54.096	0.011	0.000	0.004	100.000
Phase A	24.686	18.690	56.621	-	0.003	100.000
Phase-B	3.028	61.217	35.752	-	0.003	100.000

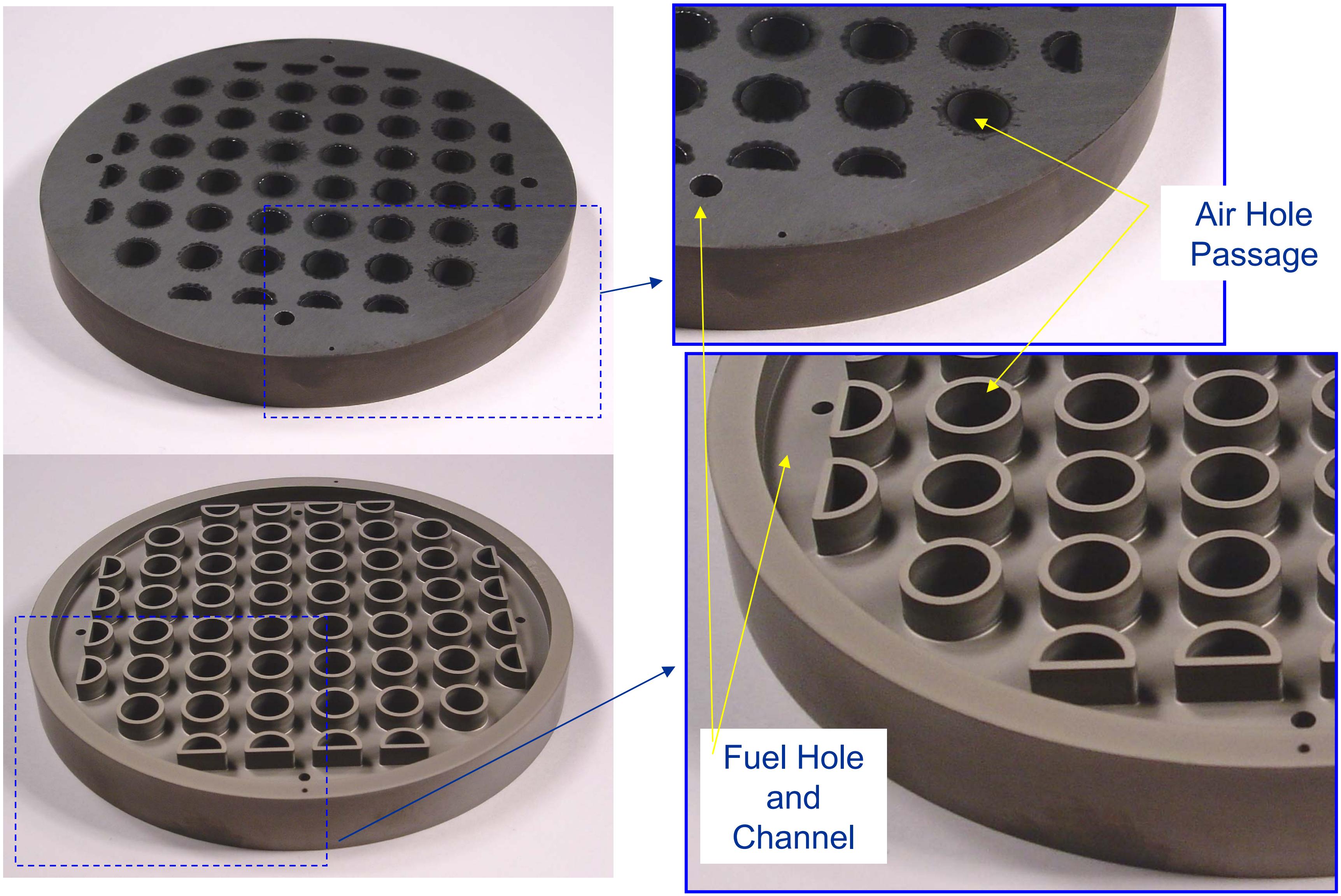
Three Part 10 cm (4”) Diameter SiC Injector



Stacking Sequence
Top to Bottom



Detail of the Thickest Injector Substrate (~0.635 cm thick)



Summary and Conclusions

- A robust method of bonding SiC to SiC has been developed and optimized that is applicable to the LDI injector application as well as a variety of other applications.
- Diffusion bonds fabricated with the alloyed Ti foil as the interlayer formed five to seven reaction formed phases in the bond.
    - Microcracks due to the formation of thermally anisotropic and low ductility 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.
- 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.