Joining of Silicon Carbide-Based Ceramics for MEMS-LDI Fuel Injector Applications

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Outline

- Background - Objectives, Challenges, and Applications
- Processing, Characterization, and Results
  - Ceramic to Ceramic Joining: Diffusion Bonding of SiC to SiC
  - SEM and TEM
  - Strength tests and NDE
  - Large geometry processing (injector component size)
- Conclusions

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Research Scope

Overall Objective: Deliver the benefits of ceramics in turbine engine applications- increased efficiency, performance, horsepower, range, operating temperature, and payload and reduced cooling and operation and support costs for future engines.

Targeted Components: Aeronautic and ground based engine applications: injectors, ceramic turbine vanes, blades, rotors, combustor liners, valves, and heat shields

Approach:

•Develop **ceramic to ceramic** joining technologies that enable the fabrication of complex shaped ceramic components.

- Fabricated ceramic shapes are currently limited to relatively small, flat, and circular shapes due to limitations in ceramic processing methods (i.e. chemical vapor deposition and hot pressing).

• Develop **ceramic to metal** joining technologies that enable ceramic components to be integrated into metallic based engine systems.

- Barriers to ceramic utilization are due to residual stresses and chemical and thermal incompatibility between ceramics and metals.




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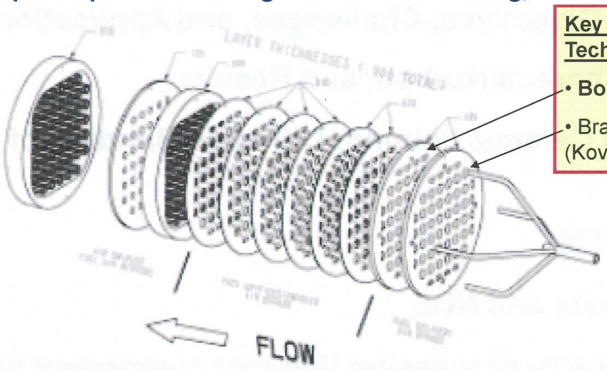
Fabrication of Lean Direct Injector Components by Diffusion Bonding of SiC Laminates

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

Goals and Advantages of Lean Direct Injector (LDI) Design

- Operability at all engine operating conditions
- Reduce NOx emissions by 90% over 1996 ICAO standard
- Does not have the problems of Lean Pre-Mixed Pre-Evaporated Injector such as auto-ignition and flashback)
- Provides extremely rapid mixing of the fuel and air before combustion occurs

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Integration Technologies for MEMS-LDI Fuel Injector

Objective:
Develop joining and integration technologies for a SiC Smart Integrated Multi-Point Lean Direct Injector (SiC SIMP-LDI)

Required capabilities of the joining technology :
- 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)

Why Silicon Carbide for a Low Temperature Application (~400ºC)
- SiC has high strength, creep resistance, and thermomechanical properties
- Allows for integration of silicon carbide based high frequency fuel actuators and sensors for monitoring and optimizing fuel flow and combustion
- Passages of any shape can be created to allow for multiple fuel circuits
- Provides thermal protection of the fuel to prevent coking
- Low cost fabrication of modules with complex internal geometries through chemical etching

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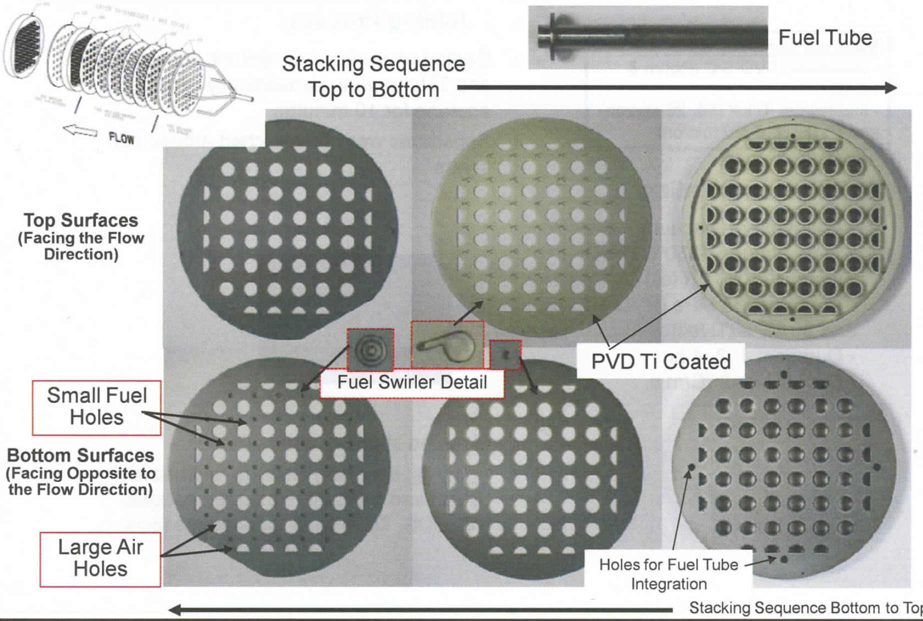
Detail of the Three Part 10 cm (4") Diameter SiC Injector



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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
- Non-flowing reaction bonded joining approach won't clog the fuel and swirler holes

Joining Challenges

- Thermal, chemical, and mechanical incompatibilities between the different materials
- Obtaining bonds that are high strength, leak free, and stable
- Excessive flow of the interlayer or reaction formed phases during processing
- Cracking in the bond layers and/or substrates
- Lack of ASTM standards for mechanical tests

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Experimental Procedure - Ceramic to Ceramic Joining

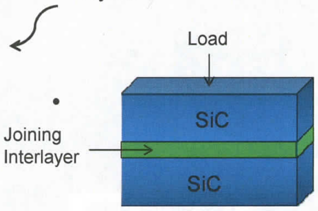
<u>Materials (dimensions 0.5" x 1")</u>
- Substrate: CVD SiC (Rohm & Haas)
- Interlayers: Ti foil (10, 20 micron) and PVD Ti (10, 20 micron)

Diffusion Bonding

- Atmosphere: Vacuum
- Temperature: 1250°C (PVD Ti) and 1200°C (Ti foil)
- Pressure: 24 MPa (PVD Ti) and 30 MPa (Ti foil)
- Duration: 1, 2, 4 hr
- Cool down: 2 °C/min

Joining Process

- Coated and uncoated ceramic substrates and Ti foils were ultrasonically cleaned in acetone for 10 minutes
- Substrates were sandwiched around foil layers



- Mounted in epoxy and polished,
- <u>Analysis:</u> scanning electron microscopy (SEM) and electron micropobe coupled with EDS, TEM, NDE, and tensile tests

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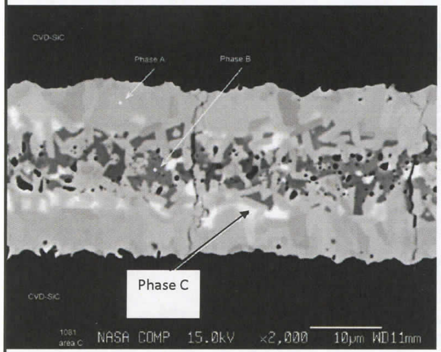
Diffusion Bonds from PVD Ti at 1250°C, 2 hr hold

20 Micron Ti Interlayer, 1250°C, 2 Hr

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

Naka et al suggest that this is an intermediate phase.

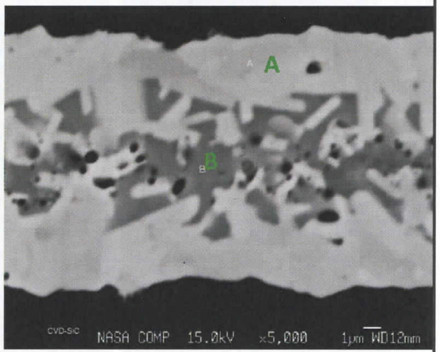


Phases in bond with the 20 μ Ti Interlayer – Atomic Percent

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 , 1250°C, 2 HR

No microcracking or phase of $Ti_5Si_3C_X$ is present.



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

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

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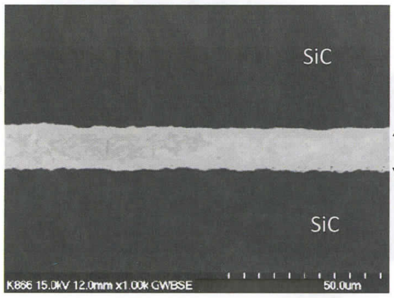
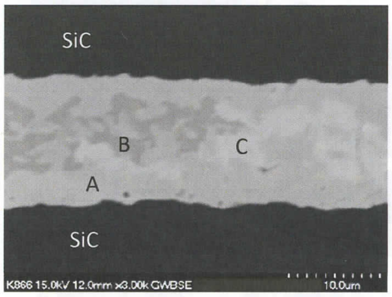
Diffusion Bonds from 10 μm Ti Foil at 1200°C, 2 hr hold



Diffusion Bond

Very minimal microcracking cracking does not traverse through the thickness of the bond. Three reaction formed phases are observed in the diffusion bond.



	C	Si	Ti
Phase A	51	14	35
Phase B	37	43	20
Phase C	38	27	35

In atomic percent

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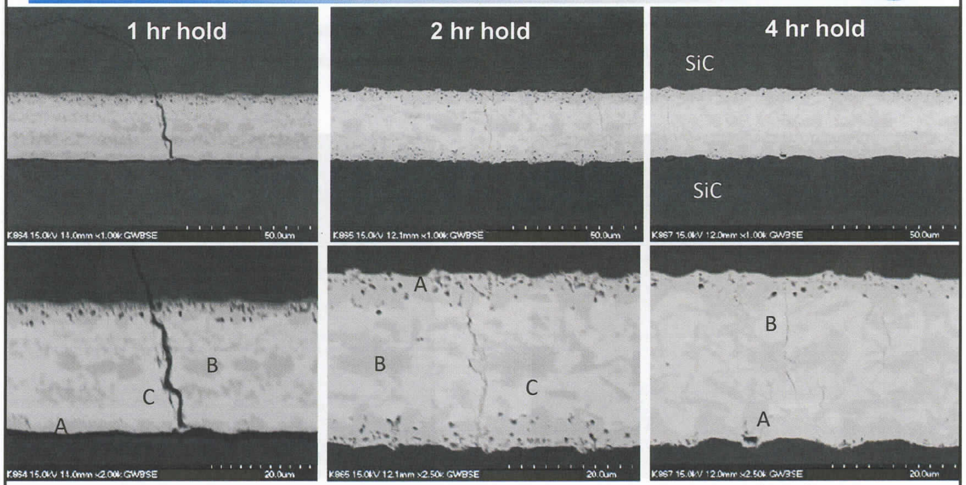
Diffusion Bonds from 20 µm Ti Foil at 1200°C



1 hr hold	C	Si	Ti
Phase A	54	13	33
Phase B	57	1	42
Phase C	44	23	33

In atomic percent

2 hr hold	C	Si	Ti
Phase A	55	13	32
Phase B	58	7	35
Phase C	47	22	31

In atomic percent

4 hr hold	C	Si	Ti
Phase A	53	14	33
Phase B	42	25	33

In atomic percent

Microcracking is lessened as titanium carbide at the core is reacted away.

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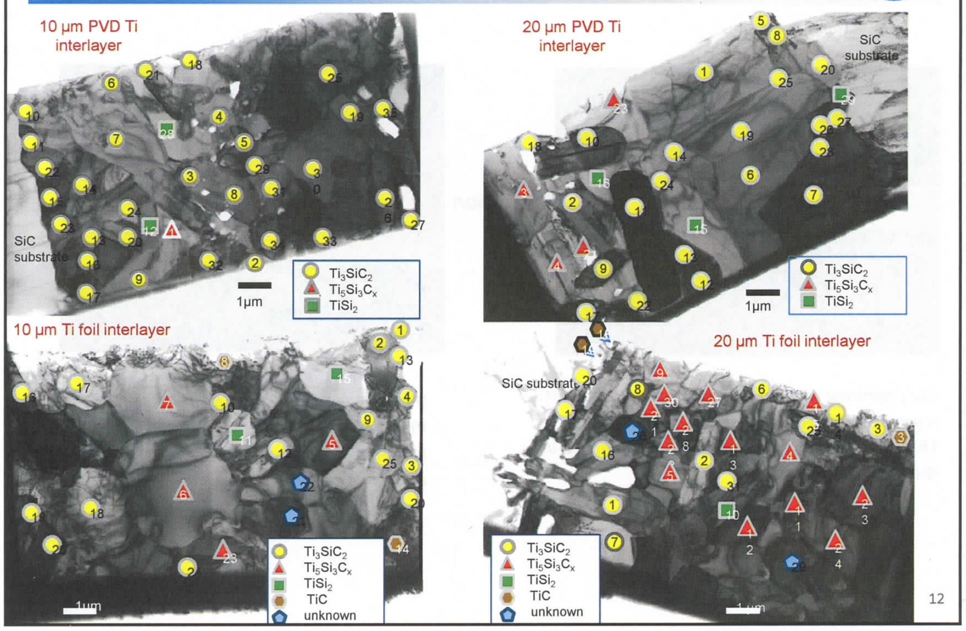
TEM Analysis of Diffusion Bonds Processed for 2 Hr



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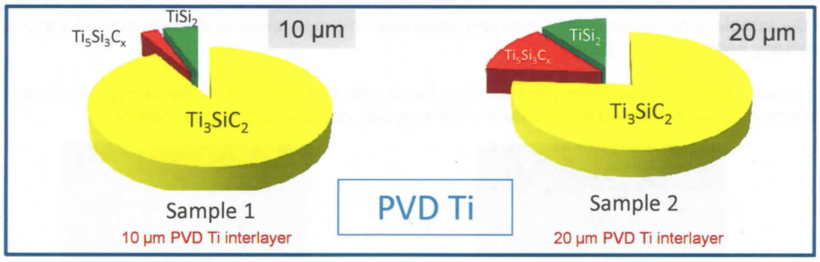
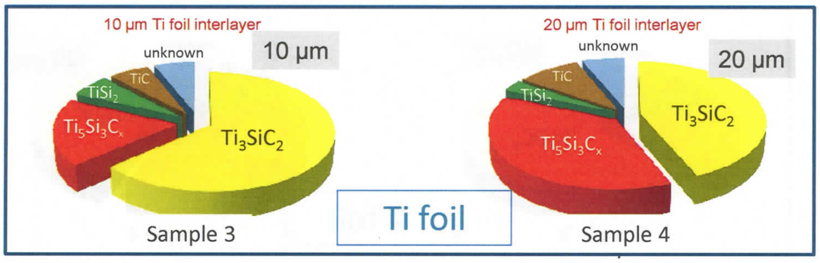
Fractions of Phases in Each Sample





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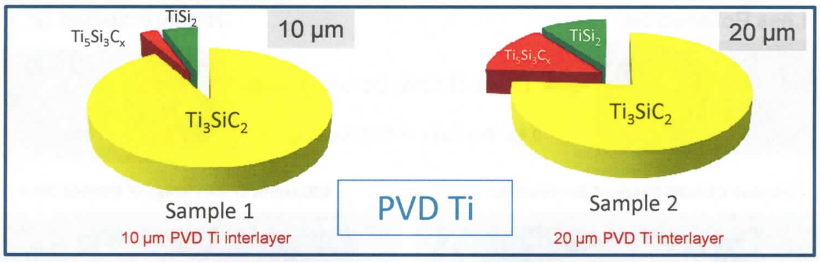
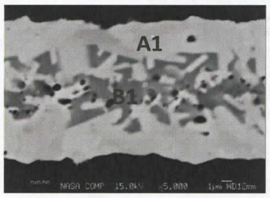
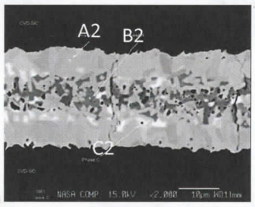
Fractions of Phases in Each Sample







In the sample 1, the fraction of Ti_3SiC_2 was highest and the fraction of $Ti_5Si_3C_x$ was lowest or zero. This suggests that the reaction may have been complete.

On the other hand, in the sample 2, less Ti_3SiC_2 and more $Ti_5Si_3C_x$ formed. Additionally, an intermediate $Ti_5Si_3C_x$ has anisotropic thermal expansion. Thus, much microcracking was observed in the sample 2.

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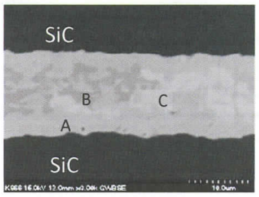
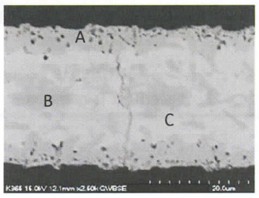
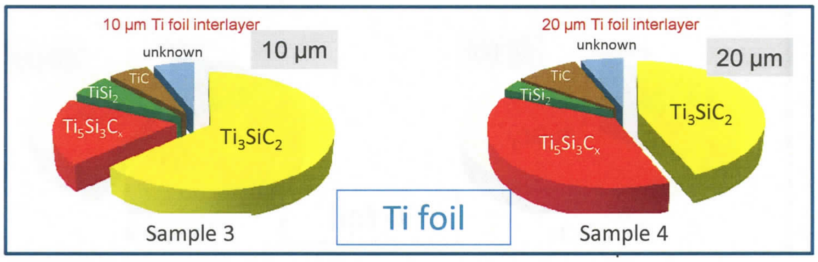
Fractions of Phases in Each Sample

In the sample 3, microcracks were scarcely observed. This may relate to the presence of a phase with relatively high Si content phase.

Moreover, if the unknown phase detected in the sample 4 has content similar to that of phase "common phase A with lower Si content", this may also cause microcracks in sample 4.







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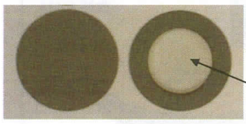
Non-Destructive Evaluation (NDE) sing Ultrasonic Immersion Shows Very Good Bonding of 1" Discs

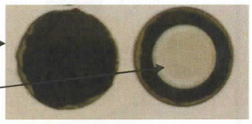
Less Polished SiC

Highly Polished SiC

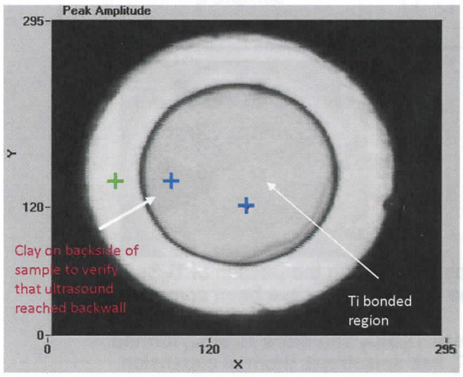


Discs Before Bonding

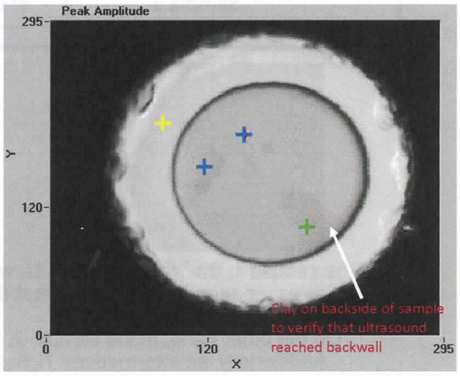
0.65" Diameter PVD Ti Coating



Ultrasonic C-scan Image of Bonded Discs



Ultrasonic C-scan Image of Bonded Discs



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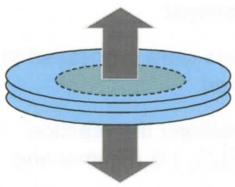
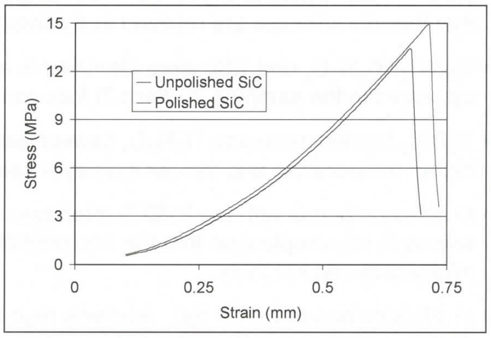
High Strength of Bonds Greatly Exceed the Application Requirements

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).

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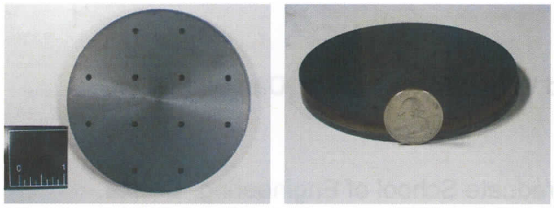
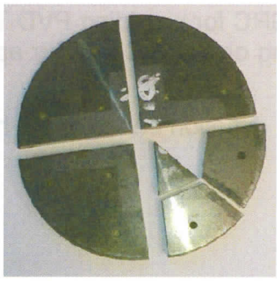
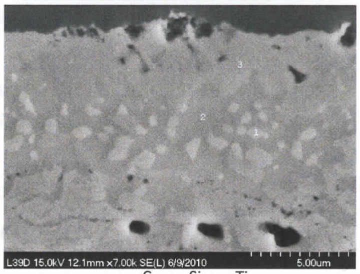
Demonstration of Diffusion Bonding on 4" Diameter CVD SiC Discs



Joined 4" diameter SiC discs.

Sectioned for microscopy and to demonstrate leak free at bonded and machined edges.





	C	Si	Ti
Phase 1	57	9	34
Phase 2	34	28	38
Phase 3	50	14	36

In atomic percent

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Conclusions

- Joining approaches using diffusion bonding are being developed and optimized so that thermal stresses are relieved and microcracks do not form.
- Ti_3SiC_2, $Ti_5Si_3C_x$ and $TiSi_2$ were identified in all samples. TiC and unknown phases appeared in the samples in which Ti foils were used as interlayer.
- Ti_3SiC_2 formed more and $Ti_5Si_3C_x$ formed less when samples were processed at higher temperature and thinner interlayer samples were used.
- In diffusion bonds with the PVD Ti Interlayer, for thinner interlayer the reaction seems to be complete so that the intermediate phase ($Ti_5Si_3C_x$) is minimal and microcracks do not form.
- In diffusion bonds with Ti foil, relatively high Si content phase may enhance ductility whereas low Si content phase may cause microcracking.
- Joining approaches will enable the fabrication and utilization of ceramic components.
- The next steps are to further evaluate the mechanical and thermal performance of the joints and to fabricate and characterize the SiC injector.

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Acknowledgements

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Thank you for your attention.