DIFFUSION BONDING AND BRAZING APPROACHES FOR SILICON CARBIDE BASED SYSTEMS

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Outline

- **Background – Objectives, Issues, and Applications**
- **Joining Challenges**
- **Processing, Characterization, and Results**
  - **Diffusion Bonding: SiC to SiC**
  - **Brazing: Kovar to SiC**
- **Conclusions**

Research Approach

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: ceramic turbine vanes, blades, injectors, 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).

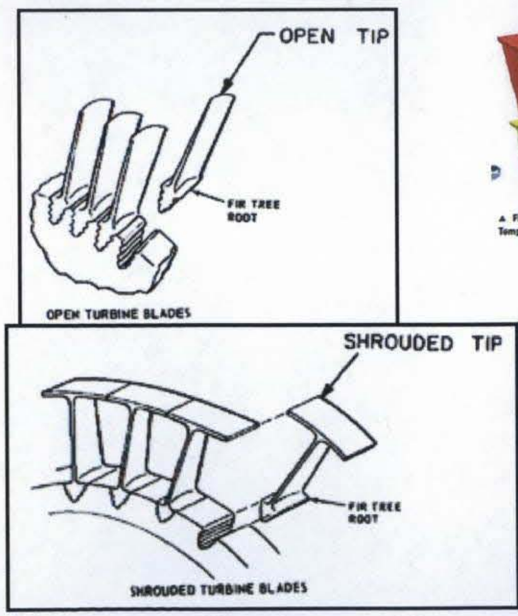
• Develop **ceramic to metal** joining technologies that enable ceramic components to be integrated into metallic based engine systems.

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

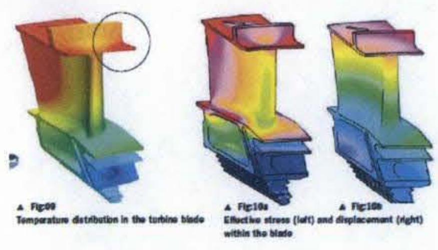
Current Turbine Blade Design Issues

Metallic Turbine Blade Tip Shroud

+ Prevent excessive blade vibrations
+ Prevents gas losses over blade tip
+ Prevents blade twisting
+ Allows for aerodynamic advantage of thinner blades
- Requires that the turbine run cooler or at lower RPM because of extra mass at the blade tip (issues for metallic materials but not ceramics – lower densities and higher temperature capability)



GlobalSucurity.org, Reference Text AL0993, paragraphs 1.15-1.25



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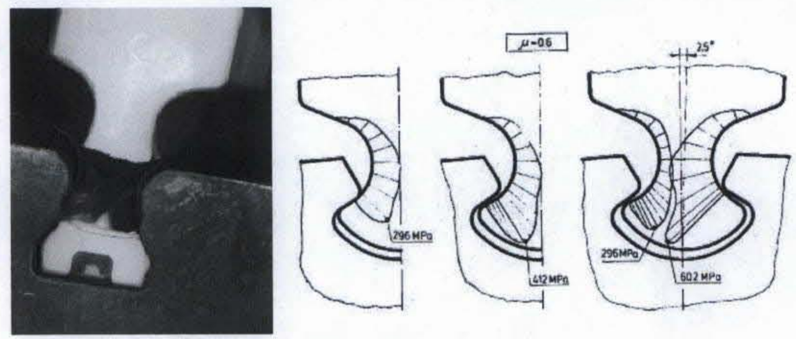
Ceramic Blades Inserted into a Metallic Disk

- High thermal stress due to thermal incompatibilities between the ceramic and metal
- Require a compliant layer however blade failures are often initiated at the compliant layer



Mark van Roode, "Advances in the Development of Silicon Nitride and Other Materials", Environmental Barrier Coatings Workshop, November 6, 2002, Nashville, TN.

Ceramic joining technologies could allow CMC blades to be fabricated with a joined tip shroud and could allow for blade insertions with lower operational stresses.

Other Ceramic Component Joining Issues

INTEGRAL ROTORS

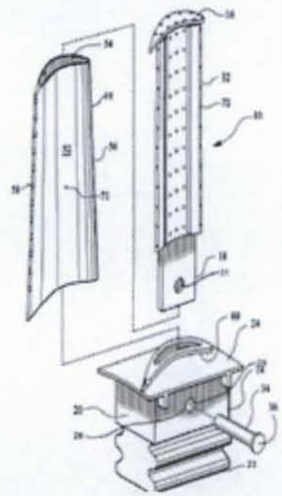
- Integral Rotors are Replacing Metal Disks with Inserted Blades
- No Compliant Layer with Disk
- Primarily Small Parts
- Ability to Fabricate Larger Parts Has Improved
- Attachment of Ceramic Rotor to Metal Shaft

Industry Direction of Silicon Nitride Integral Rotors

Mark van Roode, Solar Turbines

Ingersoll Rand Silicon Nitride Rotor, DOE Microturbine Program (top) H-T. Lin, ORNL

Hybrid Blade – Spar and Shell Concept



Preferred approach – spar and shell concept, where the shell could be a CMC and the spar could be cooled single crystal nickel alloy or cooled silicon nitride

- Potential for +250 F increase in blade temperature capability

Shell does not have high stress

Challenges:

Design and structural optimization

CMC blade architecture for small blades and manufacturing

Erosion resistant thin environmental barrier coating

Joining/attachment of metallic rotor to blade

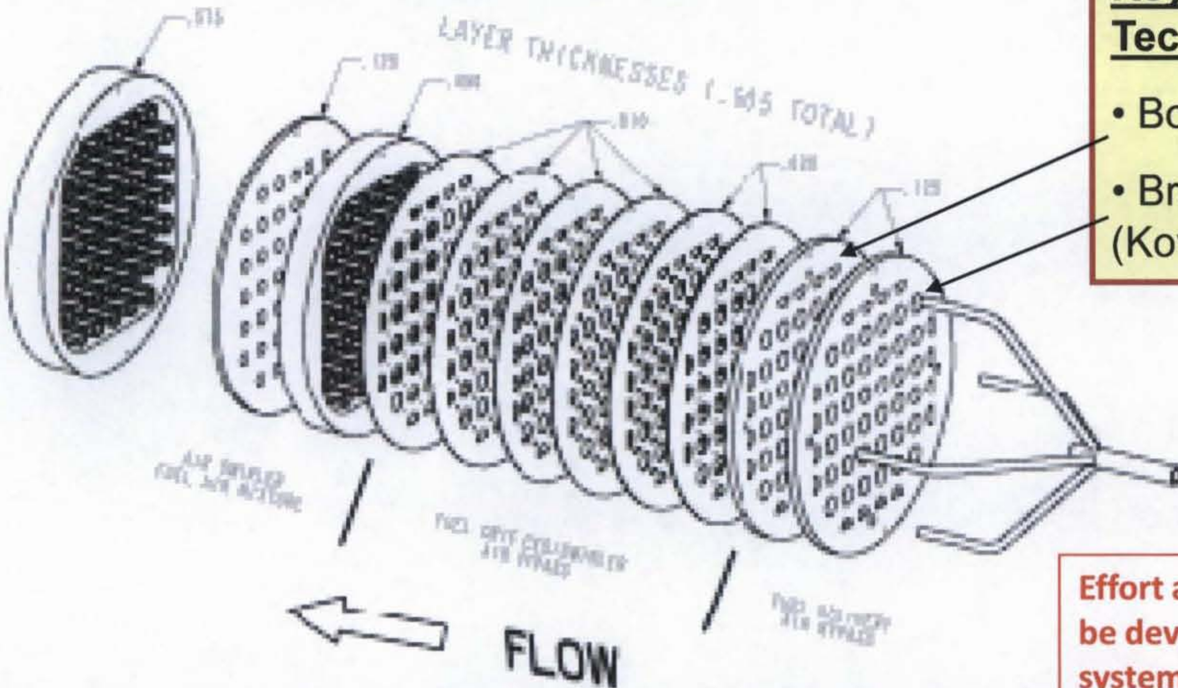
Impact resistance

Hybrid blade will enable the goal for increasing engine power by 25% and reduction of fuel consumption by 10%

Ceramic joining technologies could allow for integration of a silicon nitride integral rotor to the metallic shaft and allow for fabrication of a hybrid blade dual material concept.

Joining of Silicon Carbide to Silicon Carbide and to Metal

Fabrication and Testing of a Lean Direct Injector with MEMS Integration



Key Enabling Technologies:

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

Effort allows for joining approaches to be developed for the SiC material system for SiC to SiC and SiC to metal joining. Compatible to the CMC, SiC/SiC, material system.

Develop and characterize the joining and integration technologies required for injector fabrication:

- Diffusion Bonding of SiC Laminates

- Brazing Metallic Tubes to SiC

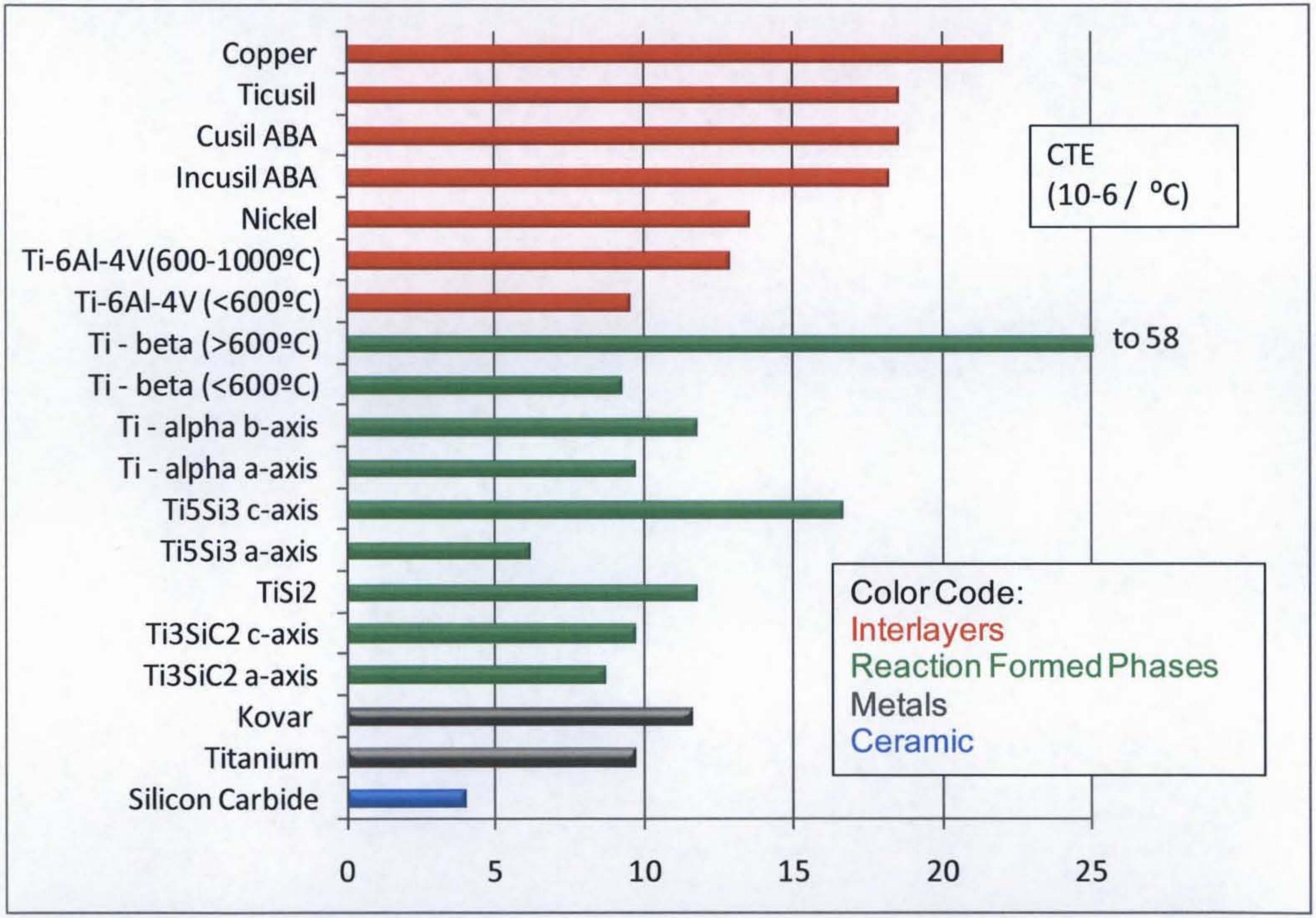
Technical Challenges in Integration of Ceramic-Metal *vs* Ceramic-Ceramic Systems

Ceramic-Metal System		Ceramic-Ceramic System
Flow and wettability		Reaction and diffusion
Roughness		Roughness
Residual stress (ΔCTE)	Common Issues	Residual stress (ΔCTE)
Multi-axial stress state	Common Issues	Multi-axial stress state
Joint design	Common Issues	Joint design
Joint stability in service	Common Issues	Joint stability in service
Metal – *forgiving*		Ceramic – *unforgiving*
Elastic-plastic system		Elastic-elastic system
Lower use temperatures		Higher use temperatures
Less aggressive environment		More aggressive environment

Joining Challenges

- Thermal and chemical incompatibilities between different materials: thermal expansion mismatches, low thermal conductivity, formation of brittle phases
- Obtaining bonds that are high strength, leak free, and stable
- Lack of ASTM standards for mechanical tests
- Non-wetting of ceramics during brazing and interlayer flow with other processing methods
- Cracking in the bond layers and/or substrates

Coefficients of Thermal Expansion



Thermal expansion mismatches contribute to thermal stresses, cracking, and debonding at the joint.

Diffusion Bonding of SiC to SiC

Diffusion Bonding of SiC

Materials (sized at 1"x1" or ½" x 1")

- SiC
  - α-SiC (CRYSTAR from Saint-Gobain)
  - CVD SiC (TREX Enterprises)
  - **CVD SiC (Rohm & Hass)**

- Titanium
  - Ti-6Al-4V (weight %), 38 micron foil
  - pure Ti, 10 and 20 micron PVD coating
  - **pure Ti, 10, 20, and 50 micron foils**

Hot Pressing Conditions

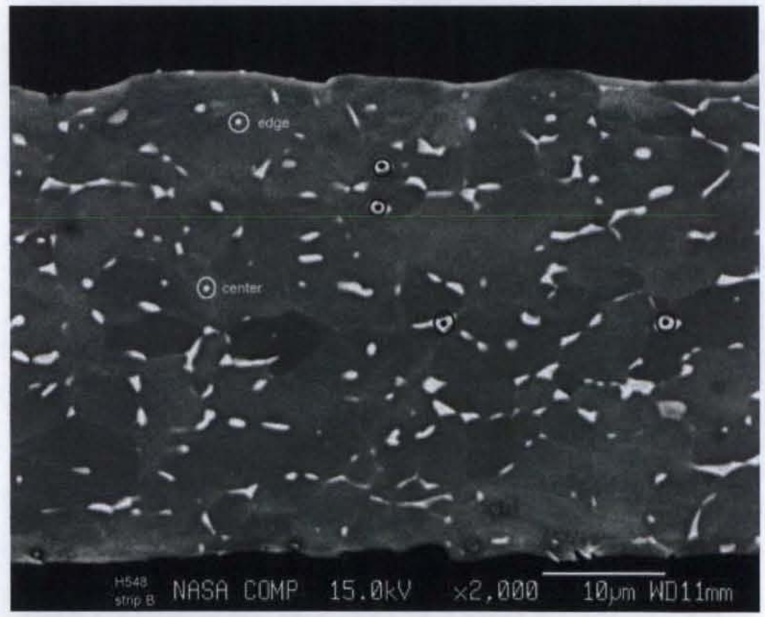
- Atmosphere: Vacuum
- Temperature: 1200ºC, 1250ºC, 1400ºC
- Pressure: 24-30MPa
- Duration: (1-16 hr)
- Cool down: 2 ºC/min

Analysis: **optical and scanning electron microscopy, microprobe,** NDE, tensile test

Processing Matrix at **1200ºC** and 1400ºC

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

Ti-6Al-4V (weight %)

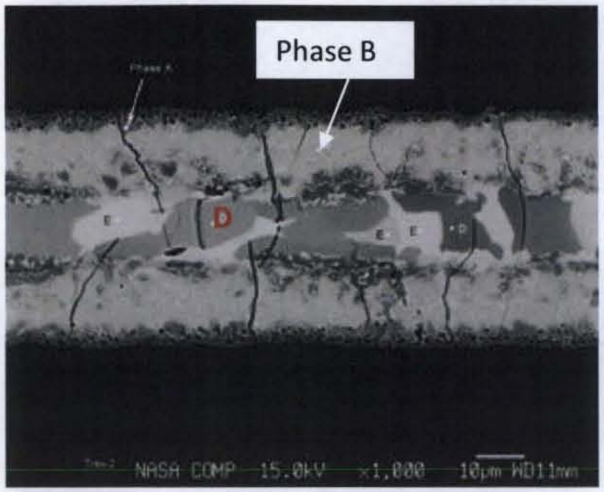


Microprobe from the cross-section of alloyed Ti foil

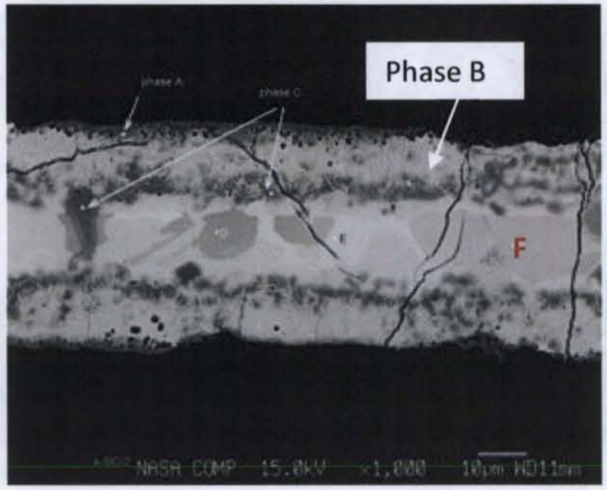
	Phase	Al	Fe	Ti	V	Total
Atomic Ratio	Grey Phase	10.196	0.042	86.774	2.988	100.000
Weight (%)	Grey Phase	5.999	0.051	90.632	3.318	100.000
Atomic Ratio	White Phase	4.841	1.850	76.507	16.803	100.000
Weight (%)	White Phase	2.748	2.172	77.084	17.997	100.000

Diffusion Bonding with the Alloyed Ti Foil

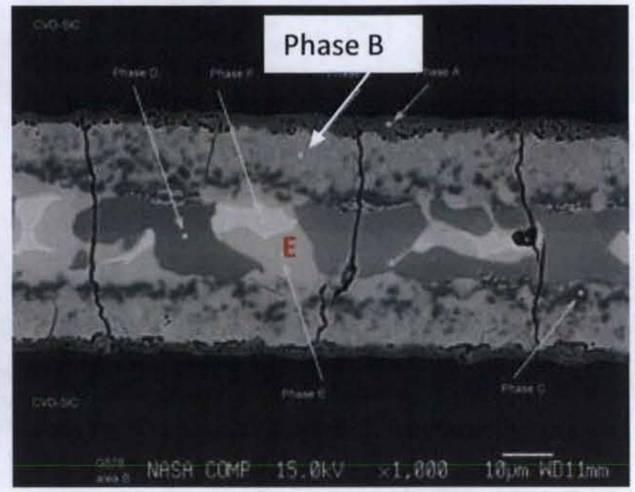
TREX CVD SiC



CRYSTAR α-SiC



Rohm & Haas CVD SIC



Microcracks formed regardless of the substrate and variations in the processing conditions: 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) - Ti5Si3CX (Ti5Si3) is highly anisotropic in its thermal expansion where CTE(c)/CTE(a) = 2.72 (Schneibel et al).

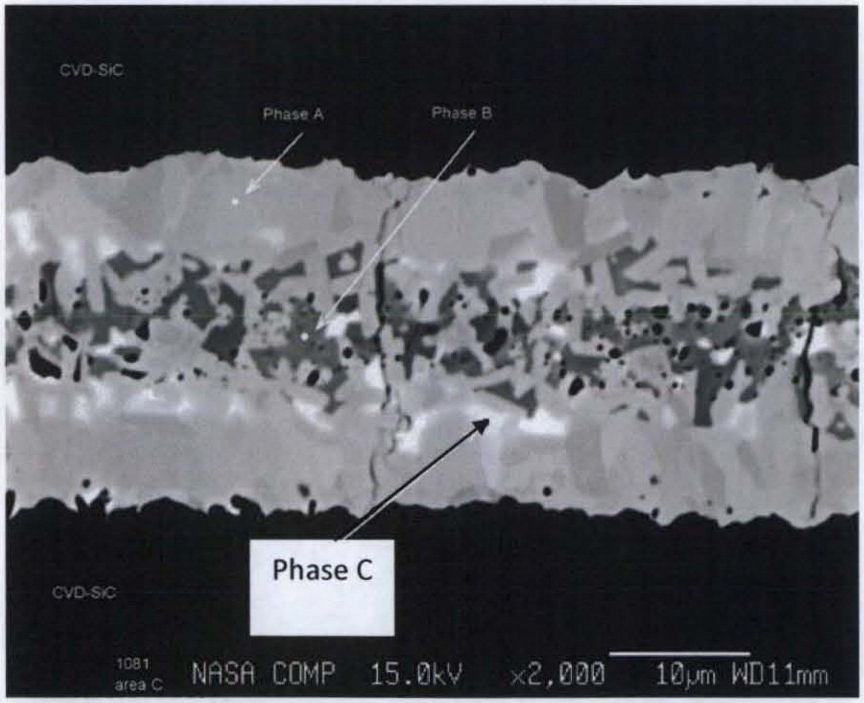
- Central core of diffusion bond has concentrated alpha and beta Ti alloy phases. The alpha phase has an anisotropic thermal expansion which is 20 % greater along the c-axis (Boyer, Welsch, and Colling). Also, the beta phase has a thermal expansion that is 6 x higher in the temperature range of 600-1000˚C (5.8x10-5/˚C) compared the thermal expansion below 600˚C (9.2x10-6/˚C) (Elmer et all).

Diffusion Bonds Using PVD Ti as the Interlayer

20 Micron Ti Interlayer

Microcracking is still present due to the presence of Ti5Si3CX.

Naka et al suggest that this is an intermediate phase.



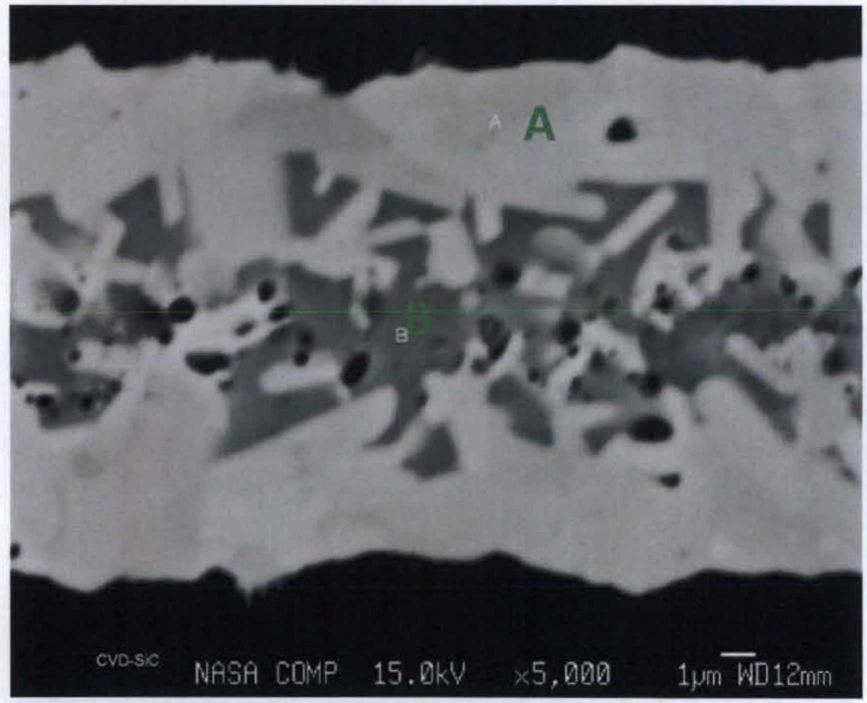
Phases in bond with the 20 μ Ti Interlayer – Atomic Ratios

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 Ti5Si3CX is present.

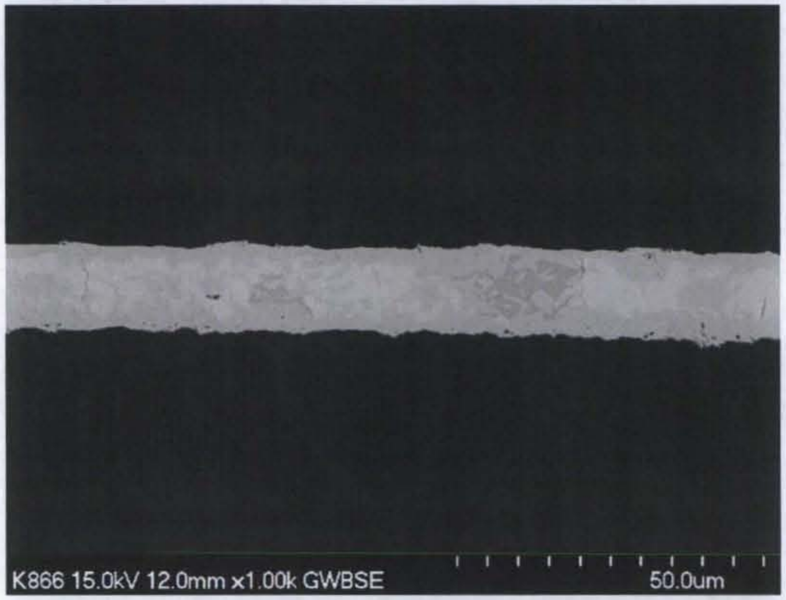
Thin interlayers of pure Ti down-selected as the preferred interlayer.

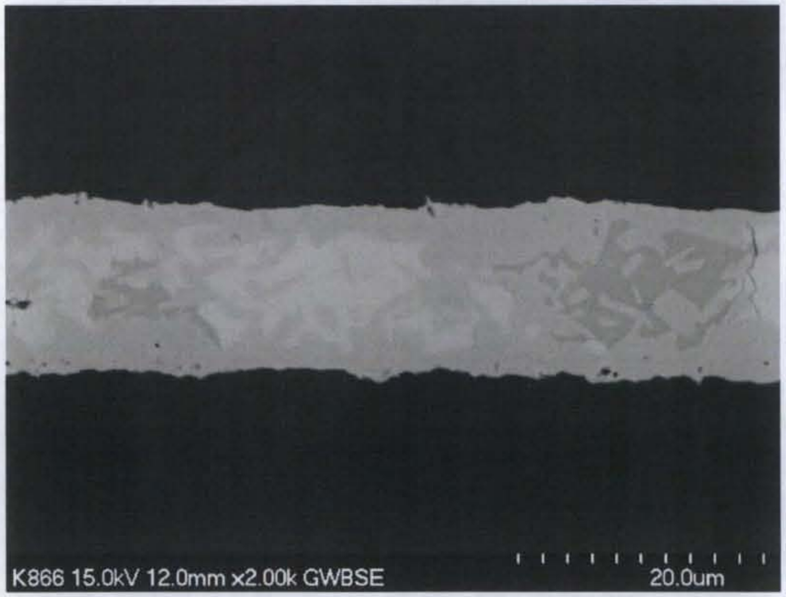


Phases in bond with the 10 μ Ti Interlayer – Atomic Ratios

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

Diffusion Bonds from 10 μm Ti Foil at 1200ºC – 2 hr hold

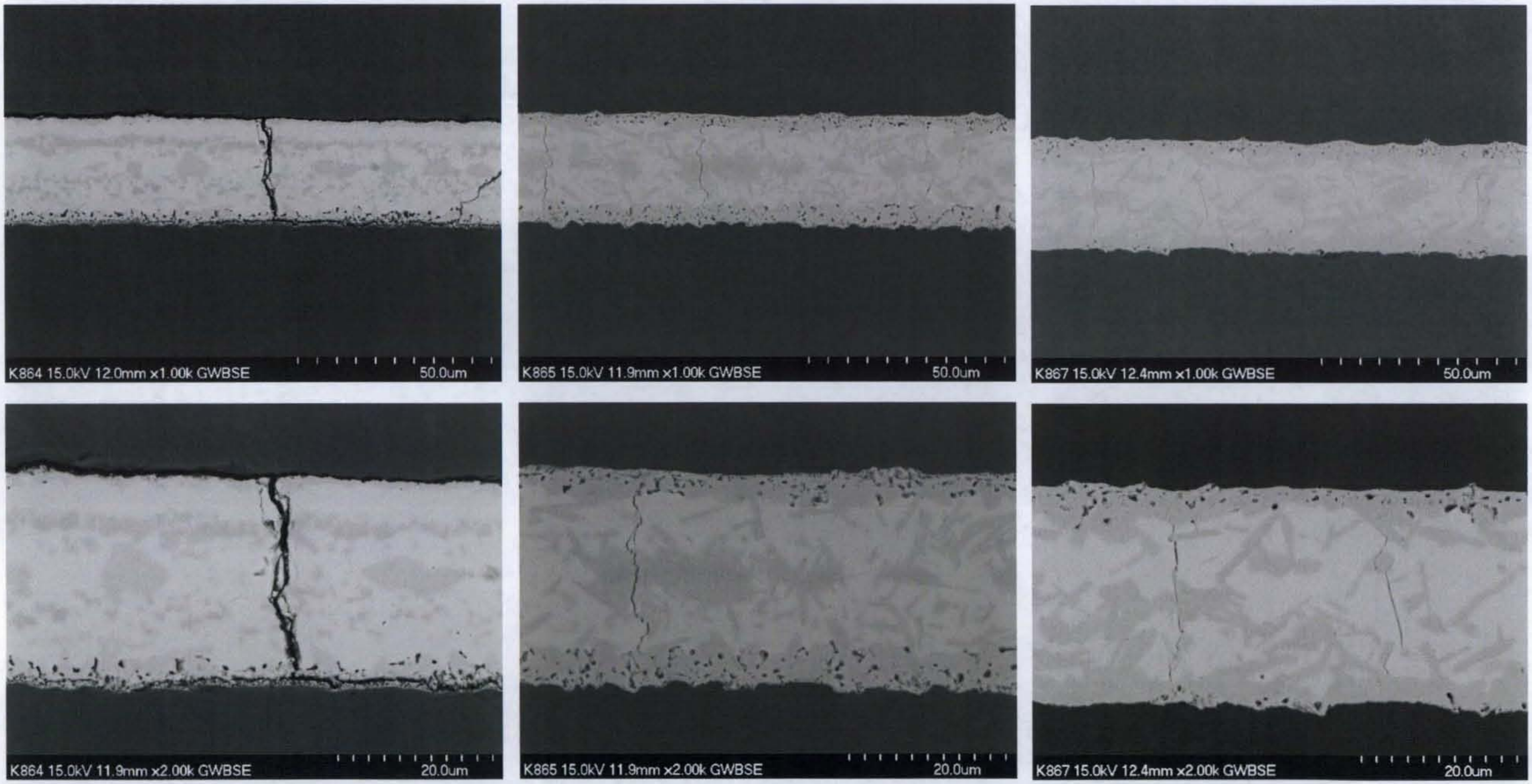




Three phases are observed with minor microcracks that do no traverse completely through the joint thickness.

Diffusion Bonds from 20 µm Ti Foil at 1200ºC

1 hr hold | 2 hr hold | 4 hr hold



Larger cracks are observed with shorter hold times. The predominance of phases and the type of phases observed changes with time. EDS will be conducted on these samples.

Summary of SiC to SiC Diffusion Bonding Results

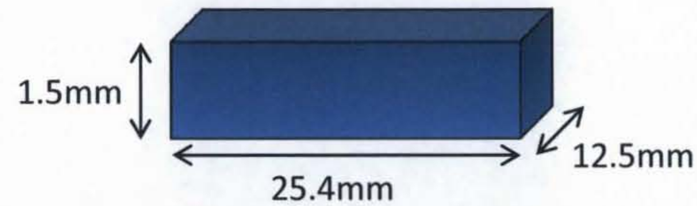
Interlayer Material	Interlayer Thickness (µM)	Temp. (ºC)	Time (hr)	Bond Quality	Comments
Ti foil	10	900	2	poor	gaps all along joint
Ti foil	10	900	4	poor	did not bond
Ti foil	10	1000	2	poor	did not bond
Ti foil	10	1000	4	poor	gaps all along joint
Ti-6Al-4V	38	1250	2	ok	microcracks
PVD Ti	10	1250	2	good	no microcracks
PVD Ti	20	1250	2	ok	microcracks
Ti foil	10	1250	2	good	no microcracks
Ti foil	20	1250	1	good	minmal microcracking
Ti foil	20	1250	2	ok	intermediat phase and microcracking
Ti foil	20	1250	4	good	minimal microcracking
Ti foil	10	1400	2	good	no microcracks
Ti foil	20	1400	1	good	no microcracks
Ti foil	20	1400	2	ok	intermediate phase and minimal microcracks
Ti foil	20	1400	4	good	minimal microcracks
Ti foil	50	1400	1	ok	microvoids and minimal microcracks
Ti foil	50	1400	2	ok	microcracks and some voids
Ti foil	50	1400	4	ok	minimal microcracks
Ti foil	50	1400	8	ok	intermediate phase and microcracks
Ti foil	50	1400	16	ok	intermediate phase and microcracks

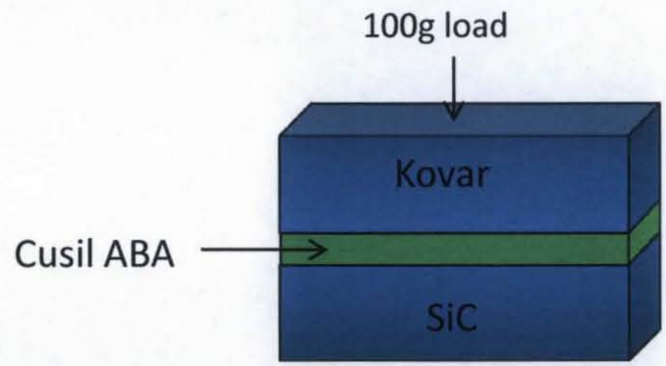
Brazing of Kovar to SiC

SiC to Kovar Joining

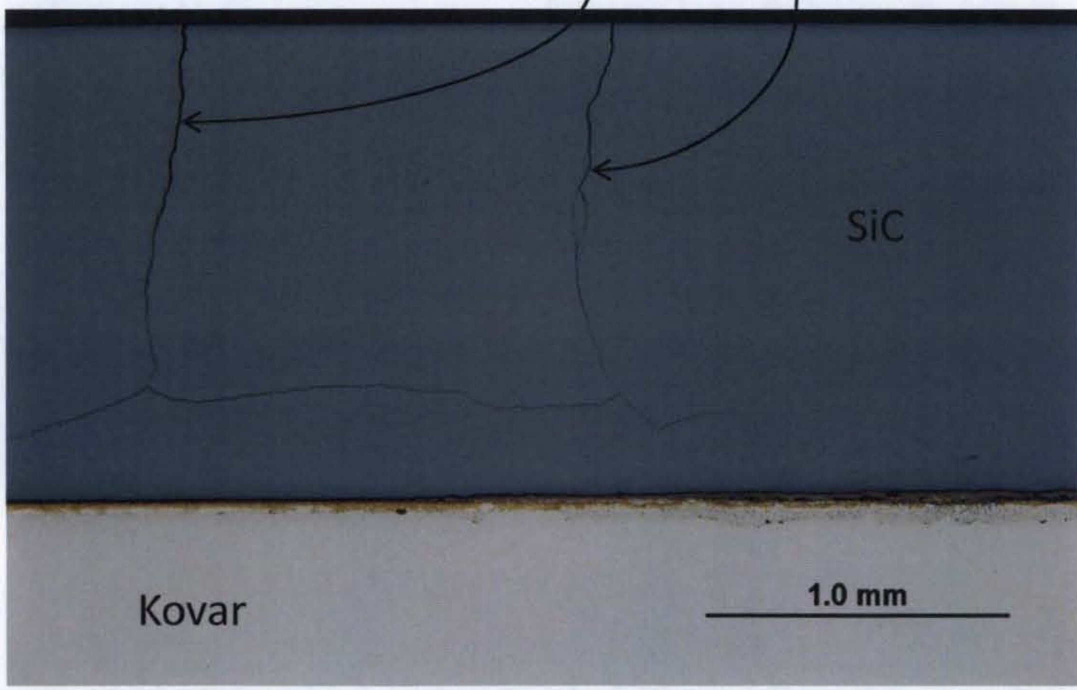
Experimental Procedure

- Ceramic and metal substrates were ultrasonically cleaned in Acetone for 10 minutes
- Substrates were sandwiched together with a 100g load applied to the assembly
- Heated to 10°C above the braze liquidus temperature
- Isothermally held for 5 minutes under vacuum
- Cooled to room temperature at 2°C/min
- Mounted in epoxy, polished and the joints were characterized





Residual Stresses in SiC



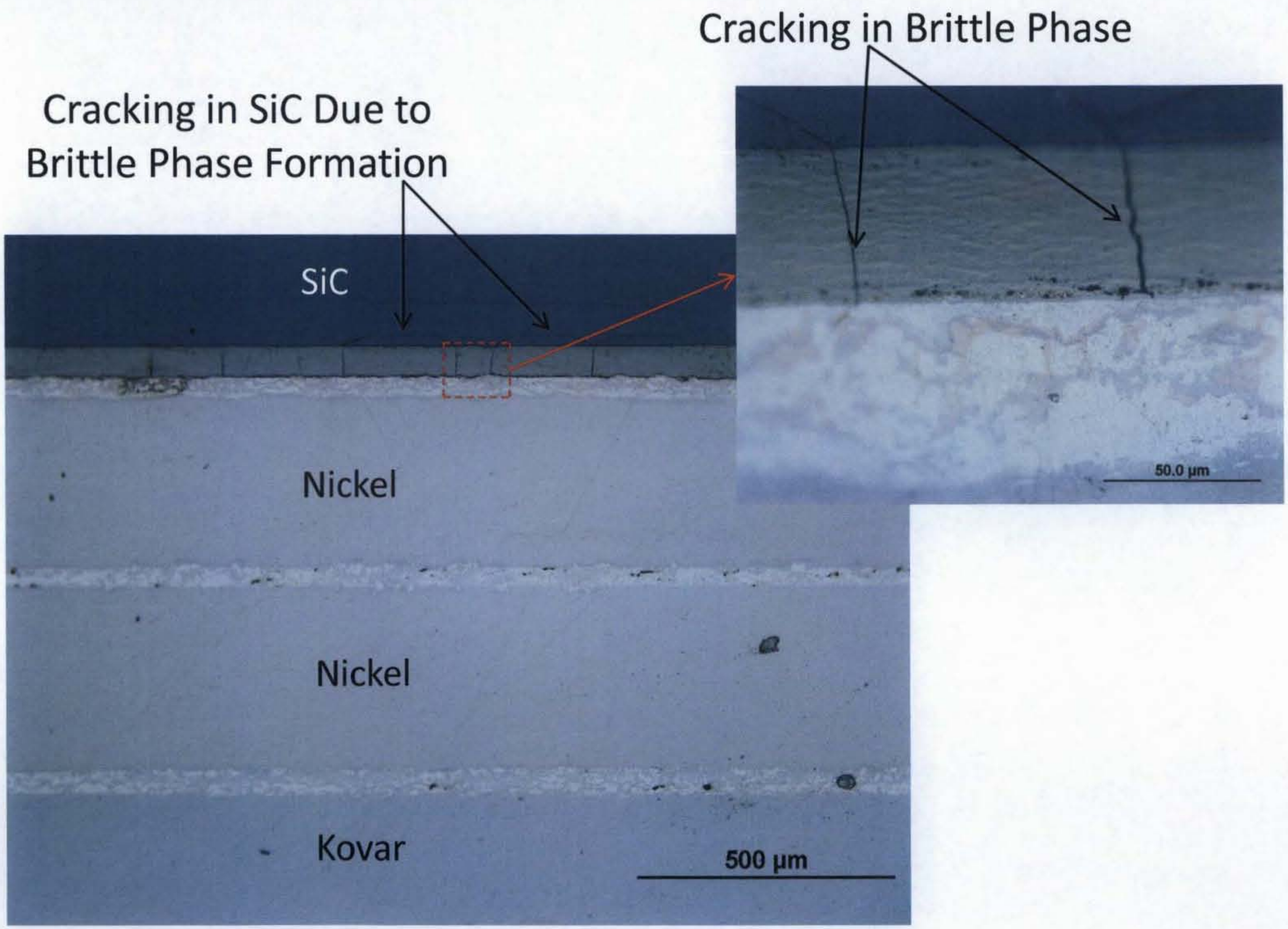
Stress Induced Cracking Due to Differing CTE

$CTE_{SiC} = 4.0 \times 10^{-6}$ /°C

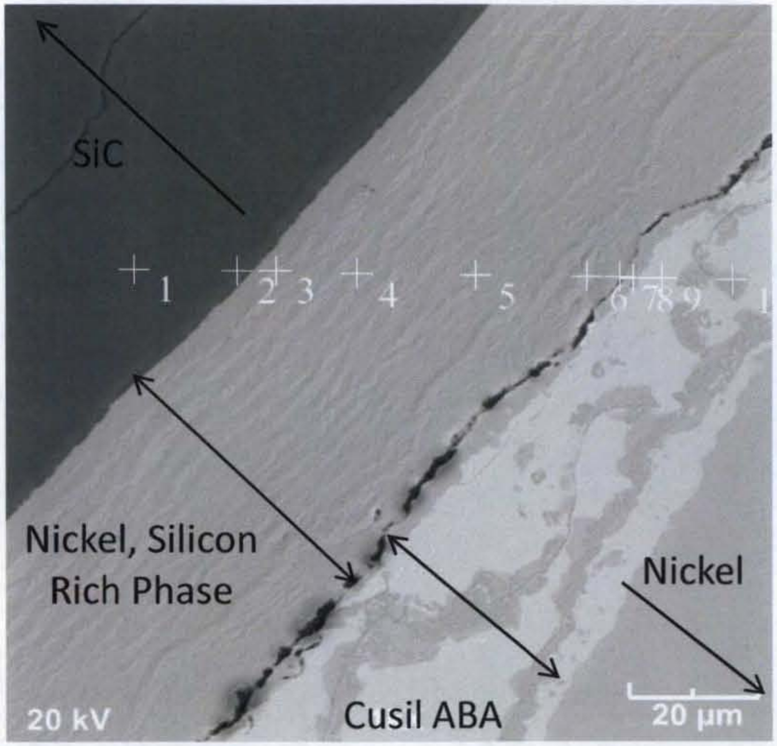
$CTE_{Kovar} = 11.5 \times 10^{-6}$ /°C

Cusil ABA

Formation of Brittle Phase



Electron Dispersion Spectrometry (EDS)



EDS analysis shows that the brittle phase is likely a Nickel Silicide

The phase is made up of about 74.5% Ni, 16.1%Si, 6.3% Cu and small amounts of Ag, Ti, Co, and Fe

Conclusion

- Joining approaches are being developed and optimized so that thermal stress are relieved and cracks do not form.
- 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 phases.
- Diffusion bonds fabricated with thinner interlayers of 10 micron pure Ti (foils and coatings) gave better diffusion bonds than the alloyed Ti foil and thick foils.
  - Fewer and preferred phases were formed which resulted in bonds without microcracks.
- For brazing metals (i.e. Kovar) to SiC a multi-layer approach looks promising for alleviating thermal stresses
- Joining approaches will enable the utilization of ceramic components

Acknowledgements

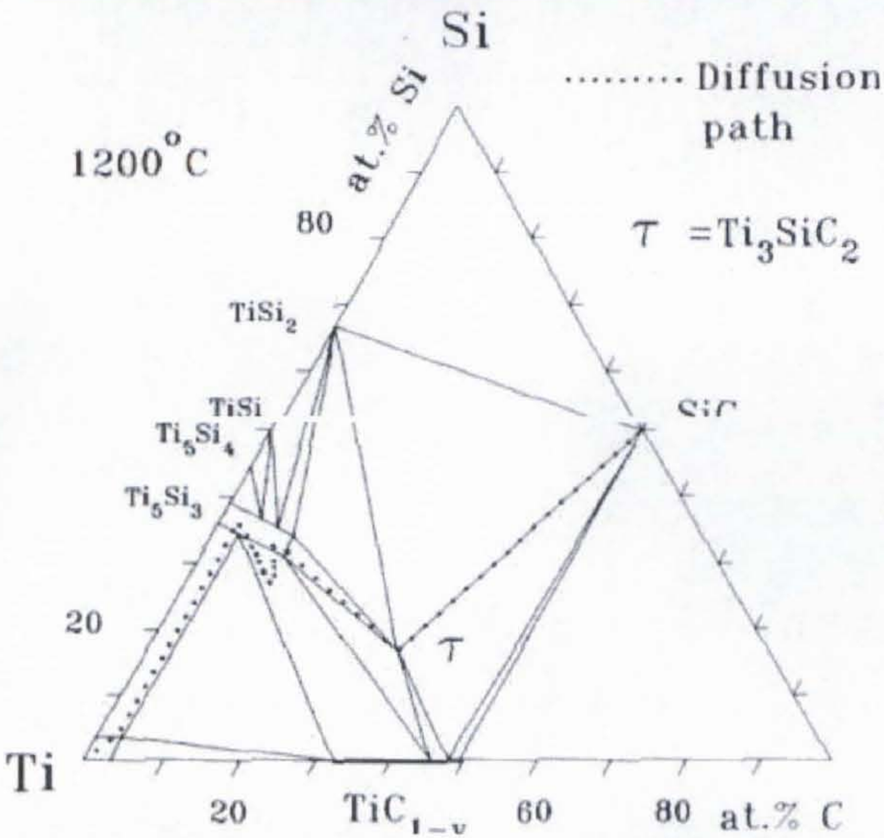
- The NASA Subsonic Fixed Wing Project and Dr. Dan L. Bulzan and at NASA GRC for providing support.

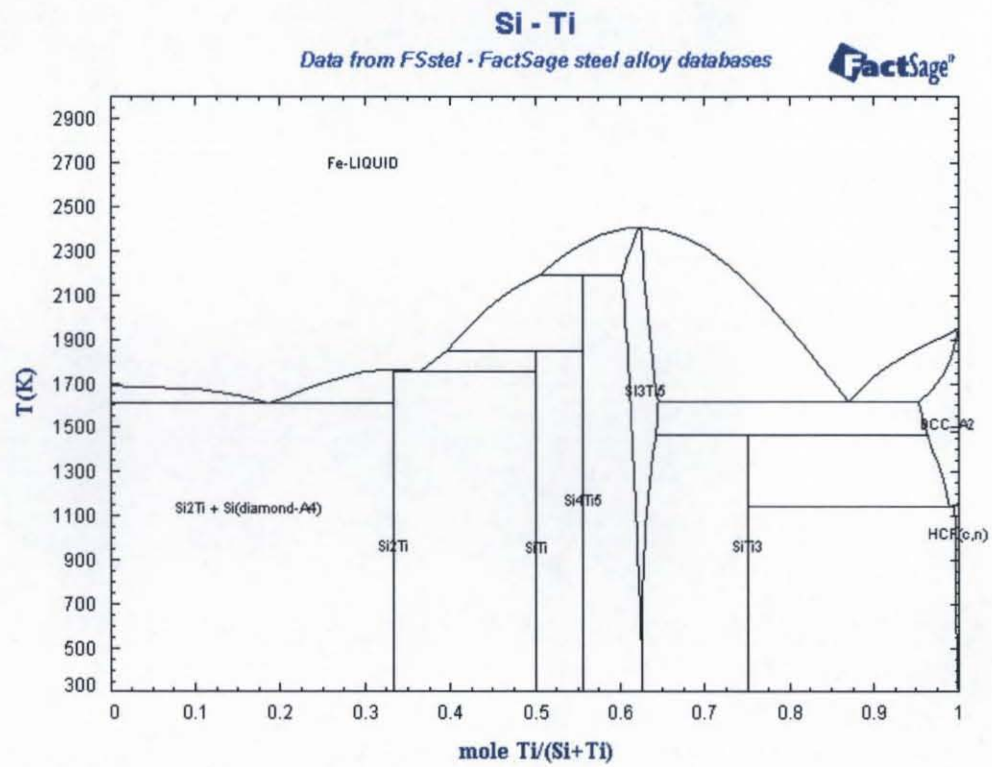
- Dr. Robert Okojie of NASA GRC for providing PVD Ti Coated CVD SiC and for collaborating on the SiC injector application.

- Robert Angus for processing diffusion bonds in the hot press.

- Dr. Rajiv Asthana from the University of Wisconsin-Stout for his helpful discussions.

The Phases of Ti-Si-C and the Intermediate Phase of Ti_5Si_3





An isothermal section of the Ti-Si-C phase diagram at 1200'C. The diffusion path is denoted by the dotted line. $SiC/Ti_3SiC_2/Ti_5Si_3$/two-phase Ti_5Si_3 plus $TiC_{1-y}/Ti_5Si_3/Ti$

F. Goesmann and R. Schmid-Fetzer, Semicond. **Sd.** Technol. **10** (1995) 1652-1658.