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Integration Science and Technology of Advanced Ceramics for Energy and Environmental Applications

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Abstract

The discovery of new and innovative materials has been known to culminate in major turning points in human history. The transformative impact and functional manifestation of new materials have been demonstrated in every historical era by their integration into new products, systems, assemblies, and devices. In modern times, the integration of new materials into usable products has a special relevance for the technological development and economic competitiveness of industrial societies. Advanced ceramic technologies dramatically impact the energy and environmental landscape due to potential wide scale applications in all aspects of energy production, storage, distribution, conservation, and efficiency. Examples include gas turbine propulsion systems, fuel cells, thermoelectrics, photovoltaics, distribution and transmission systems based on superconductors, nuclear power generation, and waste disposal.

Robust ceramic integration technologies enable hierarchical design and manufacturing of intricate ceramic components starting with geometrically simpler units that are subsequently joined to themselves and/or to metals to create components with progressively higher levels of complexity and functionality. However, for the development of robust and reliable integrated systems with optimum performance under different operating conditions, the detailed understanding of various thermochemical and thermomechanical factors is critical. Different approaches are required for the integration of ceramic-metal and ceramic-ceramic systems across length scales (macro to nano). In this presentation, a few examples of integration of ceramic to metals and ceramic to ceramic systems will be presented. Various challenges and opportunities in design, fabrication, and testing of integrated similar (ceramic-ceramic) and dissimilar (ceramic-metal) material systems will be discussed. Potential opportunities and need for the development of innovative design philosophies, approaches, and integrated system testing under simulated application conditions will also be presented.

Integration Science and Technology of Advanced Ceramics for Energy and Environmental Applications

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Outline

- **Introduction and Background**
- **Technical Challenges in Integration**
  - ***Similar vs Dissimilar Systems***
- **Ceramic Integration Technologies**
  - ***Wetting and Interfacial Effects***
  - ***Ceramic-Metal Systems***
  - ***Ceramic-Ceramic Systems***
  - ***Testing and Characterization***
- **Concluding Remarks**

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Ever Increasing Global Energy Demand….

Source: US DOE

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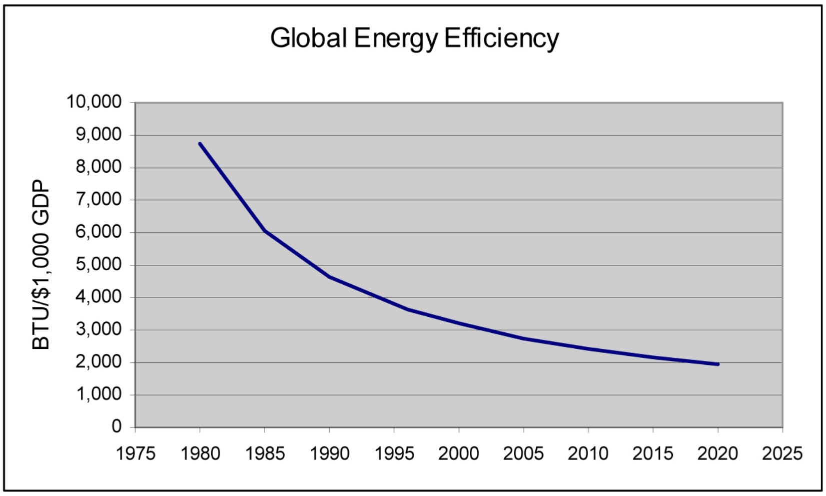
Advanced Materials and Technologies Play a Key Role in Improving Energy Efficiency



Source: Millennium Project, 2020 Global Energy Delphi Round 2

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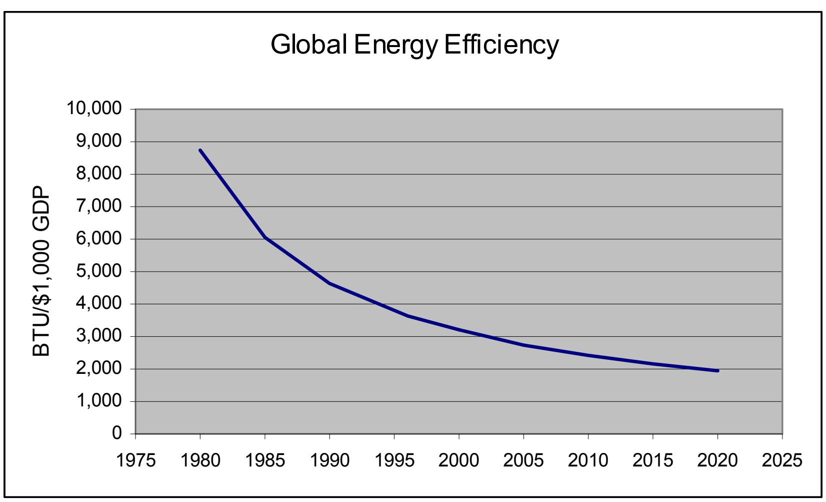

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Critical Role of Advanced Ceramic Technologies in Energy Systems

Energy Production
- Fuel Cells
- Thermoelectrics
- Photovoltaics
- Nuclear Systems
- Wind Energy
- Biomass

Energy Storage and Distribution
- Batteries
- Supercapacitors
- Hydrogen Storage Materials
- Thermal Energy Storage/PCMs
- High Temperature Superconductors

Energy Conservation and Efficiency
- Ceramic Components (Gas Turbines)
- Heat Exchangers and Recuperators
- Coatings, Bearings

Ceramic Integration Technologies

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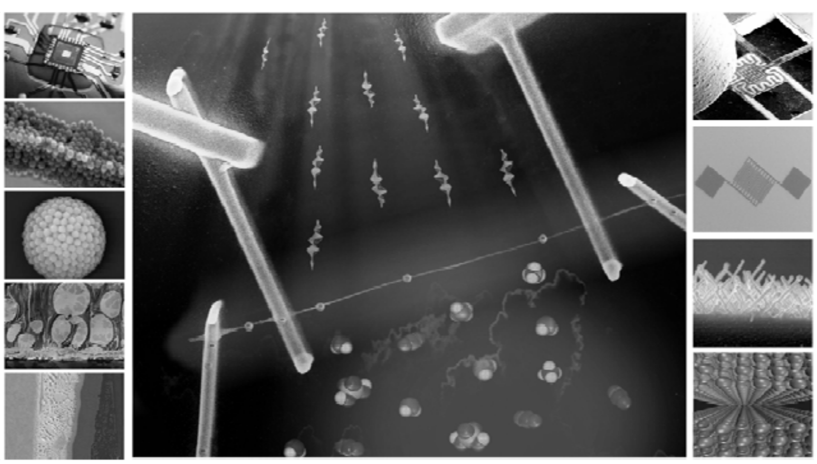
Ceramic Integration Technologies: *From Macro to Nanoscale*



A wide variety of integration technologies are needed for manufacturing and application of components of different length scales

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Overview of Key Ceramic Integration Technologies

Joining Technologies
- Ceramic-Ceramic
- Ceramic-Metal

Component & System Level Integration

Repair/Refurbishment
- In-Situ Repair
- Ex-Situ Repair

Mechanical Fastening
- Ceramic-Ceramic
- Ceramic-Metal
- Rivets/Bolts

Robust Manufacturing
- Large Components
- Complex Shapes
- Multifunctional

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

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

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Integration of Metals to Ceramics and Composites Using Metallic Interlayers

Metallic Systems:
- Titanium
- Inconel and Other Ni-Base Superalloys
- Kovar, Cu-Clad Mo,
- Stainless Steels, W

Active Metal Brazing
Soldering

Ceramics/Composite Systems:
- SiC, Si_3N_4
- YSZ, Alumina
- C/C Composites
- C/SiC, SiC/SiC

Interlayer Systems:
- Active Metal Brazes (Ag, Cu, and Pd based)
- Metallic Glass Ribbons
- Solders (Zinc based)

Technical Issues:
- **Melting range / behavior**
- **Wetting characteristics**
- **Flux or atmosphere compatibility**
- **Compositional compatibility**
- **Cost & availability**

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Wetting and Interfacial Phenomena in Ceramic-Metal System

Key Challenges:

- ***Poor Wettability of Ceramics and Composites:*** **(poor flow and spreading characteristics)**
- ***Surface Roughness and Porosity of Ceramic Substrates***
- ***Thermoelastic Incompatibility***

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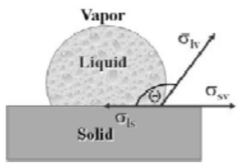
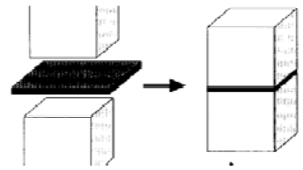
Wettability is Important Factor in Integration of Ceramics to Metals

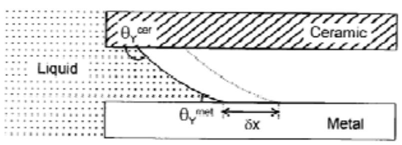
Young's equation
$\sigma_{sv}-\sigma_{sl} = \sigma_{lv}\cos\theta$



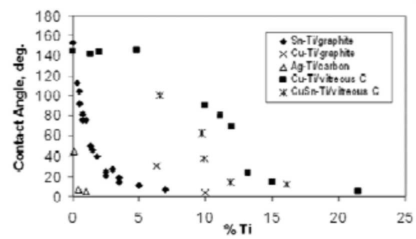
Contact angle of braze should be small



Braze layer melts and spreads between the substrates to form the joint



Ordinary braze alloys wet the metal but not the ceramic systems



Must use 'active' brazes that wet and bond with both metal and ceramics

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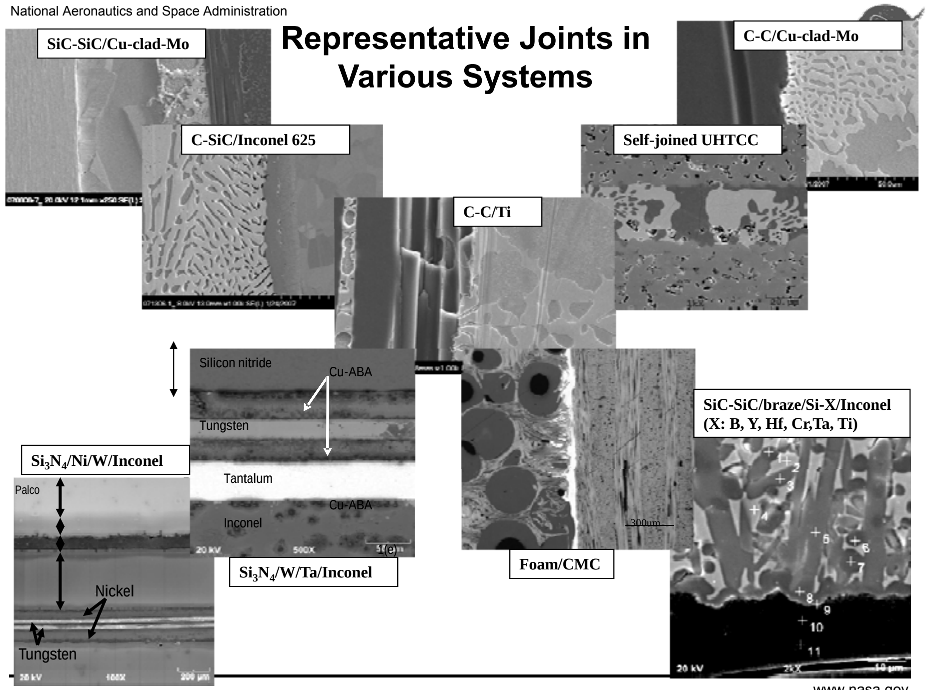
Representative Joints in Various Systems



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Integration Technologies for Improved Efficiency and Low Emissions

- Gas Turbine Components

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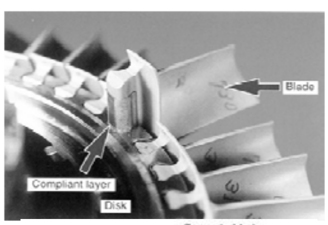
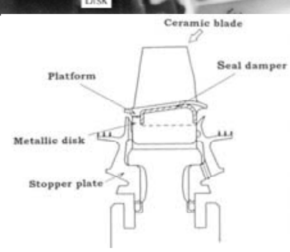
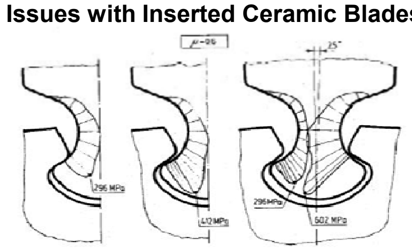
Advanced Silicon Nitride Based Components for Energy and Aerospace Systems





Hybrid Gas Turbine Blade (Ceramic Blade and Metallic Disk) in NEDO's Ceramic Gas Turbine R&D Program, Japan (1988-1999)

Issues with Inserted Ceramic Blades



There are contact stresses at the metal -ceramic interface. Compliant layers (i.e. Ni-alloy+Pt) are used to mitigate the stress and damage. Failures can occur in the compliant layer.

Mark van Roode, Solar Turbines

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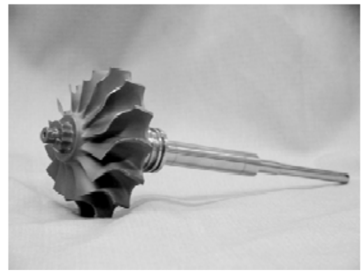
Integration Technologies for Silicon Nitride Ceramics to Metallic Components

INTEGRAL ROTORS

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

Mark van Roode, Solar Turbines

Industry Direction



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

Integration of Silicon Nitride to Metallic Systems

Approach: Use multilayers to reduce the strain energy more effectively than single layers.

Challenge: Multiple interlayers increase the number of interfaces, thus increasing the probability of interfacial defects.

Material	CTE $\times 10^{6}$/K	Yield Strength, MPa
Silicon nitride	3.3	-
Inconel 625	13.1	-
Ta	6.5	170
Mo	4.8	500
Ni	13.4	14-35
Nb	7.1	105
Kovar	5.5-6.2	270
W	4.5	550

Various combinations of Ta, Mo, Ni, Nb, W and Kovar to integrate Silicon nitride to Nickel-Base Superalloys

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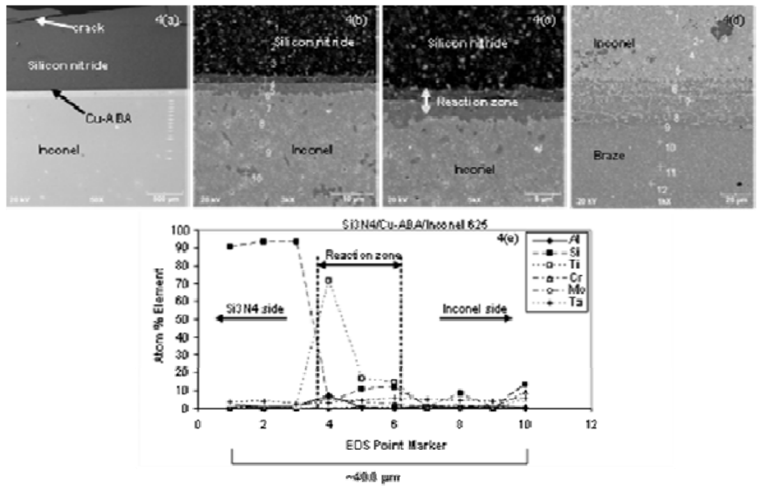
Si_3N_4 (NT 154) bonded to Inconel 625 at 1317 K for 30 min

EDS data in (e) correspond to the point markers in (b)



The reaction zone (point 4) is rich in Ti and Al, but also contains Cu, Ni, Si, and Cr. No discontinuity, cracking or microvoids are noted in the reaction zone

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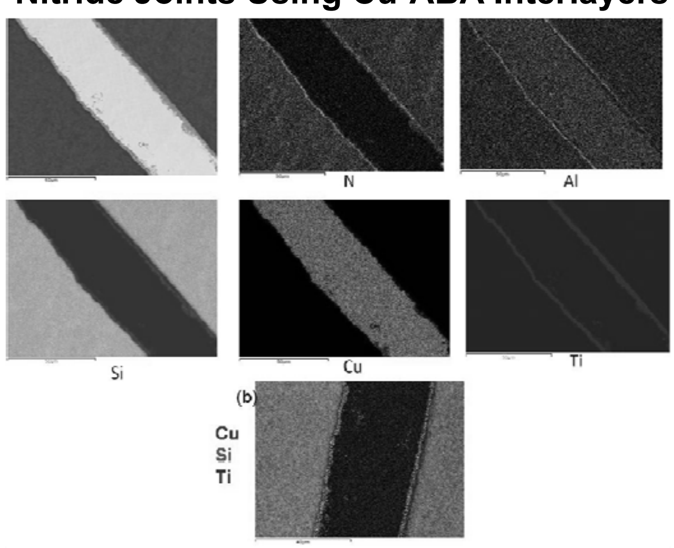
EDS Compositional Maps of Silicon Nitride/Silicon Nitride Joints Using Cu-ABA Interlayers



Titanium Segregation at the Interface

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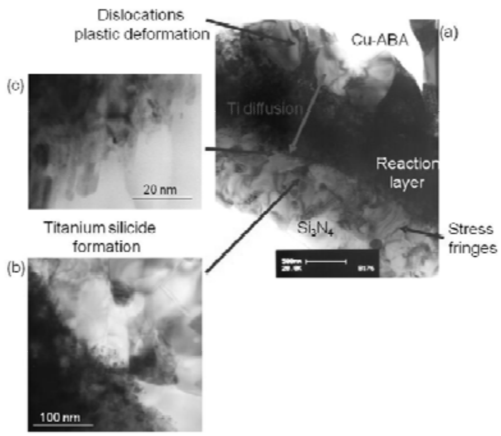
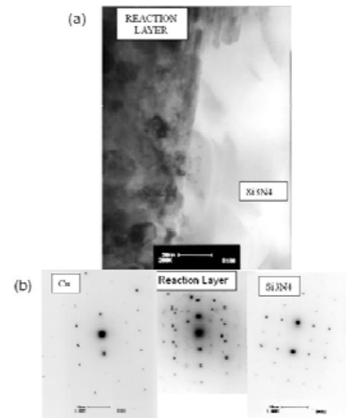
TEM Analysis Silicon Nitride/Silicon Nitride Joints Using Cu-ABA (92.75Cu-3Si-2Al-2.25Ti) Interlayers



(a) Reaction layer at the Si_3N/braze interface and dislocations in braze, (b) titanium silicide formation, and (c) a higher magnification image of region shown in (b).



Details of joint microstructure (fine grains < 50 nm) form the reaction layer. b) Electron diffraction patterns of the Si_3N_4, reaction layer and joint interior (Cu-ABA).

M. Singh, R. Asthana, F.M. Varela, J.M. Fernandez, J. Eur. Ceram. Soc. . 31 (2011) 1309-16

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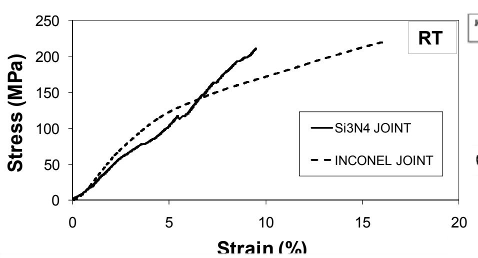
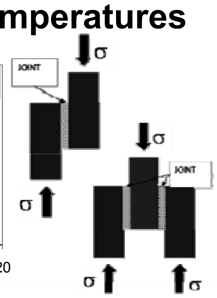
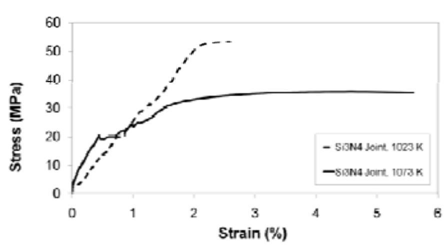
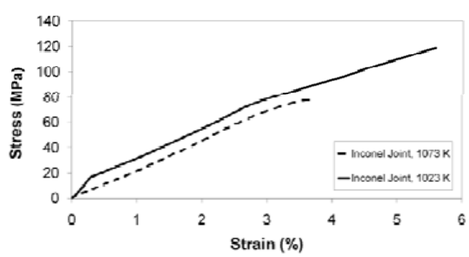
Typical Shear Behavior of Joints at Different Temperatures









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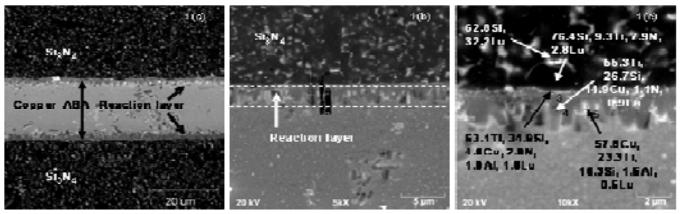
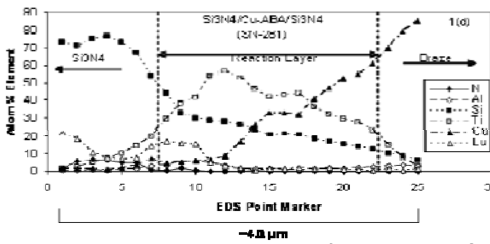
Kyocera SN-281 Si_3N_4 bonded at 1317 K for 5 min using Cu-ABA (92.75Cu-3Si-2Al-2.25Ti) Interlayers

EDS data in (d) correspond to the point markers in (b)





M. Singh, R. Asthana, J. R. Rico, J.M. Fernandez, Ceramic International, 38, 4, (2012) 2793-2802

- **An inhomogeneous reaction layer (2-2.5 µm) comprising of a dark-gray Ti-Si phase, possibly titanium silicide, and a lighter Cu (Si, Ti) phase has developed.**
- **The product phase crystals are oriented perpendicular to the interface (growth direction).**
- **No interfacial excess of Lu; exists in minute quantities in reaction layer and increases toward Si_3N_4.**

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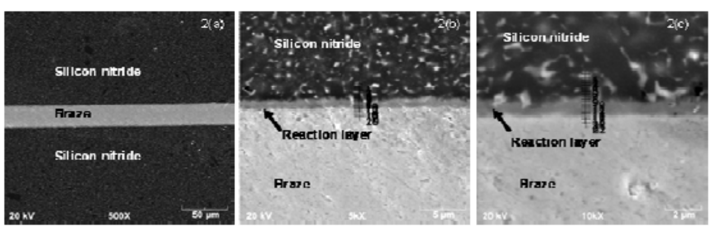
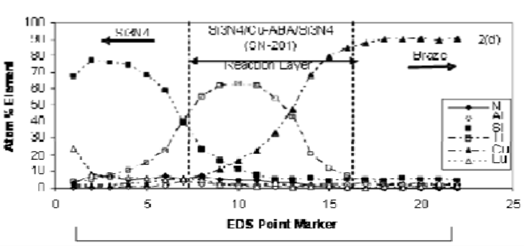
Kyocera SN-281 Si_3N_4 bonded at 1317 K for 30 min

EDS data in (d) correspond to the point markers in (C)





M. Singh, R. Asthana, J. R. Rico, J.M. Fernandez, Ceramic International, 38, 4, (2012) 2793-2802

- **No increase in reaction layer thickness for 30 min. (faster kinetics in the early stages of reaction).**
- **Morphologically a more homogeneous, compact, and featureless reaction layer (possible coalescence of coarsened silicide crystals).**

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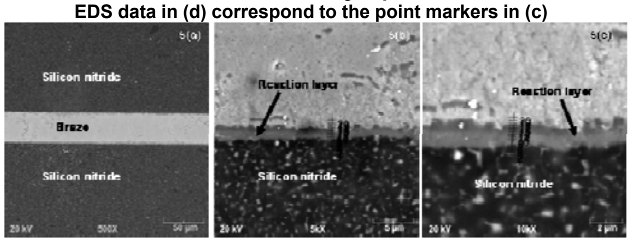
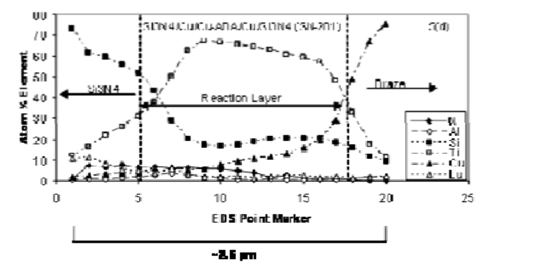
Kyocera SN-281 bonded at 1317K for 30 min.
(5 µm thick Cu foil inserts: Si_3N_4/Cu/Cu-ABA/Cu/Si_3N_4)

EDS data in (d) correspond to the point markers in (c)





- Sound joint with a compact and morphologically homogeneous reaction layer (~1-2 µm thick).
- Ti and Si enrichments at the interface (possible formation of a titanium silicide compound layer).

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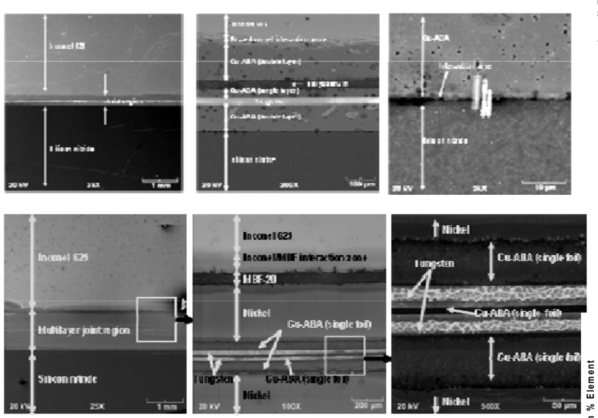
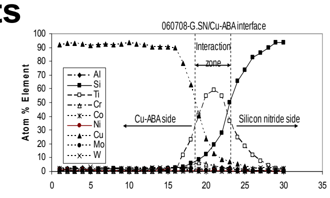
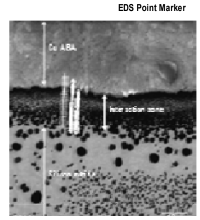
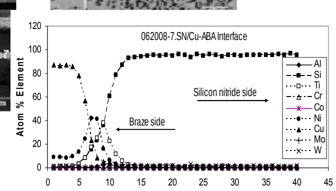
Microstructure of Silicon Nitride/Inconel 625 Multilayer Joints

Si_3N_4/W/Mo/Inconel 625 (Cu-ABA, brazed at 1044C, 30 min.)









Si_3N_4/Ni/W/Ni/Inconel 625
(with Cu-ABA on SN side and MBF on Inconel side)
(brazed at 1044C, 30 min.)

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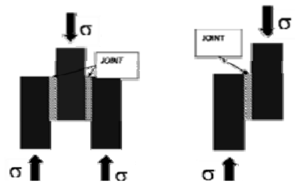
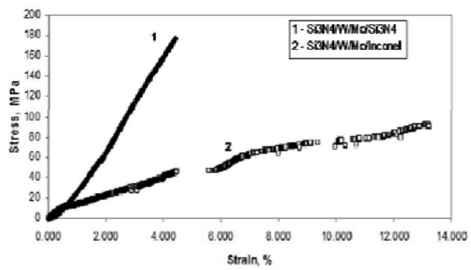
Typical Shear Behavior of Multilayer Joints at Different Temperatures





- Si_3N_4/W/Mo/Si_3N_4 joint strength (142 MPa) is comparable to Si_3N_4/Si_3N_4 strength (141 MPa)
- Si_3N_4/W/Mo/Inconel joint strength (54 MPa) is greater than strength (15 MPa) of Si_3N_4/Inconel

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Integration Technologies for Improved Efficiency and Low Emissions

- **MEMS-LDI Fuel Injector**

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Integration Technologies for MEMS-LDI Fuel Injector

Objective: Develop Technology for a SiC Smart Integrated Multi-Point Lean Direct Injector (SiC SIMP-LDI)

- 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

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

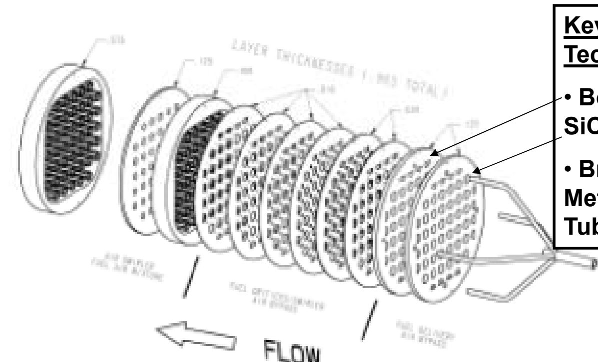
2. Lean Direct Injector (LDI)

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

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

Lean Direct Injector Fabricated by 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**

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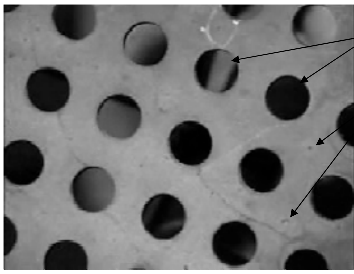
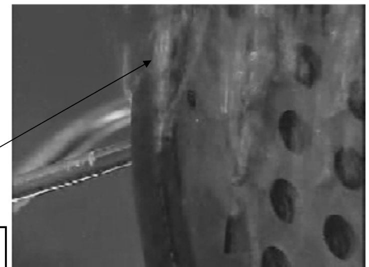
Leak Test of SiC Laminates Joined with Silicate Glass



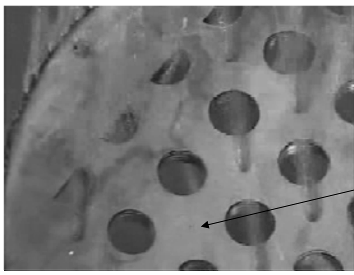


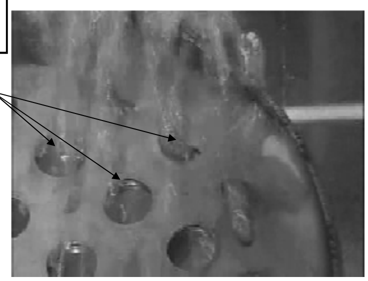
Combustion air channels

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

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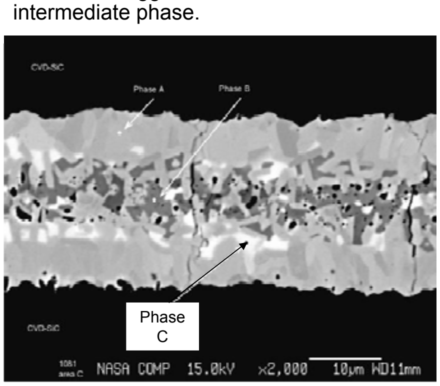
Diffusion Bonding of CVD-SiC Using PVD Ti Interlayer

20 Micron Ti Interlayer

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

Naka et al suggest that this is an intermediate phase.



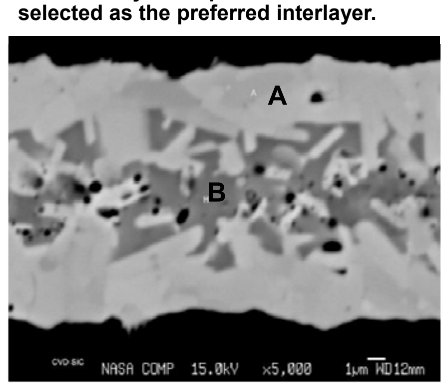
Phases in bond with the 20 μ Ti Interlayer – Atomic Ratios

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 $Ti_5Si_3C_X$ 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

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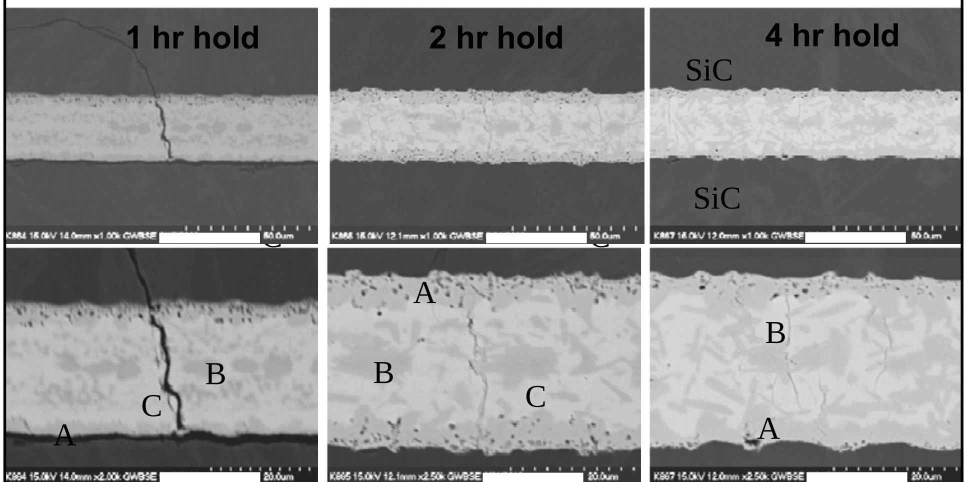
Diffusion Bonding with 20 µm Ti Foil at 1200ºC



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

In atomic percent

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

In atomic percent

	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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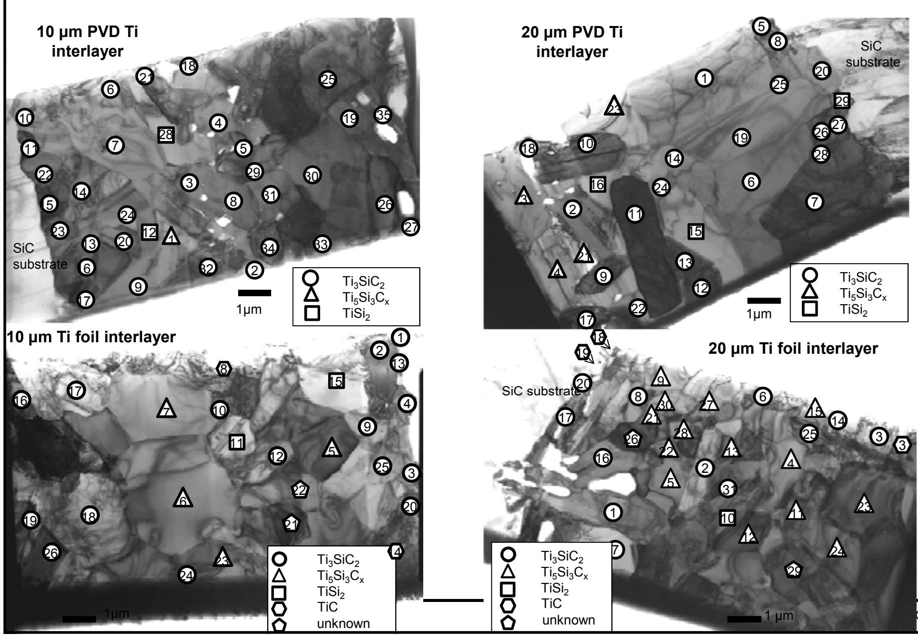
TEM Analysis of Diffusion Bonds Processed for 2 Hr



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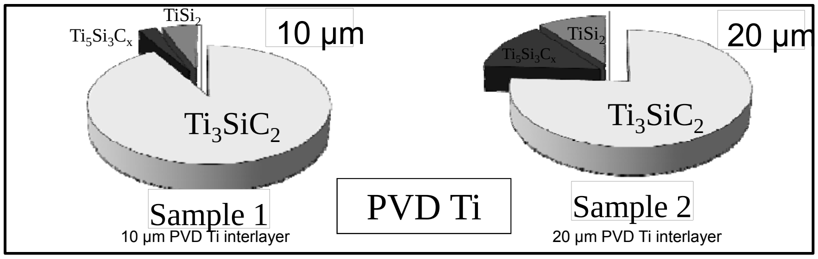
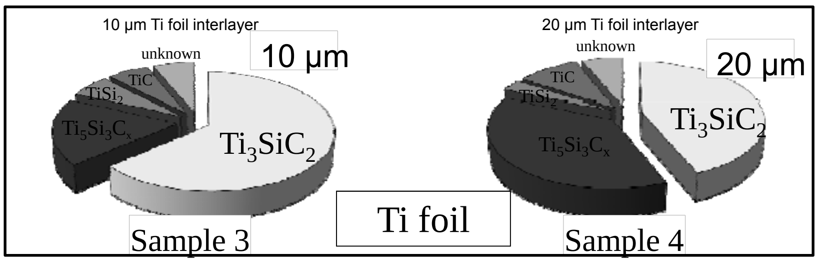
Fractions of Phases in Each Sample





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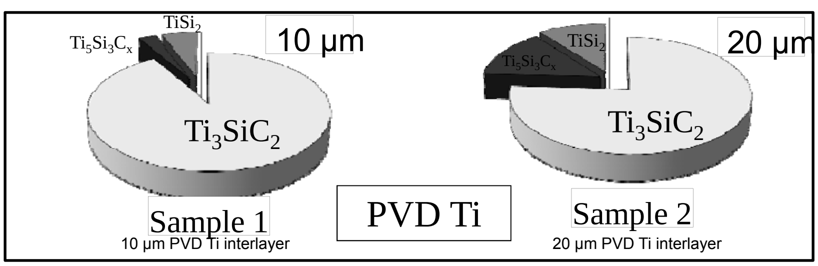
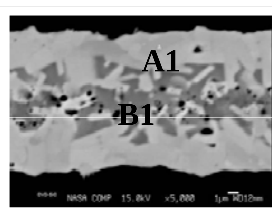
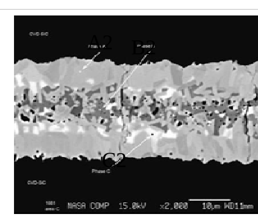
Fractions of Phases in Each Sample







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, the intermediate $Ti_5Si_3C_x$ phase has anisotropic thermal expansion. Therefore, microcracking was observed in the sample 2.

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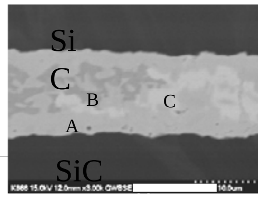
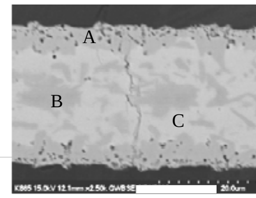
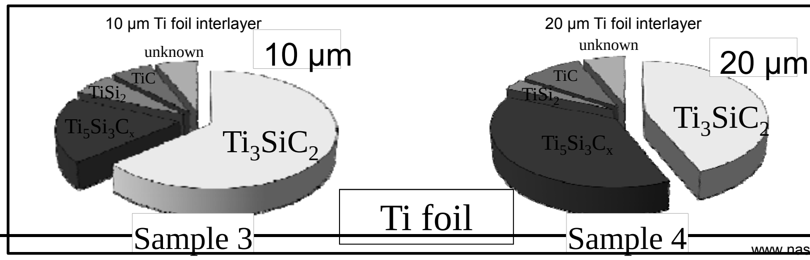
Fractions of Phases in Each Sample

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 from SEM which had lower Si content, this may also cause microcracks in sample 4.







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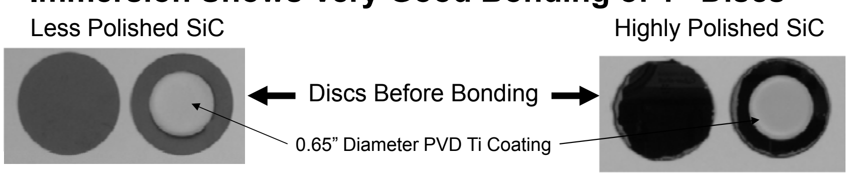
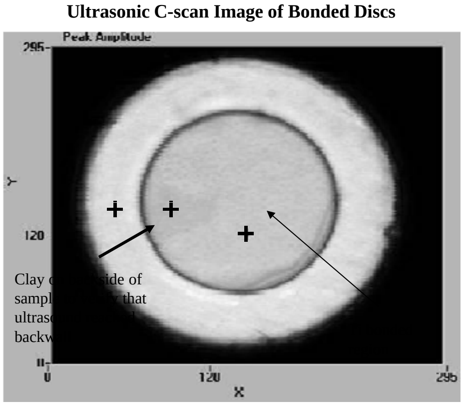
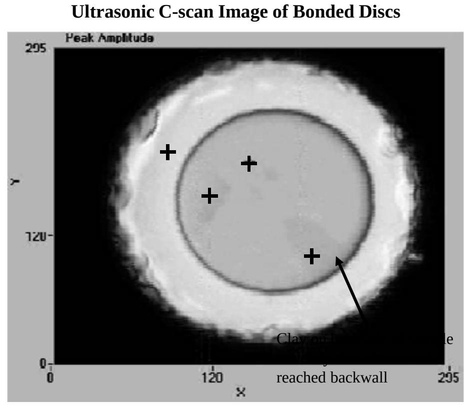
Non-Destructive Evaluation (NDE) Using Ultrasonic Immersion Shows Very Good Bonding of 1" Discs



Ultrasonic C-scan Image of Bonded Discs



Ultrasonic C-scan Image of Bonded Discs



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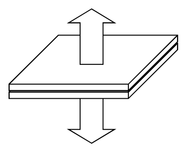
High Strength of Bonds Greatly Exceeds the Application Requirements

1” x 1” Bonded Substrates



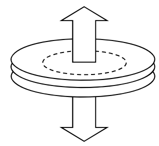
Pull test tensile strengths:

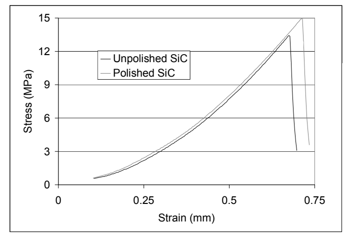
> 23.6 MPa (3.4 ksi)*

> 28.4 MPa (4.1 ksi)*

* failure in the adhesive to the test fixture

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 suggests 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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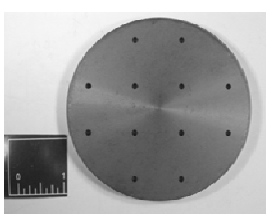
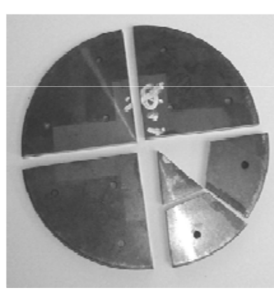
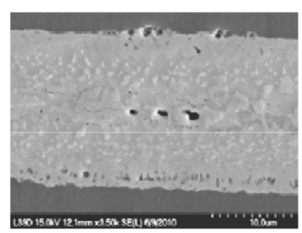
Demonstration of Diffusion Bonding of 4” Diameter CVD SiC Discs

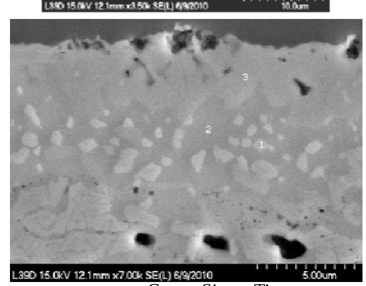


Joined 4” diameter SiC discs.

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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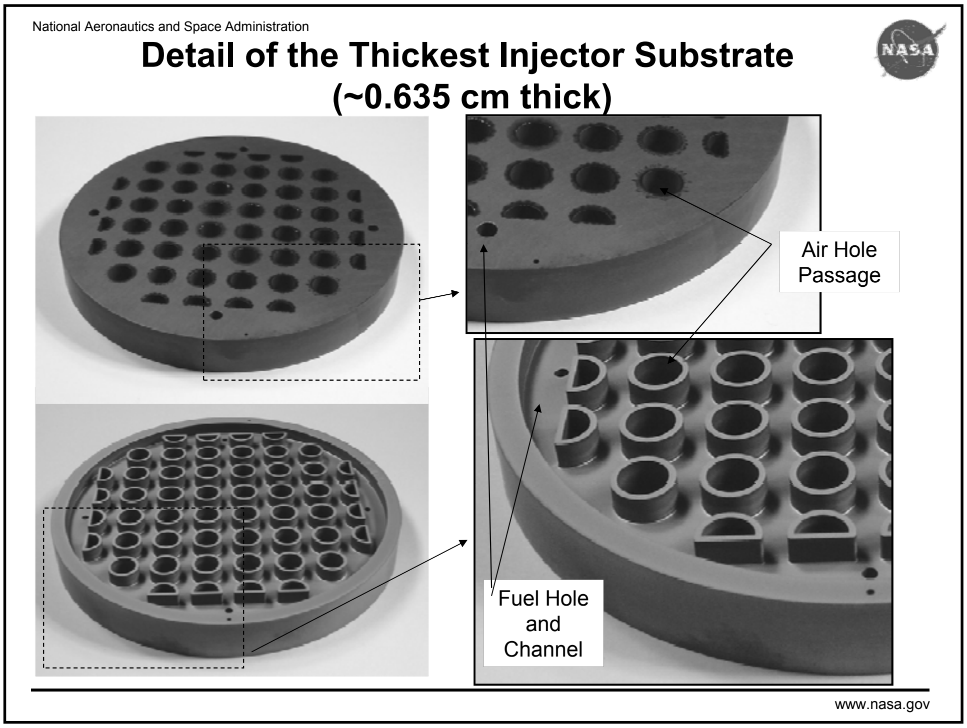
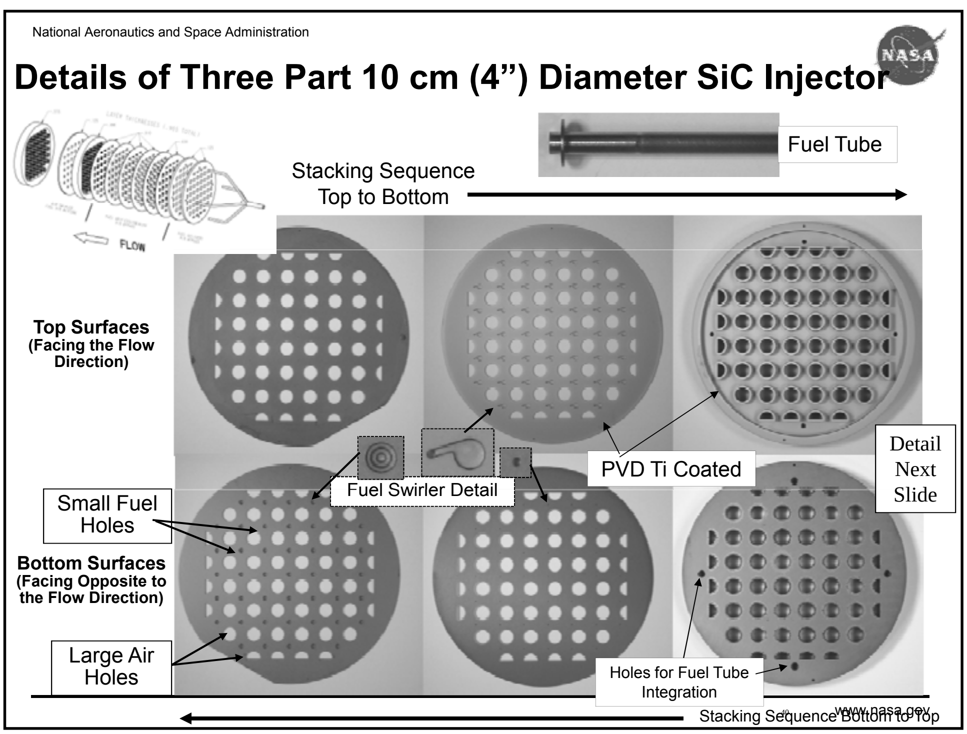

National Aeronautics and Space Administration
Detail of the Thickest Injector Substrate
(~0.635 cm thick)
Air Hole Passage
Fuel Hole and Channel
www.nasa.gov


National Aeronautics and Space Administration
Details of Three Part 10 cm (4”) Diameter SiC Injector
Fuel Tube
Stacking Sequence Top to Bottom
FLOW
Top Surfaces (Facing the Flow Direction)
PVD Ti Coated
Detail Next Slide
Fuel Swirler Detail
Small Fuel Holes
Bottom Surfaces (Facing Opposite to the Flow Direction)
Large Air Holes
Holes for Fuel Tube Integration
Stacking Sequence Bottom to Top
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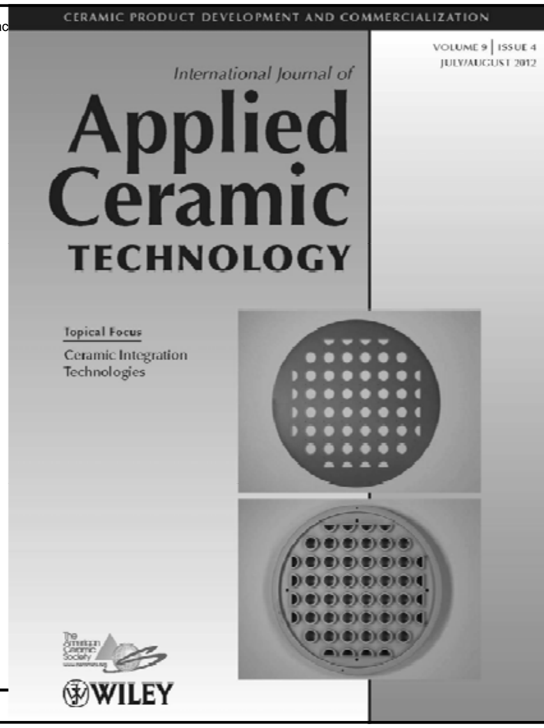

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Ceramic Integration Technologies for Alternative Energy Systems

- Solid Oxide Fuel Cells

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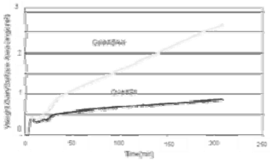
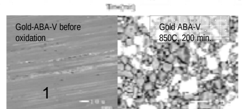
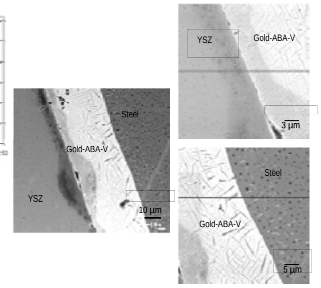
Integration of YSZ/Steel for SOFC Applications

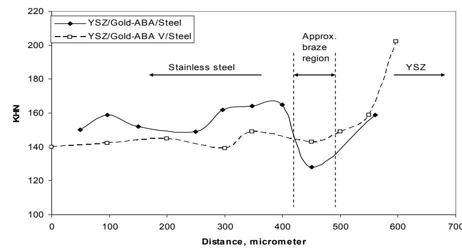
- Gold-ABA and Gold ABA-V exhibit linear oxidation kinetics at 850 C. Gold-ABA-V shows faster oxidation kinetics than Gold-ABA.
- Ti in Gold-ABA and V in Gold-ABA V caused discoloration (darkening) of YSZ.
- The darkening is caused by Ti and V that act as oxygen getters and form oxygen-deficient YSZ. No reaction layers formed at joint.
- The oxygen-deficient YSZ is better wet by gold than stoichiometric YSZ.





EDS of Gold-ABA before and after oxidation





M. Singh, T. Shpargel, R. Asthana, Int. J. Appl. Ceram. Tech. 4(2), 2007, 119-133.

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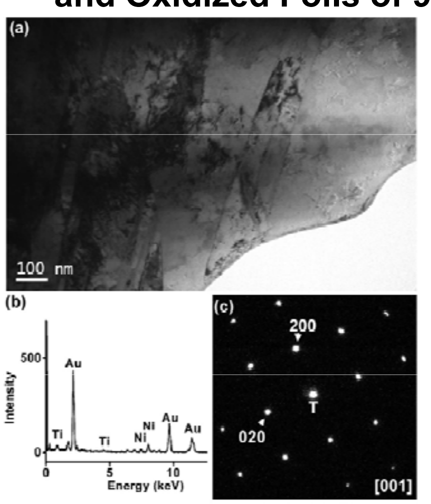
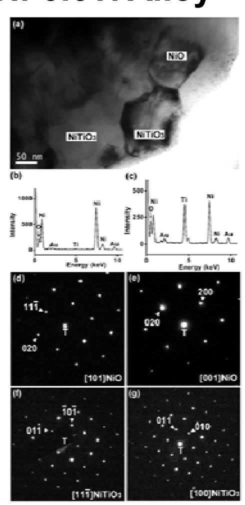
Transmission Electron Microscopy of As-Received and Oxidized Foils of 96.4Au-3Ni-0.6Ti Alloy



As Received



Oxidized

K. Lin, M. Singh, and R. Asthana, Materials Characterization 63 (2012) 105-111.

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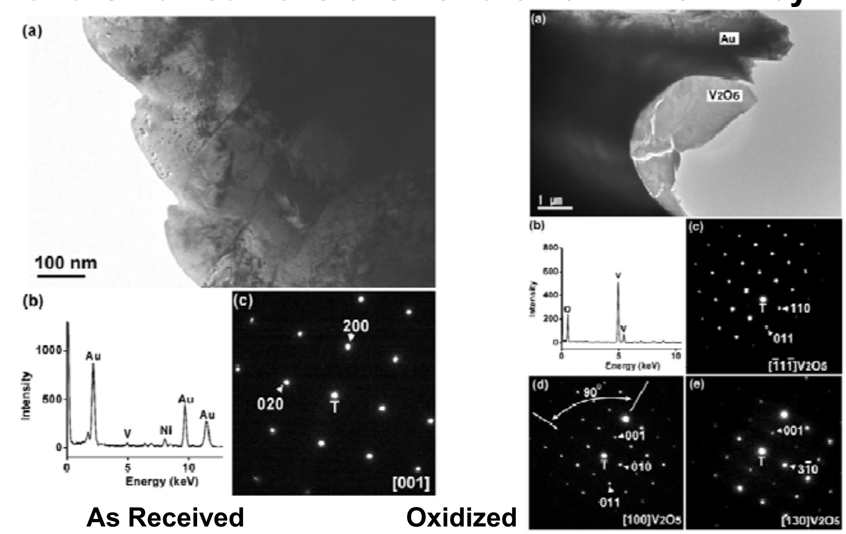
Transmission Electron Microscopy of As-Received and Oxidized Foils of 97.5Au-0.75Ni-1.75V Alloy



As Received Oxidized

K. Lin, M. Singh, and R. Asthana, Materials Characterization 63 (2012) 105-111

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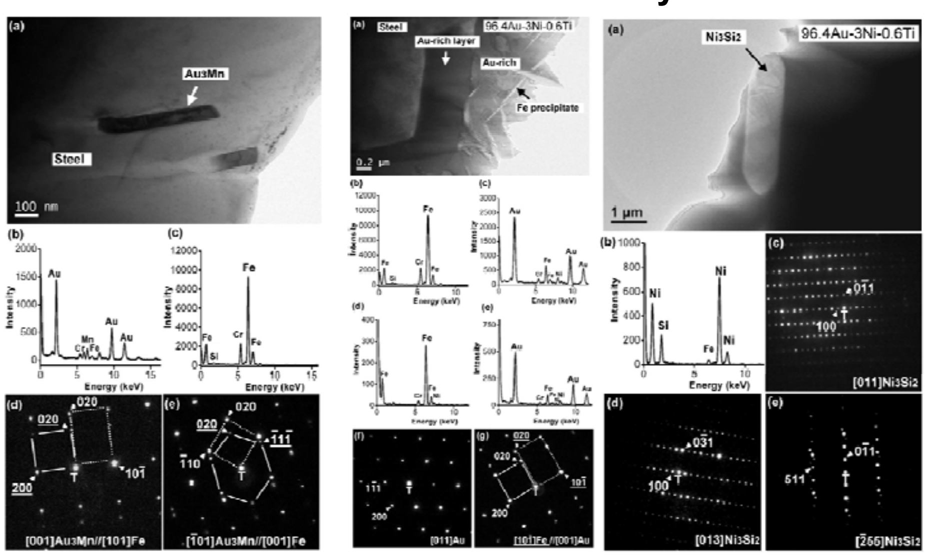
Transmission Electron Microscopy of Interfaces in YSZ/Au-Ni-Ti/Steel System

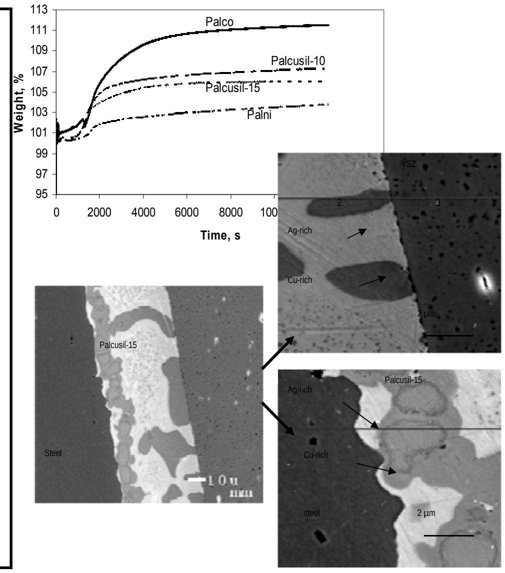


K. Lin, M. Singh, and R. Asthana, Unpublished work (2012)

www.nasa.gov

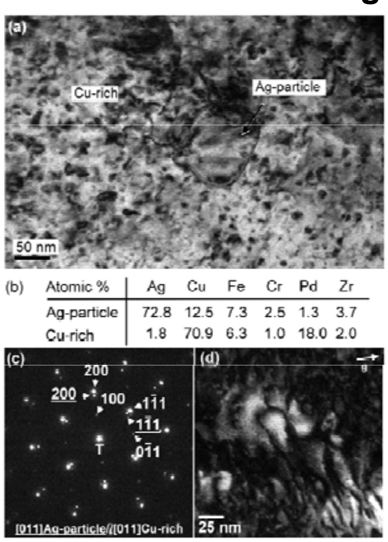
Integration of YSZ/Steel for SOFC Applications

- Pd-base brazes (Palco and Palni) and Ag-base brazes (Palcusil-10 and Palcusil-15) were characterized for oxidation at 750ºC.
- Structural changes accompany oxidation which is fastest for Palco, slowest for Palni, and intermediate for Palcusil-10 and Palcusil-15.
- All brazes were effective in joining yttria stabilized zirconia (YSZ) to stainless steel for solid oxide fuel cell (SOFC).
- Dissolution of YSZ and steel in braze, and braze constituents in YSZ and steel led to diffusion and metallurgically sound joints.
- Knoop hardness (HK) profiles are similar for all brazes, and exhibit a sharp discontinuity at the YSZ/braze interface.



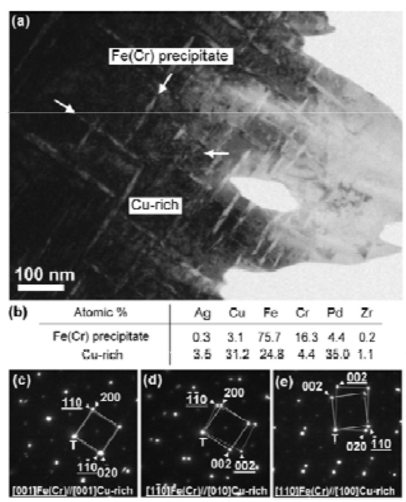
M. Singh, T. Shpargel, R. Asthana, Mater. Sci. Eng. A 485 (2008) 695-702

Transmission Electron Microscopy of Interfaces in YSZ/Ag-Cu-Pd/Steel System



(b)

Atomic %	Ag	Cu	Fe	Cr	Pd	Zr
Ag-particle	72.8	12.5	7.3	2.5	1.3	3.7
Cu-rich	1.8	70.9	6.3	1.0	18.0	2.0



(b)

Atomic %	Ag	Cu	Fe	Cr	Pd	Zr
Fe(Cr) precipitate	0.3	3.1	75.7	16.3	4.4	0.2
Cu-rich	3.5	31.2	24.8	4.4	35.0	1.1

K. Lin, M. Singh, and R. Asthana, Ceramics International, 38 (2012) 1991–1998.

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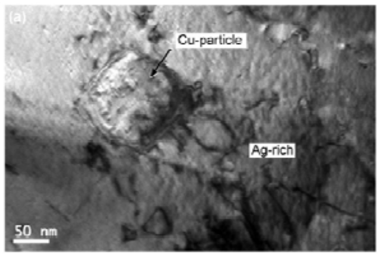
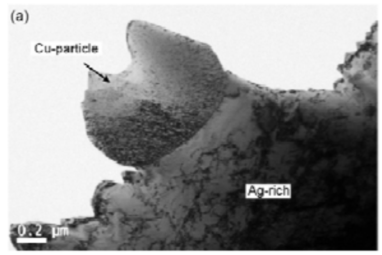
Transmission Electron Microscopy of Interfaces in YSZ/Ag-Cu-Pd/Steel System



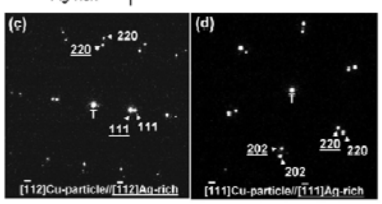


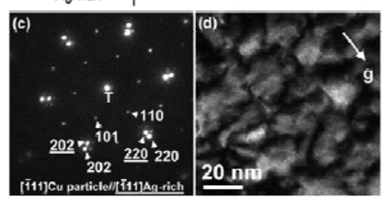
(b)

Atomic %	Ag	Cu	Fe	Cr	Pd	Zr
Cu-particle	3.8	79.8	2.2	1.5	9.8	2.9
Ag-rich	72.6	14.7	6.0	2.1	x	4.6

(b)

Atomic %	Ag	Cu	Fe	Cr	Pd	Zr
Cu-particle	2.8	72.5	3.4	1.3	19.0	1.0
Ag-rich	81.9	7.9	4.1	1.5	2.5	2.1





K. Lin, M. Singh, and R. Asthana, Ceramics International, 38 (2012) 1991–1998.

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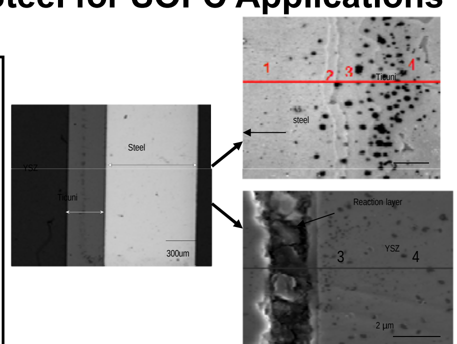
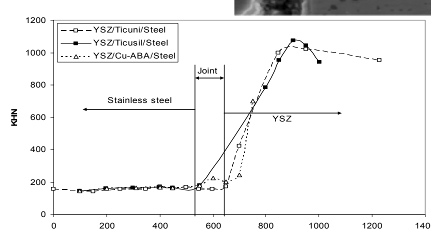
Integration of YSZ/Steel for SOFC Applications

- Active braze alloys, Cu-ABA, Ticuni and Ticusil, were characterized for oxidation at 750-850ºC.
- Oxidation is fastest for Ticusil, slowest for Cu-ABA, and intermediate for Ticuni.
- Brazes were used for joining yttria-stabilized-zirconia (YSZ) to stainless steel for Solid Oxide Fuel Cell (SOFC) applications.
- Interdiffusion, compositional changes, and reaction layer formation led to high-integrity joints.





M. Singh, T. Shpargel, R. Asthana, J. Mater. Sci 43 (2008) 23-32

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Concluding Remarks

- Ceramic integration technologies are critically needed for the successful development and applications of ceramic components in a wide variety of energy applications.
- Major efforts are needed in developing joint design methodologies, understanding the size effects, and thermomechanical performance of integrated systems in service environments.
- Development of life prediction models for integrated components is needed for their successful implementation. In addition, global efforts on standardization of integrated ceramic testing and standard test method development are also required.

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Acknowledgements

- Prof. Rajiv Asthana, University of Wisconsin-Stout
- Mr. Craig E. Smith, Ohio Aerospace Institute
- Mr. Michael H. Halbig, NASA Glenn Research Center
- Prof. J.M. Fernandez, University of Seville, Spain
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- Mr. Ray Babuder, Case Western Reserve University
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