Robust UHTC for Passive Sharp Leading Edge Applications

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Ultrahigh temperature ceramics have performed unreliably due to material flaws and attachment design. These deficiencies are brought to the fore by the low fracture toughness and thermal shock resistance of UHTCs. If these deficiencies are overcome, we are still faced with poor oxidation resistance as a limitation on UHTC applicability to reusable launch vehicles. We have been addressing the deficiencies of UHTCs for the past year via a small task at GRC that is part of the 3rd Gen TPS effort. Our focus is on composite constructions and functional grading to address the mechanical issues and on composition modification to address the oxidation issue. The approaches and progress will be reported.

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a: QSS, Inc.
b: Cleveland State University

Outline
- Introduction
  - UHTC Background
  - Performance Issues for Leading Edges
- Robust UHTC: Objective and Approaches
- Oxidation Resistance Improvement
- Functional Gradient and Composite Materials
- Summary and Conclusions

Ultra High Temperature Ceramics

- Materials consisting of refractory metal borides, refractory metal carbides, silicon carbide, and carbon which have potential use temperatures limited by the melting point of the oxide scale.
  - HfO_2 5073°F(2801°C)
  - ZrO_2 Melting points 4904°F(2707°C)
  - SiO_2 3142°F(1728°C), cristobalite
- ZrO_2 is not a highly protective oxide. Lifetimes based on ZrB_2 recession will be relatively short. 20 volume % SiC additions have been found to give lowest oxidation rates.

  Clougherty, Pober, Kaufman, Trans Met Soc AIME 242, 1077 (1968).

  Tripp, Davis, Graham, Cer Bull 52 [8] 612 (1973).
- Current Fabrication Approaches: hot pressing or chemical vapor infiltration

Potential Applications

- **Inlets, nose tips, leading edges**
- **Satellite on-board propulsion system components**
- **Nozzle throats for divert and attitude control thrusters**
- **High performance short-life turbines**

UHTC Lack Robustness

- UHTC have performed unreliably in ARC tunnels and flight due to material and attachment design flaws — ~~Not so flaws [illegible] [illegible]~~
- Oxidation resistance is unacceptable for reusable TPS leading edge applications
- Current UHTC materials are not reproducible and reliable
  - Partially intrinsic: low fracture toughness and strength
  - Partially extrinsic: process derived flaws
  - Thermal shock an area of concern

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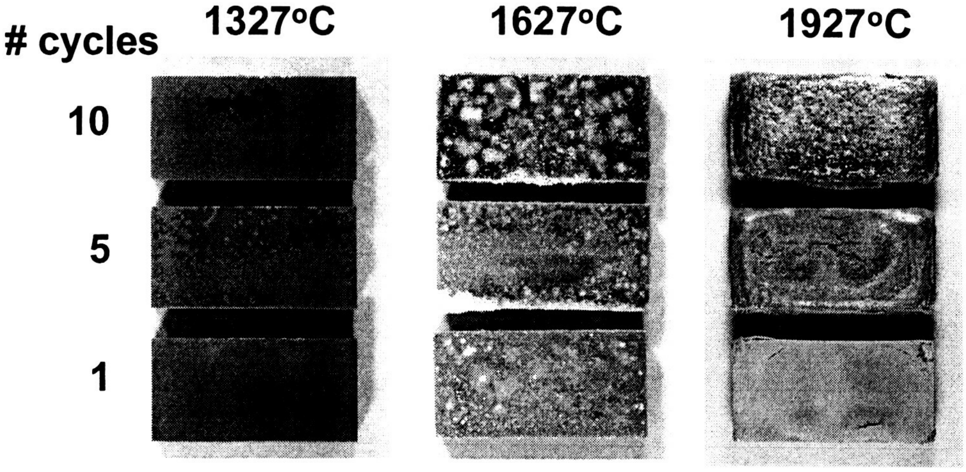
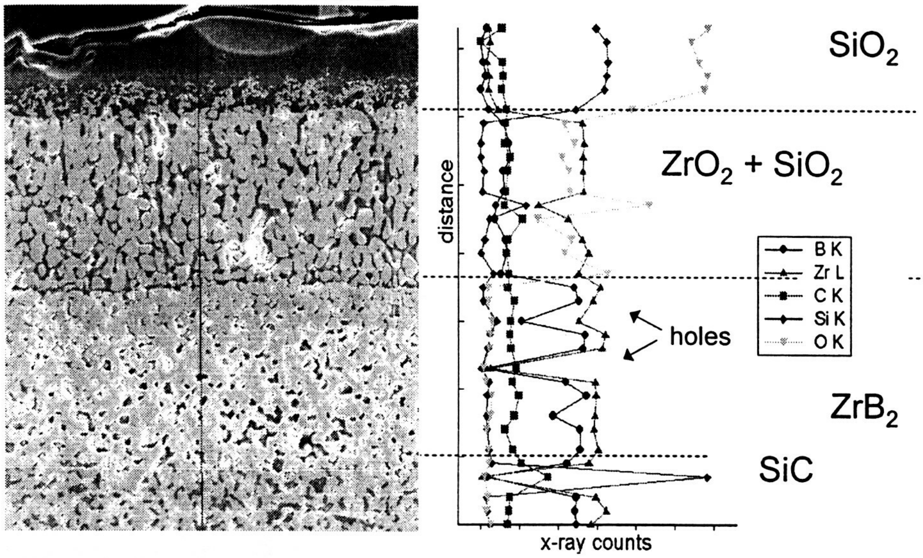
ZrB_2/ 20 $^{v}/_{o}$ SiC UHTC Oxidized in Air

10 Minute Cycles



SEM/EDS: ZrB_2 - SiC

1627°C, Air, Ten 10 min Cycles



Robust UHTC

- Objectives
  - Increase toughness and reliability
  - Improve oxidation resistance
- Approach
  - Alloying to improve oxidation resistance
  - Composites and functionally graded material (FGM)
  - Combine oxidation improvements with composite and FGM approaches

Oxidation Resistance

- Pure, dense ZrO_2 or HfO_2 scale impossible
  - Off stoichiometry
  - Porosity, cracking and spalling due to gaseous oxidation products and CTE mismatch
- Doped ZrO_2 or HfO_2
  - Limit oxygen transport via lattice vacancies
  - Still must deal with porosity, cracking, and spalling
  - Ta_2O_5 is the most practical based on melting point and formation of an intermediate ternary oxide with ZrO_2 and HfO_2
  - Potential sources of tantalum are:
    - TaB_2 or TaB : introduces B
    - TaC : introduces additional C
    - $TaSi_2$ or Ta_5Si_3 : introduces more Si + relatively low melting $TaSi_x$ (~2400 °C)
- Just beginning to think about Re_2ZrO_7 or Re_2HfO_7

Properties of Relevant Compounds

Compound	Density	CTE, 10E6*cm/cmC	MP, C
B4C	2.5	6	2350
BN	2.25	3.8	3000(s)
HfB2	**11.2**	**6.3**	**3250**
HfO2	9.7	6.5	2810
Nb2O5	4.6	1	1460
NbB2	7.2	8	>2900(d)
NbC	7.8	7.2	3500
SiC	**3.2**	**5.1**	**2500(d**
SiO2	2.7	3	1710
Ta2O5	8.7	2.4	1880
Ta5Si3	**13.1**	**6.7**	**2460**
TaB	**14.3**		**3090**
TaB2	**12.6**	**8.2**	**3000(d**
TaC	**14.5**	**7.1**	**3880**
TaSi2	**8.8**	**8.9**	**2200**
TiB2	4.5	5.2	2980
TiC	4.9	8	3250
TiO2	4.3	9.5	1920
ZrB2	**6.1**	**5.9**	**3040**
ZrO2	5.8	8.1	2690

Additives selected to replace diboride (Ta5Si3, TaB, TaB2, TaC, TaSi2)

History for Ta Additions

- Wuchina et al (NSWC) looked at TaB and TaC additions to pure HfC, HfN and HfB_2
  - Limited reporting of results
  - Denser oxide scale at 1500°C and 25% additive reported for HfB_2
- Talmy et al (NSWC) looked at up to 20% TaB_2 additions to ZrB_2 + 20%SiC
  - Only looked at furnace oxidation temperatures up to 1400°C
  - Significant improvements in oxidation resistance attributed to phase separation in the glass
  - Other mechanisms are possible

Mechanisms for ZrO_2 Protective Scale Enhancement via Ta_2O_5 Additions

- Ta_2O_5 as a glass modifier
  - Talmy et al
- Ta^{+5} as a dopant in ZrO_2 lattice
  - Fill O^{-2} vacancies
  - Decrease O^{-2} transport
- Ta_2O_5 as a major oxide scale constituent
  - Phase V
  - Low melting point

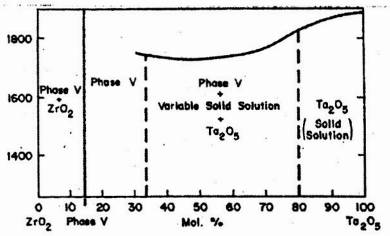
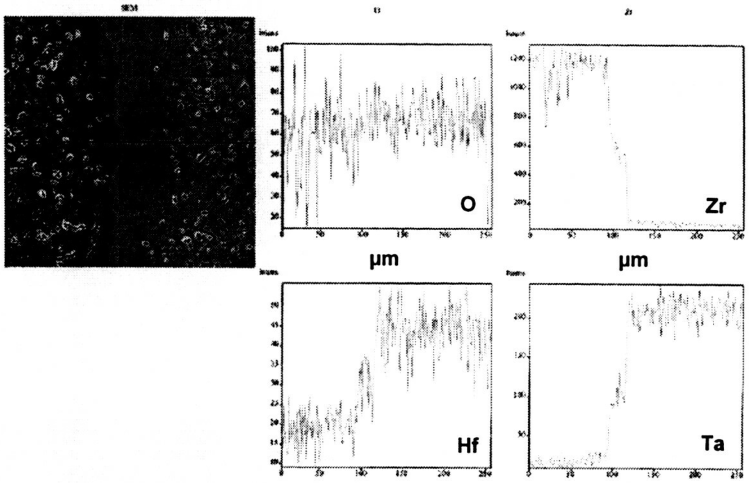


FIG. 374.—System Ta_2O_5–ZrO_2.

B. W. King, John Schultz, E. A. Durbin, and W. H. Duckworth, U. S. Atomic Energy Comm., BMI-1106, 15 (1956).

ZrO_2-Ta_2O_5 Diffusion Couple

1450°C, 1 h



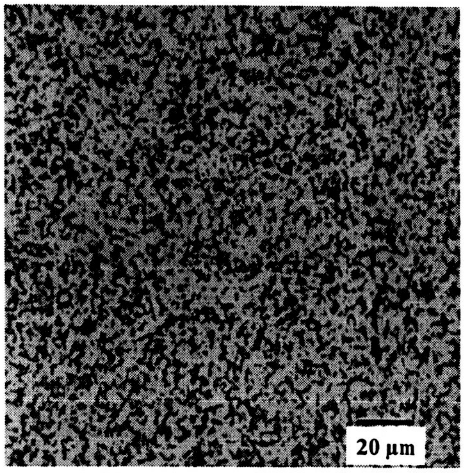
Intermediate phase formation and Ta solubility are evident

Processing

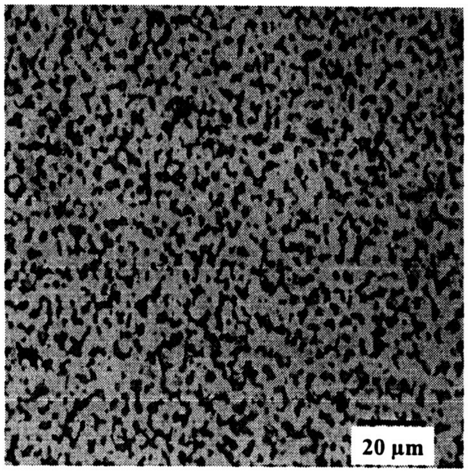
- **Baseline ZrB_2**
  - Plates hot pressed to > 95% average density at 2000°C, 10ksi, up to 2h, vac
- **Ball milling of mixed powders identified as preferred powder processing procedure**
  - One composition processed to 100% density at 1900°C, 10ksi, 2h, vac
  - Segregation observed in some early batches suggested addition of spray drying to the procedure, especially for HfB_2 based system
  - Later batches prepared by the milling of mixed powders appeared to be satisfactory
- **Compositions prepared from mixture of ball milled individual powders were unacceptable**
  - Non-homogeneous with large agglomerates and cracks
  - Further milling as mixed powders solved the problem in the one case tried.

ZrB$_2$ – 20 v/o SiC
Effect of Sintering Time

1h, 2000°C, 10 ksi

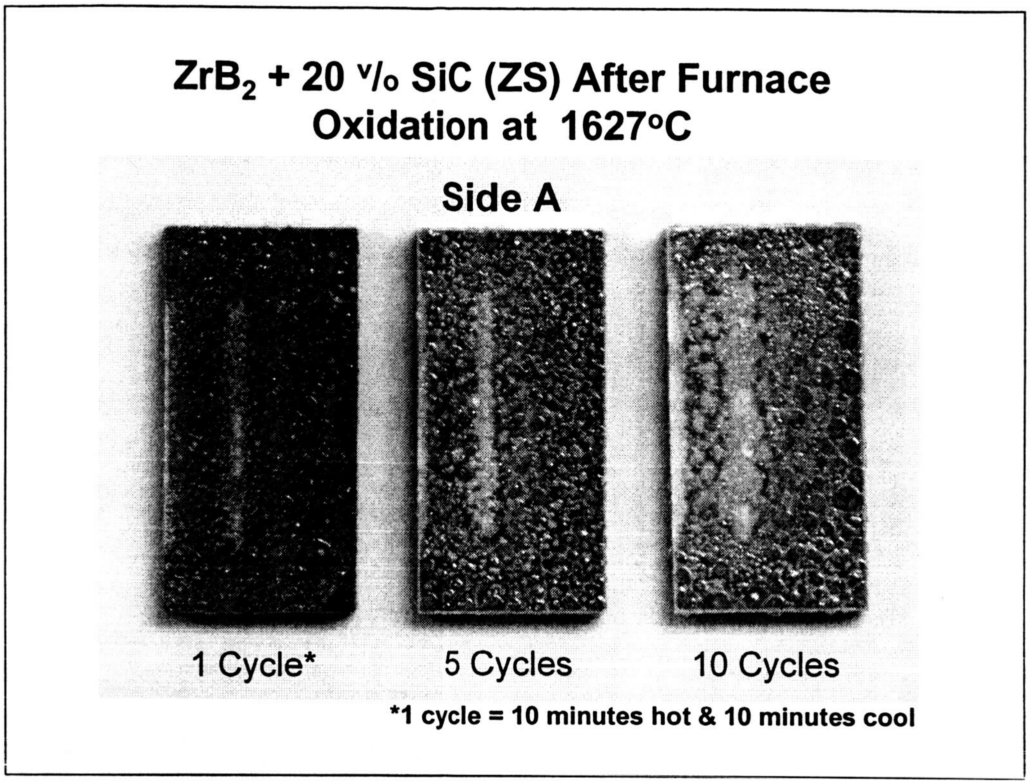


2h, 2000°C, 10 ksi



Flexural Strength Results for ZrB$_2$-based Compositions

Run	Composition	Powder Process	Processing	UTS, Mpa	% strain	E, GPA
877-1	ZrB2-20v/oSiC	batch 1, mix milled	2000C, 10ksi, 1h, vac	377.4	0.131	289.5
877-2				722.6	0.171	401.5
877-3				749.9	0.181	394.5
877-avg				616.6	0.161	361.8
882-1	ZrB2-20v/oSiC	batch 1, mix milled	2000C, 10ksi, 2h, vac	381.4	0.086	416.8
882-2				588.8	0.106	531.2
882-3				486.9	0.098	472.3
882-avg				485.7	0.097	473.4
897-1	ZrB2-20v/oSiC	batch 2, milled mix	2000C, 10ksi, 2h, vac	576.3	0.129	434.3
897-2				560.2	0.133	378.9
897-3				505.2	0.120	395.6
897-avg				547.2	0.127	402.9
878-1	ZrB2-20v/oSiC-20v/o mixed additives	milled mix	1900C, 10ksi, 2h, vac	838.7	0.178	448.0
878-2				834.3	0.173	475.7
878-3				729.3	0.144	471.1
878-avg				800.8	0.165	464.9
889-1	ZrB2-20v/oSiC-20v/oTaSi2	remill mix milled	1600C, 10ksi, 2h, vac	549.6	0.133	399.0
889-2				786.2	0.185	414.7
889-3				827.0	0.192	424.0
889-avg				720.9	0.170	412.6
908-1	HfB2-20v/oSiC	milled mix	2000C, 10ksi, 2h, vac	563.8	0.116	473.0
908-2				375.4	0.080	459.6
908-3				518.5	0.110	465.4
908-avg				485.9	0.102	466.0
911-1	HfB2-20v/oSiC	milled mix	2000C, 10ksi, 2h, vac	549.5	0.118	447.9
911-2				556.0	0.117	453.4
911-3				534.6	0.117	423.4
911-avg				546.7	0.117	441.6

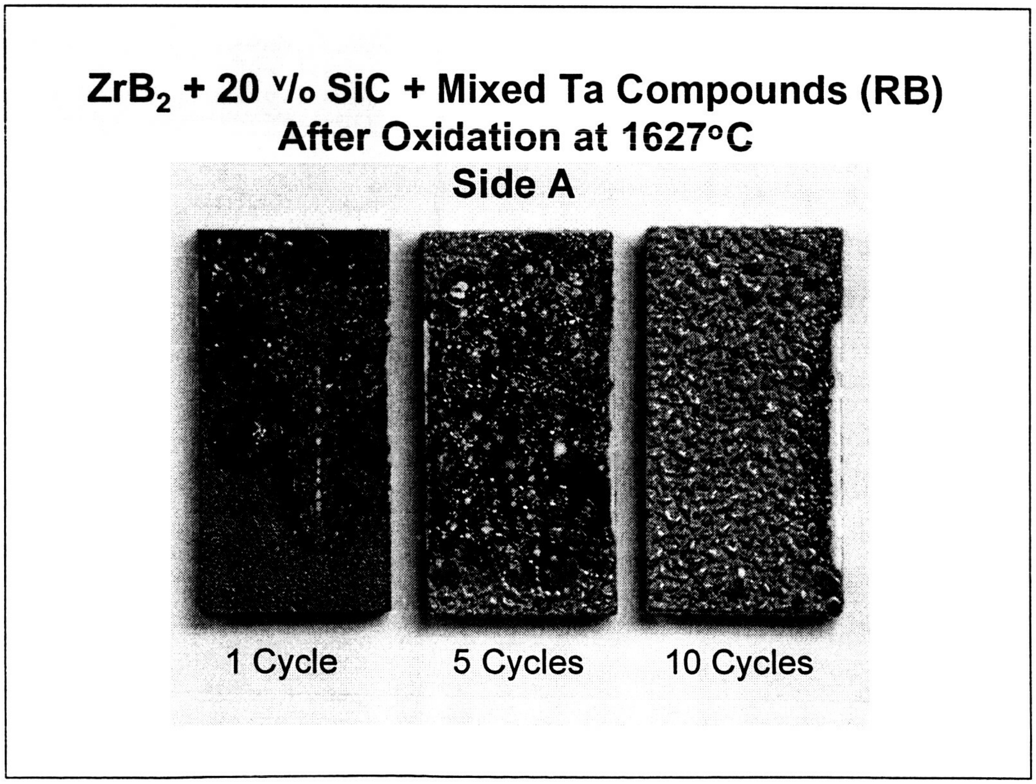


ZrB_2 + 20 v/o SiC (ZS) After Furnace Oxidation at 1627°C

Side A

1 Cycle* 5 Cycles 10 Cycles

*1 cycle = 10 minutes hot & 10 minutes cool

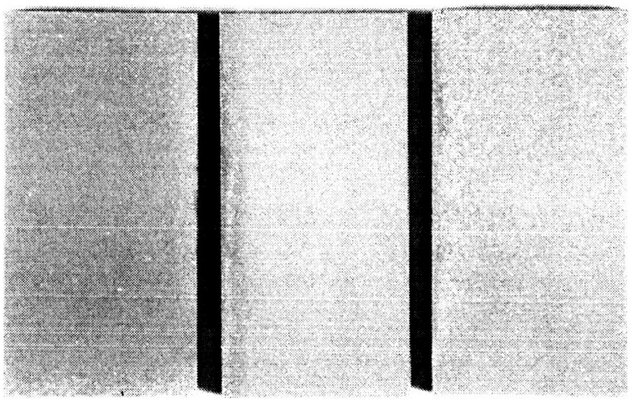


ZrB_2 + 20 v/o SiC + Mixed Ta Compounds (RB) After Oxidation at 1627°C

Side A

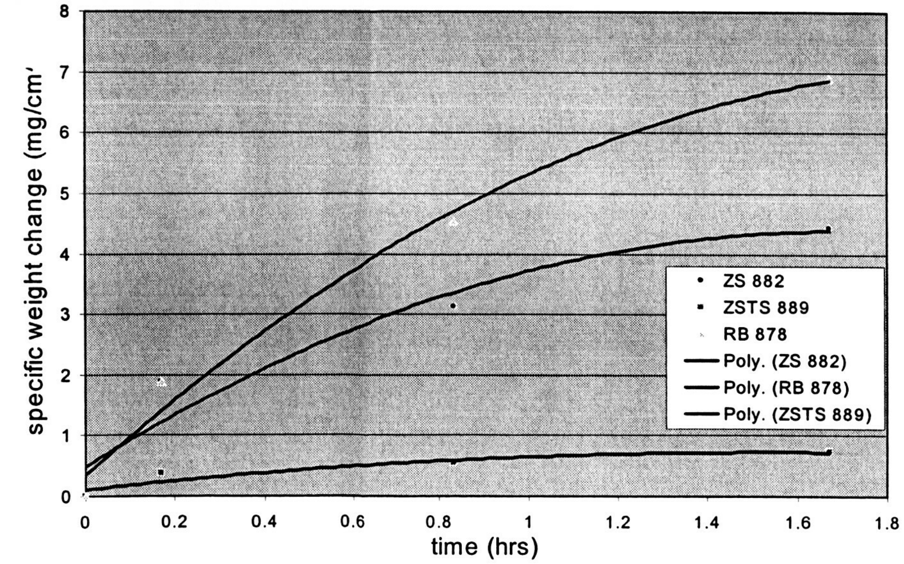
1 Cycle 5 Cycles 10 Cycles

ZrB_2 + 20 v/o SiC + 20 v/o $TaSi_2$ (ZSTS)
Oxidized at 1627°C
Side A



1 Cycle 5 Cycles 10 Cycles

UHTC Oxidation at 1627°C in Zirconia Furnace



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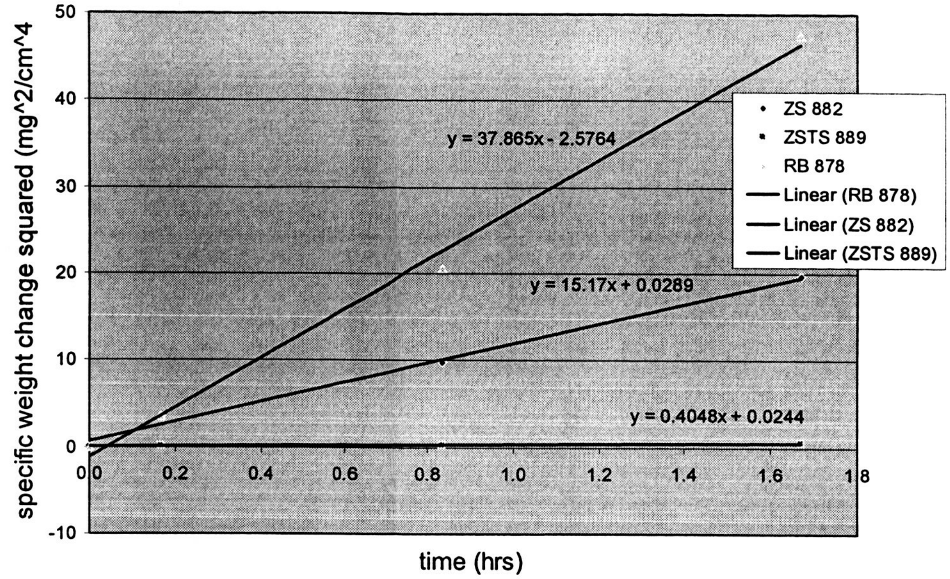
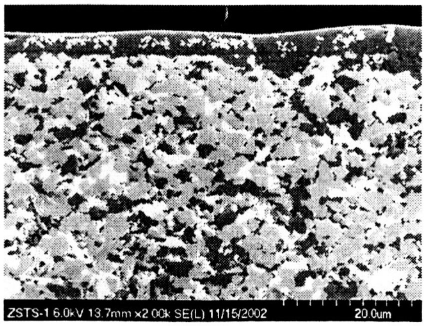
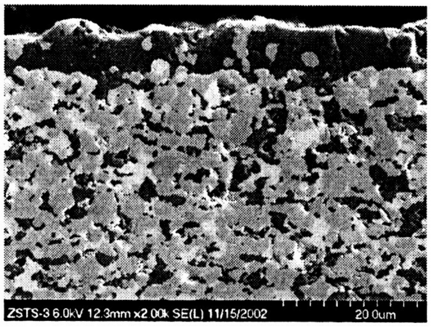


20 %TaSi$_2$ Modified ZrB$_2$ + 20% SiC After Furnace Oxidation at 1627°C in Air



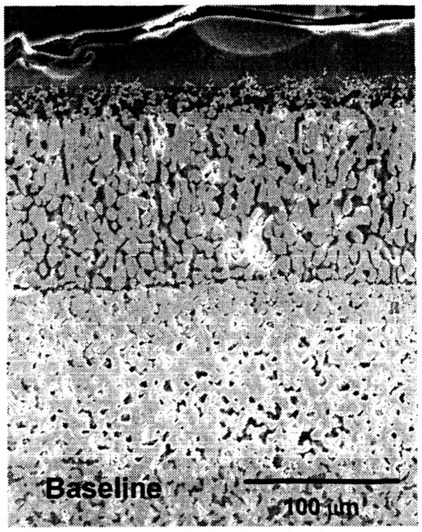
1x10 min cycle, at 1000x

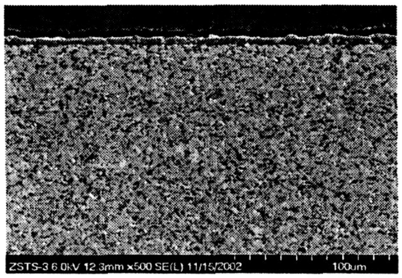


10x10min cycles, at 2000x

➢10X Improvement in Oxidation Resistance of $TaSi_2$ Modified 20 v/o SiC

10x10 minute cycles, air, 1627°C

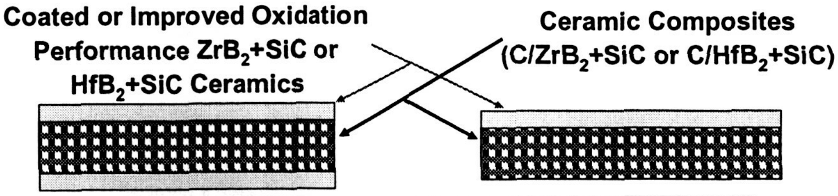




$TaSi_2$ Modified ZrB_2-20 V/o SiC

>10 X improvement in oxidation resistance relative to industry baseline based on weight change and oxidation damage

UHTCMC Hybrid System



Coated or Improved Oxidation Performance ZrB_2+SiC or HfB_2+SiC Ceramics

Ceramic Composites (C/ZrB_2+SiC or C/HfB_2+SiC)

Advantages of UHTCMC hybrids

- Thickness and composition of UHTC layers can be changed to suit the application requirements
- Improved toughness
- Improved environmental durability
  - Tailored surface coatings
  - Crack healing matrices
  - Control of residual stresses
- Smooth composite surfaces
  - Low drag
  - Machinable without fiber damage
  - Easier to bond and attach sensors and other devices
  - Critical to good attachments/seals

UHTCMC Hybrid System Processing

Carbon Fiber Preforms Made into Net Shapes

Infiltration of RB_2+SiC+C Mixtures

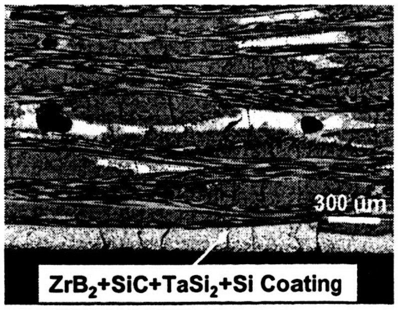
Curing and Pyrolysis

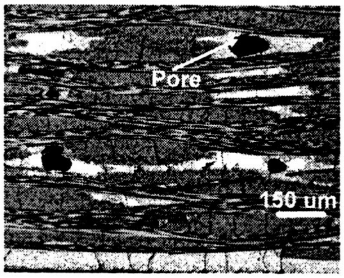
Surface Treatment (RB_2+SiC+C)

Melt Infiltration (R or R-Si alloys)

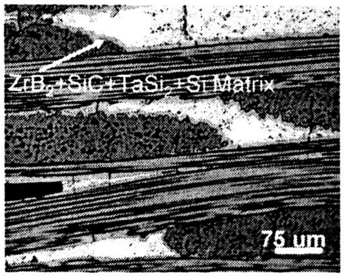
Ultra High Temperature Ceramic Matrix Composites (UHTCMCs)

Microstructure of UHTCMC Hybrid System

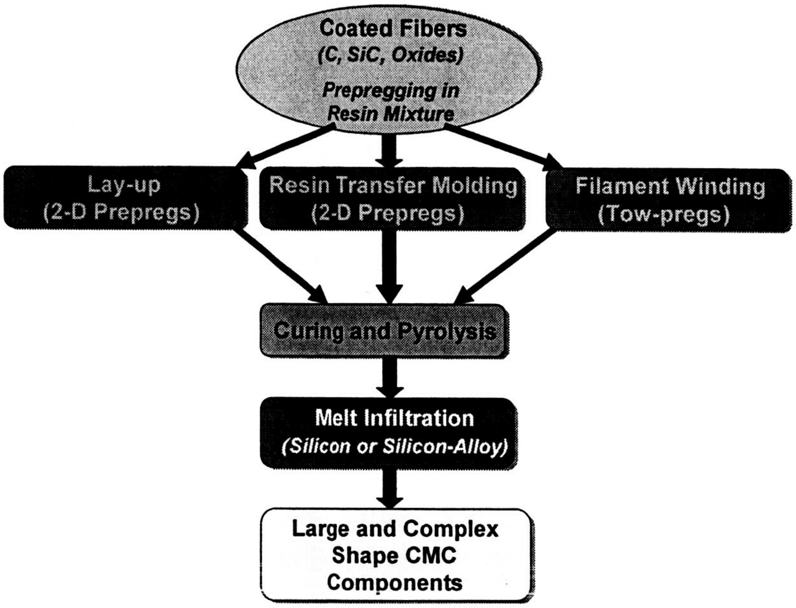




- ZrB_2 + SiC slurry infiltrant in C/SiC preform
- Similar microstructures in silicon and Si-Ta alloy melt infiltrated systems
- Cracked coatings and matrix due to thermal expansion mismatch
- Process optimization and property characterization underway



CMCs by Prepregging and Melt Infiltration (PREMI)



Coated Fibers
(C, SiC, Oxides)
Prepregging in Resin Mixture

Lay-up (2-D Prepregs)

Resin Transfer Molding (2-D Prepregs)

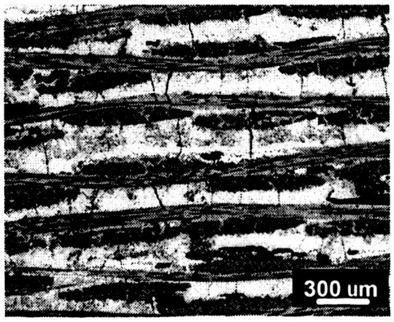
Filament Winding (Tow-pregs)

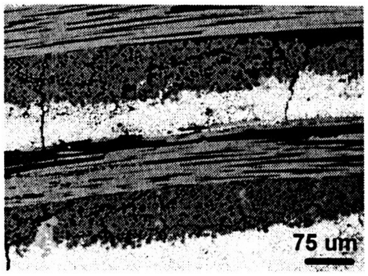
Curing and Pyrolysis

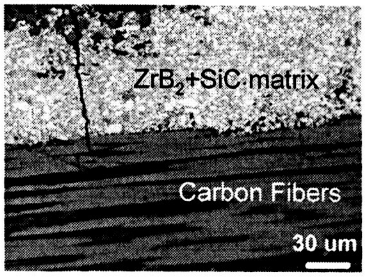
Melt Infiltration
(Silicon or Silicon-Alloy)

Large and Complex Shape CMC Components

Microstructure of UHTCMCs by Prepregging and Melt Infiltration (PREMI)







- 12 layers of T-300, 5-HS cloth prepregged with ZrB_2+SiC or ZrB_2+$TaSi_2$ mixture
- Warm pressed, pyrolyzed, and melt
- Infiltrated with Si or Si-Ta alloy

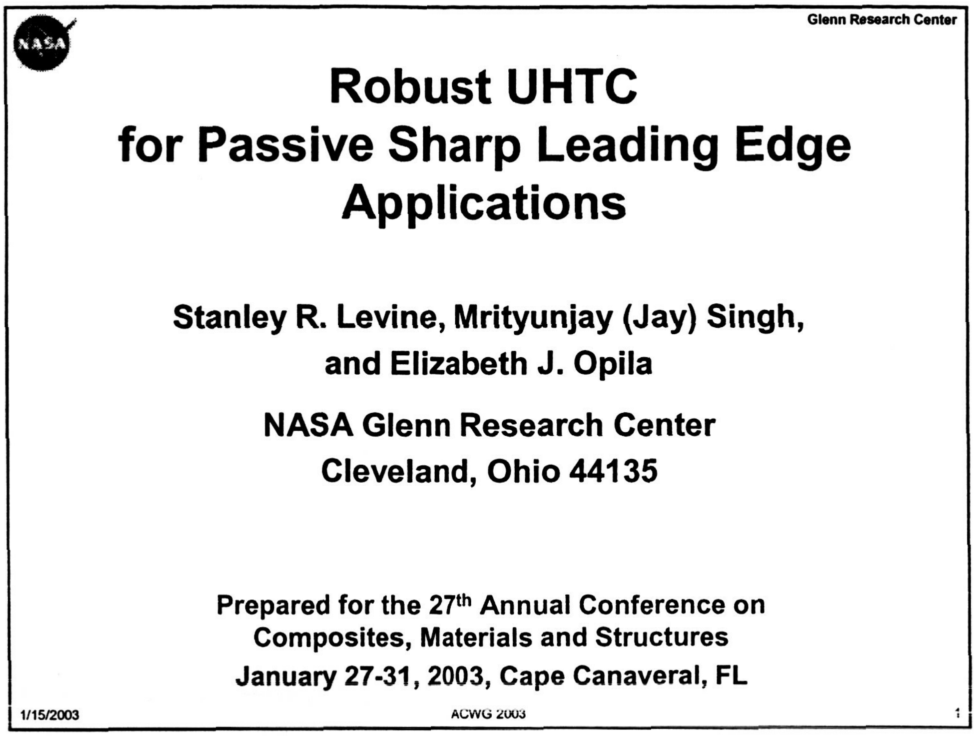
Cracking –
Maybe the
powder did
not w Si.

Summary

- Current UHTCs lack robustness (low fracture toughness, reliability and oxidation resistance)
- Alloy and functionally graded materials approaches have been identified to improve oxidation resistance
  - Ta addition appears to be promising with oxidation rate reduced by > 10X @ 1627°C
  - FGM approaches in processing
- Several composites approaches have been identified to increase mechanical robustness
  - Infiltration processing and prepregging have produced materials with interesting microstructures
  - Further characterization of microstructures, and mechanical property and environmental durability is needed to guide the next processing cycle

Conclusions

- Alloying can yield a large improvement in oxidation resistance in a static environment
  - Need to demonstrate in a flowing environment representative of the application
- Several approaches to fabrication of UHTC composites appear to be promising in so far as microstructural appearance
  - Mechanical property and environmental durability evaluations will guide future directions



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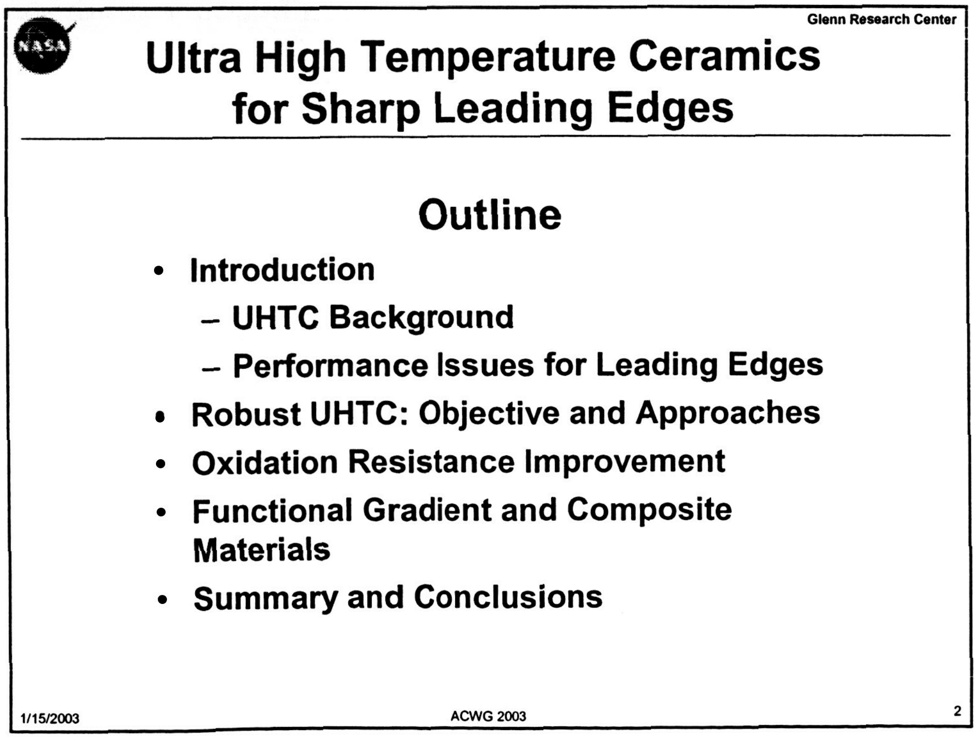

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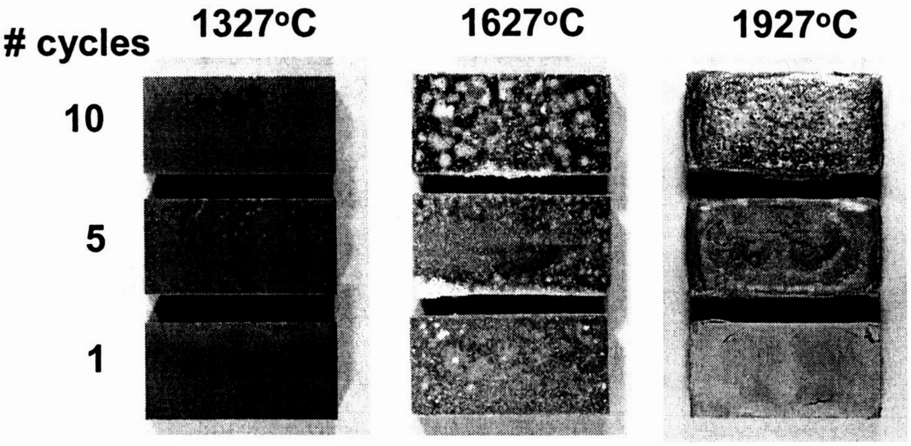

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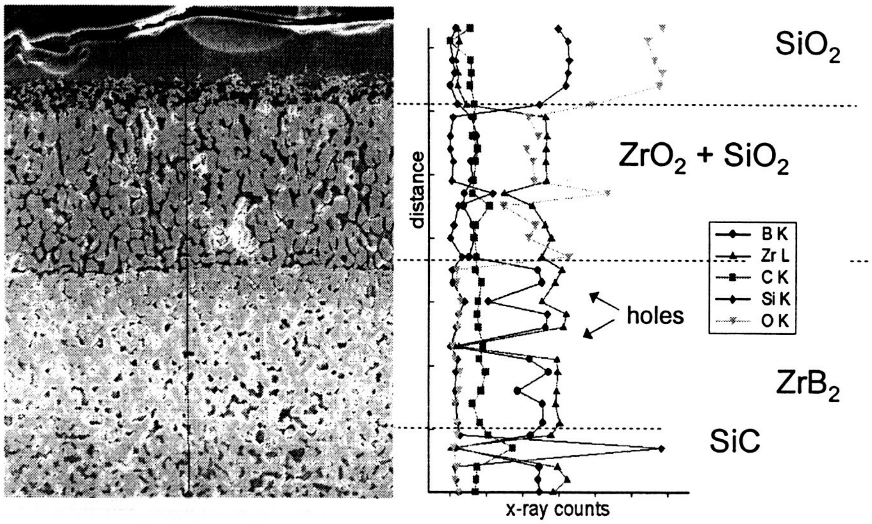

Robust UHTC

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  - **Improve oxidation resistance**
- **Approach**
  - **Alloying to improve oxidation resistance**
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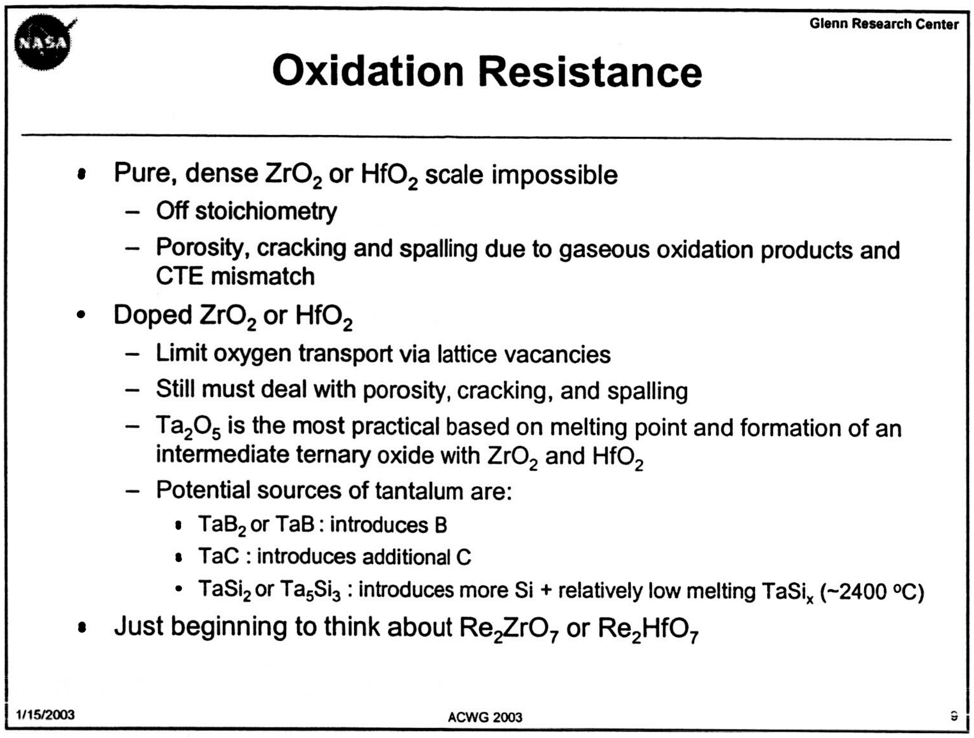
  - **Combine oxidation improvements with composite and FGM approaches**



Oxidation Resistance

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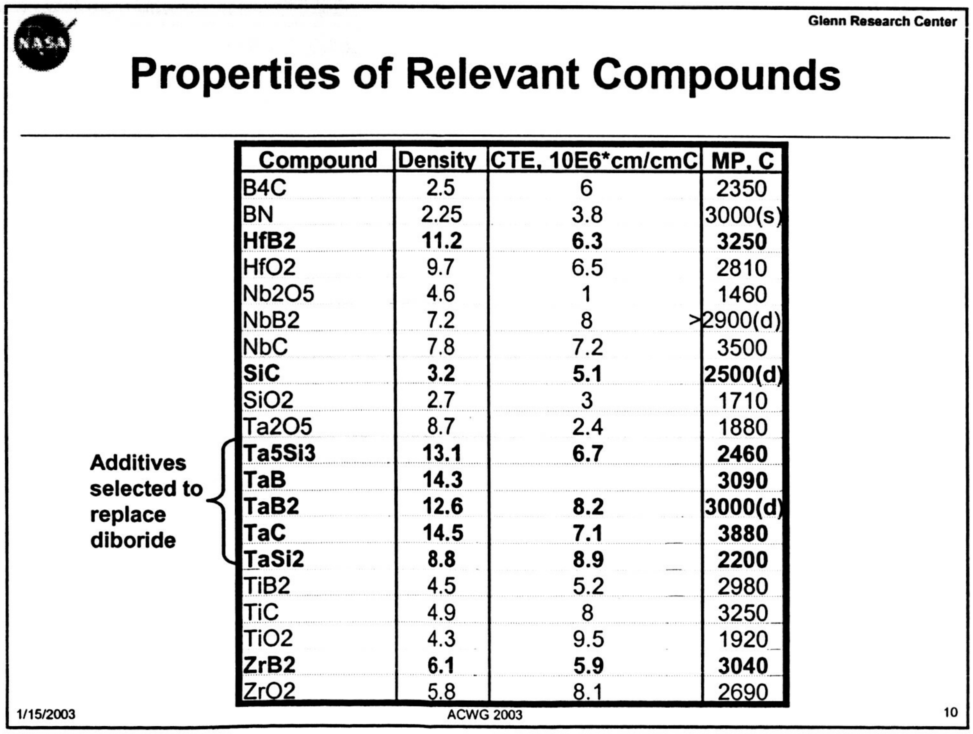
  - Off stoichiometry
  - Porosity, cracking and spalling due to gaseous oxidation products and CTE mismatch
- Doped ZrO_2 or HfO_2
  - Limit oxygen transport via lattice vacancies
  - Still must deal with porosity, cracking, and spalling
  - Ta_2O_5 is the most practical based on melting point and formation of an intermediate ternary oxide with ZrO_2 and HfO_2
  - Potential sources of tantalum are:
    - TaB_2 or TaB : introduces B
    - TaC : introduces additional C
    - $TaSi_2$ or Ta_5Si_3 : introduces more Si + relatively low melting $TaSi_x$ (~2400 °C)
- Just beginning to think about Re_2ZrO_7 or Re_2HfO_7



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NbC	7.8	7.2	3500
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Ta5Si3	**13.1**	**6.7**	**2460**
TaB	**14.3**		**3090**
TaB2	**12.6**	**8.2**	**3000(d**
TaC	**14.5**	**7.1**	**3880**
TaSi2	**8.8**	**8.9**	**2200**
TiB2	4.5	5.2	2980
TiC	4.9	8	3250
TiO2	4.3	9.5	1920
ZrB2	**6.1**	**5.9**	**3040**
ZrO2	5.8	8.1	2690

Additives selected to replace diboride { Ta5Si3, TaB, TaB2, TaC, TaSi2

History for Ta Additions

- Wuchina et al (NSWC) looked at TaB and TaC additions to pure HfC, HfN and HfB_2
  - Limited reporting of results
  - Denser oxide scale at 1500°C and 25% additive reported for HfB_2
- Talmy et al (NSWC) looked at up to 20% TaB_2 additions to ZrB_2 + 20%SiC
  - Only looked at furnace oxidation temperatures up to 1400°C
  - Significant improvements in oxidation resistance attributed to phase separation in the glass
  - Other mechanisms are possible

Mechanisms for ZrO_2 Protective Scale Enhancement via Ta_2O_5 Additions

- Ta_2O_5 as a glass modifier
  - Talmy et al
- Ta^{+5} as a dopant in ZrO_2 lattice
  - Fill O^{-2} vacancies
  - Decrease O^{-2} transport
- Ta_2O_5 as a major oxide scale constituent
  - Phase V
  - Low melting point

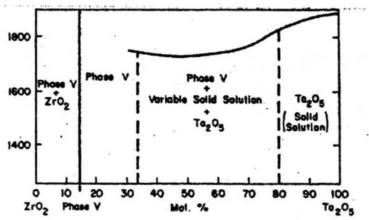
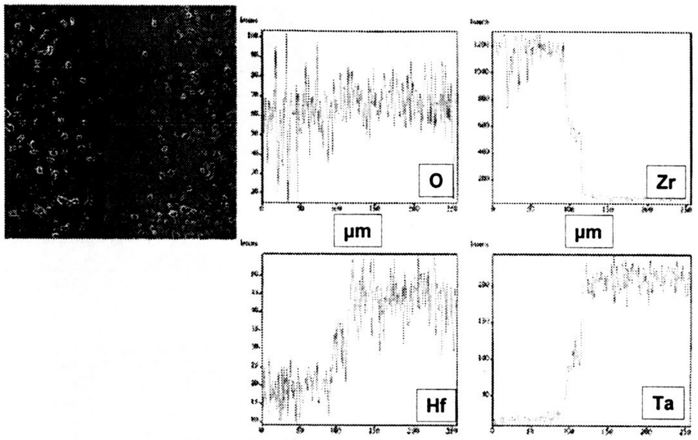


FIG. 374.—System Ta_2O_5-ZrO_2.

B. W. King, John Schultz, E. A. Durbin, and W. H. Duckworth, U. S. Atomic Energy Comm., BMI-1106, 15 (1956).

ZrO_2-Ta_2O_5 Diffusion Couple

1450°C, 1 h



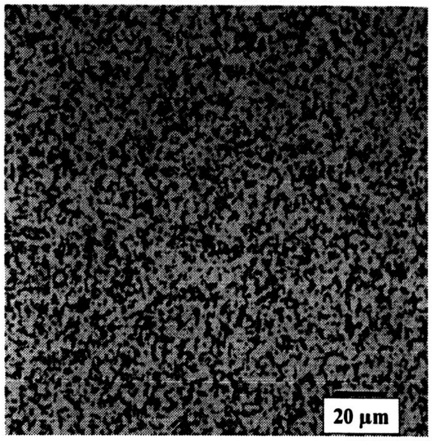
Intermediate phase formation and Ta solubility are evident

Processing

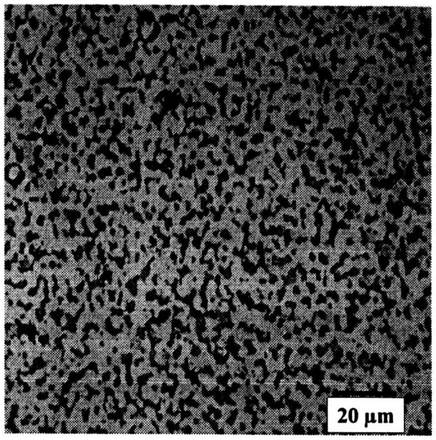
- **Baseline ZrB_2**
  - Plates hot pressed to > 95% average density at 2000°C, 10ksi, up to 2h, vac
- **Ball milling of mixed powders identified as preferred powder processing procedure**
  - One composition processed to 100% density at 1900°C, 10ksi, 2h, vac
  - Segregation observed in some early batches suggested addition of spray drying to the procedure, especially for HfB_2 based system
  - Later batches prepared by the milling of mixed powders appeared to be satisfactory
- **Compositions prepared from mixture of ball milled individual powders were unacceptable**
  - Non-homogeneous with large agglomerates and cracks
  - Further milling as mixed powders solved the problem in the one case tried.

ZrB$_2$ – 20 v/o SiC
Effect of Sintering Time

1h, 2000°C, 10 ksi



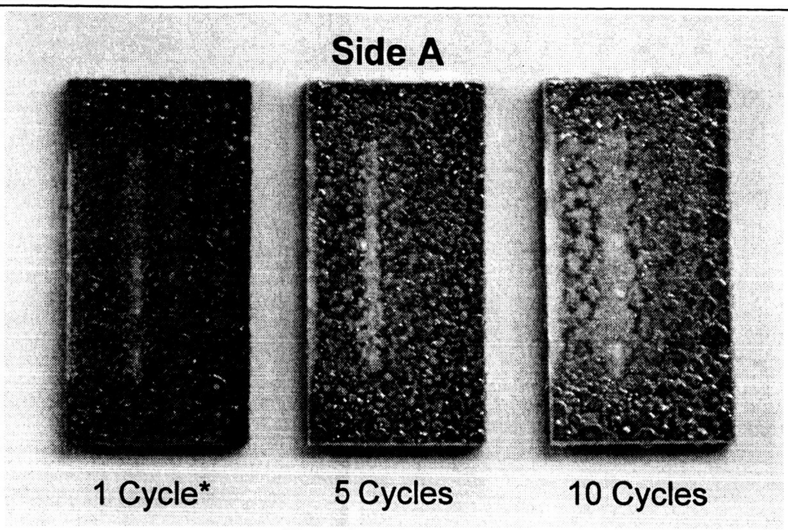
2h, 2000°C, 10 ksi



Flexural Strength Results for ZrB$_2$-based Compositions

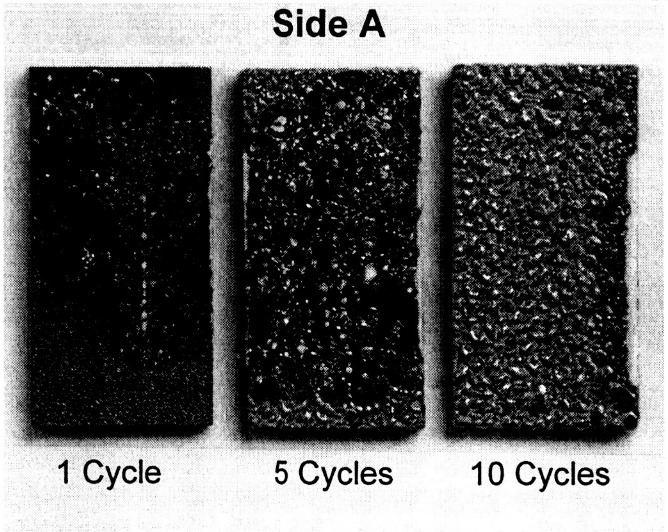
Run	Composition	Powder Process	Processing	UTS, Mpa	% strain	E, GPA
877-1	ZrB2-20v/oSiC	batch 1, mix milled	2000C, 10ksi, 1h, vac	377.4	0.131	289.5
877-2				722.6	0.171	401.5
877-3				749.9	0.181	394.5
877-avg				616.6	0.161	361.8
882-1	ZrB2-20v/oSiC	batch 1, mix milled	2000C, 10ksi, 2h, vac	381.4	0.086	416.8
882-2				588.8	0.106	531.2
882-3				486.9	0.098	472.3
882-avg				485.7	0.097	473.4
897-1	ZrB2-20v/oSiC	batch 2, milled mix	2000C, 10ksi, 2h, vac	576.3	0.129	434.3
897-2				560.2	0.133	378.9
897-3				505.2	0.120	395.6
897-avg				547.2	0.127	402.9
878-1	ZrB2-20v/oSiC-20v/o mixed additives	milled mix	1900C, 10ksi, 2h, vac	838.7	0.178	448.0
878-2				834.3	0.173	475.7
878-3				729.3	0.144	471.1
878-avg				800.8	0.165	464.9
889-1	ZrB2-20v/oSiC-20v/oTaSi2	remill mix milled	1600C, 10ksi, 2h, vac	549.6	0.133	399.0
889-2				786.2	0.185	414.7
889-3				827.0	0.192	424.0
889-avg				720.9	0.170	412.6
908-1	HfB2-20v/oSiC	milled mix	2000C, 10ksi, 2h, vac	563.8	0.116	473.0
908-2				375.4	0.080	459.6
908-3				518.5	0.110	465.4
908-avg				485.9	0.102	466.0
911-1	HfB2-20v/oSiC	milled mix	2000C, 10ksi, 2h, vac	549.5	0.118	447.9
911-2				556.0	0.117	453.4
911-3				534.6	0.117	423.4
911-avg				546.7	0.117	441.6

ZrB_2 + 20 $^{v}/_{o}$ SiC (ZS) After Furnace Oxidation at 1627°C



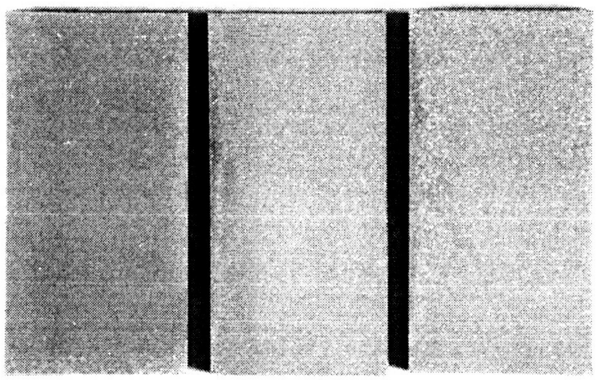
*1 cycle = 10 minutes hot & 10 minutes cool

ZrB_2 + 20 $^{v}/_{o}$ SiC + Mixed Ta Compounds (RB) After Oxidation at 1627°C



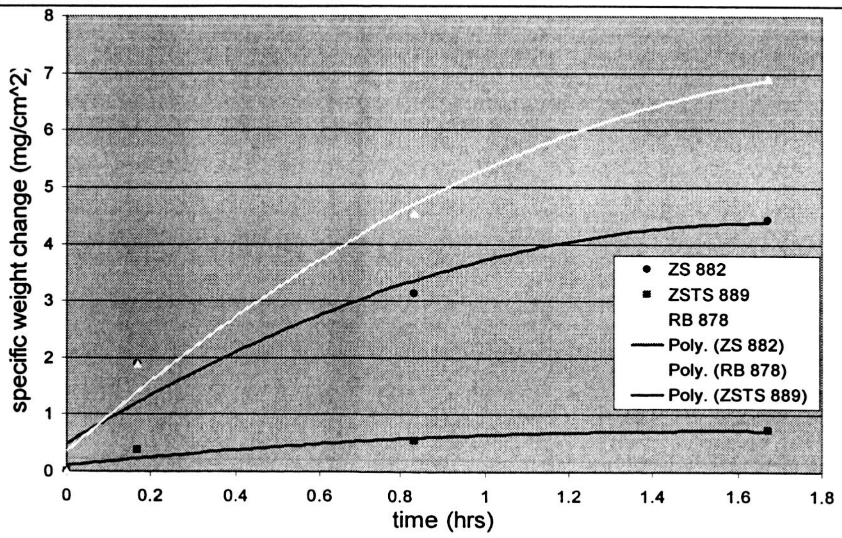
ZrB_2 + 20 v/o SiC + 20 v/o $TaSi_2$ (ZSTS) Oxidized at 1627°C

Side A

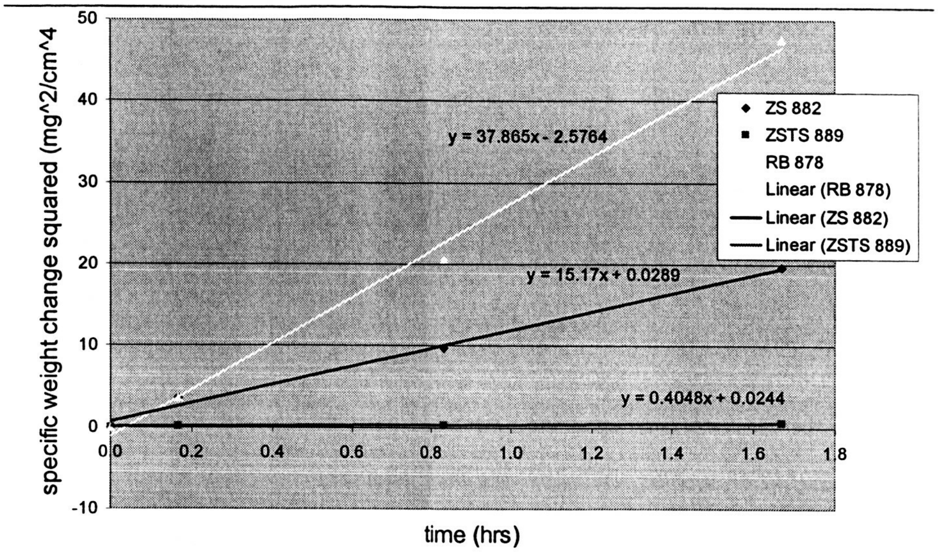


1 Cycle 5 Cycles 10 Cycles

UHTC Oxidation at 1627°C in Zirconia Furnace



UHTC Oxidation at 1627°C in Zirconia Furnace



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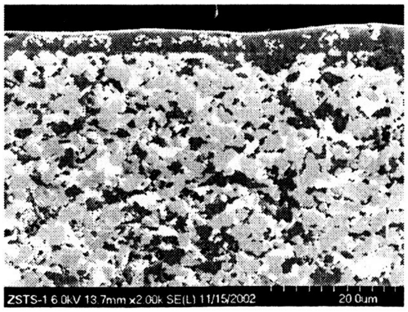
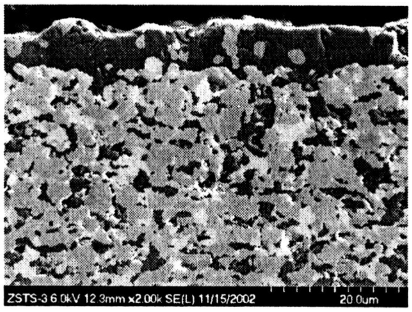
20 %$TaSi_2$ Modified ZrB_2 + 20% SiC After Furnace Oxidation at 1627°C in Air



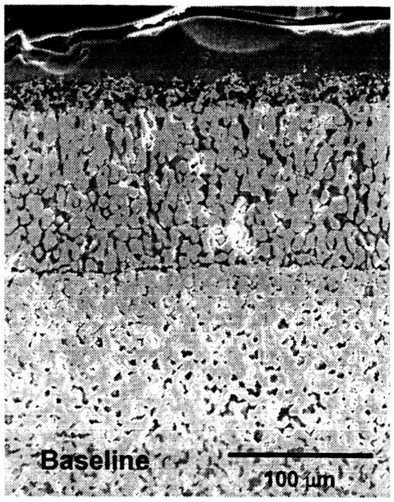
1x10 min cycle, at 1000x

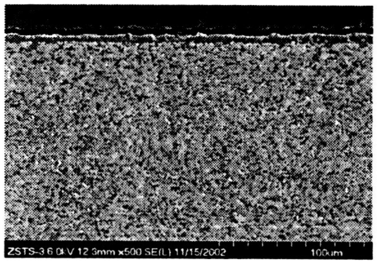


10x10min cycles, at 2000x

➢10X Improvement in Oxidation Resistance of $TaSi_2$ Modified 20 $^{v}/_{o}$ SiC

10x10 minute cycles, air, 1627°C





$TaSi_2$ Modified ZrB_2-20 $^{V}/_{o}$ SiC

>10 X improvement in oxidation resistance relative to industry baseline based on weight change and oxidation damage

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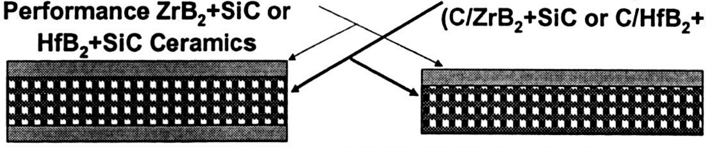
UHTCMC Hybrid System



Coated or Improved Oxidation Performance ZrB_2+SiC or HfB_2+SiC Ceramics

Ceramic Composites (C/ZrB_2+SiC or C/HfB_2+SiC)

Advantages of UHTCMC hybrids

- Thickness and composition of UHTC layers can be changed to suit the application requirements
- Improved toughness
- Improved environmental durability
  - Tailored surface coatings
  - Crack healing matrices
  - Control of residual stresses
- Smooth composite surfaces
  - Low drag
  - Machinable without fiber damage
  - Easier to bond and attach sensors and other devices
  - Critical to good attachments/seals

UHTCMC Hybrid System Processing

Carbon Fiber Preforms Made into Net Shapes

Infiltration of RB_2+SiC+C Mixtures

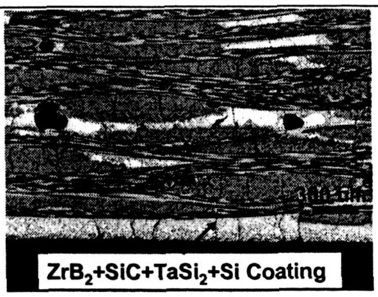
Curing and Pyrolysis

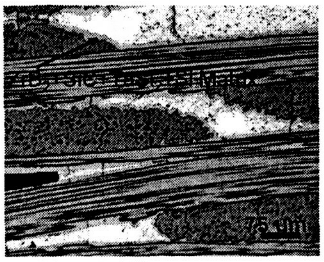
Surface Treatment (RB_2+SiC+C)

Melt Infiltration (R or R-Si alloys)

Ultra High Temperature Ceramic Matrix Composites (UHTCMCs)

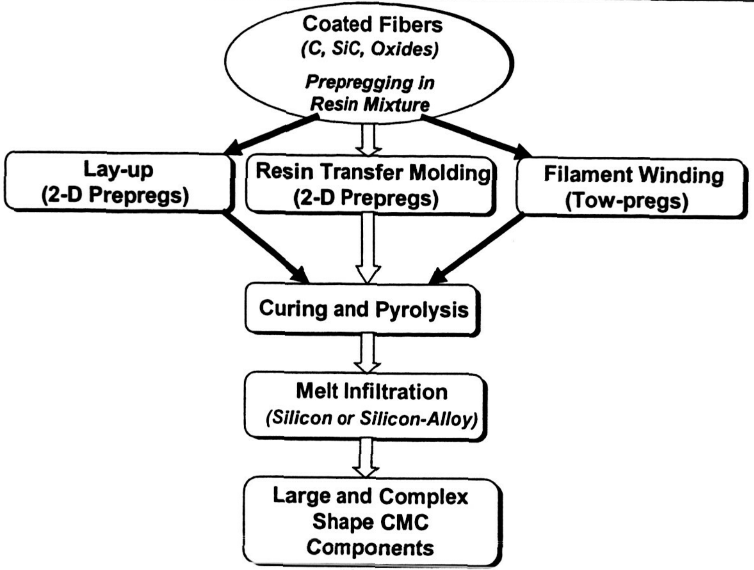
Microstructure of UHTCMC Hybrid System





- ZrB_2 + SiC slurry infiltrant in C/SiC preform
- Similar microstructures in silicon and Si-Ta alloy melt infiltrated systems
- Cracked coatings and matrix due to thermal expansion mismatch
- Process optimization and property characterization underway

CMCs by Prepregging and Melt Infiltration (PREMI)



Coated Fibers
(C, SiC, Oxides)
Prepregging in Resin Mixture

Lay-up
(2-D Prepregs)

Resin Transfer Molding
(2-D Prepregs)

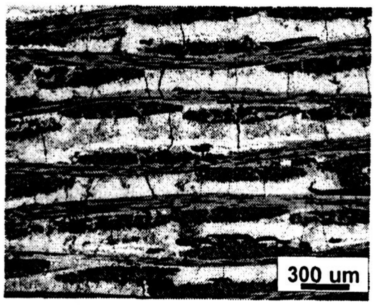
Filament Winding
(Tow-pregs)

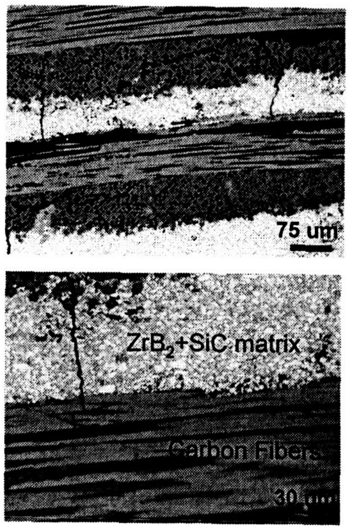
Curing and Pyrolysis

Melt Infiltration
(Silicon or Silicon-Alloy)

Large and Complex
Shape CMC
Components

Microstructure of UHTCMCs by Prepregging and Melt Infiltration (PREMI)





- 12 layers of T-300, 5-HS cloth prepregged with ZrB_2+SiC or ZrB_2+$TaSi_2$ mixture
- Warm pressed, pyrolyzed, and melt
- Infiltrated with Si or Si-Ta alloy

Summary

- Current UHTCs lack robustness (low fracture toughness, reliability and oxidation resistance)
- Alloy and functionally graded materials approaches have been identified to improve oxidation resistance
  - Ta addition appears to be promising with oxidation rate reduced by > 10X @ 1627°C
  - FGM approaches in processing
- Several composites approaches have been identified to increase mechanical robustness
  - Infiltration processing and prepregging have produced materials with interesting microstructures
  - Further characterization of microstructures, and mechanical property and environmental durability is needed to guide the next processing cycle

Conclusions

- Alloying can yield a large improvement in oxidation resistance in a static environment
  - Need to demonstrate in a flowing environment representative of the application
- Several approaches to fabrication of UHTC composites appear to be promising in so far as microstructural appearance
  - Mechanical property and environmental durability evaluations will guide future directions