Ultra High Temperature Ceramics: Application, Issues and Prospects

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- Michael Cinibulk, T. A. Parthasarathy, Allan Katz: AFRL
Outline

• What are UHTCs?
  – Background and features
• Aerospace applications
  – Sharp leading edges
• Properties
• Thoughts on materials development
• Specific issues with UHTCS and approaches
  – Design issues
  – Material issues
  – Modeling
• Thoughts on future directions
  – Technical
  – Application
• Concluding remarks
Ultra High Temperature Ceramics (UHTCs) : A Family of Materials

• Borides, carbides and nitrides of transition elements such as hafnium, zirconium, tantalum and titanium.
• Some of highest known melting points
• High hardness, good wear resistance, good mechanical strength
• Good chemical and thermal stability under certain conditions
• High thermal conductivity (diborides).
  – good thermal shock resistance
Diborides Have Very High Melting Temperatures

\[ \text{ZrB}_2 \]

\[ \text{HfB}_2 \]
Aerospace Application

- The diborides of hafnium and zirconium are of particular interest to the aerospace industry for sharp leading edge applications which require chemical and structural stability at extremely high operating temperatures.

- Some can be used as a monolith or matrix; some are more appropriate as a coating.

- Thermal properties have a significant impact on the surface temperatures.

UHTC billets, quarter for scale
Materials for Sharp Leading Edges

Sustained Hypersonic Flight Limited by Materials
- High heat flux over small area
- High temperature, oxidation, erosion
- Very high temperature gradients

UHTCs (ZrB$_2$/HfB$_2$-based composites)
- High temperature capability and high thermal conductivity
- Poor oxidation resistance
- Low fracture toughness

Modeling/Validation
- Fiber Reinforcement

Courtesy: AFRL
• Sharp leading edge technology
  – Enhances vehicle performance
  – Leads to improvements in safety
    • Increased vehicle cross range
    • Greater launch window with safe abort to ground
• Sharp leading edges place significantly higher temperature requirements on the materials:
  – Current shuttle RCC leading edge materials: T~1650 °C
  – Sharp leading edged vehicles will require: T>2000 °C
• Ultra High Temperature Ceramics (UHTCs) are candidates for use in sharp leading edge applications.
- There are multiple options to manage the intense heating on sharp leading edges.
- Simplest option is passive cooling.
Insulators and UHTCs manage energy in different ways:

- Insulators store energy until it can be eliminated in the same way as it entered
- UHTCs conduct energy through the material and reradiate it through cooler surfaces

UHTC Suitability for TPS

• UHTCs are only for specialized TPS applications for which other material systems are not as capable or straightforward or their capabilities are required when active cooling is not feasible.

• Choice of materials driven by design, environment, and material properties.
  – Feasible simple nose-cone and passive-leading-edge designs have been developed. (UHTC leading edge designs use small volumes of material.)
  – UHTCs have high temperature capabilities (> 2000 °C / 3600 °F)

• Material selection should be based on appropriate testing of matured material in relevant environment.

• Concerns about monolithic UHTC properties are being addressed by processing and engineering improvements (ceramic matrix composites [CMCs])

• Use will depend upon level of maturity relevant to specific application
HfB$_2$ has a narrow range of stoichiometry with a melting temperature of 3380°C. Density = 11.2 g/cm$^3$

Silicon carbide is added to boride powders:
- Promotes refinement of microstructure
- Decreases thermal conductivity of HfB$_2$
- 20v% may not be optimal but is a common amount added
- SiC will oxidize either passively or actively, depending upon the environment

Density = 3.2 g/cm$^3$
Role of SiC in UHTCs

• Silicon carbide is added to boride powders
  • Promotes refinement of microstructure
  • Decreases thermal conductivity of HfB$_2$
  • 20v% may not be optimal but is common amount added
  • SiC will oxidize either passively or actively, depending upon the environment

Baseline hot pressed UHTC microstructure
Dark phase is SiC
UHTC Material Properties

Sharp leading edges require:
- High thermal conductivity (directional)
- High fracture toughness/mechanical strength/hardness
- Oxidation resistance (in reentry conditions)

<table>
<thead>
<tr>
<th>Property</th>
<th>$\text{HfB}_2/20\text{vol}%\text{SiC}$</th>
<th>$\text{ZrB}_2/20\text{vol}%\text{SiC}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cc)</td>
<td>9.57</td>
<td>5.57</td>
</tr>
<tr>
<td>Strength (MPa) at 21°C</td>
<td>356±97*</td>
<td>552±73*</td>
</tr>
<tr>
<td></td>
<td>1400°C 137±15*</td>
<td>240±79*</td>
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<tr>
<td>Modulus (GPa) at 21°C</td>
<td>524±45</td>
<td>518±20</td>
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<tr>
<td></td>
<td>1400°C 178±22</td>
<td>280±33</td>
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<tr>
<td>Coefficient of Thermal Expansion (x10^6/K) at RT</td>
<td>5.9</td>
<td>7.6</td>
</tr>
<tr>
<td>Thermal Conductivity (W/mK) at RT</td>
<td>80</td>
<td>99</td>
</tr>
</tbody>
</table>

Source: ManLabs and Southern Research Institute

* Flexural Strength

HfB$_2$/SiC thermal conductivity was measured on material from the SHARP-B2 program.

Thermal Diffusivity and Heat Capacity of HfB$_2$/SiC were measured using Laser Flash.

HfB$_2$/SiC materials have relatively high thermal conductivity.
Some UHTC Development History

• Hf and ZrB$_2$ materials investigated in early 1950s as nuclear reactor material

• Extensive work in 1960s & 1970s (by ManLabs for Air Force) showed potential for HfB$_2$ and ZrB$_2$ for use as nosecones and leading edge materials (Clougherty, Kaufman, Kalish, Hill, Peters, Rhodes et al.)

• Gap in sustained development during 1980s and most of 1990s
  – AFRL considered UHTCs for long-life, man-rated turbine engines

• During late 1990s, NASA Ames revived interest in HfB$_2$/SiC, ZrB$_2$/SiC ceramics for sharp leading edges

• Ballistic flight experiments: Ames teamed with Sandia National Laboratories New Mexico, Air Force Space Command, and TRW

• Space Launch Initiative (SLI, NGLT, UEET programs: 2001-5

• NASA’s Fundamental Aeronautics Program funded research until 2009

• Substantial current ongoing effort at universities, government agencies, & international laboratories

* Slender Hypervelocity Aerothermodynamic Research Probes
Where are we going?

• What does a UHTC need to do?
  • Carry engineering load at RT - √
  • Carry load at high use temperature
  • Respond to thermally generated stresses (coatings)
  • Survive thermochemical environment - √

• High Melting Temperature is a major criterion, but not the only one
  • Melting temperature of oxide phases formed
  • Potential eutectic formation

• Thermal Stress – $R' = \sigma k/(\alpha E)$
  • Increasing strength helps, but only to certain extent

• Applications are not just function of temperature

• Materials needs for long flight time reusable vehicles are different to those for expendable weapons systems
Why Continue to Develop UHTCs Now?

Given that …

• *Sharp leading edges require refractory materials.*
• *UHTCs have required temperature capability.*

And history tells us …

• Material development is a time-consuming process — 20 years is typical.
• Improvements in ceramic materials and design approaches over time have enabled many advanced applications.

*We need to develop UHTCs now if we want materials to be available for applications.*
Example of Material Development Success – Silicon Nitride

- Intensive research over the past 50 years
- 1950s–1970s: early and substantial research
- 1980s: programs to use material in engines
  - US (turbocharger rotors, cylinder liners)
  - Japan (government and industry). Substantial progress made but applications failed (rotating)
  - Estimated costs of ceramic engine programs: “several thousand million dollars” (ca 2000, F.L. Riley)
- Recent research: substantial improvements in properties leading to significant applications
SiC/SiC and C/SiC Development

- Started with fiber technology — fibers still an issue
- Numerous tech driven projects performed over the past 2+ decades in Europe, Japan, and the US
- SiC/SiC and C/SiC extensively studied since discovery in the mid 70s (French pat. 77/26979 Sept. 1977)
- NASA Enabling Propulsion Materials (EPM) Program: identifying proper CMC constituent materials and processes
  - EPM program terminated in 1999
  - Subsequent Ultra Efficient Engine Technologies (UEET) program built on EPM success
  - US Air Force has built on EPM success
- Hot structures of NASA X38 as example of combined efforts (nose cap, 2 leading edge segments manufactured and ground tested by German consortium, as examples)
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Design Challenges for UHTC Flight Components

• Integrated approach that combines:
  – Mission requirements
  – Aerothermal and aerodynamic environments
  – Structural material selection
  – Component serviceability requirements
  – Safety requirements

• Size of UHTC billets limited to several centimeters — wing leading edges and nosetips must be segmented
  – The design of interfaces between segments is critical

• The mechanical loads on small UHTC components during flight are primarily result of differential thermal expansion within material

• High temperature UHTC components must be attached to vehicle structure (with lower operating temperature limits)
  – Design issue, not materials issue
  – Design concepts developed showed feasibility
UHTC Wing Leading Edge Concept

UHTC wing leading edge (WLE) concept for a hypersonic aircraft:
- UHTC segmented leading edge attached to carbon-based hot structure
- Nose radius ~1cm
UHTC WLE Concept

UHTC wing leading edge component concepts — intersegment faces with interlocking geometric features — would aid in assembly and mitigate hot gas flow through the gap from the windward side to leeward side.
Example of Predicted UHTC WLE Component Performance

- UHTC WLE under reentry heating conditions
- Peak predicted thermal stress of 80 MPa was well below demonstrated UHTC strengths between 300 to 400 MPa
Improving Processing and Microstructure

• Initial focus on improving material microstructure and strength
• HfB$_2$/20vol%SiC selected as baseline material for project constraints
• Major issue was poor mixing/processing of powders with different densities
  - Used freeze-drying to make homogenous powder granules
  - Developed appropriate hot pressing schedules

Granulated HfB$_2$/SiC Powder
Early HfB$_2$ - 20% SiC Materials

- Early and SHARP materials made by an outside vendor
- Improvements in powder handling provide a more uniform microstructure

Understand what you are testing!
UHTCs with Improved Strength (MS&T)

- Historic strengths were modest
  ~415 MPa reported in 1971*
  ZrB$_2$ grain size ~10 μm

- Strengths higher for recent materials
  >1000 MPa for ZrB$_2$-30 vol% SiC
  Control of microstructure is key
  ZrB$_2$ grain size ~3 μm

- Strength controlled by SiC particle size
  Stronger as SiC gets smaller

- Finite element modeling shows that
  residual stresses arise due to the CTE
  difference between ZrB$_2$ and SiC
  Compressive in SiC
  Tensile in ZrB$_2$ matrix

- Residual stresses affected by size and
  shape of SiC inclusions

Pressureless Sintering for Cost Reduction & Complex Shapes at MS&T

• Pressureless sintering methods have been developed for ZrB$_2$ and ZrB$_2$-SiC

• Sintering enables fabrication of complex shapes
  – Conventional powder processing, uniaxial pressing, and pressureless sintering
  – Wedges produced to near net shape with finish machining after sintering to produce the desired radius
  – Mushrooms produced by green machining, sintering, and polishing the top

• Sintered wedges were tested at Boeing Large Core Arc Tunnel in St. Louis
Need for Arc Jet Testing

• Arc jet testing is the best ground-based method of evaluating a material’s oxidation/ablation response in re-entry environments
• A material’s oxidation behavior when heated in static or flowing air at ambient pressures is likely to be significantly different than in a re-entry environment.
• In a re-entry environment:
  – Oxygen and nitrogen may be dissociated
    • Catalycity of the material plays an important role
    • Recombination of O and N atoms adds to surface heating
  – Stagnation pressures may be less than 1 atm.
    • Influence of active to passive transitions in oxidation behavior of materials
      – SiC materials show such a transition when the protective SiO$_2$ layer is removed as SiO
Simulates reentry conditions in a ground-based facility

Method: Heat a test gas (air) to plasma temperatures by an electric arc, then accelerate into a vacuum chamber and onto a stationary test article

UHTC Cone After 9 Arc Jet Exposures
(89 minutes total run time)

Runs 4 and 5 lasted ~ 2 min. each

<table>
<thead>
<tr>
<th>HSp-45 Pretest</th>
<th>Run 1 Post-Test</th>
<th>Run 2 Post-Test</th>
<th>Run 3 Post-Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>300 sec % Δwt = 0</td>
<td>600 sec % Δwt = 0</td>
<td>600 sec % Δwt = 0</td>
<td>600 sec % Δwt = -0.06</td>
</tr>
<tr>
<td>T_{ss} = 1280°C</td>
<td>T_{ss} = 1220°C</td>
<td>T_{ss} = 1325°C</td>
<td>T_{ss} = 1970°C</td>
</tr>
</tbody>
</table>

Run 6 Post-Test
600 sec
% Δwt = -0.2
T_{ss} > 2000°C

Run 7 Post-Test
1200 sec
% Δwt = -0.32
T_{ss} > 2000°C

Run 8 Post-Test
1200 sec
% Δwt = -1.24
T_{ss} > 2000°C

Run 9 Post-Test
600 sec
% Δwt = -0.32
T_{ss} > 2000°C

Increasing heat flux

2.54 cm

Runs 4 and 5 lasted ~ 2 min. each

Increasing heat flux
Reducing Oxide Formation

* Post-test arc jet nosecone model after a total of 80 minutes of exposure. Total exposure the sum of multiple 5 and 10 minute exposures at heat fluxes from 200W/cm²

- In baseline material:
  - SiC depleted during arc jet testing
  - Surface oxide is porous

- Potential solution: Reduce amount of SiC below the percolation threshold while maintaining mechanical performance

*Arc jet test data from Space Launch Initiative program
UHTC Evaluation under Service-Relevant Conditions

Understand behavior of UHTCs in hypersonic environments

- Evaluate response under realistic hypersonic conditions in various rigs (scramjet, arc-jet, laser, etc.)

- Develop robust models using responses from rig testing to predict performance in actual service
Pressure-Time Traces of Graphite and HfB$_2$
Throats Showing Non-eroding Behavior of Ceramic
What About Active Oxidation?

- Silicon-containing materials will actively oxidize under high temperature, low pressure conditions, forming SiO as gas
- Most problematic during re-entry (not during cruise)
- Mitigation approaches:
  - Reduce volume of SiC
    - Reduce overall oxidation
    - Below percolation threshold
  - Reduce scale of SiC particles
    - Allows formation of protective oxide sooner
    - Increase tortuosity of diffusion path
    - Balance between control of grain size and limit of oxidation
  - Additives
    - To change viscosity of the oxide
      - Change emissivity (lower surface temperature)
      - Change diffusivity of species through the oxide
    - To form a physical barrier
    - To change sintering behavior of UHTC with consequent reduction in SiC
Modeling Oxidation Kinetics

Model MB$_2$ (isothermal)

Model MB$_2$-SiC (isothermal)

Model MB$_2$-SiC

high velocity air

Model MB$_2$-SiC
high velocity air
in temperature gradient

Adequate literature data available on ZrB$_2$, HfB$_2$
(but scattered)

Limited data (none @ T >1650C)
on ZrB$_2$-SiC, HfB$_2$-SiC

No data under well-controlled conditions

No data under “well-controlled” conditions;
some arc-jet data on ZrB$_2$-SiC, HfB$_2$-SiC

Parthasarathy et al., Acta Mater., 2007
Parthasarathy et al., J. Am. Ceram. Soc., in review

Courtesy AFRL
Arcjet Characterization: Additives & Influence of Microstructure

Both oxide scale and depletion zone can be reduced.
In Situ Composite for Improved Fracture Toughness

Evidence of crack growth along HfB$_2$-SiC interface, with possible SiC grain bridging
Ultra High Temperature Continuous Fiber Composites

- Image at top right shows dense UHTC matrix with indications of high aspect ratio SiC.
- Image at bottom right shows the presence of C fibers after processing.
• Nicalon SiC fiber
• HfB$_2$-filled SMP-10 (SiC precursor) slurry
• Tape wound infiltrated tows
• Unidirectional composite panels

SiC$_f$/HfB$_2$-SiC Composites

Courtesy AFRL
Engineered Architectures for Improved Thermal Shock Resistance at MS&T

- Thermal stress resistance can be improved
  \( \Delta T_{\text{critical}} \approx 400 ^\circ \text{C} \) for conventional ceramic
  Improves to \( \approx 1400 ^\circ \text{C} \) for cellular architecture

- Fiber reinforcement could produce additional gains
Arc Welding of UHTCs (MS&T)

- ZrB$_2$-based UHTCs, up to 3 mm thick, have been joined by gas tungsten arc welding (GTAW).
- Joint strengths were lower than the parent materials due to the formation of voids at the interface between the heat affect zone and the parent material.

Joint Strengths

- Should be reduced by optimizing the starting composition, atmosphere, or heat input.
Modeling of UHTCs Will Enhance Development

Goals
• Reduce materials development time
• Optimize material properties/tailor materials
• Guide processing of materials
• Develop design approaches

Approach
• Develop models integrated across various length scales
• Correlate models with experiment whenever possible
Multiscale Modeling of Materials

• **Ab initio calculations** — intrinsic material properties
  – *Enables:* structure, bonding, optical and vibrational spectra, chemical reactions, etc
  – *Challenges:* computationally very demanding (very small systems only — $10^2$ atoms)

• **Atomistic simulations** — localized interfaces, defects, transport, and so forth
  – *Enables:* thermal transport, mechanical properties, interface (for example, grain boundary) adhesion, impurities effects
  – *Challenges:* requires difficult interatomic potential development (except for C, Si, and so forth) (small systems and short time scales — $10^8$ atoms and $10^{-9}$ sec)

• **Image-based FEM** — microstructural modeling
  – *Enables:* thermal, mechanical, fracture analysis based on microstructure
  – *Challenges:* requires large database of materials parameters (from experiment or modeling). Nonlinear problems (fracture, plasticity) are very challenging. Macroscopic limit may be difficult.
Modeling UHTCs – What’s Next?

• **Accomplishments**
  - *Ab initio* calculations of lattice structure, bonding characteristics, elastic constants, phonon spectra and thermal properties of ZrB$_2$ and HfB$_2$
  - *Ab initio* calculations of formation and migration energies for simple defects (vacancies)
  - Development of interatomic potentials for ZrB$_2$ and HfB$_2$ for atomistic simulations

• **Opportunities**
  - *Ab initio* calculations of simple/ideal grain boundary structures with and without chemical impurities
  - *No UHTC atomistic simulations exist in the literature.* *New potentials mean the field is wide open!*
  - FEM modeling of microstructure to relate processing and properties
What are the issues with use of UHTCs?

• Similar to the risk aversion in many industries in using structural ceramics!
• Designers prefer to use metals or complex systems to avoid using advanced ceramics and composites.
  – Industry is conservative
  – Building a system, not developing materials
  – Unfamiliarity with designing with brittle materials - safety factor.
  – Advantages of weight savings and uncooled temperature capability not high enough to overcome risk aversion
• Using monolithic ceramics and CMCs requires a different design approach, not straight replacement of a metal part
• Need for subscale materials/component testing in realistic environments is imperative
• Must develop materials and test them such that designers can increase their comfort level
  – Must do in advance of need!
• Must have ways of moving materials from research and development (low technology readiness level) to demonstration of applications through testing in realistic environments
UHTC Challenges: What will make designers use these materials?

1. Fracture toughness: Composite approach is required
   - Integrate understanding gained from monolithic materials
   - Need high temperature fibers
   - Need processing methods/coatings

2. Oxidation resistance in reentry environments
   reduce/replace SiC

3. Modeling is critical to shorten development time, improve properties and reduce testing

4. Joining/integration into a system

5. Test in relevant environment—test data!
Some Recent Research Efforts in UHTCs:
Materials and Properties

<table>
<thead>
<tr>
<th>ZrB₂ Based Ceramics</th>
<th>Catalytic Properties of UHTCs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Missouri University of Science &amp; Technology</td>
<td>PROMES-CNRS Laboratory, France</td>
</tr>
<tr>
<td>US Air Force Research Lab (AFRL)</td>
<td>CNR-ISTEC</td>
</tr>
<tr>
<td>NASA Ames &amp; NASA Glenn Research Centers</td>
<td>CIRA, Capua, Italy</td>
</tr>
<tr>
<td>University of Illinois at Urbana-Champaign</td>
<td>SRI International, California</td>
</tr>
<tr>
<td>Harbin Institute of Technology, China</td>
<td>Imaging and Analysis (Modeling)</td>
</tr>
<tr>
<td>Naval Surface Warfare Center (NSWC)</td>
<td>University of Connecticut</td>
</tr>
<tr>
<td>NIMS, Tsukuba, Japan</td>
<td>AFRL</td>
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<tr>
<td>Imperial College, London, UK</td>
<td>NASA Ames Research Center</td>
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<tr>
<td>Korea Institute of Materials Science</td>
<td>Teledyne (NHSC-Materials and Structures)</td>
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<tr>
<td>CNR-ISTEC</td>
<td>Oxidation of UHTCs</td>
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<td>Texas A &amp; M University</td>
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<th>Fiber Reinforced UHTCs</th>
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### Some Recent Research Efforts in UHTCs: Processing

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<th>UHTC Polymeric Precursors</th>
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<td><strong>Pressureless Sintering</strong></td>
<td><strong>Nano &amp; Sol Gel Synthesis of UHTCs</strong></td>
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<td><strong>Reactive Hot-Pressing</strong></td>
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UHTC Researchers Throughout the World
Summary

- Work on UHTC-type compositions decades in development, but non-continuous.
- Significant expansion of interest in UHTCs in past 10 years — multinational research.
- Considerable improvements have been made in processing and properties.
- Must develop materials to meet needs of application
- Must test in relevant environment
- UHTCs may not find application by themselves but as parts of systems, and thus continued research is critical to the success of future applications.

Long and winding road to applications!