



Ultra High Temperature Ceramics: Application, Issues and Prospects

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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

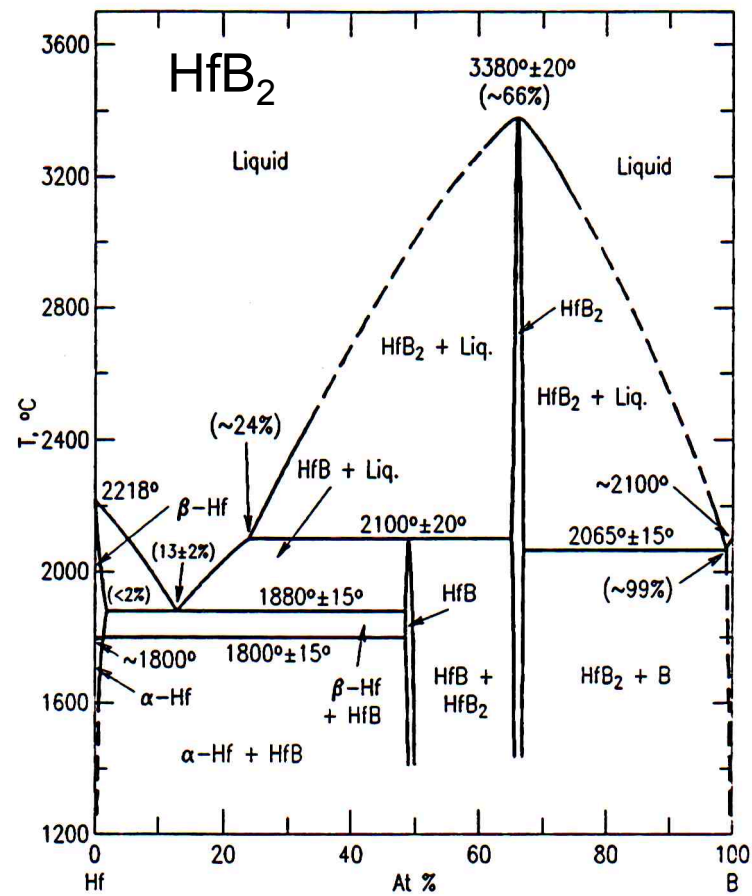
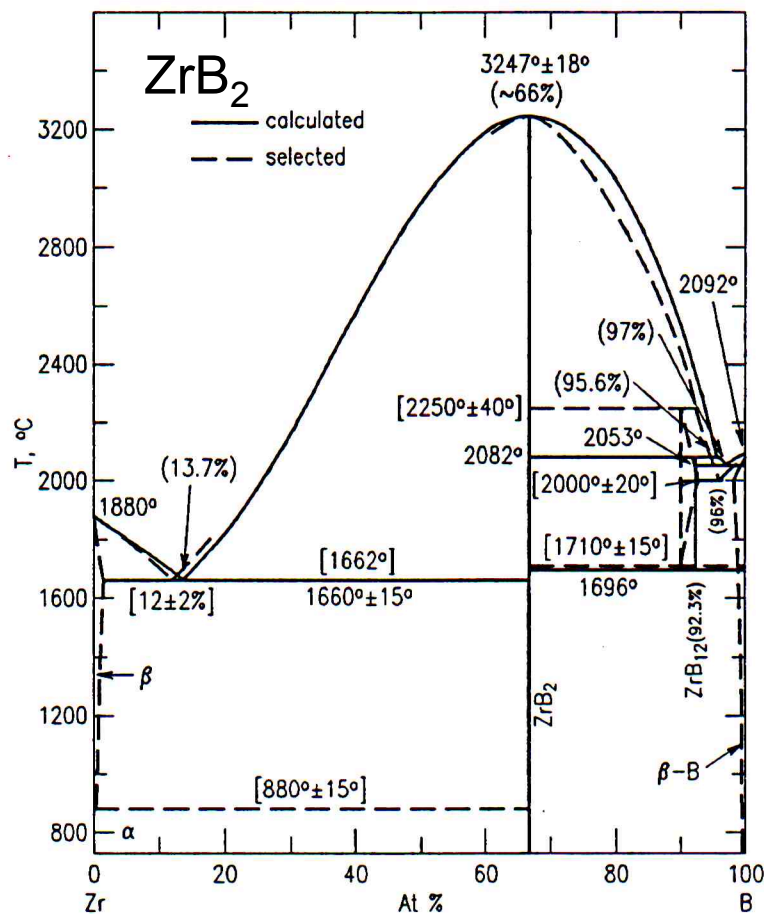


Ultra High Temperature Ceramics (UHTCs) : A Family of Materials

- 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



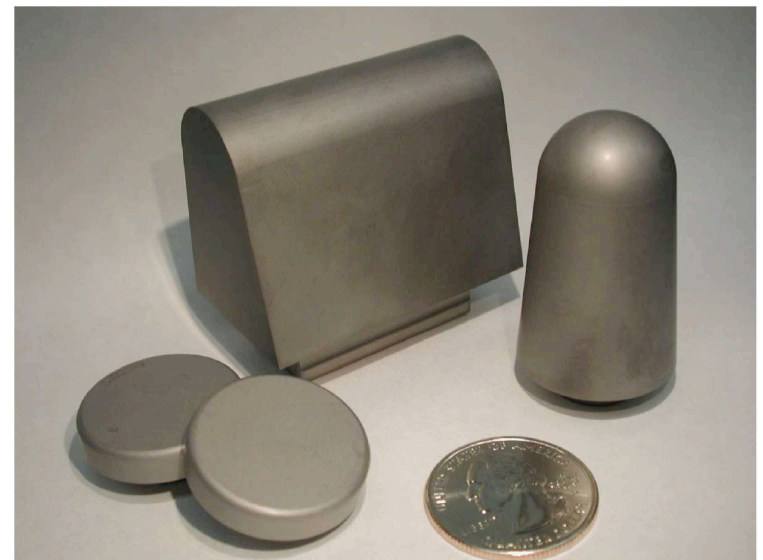
Diborides Have Very High Melting Temperatures





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

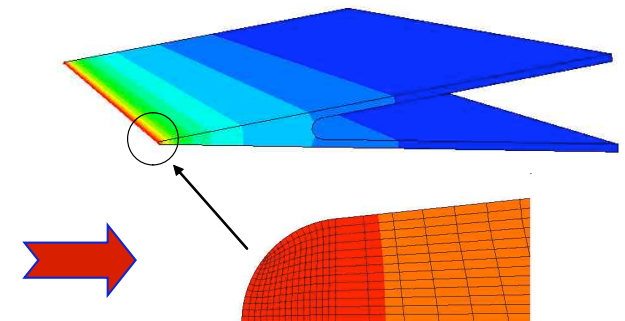
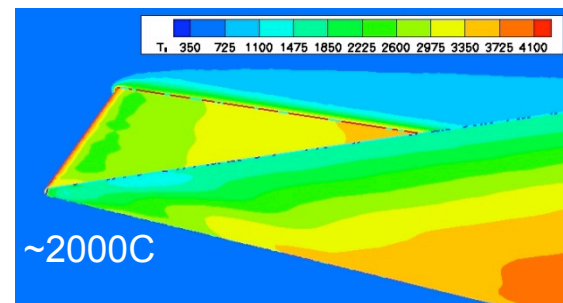
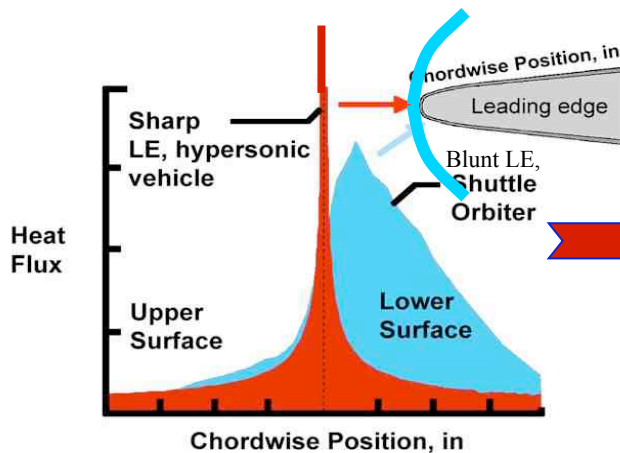
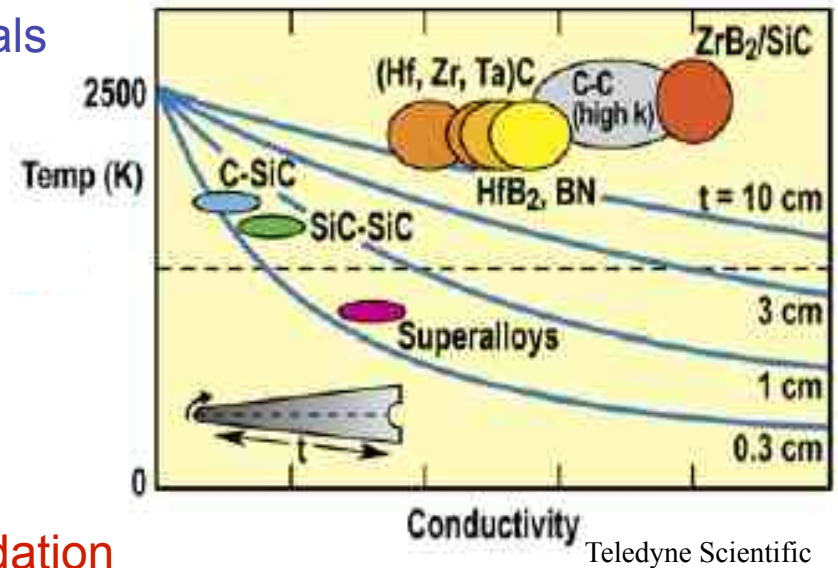
Materials for Sharp Leading Edges

Sustained Hypersonic Flight Limited by Materials

- High heat flux over small area
- High temperature, oxidation, erosion
- Very high temperature gradients

UHTCs (ZrB₂/HfB₂-based composites)

- High temperature capability and high thermal conductivity
- Poor oxidation resistance → Modeling/Validation
- Low fracture toughness → Fiber Reinforcement



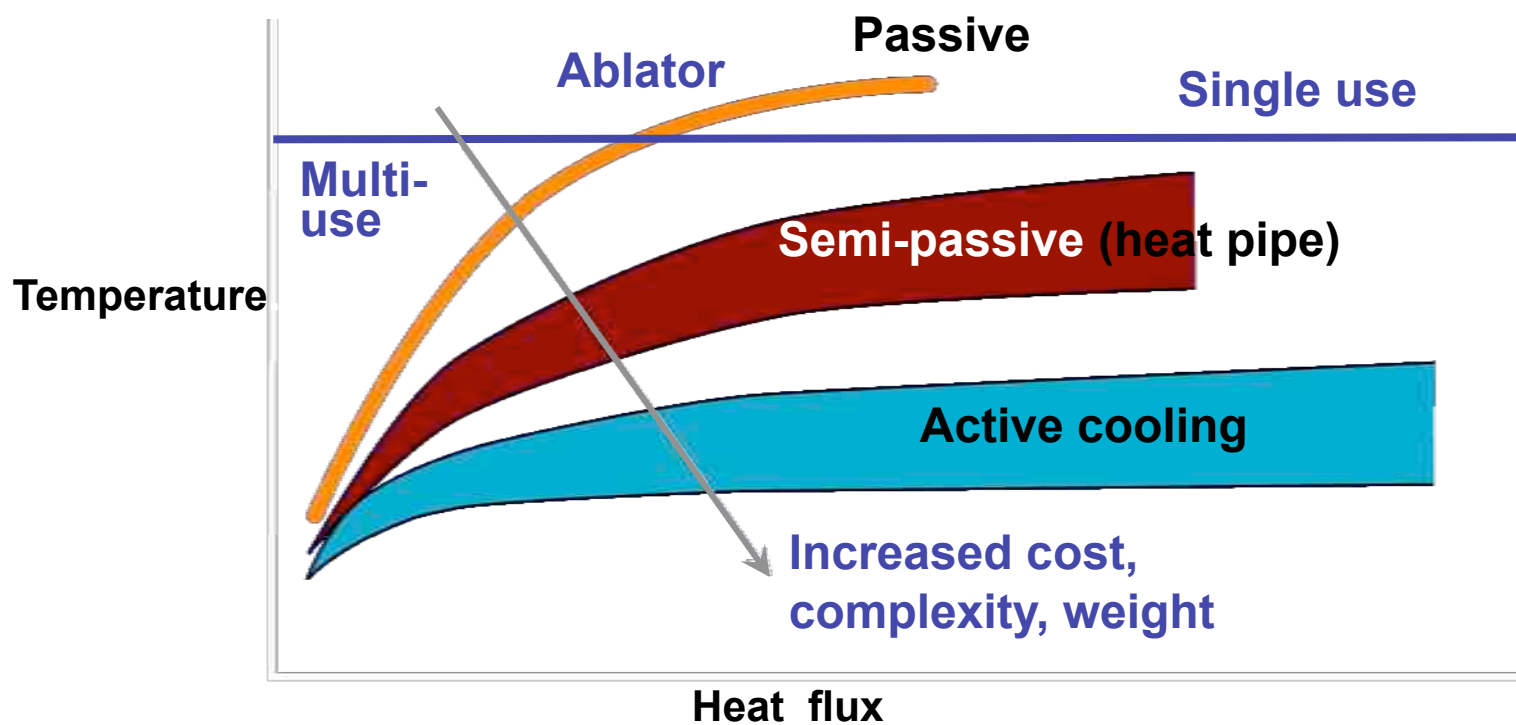


Sharp Leading Edge Technology / Review

- 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: $T \sim 1650$ °C
 - Sharp leading edged vehicles will require: $T > 2000$ °C
- Ultra High Temperature Ceramics (UHTCs) are candidates for use in sharp leading edge applications.



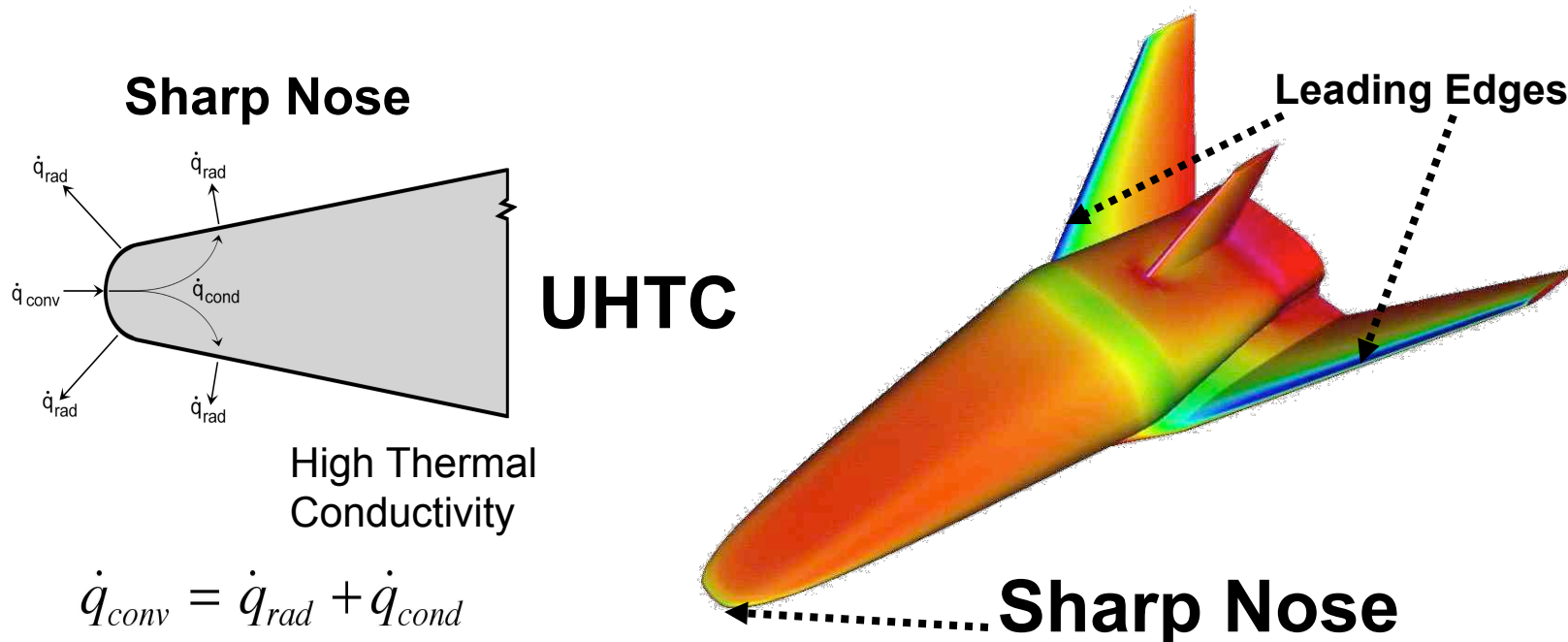
Leading-Edge Thermal Management Options



- There are multiple options to manage the intense heating on sharp leading edges.
- Simplest option is passive cooling.



Sharp Leading Edge Energy Balance



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

Dean Kontinos, Ken Gee and Dinesh Prabhu. "Temperature Constraints at the Sharp Leading Edge of a Crew Transfer Vehicle." AIAA 2001-2886 35th AIAA Thermophysics Conference, 11-14 June 2001, Anaheim CA



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 ($> 2000\text{ }^{\circ}\text{C}$ / $3600\text{ }^{\circ}\text{F}$)
- 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



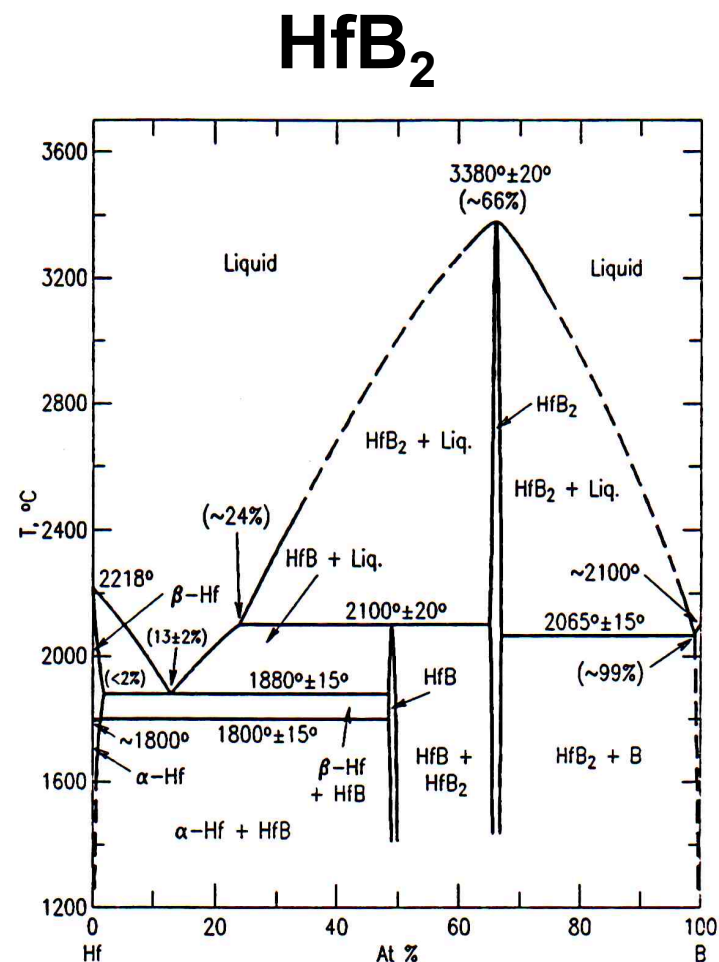
Processing of HfB_2 -SiC

- 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 common amount added
 - SiC will oxidize either passively or actively, depending upon the environment

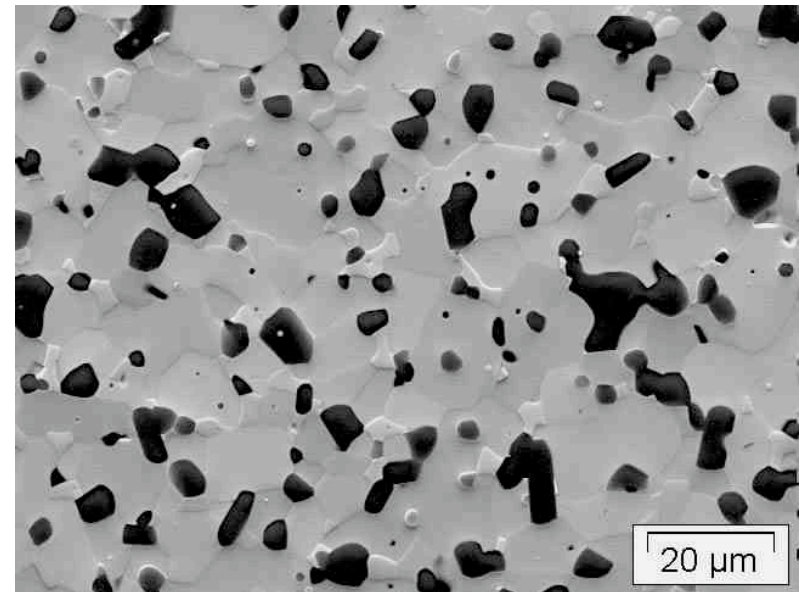
Density = 3.2 g/cm^3





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)

Property	HfB ₂ /20vol%SiC	ZrB ₂ /20vol%SiC
Density (g/cc)	9.57	5.57
Strength (MPa) 21°C	356±97*	552±73*
1400°C	137±15*	240±79*
Modulus (GPa) 21°C	524±45	518±20
1400°C	178±22	280±33
Coefficient of Thermal Expansion (x10 ⁻⁶ /K) RT	5.9	7.6
Thermal Conductivity (W/mK) [#] RT	80	99

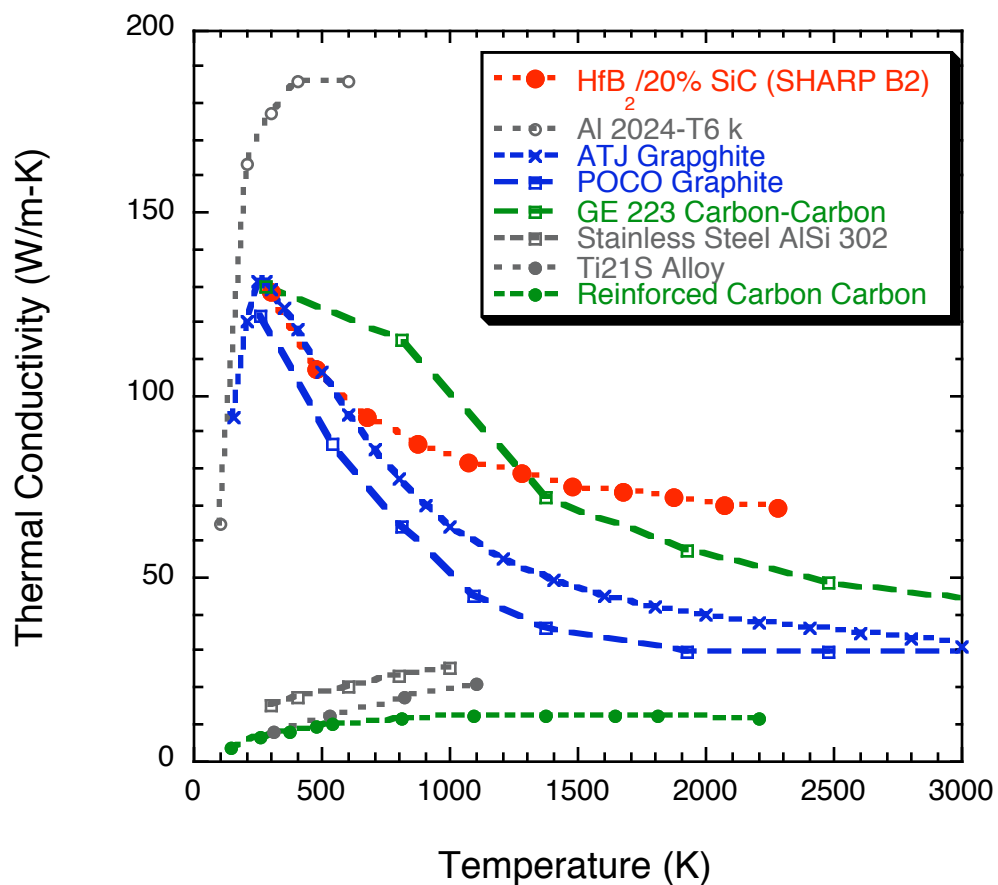
Source: ManLabs and Southern Research Institute

* Flexural Strength

R. P. Tye and E. V. Clougherty, "The Thermal and Electrical Conductivities of Some Electrically Conducting Compounds." Proceedings of the Fifth Symposium on Thermophysical Properties, The American Society of Mechanical Engineers, Sept 30 – Oct 2 1970. Editor C. F. Bonilla, pp 396-401.



Thermal Conductivity Comparison



HfB₂/SiC materials have relatively high thermal conductivity

- HfB₂/SiC thermal conductivity was measured on material from the SHARP-B2 program.
- Thermal Diffusivity and Heat Capacity of HfB₂/SiC were measured using Laser Flash.



Some UHTC Development History

- Hf and ZrB₂ materials investigated in early 1950s as nuclear reactor material
- Extensive work in 1960s & 1970s (by ManLabs for Air Force) showed potential for HfB₂ and ZrB₂ 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 HfB₂/SiC, ZrB₂/SiC ceramics for sharp leading edges
- Ballistic flight experiments: Ames teamed with Sandia National Laboratories New Mexico, Air Force Space Command, and TRW
 - SHARP*-B1 (1997) UHTC nosetip & SHARP-B2 (2000) UHTC strake assembly
- 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 - \checkmark
 - Carry load at high use temperature
 - Respond to thermally generated stresses (coatings)
 - Survive thermochemical environment - \checkmark
- 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)



DARPA/Air Force Falcon HTV-2 C/C aeroshell



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Design Challenges for UHTC Flight Components

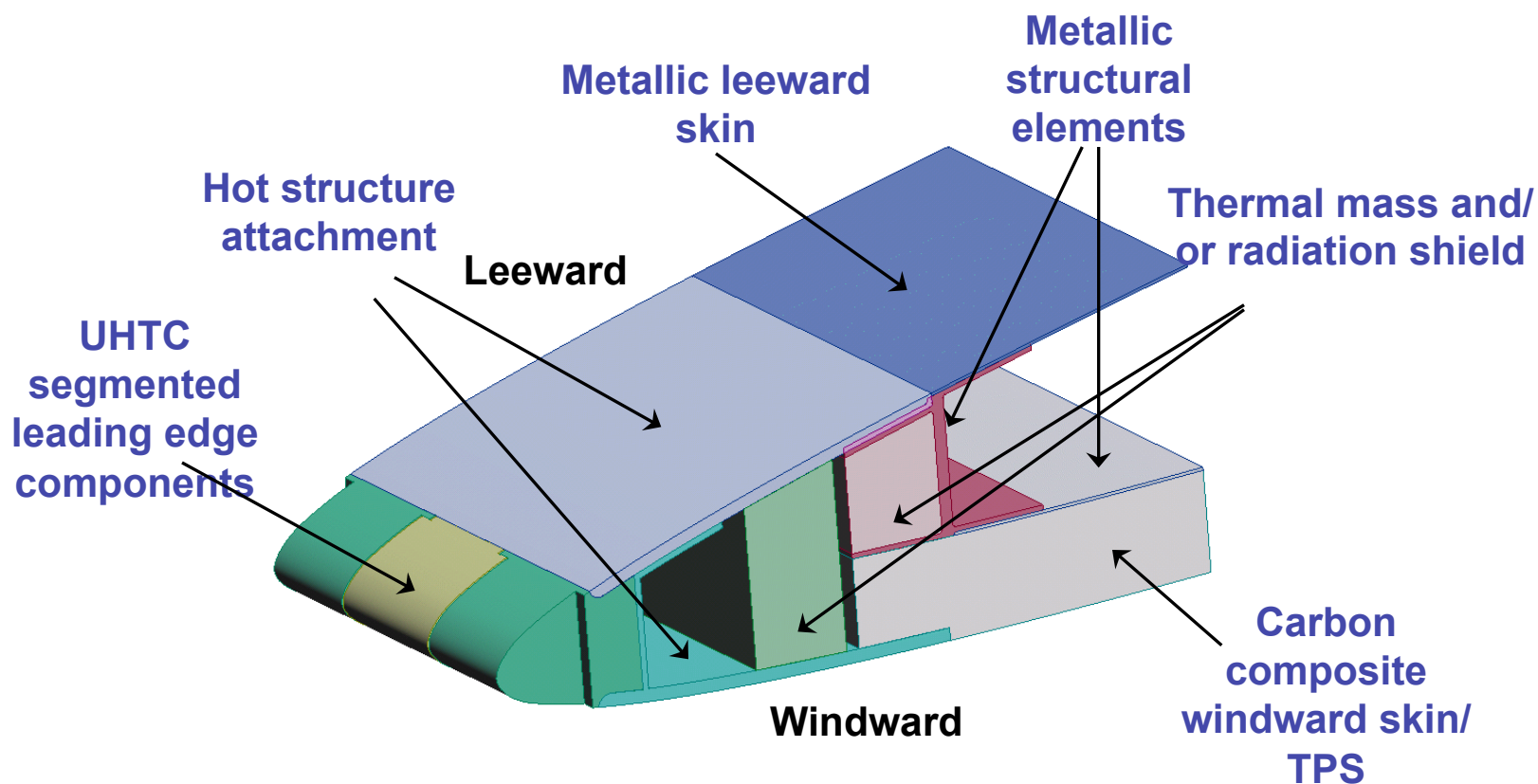
- Integrated approach that combines:
 - Mission requirements
 - Aerothermal and aerodynamic environments
 - Structural material selection
 - Component serviceability requirements
 - Safety requirements
- Size of UHTC billets limited to several centimeters — wing leading edges and nosetips must be *segmented*
 - 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:

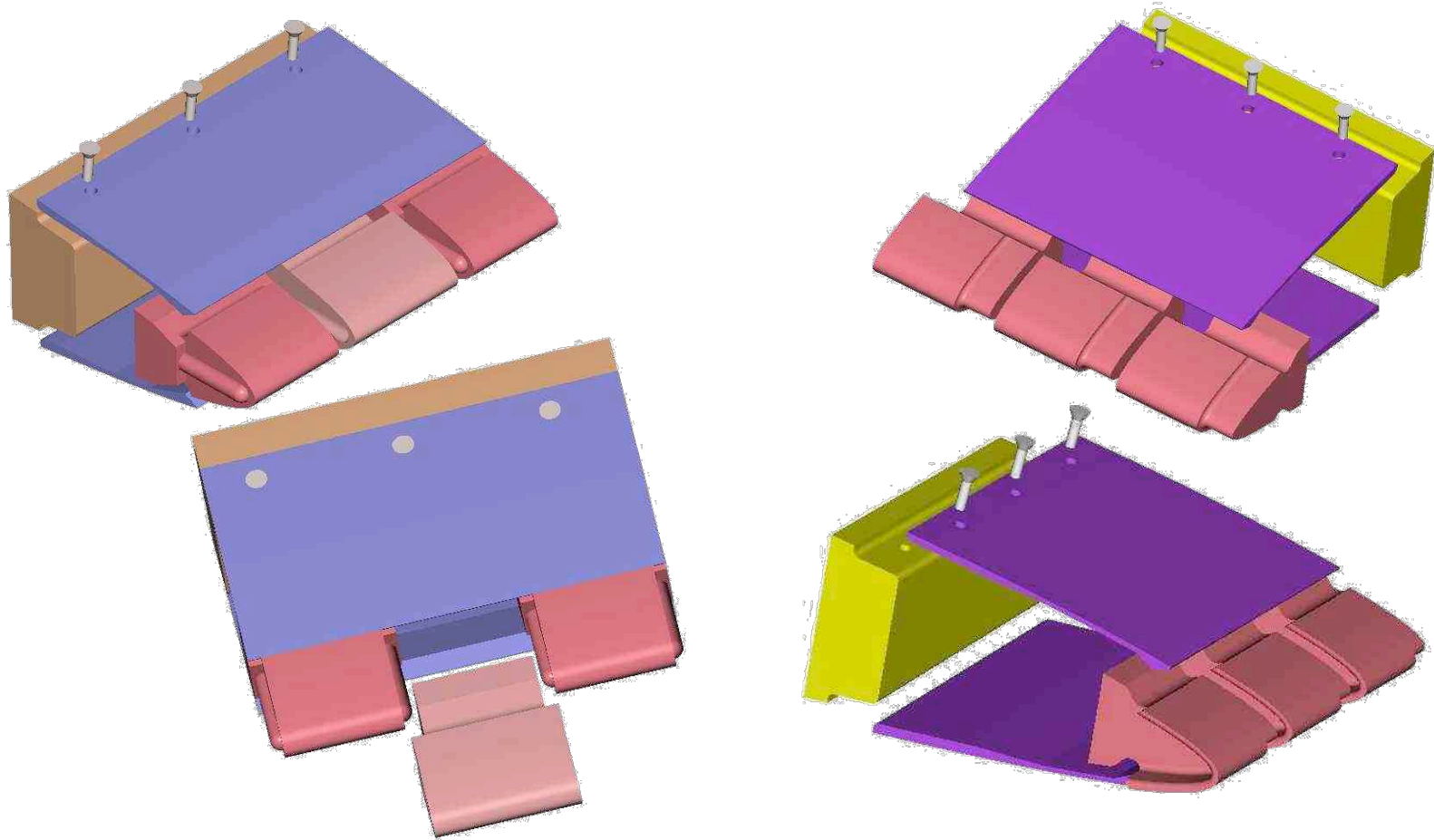
- UHTC segmented leading edge attached to carbon-based hot structure
- Nose radius $\sim 1\text{cm}$





UHTC WLE Concept

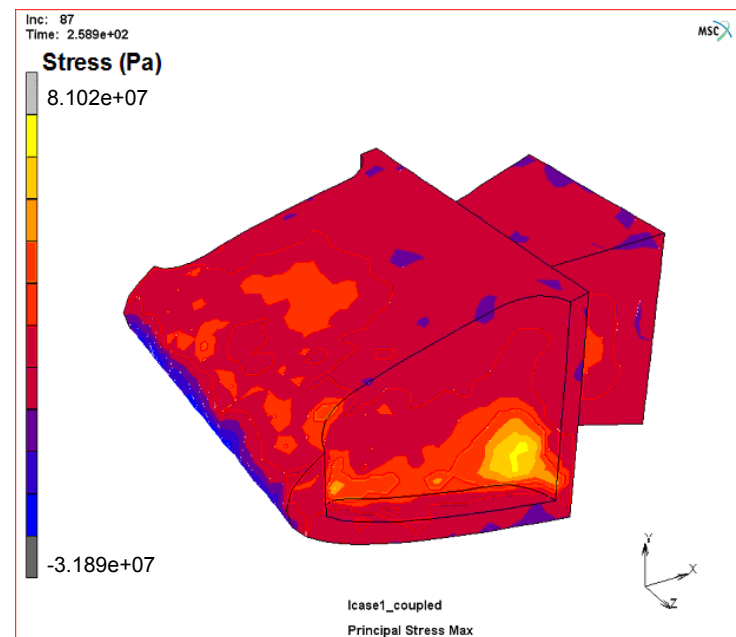
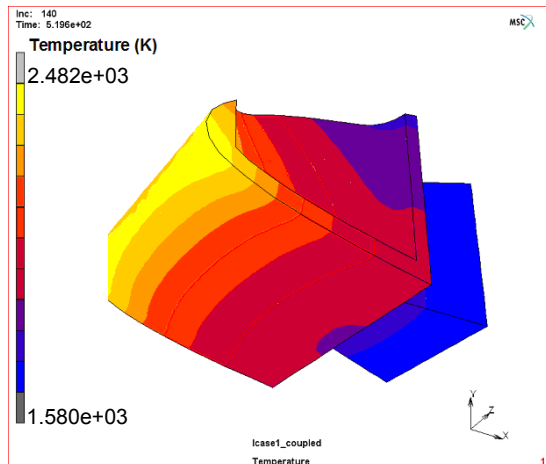
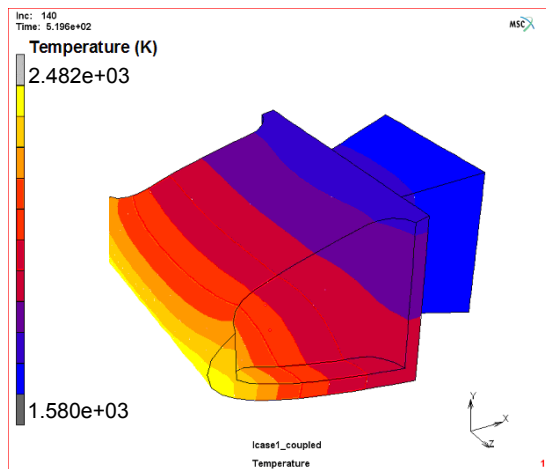
UHTC wing leading edge component concepts — intersegment faces with interlocking geometric features — would aid in assembly and mitigate hot gas flow through the gap from the windward side to leeward side.





Example of Predicted UHTC WLE Component Performance

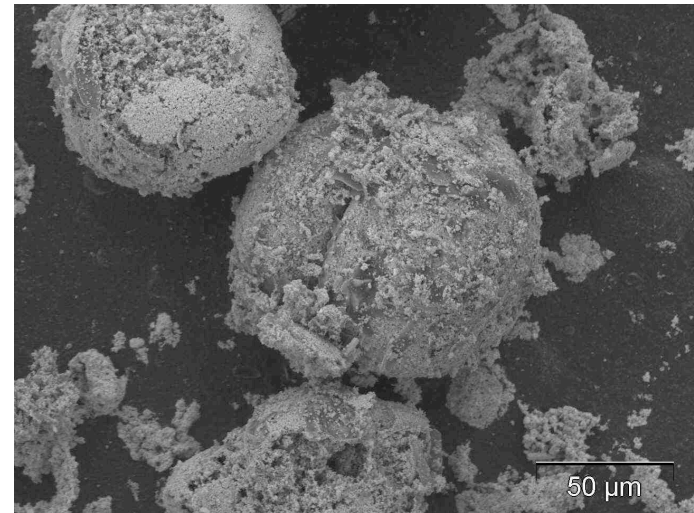
- UHTC WLE under reentry heating conditions
- Peak predicted thermal stress of 80 MPa was well below demonstrated UHTC strengths between 300 to 400 MPa





Improving Processing and Microstructure

- Initial focus on improving material microstructure and strength
- $\text{HfB}_2/20\text{vol}\%\text{SiC}$ 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

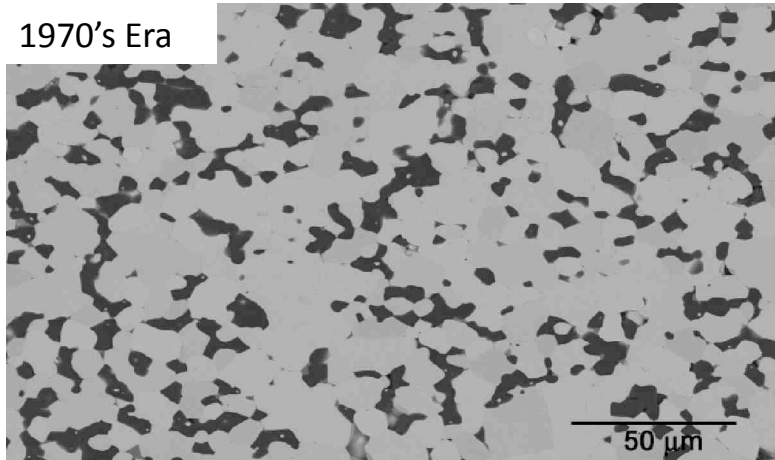


Granulated HfB_2/SiC Powder

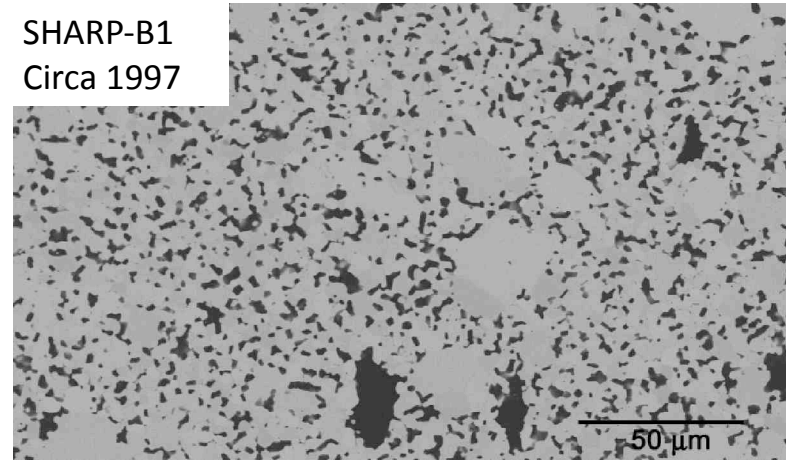


Early HfB₂ - 20% SiC Materials

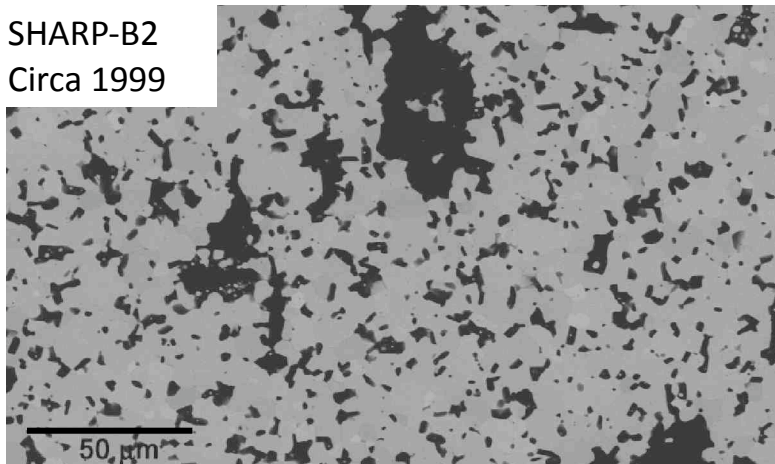
1970's Era



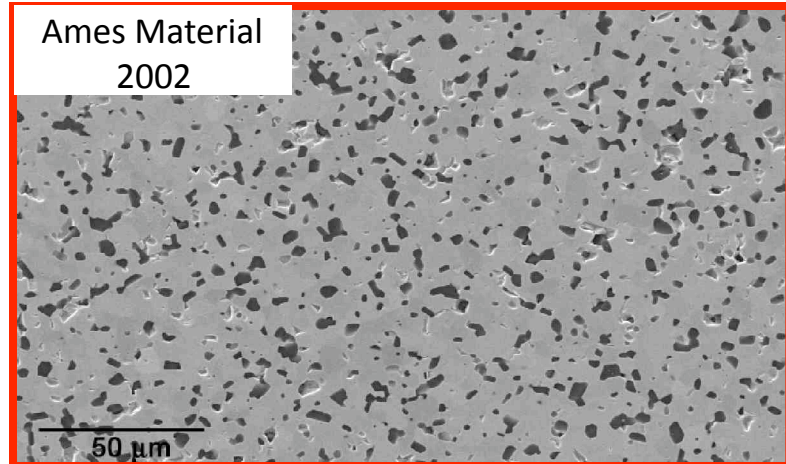
SHARP-B1
Circa 1997



SHARP-B2
Circa 1999



Ames Material
2002



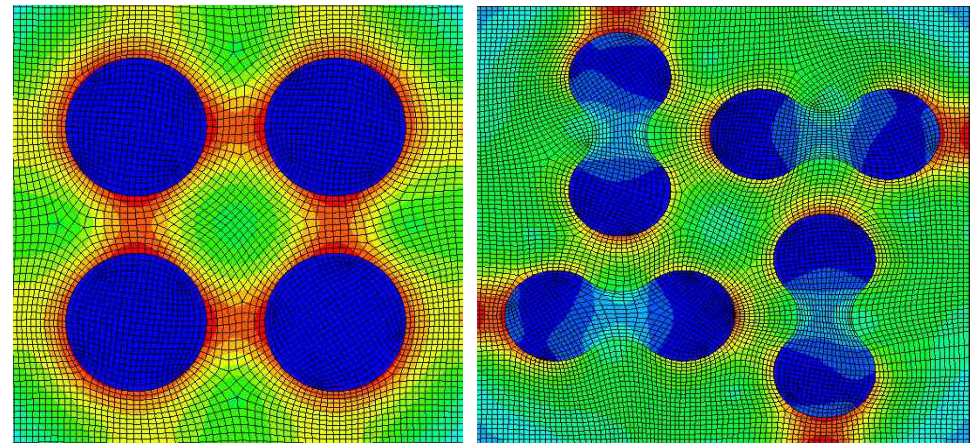
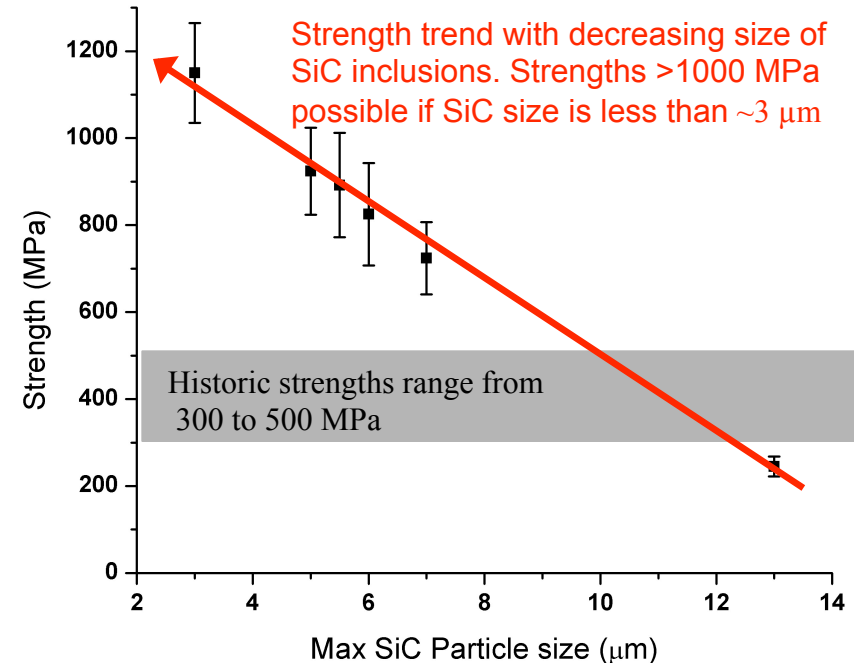
- Early and SHARP materials made by an outside vendor
- Improvements in powder handling provide a more uniform microstructure

Understand what you are testing!

UHTCs with Improved Strength (MS&T)

- Historic strengths were modest
~415 MPa reported in 1971*
ZrB₂ grain size ~10 μm
- Strengths higher for recent materials
>1000 MPa for ZrB₂-30 vol% SiC
Control of microstructure is key
ZrB₂ 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 ZrB₂ and SiC
Compressive in SiC
Tensile in ZrB₂ matrix
- Residual stresses affected by size and shape of SiC inclusions

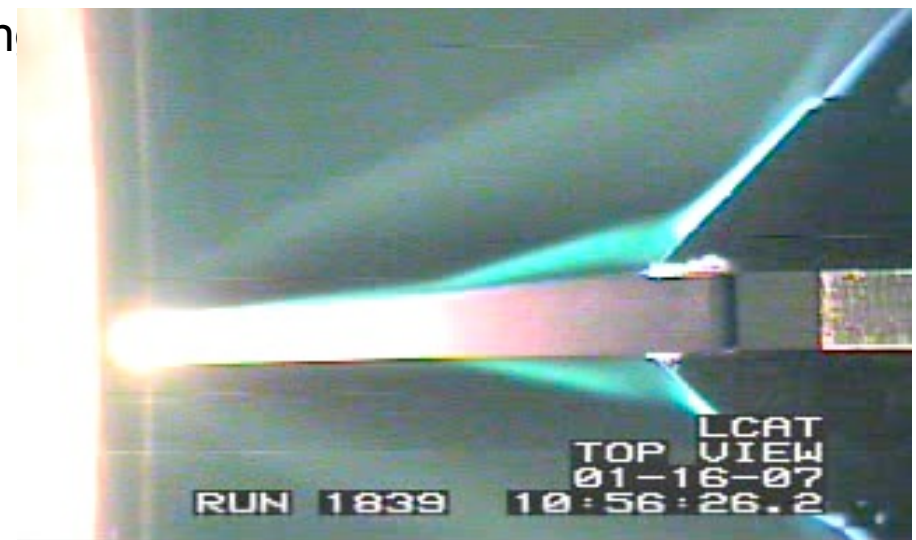
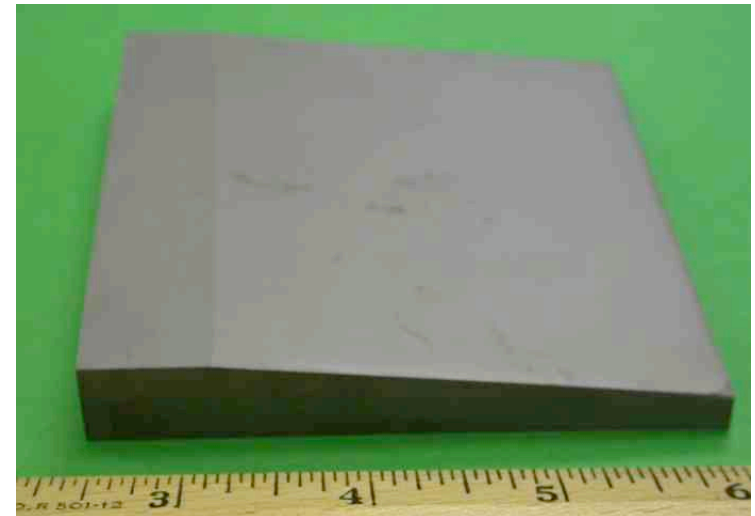
* J.R. Fenter, "Refractory Diborides as Engineering Materials," SAMPE Quarterly, **2**, 1-15 (1971).



Courtesy Missouri S&T

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





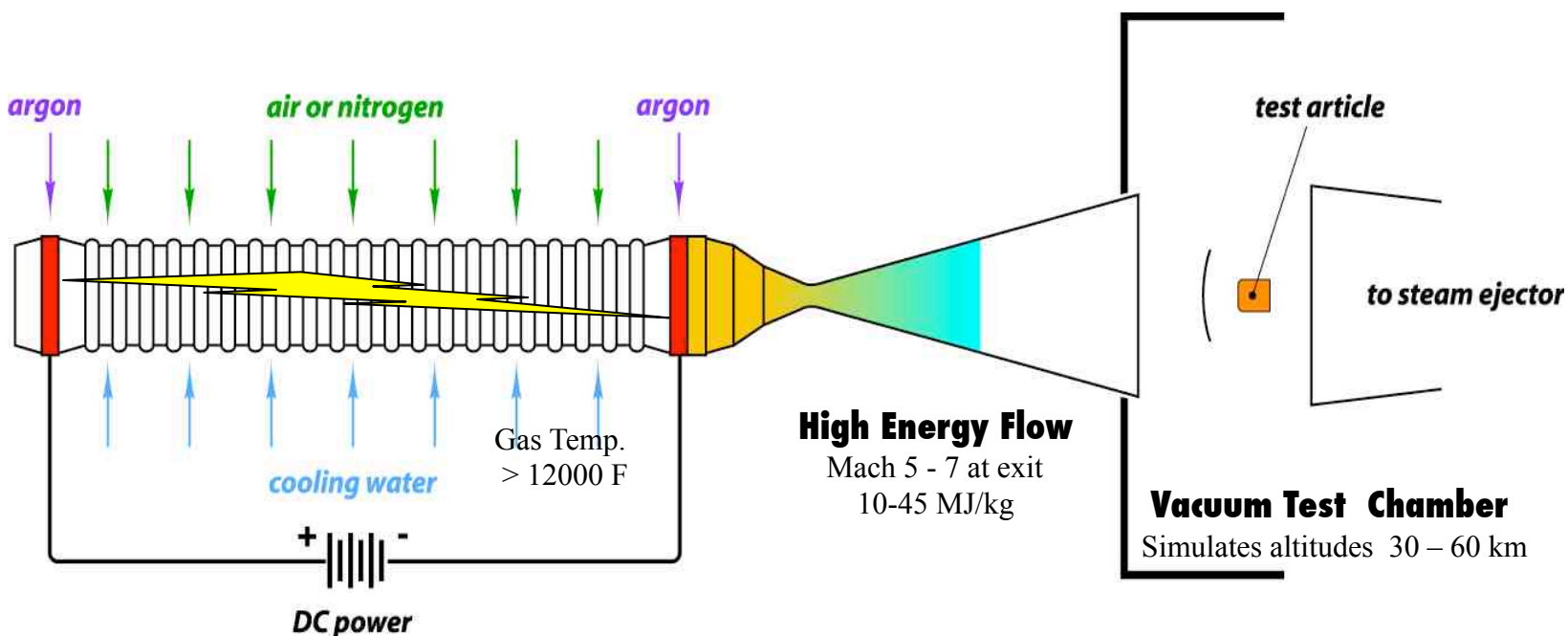
Need for Arc Jet Testing

- 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 SiO₂ layer is removed as SiO



Arc Jet Schematic

Simulates reentry conditions in a ground-based facility



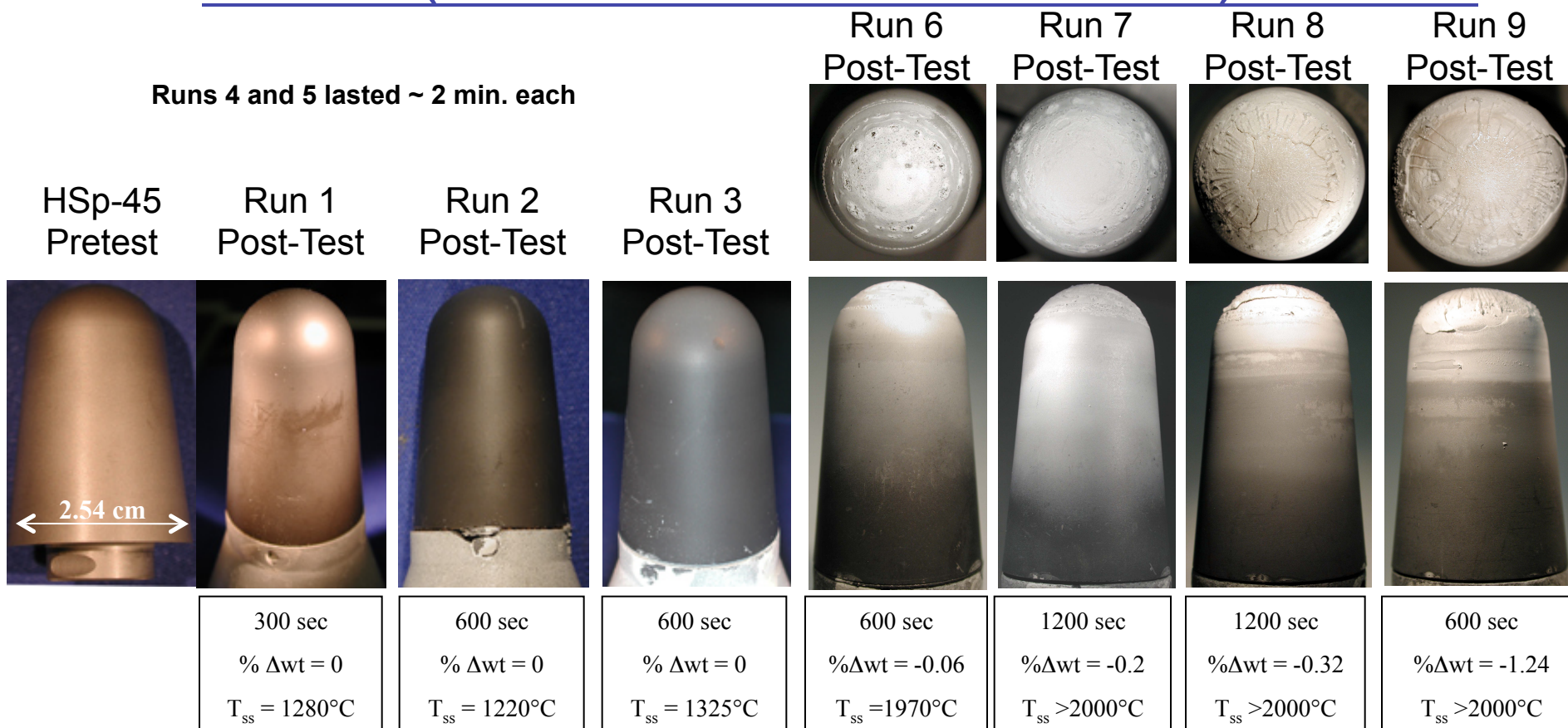
Method: Heat a test gas (air) to plasma temperatures by an electric arc, then accelerate into a vacuum chamber and onto a stationary test article

Stine, H.A.; Sheppard, C.E.; Watson, V.R. Electric Arc Apparatus. U.S. Patent 3,360,988, January 2, 1968.



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

Runs 4 and 5 lasted ~ 2 min. each



Increasing heat flux



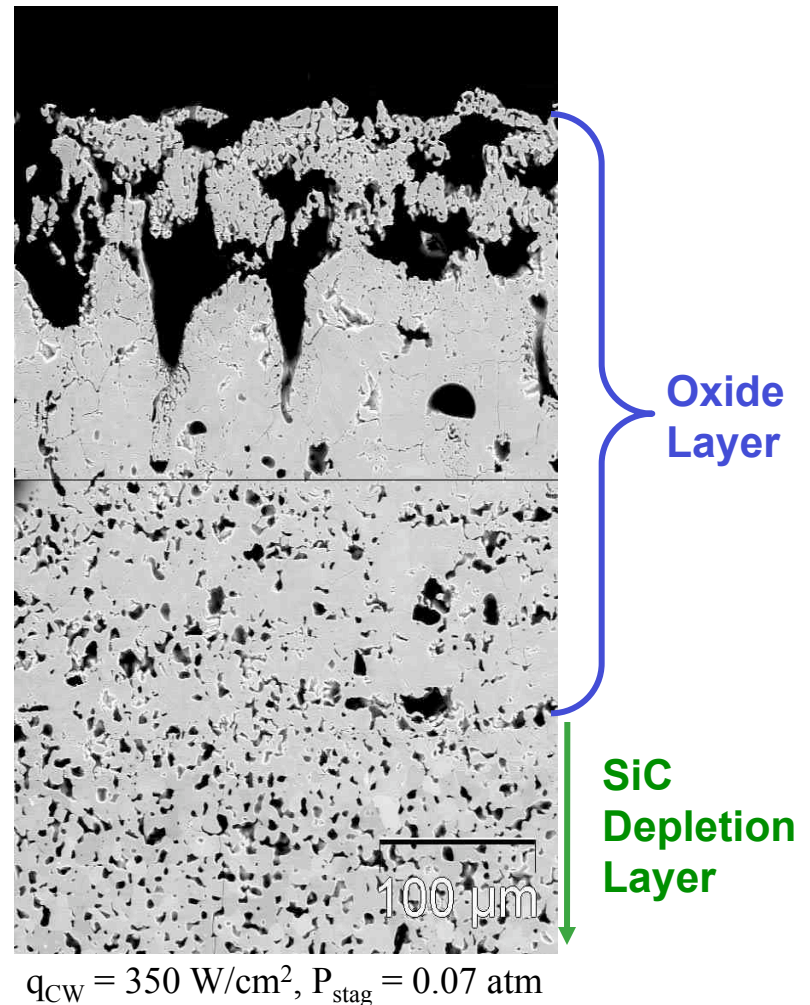


Reducing Oxide Formation



* 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 $200\text{W}/\text{cm}^2$

- 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



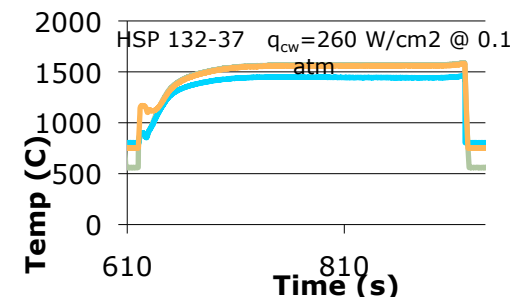
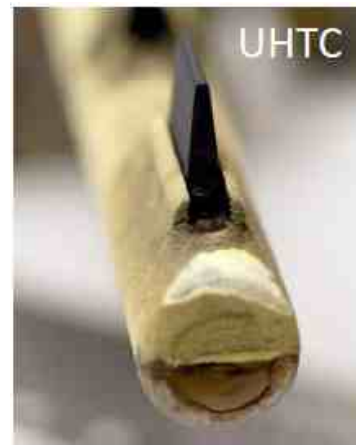
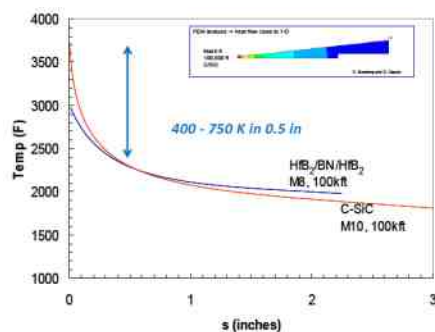
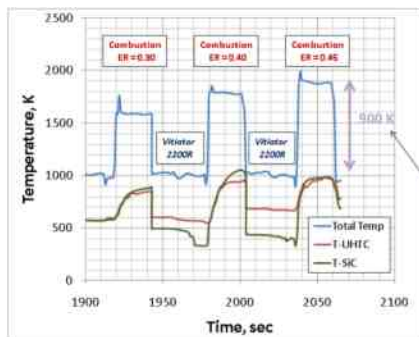
*Arc jet test data from Space Launch Initiative program

UHTC Evaluation under Service-Relevant Conditions

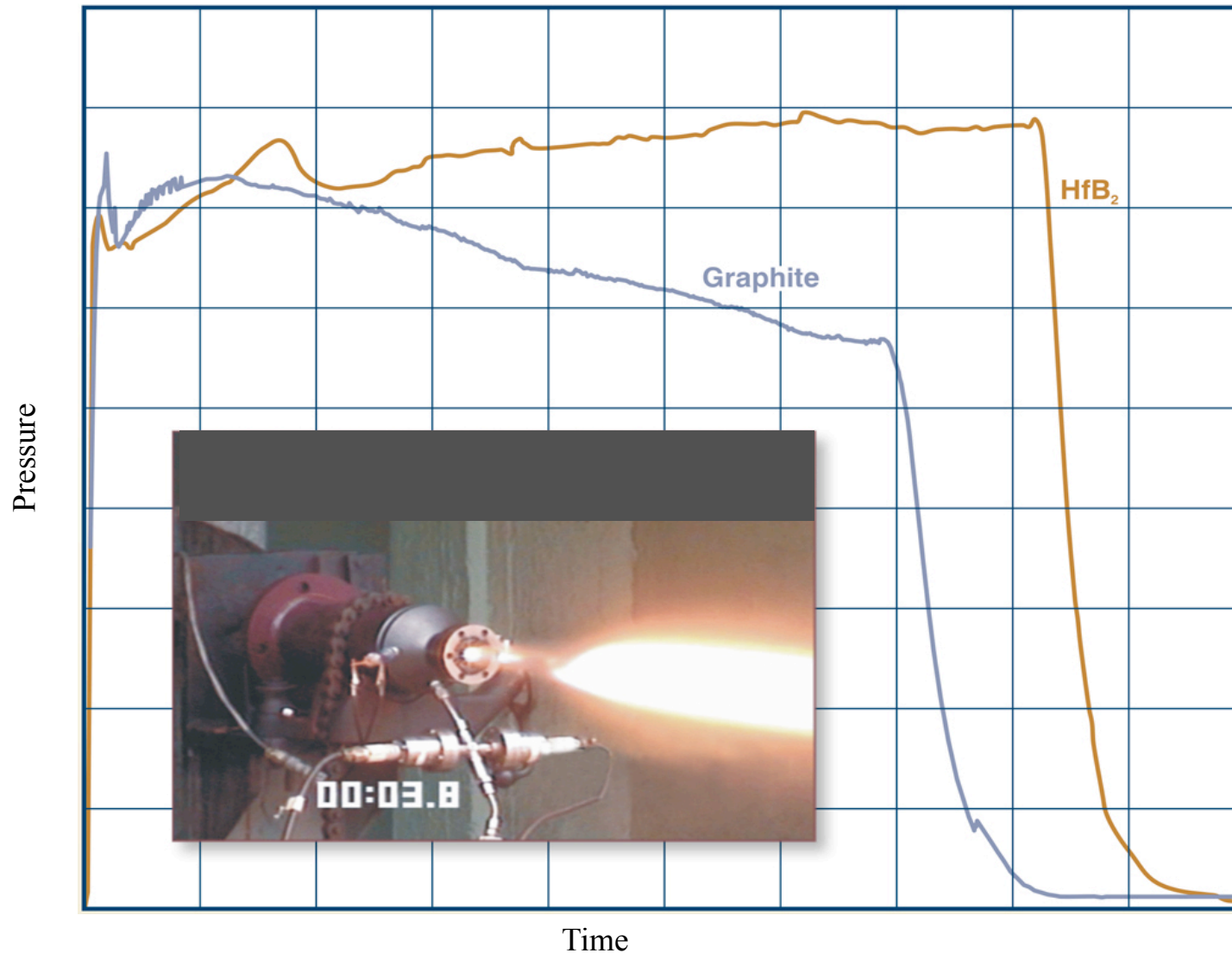


Understand behavior of UHTCs in hypersonic environments

- 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 HfB₂ Throats Showing Non-eroding Behavior of Ceramic



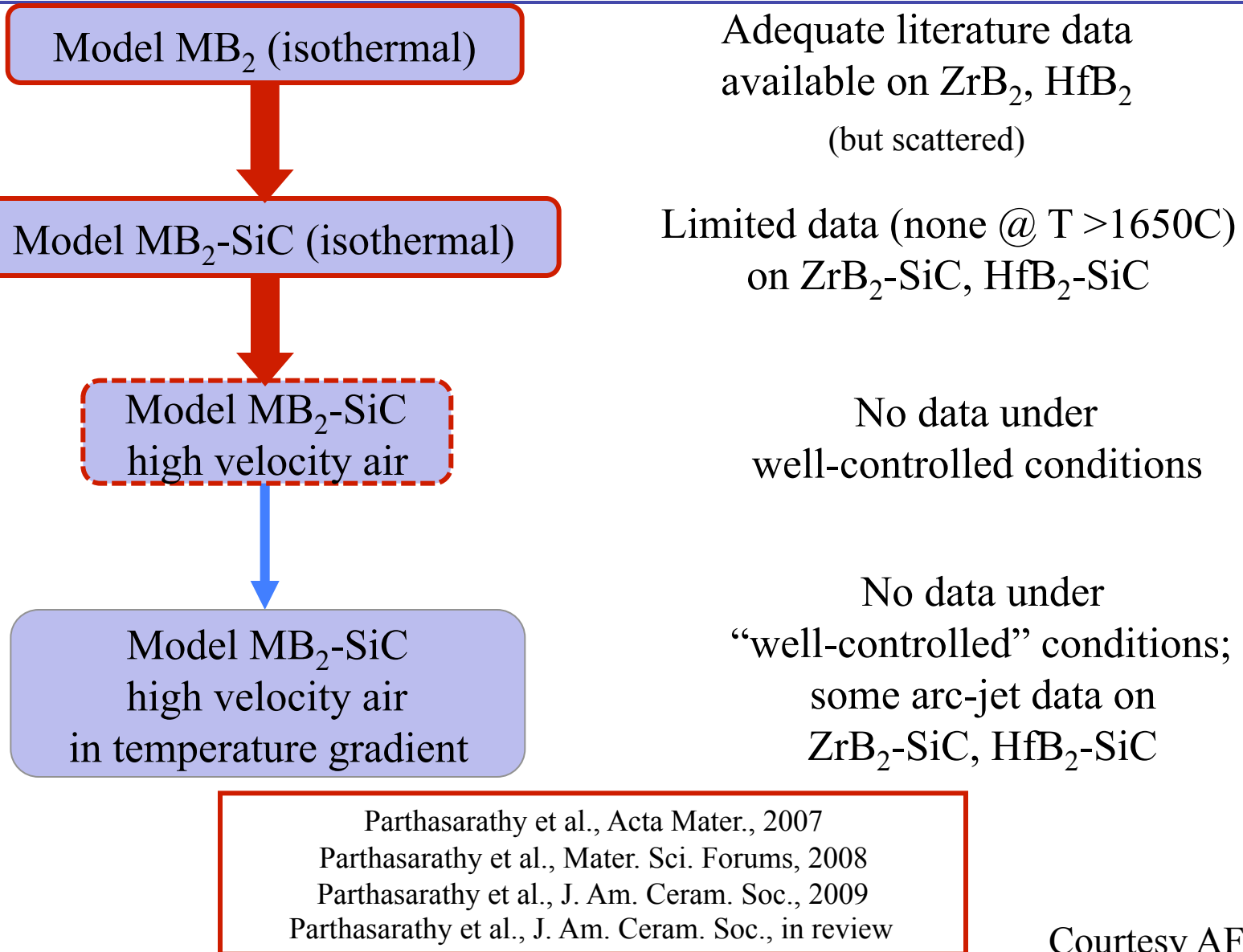


What About Active Oxidation?

- 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



Courtesy AFRL

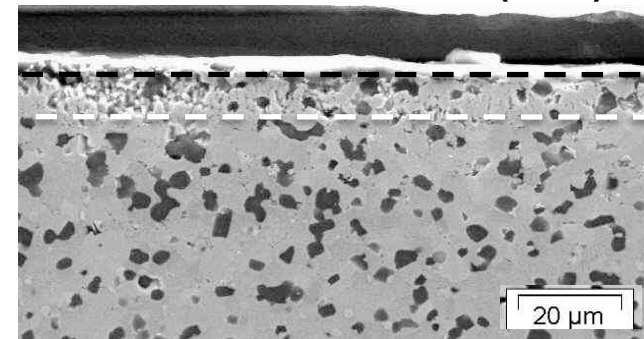
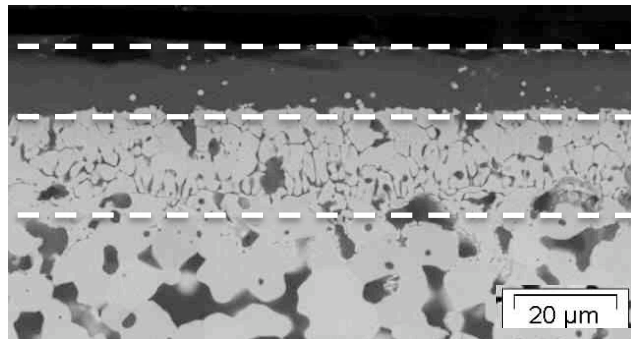


Arcjet Characterization: Additives & Influence of Microstructure

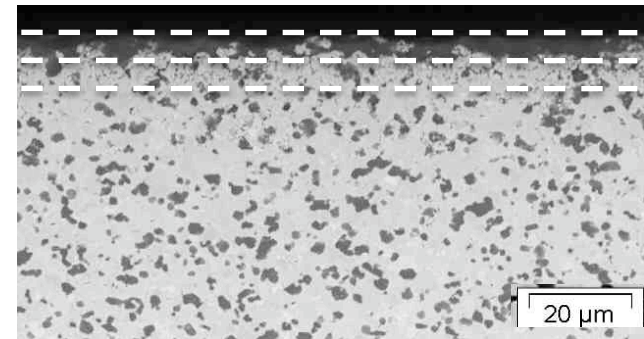
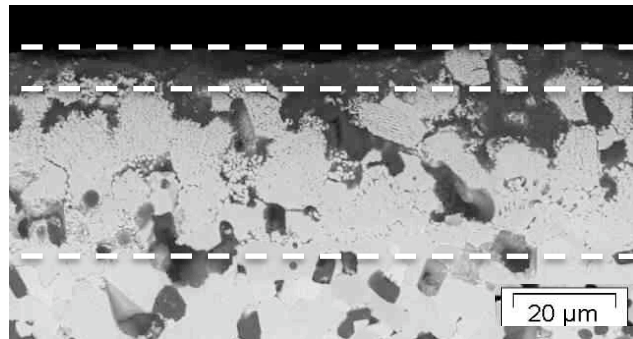
Hot Pressed

Field Assist Sintered (FAS)

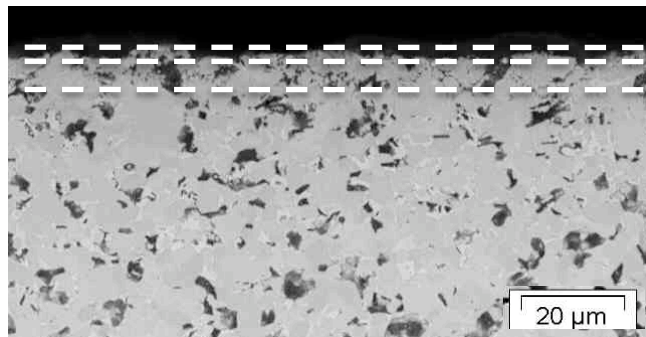
HfB₂-SiC
Baseline



HfB₂-SiC-TaSi₂



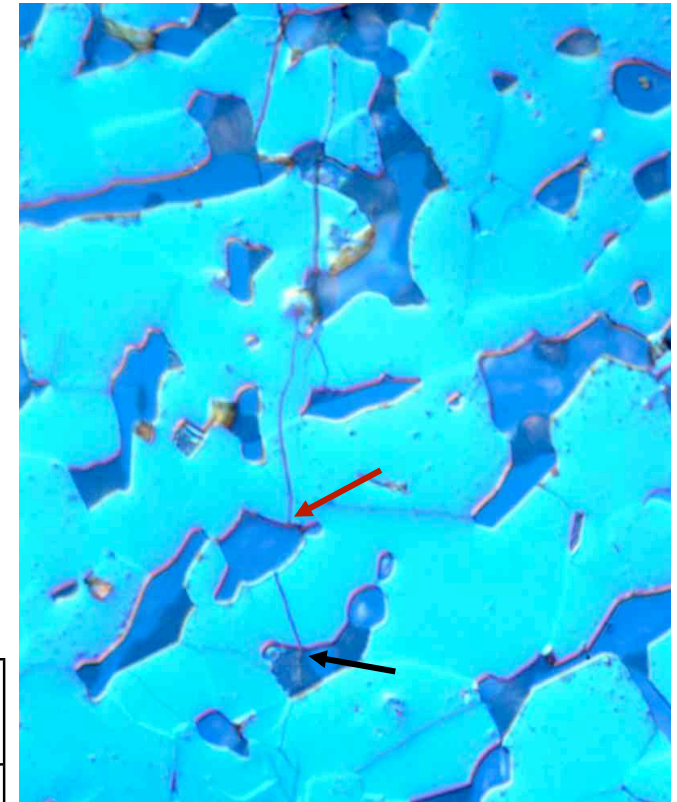
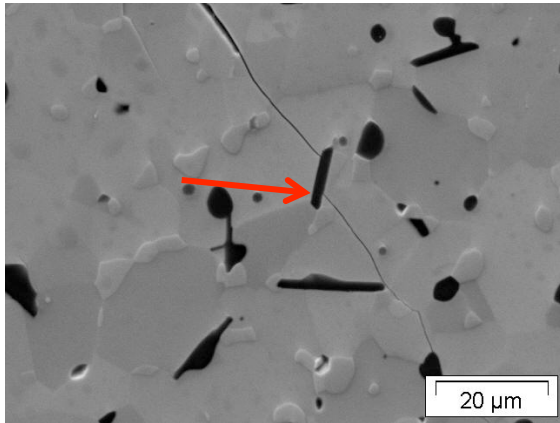
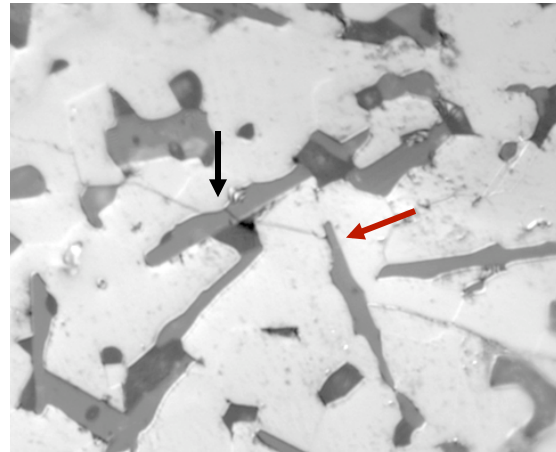
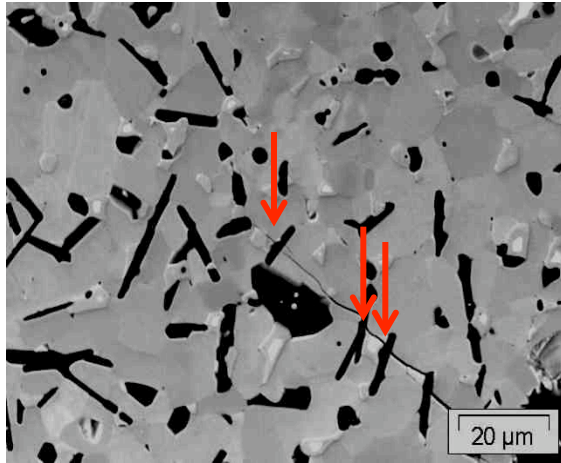
HfB₂-SiC-
TaSi₂-Ir



Both oxide scale and
depletion zone can be
reduced.



In Situ Composite for Improved Fracture Toughness



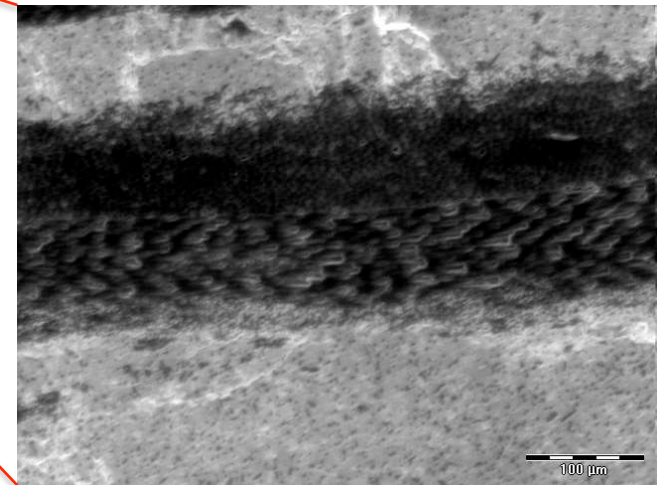
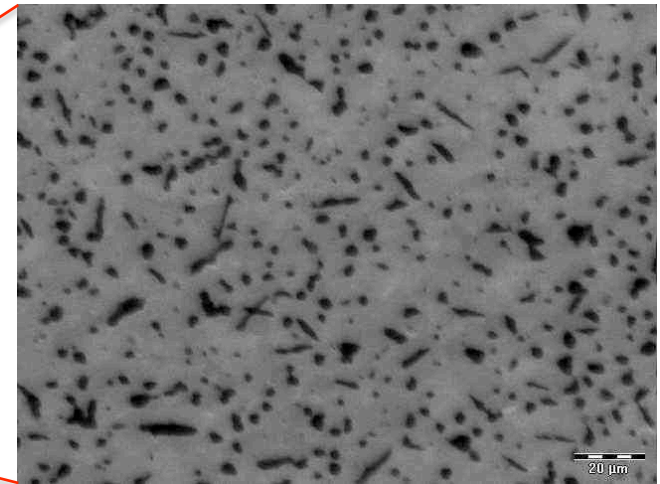
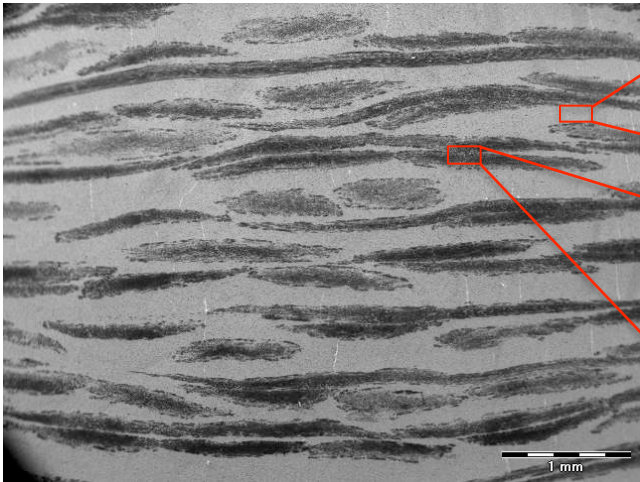
Oak Ridge National Laboratory

SiC Content	Fracture Toughness (MPam ^{1/2})
5%	3.61
10%	4.06
15%	4.47
Baseline UHTC (20%)	4.33

Evidence of crack growth along HfB₂-SiC interface, with possible SiC grain bridging



Ultra High Temperature Continuous Fiber Composites

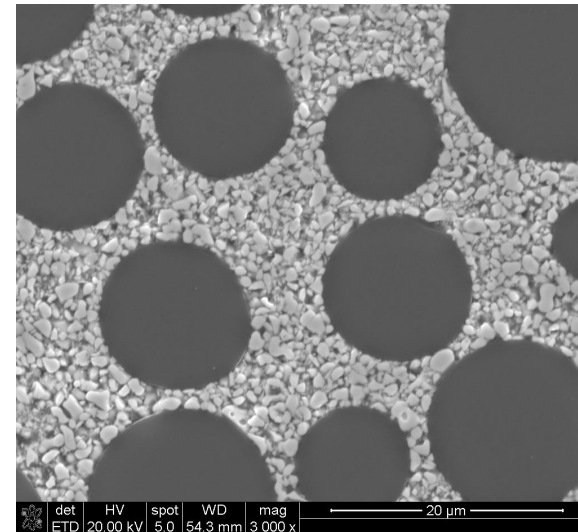
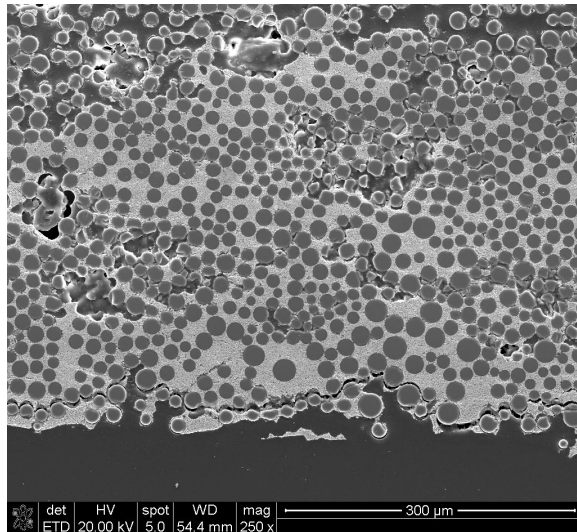


- Image at top right shows dense UHTC matrix with indications of high aspect ratio SiC.
- Image at bottom right shows the presence of C fibers after processing.



UHTC Composite Processing Preliminary Studies (AFRL)

- Nicalon SiC fiber
- HfB₂-filled SMP-10 (SiC precursor) slurry
- Tape wound infiltrated tows
- Unidirectional composite panels

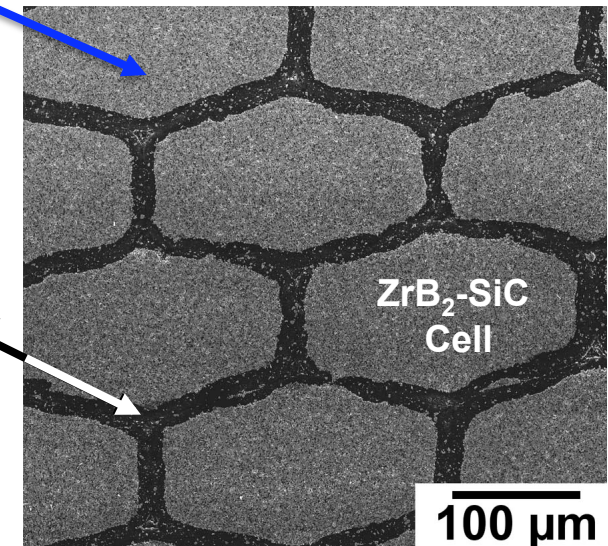
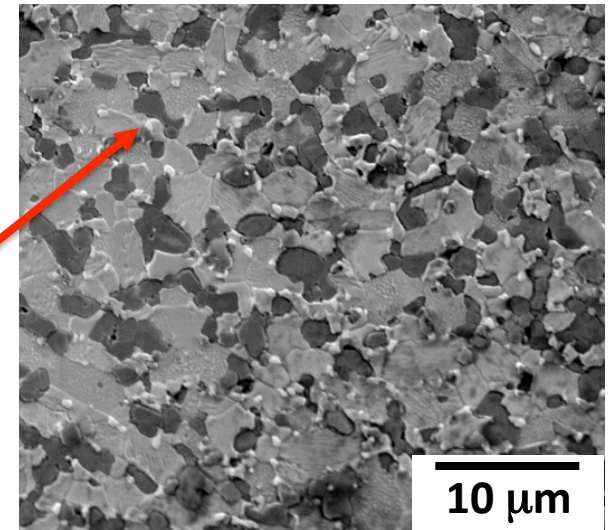
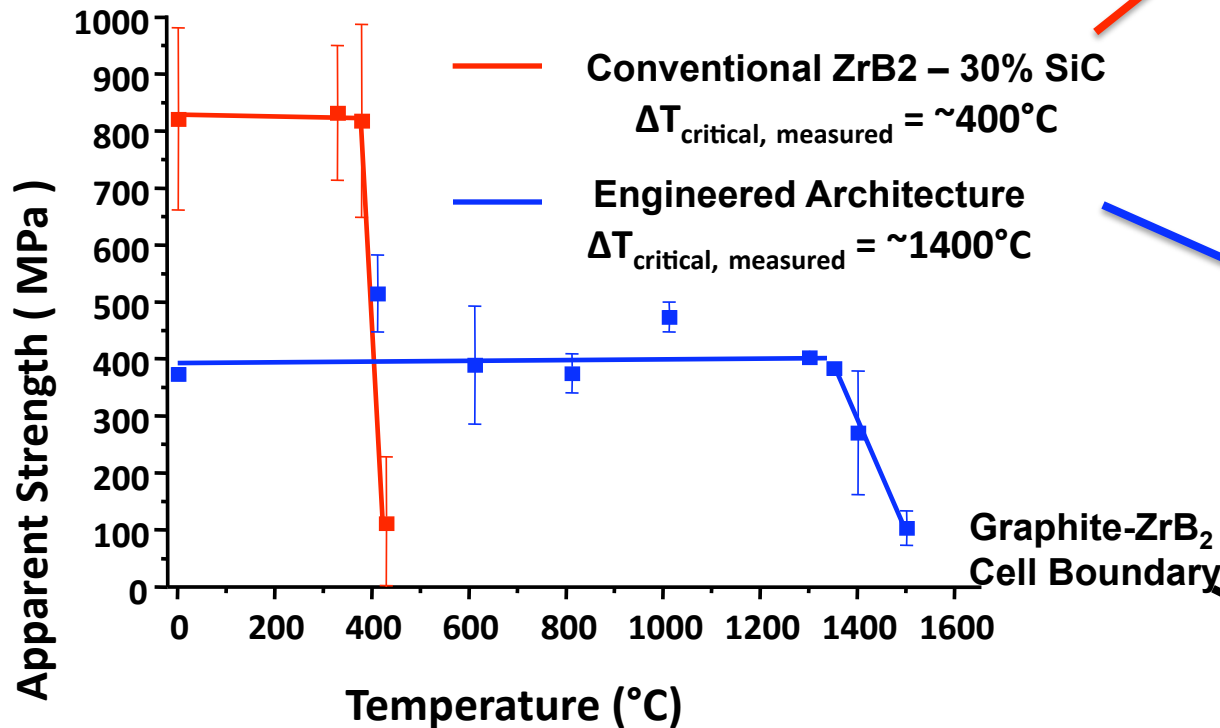


SiC_f/HfB₂-SiC Composites

Courtesy AFRL

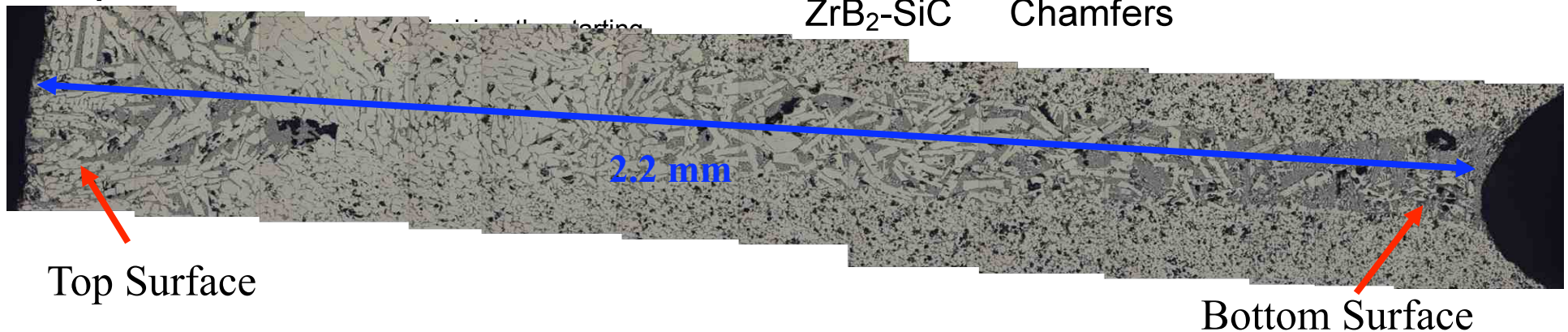
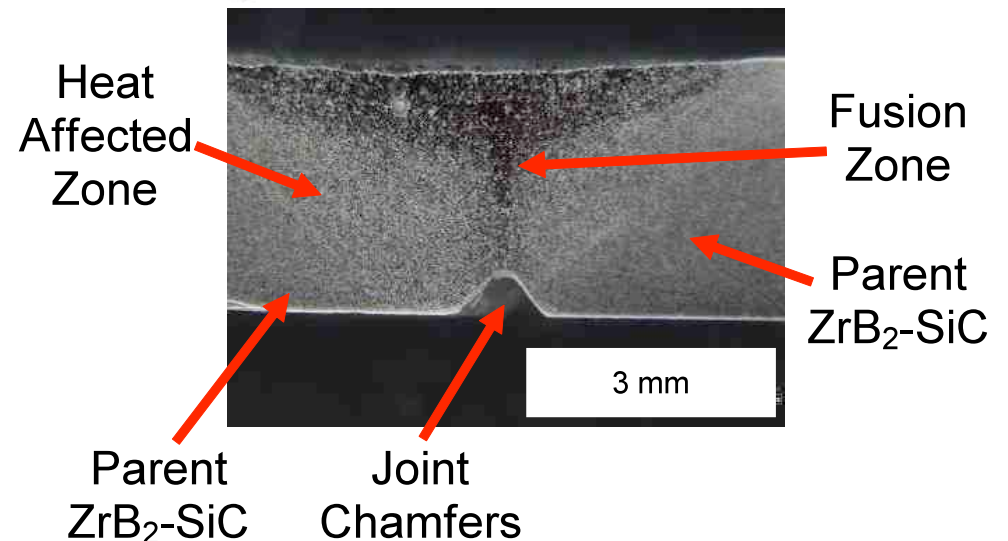
Engineered Architectures for Improved Thermal Shock Resistance at MS&T

- Thermal stress resistance can be improved
 $\Delta T_{\text{critical}} \sim 400^\circ\text{C}$ for conventional ceramic
Improves to $\sim 1400^\circ\text{C}$ for cellular architecture
- Fiber reinforcement could produce additional gains



Arc Welding of UHTCs (MS&T)

- ZrB₂-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





Modeling of UHTCs Will Enhance Development

Goals

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

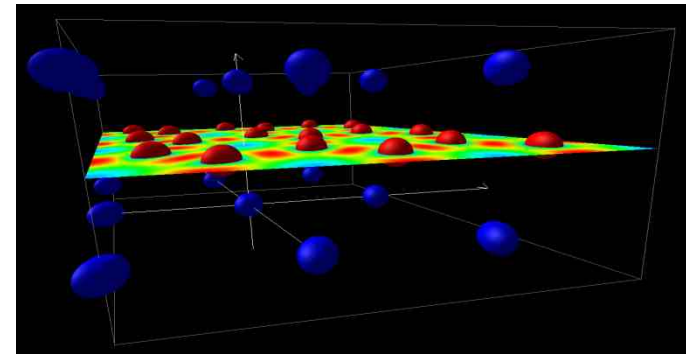
Approach

- Develop models integrated across various length scales
- Correlate models with experiment whenever possible

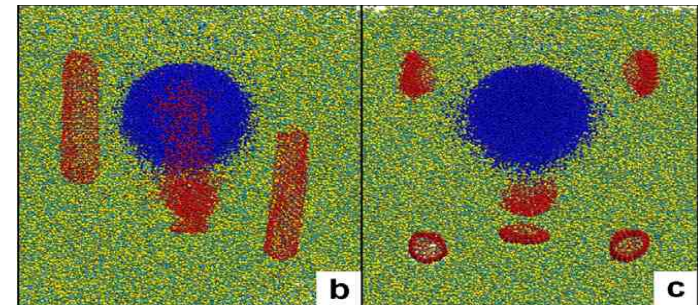


Multiscale Modeling of Materials

- **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.



Lawson, publication in preparation (2010)



Makeev, Sundaresh, and Srivastava, J. Appl. Phys. 106, 014311 (2009)



Lewis and Geltmacher, Scripta Materialia 55 (2006)



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

ZrB₂ Based Ceramics	Catalytic Properties of UHTCs
Missouri University of Science & Technology	PROMES-CNRS Laboratory, France
US Air Force Research Lab (AFRL)	CNR-ISTEC
NASA Ames & NASA Glenn Research Centers	CIRA, Capua, Italy
University of Illinois at Urbana-Champaign	SRI International, California
Harbin Institute of Technology, China	
Naval Surface Warfare Center (NSWC)	Imaging and Analysis (Modeling)
NIMS, Tsukuba, Japan	University of Connecticut
Imperial College, London, UK	AFRL
Korea Institute of Materials Science	NASA Ames Research Center
CNR-ISTEC	Teledyne (NHSC-Materials and Structures)
	Oxidation of UHTCs
HfB₂ Based Ceramics	AFRL
NASA Ames Research Center	NASA Glenn Research Center
NSWC—Carderock Division	Georgia Institute of Technology
Universidad de Extramadura, Badajoz, Spain	Missouri University of Science & Technology
CNR-ISTEC, Italy	Texas A & M University
	CNR-ISTEC, Italy
Fiber Reinforced UHTCs	
Chinese Academy of Sciences, Shenyang	University of Michigan, Ann Arbor, Michigan
University of Arizona	NSWC—Carderock
MATECH/GSM Inc., California	Harbin Institute of Technology, China
AFRL	University of Illinois at Urbana-Champaign

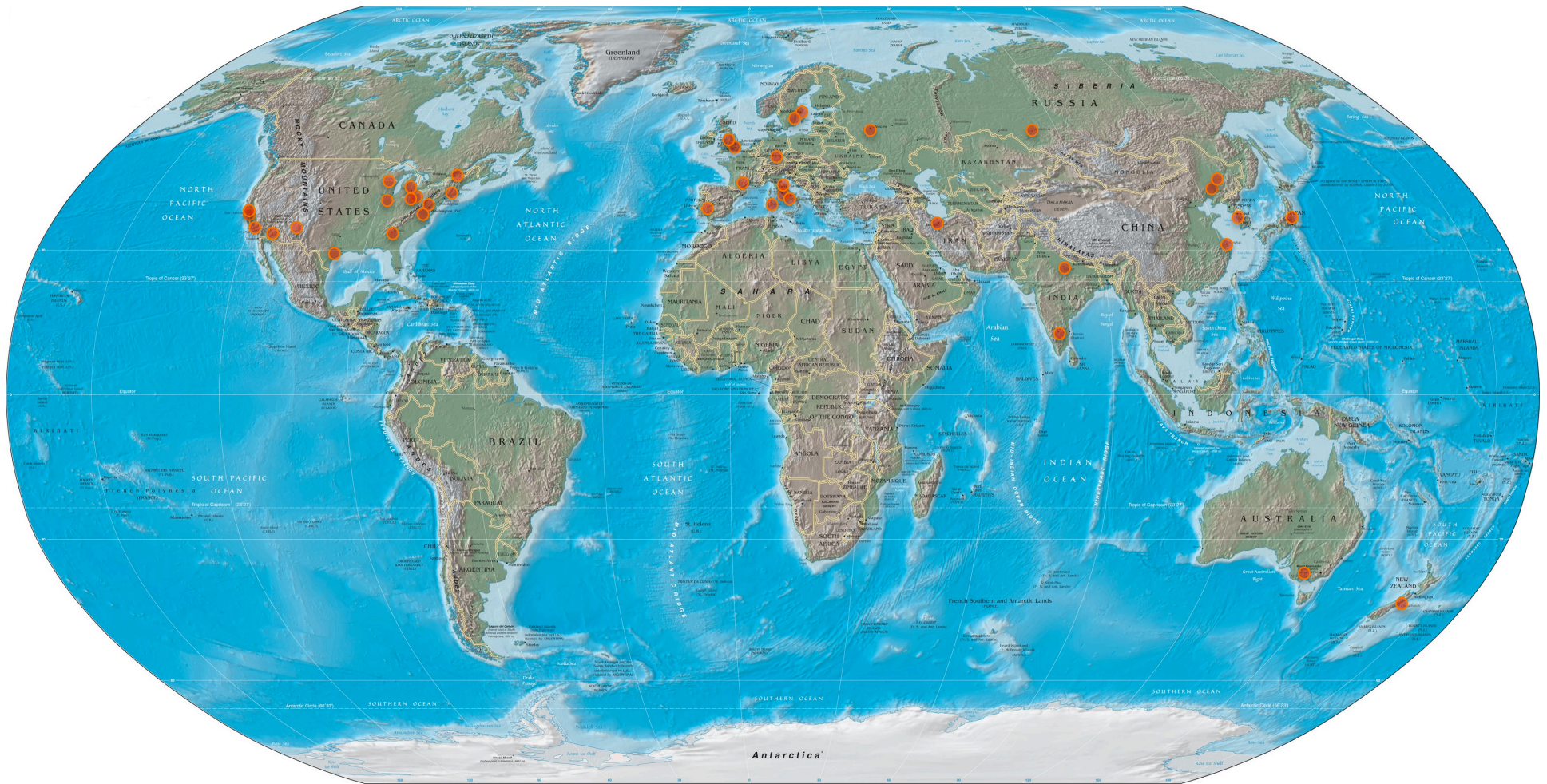


Some Recent Research Efforts in UHTCs: Processing

Field Assisted Sintering	UHTC Polymeric Precursors
University of California, Davis	SRI International, California
Air Force Research Laboratory (AFRL)	University of Pennsylvania
CNR-ISTEC, Italy	Missouri University of Science & Technology
Stockholm University, Sweden	MATECH/GSM Inc., California
NIMS, Tsukuba, Japan	Teledyne (NHSC)
Pressureless Sintering	Technische Universität Darmstadt, Germany
Missouri University of Science & Technology	Nano & Sol Gel Synthesis of UHTCs
Politecnico di Torino, Italy	Loughborough University, U.K.
Reactive Hot-Pressing	IGIC, Russian Academy of Science
Shanghai Institute of Ceramics, China	University of Erlangen-Nürnberg, Germany
NASA Ames Research Center	Korea Institute of Materials Science
National Aerospace Laboratories, India	Iran University of Science and Technology
Sandia National Laboratories, New Mexico	
McGill University, Montreal, Canada	
University of Erlangen-Nürnberg, Germany	



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! 52