

UHTCs: Ultra-High Temperature Ceramic Materials for Extreme Environment Applications

by E. Wuchina, E. Opila, M. Opeka, W. Fahrenholtz, and I. Talmy

3000°C That's not just hot – it's EXTREMELY hot. It is above the melting or decomposition temperatures for most of the materials known to man. But in the world of extreme environment engineering, it is just a baseline. The list of materials with melting temperatures above 3000°C is limited to perhaps 15 elements or compounds (Table I). That is a pretty small palette of materials to draw from for an engineer or designer.

Table I. Materials with melting temperatures above 3000°C.

Carbon	TaB ₂	Re
W	HfC	BN
HfB ₂	HfN	ZrC
TaC	ZrB ₂	TiC
TaN	NbC	ThO ₂

Since the late 1960s, the world of high-temperature materials has focused primarily on SiC and Si₃N₄ as the materials of choice. Entire industries have developed to produce ball bearings, armor, fibers, and even turbine blades. But recently, there has been a revival of sorts in materials originally studied in the 1960s for potential aerospace applications, driven by “the need for speed”—with new propulsion and hypersonics concepts as shown in Fig. 1. Current increasing interest in hypersonic vehicles and weapons points to the need for new ultra-high-temperature materials for wing leading edges and nosetips, as well as propulsion system components.

There are more than 300 materials with melting temperatures over 2000°C, including the aforementioned SiC, refractory metals (Hf, Nb, Ir, Re, Ta, W), oxides (HfO₂, ZrO₂, UO₂, ThO₂), a variety of transition metal carbides, nitrides, and borides as well as other compounds. For real engineering applications, though, melting temperature is only one of many of properties used in the materials selection process.³ As most engine and hypersonic leading edge applications will involve exposure to oxidizing fuels or aeroheating, all nonoxide materials will undergo oxidation to form some combination of solid, liquid, or gaseous reaction product. It is the oxidation behavior that is a second primary property associated with the materials selection process. Strength (room temperature and at application temperature), thermal conductivity,



Fig. 1. Conceptualized drawings of hypersonic vehicle¹ and missile² as potential applications for UHTC leading edge and nozzle components.

thermal expansion, density, fabricability, and cost are also important factors in determining the optimal material for a given application.

For the purposes of this paper, we will simply define UHTC materials by their usefulness in a real structural (load-bearing) application where the very high temperatures are generated rapidly by burning fuels or friction with the atmosphere (not steady state). This will quickly eliminate most of the materials mentioned above. While oxides are reasonable to consider for use in oxidizing environments, poor thermal shock resistance due to high thermal expansion and low thermal conductivity eliminates them from further discussion. The silicon based refractory compounds (SiC, Si₃N₄, MoSi₂, etc.) possess excellent oxidation resistance up to 1700°C due to the formation of a layer of SiO₂ glass that inhibits oxygen diffusion to the parent material.⁴ This is the primary reason for the popularity of these materials for a wide variety of applications. However, active oxidation (the direct formation of

SiO(g) instead of a protective SiO₂ layer) can occur at very high temperatures (> 1350°C, depending on P_{O2}) and reduced system pressures. In addition, decomposition of already-formed SiO₂, or the interface reaction between SiC and SiO₂ results in SiO(g) formation at high temperatures and reduced pressure environments. Other materials, such as TiB₂, TiC, NbB₂, NbC, while having high melting temperatures, form oxides with low melting points (TiO₂ – T_m = 1840°C and Nb₂O₅ – T_m = 1485°C). Graphite has the highest melting temperature of any material known, but starts to burn at 800°C. While it is a most widely used material in high-temperature applications, it must be protected by coatings for long-term use.

What we are left with are the borides, carbides, and nitrides of the Group IV & V elements, as well as mixtures based on these compounds. While these materials of interest have been known since the 1930s, it wasn't until the seminal works sponsored by the U.S. Air Force⁶ as well as the work of Samsonov⁷ in the 1960s that the class of UHTC materials became more widely known. As the ZrB₂ and HfB₂-based UHTCs are the most widely studied of these materials due to their good oxidation resistance from room temperature to over 2000°C, we will start by discussing them.

Borides

It is well known that Zr and Hf are very chemically similar elements, with their primary differences being density and nuclear capture cross section, so it is not surprising that the borides of these elements are also very similar. Both exist in hexagonal crystal structures of the AlB₂ prototype, with layers of B atoms in 2D graphite-like rings, alternating with Zr or Hf layers in a hexagonally close-packed array. Because of the very high strengths of the B-B rings and M-B bonds, these materials have very high hardness and temperature stability. While most extrinsic properties such as strength are dependent on processing and microstructure, the intrinsic thermal conductivities of the diborides are very high, approaching copper at room temperature, with little dropoff up to 2500°C. This makes ZrB₂ and HfB₂ very appealing for applications where thermal stress response is an important issue. A good example of this is rocket motor nozzles. In these applications, the temperature rise on the inside surface can approach 2000°C in less than 0.15

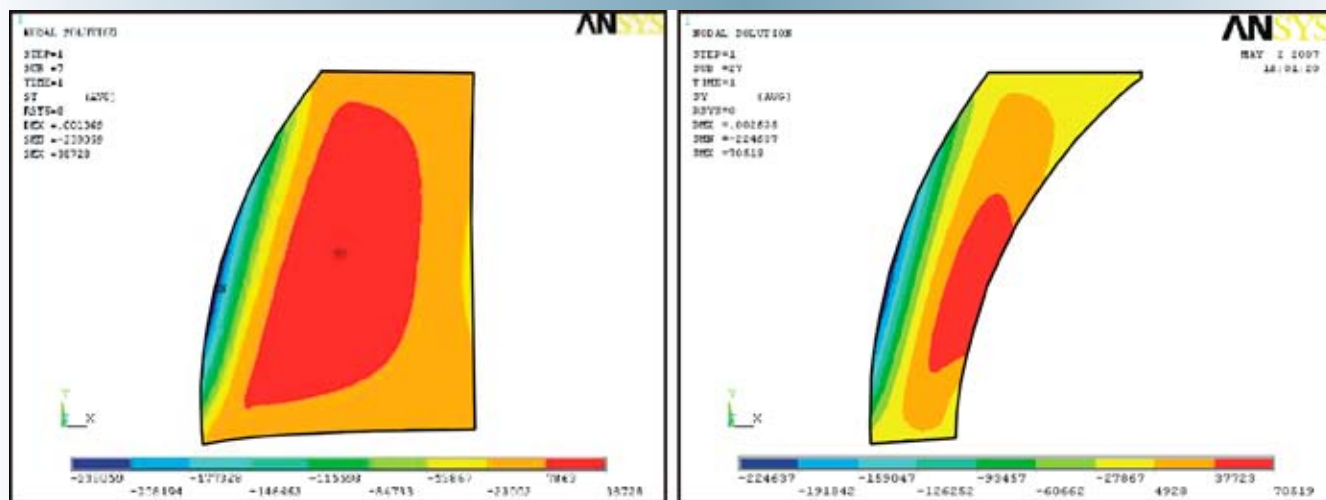


Fig. 2. Finite element modeling results showing the peak tensile stresses generated in rocket motor nozzles during the early stages of firing.⁸

seconds, while the outer wall is still at room temperature. This ΔT generates a compressive stress on the inside (hot) surface, while the outside (cool) surface carries a tensile stress. Depending on the thickness of the nozzle, the thermal conductivity and strength will determine the success or failure of the part. A finite element model of thick and thin-walled cross-section (Fig. 2) shows the highest tensile loads in red.⁸ With higher thermal conductivity, the borides can more readily transmit the heat through the part and equilibrate the temperature within the cross-section, thereby reducing the thermal stress.

As mentioned previously, the strength of materials is dependent on

processing and microstructure (as well as test methods used to measure the property). It is not surprising, then, that the strength values reported in the literature have a wide range. Because ZrB_2 and HfB_2 are hexagonal crystals, the properties exhibit anisotropic behavior within individual crystals. However, the bulk data reported are traditionally gathered from polycrystalline ceramics, and therefore do not show anisotropic behavior. As is common in ceramic systems, the strength of the diborides is generally shown to increase with decreasing grain size according to the Hall-Petch relationship. The influence of second-phase additions has also been reported. Silicon carbide was originally incorporated into HfB_2 and ZrB_2 by

Cougherty, et al.,⁹ in the 1960s as a grain refiner to improve its strength. More recent second phase additions include carbides and silicides. While these additions also influence the strength of the diboride ceramics, the primary interest of research on ZrB_2 and HfB_2 has primarily focused on improving their oxidation resistance.

The presence of a silica-forming species in the diborides greatly increased their oxidation resistance due to the in-situ formation of a borosilicate glass on the surface of the material, which impeded oxygen diffusion to the parent material. It was found that an optimal composition of ZrB_2 plus 20 to 30 vol% SiC, produced the highest oxidation resistance up

to 2000°C.^{10,11} Pure diborides form liquid B_2O_3 during oxidation that can be protective up to around 1200°C, but the B_2O_3 evaporates at higher temperatures and no longer provides a diffusion barrier. The addition of the SiO_2 -forming species helps increase the oxidation resistance by “tying-up” the boron by the formation of a borosilicate glass that can be stable up to at least 1600°C. A cross-section of a ZrB_2 -SiC material oxidized at 1627°C for ten 10-minute cycles in air is shown in Fig. 3.¹² Efforts to model this oxidation behavior and the complex porous ZrO_2 -glass scale are being undertaken by a variety of

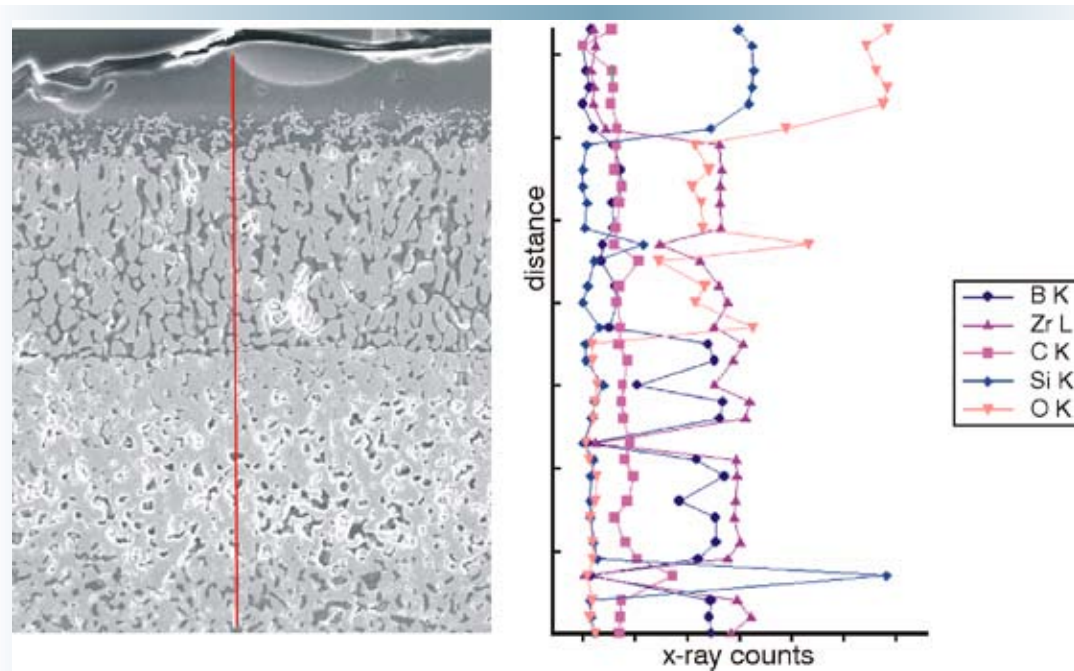


Fig. 3. Scanning electron micrograph and composition profile of a cross section of ZrB_2 -SiC material after ten 10-minute cycles at 1627°C in air.¹²

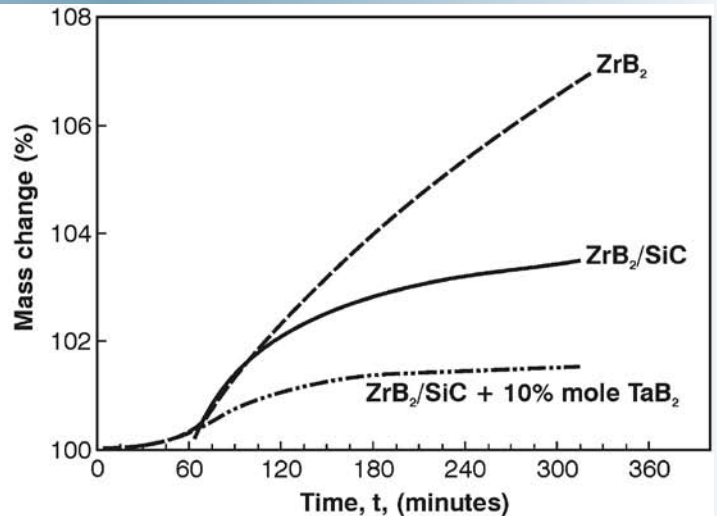
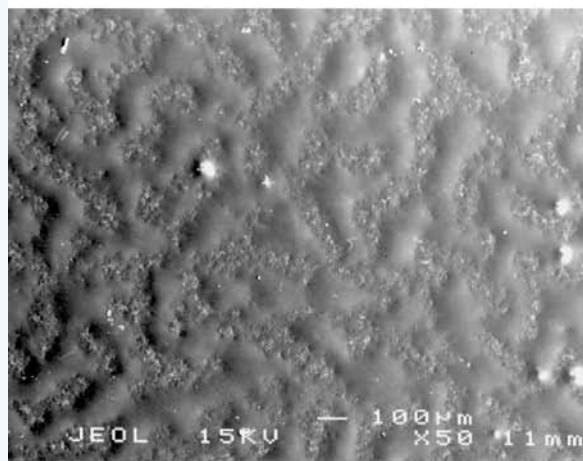


Fig. 4. Scanning electron micrographs of top surface of ZrB₂-SiC modified with TaB₂ oxidized at 1300°C in air.¹⁴

researchers¹³ and will hopefully lead to further improvements in oxidation resistance. Hypersonic aerosurfaces will need to have lifetimes on the order of hundreds to thousands of hours to become cost-effective on reusable structures. One avenue to further increase the oxidation resistance has been reported by Talmy and Zaykoski.¹⁴ This involves the addition of transition-metal elements in the form of borides or silicides to produce complex immiscible

glasses during oxidation to further reduce the oxygen diffusion rates through the glass. An example of the immiscible glass microstructure and a thermogravimetric oxidation curve showing the improvement of oxidation resistance is presented in Fig. 4.

Processing—While there has been a small amount of work describing the chemical vapor deposition of boride coatings,¹⁵ the vast majority of research on dense bulk boride materials has

been in the form of powder-processed materials. Most boride powders have been synthesized by reduction, chemical or reactive processes. Carbothermal reduction of ZrO₂ + B₂O₃ is the most commercially viable process to produce ZrB₂, but often leaves B₂O₃ and carbide impurities. Other economically feasible processes for powder production¹⁶ include self-propagating high-temperature synthesis¹⁷ from the pure elements. The highly exothermic nature

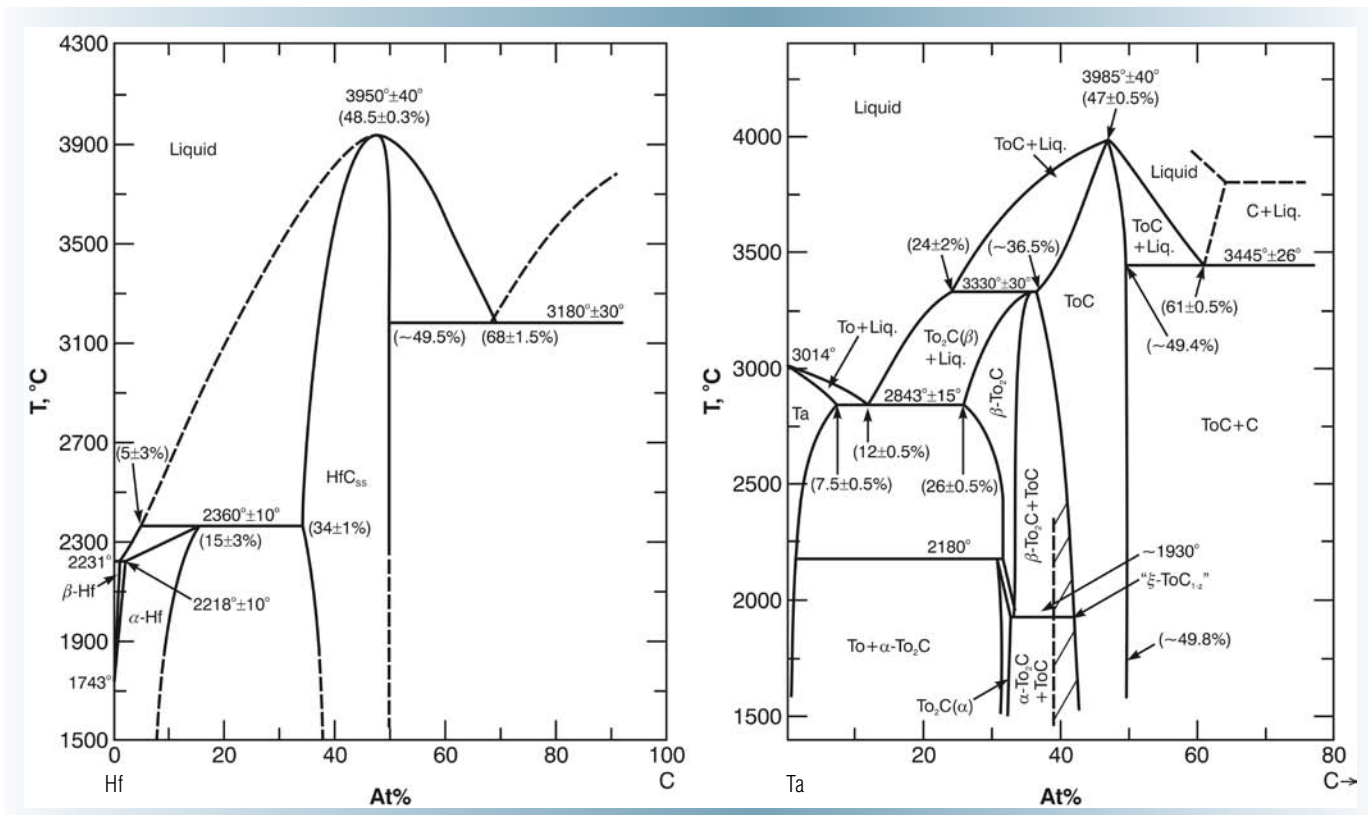


Fig. 5. The phase equilibrium diagrams for the Hf-C²⁴ and Ta-C²⁵ systems.

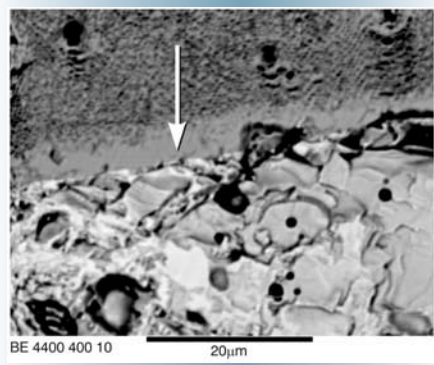


Fig. 6. SEM of HfC/HfO₂ interface showing evidence of oxycarbide interphase. (Arrow indicates interphase layer.)

of these reactions makes them difficult to control, but can result in highly defective, very small crystals that have been linked to improved sinterability.

Densification of these materials has traditionally involved uniaxially hot-pressing in graphite molds at temperatures above 1800°C. However, recent work¹⁸ on the use of spark-plasma to minimize grain growth during hot pressing has drawn interest, and the reports of pressureless sintering by groups in Italy and the U.S.^{19,20} could lead to a wider use of borides in engineering applications by lowering processing temperatures and increasing the size and shape of articles that can be produced commercially.

Carbides

The transition-metal carbides have also been considered as UHTC materials of interest due to their extremely high melting points—exceeding those of the aforementioned refractory borides. The monocarbides of Ta and Hf are of particular interest because they possess the highest melting temperatures of any compounds (3980 and 3928°C, respectively), as well as very high hardness and elastic modulus. Many of the monocarbides exist in the NaCl-type face-centered cubic crystal structure. The carbides are not as commonly studied as the borides due to the low-temperature “pestring” that occurs during oxidation, which will be described later. However, for applications where there is a rapid rise to temperatures above 2000°C, the carbides have become of considerable interest.

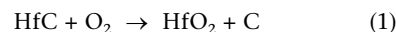
Many transition-metal carbides have potentially tailorable thermomechanical and thermophysical properties due to a wide phase stability field that allows a large number of vacancies to exist in the crystal lattice. The extremely high melting temperatures of TaC and HfC are due to their interatomic bonding, which while most researchers agree is a mixture of metallic, covalent, and ionic—the strongest component in the monocarbides has been determined to

be covalent.²¹ These highly covalent bonds not only affect the melting temperature, but contribute to the high elastic modulus of these materials as well. However, the precise nature of bonding in these compounds is not well understood, as discussed in these recent reviews.^{22,23}

As shown in Fig. 5, the phase diagrams for Hf-C²⁴ and Ta-C²⁵ show a wide homogeneity region for the stability of the monocarbides. While the chemical formula is generally written as MeC, MeC_x is more appropriate, where x is the C/Me ratio. The properties of the materials across this stability range will obviously be a function of the interaction between the dislocations and vacancies in the lattice. This interaction increases the Peierls stress (the force needed to move a dislocation through the lattice) and also changes the bonding character (reduced contribution of C atoms to cohesion by reducing the number of Me-C bonds that need to be broken during dislocation motion as well as reducing the strength of the Me-Me bond by increasing vacancy concentration) that will decrease the Peierls stress.²¹

A number of studies on the oxidation behavior of HfC over a wide range of temperatures have shown excellent oxidation behavior at temperatures above 1800°C, when the oxide formed can densify. At temperatures below 1500°C, the oxide grains formed are not able to sinter, causing them to spontaneously spall off the parent

main rate controlling mechanism for the oxide growth. While others²⁷ have also found evidence of oxycarbide at this interface (Fig. 6) after arcjet testing at higher temperatures (> 2200°C) in reduced pressure environments (using NASA Ames AHF arc-jet facility), both Shimada²⁸ and Wuchina²⁷ noted the presence of carbon (both graphitic and amorphous) at the interface after furnace oxidation at lower temperatures (800-1500°C). The effect of pressure on oxidation of the carbides has not been well studied, but the oxidation of carbon and HfC is controlled by the P_{O₂} at the interface, with the reaction:



describing the oxidation reaction at low P_{O₂}, while at higher oxygen pressures, the C would oxidize as well, leaving HfO₂ and CO₂ as the reaction products.

The effect of carbide stoichiometry has also been studied. In furnace testing (below 1600°C), HfC_{.67} had a thinner oxide scale than HfC_{.98}, as shown in Fig. 7. The oxide on the subcarbide was also denser, with less porosity and cracking, than on the higher carbide. The main difference between HfC and TaC is the melting temperature of the oxide formed. While HfO₂ is itself a high-temperature ceramic, with a melting temperature of 2758°C, Ta₂O₅ has a much lower melting point of 1872°C. This difference is a major component in the materials selection process. For high-temperature applications in

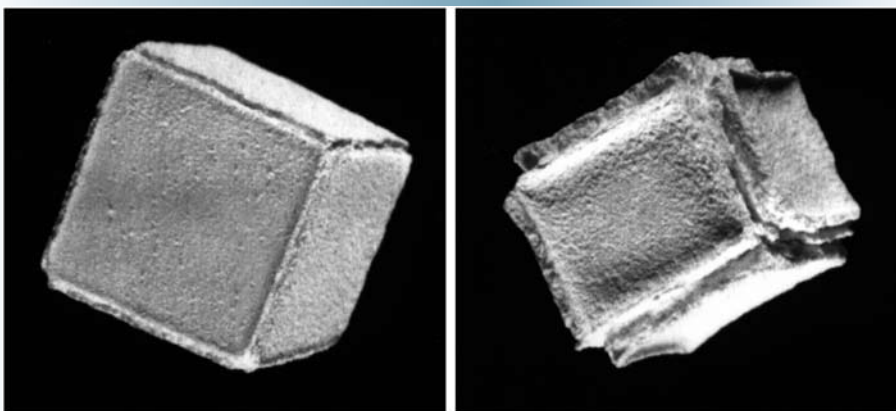


Fig. 7. Photographs of HfC_{.67} and HfC_{.98} after furnace oxidation at 1500°C for 15 minutes in air.

carbide, a process known as pestring. At low temperatures, the HfO₂ forms a porous scale (due to the evolution of CO₂ during the oxidation), and the oxidation kinetics are linear. At higher temperatures, the kinetics can be parabolic due to the slower diffusion of oxygen through a dense scale. Barger²⁶ studied the oxidation behavior of HfC films, and described the presence of an oxycarbide interlayer between the HfO₂ outer layer and the parent carbide. He concluded that the diffusion of oxygen through that the oxycarbide was the

oxidizing environments, such as engine propulsion or hypersonic leading edges, where a shape-stable, non-eroding oxide is needed, TaC is not likely to be used because the oxide formed will readily slough off, especially in high-shear flight environments, as shown in Fig. 8. HfC is a much better choice due to the HfO₂ stability. However, when P_{O₂} is considerably lower, it is possible that the oxide will not form on TaC, whereas HfO₂ will form on HfC at even very low oxygen pressures. Under these conditions, TaC would be a better

structural material. Agte²⁹ reported a maximum in the melting temperature in TaC-HfC solid solutions, but more recent studies in this system have indicated that the melting temperatures fall within the melting temperatures of the pure components.³⁰

Processing—As with the diborides, monocarbides powders are commercially produced by either carbothermal reduction (sometimes with CaCO₃) or direct reaction (SHS synthesis).^{31,32} Densification is typically done by hot pressing, but plasma spraying and hot isostatic processing (HIP) have also been employed. Plasma spraying has the advantage of producing near net-

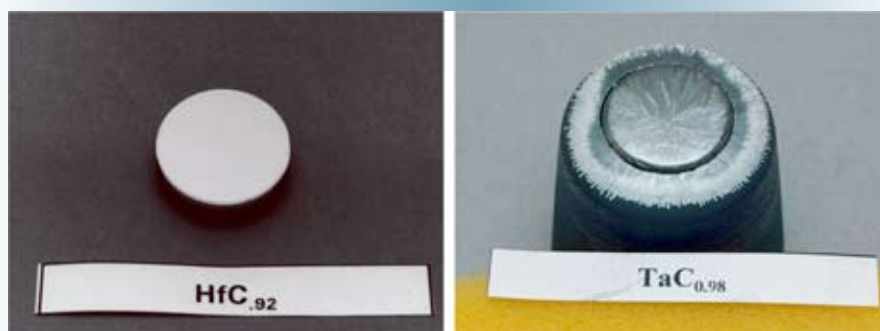


Fig. 8. Optical micrographs of HfC and TaC after 3 minute exposure in NASA-Ames arcjet facility ($T > 2000^{\circ}\text{C}$) showing presence of dense, adherent HfO₂ scale on HfC and evidence of residual molten Ta₂O₅ on TaC.

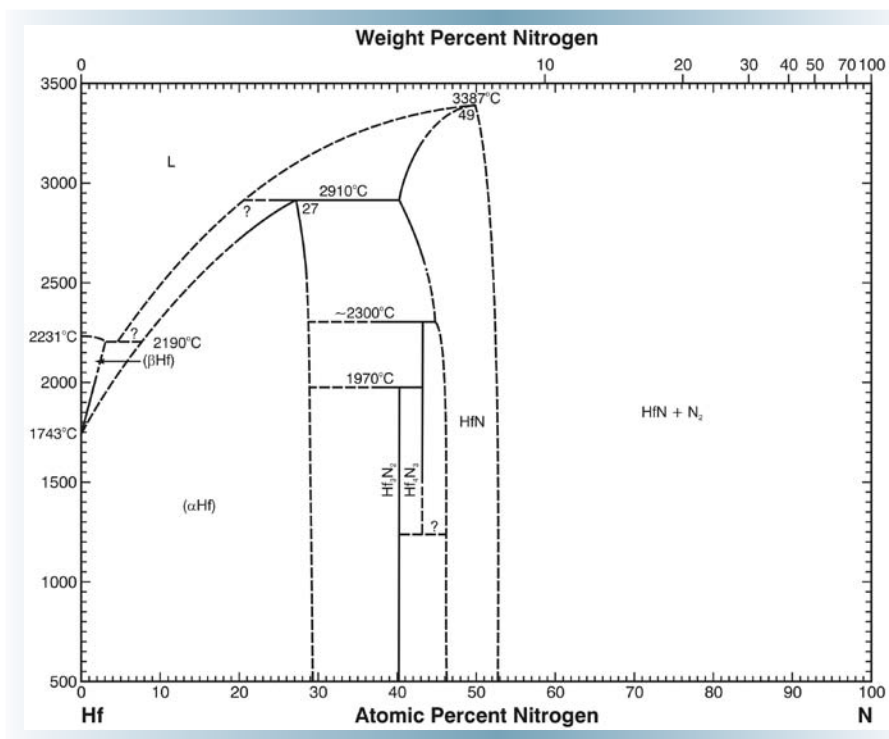


Fig. 9. The phase equilibrium diagram for the Hf-N system.³⁶

shape articles, but stoichiometry tends to be difficult to control due to loss of C at extremely high-temperatures in the plasma, while the HIP process is used when control of carbon content is of high importance. The metallic encapsulation necessary to densify the carbides from the powders is an effective diffusion barrier to the carbonizing atmosphere in the HIP vessel from the heating elements.

Nitrides

Transition metal nitrides are of critical importance in the microelectronics industry as a diffusion barrier between Cu interconnects and the SiO₂ insulation layer, as well as an interlayer/diffusion

barrier in magnetic recording devices. In the world of structural ceramics, these materials offer a wide range of thermal and mechanical properties that are of use to a systems designer. As structural ceramics, the UHTC nitrides (ZrN, HfN, and TaN) are less well known than the diborides or monocarbides, but they do offer some advantageous properties.

Like the monocarbides, HfN (and ZrN) can exist over a range of stoichiometries, as shown in Fig. 9. With a melting temperature of 3387°C, it certainly qualifies as a UHTC material. That temperature was measured in an atmospheric pressure environment: at an overpressure of nitrogen at 60 atmospheres, the melting point increases to 3800°C!³³ For long temperature applications at high temperatures, the loss of N from the lattice is thought to be a considerable problem to overcome. It is interesting to note that small N additions to the αHf with N additions lattice dramatically raise the melting temperature—from 1743°C for the pure metal to 2910 for 30%N. The thermomechanical properties of αHf have been described in the literature.³⁴ For the HfN phase, the thermal expansion and strength and modulus are all very close to that measured for HfC, while the thermal

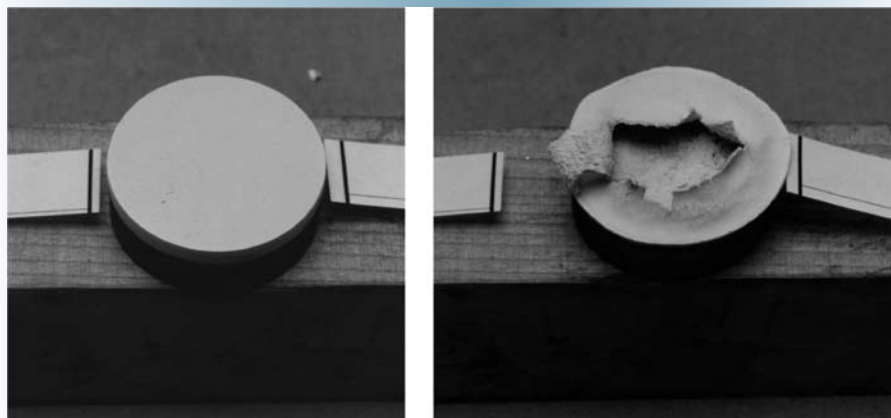


Fig. 10. Post-test photographs showing evidence of porous, adherent scale on HfN_{0.95} and dense oxide that formed on HfN_{0.75}. The buildup of N₂ behind the oxide burst during testing.

conductivity is slightly higher.³⁵ TaN is considerably less stable than HfN, with decomposition temperatures below 2700°C at atmospheric pressure.

Oxidation testing of HfN was carried out in a similar method as described for the HfC materials. While HfN_{0.92} did not have as significant of a peeling problem as HfC_{0.98} did, the scale formed was not protective and showed evidence of porosity and cracking. In higher-temperature arcjet testing, however, the HfN_{0.75} did show a very different response in forming an oxide scale. While the oxygen diffusion inward / N₂ evolution and NO_x species formation / outward diffusion are not well-understood, the gas pressures at the interface must be significantly lower as the oxide that formed during testing formed a dense, adherent scale—so much so that subsequent oxidation caused the formation of a gas bubble behind the oxide scale, resulting in a “blowout” of the oxide towards the end of the 3 minute test, as shown in Fig. 10.

Summary

Ultra-High Temperature Ceramics are a family of compounds that display a unique set of properties, including extremely high melting temperatures (> 3000°C), high hardness, and good chemical stability and strength at high temperatures. Structural materials for use in high-temperature oxidizing environments are presently limited mostly to SiC, Si₃N₄, oxide ceramics, and composites of these materials. The maximum-use temperatures of silicon-based ceramics are limited to approximately 1700°C due to the onset of active oxidation (lower temperatures in water vapor environments). The development of structural materials for use in oxidizing and rapid heating environments at temperatures above 1700°C is therefore of great engineering importance. UHTC materials are typically considered to be the carbides, nitrides, and borides of the transition metals, but the Group IV-V compounds (Ti, Zr, Hf, Ta) are generally considered to be the main focus of research due to the superior melting temperatures and formation of stable high-melting temperature oxides. The combination of properties make these materials potential candidates for a variety of high-temperature structural applications, including engines, hypersonic vehicles, plasma arc electrodes, cutting tools, furnace elements, and high temperature shielding. ■

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About the Authors

ERIC WUCHINA is a materials research engineer at the Naval Surface Warfare Center, Carderock Division in West Bethesda, MD. He received his BS in 1988 and PhD in 1995, both from Virginia Tech. His research interests include chemical vapor deposition, ultra-high temperature ceramics processing and characterization, thermochemistry, and oxidation behavior of non-oxide ceramics in extreme environments. He currently serves as the chair of the ECS High Temperature Materials Division and is the organizer of the ECS symposium *High-Temperature Corrosion and Materials Chemistry VII and Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications*, both to be held in 2008. He may be reached at eric.wuchina@navy.mil.

ELIZABETH OPILA is a materials research engineer at the NASA Glenn Research Center in Cleveland, Ohio. She received her BS in ceramic engineering at the University of Illinois in 1981, her MS in materials science at the University of California, Berkeley in 1983, and

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her PhD in materials science at the Massachusetts Institute of Technology in 1991. Her research interests include the thermodynamics and kinetics of high temperature materials oxidation and corrosion reactions. She may be reached at opila@nasa.gov.

MARK OPEKA is a research materials engineer for the Naval Surface Warfare Center, Carderock Division, in West Bethesda, MD. He earned his BS and MS in mechanical engineering, and his PhD in materials science, all from the University of Maryland. His PhD included significant studies at Ohio State University in metallurgical thermodynamics and oxidation kinetics. He has been employed by the Navy for 30 years and has conducted research and development on high temperature materials, including refractory metals, cermets, ceramics and ceramic composites, carbon-carbon composites, and carbon-phenolic composites. He has authored or co-authored 30 publications on materials selection for high temperature systems and oxidation properties of high temperature materials. He may be reached at mark.opeka@navy.mil.

BILL FAHRENHOLTZ is an associate professor of ceramic engineering at the University of Missouri-Rolla. He teaches undergraduate and graduate courses on thermodynamics as well as a required sophomore level laboratory on traditional ceramics. His research focuses on the processing and characterization of ceramics and ceramic-metal composites. He has current projects related to ultra-high temperature ceramics as well as the use of cerium oxide coatings for the corrosion protection of high strength aluminum alloys. He has published over 50 papers on his research. He may be reached at billf@umr.edu.

INNA TALMY is the senior research ceramist and group leader of the Ceramic Science and Technology Group at NSWCCD. She received both her MS (1957) and PhD (1965) degrees in ceramic science and engineering from the Mendeleev Institute of Chemical Technology in Moscow, Russia. Previously, she worked in the ceramic departments of Institutes of Chemical Technology in Moscow and Prague, Czechoslovakia. Her primary research efforts are in the field of structural non-oxide ceramics, cermets, dielectric ceramics, superconductors, and ceramic-matrix composites. Inna directed the development of celsian and phosphate ceramics as candidates for next generation tactical missile radomes. Her work has resulted in over 80 publications and 21 patents. She may be reached at inna.talmy@navy.mil.



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Pennington, NJ 08534-2839, USA
Tel: 609.737.1902
Fax: 609.737.2743
E-mail: interface@electrochem.org

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