SPA 3 Activities DIFFER 2023

T.W. Morgan^{1,2}, L. Nuckols³, J. Rapp³

¹Dutch Institute for Fundamental Energy Research, Eindhoven, The Netherlands ²Eindhoven University of Technology, The Netherlands ³Oak Ridge National Laboratory, USA









UHTC Overview/Background

- <u>U</u>ltra-<u>H</u>igh <u>T</u>emperature <u>C</u>eramics (UHTC)
- Defined by:
 - Melting temperatures: > 3000 °C
 - Application: Sustained working temperatures > 1600
 °C
 - Chemistry: Largely binary compounds of Boron, Nitrogen, or Carbon bonded with an early transition metal
- Historic and current materials of interest for leading edges, heat shields, and thermal protection systems for hypersonic and atmospheric re-entery vehicles

1 Н Нудгарая Интенни		PubChem								(2 He Helan						
3 Lii Linna Anal Vale	4 Be Beryflum Metro toro total			H	ydrogen	Nam	ie					5 B Baron Veistad	6 C Carbon Norrelat	7 N Ntrogen Norrese	8 O Crygen Herrord	9 Fisecine scope	10 Neo Naor 100
11 Na Solum	12 Mg Magnesium Magnesium	Nonmetal Chemical Group Block						13 Alectron Ref Teacher Hard	14 Sileon Marcure	15 P Pheophorus Normer	16 Softer	17 Clippine Patripe	18 Ar Argon Mar ta				
19 K Petessium Aust More	20 Ca Criteium Kinetie Konto Konto	21 Scanduars Transfer trad	22 Ti Titarium Trevettan Mona	23 V Vanadium Terrelitice Metal	24 Cr Crr Crrsolum Territor Ment	25 Mn Marganese Transition	26 Fe	27 Co Cobell Tracester Meter	28 Nicket Traveline Konic	29 Cu Conser Technologia	30 Zn 2re	31 Ga Dailture	32 Ge Osementum Martin	33 As Armenic Nonice	34 See Selenium	35 Br Bromine Recent	36 Krater
37 Rb Rubidium	38 Sr Strotture	39 Yutikan	40 Zrcontam Treastant Minut	41 Nbb Nobium	42 Mo Moryladerati	43 TC Technotium	44 Ru Partiere and	45 Rh Produm	46 Pd Patiadium	47 Ag	48 Cd Cadmium	49 In 149	50 Sn	51 Sb Antimery	52 Telarlari visual	53	54 Xe
55 Cs Cesture	56 Ba Batun	•	72 Hff Hafniam	73 Ta Tantalum Tunuttee Metal	74 W Tungsten	75 Re Aberium	76 Os oumum	77	78 Pt Platnum	79 Au _{Gold}	80 Hg Mercury	81 TI The furr	82 Pb	83 Bi Banuth	84 Pool	85 At Astatione	86 Rr Radon
87 Fr Francium	88 Ra Pacture		104 Rf Rutherfordiare	105 Db Debnium Transition Intel	106 Sg	107 Bh Bohrion Transition	108 Hs Hassian Transition	109 Mt	110 DS Dermittadfiller Teacher Here	111 Rg Reenspecture Investigation	112 Con Coperations Transmission	113 Nh Nitorium Hitorium	114 Fl Flerevium Francharothere there	115 Mc Maconfum Maconfum	116 Lv Liverenterium Part Transferre Market	117 TS Tornecoline Heaper	
			57 La	58 Ce	59 Pr Praseotymian Lastans	60 Nd Neodymian Latitatik	61 Pm Promotivium	62 Sm Benarlan	63 Europian	64 Gd Badolistum	65 Tb Terbian	66 Dy Dysprosium	67 Ho Halinian Larrania	68 Er	69 Tm Thailum	70 Yb	71 Luc Latelier
			89 Activities	90 Th	91 Pa	92 U Uranium	93 Np Neptunkers	94 Put Putrostum	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr



UHTCs Material Properties and Fusion Plasma Facing Applications

- Several UHTC chemistries have appealing material properties for fusion PFM applications
 - High temperature strength and thermal conductivity
 - tunable neutronic properties
 - potentially composed of low to mid Z elements
- However, uncertainties on UHTC PMI response needs to be studied prior to qualification/disqualification
 - chemical erosion processes
 - failure mechanisms at high temperatures
 - microstructural response to plasma transients



Material	Melting Temperature (°C)	RT Flexural Strength (MPa)	HT Flexural Strength (MPa)	RT Thermal Conductivity (W/m-K)	HT (1000 °C) Thermal Conductivity (W/m-K)
ZrB ₂	~3245	300 – 500	~450 (800 °C)	60 – 105	~50
TiB ₂	~3225	375 – 1000	~550 (1000 °C)	60 – 120	~67
Sintered SiC	2700 (sublimation)	325 – 400	350 – 450 (1000 °C)	~400	~80
Tungsten	~3422	460-600	~200 (1000 °C)	~180	~110 /23

UHTCs for Sacrificial Limiters

- Moving towards a fusion pilot plant (FPP) means means more demands on first wall (FW) blanket
 - Heat transfer and tritium breeding requirements verses just surviving
 - Stricter heat flux constraints
- Sacrificial limiters will likely be used to protect the blanket from transients
 - Limiter Drivers:
 - 1) Withstand thermal and particle loads from steady state exposures and ramp-up and ramp-down
 - 2) Following intense/unexpected transients, protect the blanket from damage extending to cooling channels





Starting Materials

- ZrB₂ and TiB₂ samples formed via reaction hot pressing
 - $MeH_2 + 2B \rightarrow MeB_2 + H_2(gas)$
 - No sintering additives used to ensure chemical purity
 - Pristine grain sizes ~3 10 μ m (TiB₂) and ~ 10 30 μ m (ZrB₂)





Proposed Magnum PSI Exposure Conditions

- Experimental Goals:
 - 1) Analyze material surface microstructure evolution as a response to coupled steady-state and transient heat fluxes
 - 2) Determine transient load limit in diborides
 - 3) Measure erosion products in-situ and net erosion ex-situ to determine changes in erosion behavior as a response to coupled HFs
- Utilizes the pulsed plasma system in tandem with steady-state exposures
- Experimental Controls
 - Plasma composition
 - Steady-state loading conditions (~5 MW/m², ion flux, ion energy, ion fluence)
 - Transient pulse conditions (~10 Hz, 0.25 GW/m², 1 ms pulses)
- Experimental Variables
 - Presence of pulses
 - Number of total pulses





Thank you for your attention



Sample Name	Chemistry	# of transient pulses	Steady State Heat Flux (MW-m ⁻²)
TB-1	TiB ₂	0	5
TB-2	TiB ₂	10	5
TB-3	TiB ₂	100	5
TB-4	TiB ₂	1000	5
TB-5	TiB ₂	10000	5
TB-A*	TiB ₂	0	5
TB-B*	TiB ₂	100	5
ZB-1	ZrB ₂	0	5
ZB-2	ZrB ₂	10	5
ZB-3	ZrB ₂	100	5
ZB-4	ZrB ₂	1000	5
ZB-5	ZrB ₂	10000	5
ZB-A*	ZrB ₂	0	5
ZB-B*	ZrB ₂	100	5

Desired in-situ diagnostics

- Fast IR imaging of sample surfaces
- Thomson scattering on plasma near target surface
- OES tuned to target elements

Controls (excluding *'d samples)

- H plasma
- Pulse magnitude (0.25 GW/m²), frequency (10 Hz) and duration (1 ms)
- Steady state conditions
 - HF, ion energy, ion flux, fluence
 - 1000 °C ambient surface temperature

*D plasma exposures with transient heat flux simulation from LASGAG system



Planned Post-mortem Analysis

- IBA-NRA
- Mass loss measurements
 - Net erosion
- X-ray photoelectron spectroscopy (XPS)*
 - Measures changes in areal-averaged surface chemistry/stoichiometry, indicating preferential erosion
- Scanning electron microscopy (SEM)*
 - Examine plasma-induced microstructural evolution of sample surfaces
- SEM-based techniques: energy dispersive x-ray spectroscopy (EDS)* and electron backscattered diffraction (EBSD)*
 - EDS: Surface chemistry mapping to determine the presence and magnitude of preferential erosion
 - EBSD: Surface crystallographic mapping to measure changes crystallographic orientation
- Focus ion beam (FIB)*
 - Determine damage depth
- Transient grating spectroscopy (TGS)
 - Measures surface thermal transport properties
- Thermal desorption spectroscopy (TDS)*
 - Measures light atom retention in material
- Atomic Force Microscopy (AFM)*
 - Measures/maps surface roughness

*can be performed at ORNL