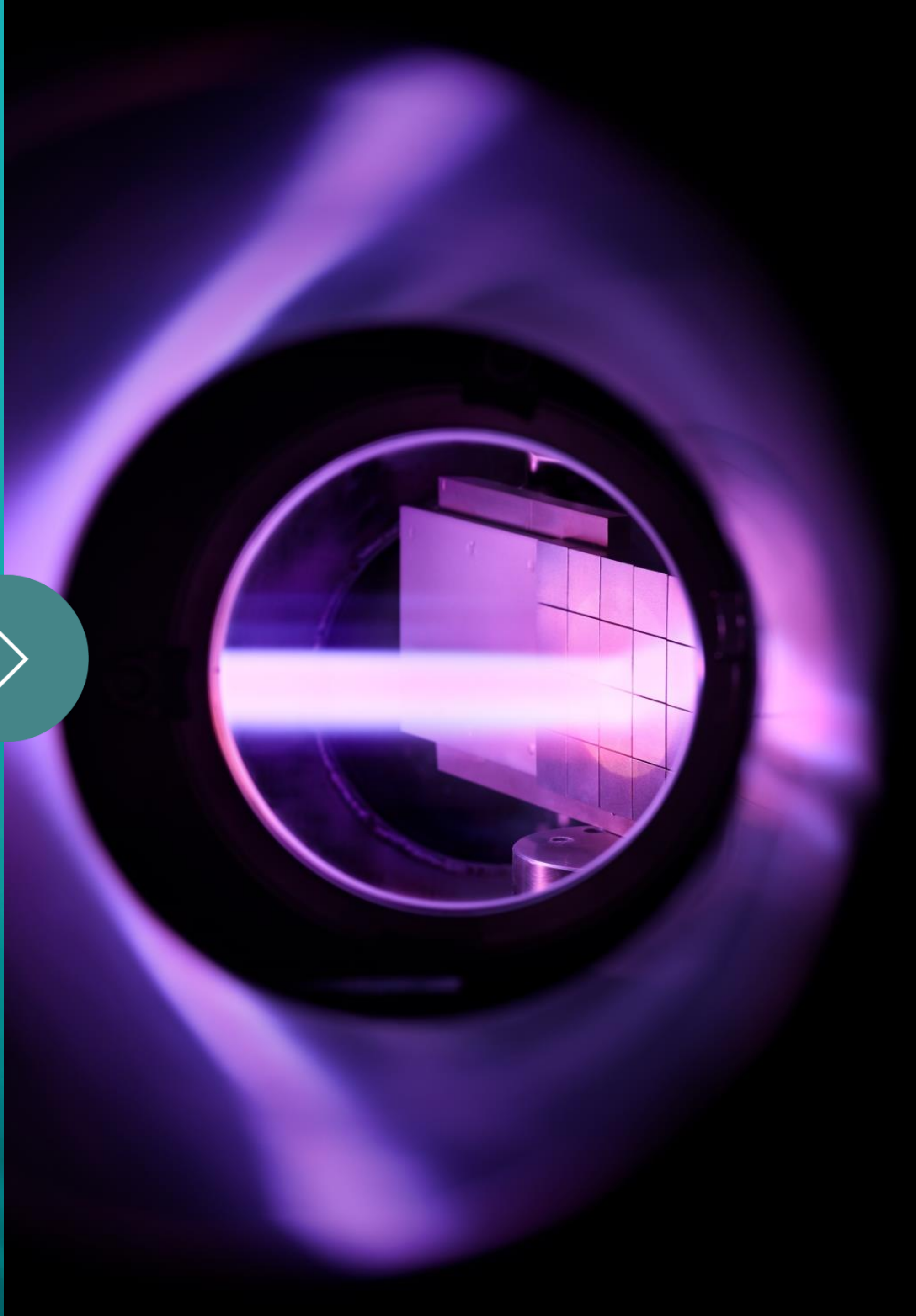
SPA 3 Activities DIFFER 2023

T.W. Morgan[1,2], L. Nuckols[3], J. Rapp[3]

[1]Dutch Institute for Fundamental Energy Research, Eindhoven, The Netherlands

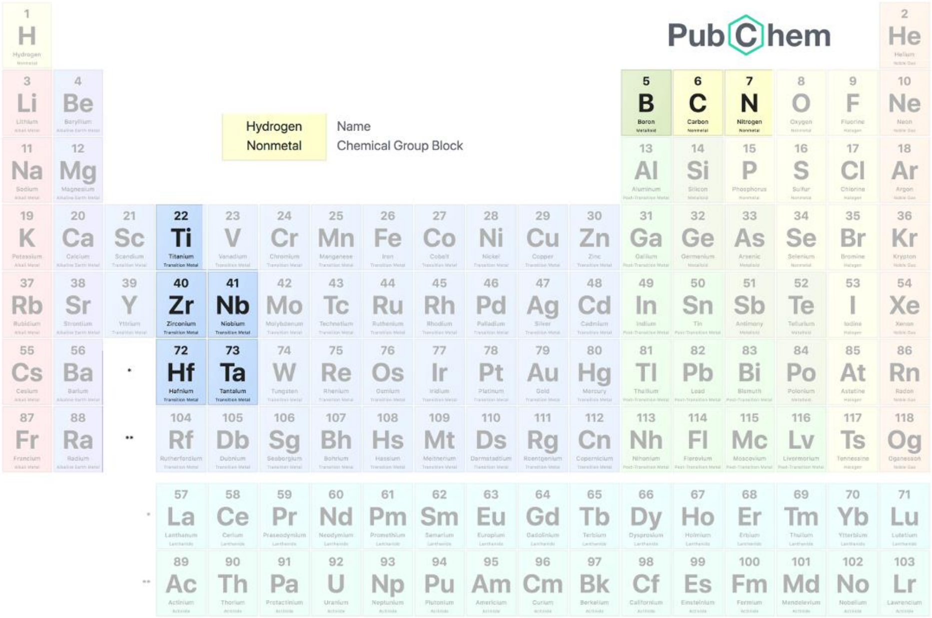
[2]Eindhoven University of Technology, The Netherlands

[3]Oak Ridge National Laboratory, USA



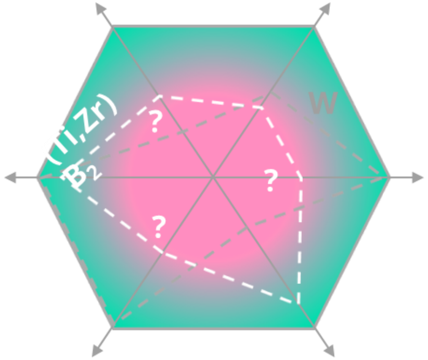
UHTC Overview/Background

- **U**ltra-**H**igh **T**emperature **C**eramics (UHTC)
- Defined by:
  - Melting temperatures: > 3000 ºC
  - Application: Sustained working temperatures > 1600 ºC
  - 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



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



Oxidation
Oxide Stability,
Oxidation Mechanism

Mechanical Properties
Flexural strength,
Elastic modulus,
Machinability

Thermal Properties
Thermal conductivity,
Thermal expansion,
Melting temperature

H/D/D
Interaction
Retention,
Diffusion,
Co-deposition

Sputtering
Net and Gross Erosion,
Chemical and physical erosion,
Surface compositional change

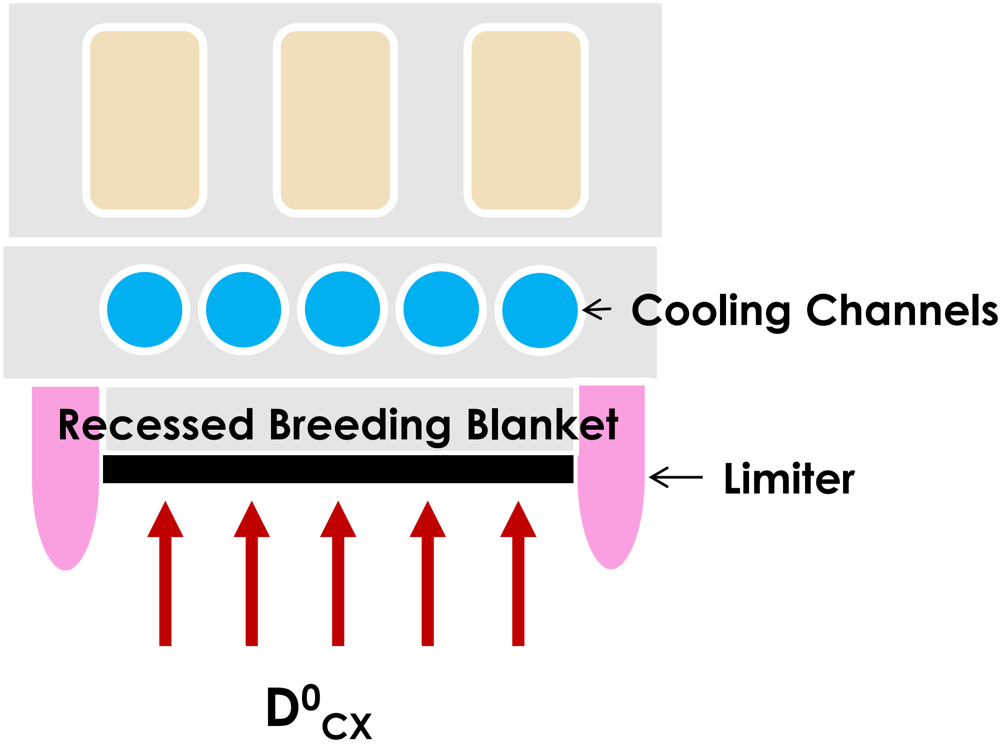
Transmutation/Activation

Plot concept and W info from: juser.fz-juelich.de/record/283652/files/Coenen_MA-1

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

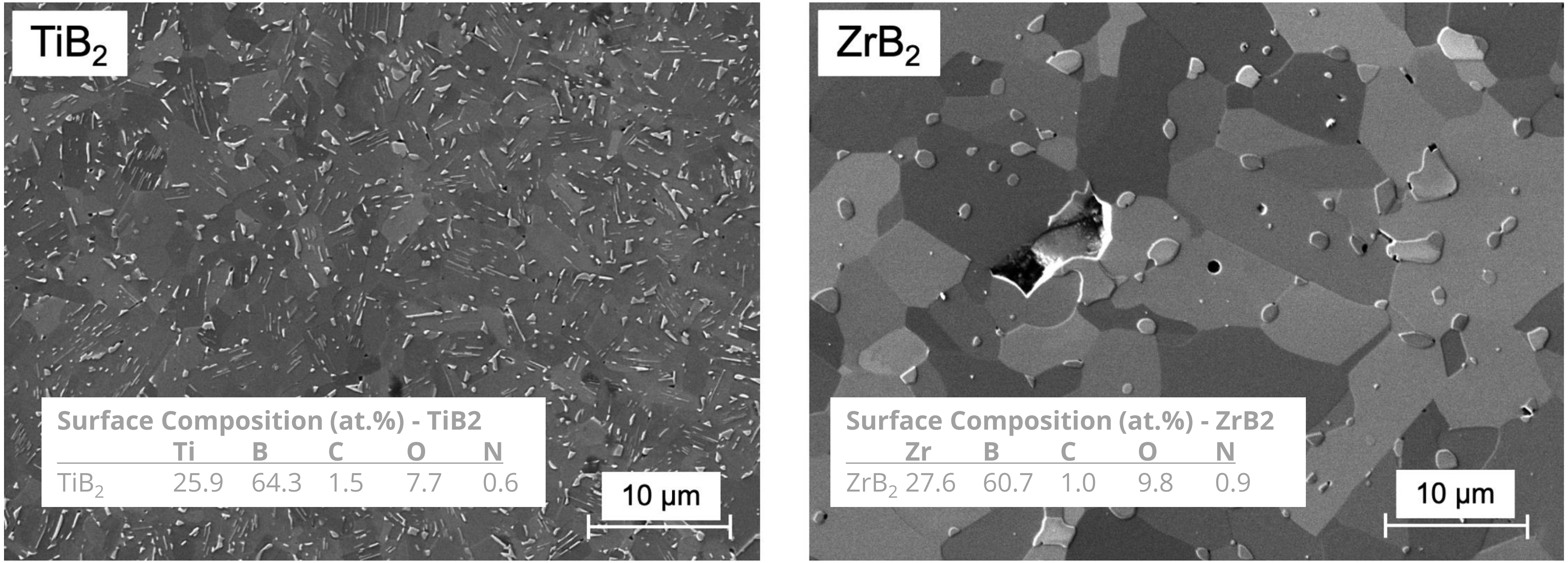
UHTCs for Sacrificial Limiters

- 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

- ZrB_2 and TiB_2 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 µm (TiB_2) and ~ 10 – 30 µm (ZrB_2)



Surface Composition (at.%) - TiB2

	Ti	B	C	O	N
TiB_2	25.9	64.3	1.5	7.7	0.6

Surface Composition (at.%) - ZrB2

	Zr	B	C	O	N
ZrB_2	27.6	60.7	1.0	9.8	0.9

SEM micrographs taken with Tescan Mira 3 at LAMDA labs (ORNL), surface chemistry measurements taken via x-ray photoelectron spectroscopy.

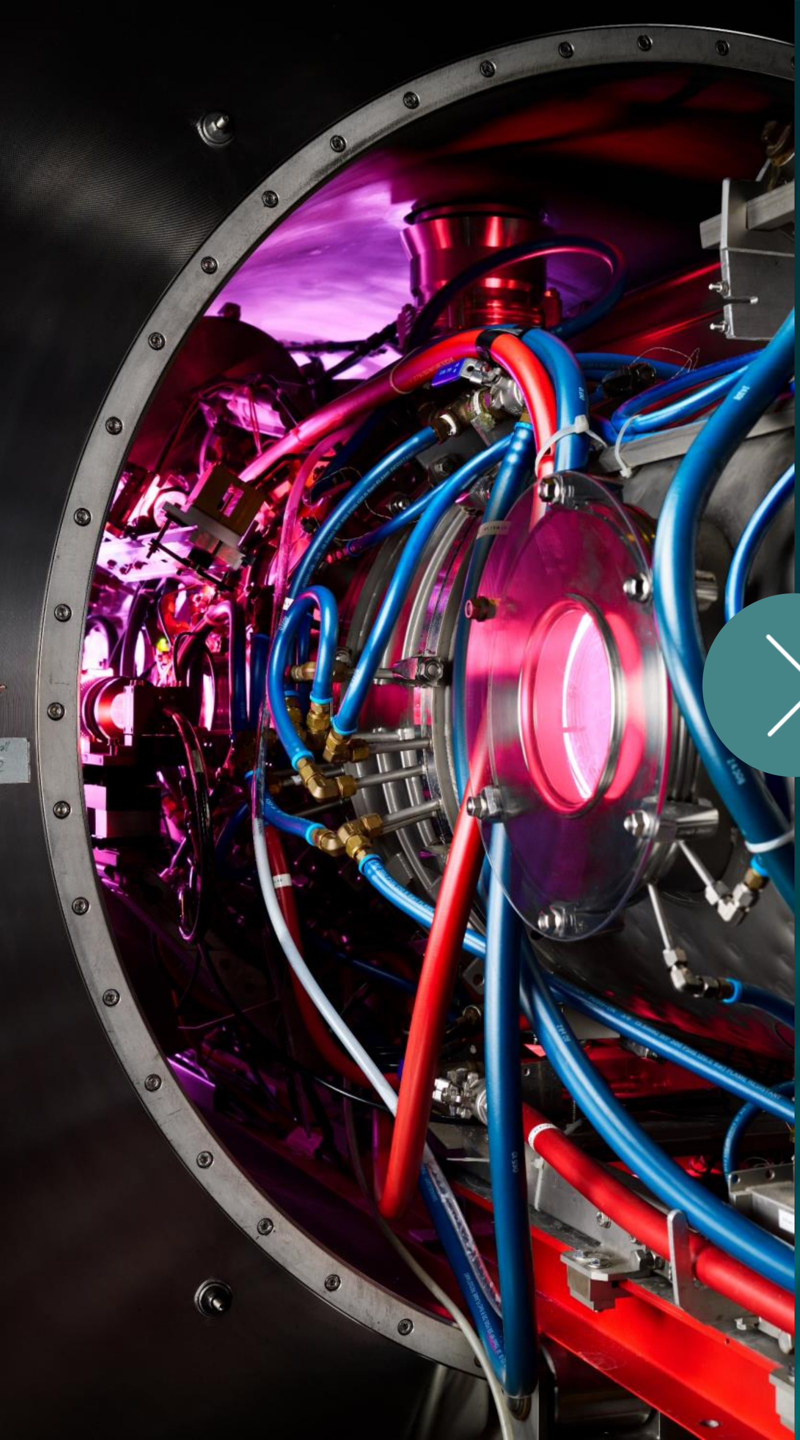
Proposed Magnum PSI Exposure Conditions

- 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/m^2, ion flux, ion energy, ion fluence)
  - Transient pulse conditions (~10 Hz, 0.25 GW/m^2, 1 ms pulses)

- Experimental Variables
  - Presence of pulses
  - Number of total pulses



Thank you for your attention

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Preliminary Shot Plan

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*

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

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/m^2), frequency (10 Hz) and duration (1 ms)**
- Steady state conditions
  - HF, ion energy, ion flux, fluence
    - 1000 °C ambient surface temperature

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