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Homogeneity Study of ZrC, NbC, and TaC Binary Carbides

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Motivation and Background

- Nuclear Thermal Propulsion (NTP) requires a fuel form that survives extreme conditions
- Cercer (ceramic-ceramic), Cermet (ceramic-metallic), and solid solution carbide fuel forms have previously been considered
- The database for solid-solution carbides is limited in information
- Does consolidation to >95%TD lead to full homogenization of bi-carbides?

Experimental Methods

- US Research Nanomaterials Inc. Sourced Carbide Powders

Carbide	Particle Size (nm)	Anion : Cation Ratio
ZrC (excl. ZrO_x)	400 – 1200	0.85
NbC	500	0.98
TaC	300	0.98

- Notable Impurities
  - ZrC Impurities >1wt%

Impurity	Quantity (wt%)
Hf	~3%
ZrO_x	~3%
W	~2.5%

- Samples consolidated in Spark Plasma Sintering (SPS or Field Assisted Sintering Technology)

- Minimum Conditions used to consolidate Bi-Carbides to >95% Theoretical Density (%TD)

Carbide	Temperature (°C)
$(Nb_{50},Zr_{50})C$	1600
$(Ta_{50},Zr_{50})C$	1700
$(Ta_{50},Nb_{50})C$	2200

Conditions at 30 MPa with 100 °C/min ramp and 15-minute hold at temperature

- Samples characterized through
  - Archimedes Density Method
  - X-Ray Diffraction
  - SEM and EDS

Densification vs Homogenization

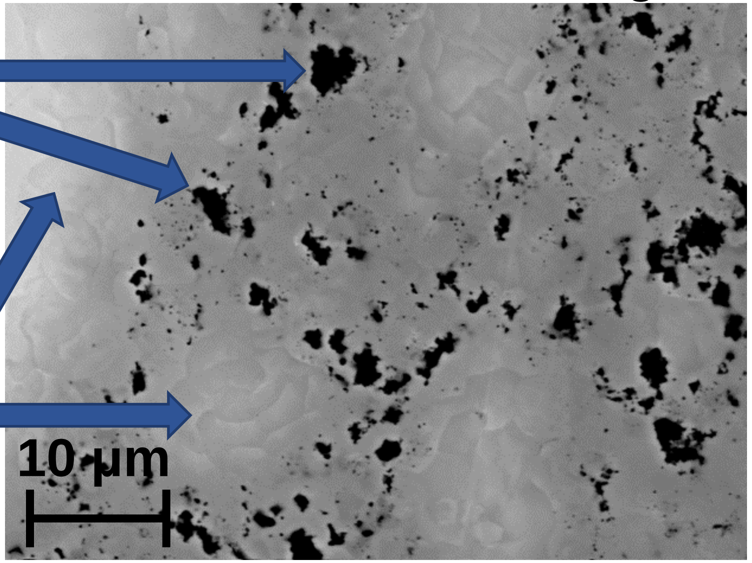
- Densification typically occurs faster than homogenization
  - Surface diffusion (for sintering near ideal density) tends to occur at lower temperatures than bulk diffusion (required for solute interdiffusion)

$(Ta_{0.5},Zr_{0.5})C$ sintered to ≈99%TD

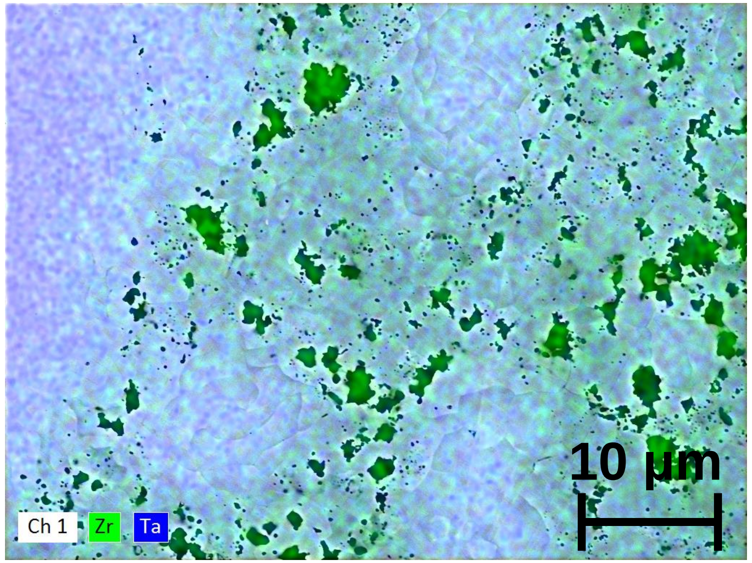
SEM Backscatter Image

Zr rich regions

Ta rich regions



EDS Atom Map



Zr

Ta

Chemical segregation is still present in densified specimens
Homogenization not complete

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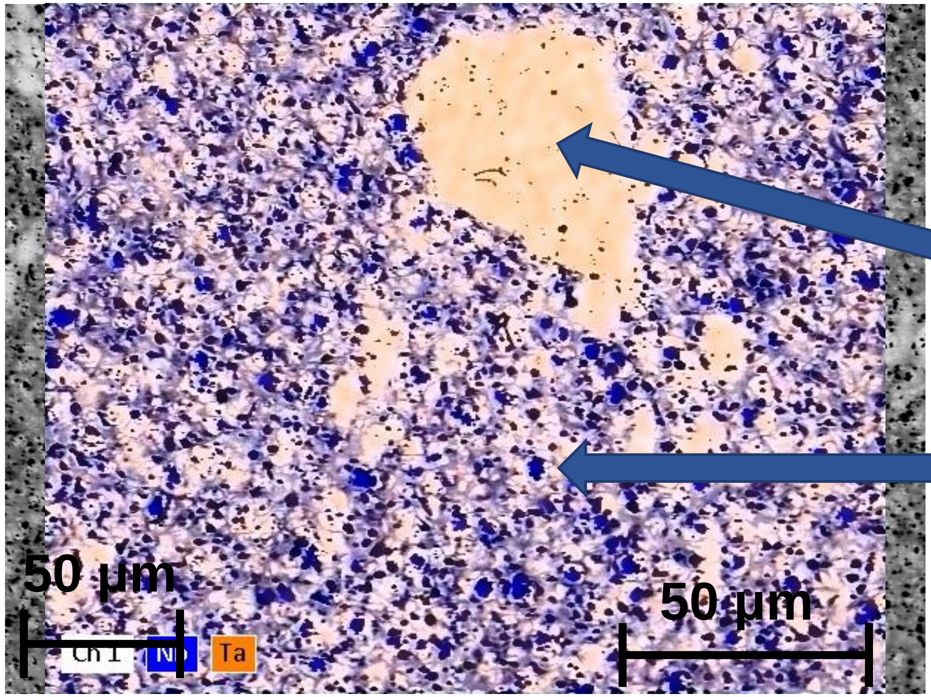
Substantial compositional heterogeneity observed within >95%TD sintered specimens

$(Nb_{0.5},Ta_{0.5})C$ sintered to ≈96%TD



Nb

Ta

Large (~5 – 50 μm) heterogeneity potentially due to inadequate mixing, and fine-scale (~0.5 – 5 μm) heterogeneity due to initial NbC, TaC particles

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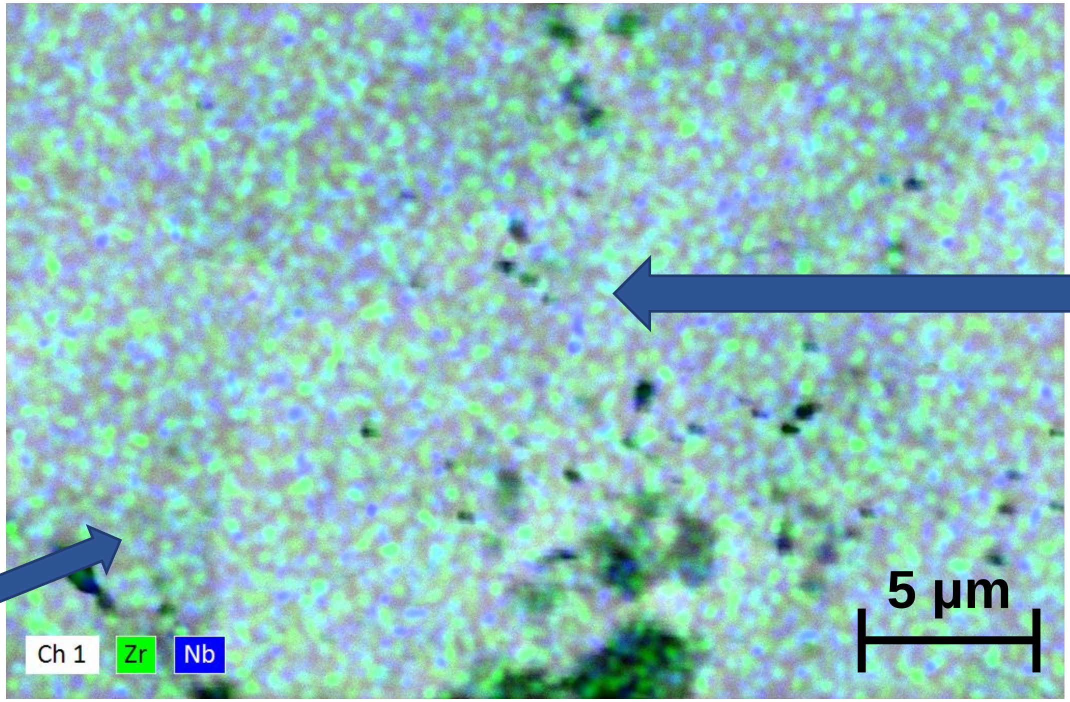
Substantial compositional heterogeneity observed within >95%TD sintered specimens

$(Nb_{0.5},Zr_{0.5})C$ sintered to ≈100%TD



Apparent ZrO_x aggregation

Apparent fine-scale heterogeneity (similar to particle size)

Carbide	Particle Size (µm)
ZrC	0.4 – 1.2
NbC	0.5

Zr
Nb

Microstructure at different magnifications exhibit different length scale perspectives with respect to homogeneity

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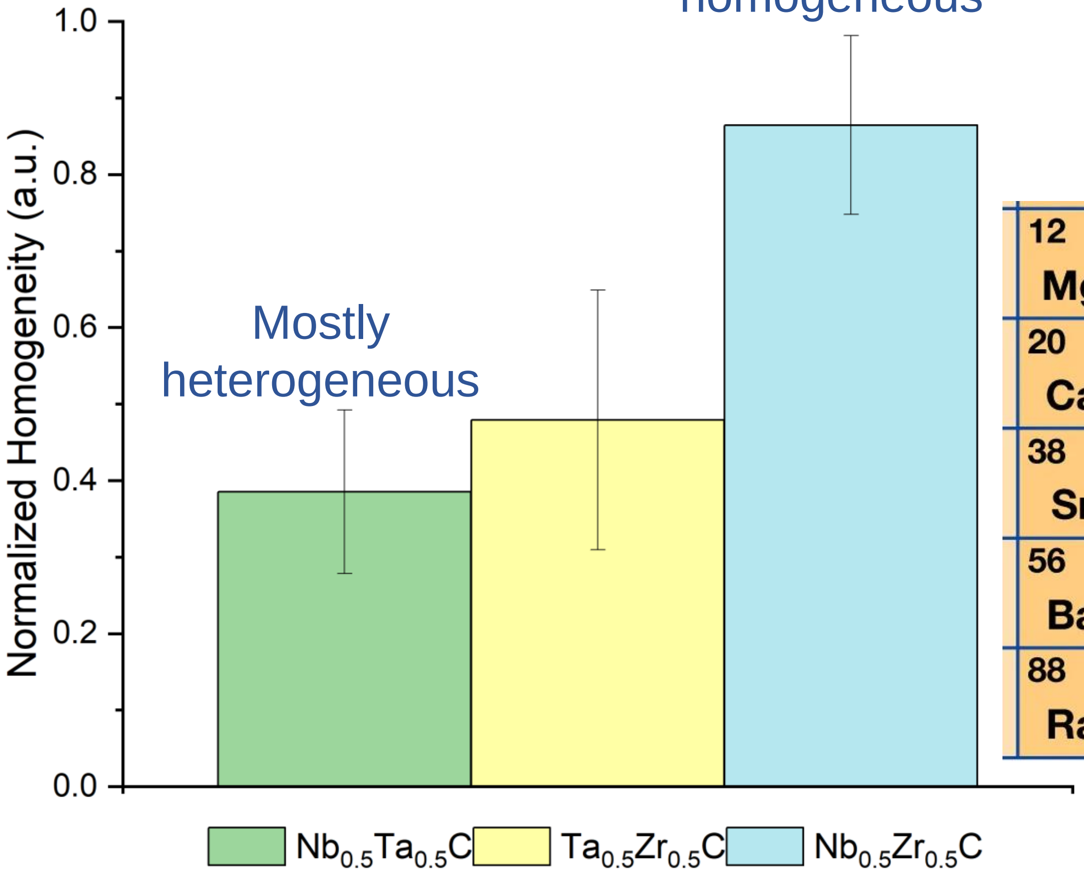
Effect of monocarbides on degree of homogenization

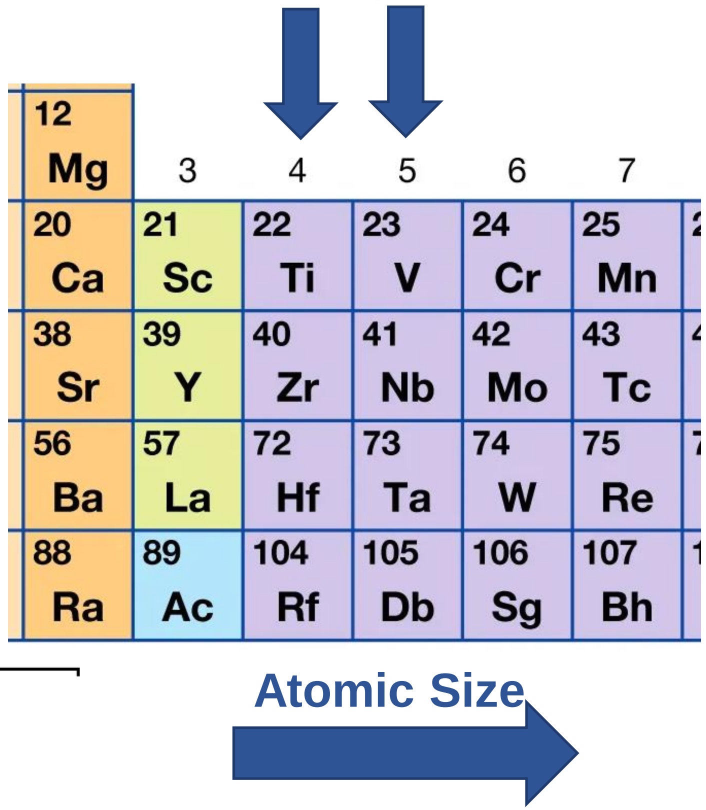
Methodology

- EDS Lines Scans of the samples were normalized to the cation-cation ratios found in XRD
- EDS Point Scans from the Line Scan that fell within **+/- 5 atom %** from stoichiometry are consider **homogeneous**

Carbide	Melting Temperature (°C)
ZrC	3420
NbC	3600
TaC	3950



Group IV metallic bonding **weaker** than Group V



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Diffusivity controls cation-cation diffusion by the **physical size** of the cations, **strength of the bonds** between the cations, and difference in **melting temperature**.

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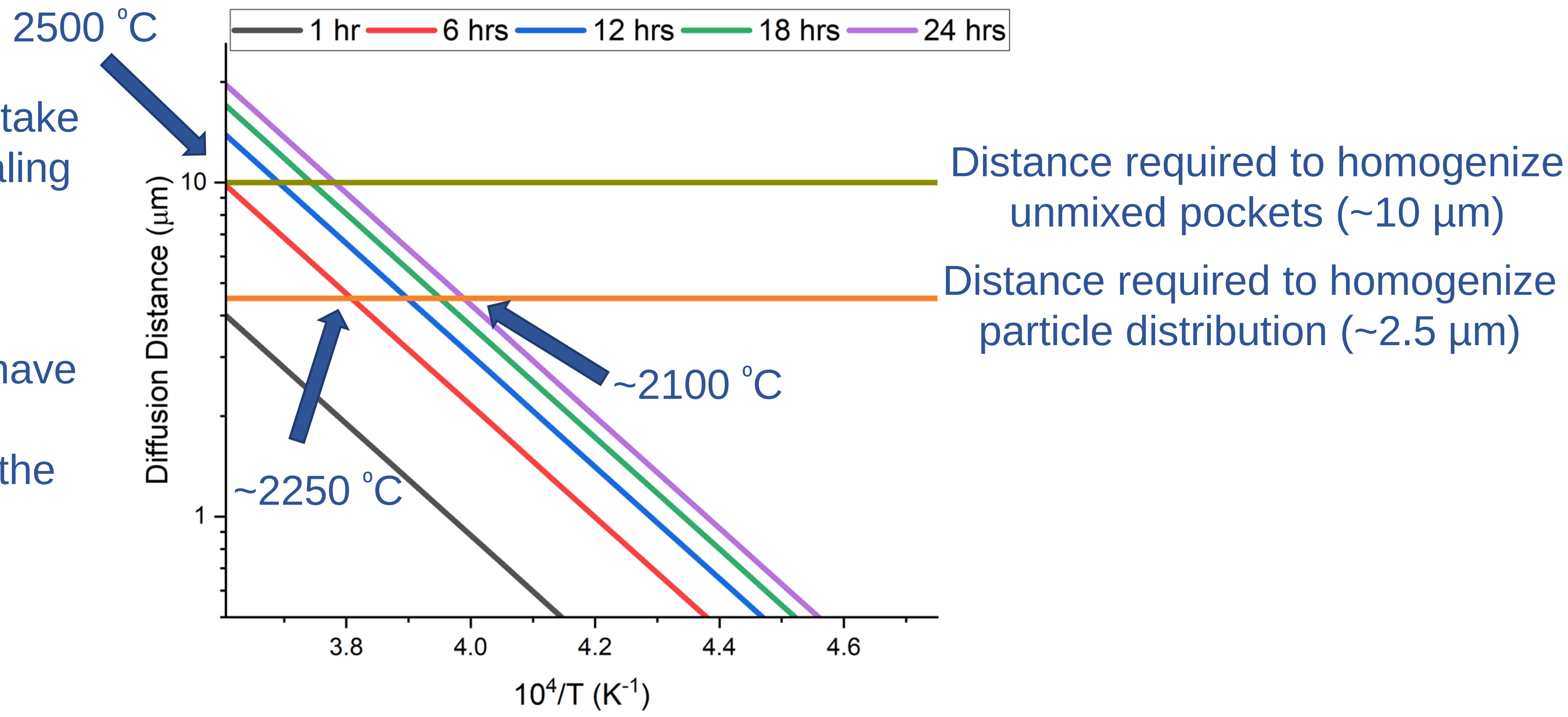
Estimated annealing conditions to achieve homogenization
(to be confirmed in upcoming research)

- $(Nb_{0.5},Ta_{0.5})C$ expected to take longest and highest annealing times and temperatures
- $(Ta_{0.5},Zr_{0.5})C$ expected to have moderate homogenization conditions with respect to the $(Nb_{0.5},Zr_{0.5})C$ system

Calculated diffusion[1] of Nb in $(Nb_{0.5},Zr_{0.5})C$



Annealing at 2250 °C for ~18 hours should produce chemical homogenization

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[1] Lanin, A. (2014). *Nuclear Rocket Engine Reactor.* Springer. https://doi.org/10.1007/978-3-642-32430-7

Future Work

- Further analysis on annealing parameters
- Homogeneity for ternary carbide systems

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