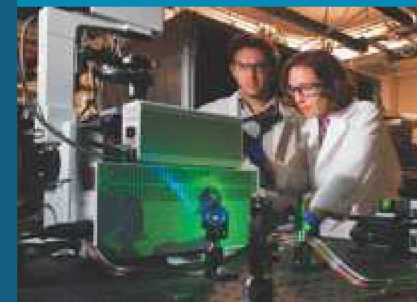
SAND2018-11758C

Analysis of Defects in Tungsten Heavy Alloy



PRESENTED BY

Zahra Ghanbari, Donald Susan, Alice Kilgo, Christina Profazi and Michael Heiden

Acknowledgements: *Jay Carroll, Zach Casias, Bonnie McKenzie, Michael Parrish*

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Background on Tungsten Heavy Alloy

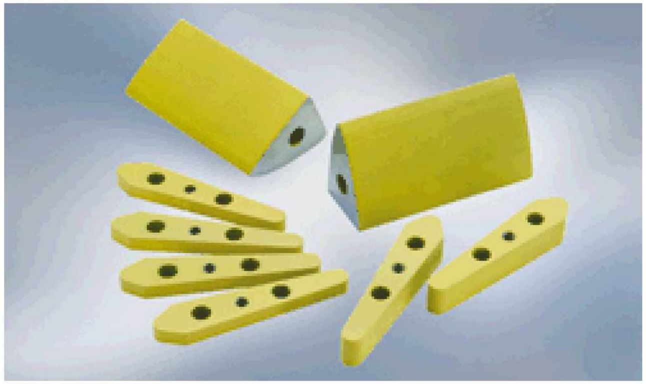
Tungsten Heavy Alloy (WHA) is a metal matrix composite comprised of W particles in a binder comprised of metals with lower melting points

- Commonly Ni-Fe or Ni-Cu

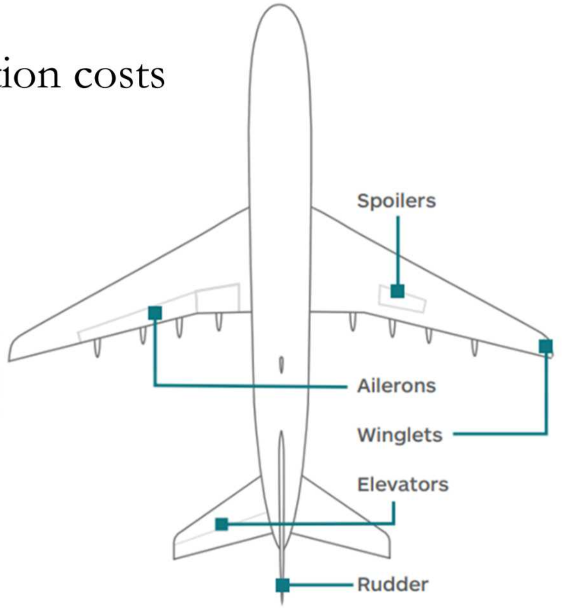
WHA's are used in applications that require **high density** (16.85-18.85g/cc)*, where tungsten (W) or tungsten carbide (WC) are not suitable. Compared to these alternatives, WHA provides:

- Lower sintering temperatures than W, and thus lower fabrication costs
- Increased flexibility in size and shape of manufacturers parts

Syringe Radiation Shielding *Kennemetal*



H.C. Starck



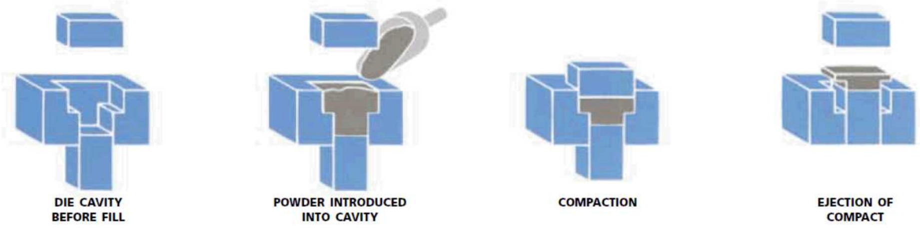
Wolfmet, M&I Materials Ltd

*ASTM B777

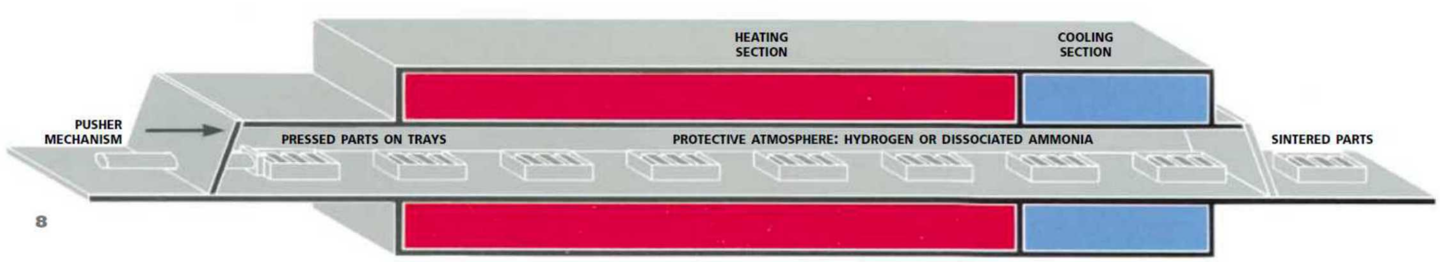
Fabrication of WHAs via Sintering

The W and binder powders (often Ni, and Fe or Cu) are pressed in either die cavities (i.e. small parts) or cold isostatic presses (i.e. large rods), and then sintered in large furnaces (often belt furnaces)

Die Pressing of Powder*



Sintering of Compact*



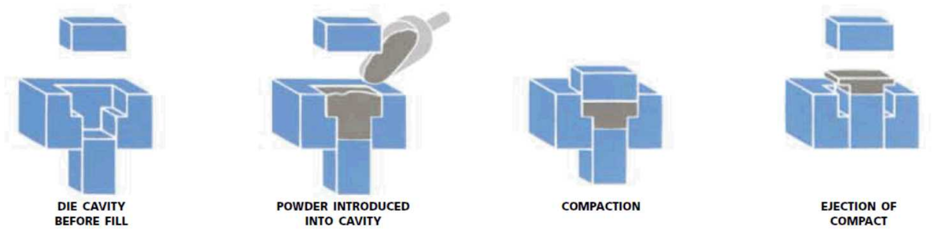
*Images from H.C. Starck, Inc
+ASM Phase Diagram Database

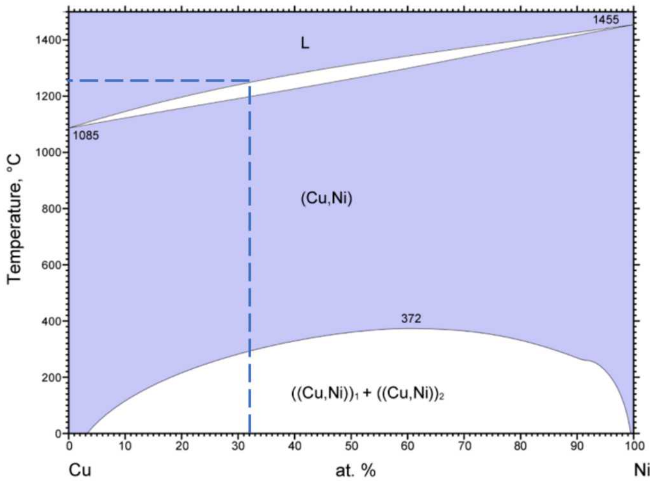
Fabrication of WHAs via Sintering

The W and binder powders (often Ni, and Fe or Cu) are pressed in either die cavities (i.e. small parts) or cold isostatic presses (i.e. large rods), and then sintered in large furnaces (often belt furnaces)

- Liquid phase sintering: $T_{sinter} > T_{melt-binder}$, to allow flowing of binder alloy around the W particles

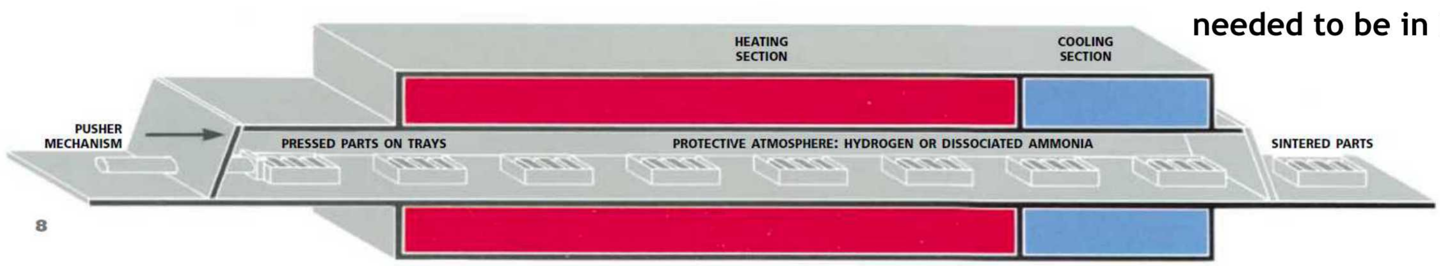
Die Pressing of Powder*





For a W-7wt%Ni- 3wt%Cu, $T_{sinter} \geq \sim 1250°C$ needed to be in liquid-phase field+

Sintering of Compact*



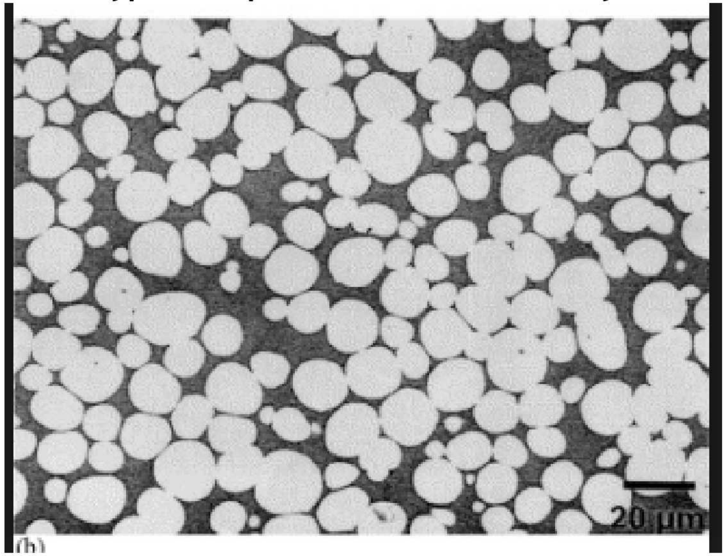
*Images from H.C. Starck, Inc
+ASM Phase Diagram Database

Microstructural Features of As-Sintered WHA

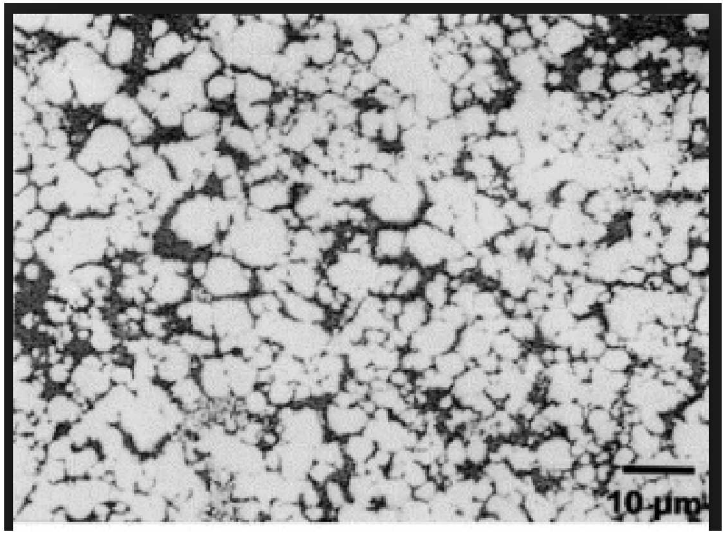
If proper sintering is performed (i.e. liquid-phase sintering), particles of W are suspended in the binder alloy.

If the sintering temperature is too low, termed solid-state sintering, a reduction in properties can also be realized

Typical Liquid-Phase Sintered Alloy*



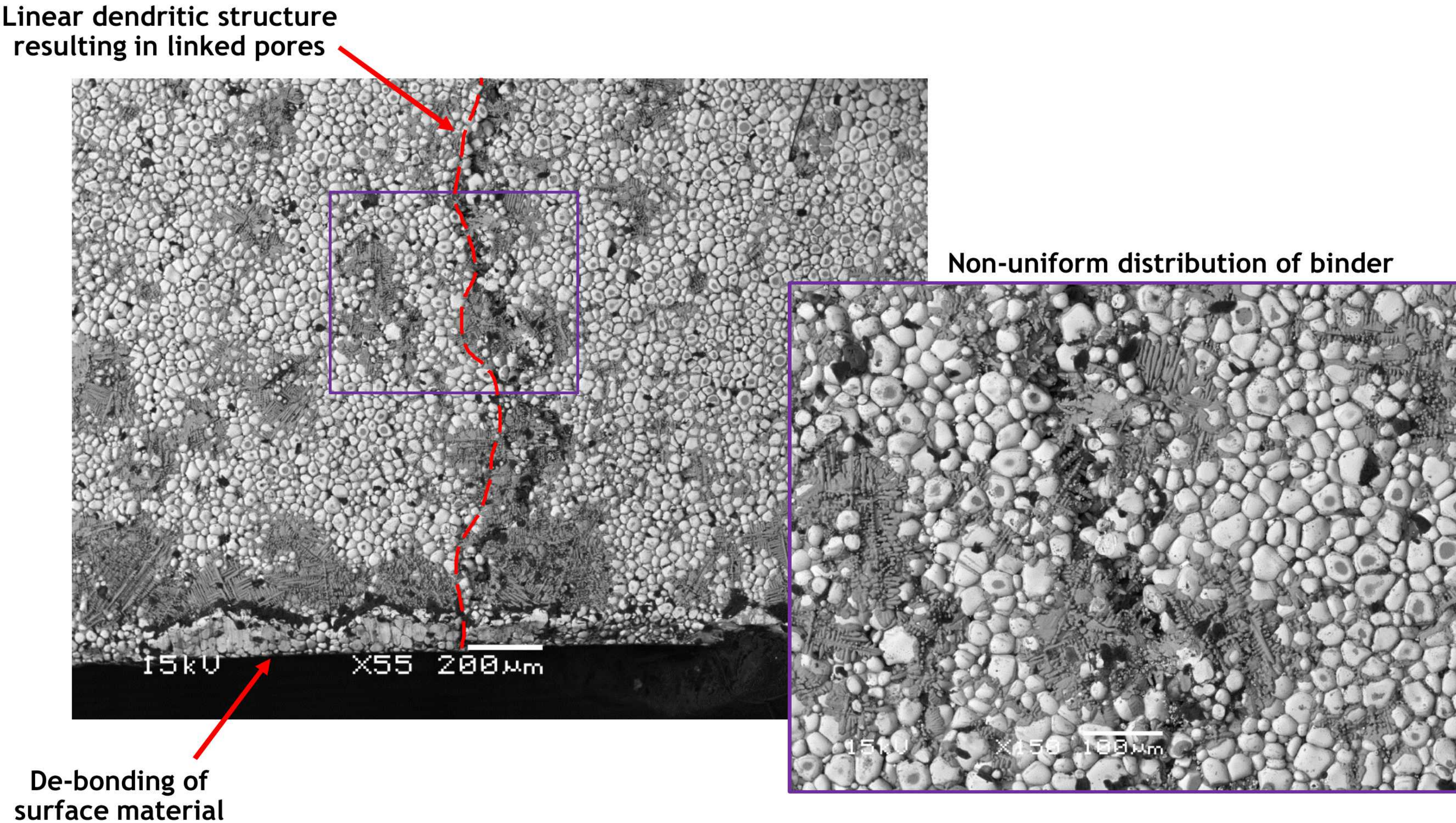
Typical Solid-State Sintered Alloy *



** Upadhyaya*

Defects from Fabrication

If sintering is performed incorrectly, several defects can be observed, including: non-uniform binder distribution, linear defects, porosity, and de-bonding of surface material:



Defect Implications: Density Requirements, Presence of Porosity

ASTM B777 calls out weight percent of W and density requirements

- The requirements can still be met with some volume fraction of pores present

Table 1 Composition, Density, Hardness*

Evaluated at SNL (Classes 1 and 2)

Class	Nominal Tungsten (wt %)	Density (g/cc)	HRC (max)
1	90	16.85-17.30	32
2	92.5	17.15-17.85	33
3	95	17.75-18.35	34
4	97	18.25-18.85	35

Assuming all porosity comes from the Ni-Cu binder*, we can calculate a max volume fraction of pores possible, such that the material still meets the requirements:

Ex. Class I:

For 90W-6Ni-4Cu	"Full Density" Volume %	Volume % with Pores	Density (g/cc)
W	80.0	80.0	19.3
Ni	11.8	8.7	8.91
Cu	7.4	5.4	8.96
Pores+	0	5 ← Max. Vol% Porosity	
Mass% W:	90.1	92.5	
WHA Density (g/cc)	17.03	16.85	

*ASTM B777

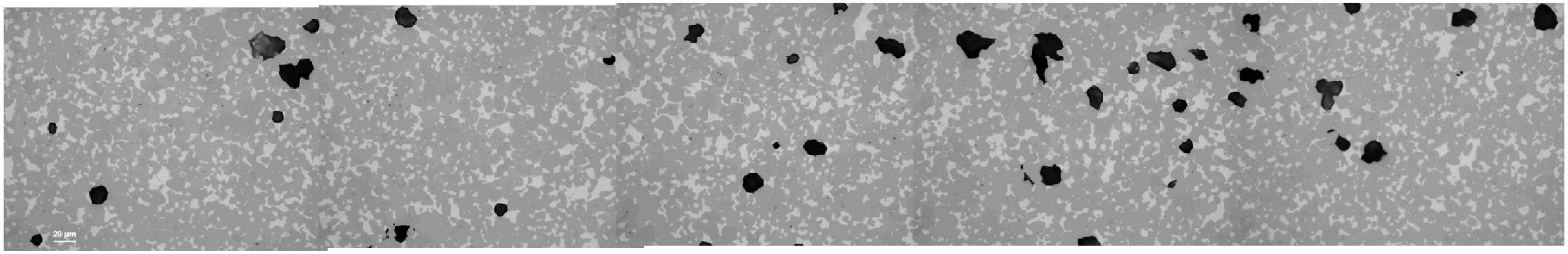
+*Note: This calculation also assumes that the composition of Ni-Cu binder has the same ratio as the added powder*

Class I: Porosity Measurements Using Quantitative Image Analysis

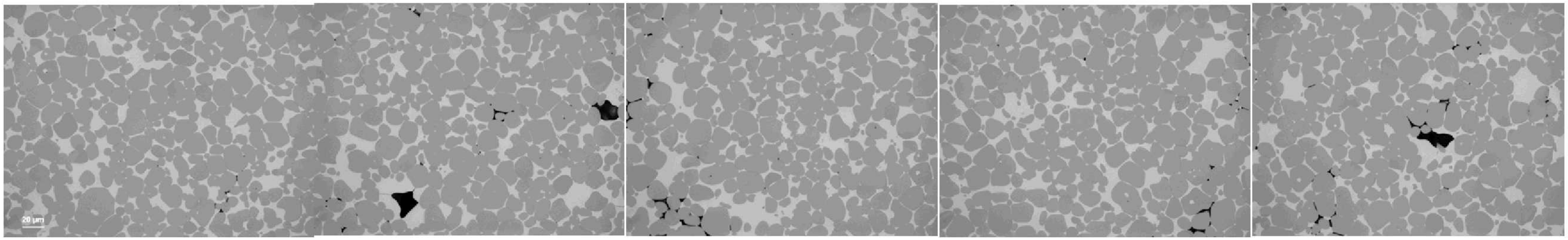
Porosity was measured on polished cross-sections on Class I WHA material using quantitative image analysis (via gray thresholding)

- Parts from two different material lots were assessed; notice the order of magnitude difference in porosity

High Porosity Specimen: 2.7% porosity



Low Porosity Specimen: 0.52% porosity

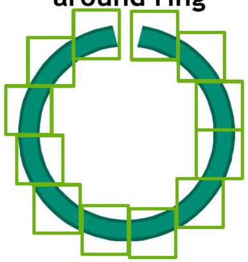


Class II: Measurement of Porosity

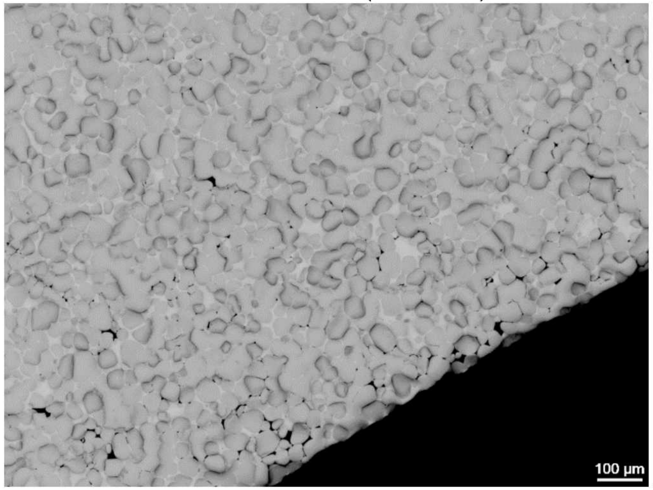
Class II material was measured on transverse and axial views of a sintered threaded ring

- Compared to the Class I material, the Class II material had a lower area percent porosity:
- Average Area Percent Porosity (Planar): **0.06%**
- Average Area Percent Porosity (Planar): **0.04%**

Images were taken around ring



Example image from Planar section of Class II Material (W-Ni-Cu)



Transverse section of Class II Material (W-Ni-Cu)

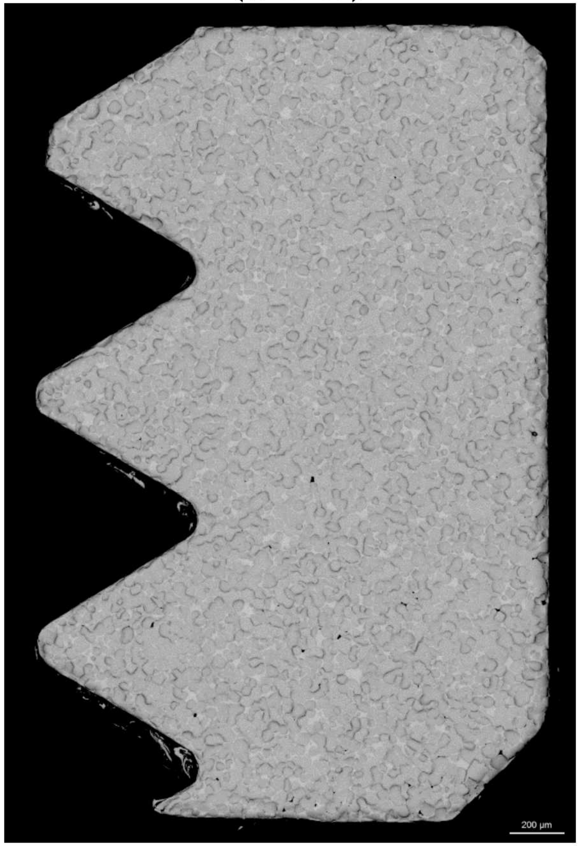
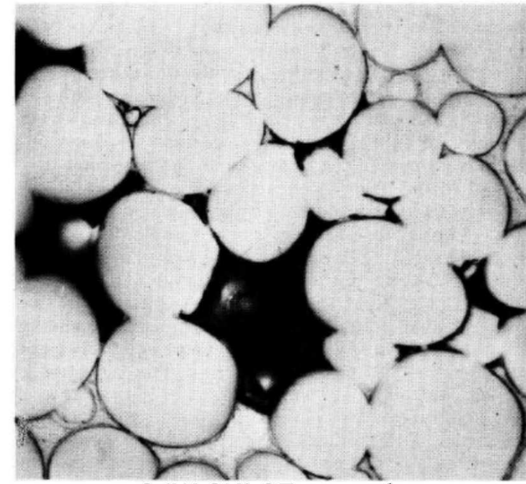


Image	Area Percent Porosity (%)
1	0.036
2	0.007
3	0.009
4	0.074
5	0.027
6	0.007
7	0.011
8	0.013
9	0.007
10	0.042
11	0.030
12	0.373
13	0.112
14	0.009
15	0.044
16	0.223
Avg. Area Percent Porosity	0.064
Transverse	0.044

Defect Implications: Possible Reduction in Mechanical Properties

Churn and Yoon reported that a 94W-3Ni-3Fe alloy, reduced fracture strength and elongation were observed with 1.25-2% porosity.

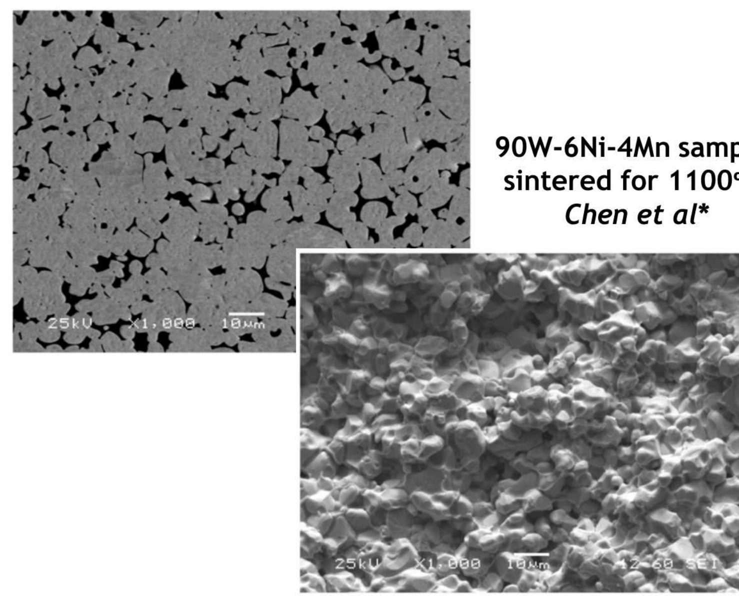
- Fracture strength decreased from 900 to 200MPa
- Elongation decreased from ~12% to 0%
- Attribute loss of properties to sharp tips in pores that act as stress concentrators



94W-3Ni-3Fe sample, sintered for 4hr at 1450°C (400x original magnification), *Churn and Yoon**

Chen *et al* reported that in solid state sintering (T=1100°C) of a 90W-6Ni-4Mn heavy alloy, insufficient bonding between the binder and the W particles, and significant porosity (5%), occurred.

- The 1100°C sample had a UTS of 61 ksi (426 MPa), and no measurable elongation
- The authors suggested that high porosity (lower density) responsible for lower stress failure
- The authors did not report porosity levels for specimens sintered at higher temperatures (when liquid phase sintering was achieved.



90W-6Ni-4Mn sample, sintered for 1100°C, *Chen et al**

Tensile Testing of Class I and Class II Material

Class I material was machined into small dogbones from the wall of a cylinder and tensile tested in a hydraulic load frame

- Both extensometer and digital image correlation (DIC) used to measure extension of sample. The max ductility (higher of the measurements) is reported.

Quarter inch round tensile specimens of Class II material were tested during product acceptance, and reported properties are listed for comparison.

Neither material examined met the ASTM B777 mechanical property requirements

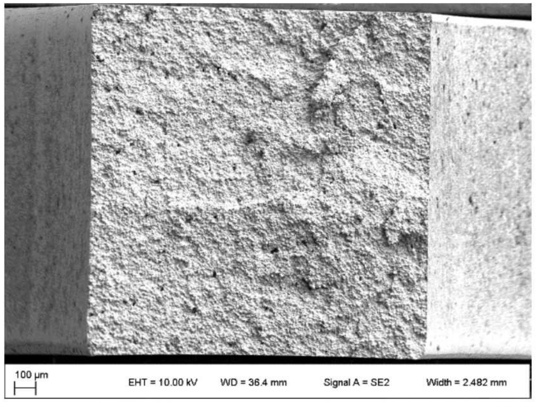
Class I, High porosity sample

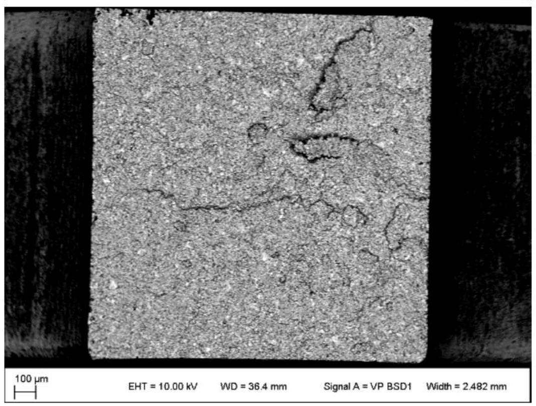
Specimen	Avg. Percent Porosity (QIA)	Avg. UTS, ksi (MPa)	Avg. Maximum Ductility (%)
Class I: High Porosity	2.7	56.1 (386.9)	0.25
Class I: Low Porosity	0.51	59.1 (407.2)	0.38
Class II (acceptance test)	0.06	89.8 (619.1)	0.75
ASTM B777, Class I & II		*94 (648)*	2

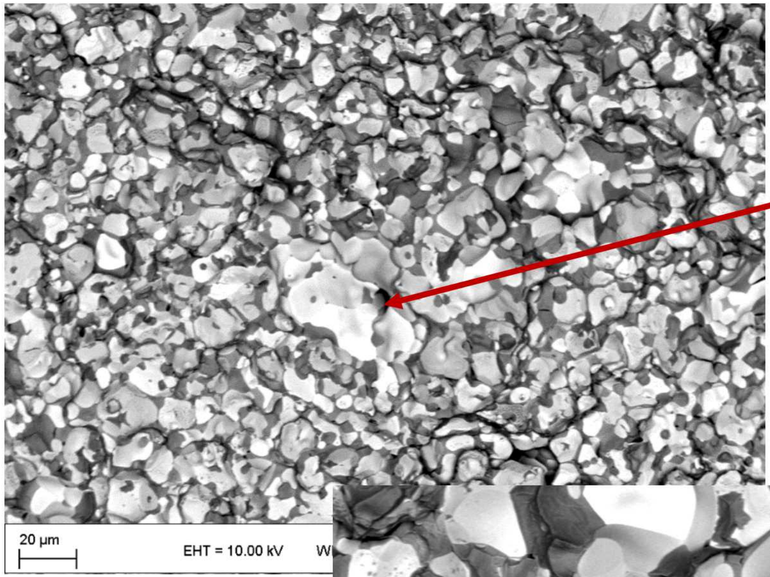
Special thanks to Zach Casias and Jay Carroll for performing tensile tests

Class I: Porosity on Fracture Surfaces of Tensile Specimens

Class I, High Porosity Sample

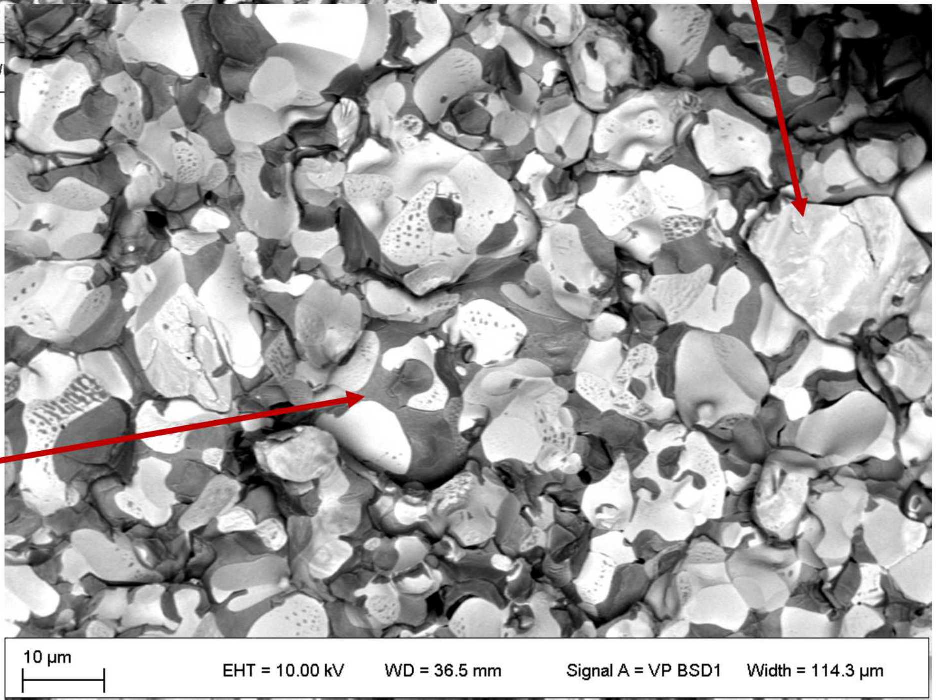






Faceted "pores", with W particles at bottom

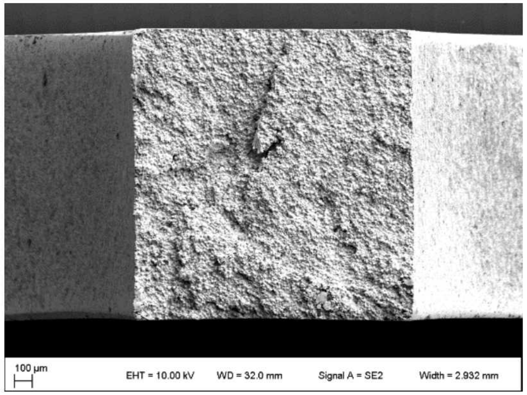
Example of sheared W particle, indicates region where particle-binder bond was strong

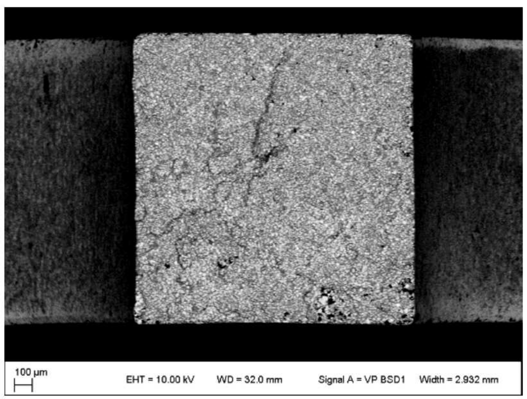


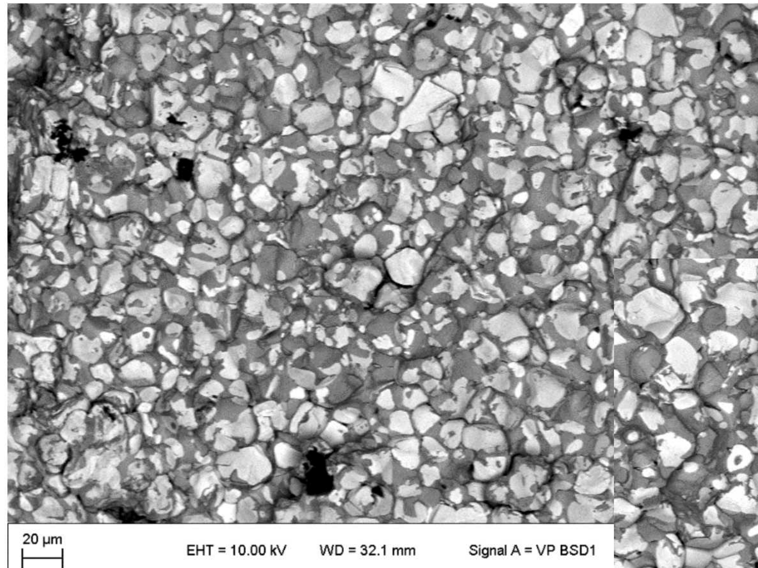
W particles covered with some Ni-Cu binder

Class I: Ductile Tearing of Binder Observed on Fracture Surfaces

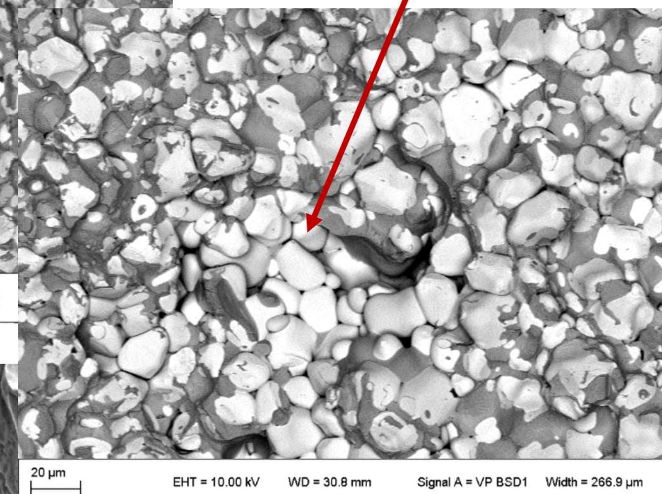
Class I, Low Porosity Sample



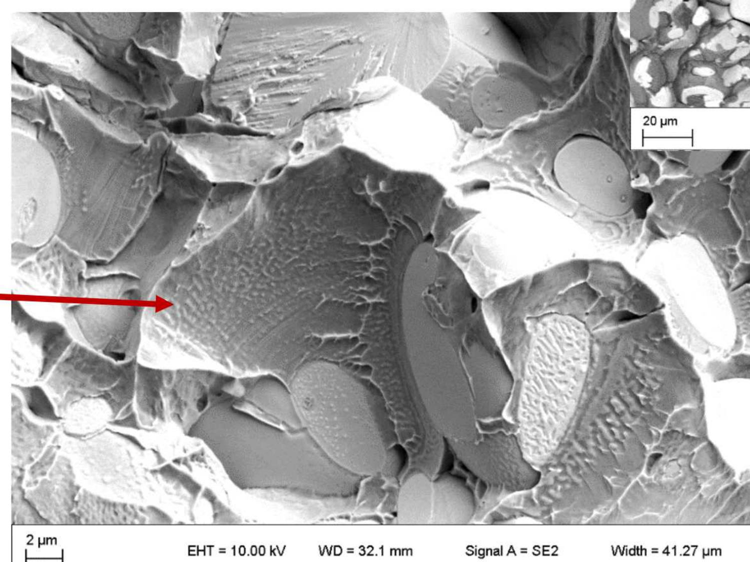




Rounded W particles, not covered with binder - indicative of pore



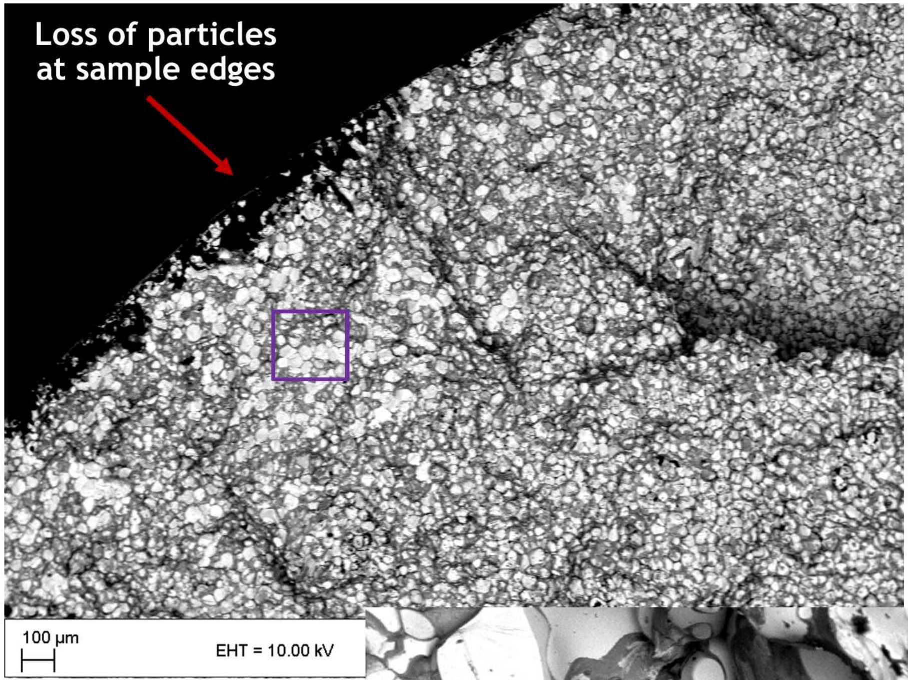
Ductile tearing of Ni-Cu binder



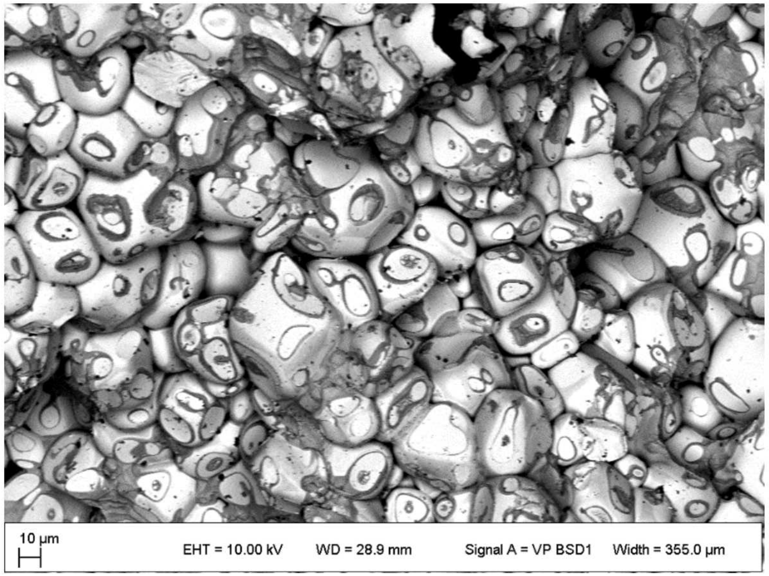
Class I material has the lowest density (i.e. highest binder content), and increased elongation due to ductility of binder alloy.

Class II: Less Binder on Tensile Specimen Fracture Surfaces

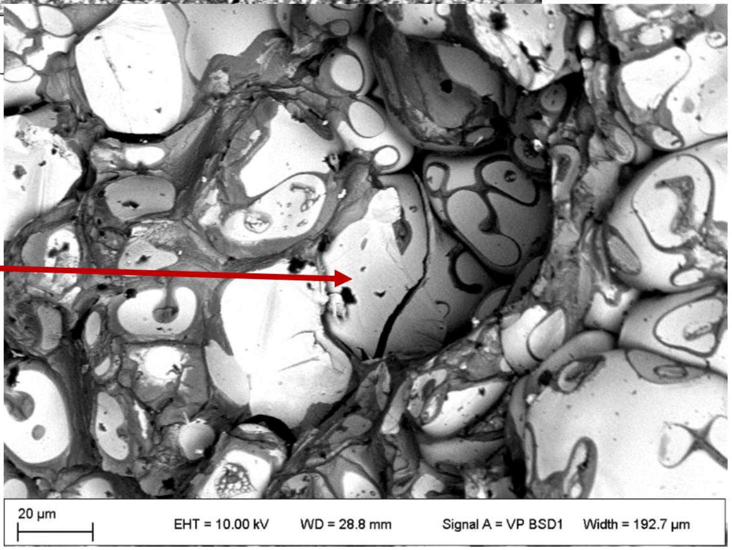
Loss of particles at sample edges



Rounded W particles, have less binder coverage compared to Class I



Cracking and shearing of W more prevalent in Class II material



Class II has higher W content (and increased density), but less binder material compared to Class I material. Thus, less coverage of W particles, and less binder ductility is also observed.

Summary

Preliminary characterization of Class I and Class II W-Ni-Cu WHA was performed

- Class I had 0.52-2.7% porosity, while Class II had much lower 0.04-0.06% porosity

Minor differences in mechanical properties were observed in Class I material, despite the noticeable difference in porosity

Fracture surfaces of both Class I and Class II material were examined

- Ductile tearing of the Ni-Cu binder was observed in Class I material, while more shearing of W particles appeared to be prevalent in Class I material

Future Work

Compositional analysis of the Ni-Cu binder for refinement of density calculations

- Refined calculations may lead to a more accurate maximum porosity threshold

Mechanical testing of different sample geometries to better understand the contribution of porosity to mechanical behavior

- Is there are pore size effect that is more pronounced in smaller specimens?

Characterization of additional Class I and Class II material to look further into particle size, pore size, and pore fraction

Outline

1. Main properties of interest
    1. Density
2. Fabrication/synthesis
3. Microstructure
    1. Composition/distribution of particles
4. Liquid vs solid state sintering
    1. Porosity
    2. Particle agglomeration
5. Linear defects – what causes them?
6. Tensile properties
7. Plating issues/tribology properties