Refractory Alloys
(collaboration with Naval Reactor Program)

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Three systems being studies:

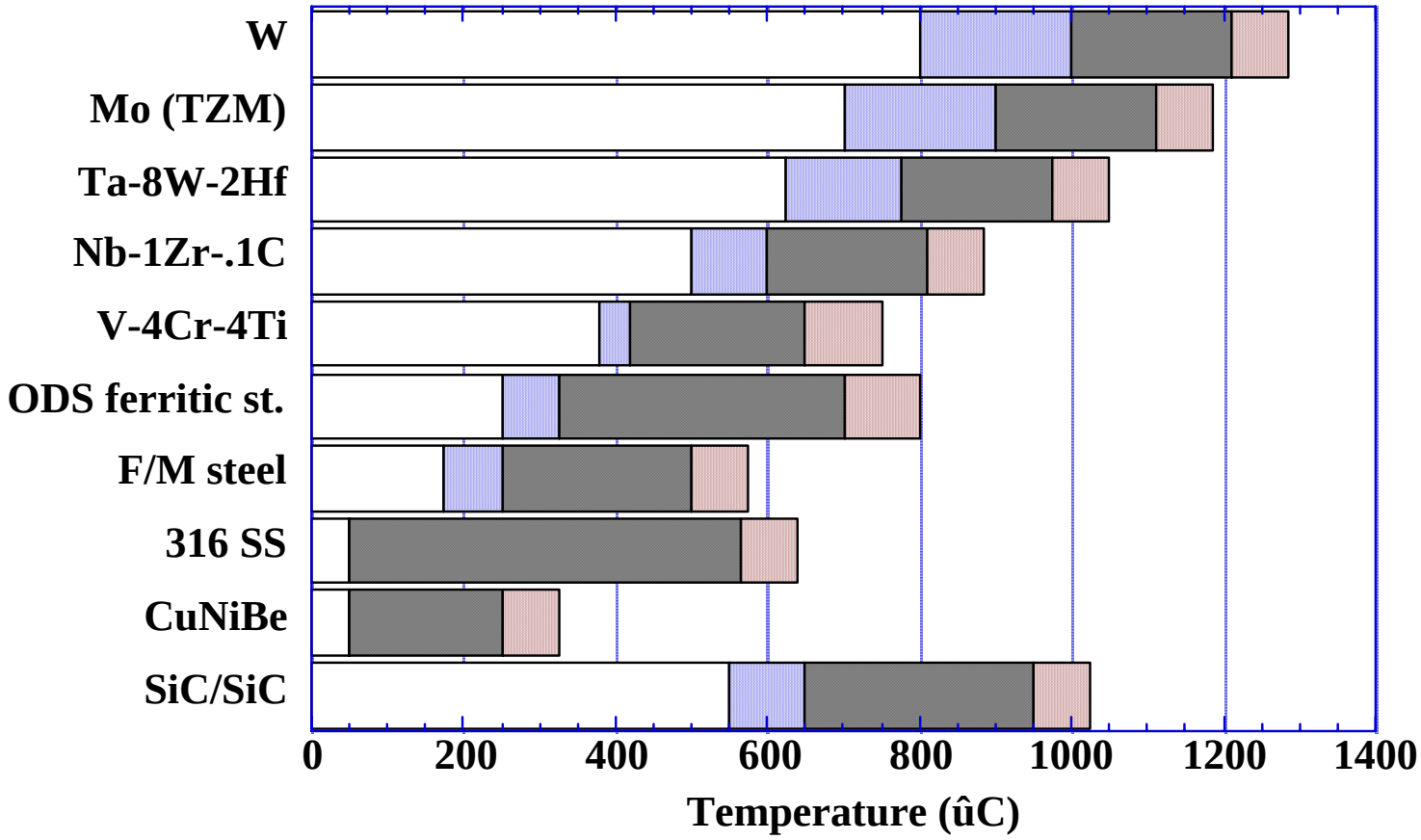
- SiC/SiC composites (2D, stoichiometric fibers)
- Molybdenum alloys (LCAC,ODS, and advanced alloys)
- Superalloys (MA-956, INOR)

Studies include:

- Radiation effects and processing of SiC
  Tirr 300-1600°C, up to 10 dpa
- Radiation effects on Superalloys, ODS and LCAC moly
  Tirr 300-1200°C, up to 15 dpa
- Processing of molybdenum for enhanced ductility
- Welding of molybdenum

Multiyear, restricted program : 2-3 M$/yr

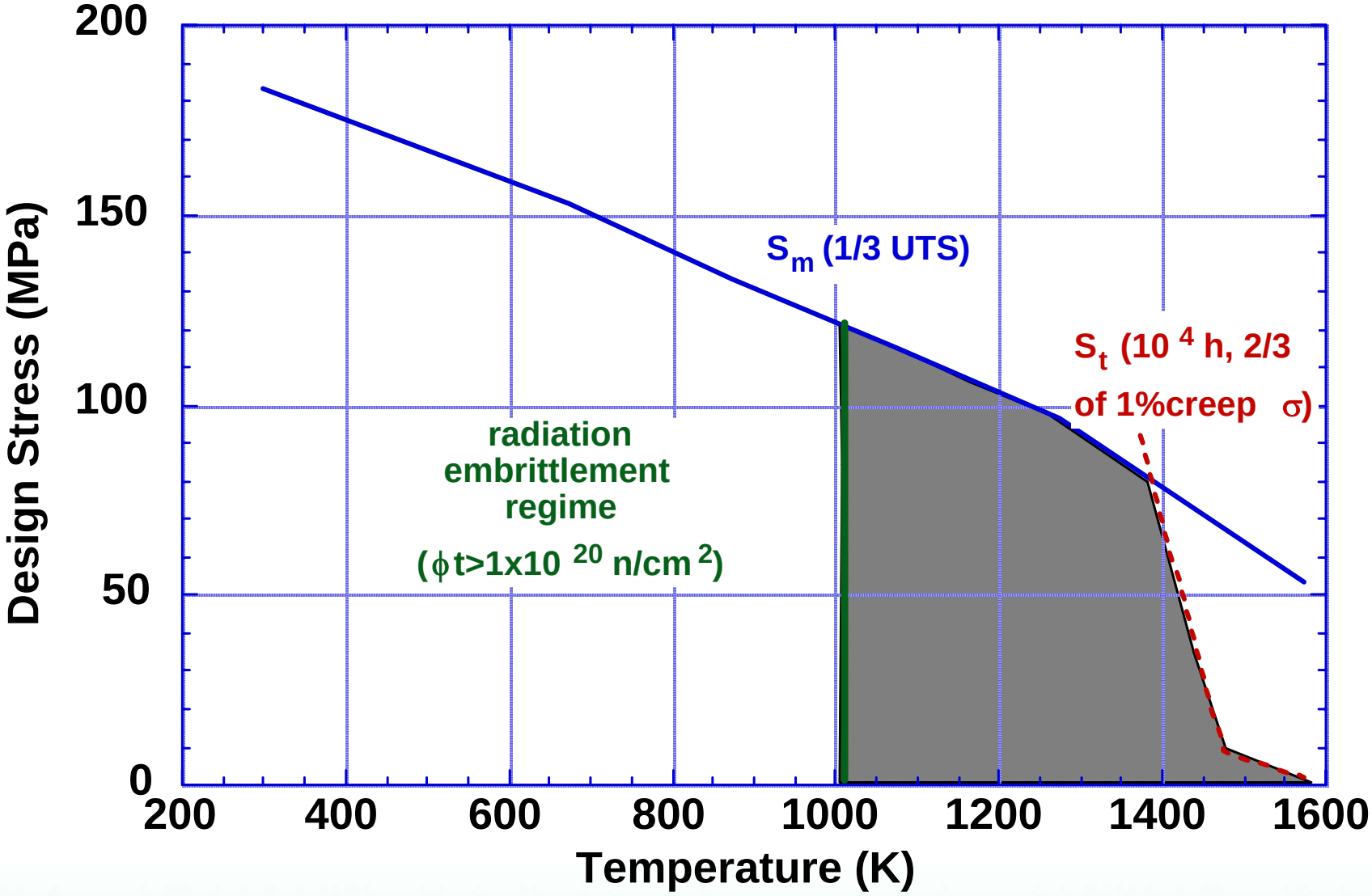
Operating Temperature Windows for Structural Alloys



• Lower temperature limit of alloys based on radiation hardening/ fracture toughness embrittlement (K_{1C}< ~30 MPa-$m^{1/2}$)—large uncertainty for W,Mo due to lack of data
• Upper temperature limit based on 150 MPa creep strength (1% in 1000 h); chemical compatibility considerations may cause further decreases in the max operating temp.

S.J. Zinkle and N.M. Ghoniem
Fus. Eng. Des. 51-52 (2000) 55.

Structural Design Window for TZM Mo Alloy



Naval Reactor (Bettis/KAPL) Moly Irradiation

Completed

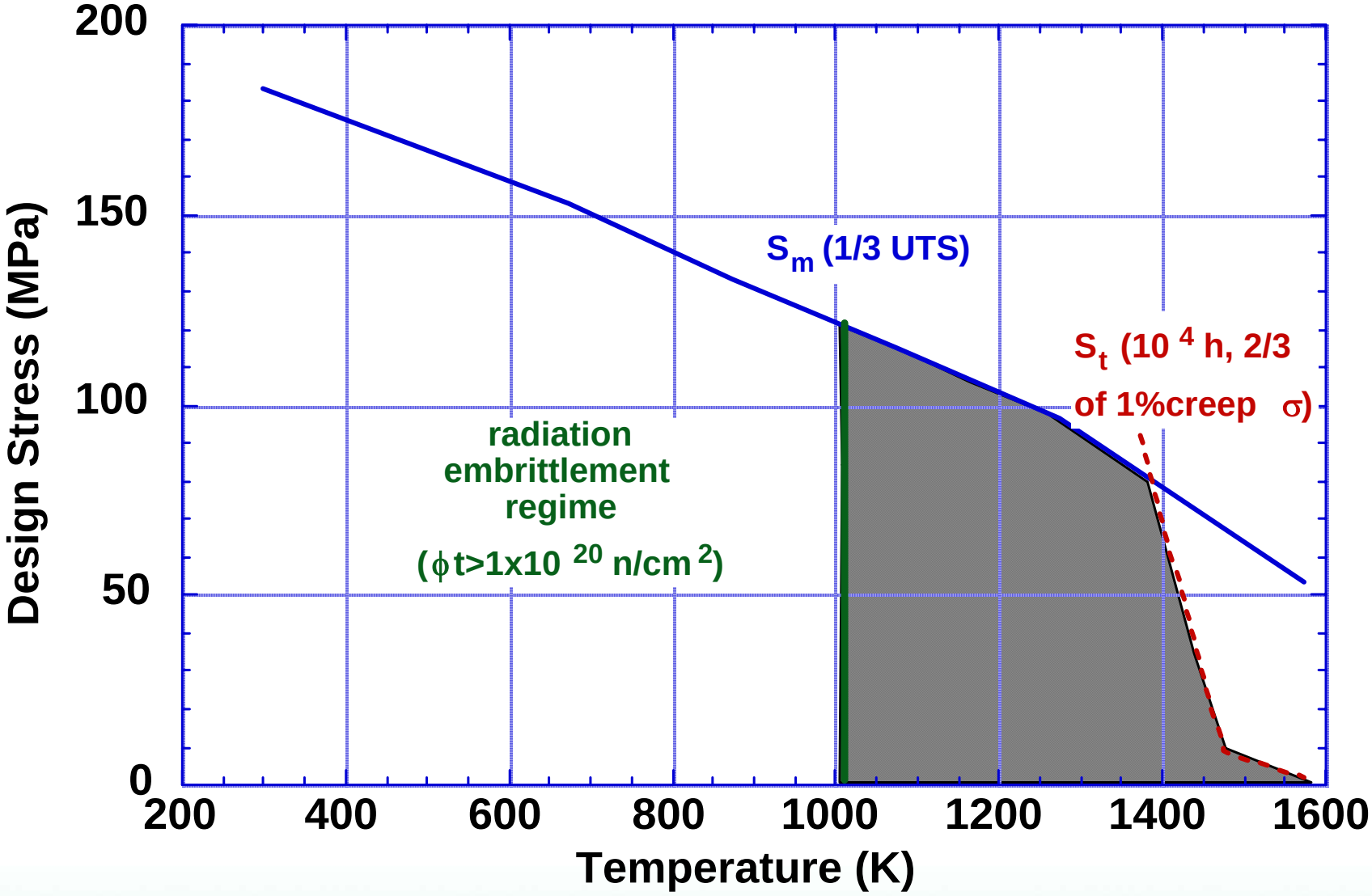
- Materials : Low Carbon Arc Cast Moly (Climax Metals)
  ODS (yttria oxide) Moly
  »
- Irradiation : 300 - 1200°C in HFIR PTP rabbits
  - 1-6 E 25 n/m2 (E>0.1 MeV)

Underway

- Materials : Low Carbon Arc Cast Moly (Climax Metals)
  ODS (yttria oxide) Moly
  TZM, Isotopically tailored, Zr-B alloys
- Irradiation : 300 - 1200°C in HFIR PTP rabbits
  - Up to 20 E 25 n/m2 (E>0.1 MeV)

Tools : Tensile (RT, IT), Creep(ATR), Hardness, SEM, TEM, Atom Probe, Swelling

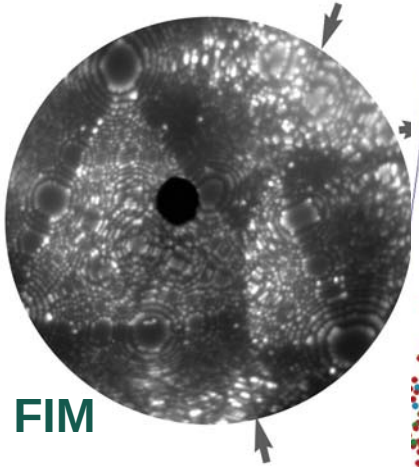
Structural Design Window for TZM Mo Alloy



ATOM PROBE TOMOGRAPHY REVEALS Zr, B and C SEGREGATION TO THE GRAIN BOUNDARIES

- **Base metal: Zr, C and B enrichments and O depletion**
- **HAZ: Heavy B and moderate Zr enrichments**

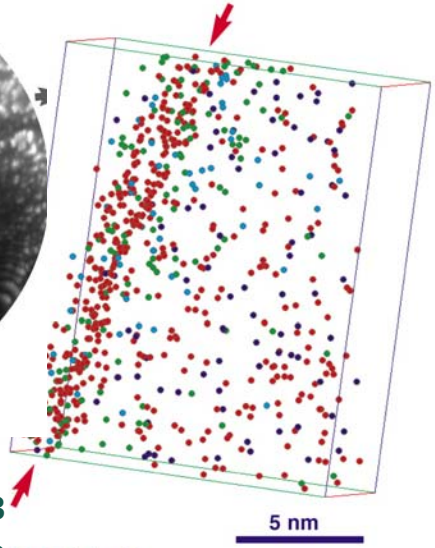
BASE METAL



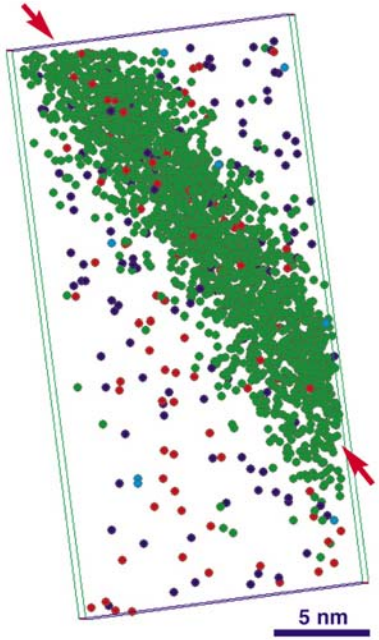
FIM

GIE (atoms m^{-2})

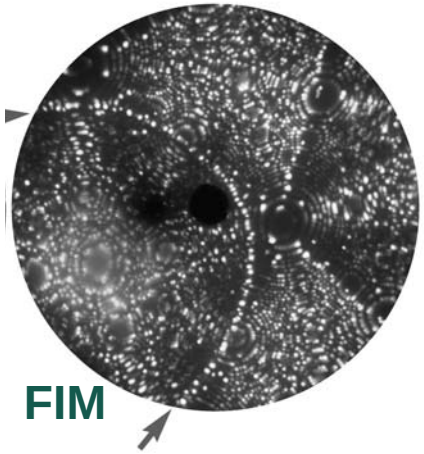
Zr	7.6×10^{13}
B	7.3×10^{12}
C	1.1×10^{13}
O	-3.9×10^{12}



HEAT AFFECTED ZONE



APT atom maps



FIM

GIE (atoms m^{-2})

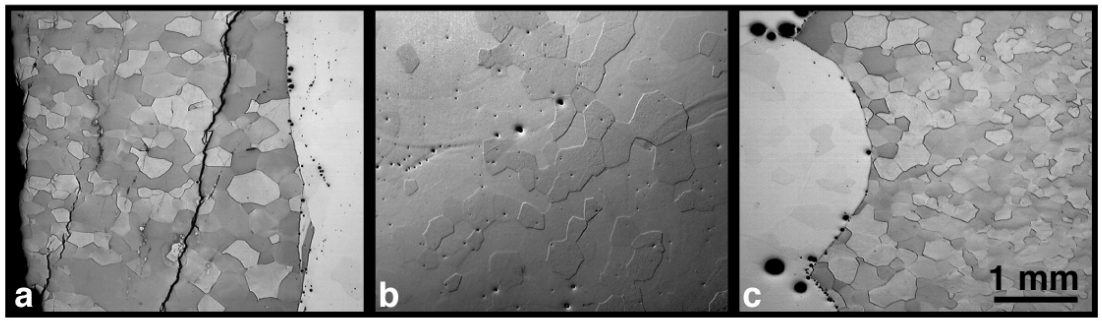
Zr	1.3×10^{13}
B	9.9×10^{14}
C	9.9×10^{11}
O	1.1×10^{13}

Research performed by
M. K. Miller, Oak Ridge National Laboratory
and A. J. Bryhan, Applied Materials

IMPROVEMENTS IN THE DUCTILITY OF MOLYBDENUM WELDMENTS BY ALLOYING ADDITIONS OF Zr, B and C

ALLOY COMPOSITION

Zr	1600 appm
C	96 appm
B	53 appm
O	250 appm
N	178 appm
Mo	balance



HAZ Weld HAZ

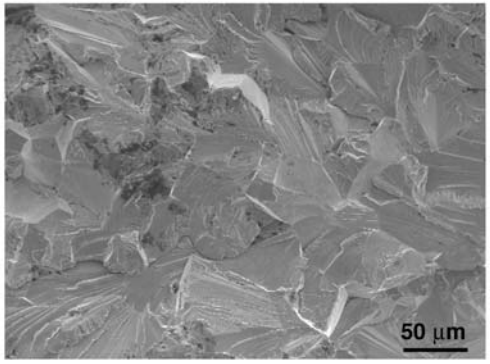
Fracture occurred predominantly in the heat affected zones

MECHANICAL PROPERTIES

Strain rate: 8.3×10^{-4} s^{-1}
Mo- 30% Re filler

Ductility	19.5%
Yield Stress	481 MPa
UTS	544 MPa

Commercial Mo weld: 3% Ductility



The fracture mode was transgranular cleavage with only small regions of intergranular fracture. This contrasts the intergranular fracture typically found in commercial Mo welds.

Research performed by
M. K. Miller, Oak Ridge National Laboratory
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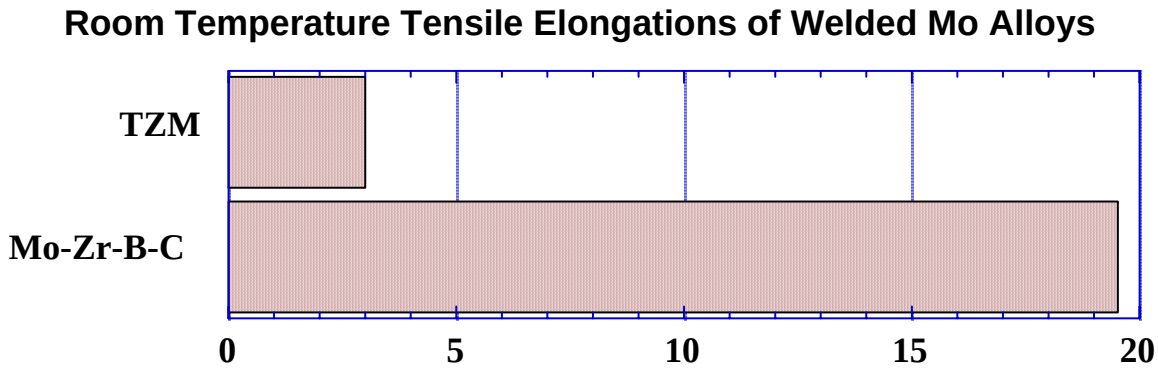
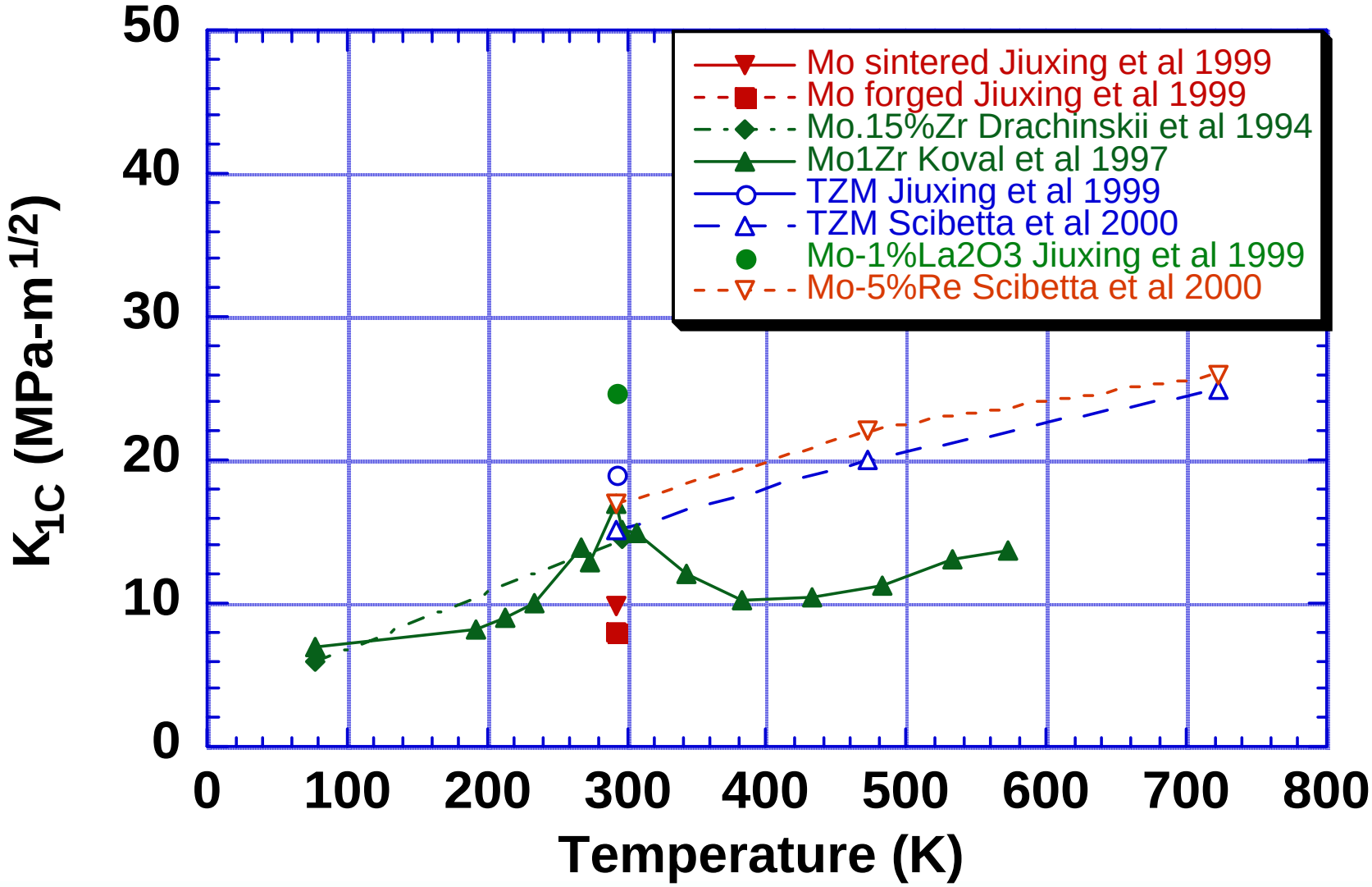
Recent research offers promise for developing refractory alloys with improved ductility

- Controlled (50-1600 appm) additions of Zr, B, C to molybdenum increases room temperature ductility of weldments from nearly zero to e_{tot}~20% (M.K.Miller and A.J.Bryhan, 2001)



- Mechanically alloyed W-0.3wt%Ti-0.05wt%C (H. Kurishita et al., ICFRM-10, Baden-Baden, Oct. 2001)
  - Avoid $(W,Ti)_2C$ brittle phase by limiting max concentration of carbon
  - Small grain size (~2 µm) helps to dilute harmful oxygen grain boundary segregation
  - TiC dispersed particles provide increased toughness (appropriate fracture mechanics tests needed to verify preliminary smooth bend bar results)

Summary of Fracture Toughness Data for Mo Alloys



Vacuum Arc Remelted Mo-Re Alloys Have Been Produced and Fabricated for Thermal-to-Electric Power Conversion Systems

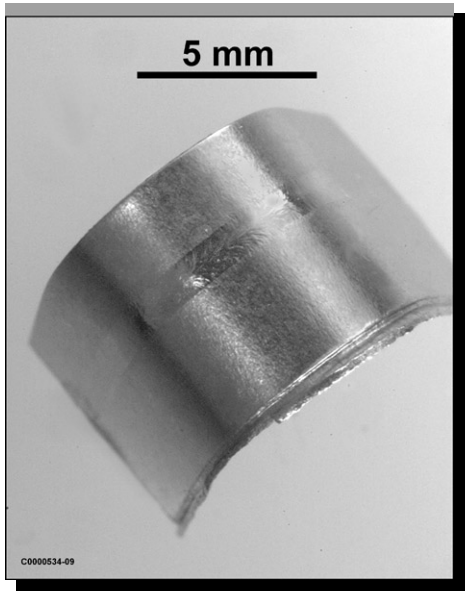
Ingots of arc melted Mo-Re are routinely processed from powder

These ingots have been fabricated into precision components by extrusion, rolling, and machining

Vacuum Arc Remelted Mo-Re Has Attractive Properties for Thermal-to-Electric Power Conversion Systems

- Weldability **– VAR Mo-Re has exhibited good weldability and weldment ductility**

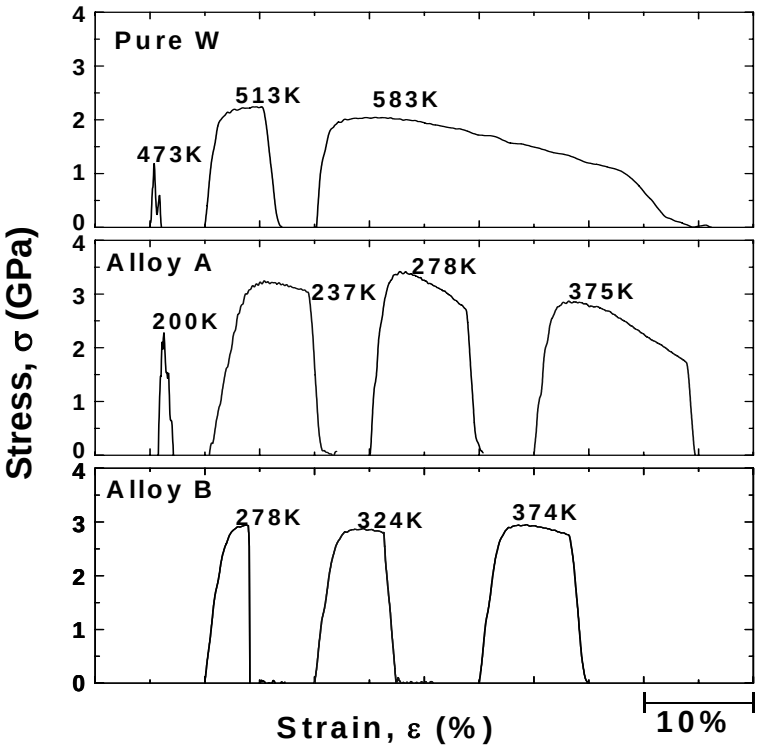
- Oxidation Resistance – Research has indicated that Mo-Re is more oxidation resistant than Nb and Ta alloys



- Sodium Compatibility **– Mo-Re has better compatibility than Nb-1Zr because it is not subjected to attack by impurities in Na at the operating temperatures of 350°C to 950°C**

Mechanically alloyed W-0.3wt%Ti-0.05wt%C exhibits good low temperature ductility

Example of the as-rolled sheets.



Three-point bending stress-strain curves for pure tungsten and developed alloys A and B.

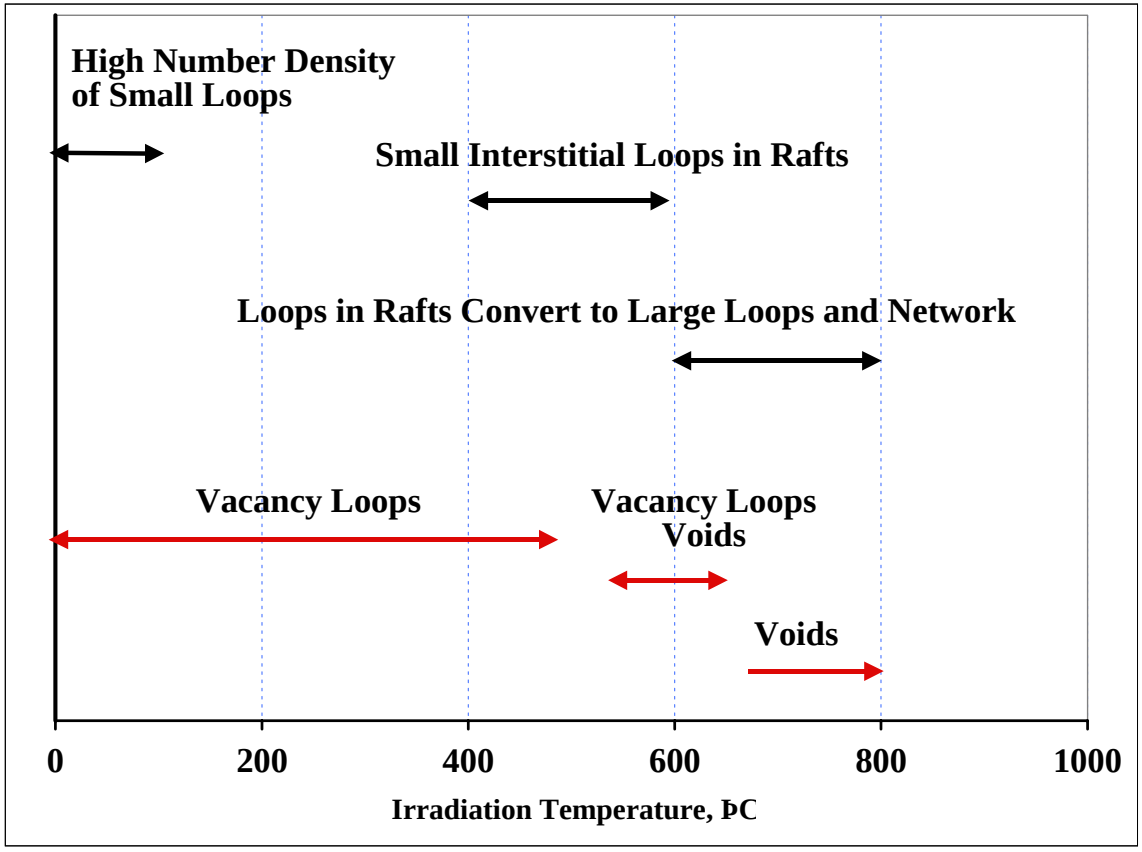
Comparison of test temperature dependence of total absorbed energy and maximum strength among alloy A, W-0.2wt%TiC and pure tungsten (un-notched bend bar impact tests).

H. Kurishita et al., 2001

Low-Dose Neutron-Irradiated Microstructure in Mo

3-6 x 10^{23} n/m^2 E>1 MeV

Brimhall/Mastel 1970



High-Dose Damage in Neutron-Irradiated Mo

1×10^{26} n/m^2 E>1MeV

Sikka/Moteff 1974

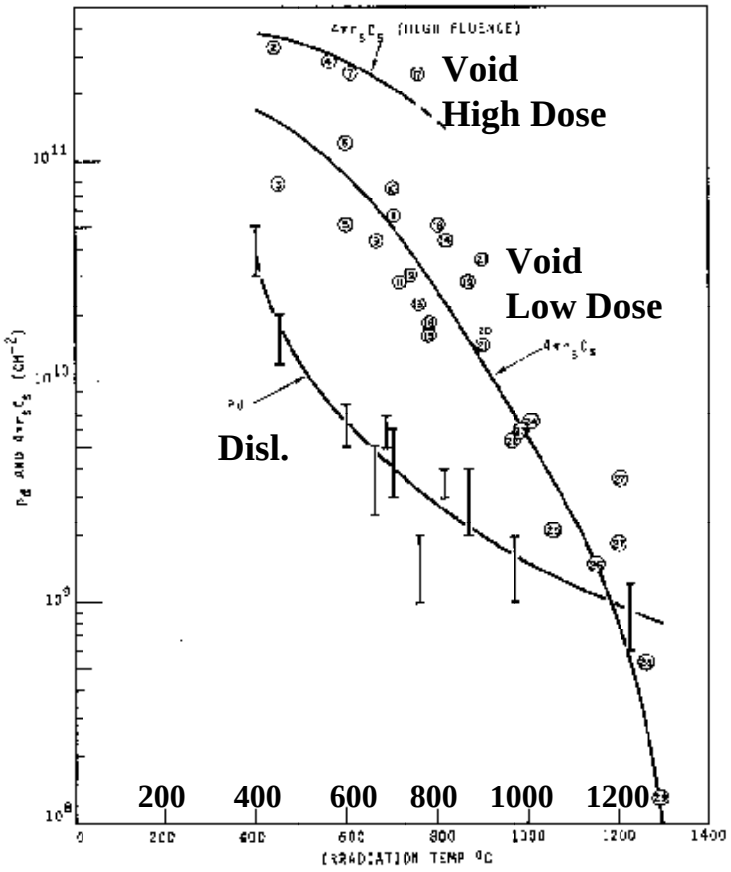
Damage	430 ˚C	580 ˚C	700 ˚C	800 ˚C	900 ˚C	1000 ˚C
Rafts	observed					
Black Spot Clusters	observed	observed				
Loops	small	average	average	large	very large	
Voids	ordered	ordered	ordered	partial order	partial order	partial order
Dislocations	observed	observed	observed	observed	observed	observed

Neutron-Irradiated Microstructural Sink Strength in Mo

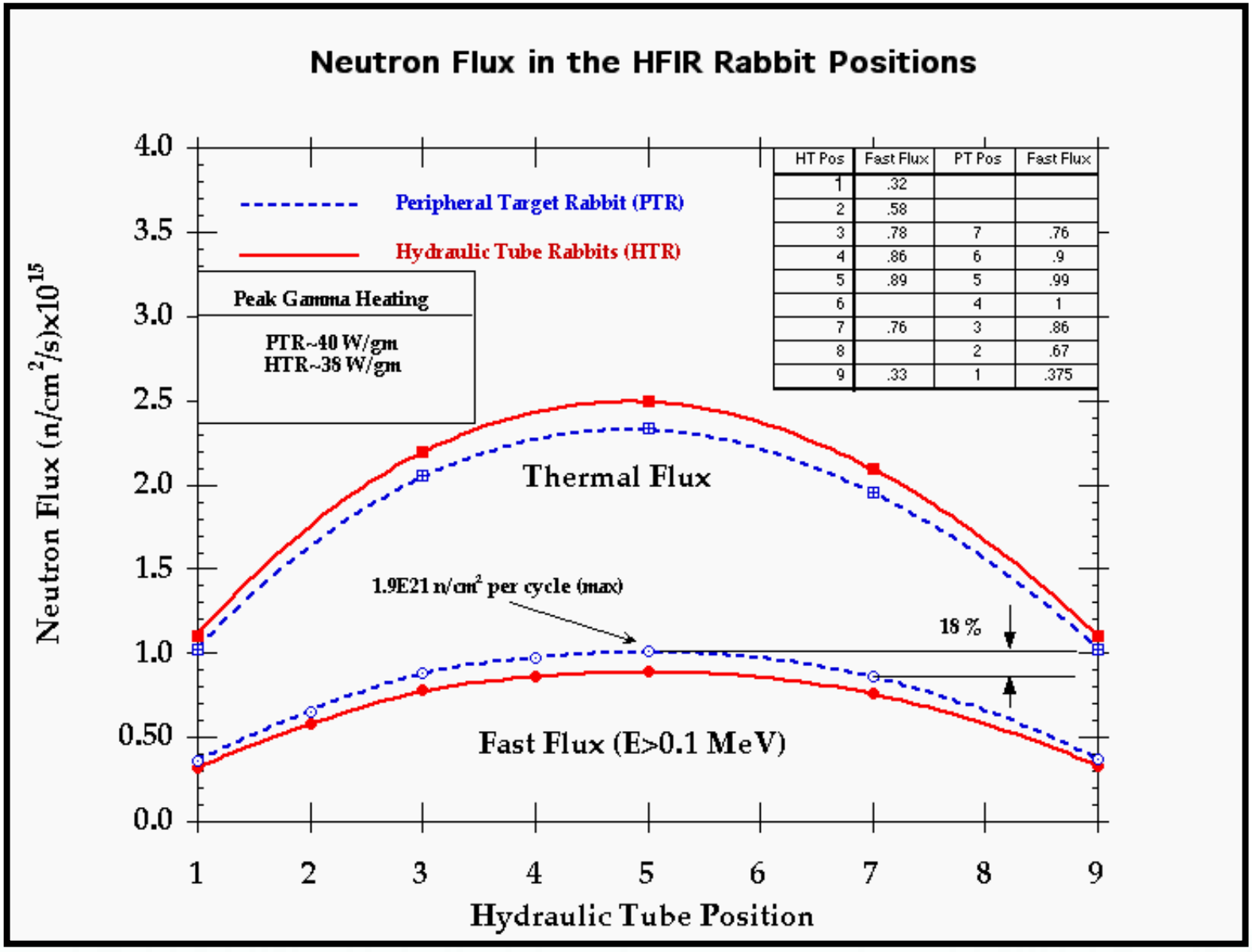
Temperature Dependence

Brimhall 1973

Sink Strength



Irradiation Temperature, ˚C



HT Pos	Fast Flux	PT Pos	Fast Flux
1	.32		
2	.58		
3	.78	7	.76
4	.86	6	.9
5	.89	5	.99
6		4	1
7	.76	3	.86
8		2	.67
9	.33	1	.375