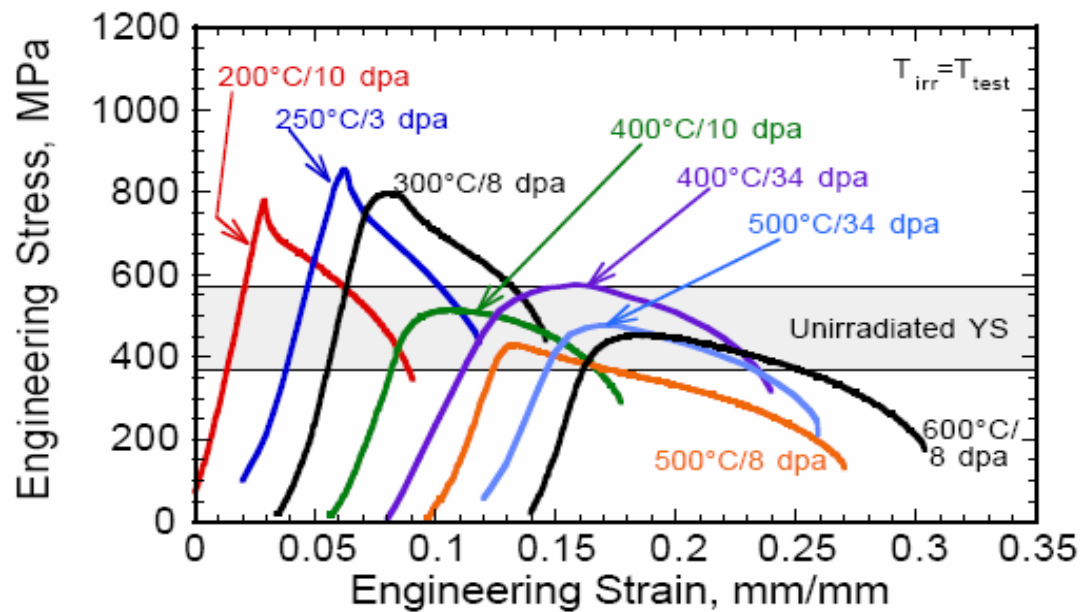


**Material:** Ferritic Steel: F82H  
**Property:** Engineering Stress vs. Engineering Strain  
**Condition:** Irradiated  
**Data:** Experimental



**Source:**

Fusion Materials Sciences Peer Review, University of California Santa Barbara  
(August 27-28, 2001)

**Title of paper (or report) this figure appeared in:**

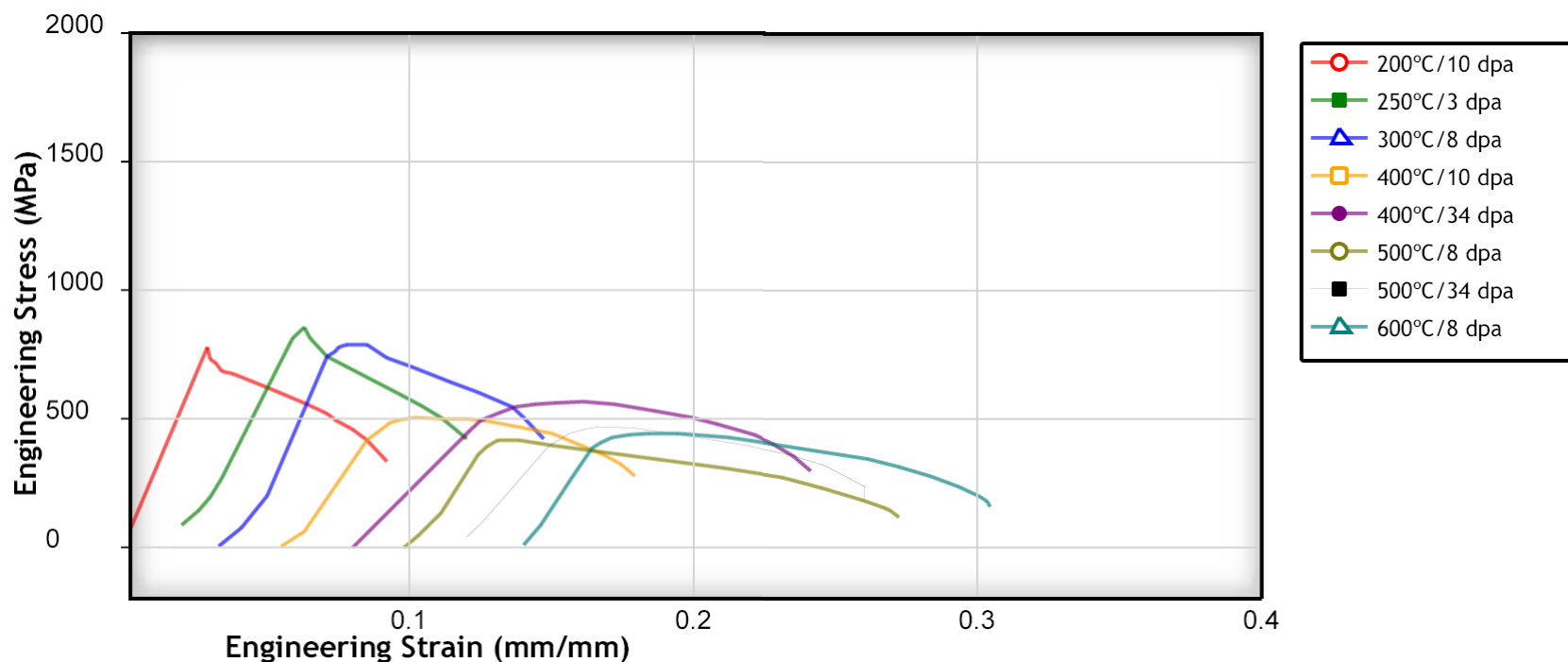
Overview of Current BCC Structural Alloys

**Author of paper or graph:**

A.F. Rowcliffe

**Caption:**

Stress-Strain Curves for Neutron Irradiated F82H



**Stress-Strain Curves for Neutron Irradiated F82H ( $T_{\text{irr}} = T_{\text{test}}$ )**

**Reference:**

**Author:** A.F. Rowcliffe

**Title:** Overview of Current BCC Structural Alloys

**Source:** Fusion Materials Sciences Peer Review, University of California Santa Barbara  
(August 27-28, 2001), [\[PDF\]](#)

[View Data](#)

[Author Comments](#)

**Plot Format:**

**Y-Scale:** ☒ linear ☐ log ☐ ln

**X-Scale:** ☒ linear ☐ log ☐ ln

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# **Overview of Current BCC Structural Alloys**

**Presented by  
A.F. Rowcliffe  
Oak Ridge National Laboratory**

**Contributors:**  
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G.R. Odette (UCSB)  
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D.L. Smith (ANL)

**Fusion Materials Sciences Peer Review**  
August 27<sup>th</sup>-28<sup>th</sup>, 2001  
University of California Santa Barbara

# Development of Current BCC Alloy Compositions

- Current alloy compositions evolved from strong U.S. leadership in the development of reduced activation options for Tempered Martensitic Steels (TMS) and Vanadium alloys
- Empirically-based development programs in the U.S., EU and Japan have addressed wide range of issues
  - reduced activation (safety, waste disposal)
  - fabrication/joining
  - mechanical behavior and thermophysical properties
  - radiation damage and helium effects
  - chemical compatibility and corrosion

# Composition of Tempered Martensitic Steels (TMS)

- Current primary compositions have evolved empirically from studies on a wide range of compositions

USA	EU	Japan
2-9Cr-V	9Cr-W-V-Ta-N	2-15Cr-W
2-9Cr-W	12Cr-W-V-Ta-N	2-3Cr-W-V-Ta
2-12Cr-W-V	9-10Cr-W-V-Ta-Ti-Ce	7-9Cr-W-V-Ta
9Cr-W-Mn	9Cr-W-V-Mn-Ti	11Cr-W-V-Ta
9Cr-V-Mn		
12Cr-W-Mn		
12Cr-V-Mn		

# Current Composition of US Program Model TMS

- Current compositions are all variants of the US-developed alloy
- 5000kg Heat of F82H is source of U.S. program model TMS

	Designation	Cr	W	V	Ta	Si	Mn	C	N
USA	9C-2WVTa	9.0	2.0	0.25	0.07	0.03	0.60	0.1	0.01
Japan	F82H	8.0	2.0	0.20	0.04	0.20	.050	0.1	<0.01
	JLF-1	9.0	2.0	0.20	0.07	0.08	0.65	0.1	0.05
EU	EUROFER	8.5	1.1	0.25	0.08	0.05	0.50	0.15	0.03

# Composition of US Program Model Vanadium Alloy

- Model V-4Cr-4Ti, composition evolved empirically from US-led studies covering a wide range of compositions

**Substitutional:** Cr(0-20); Ti (1-15); Si(0-1)  
(wt%)

**Interstitial:** C(50-100); O(100-500); N(50-100)  
(wt.ppm)

- Composition of U.S. 500kg heat

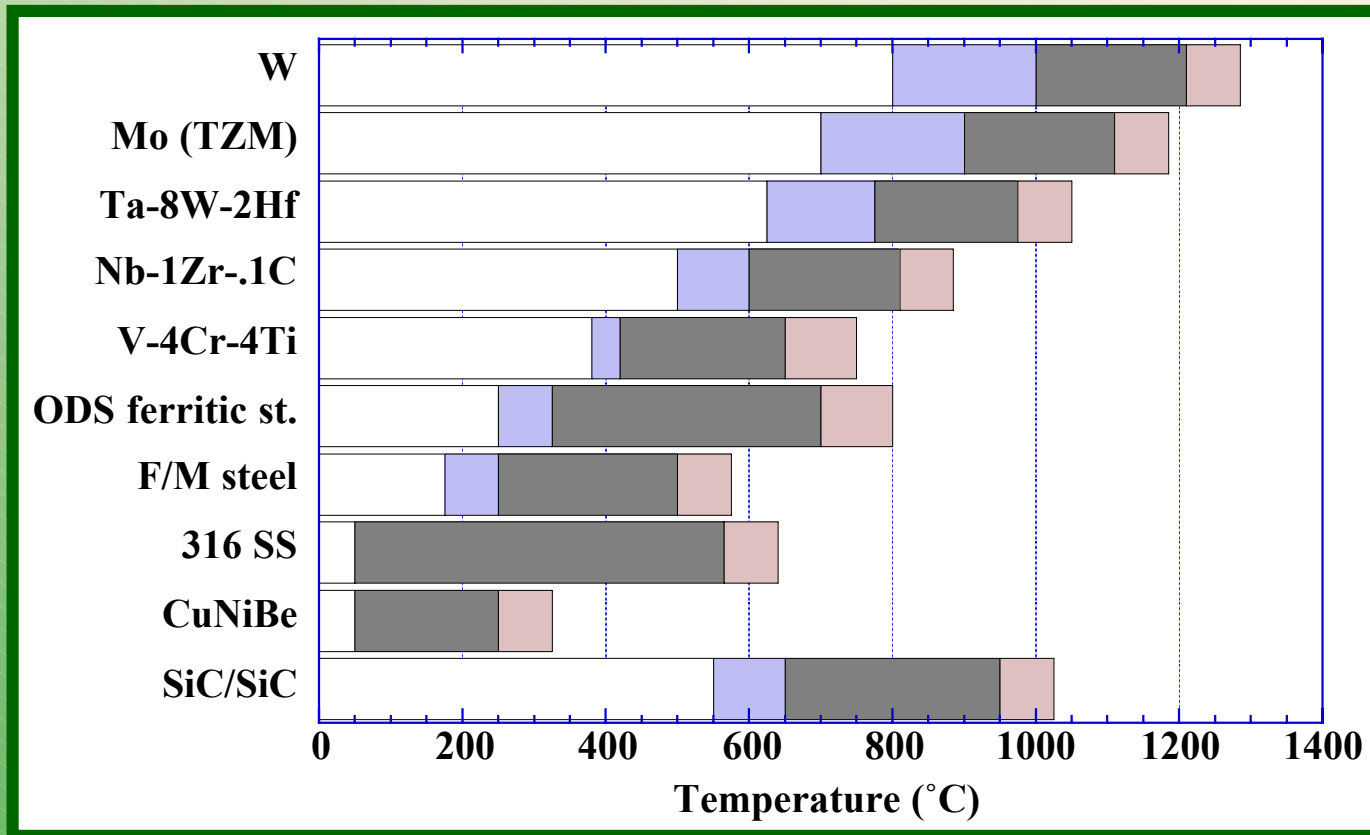
<u>Cr</u>	<u>Ti</u>	<u>C</u>	<u>O</u>	<u>N</u>
(wt%)		(wt.ppm)		
3.8	3.9	80	310	85

# **TMS and V alloys are the Focus of International Programs Co-ordinated through IEA Working Groups**

- **EU**
  - Large program on TMS
  - Materials engineering data base for 2010 DEMO breeding blanket, Pb-Li and water-cooled options
  - No technological interest in V
- **Japan**
  - Program on TMS led by JAERI
  - Materials engineering data base for 2015 DEMO, Water-cooled blanket (SSTR)
  - Program on V Alloys led by MONBUSHO
  - Materials development phase for a Li-cooled blanket (ARIES-RS)
- **US**
  - Advancement of materials science base for BCC model structural alloys; integrated theory/modeling/experimental approach to resolve feasibility issues; development of innovative materials



# Operating Temperature Windows for Structural Alloys in Fusion Reactors



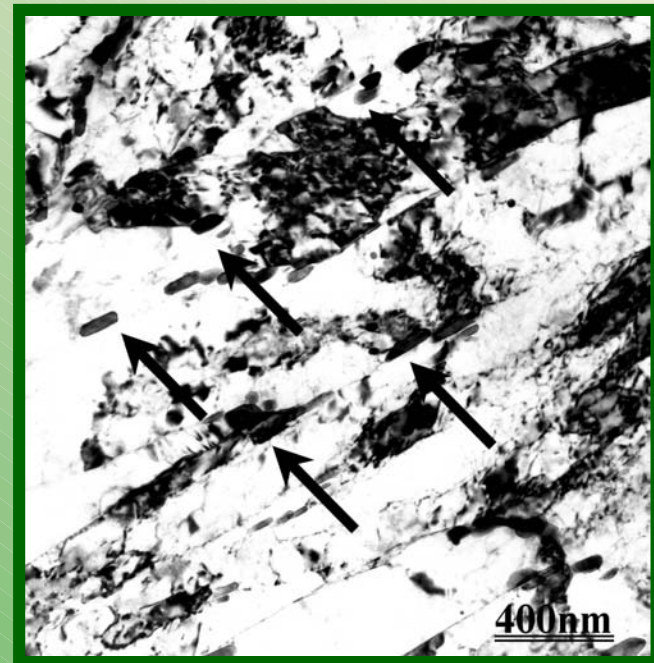
- Lower temperature limit of alloys based on radiation hardening/ fracture toughness embrittlement ( $K_{1C} < \sim 30 \text{ MPa}\cdot\text{m}^{1/2}$ )—large uncertainty for W, Mo due to lack of data
- Upper temperature limit based on 150 MPa creep strength (1% in 1000 h); chemical compatibility considerations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

# Critical Performance-Limiting Phenomena in BCC Alloys

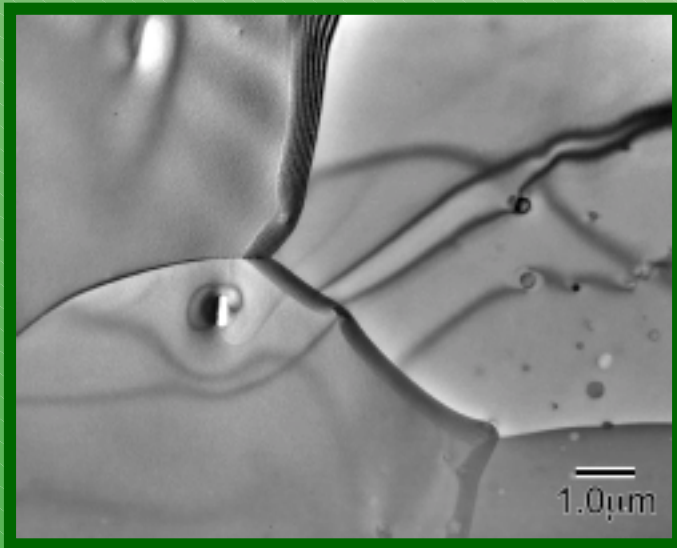
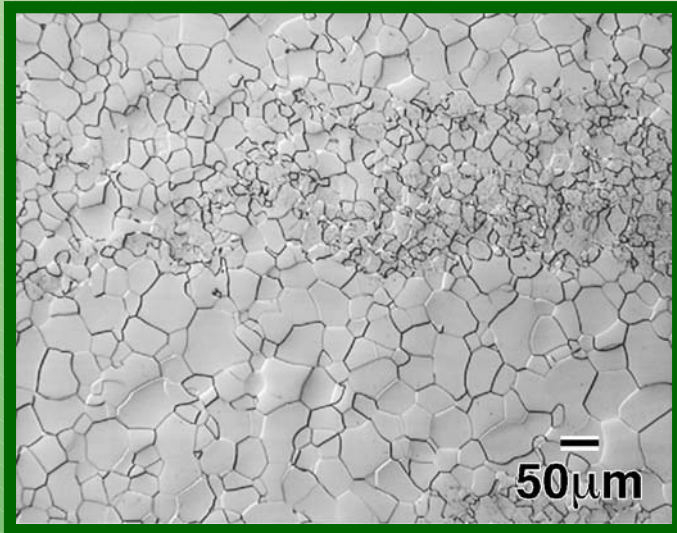
- **Low Temperature Regime:**
  - Radiation hardening and flow localization
  - Hardening-induced shifts in fracture toughness transition temperature
  - Effects of helium and hydrogen generation on fracture properties
- **High Temperature Regime:**
  - Loss of creep strength in TMS due to recovery of martensite structure
  - Loss of creep strength in V-4CrO<sub>4</sub>Ti; low barrier density
  - Helium-induced swelling and grain-boundary embrittlement

# Processing and Start-of-Life Microstructure for F82H

- **Processing**
  - Hot rolling in the austenite range at 1200°C-1050°C
  - Normalizing at 1040°C (fully austenitic); martensitic transformation on cooling; tempering at 740°C (below  $A_c$ ).
- **Microstructure**
  - Prior austenite grain size  $\sim 100\mu$ ; lath packets/partially recovered dislocation structure; precipitation of  $M_{23}C_6$ ;
  - Dislocation density  $\sim 5 \times 10^{14} m^{-2}$
  - $M_{23}C_6$  number density  $\sim 1 \times 10^{20} m^{-3}$



# Processing and Start-of-Life Microstructures for V-4Cr-4Ti

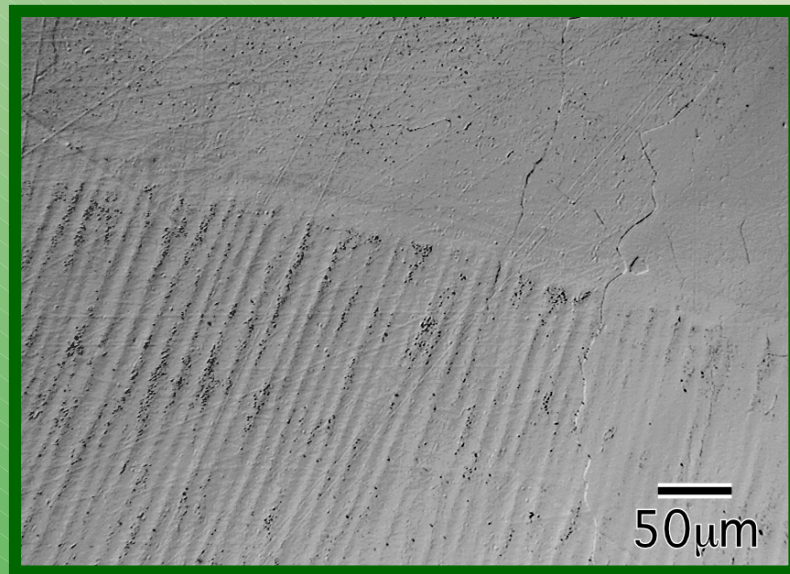


- **Processing**
  - 500kg ingot hot extruded at 1100°C
  - Cold rolling and recrystallization in the range 950°C-1050°C
  - Primary globular Ti(OCN) phase solvus temperature >1150°C
- **Microstructure**
  - Ti segregation during ingot solidification results in formation of bands of Ti(OCN) during extrusion
  - Recrystallized grain size 20-30μm; low sink strength microstructure

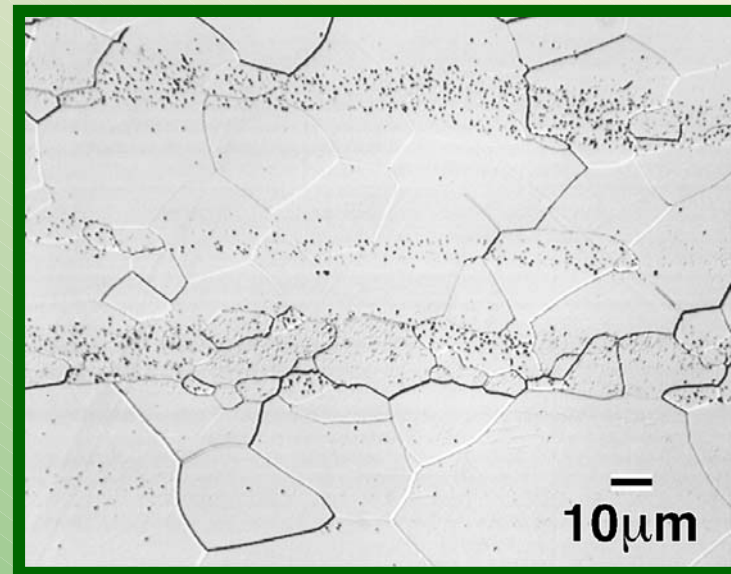


# Formation of Banded Microstructures in V-4Cr-4Ti

GTA Weld



Recrystallized at 1000°C

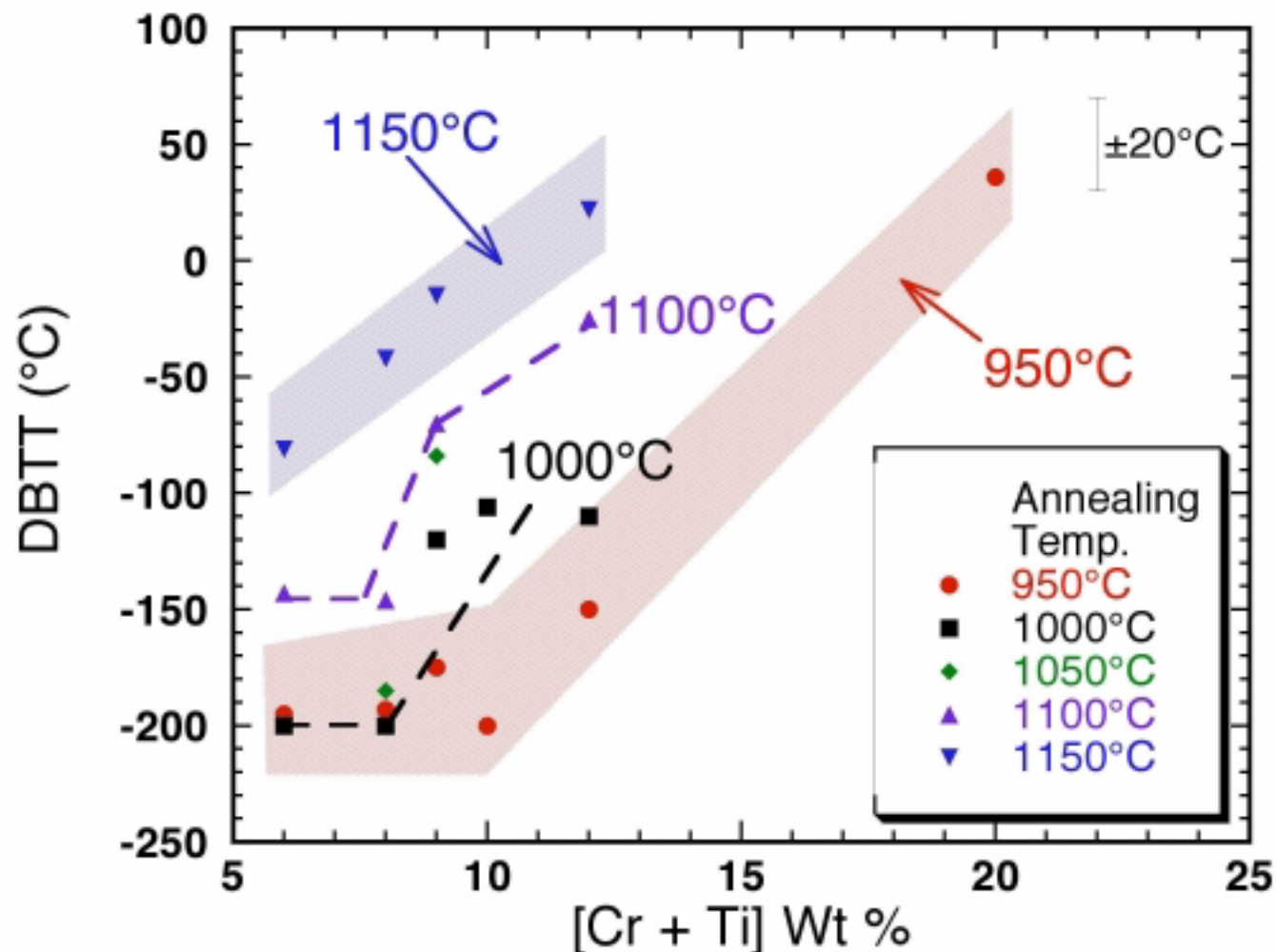


- Evidence for Ti segregation during solidification in GTA weld zones and also from microprobe analysis of ingots prepared in RF
- Globular Ti(OCN) develops in Ti-rich regions; bands of particles form during hot extrusion

# Sensitivity of Fracture Properties to SOL Microstructures

- Fracture properties of V-4Cr-4Ti strongly dependent on final heat treatment conditions
  - DBTT variations  $>200^{\circ}\text{C}$  due to change in grain size and interstitial content
- Fracture properties of TMS much less sensitive to SOL microstructure
  - DBTT variations  $10^{\circ}\text{-}50^{\circ}\text{C}$  produced by variations in prior austenite grain size and final tempering conditions.

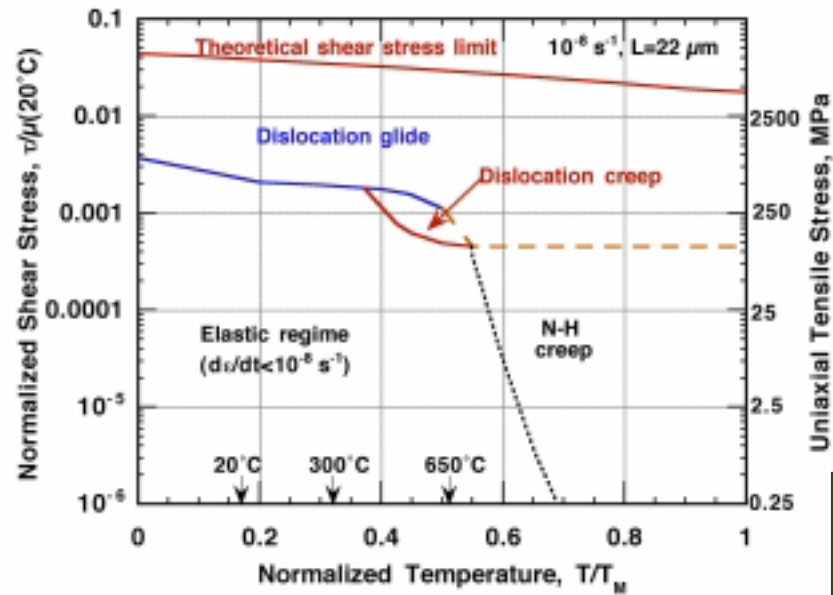
## Summary of Effect of Heat Treatment and [Cr+Ti] Concentration on Impact Properties



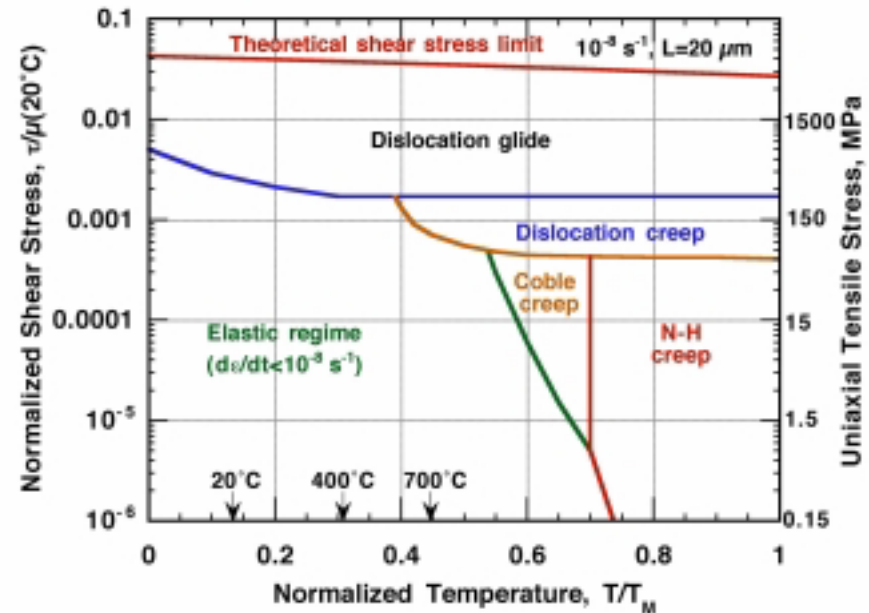
- Optimal impact properties are produced at annealing temperature of 950-1000°C for a wide range of V-Cr-Ti solid solution alloys

# Mechanical Behavior of BCC Structural Alloys

Deformation Map for Fe-8Cr-2WVTa  
Ferritic-Martensitic Steel



Deformation Map for V-4Cr-4Ti ( $d\varepsilon/dt=10^{-8} \text{ s}^{-1}$ )



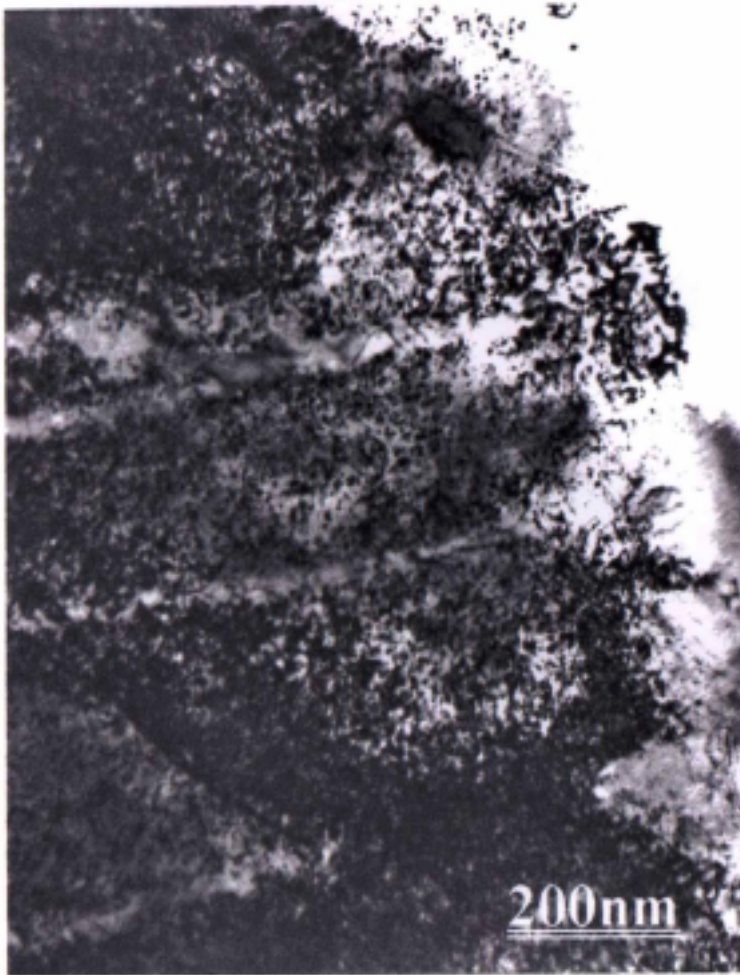


# Microstructural Stability in F82H

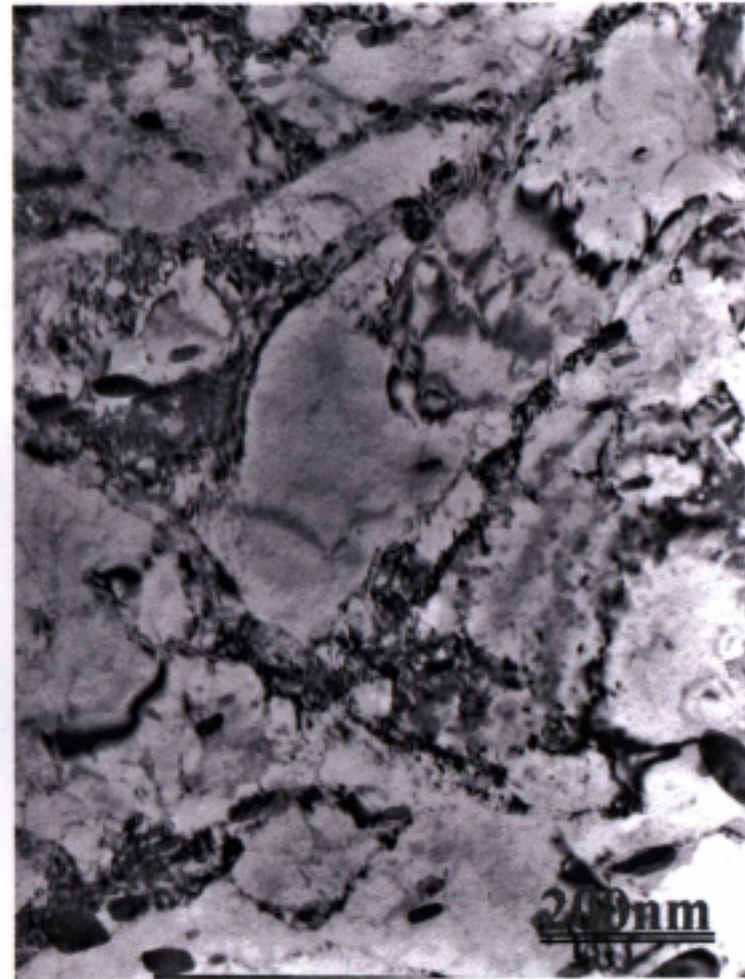
- **Thermal**
  - Tempered martensite structure relatively stable up to 550°C (5000h)
  - Intermetallic Laves phase develops > 600°C after 10<sup>4</sup> hours
- **Irradiation**
  - Lath, dislocation and precipitate structure relatively stable during neutron irradiation up to 500°C
  - Populations of  $a_0\langle 100 \rangle$  and  $(a_0/2)\langle 111 \rangle$  loops are the principal source of hardening 100°C - 400°C
- **Helium Generation**
  - Doping with Ni or B produces fairly uniform distributions of helium bubbles via (n,α) reactions between <sup>58</sup>Ni, <sup>10</sup>B and thermal neutrons

# Microstructure of F82H Irradiated to 5 dpa and Tensile Tested at R.T.

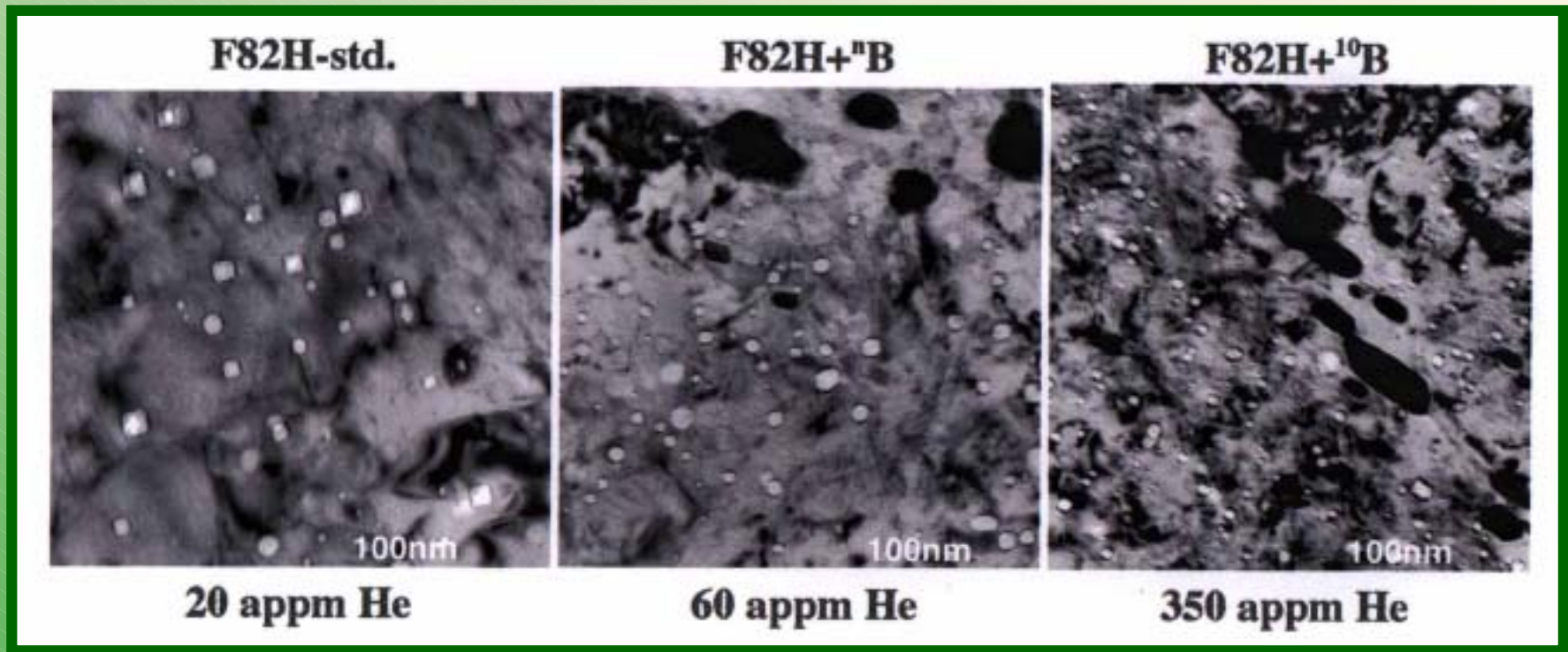
**Irradiated at 300°C**



**Irradiated at 500°C**



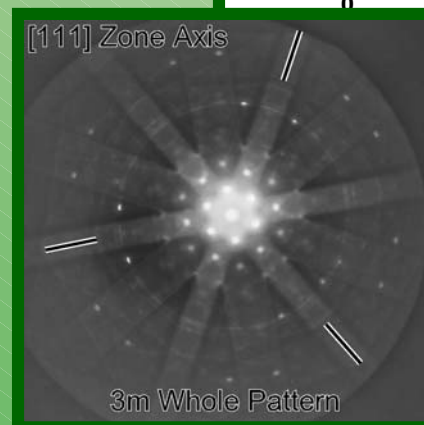
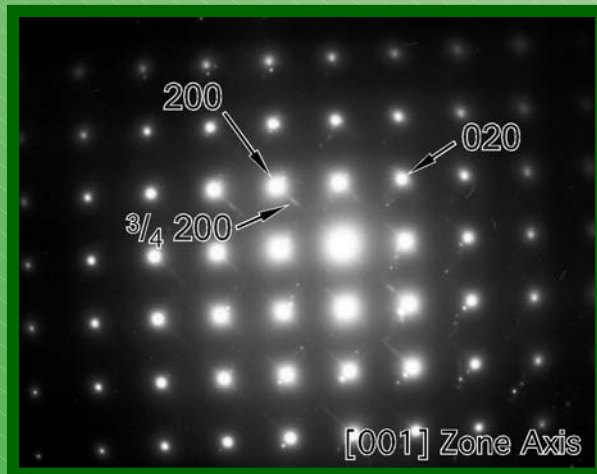
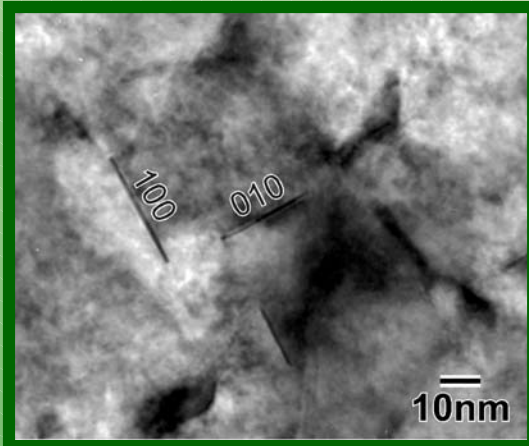
# Microstructure of F82H Irradiated at 400°C to 52 dpa in HFIR



- Doping with B produces fairly uniform bubble distributions and provides an important means of investigating the effect of helium on fracture behavior.

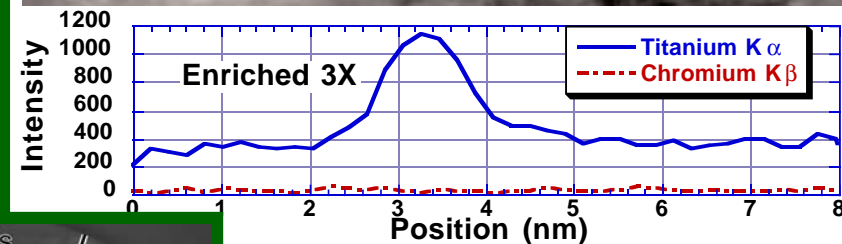


# Advanced Analytical Electron Microscopy Techniques are being used to Examine Precipitates in V alloys



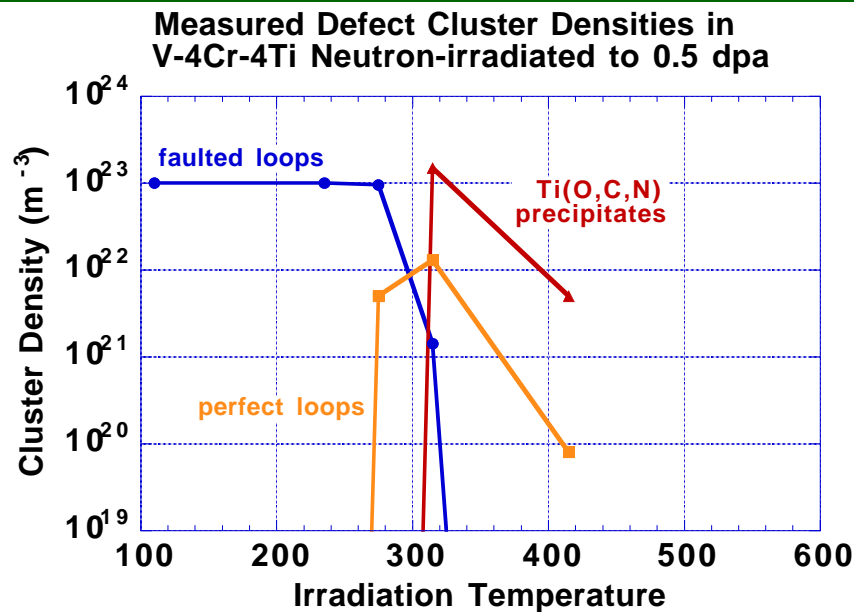
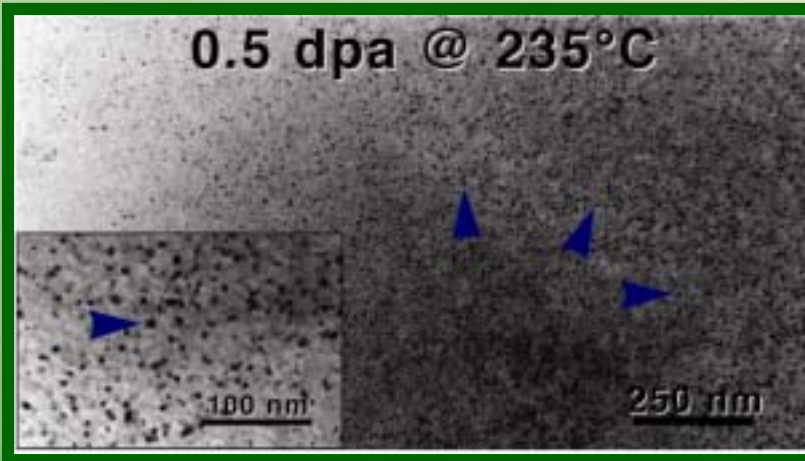
Solute Segregation Was Detected in V-4Cr-4Ti Following Neutron Irradiation to 0.5 dpa at Elevated Temperatures

**Dark Field STEM Image**

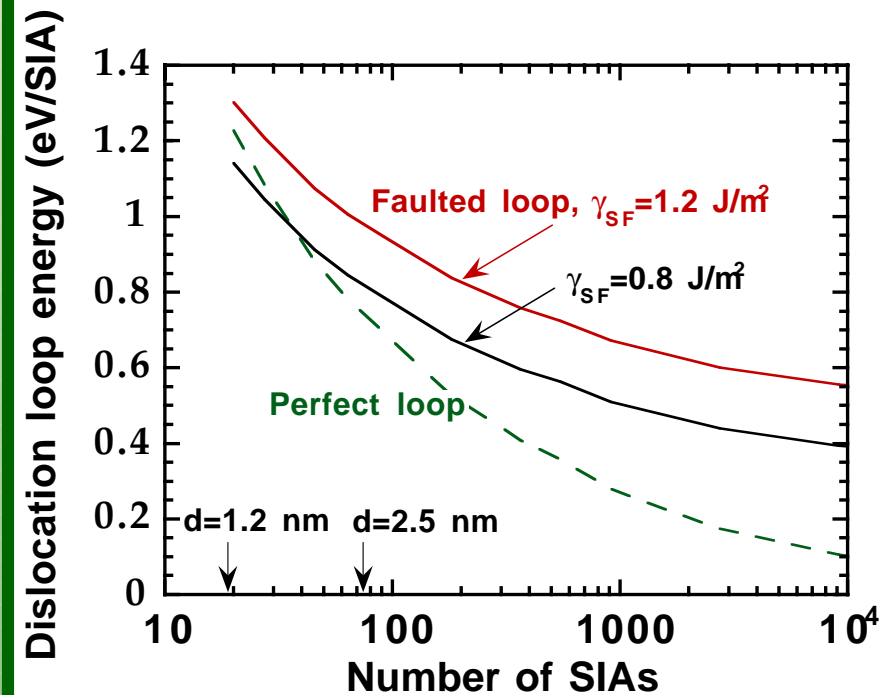


- Analytical microscopy reveals Ti-rich precipitates with Fm3m space group

# Experimental investigation of stacking fault energies of BCC metals



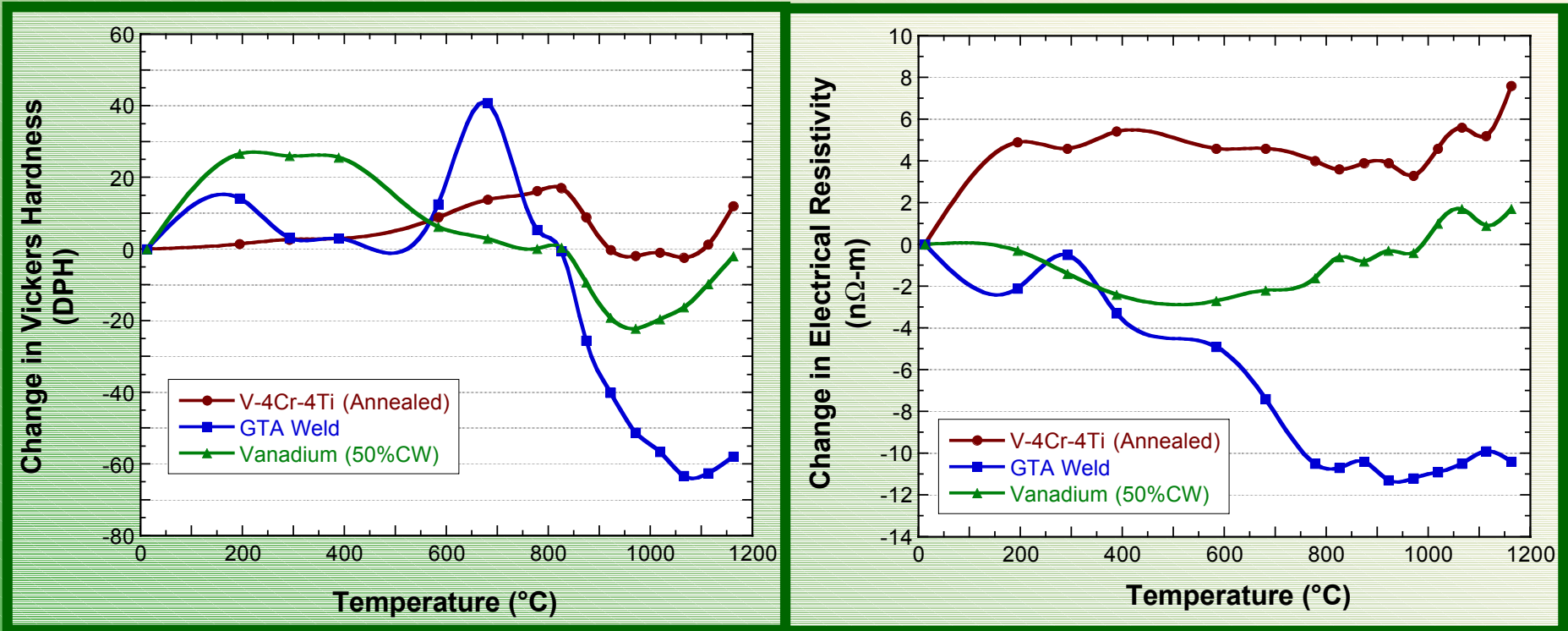
Continuum mechanics calculation of dislocation loop energies in vanadium



# Microstructural Stability in V-4Cr-4Ti

- **Thermal**
  - Segregation of interstitials to dislocations beginning at  $\sim 200^{\circ}\text{C}$  (static and dynamic strain aging)
  - Precipitation of a range of plate shaped Ti(OCN) phase  $700^{\circ}\text{-}950^{\circ}\text{C}$
- **Irradiation**
  - $\langle 110 \rangle$  faulted and  $\langle 111 \rangle$  perfect interstitial loops primarily responsible for radiation hardening  $60^{\circ}\text{-}350^{\circ}\text{C}$
  - Ti-enriched  $\langle 001 \rangle$  defects develop  $300^{\circ}\text{-}400^{\circ}\text{C}$ ; Ti-rich oxycarbonitride plates develop  $400^{\circ}\text{-}550^{\circ}\text{C}$

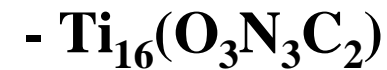
# Electrical Resistivity and Hardness in V-4Cr-4Ti as a Function of Temperature



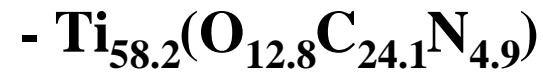
- Variations in electrical resistivity and hardness reflect mobility of interstitials and interactions with Ti and provide information relevant to DSA, recovery and recrystallization processes and precipitation reactions

# Plate Formation on {001} Habit in V-4Cr-4Ti

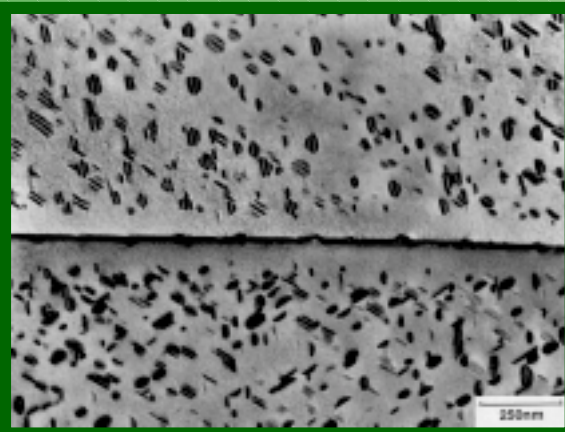
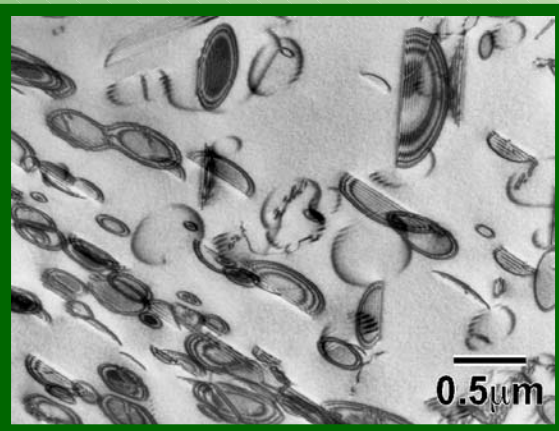
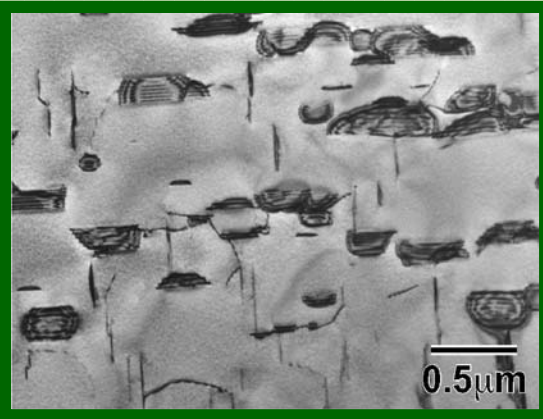
**GTA Weld**



**Oxidized**



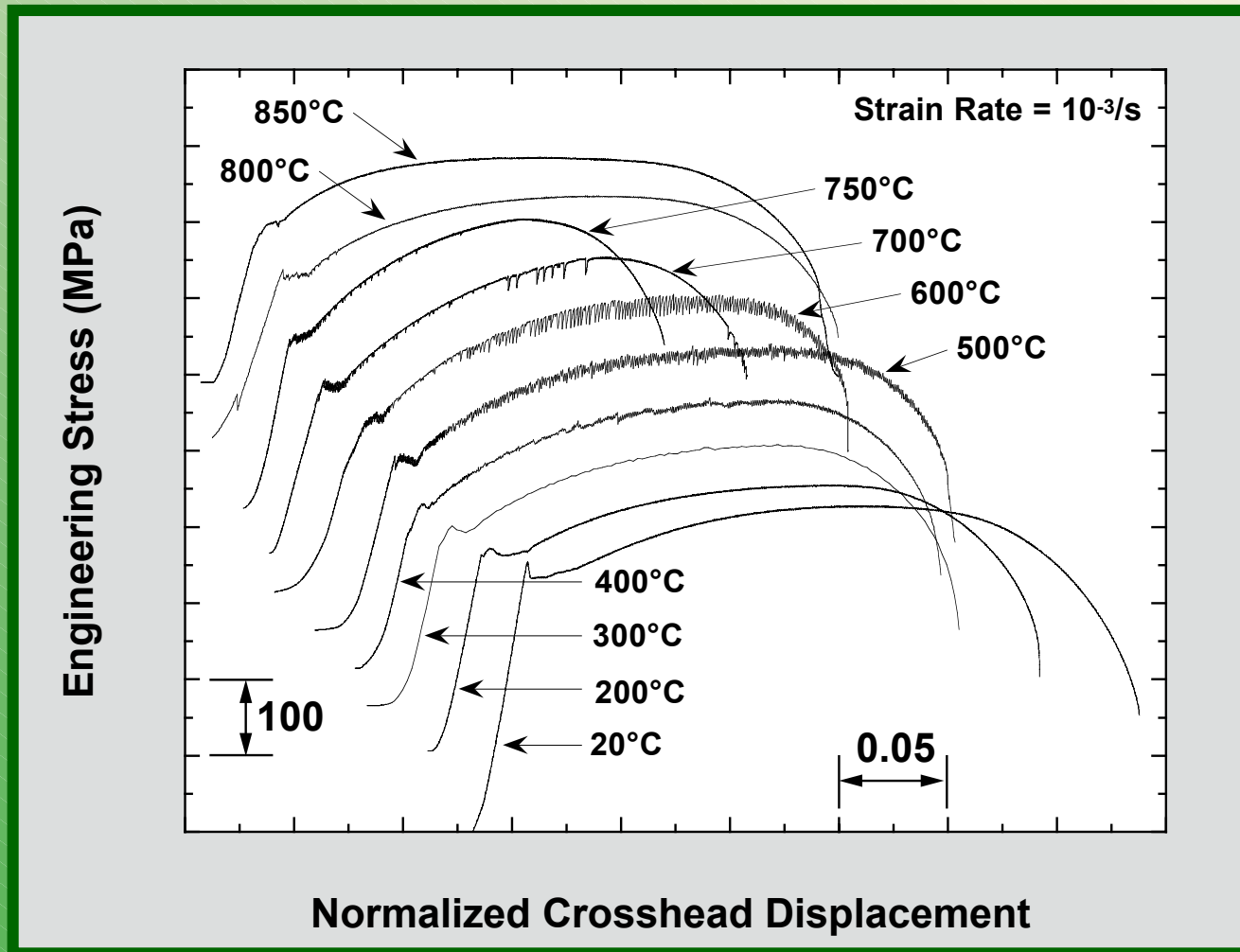
**Neutron Irradiated**



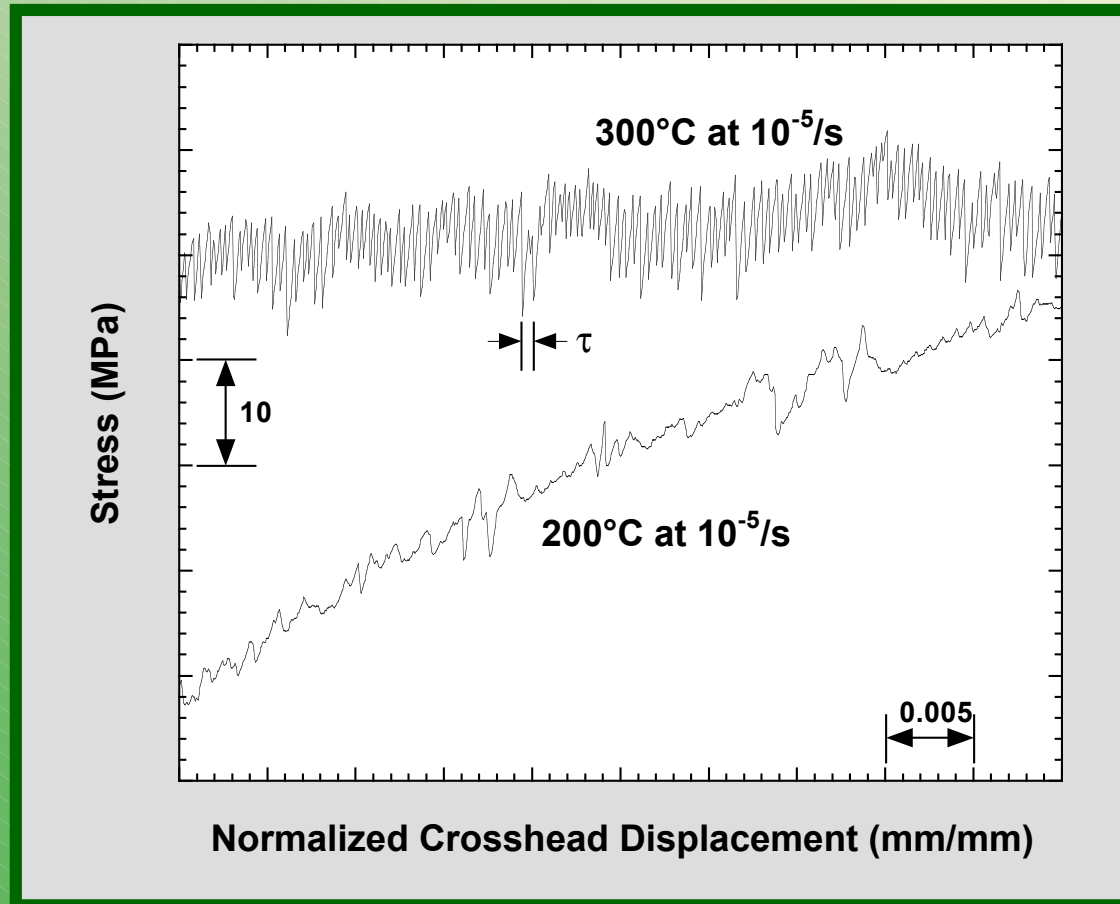


# Dynamic Strain Aging in V-4Cr-4Ti

- Maximum in strain-rate sensitivity occurs at 600°C in V-4Cr-4Ti



# Magnified View of Serrated and Jerky Flow Behavior in Vanadium



- Estimate time for diffusion of interstitials

- segregate to immobile and mobile dislocations

- identify elements responsible for DSA

- Calculate average time between minima ( $t$ )

- 300°C and 10<sup>-5</sup>/s

$$\tau = \sim 37.5s$$

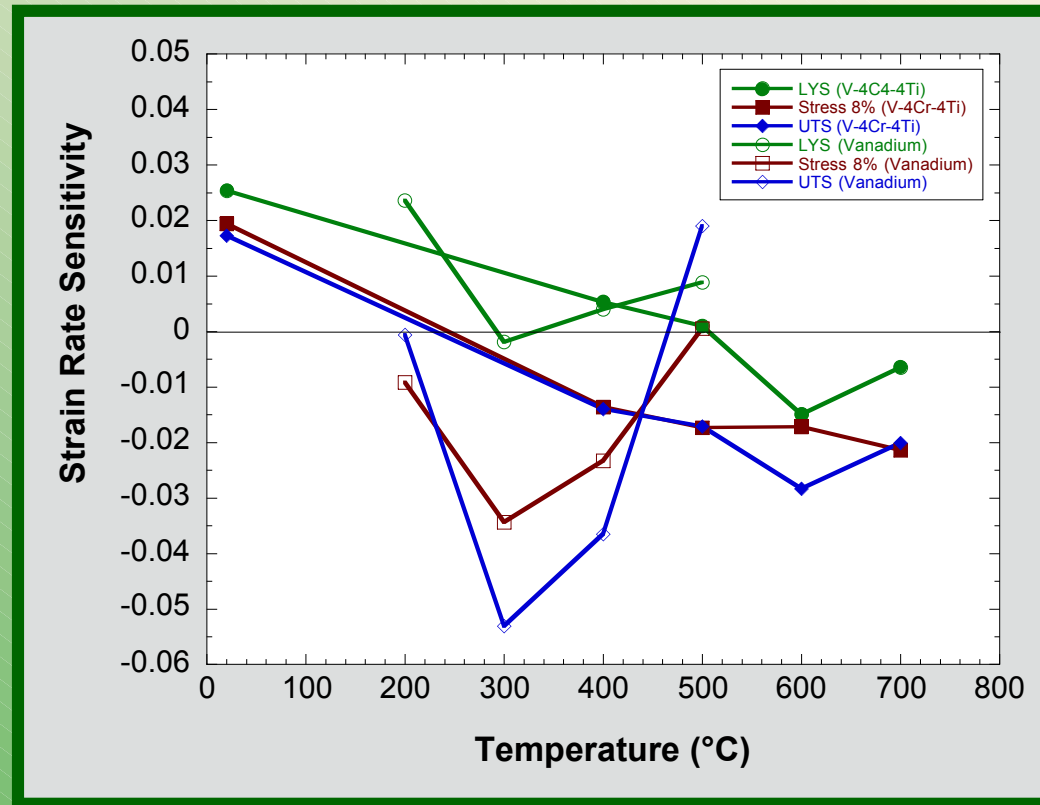
- 200°C and 10<sup>-5</sup>/s

$$\tau = \sim 129s$$

- Diffusivity calculations show:

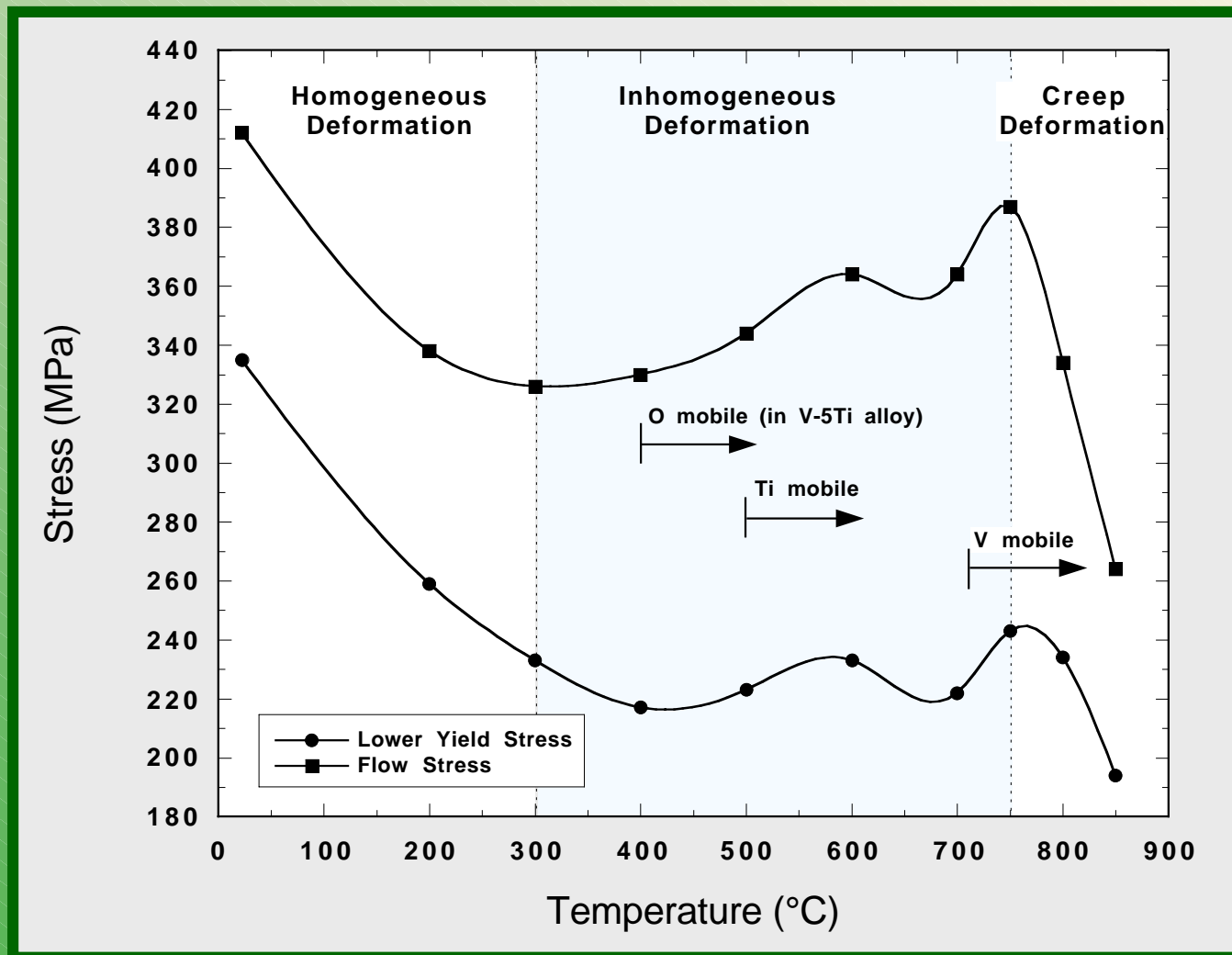
- Carbon and oxygen are primarily responsible for DSA effect below 300°C
- Nitrogen has similar mobility at a higher temperature of 400°C

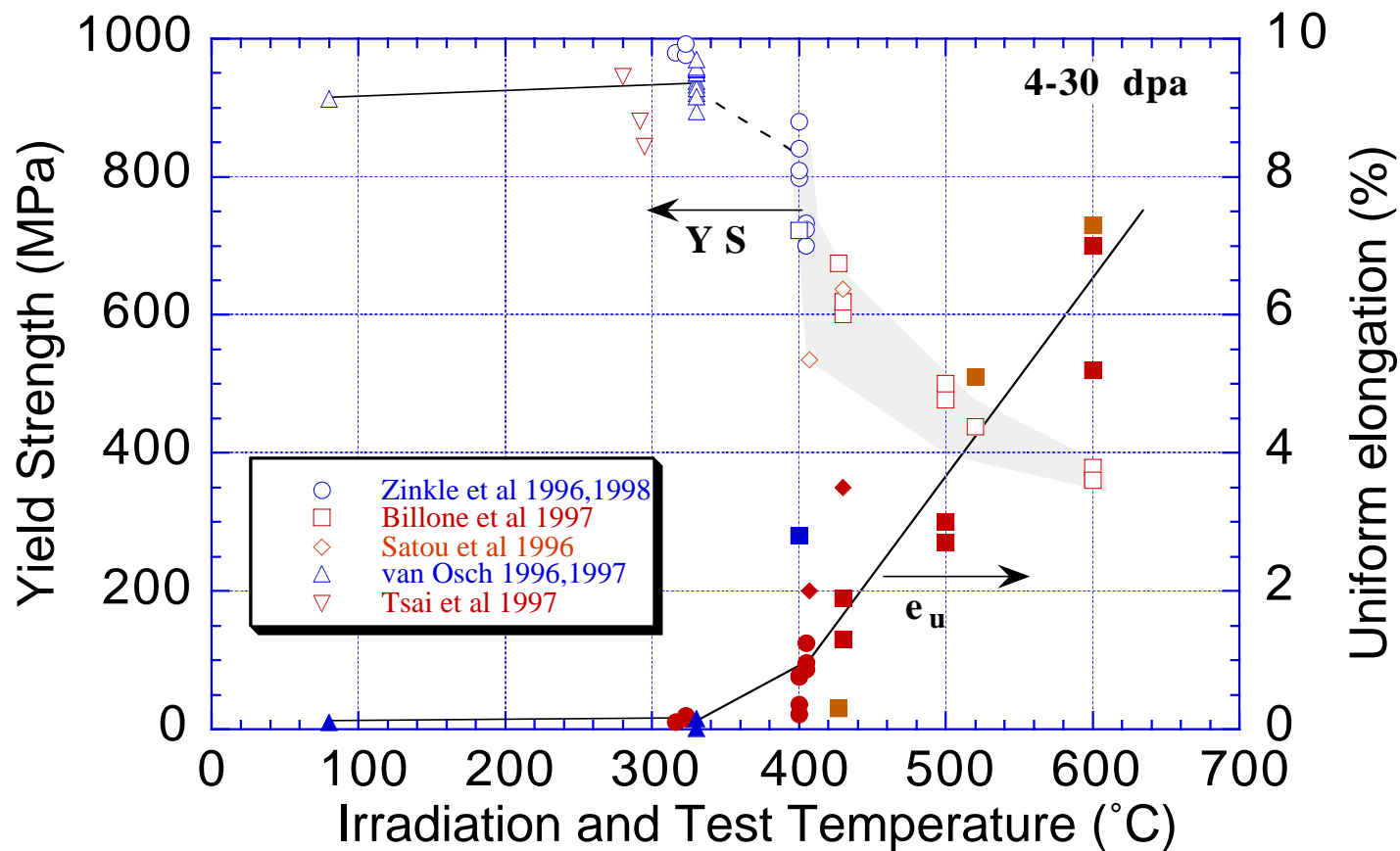
# Strain Rate Sensitivity for V and V-4Cr-4Ti



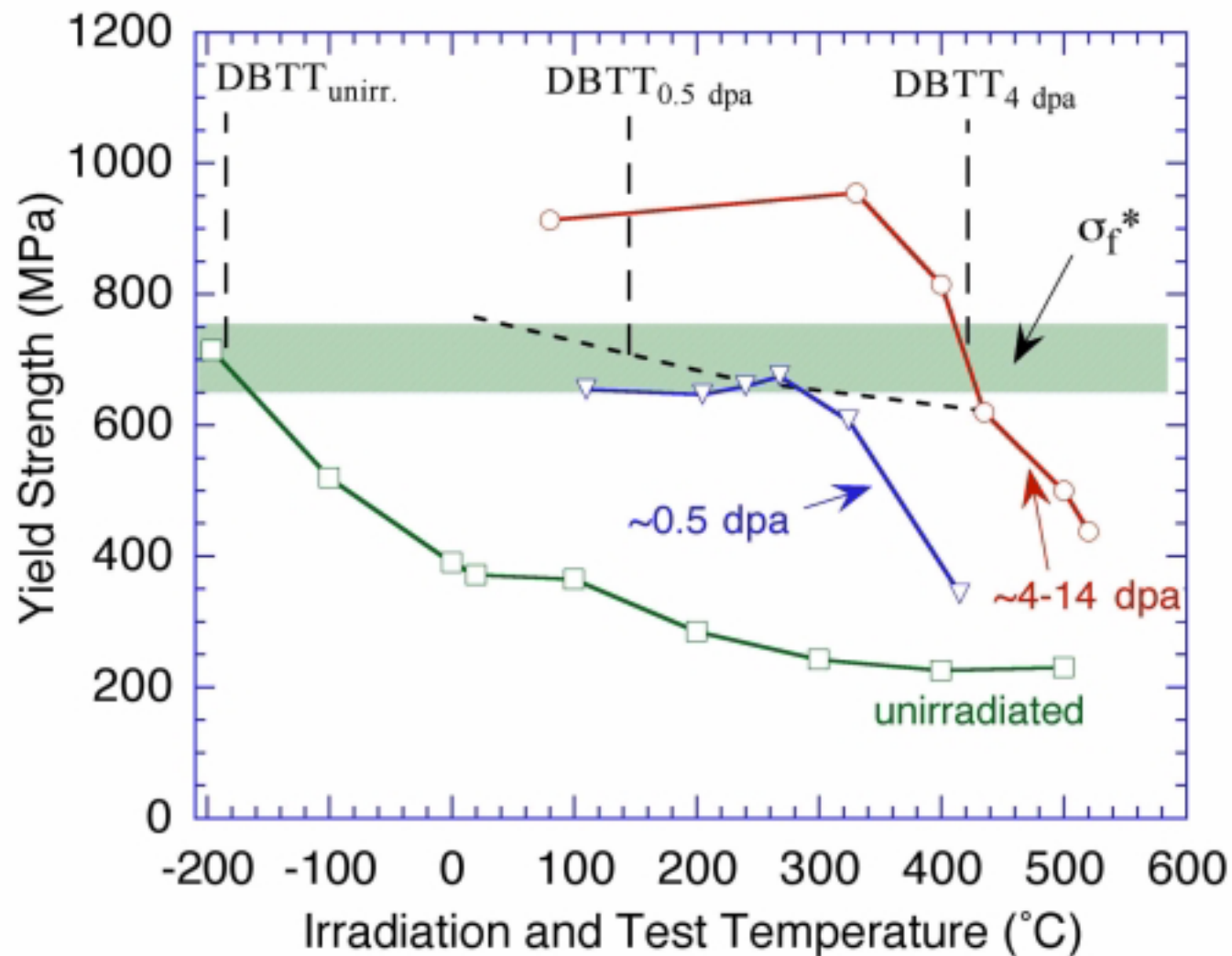
- Alloying with Ti shifts the maximum negative SRS by ~300°C
- Calculations based on stereology measurements indicate that ~50% of the interstitials are retained in the globular Ti(OCN)
  - mobility of the remaining interstitial content is reduced by interactions with Ti
- Possible benefits of lowering Ti concentration are being investigated

# Temperature Dependence of $\sigma_Y$ and $\sigma_f$ at $10^{-3} \text{ s}^{-1}$

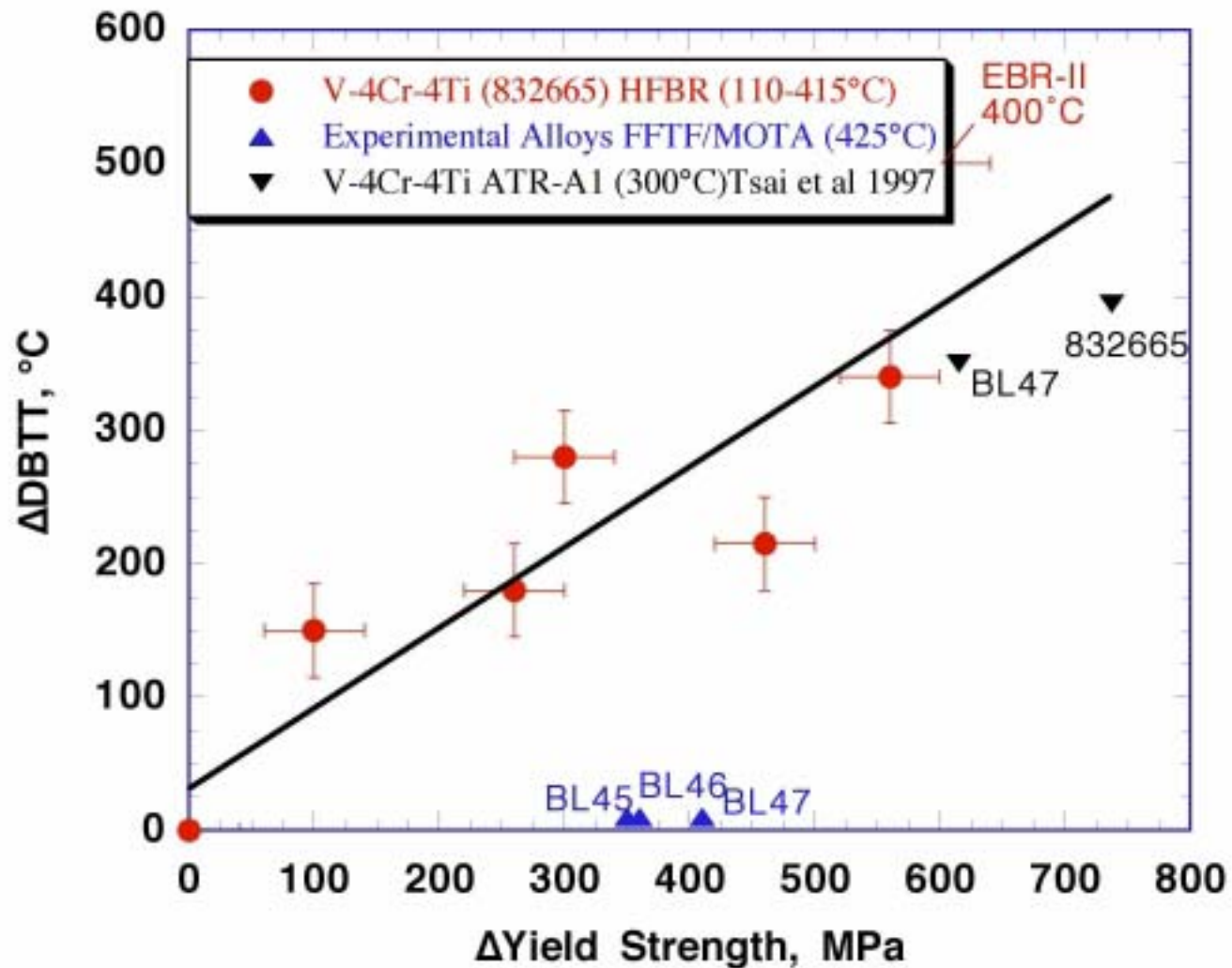




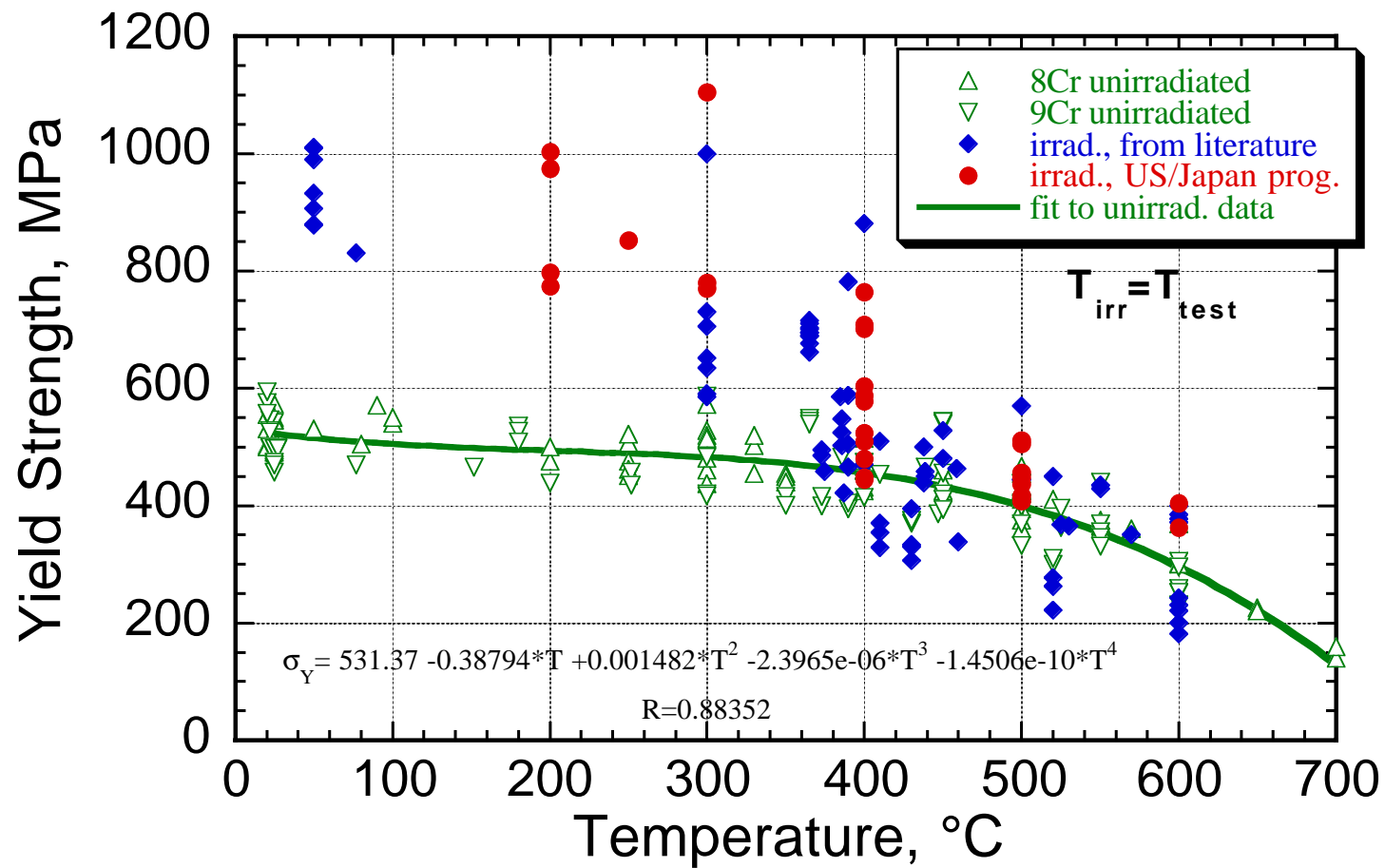
**Low-Temperature Radiation Hardening Causes a Large Increase in the Ductile-to-Brittle-Transition Temperature in V-4%Cr-4%Ti Alloys**



## Relationship Between Radiation Hardening and DBTT Shift for V-4Cr-4Ti Irradiated in Various Reactors

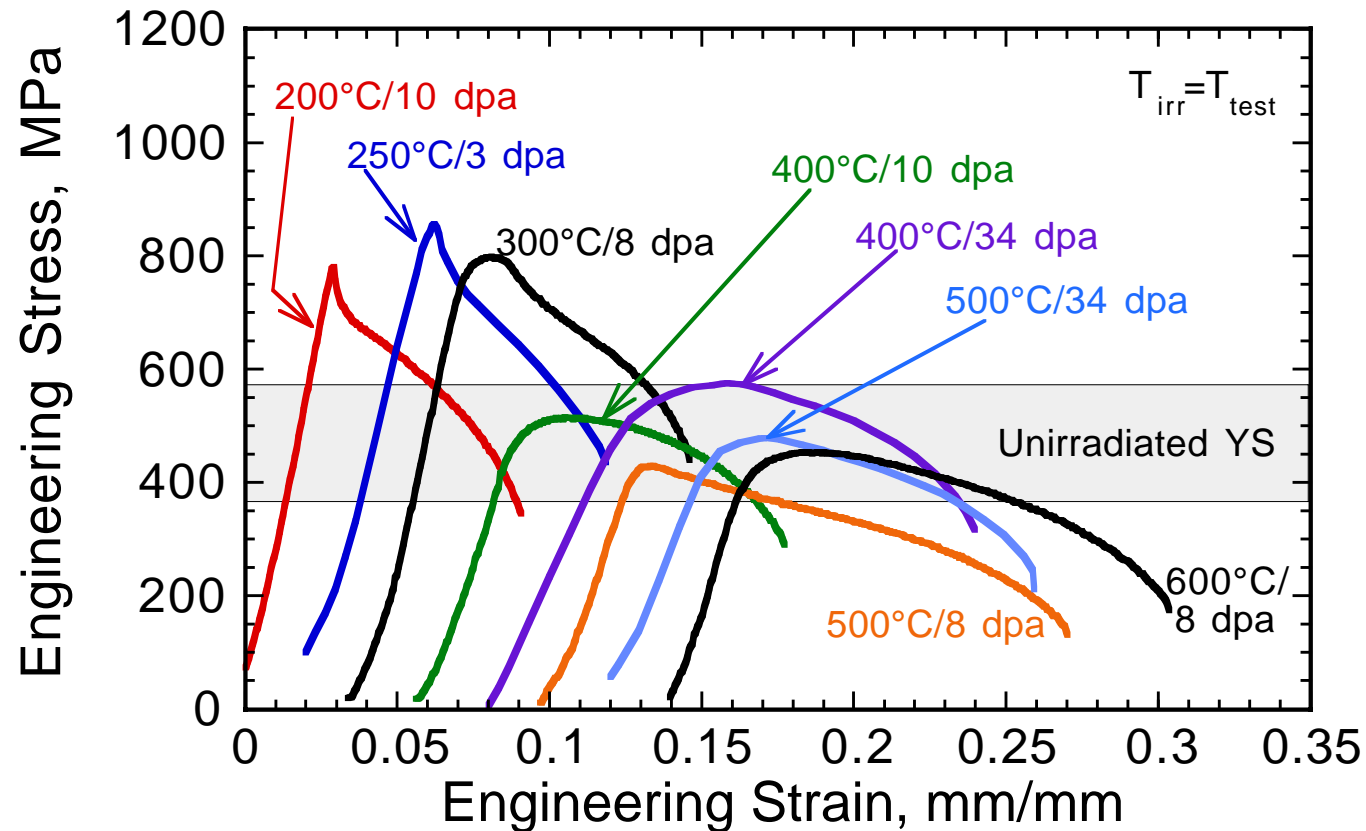


# Radiation Hardening in TMS

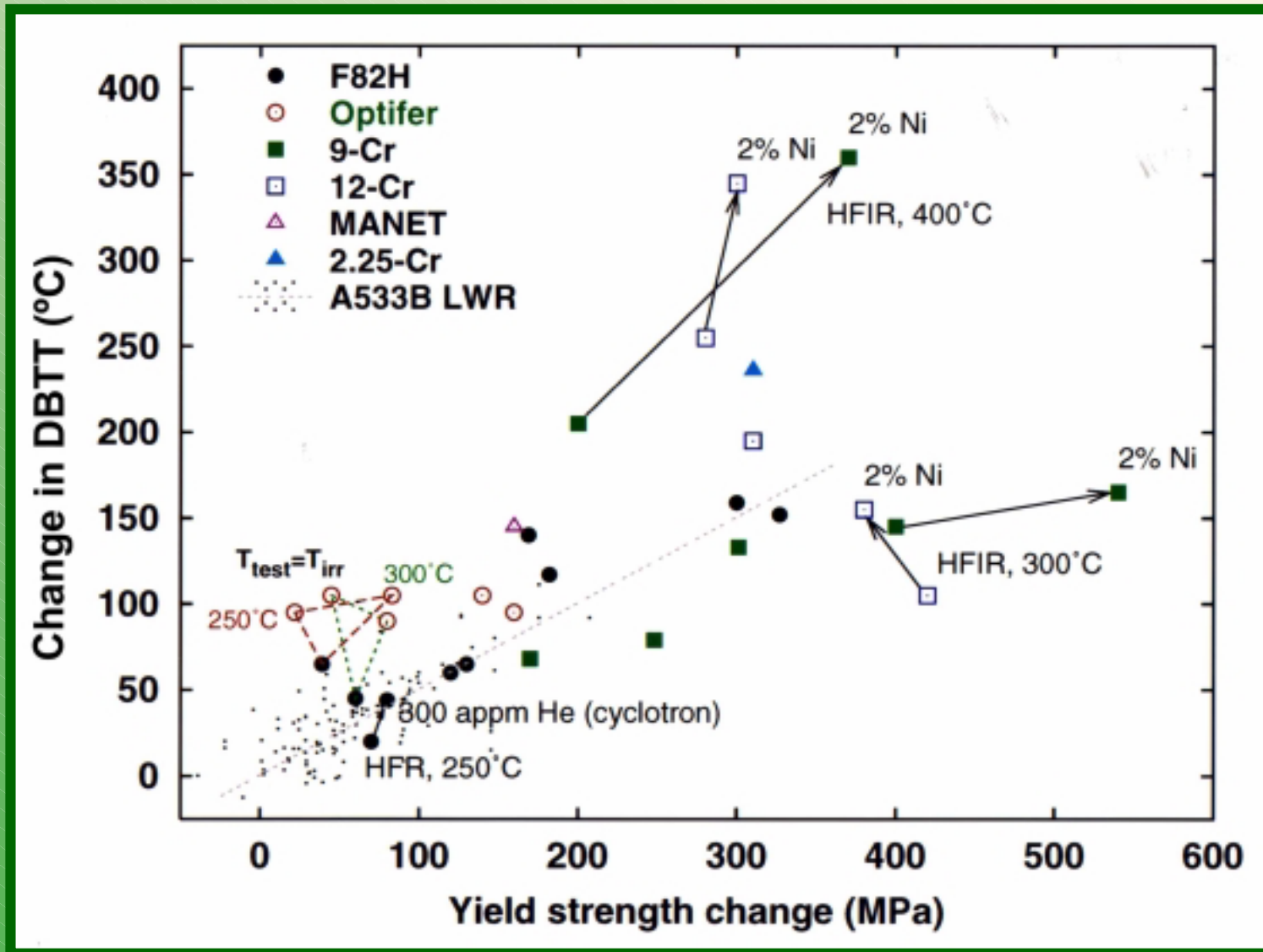




# Stress - Strain Curves for Neutron Irradiated F82H



# Radiation Hardening-DBTT Shifts Relationship for TMS Including Helium Effects



# Future Work

- Investigation of performance limiting, radiation-induced phenomena in model alloys
  - Broad portfolio of miniaturized property measurements and characterization methods
  - Fully integrated with theory/modeling activities
- Fundamental issues of flow and fracture in TMS and V-4Cr-4Ti model alloys including helium effects at 300°-400°C
- Fundamental issues related to helium migration, trapping and bubble formation in TMS and V-4Cr-4Ti model alloys at 400°-650°C
- Initial studies of dispersoid and nanocluster stability, helium trapping in advanced alloys at 300°-800°C

# Future Work

- Irradiation program to be carried out under a 5-year shared-cost program with JAERI/MONBUSHO
  - 5 Li-bonded small volume HFIR experiments (25cm<sup>3</sup> specimens/capsule)
  - Temperature monitored and controlled; neutron doses (1-10 dpa)
  - Spectrally tailored and isotopically- doped to vary helium production
- Continuing investigation of substitutional solute-interstitial interactions in V-4Cr-4Ti model alloy
  - Immobilization of interstitials in uniformly-dispersed oxycarbonitride phases
  - Improved creep strength and helium management
- Expanding upper temperature operating limits of TMS through nano-phase engineering