Creep and Swelling of Vanadium Alloys

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Pacific Northwest National Laboratory

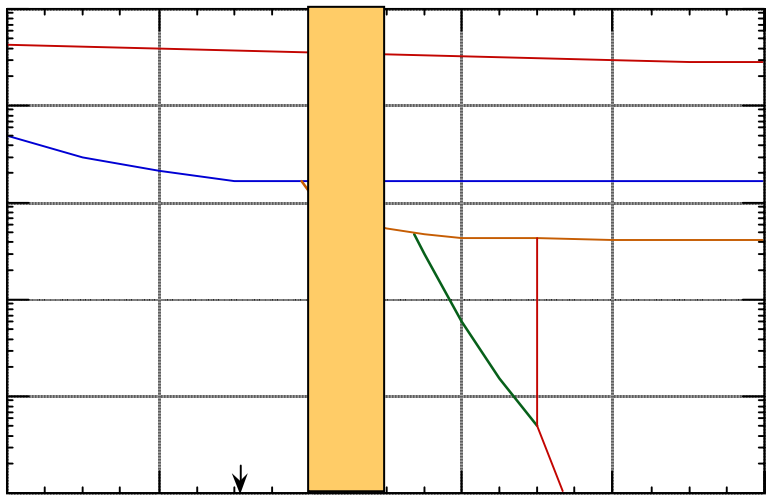
Outline

- Thermal creep data base and ongoing experiments
- Irradiation creep data base and planned experiments
- Creep-swelling interactions
- High-temperature helium embrittlement
- Swelling data base

Deformation Mechanism Map for V-4Cr-4Ti

Zinkle and Lucas

$$\dot{\varepsilon} = 10^{-8} s^{-1},\ \mathrm{L} = 20\,\mu m$$



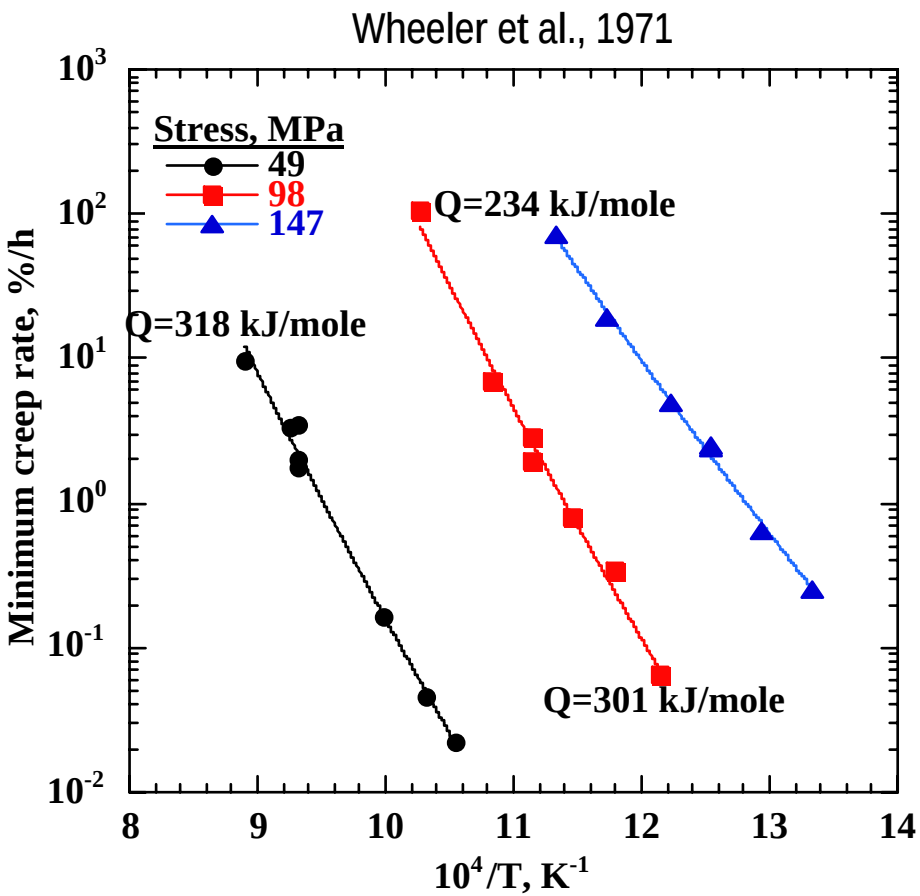
Diffusion-Controlled Creep Mechanisms

Murty, Mohamed and Dorn, 1972

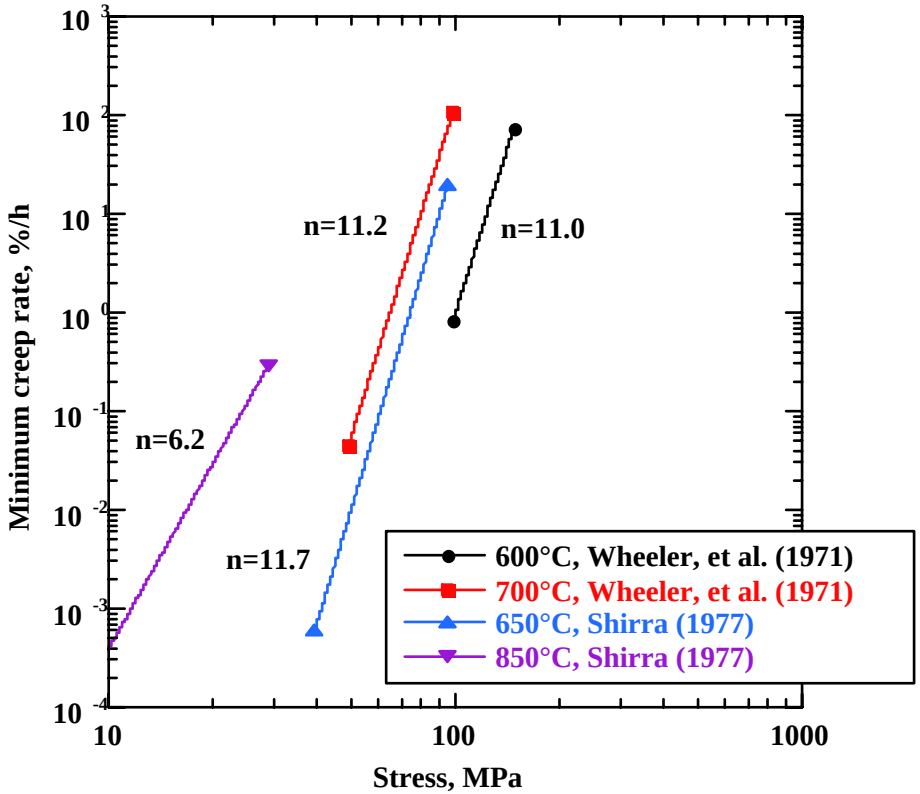
$$\frac{\dot{\varepsilon} kT}{DGb} = A\left(\frac{b}{d}\right)^m \left(\frac{\sigma}{G}\right)^n$$

Mechanism	D	n	A	m
Climb of Edge Dislocations	D_L	5	6×10^{7}	0
Viscous Glide (Microcreep)	D_S	3	6	0
Low-Temperature Climb	D_d	7	2×10^{8}	0
Harper-Dorn	D_L	1	3×10^{-10}	0
Nabarro-Herring	D_L	1	12	2
Coble	D_b	1	100	3
GBS (Superplasticity)	D_b	2	200	2
Nabarro-Subgrain	D_L	1	12	2
Nabarro-Bardeen-Herring	D_L	3	10	0

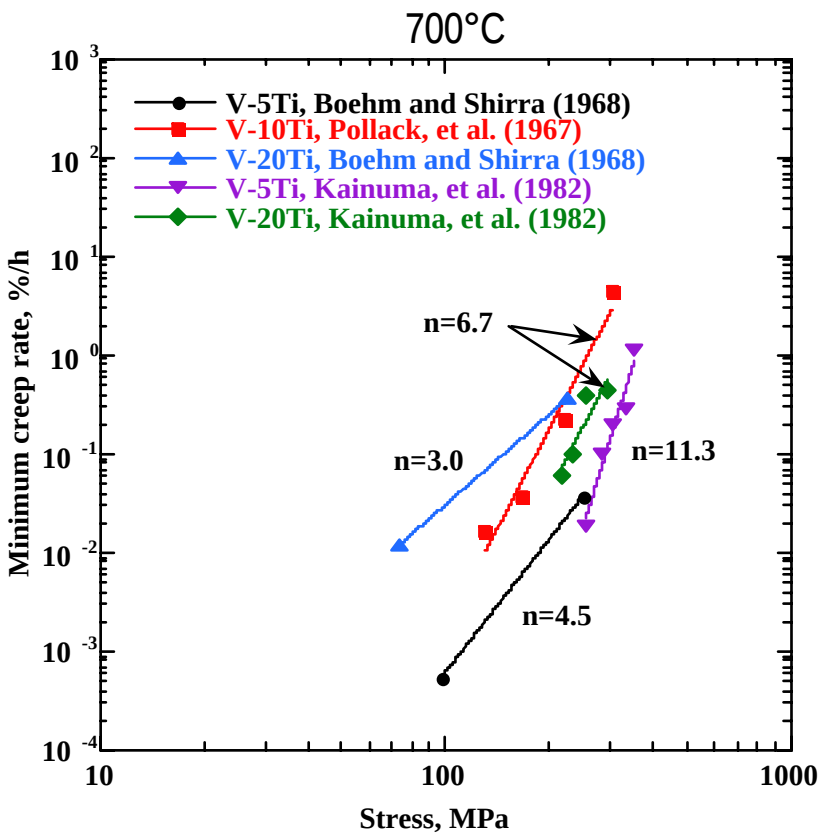
Temperature Dependence of Creep Rate in Pure Vanadium

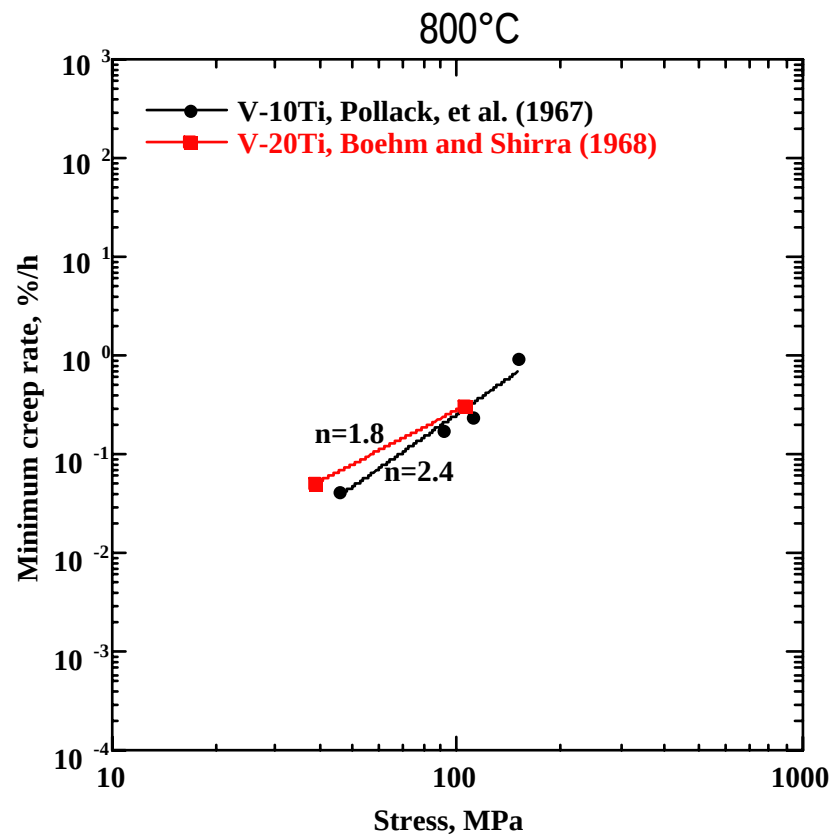


Stress Dependence of Creep Rate in Pure Vanadium

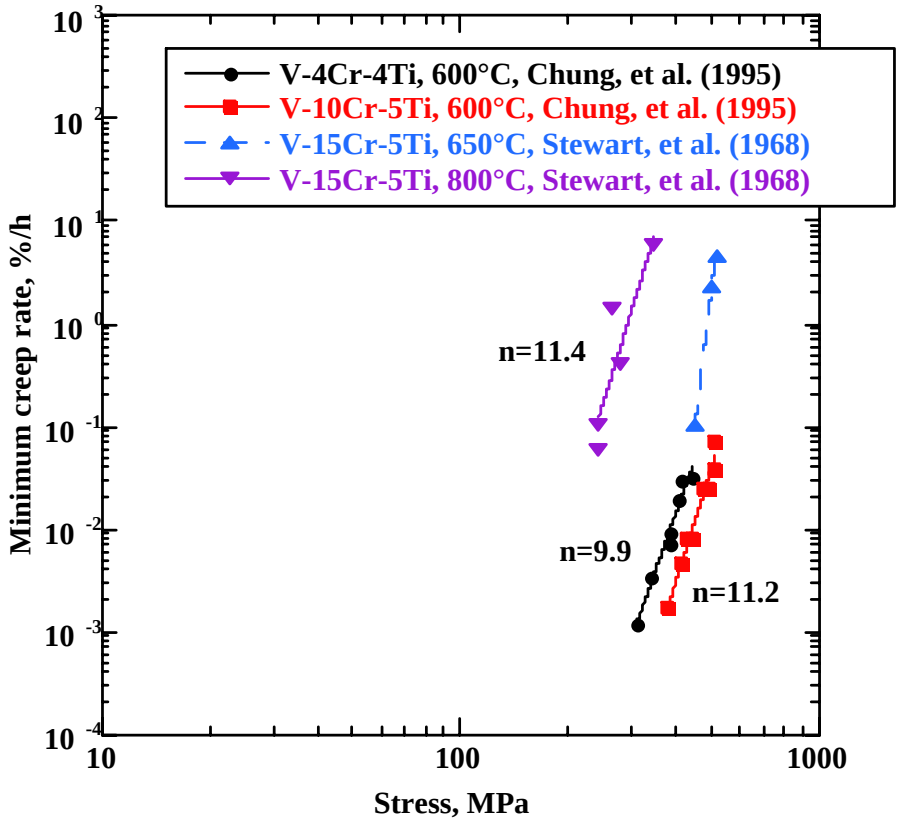


Stress Dependence of Creep Rate in V-Ti Alloys at 700 and 800°C

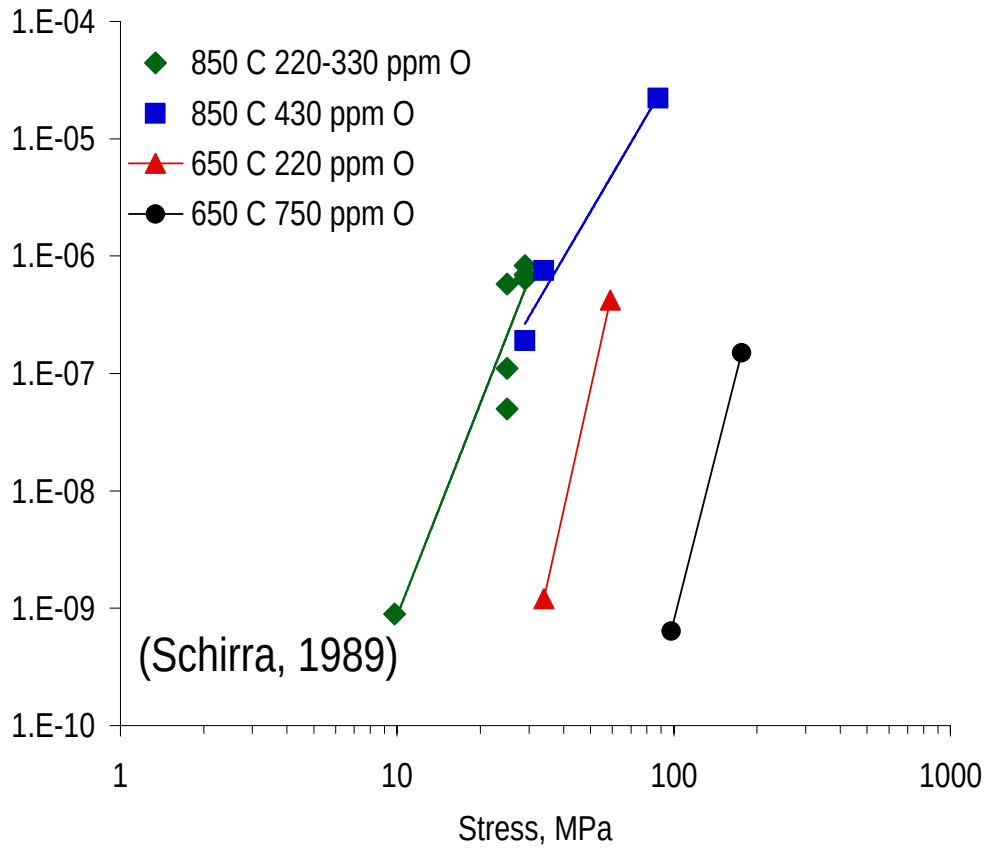




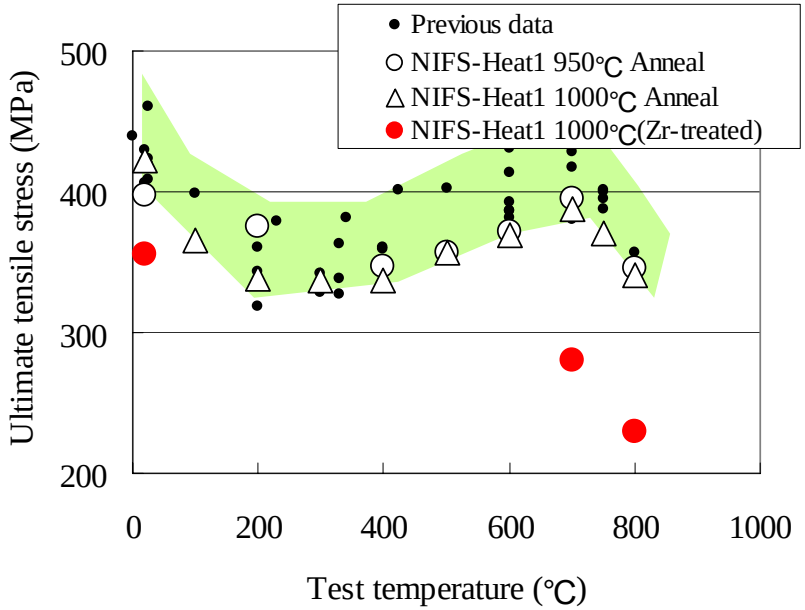
Stress Dependence of Creep Rate in V-Cr-Ti Alloys Between 600 - 800°C

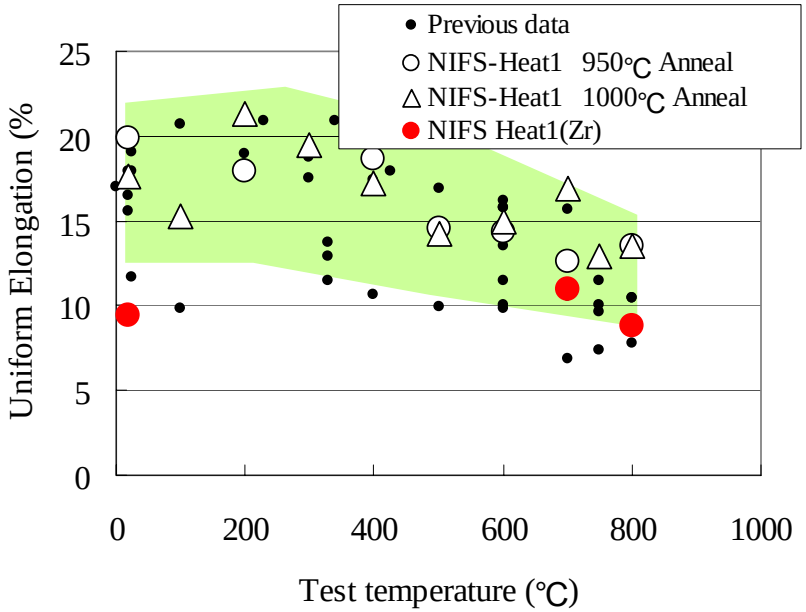


Effect of Oxygen on Creep Rate of Vanadium



Effect of Alloy Purity on Tensile Properties of NIFS Heat-1





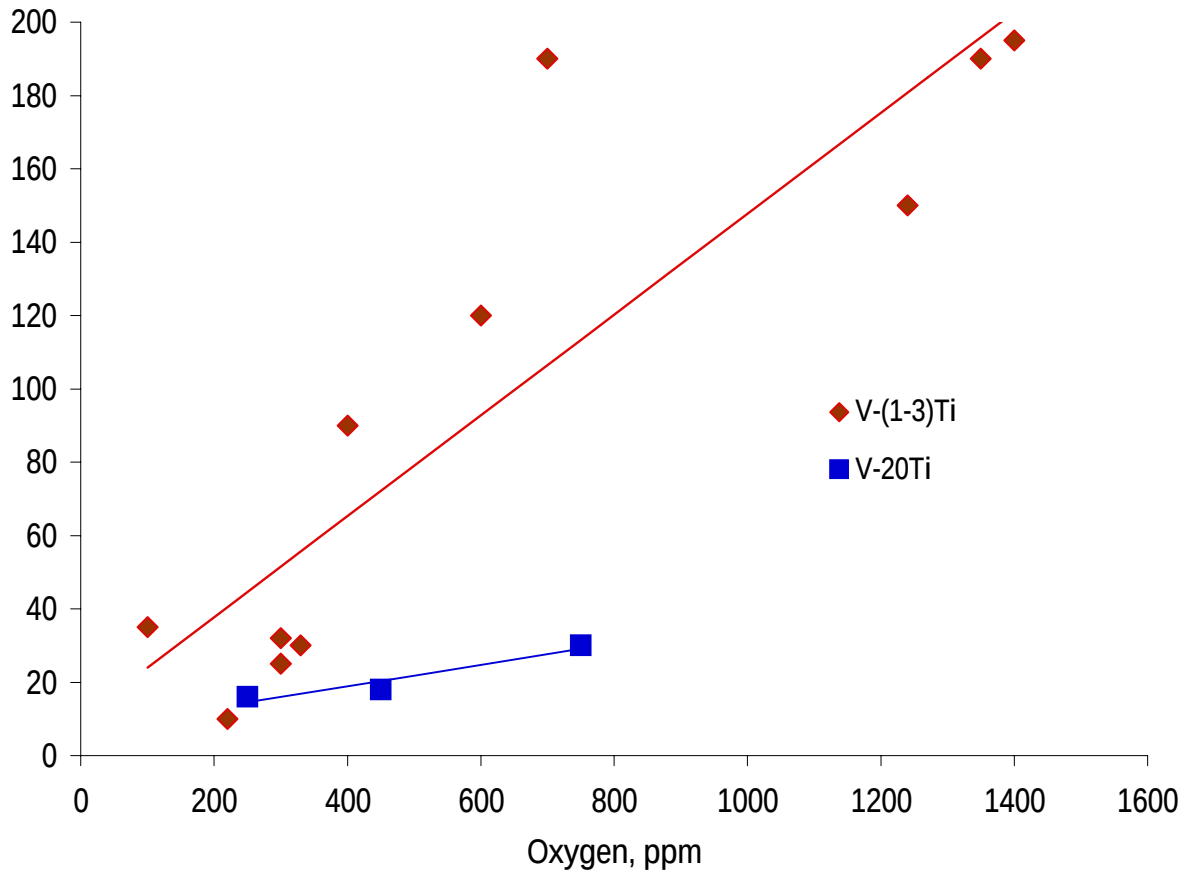
Purification of NIFS Heat-1 by Zr-treatment

Fukumoto, et al.

- Decreased yield stress and UTS at high temperature
- Supression of dynamic strain aging behavior at 700°C

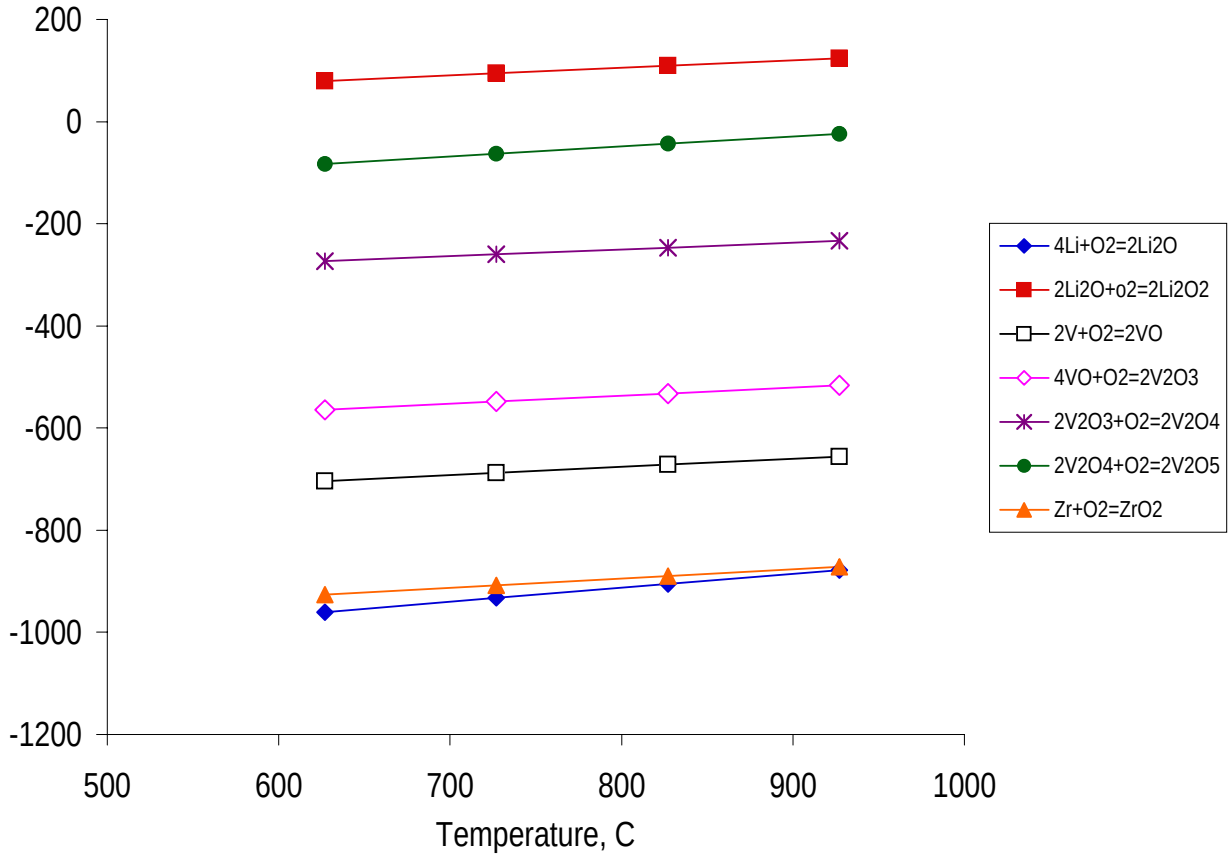
The effect of interstitial impurities on the mechanical properties of V-4Cr-4Ti alloys is eliminated at the 20 wppm level.

Dependence of Creep Rupture Time on Oxygen Concentration



Even though creep rupture time is extended, oxygen can severely embrittle vanadium.

In a Lithium Environment, Oxygen is Removed From Vanadium



Experiments Needed to Separate the Effects of Irradiation and Environment

1. DHCE -- Irradiation, He, Li, Thermal

2. RTHD (Real-Time Helium Doping)
   -- He, Li, Thermal

3. LTCE (Li Thermal Creep Experiment)
   -- Li, Thermal (low oxygen)

4. Thermal Creep Experiment
   -- Thermal effects (oxygen increases)

Thermal Creep Experiments

- Characterize the thermal creep behavior of vanadium alloys from 650°C to 800°C at effective stress levels below yield
- Elucidate fundamental mechanisms controlling creep behavior of vanadium alloys
- Three experimental studies:
  - Biaxial creep tests in Li (ORNL)
  - Uniaxial creep tests in vacuum (ANL)
  - Biaxial creep tests in vacuum (PNNL)

Creep Matrix in Vacuum

- Pressurized tubes: 4.57 mm diameter x 25.4 mm long of V-4Cr-4Ti and V-3Fe-4Ti (PNNL)
- Flat tensile specimens of V-4Cr-4Ti: 19 mm x 4.5 mm x 1 mm gauge section (ANL)
- Round tensile specimens of V-4Cr-4Ti: 2.54 mm diameter x 19 mm long (ANL)
- Temperatures: 650, 700, 725, 800°C
- Stresses:

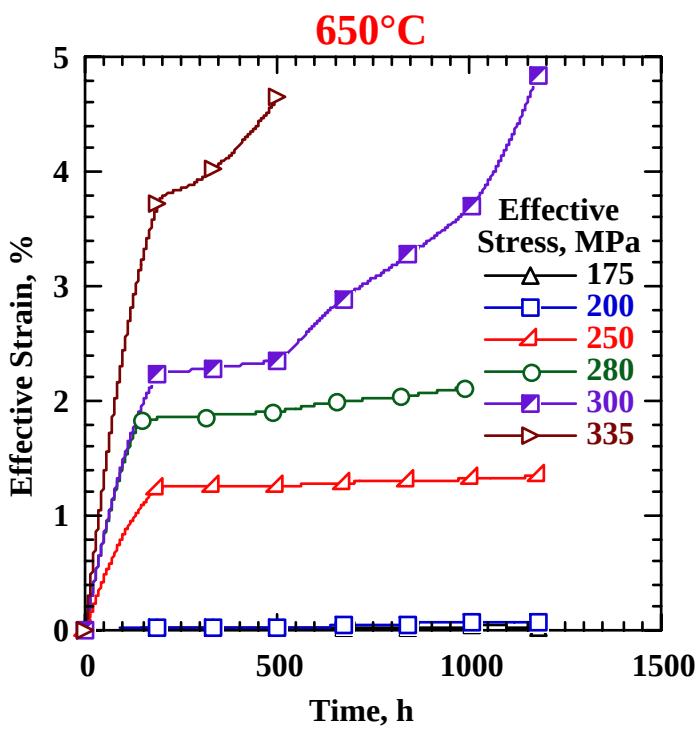
650	700	725	800°C
175-335	67-159	150-200	26-137 MPa

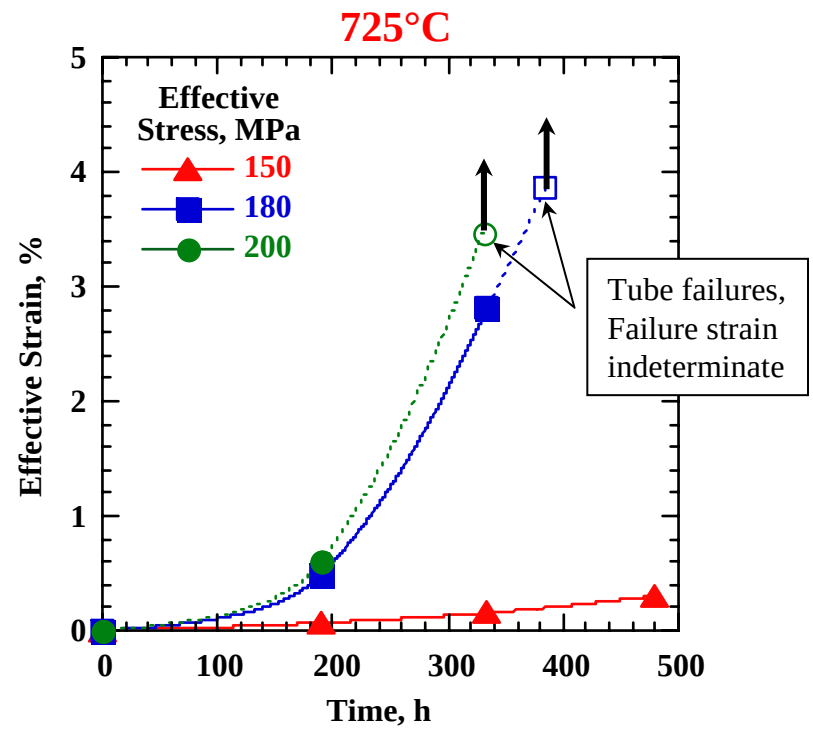
Creep Matrix in Lithium

- Pressurized tubes of V-4Cr-4Ti, 4.57 mm diameter x 25.4 mm long to match vacuum tests (ORNL)
- Temperatures: 665, 765, 800°C
- Stresses:

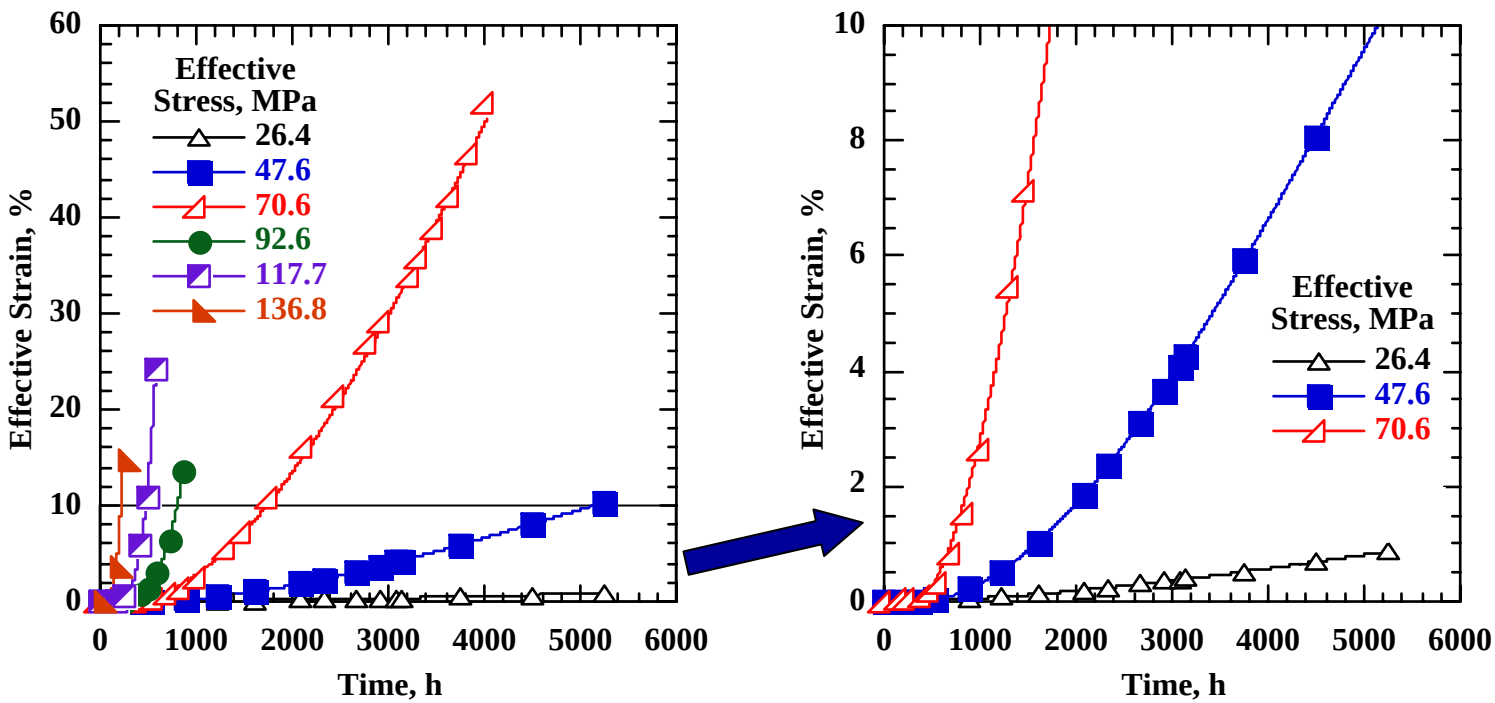
665	765	800°C
59-117	25-84	30-100 MPa

Time Dependence of Effective Creep Strain at 650 and 725°C for V-4Cr-4Ti



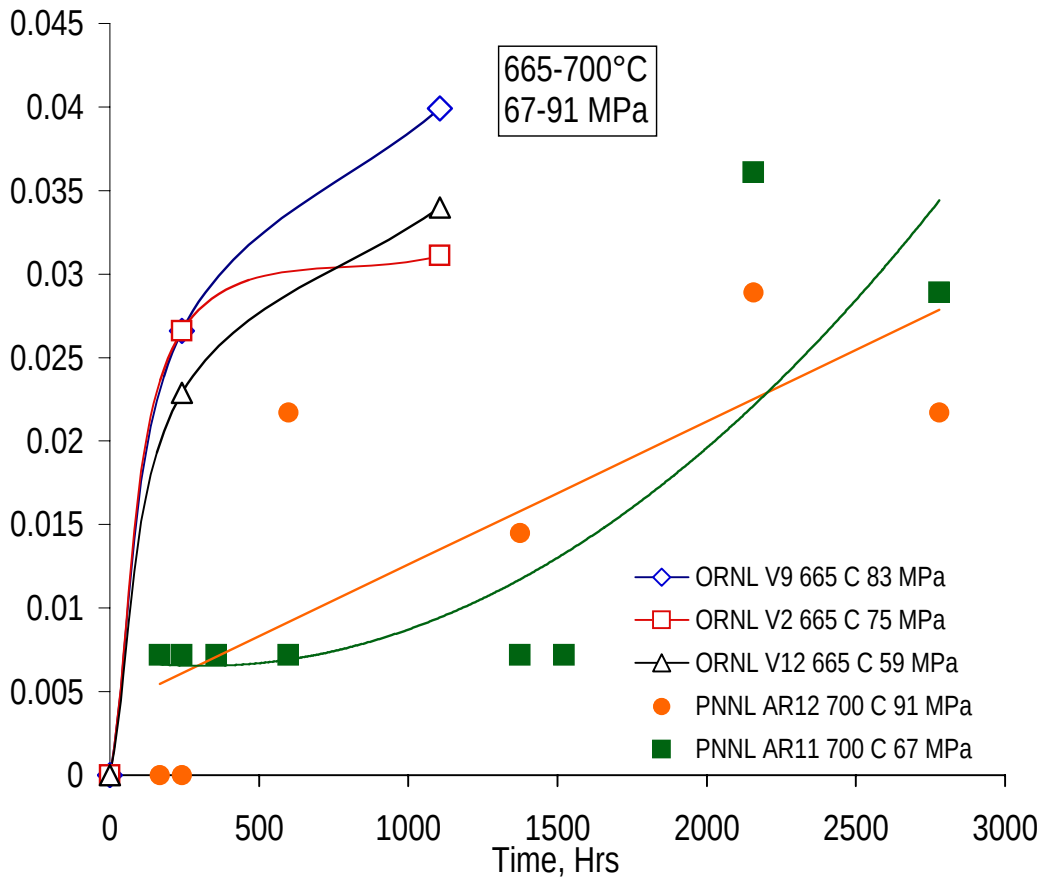


Time Dependence of Effective Creep Strain at 800°C for V-4Cr-4Ti



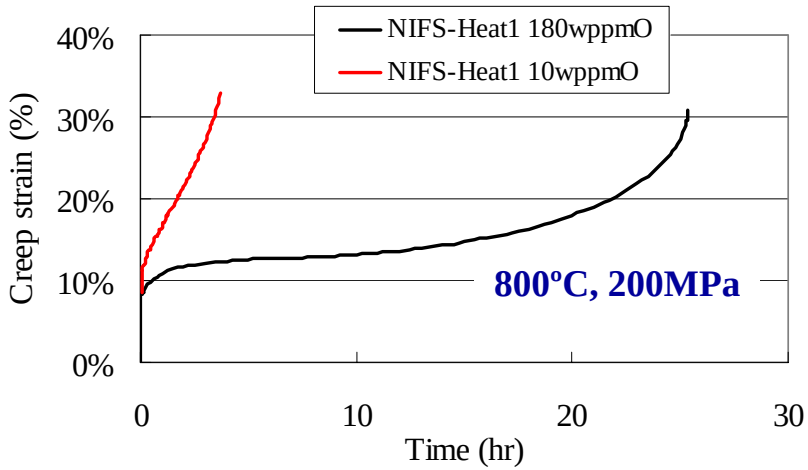
Effect of Li on Creep at 665-700°C

At 665-700°C strains are very small, but the trend of higher creep rates is again shown. Some primary creep is suggested.



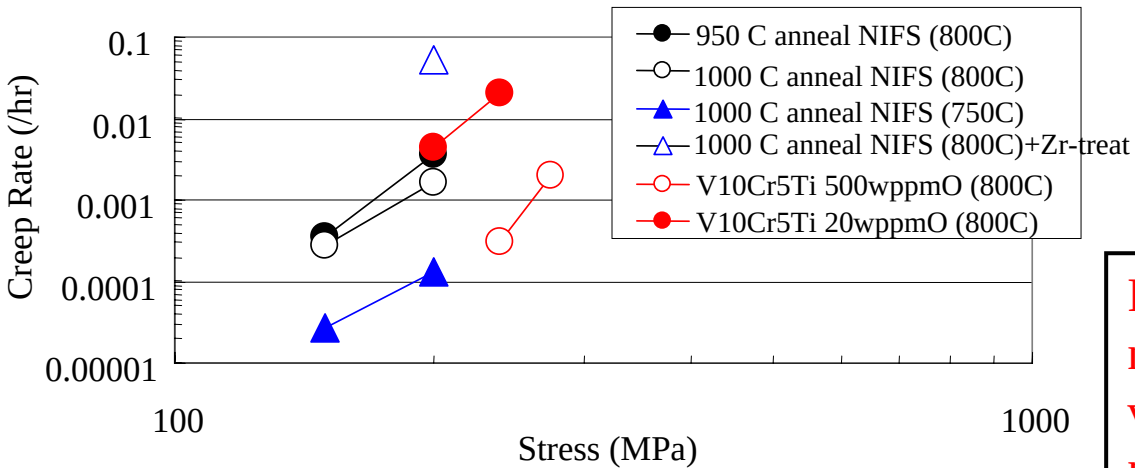
Grossbeck

Effect of Alloy Purity and Cr Content on Creep of NIFS Heat-1



The rupture time and creep strain rate of highly-purified NIFS Heat-1 are shorter and higher, respectively than NIFS Heat-1 with the original heat-treatment (1000° C,2hr).

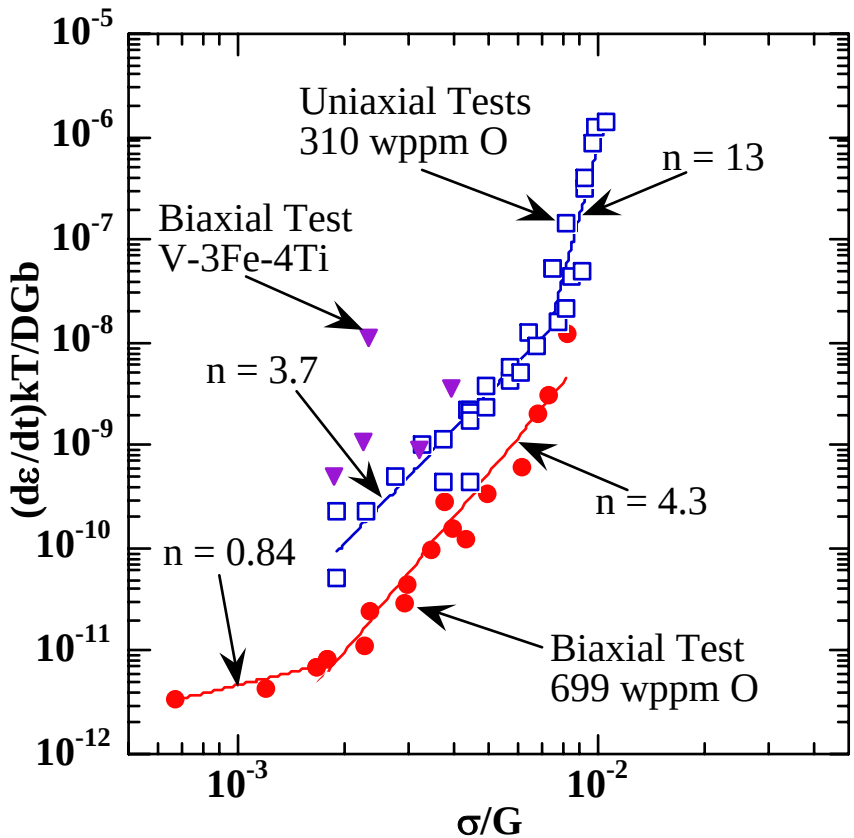
Fukumoto, et al.



- Increasing the Cr content lowers the creep strain rate
- Increasing alloy purity increases the creep strain rate

Purification below 300 wppm may not be suitable from the point of view of high-temperature mechanical properties

Stress Dependence and Activation Energy for Creep



Creep tubes in ~10^{-8} torr vacuum

Activation Energy

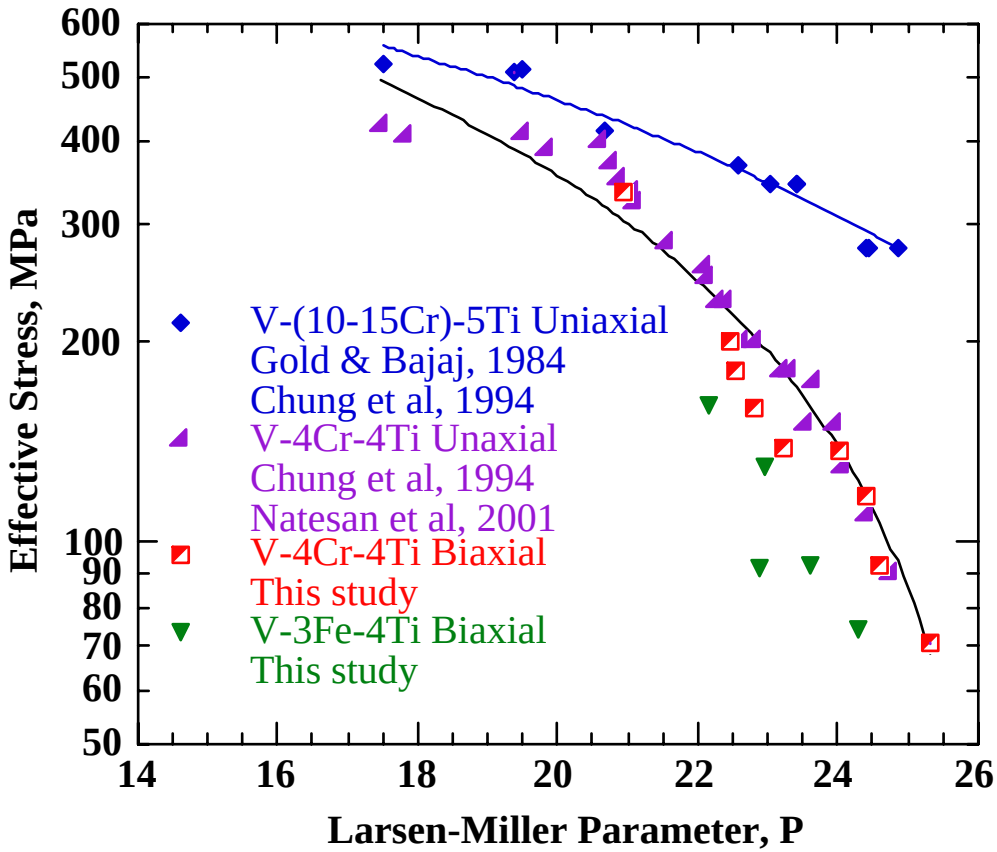
$$Q = \frac{R * Ln(\dot{\varepsilon}_1/\dot{\varepsilon}_2)}{1/T_1 - 1/T_2}$$

Stress, MPa	Activation Energy, kJ/mole
70	272
90	326
120	300

Q_{SD} ~ 270 kJ/mole (Pure V)

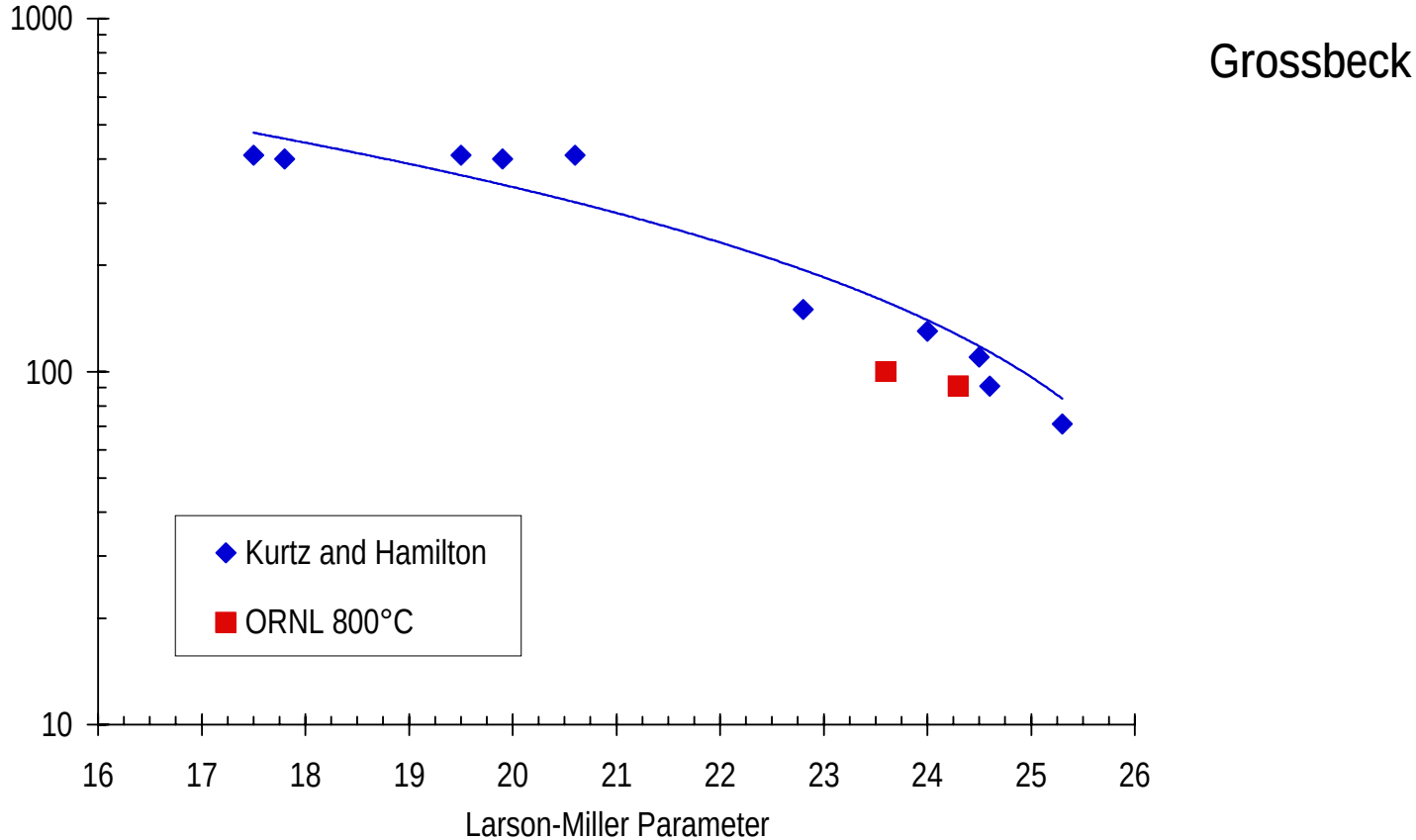
Creep-Rupture of Vanadium Alloys

$$P = \frac{T(\log t_r + 20)}{1000}$$



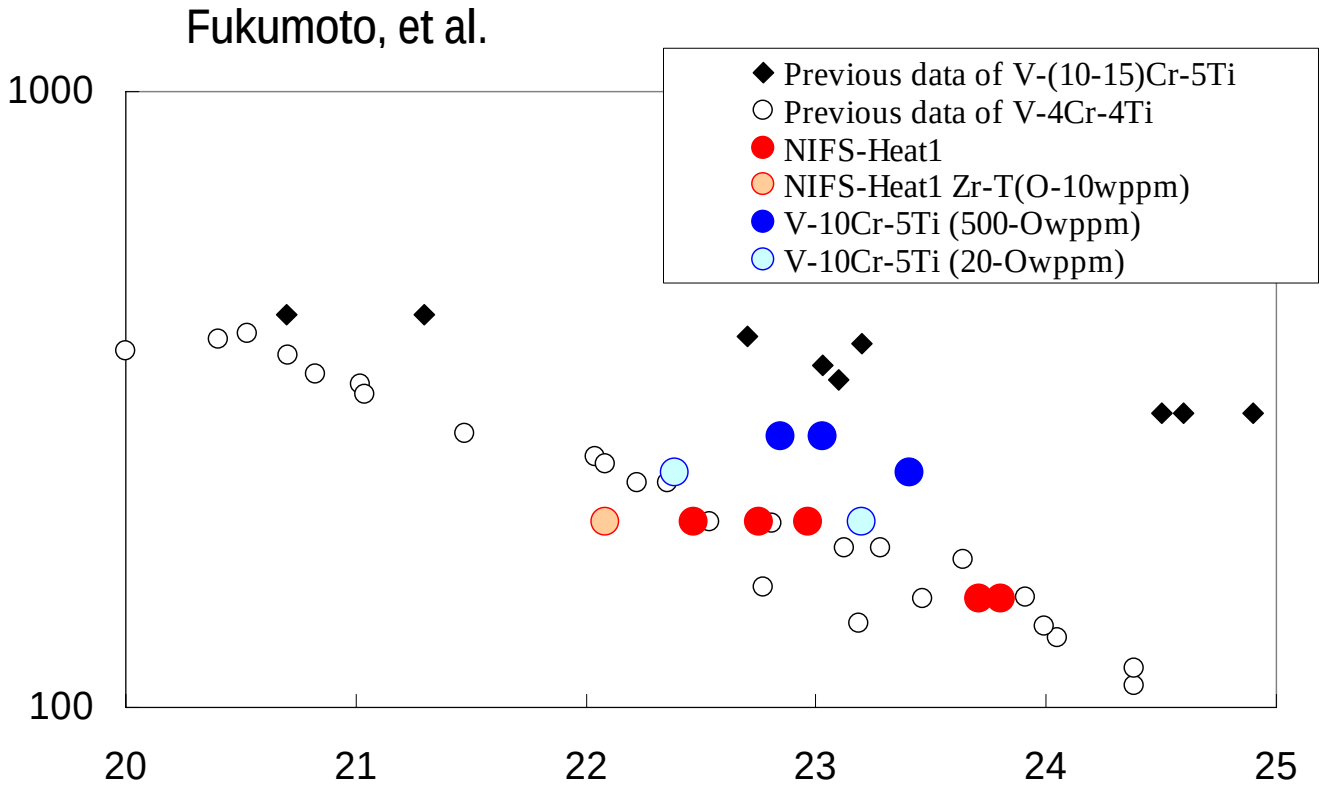
Creep Rupture in Lithium

A Larson-Miller presentation shows that failure in Li occurs slightly prior to vacuum tests.



$P=T\ (\log t_r + 20)/1000$

Effect of Increased Purity and Cr Content on Creep Rupture



- Increasing Cr level improves creep resistance
- Lowering interstitial levels degrades creep performance

Summary of Thermal Creep Tests

- The normalized secondary creep rate ($\varepsilon kT/DGB$) is power-law dependent on stress with n ~ 4 at normalized stresses (σ/G) greater than ~$2x10^{-3}$
- The activation energy, Q, for creep at 700 - 800°C is ~ 300 kJ/mole which is similar to the self diffusion activation energy for pure vanadium
- The predominant creep mechanism appears to be climb-assisted dislocation motion at normalized stresses (σ/G) greater than 10^{-3}
- The creep mechanism may change at lower stresses where the stress exponent is near unity
- The creep rates from tensile samples are several times greater than creep rates from pressurized tubes reflecting the differences in oxygen concentration
- Consistent with low-oxygen and lower strength, creep rates in Li are lower than those in vacuum
- Higher levels of Cr improve creep resistance but increased purification degrades creep performance

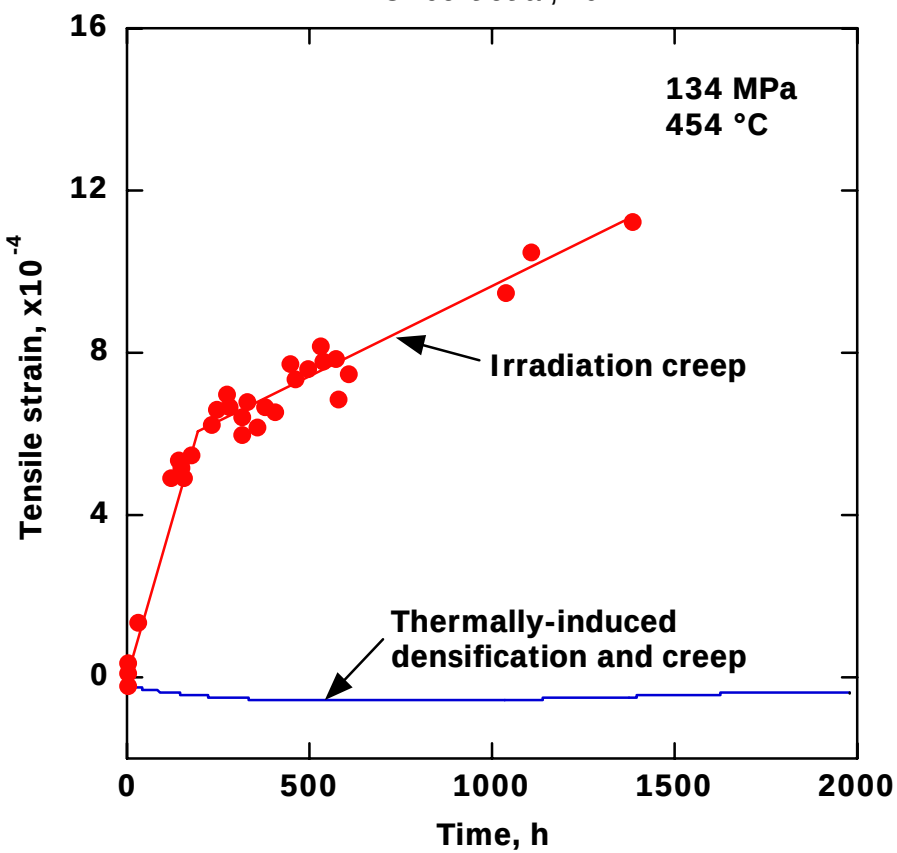
Irradiation Creep - Phenomenological Description

- Orders of magnitude increase in creep strain during relatively low-temperature irradiation
- Degree of creep enhancement is greatest at low temperature, decreasing as the thermal creep regime is approached
- Constitutive equation:

$$\frac{\partial \varepsilon_c}{\partial \phi} = B(\phi, T)\sigma^n, n \approx 1$$

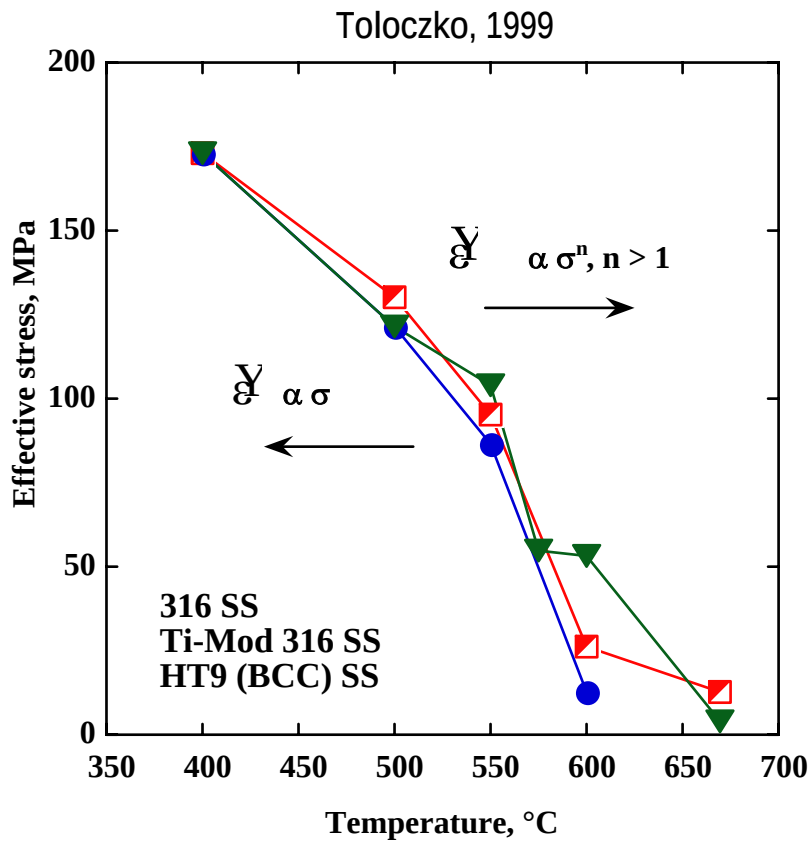
$$B(\phi, T) = B_o + D\frac{\partial S(\phi, T)}{\partial \phi}$$

Gilbert et al, 1972

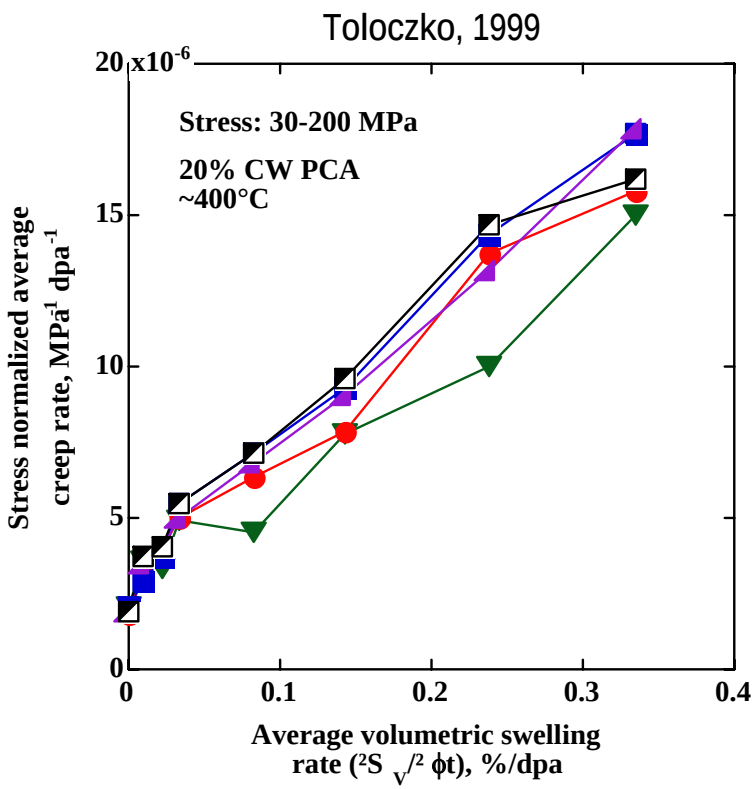


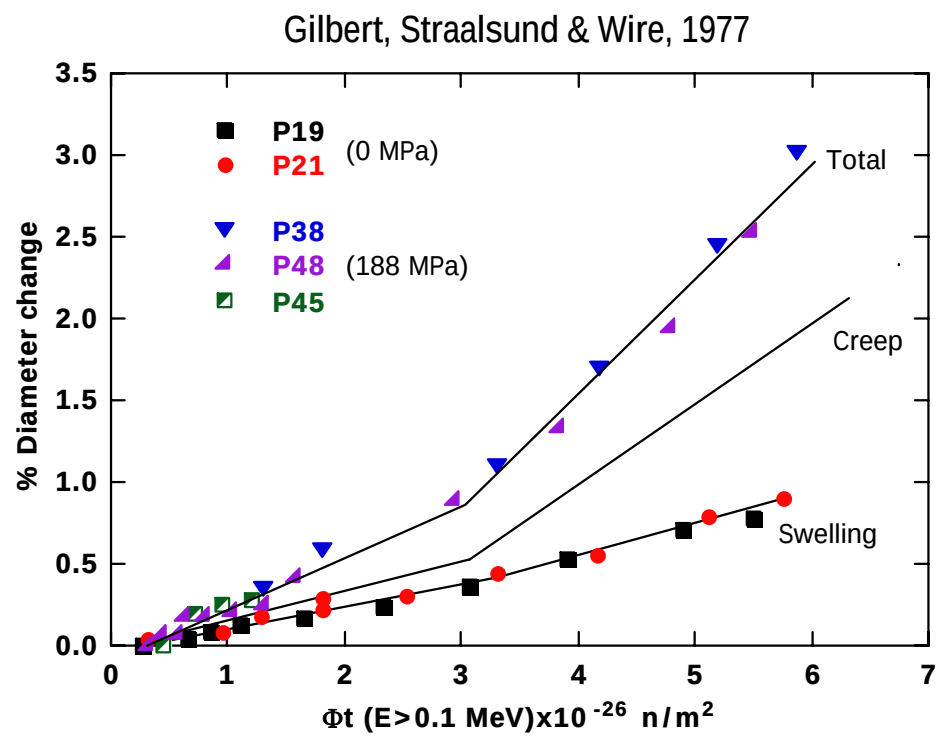
Stress Dependence of Irradiation Creep in the Absence of Swelling

- Irradiation creep exhibits a linear stress dependence over a limited stress range that is temperature dependent
- Similar behavior for FCC and BCC stainless steels

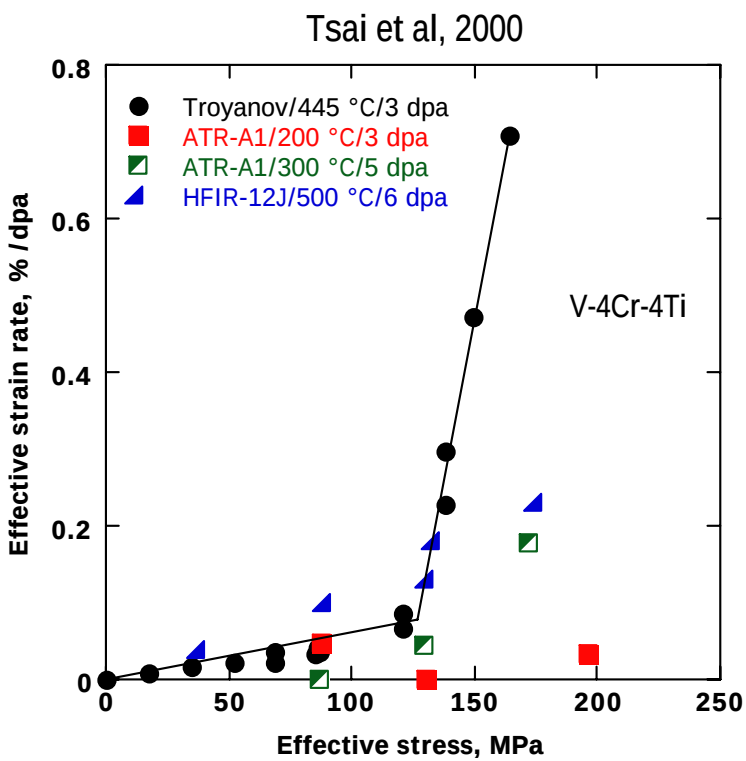


Creep-Swelling Coupling



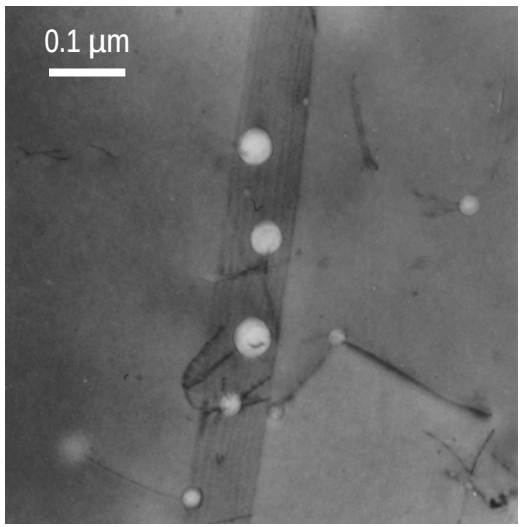


Limited Irradiation Creep Data on Vanadium Alloys

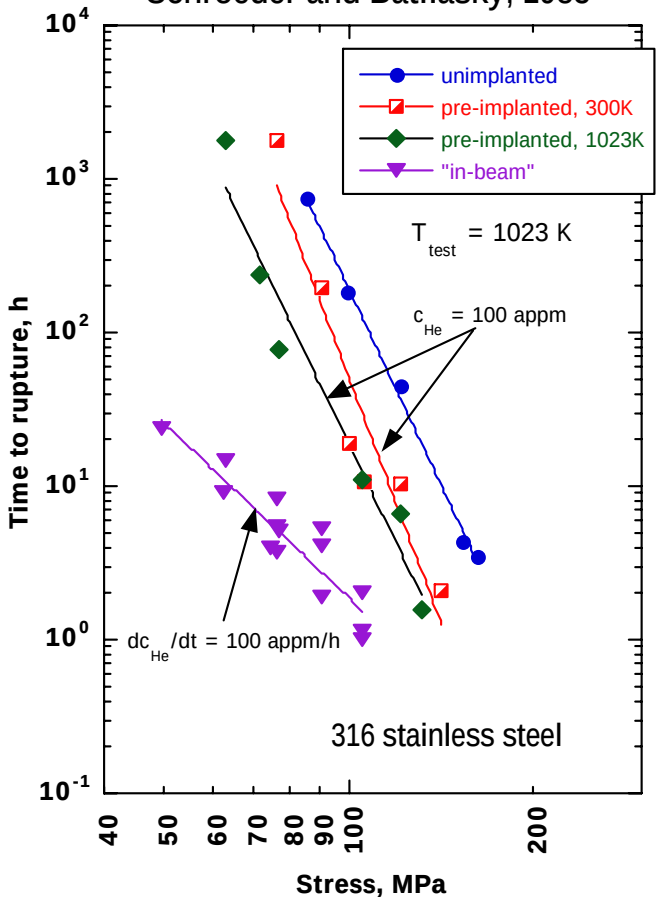


Mechanical Property Degradation Due to He

- Above 0.5 T_m , the coalescence of cavities at GBs can result in intergranular fracture
- The life time under creep and creep fatigue conditions is dominated by the time of stable gas driven growth of bubbles on GBs (rather than by gas driven growth of intergranular cracks

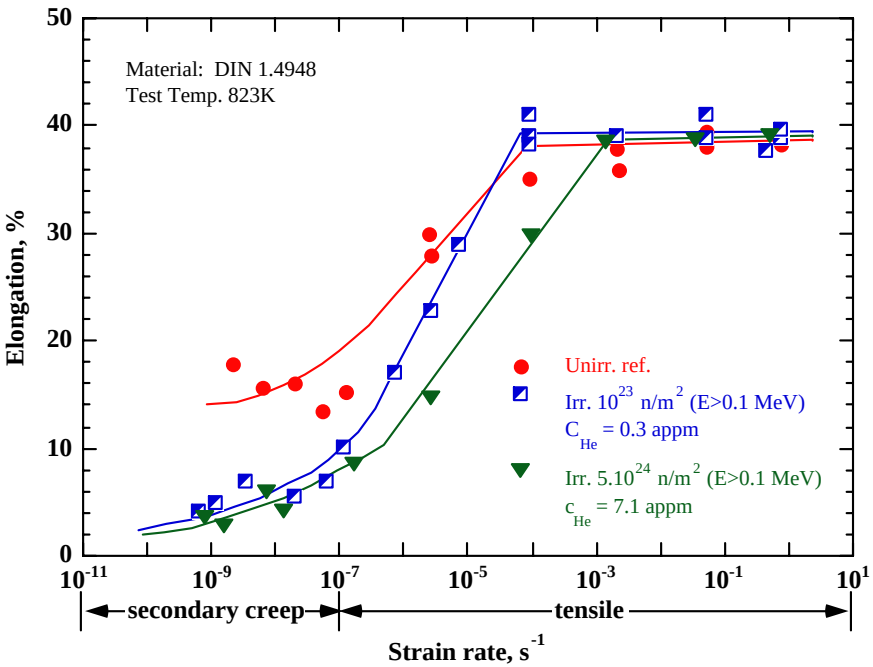


Schroeder and Batflasky, 1983

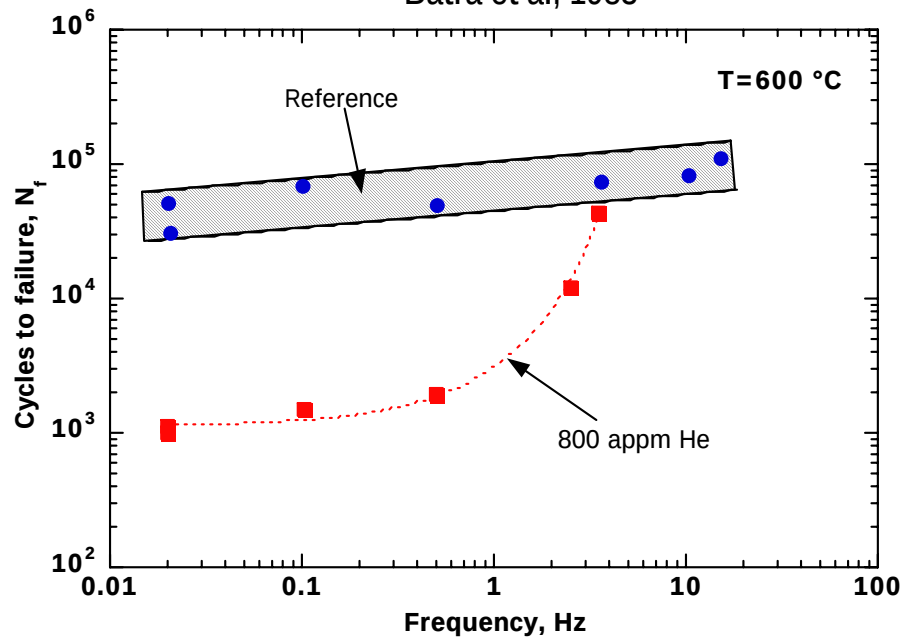


Effect of Strain Rate

Van der Schaaf et al, 1977



Batra et al, 1983

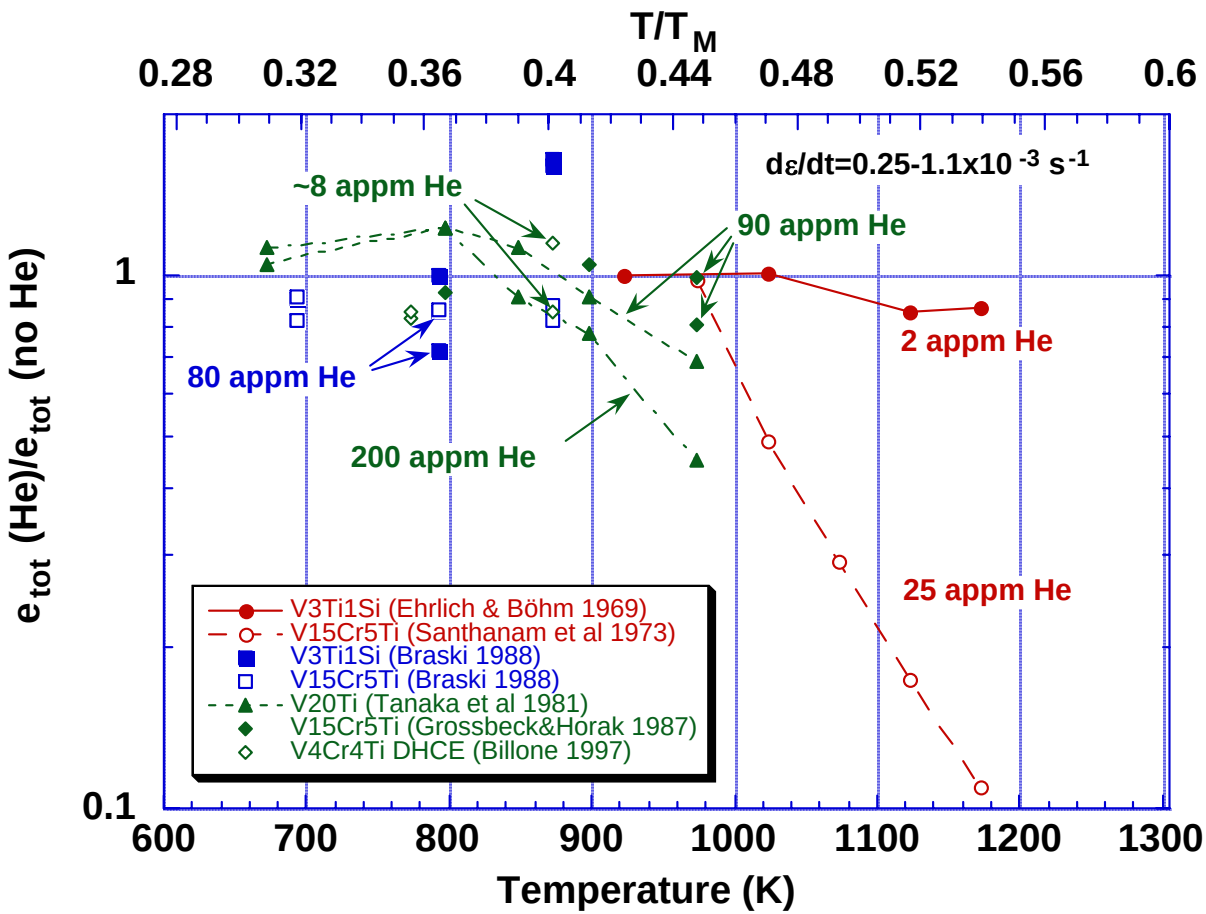


Effect of He on Tensile Properties of Vanadium Alloys

He embrittlement in V alloys becomes evident in tensile tests for:

T > 700°C

C_{He} > 100 appm



Overview of DHCE Concept

- Goal is to simulate fusion reactor He/dpa for V alloys
- Specimens are immersed in Li
- Initial tritium loaded into mother alloy
- Tritium equilibrates with Li/V system when the irradiation starts and the temperature rises
- Tritium in V decays to ^{3}He
- Tritium loss due to decay and diffusion can be replaced by generation from ^{6}Li
- Ideally the He/dpa rate is nearly constant at the desired fusion rate for the entire experiment

DHCE-1 Design Parameters

Parameter	Capsule						
	4D1	**4D2**	**5E2**	**5D1**	**5E1**	**5C1**	**5C2**
DPA	25	27	13	14	18	18	14
Temperature, °C	430	430	430	500	500	600	600
^{6}Li fraction	0.05	0.045	0.01	0.065	0.01	0.08	0.08
Initial Tritium,Ci	99	70	26	74	57	16	18
Final Tritium,Ci	90	66	22	55	37	8.7	10.4
Post-test Tritium, Ci*	46.3	38.4	3.4	ND	~24.1	4.5	12.3
^{3}H / Li,appm (desired)	31,000	22,000	9,300	19,000	14,300	4,900	4,500
^{3}H / Li,appm (limit)**	17,400	17,400	17,400	34,450	34,450	75,700	75,700
K_A* **	0.0136	0.0136	0.0136	0.0197	0.0197	0.0294	0.0294
^{3}H, leakage	4.9×10^{-9}	4.9×10^{-9}	4.9×10^{-9}	2.0×10^{-8}	2.0×10^{-8}	8.9×10^{-8}	8.9×10^{-8}

* Partial measurement of post-test tritium levels. Some tritium remained in solid residues.
** Solubility limit of H in Li.
***K_A is the tritium distribution coefficient between the liquid lithium and the vanadium alloys.

DHCE-1 ^{3}He Data

	Capsule						
	4D1	**4D2**	**5E2**	**5D1**	**5E1**	**5C1**	**5C2**
Calculated ^{3}He	10.1	10.1	5.38	14.2	10.0	4.55	4.34
V	8.83	15.2	2.54	31.6	12.3		10.4
V-5Ti	18.0	12.2	3.05	19.2	7.19		8.10
V-5Ti (BL46)		15.5					8.59
V-5Cr	6.45				3.35		26.3
V-5Nb	7.65				6.78		5.70
V-5Mo	9.81				3.56		8.22
V-5Mo							5.02
V-3Ti-1Si	34.7				5.02		6.25
V-4Cr-4Ti (BL47)	11.6	9.04	2.46	14.0	5.51	8.02	6.80
V-4Cr-4Ti (ANL)	9.86	20.9	2.48	14.1	5.58	7.90	74.0
High ^{3}He Alloys							
V-1Si	105				59.9		48.8
V-5Fe	80.6				34.9		93.4
V-5Cr-5Ti(Si,Al,Y)	177				108		260

Main Conclusions from DHCE-1

- Experiment did achieve accelerated He/dpa levels of 0.3 to 1.0 for most alloys
- Most alloys below fusion He/dpa ratio of 4-5 since K_A lower than expected for most alloys
- Data on most alloys reasonably well fit (factor of 2) by present model of K_A and tritium leakage
- Hydrogen solubility limit needs to be considered
- Three alloys showed much higher ^{3}He, V-1Si, V-5Fe, and V-5Cr-5Ti (Si,Al,Y) (He/dpa = 3-18)
  - Possible explanations: hydride formation, tritium trapping at voids for alloys that swell (V-Cr, V-Si, V-Fe)
- Tritium leakage may be overestimated at 600°C since more ^{3}He in samples than calculated

Ongoing Experiments & Modeling

Summary of Planned US/Japan Irradiation Experiments

- Predominantly US/Japan collaborative irradiation capsules
  - Smaller efforts using informal EU collaborations and HFIR rabbit capsules
- DOE/JAERI Phase IV fusion materials collaboration
  - Focus is on deformation and fracture behavior of ferritic steels (and other alloys), including He effects
  - Three HFIR target capsules (~25 cm3 volume each; 10, 40dpa, 300/400/500˚C
  - Two HFIR RB* capsules: with, without Eu shielding (~200 cm3 volume each), 6dpa, 300/400˚C
  - Typical irradiation matrix: tensiles, fracture, DFMBs
- DOE/MEXT JUPITER-II "ALiVE" capsule
  - Focus is on deformation and fracture behavior of V alloys, including irrad. creep
  - Eu-shielded HFIR RB* capsule, 450/600/700˚C
- DOE/MEXT JUPITER-II HFIR PT-rabbit and RB* capsules
  - Focus is on radiation effects in 3rd-generation SiC composites (and other materials at 600-1400˚C, dpa
  - Tensile, fracture toughness, thermal conductivity

Ongoing and Planned Thermal Creep Studies

- Long-term tests in Li planned to develop more complete data base
- Long-term tests in vacuum at low-stress to explore transition in stress exponent
- Investigation of the affect of metallurgical variables:
  - Characterize NIFS Heat-2 creep tubing
  - Effect of grain size on creep properties
  - Alternative heat treatments to render interstitial impurities benign at low-temperature and also create obstacles to dislocation motion at high-temperature
- Real-Time Helium Doping Experiment
  - He, Li, Thermal
  - Major impediment - funding required

What Needs to Be Done?

What Needs to be Done?

- Thermal creep
  - Alternative compositions and microstructures (heat treatments) for improved performance
- Irradiation creep
  - Lack of data at any dose
  - Effect of alloy composition and heat treatment on irradiation creep
  - Creep-swelling coupling effects
- Helium effects
  - Effect of He on creep and creep-rupture
  - Remaining uncertainties associated with DHCE-1
  - Optimal 2nd phase microstructure for He trapping