Qualification of New Structural Materials

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Joint IAEA-ICTP Advanced Workshop on
Development of Radiation Resistant Materials

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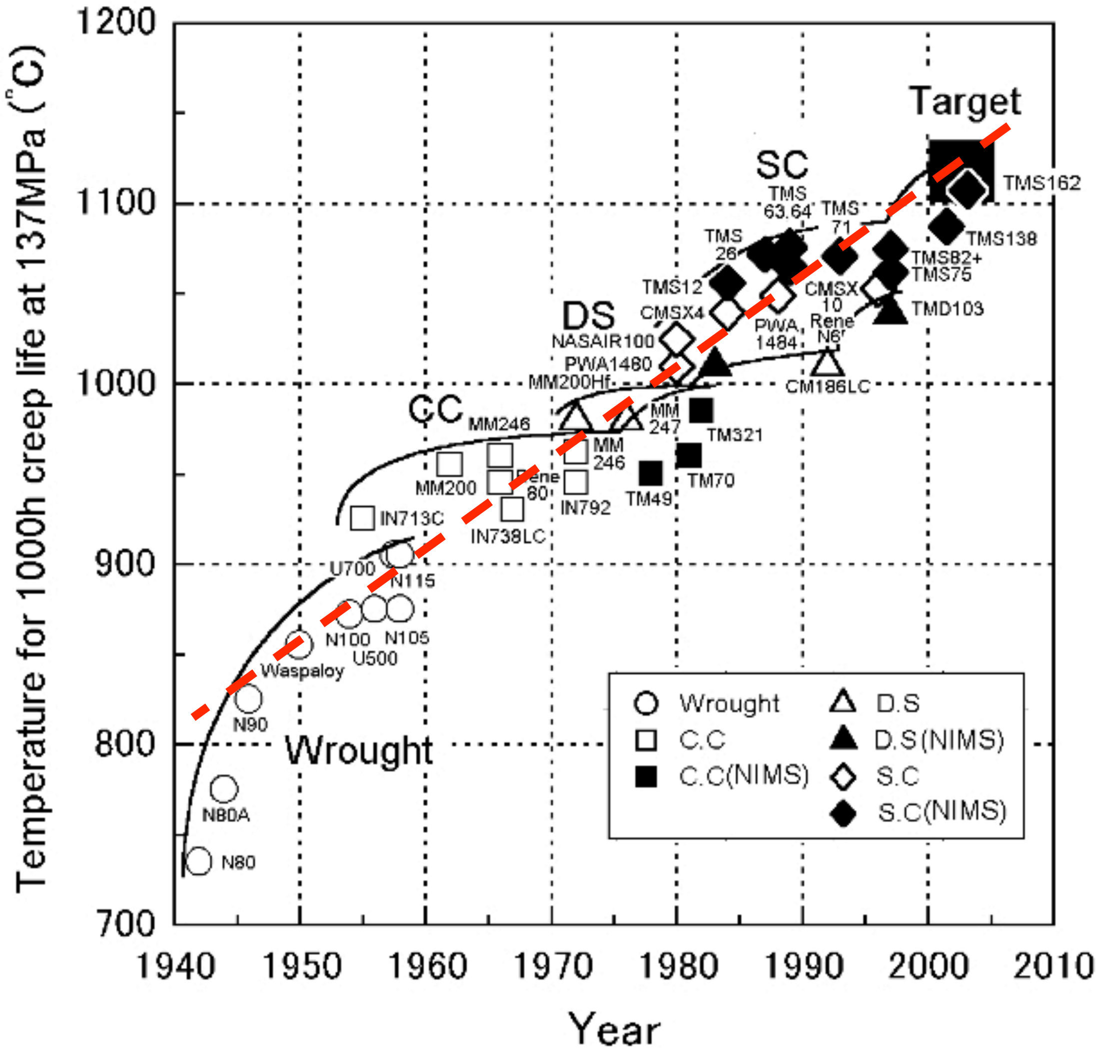
Development of structural materials for applications involving public safety is historically a long process

- "When you hear something about a new material, write it down because it will be the best thing you'll ever hear about it" (Jim Williams, paraphrasing Bob Sprague of General Electric)
- Aerospace structural materials
  - Over 50 years to develop TiAl intermetallics from initial studies in 1950s
  - Design cycle times have been reduced to 3-5 years, but development and qualification of new materials still requires >7 years
    - Qualification time dominated by creep and fatigue testing
- Structural materials for nuclear reactors
  - Qualification requires all of the mechanical property testing on unirradiated material, plus neutron irradiation and testing of irradiated material
    - Sequential approach would lead to unacceptably long qualification times

History of improvement in temperature capability of Ni-base superalloys

- Historical rate of improvement is ~5^{o}C/year



Y. Koizumi et al., Proc. Int. Gas Turbines Conf., 2003, paper TS-119

Qualification of new structural materials involves two considerations based on safety and financial protection

- Cognizant licensing authority
  - Considers public safety aspects
  - Generally requires the structural material to be evaluated by an appropriate independent engineering society (e.g., ASME, ASTM, etc.)
- Capital investment organization (federal government, utility, etc.)
  - Considers potential risk to their investment if a structural material fails
  - Generally requires the structural material to qualified using well-established engineering procedures (e.g., ASME, RRC-MR, JSME, etc.)

The list of structural materials qualified by ASME for high temperature service in nuclear reactors is very small

- Fully qualified (note all of these alloys were developed over 40 years ago!)
  - Type 304 austenitic stainless steel
  - Type 316 austenitic stainless steel
  - 800H austenitic Fe-base superalloy (unacceptable void swelling for doses >50 dpa)
  - 2 1/4 Cr- 1 Mo bainitic steel
- Partially qualified (~30 year old alloy)
  - 9Cr-1Mo ferritic-martensitic steel

ASME Boiler & Pressure Vessel Code Section III, subsection NH

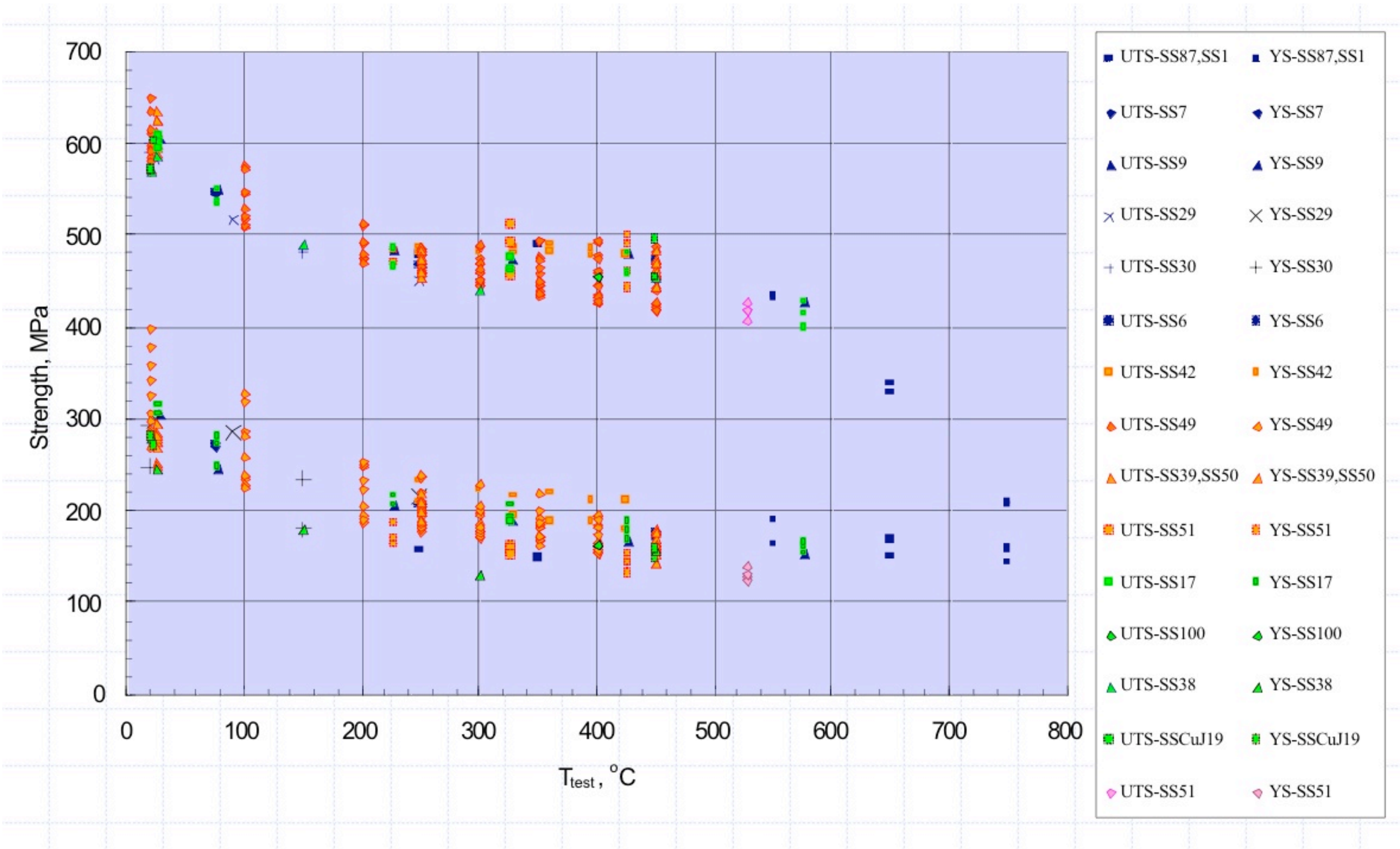
Summary of key steps for ASME code qualification

- Full set of data must be obtained on multiple heats, independently produced
  - Intention is to assure reproducibility of processing procedure
  - Potential effects of product form (e.g., plate vs. tubing) and welding are also assessed
- Key mechanical property test data include:
  - Tensile properties (yield strength, ultimate strength, uniform elongation, total elongation, reduction in area) over the range of anticipated operating temperatures
  - Thermal creep properties (typically 1% deformation, minimum creep rate and creep rupture lifetime) at suitable stresses, temperatures
  - Cyclic fatigue properties (low-cycle and high-cycle)
  - Combined creep-fatigue tests

Determination of design curves

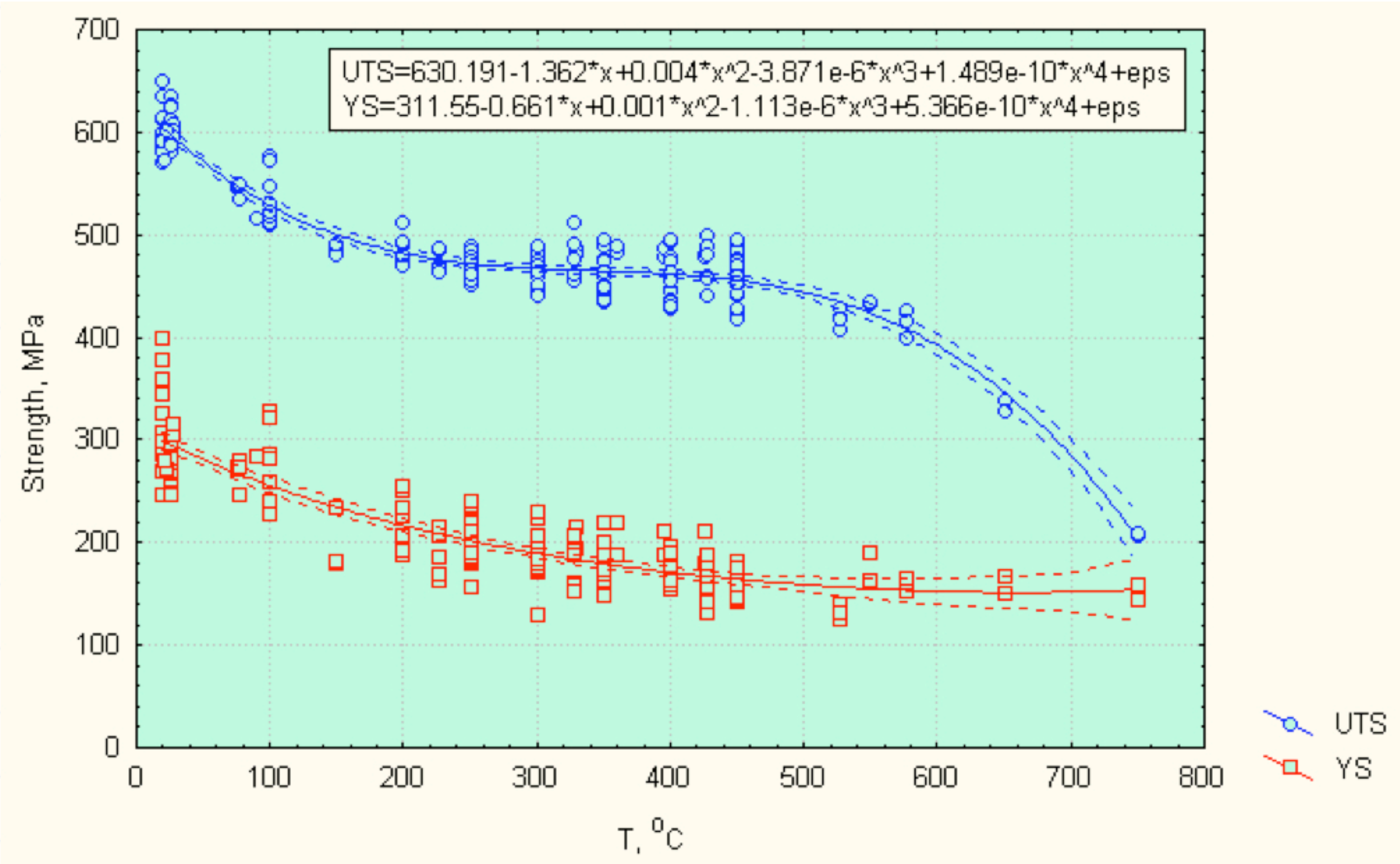
- Tensile strength
  - If large number of test data are available, then design curve can be set at a value equal to two standard deviations below the mean value (represents 97.5% confidence limit)
  - Alternatively, the design curve can be set at the minimum strength values in the data base
- Fatigue data
  - Strain range vs. fatigue cycle design curve is determined by the minimum of either $\varepsilon_t/2$ or $N_f/20$

Example tensile data base for Type 316 stainless steel



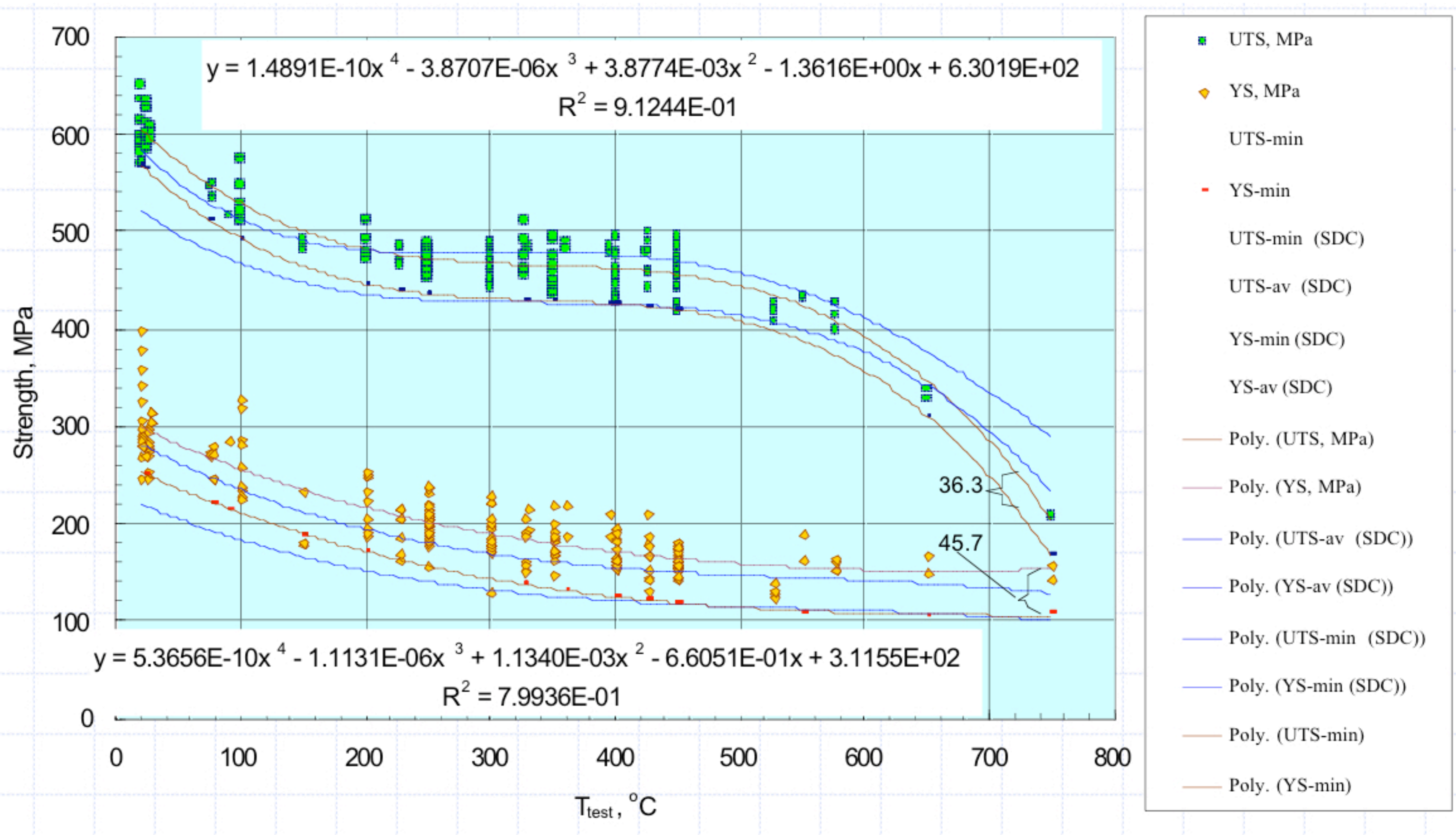
G.M. Kalinin

Mean tensile strengths for Type 316 stainless steel



G.M. Kalinin

Design tensile strengths for Type 316 stainless steel



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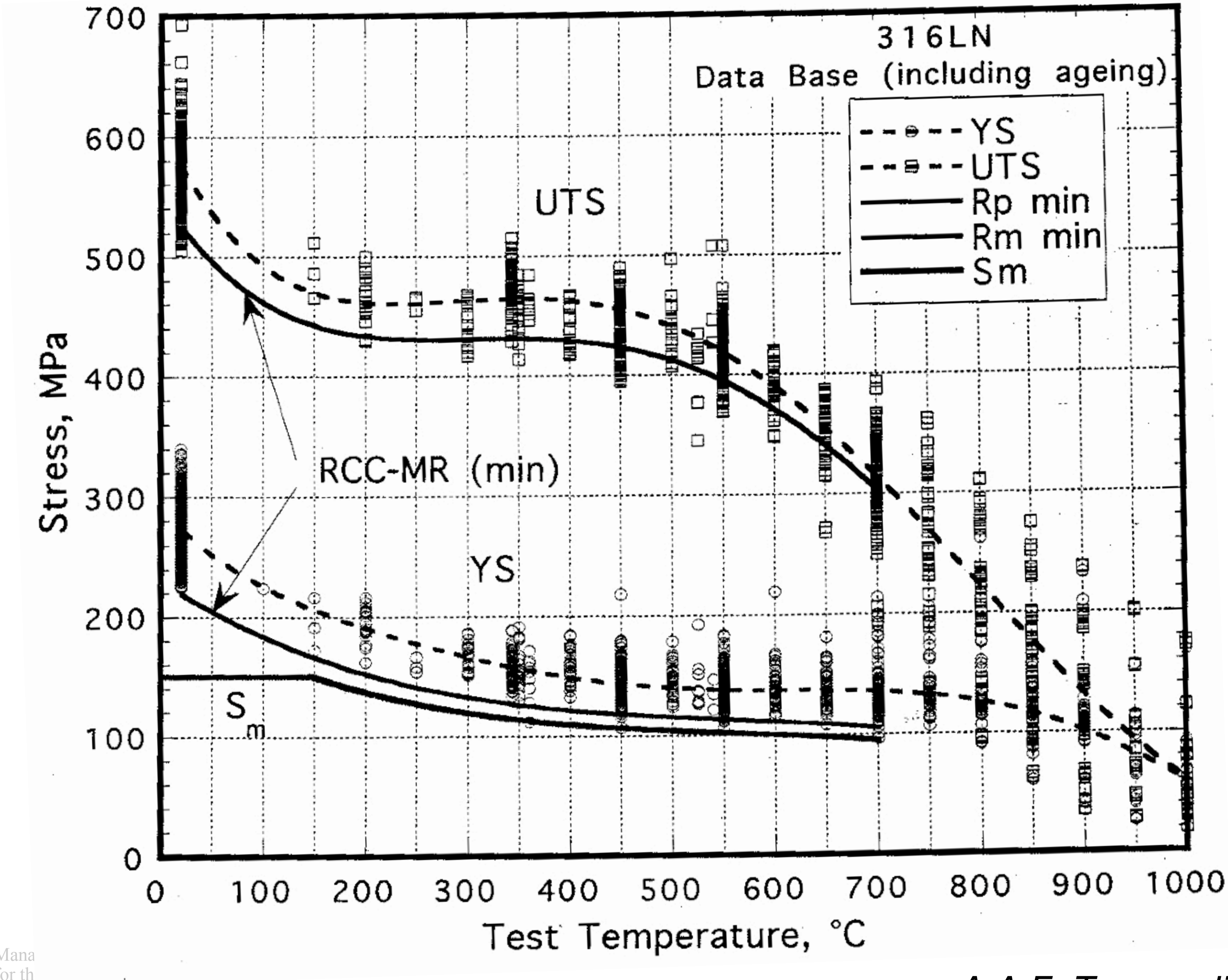
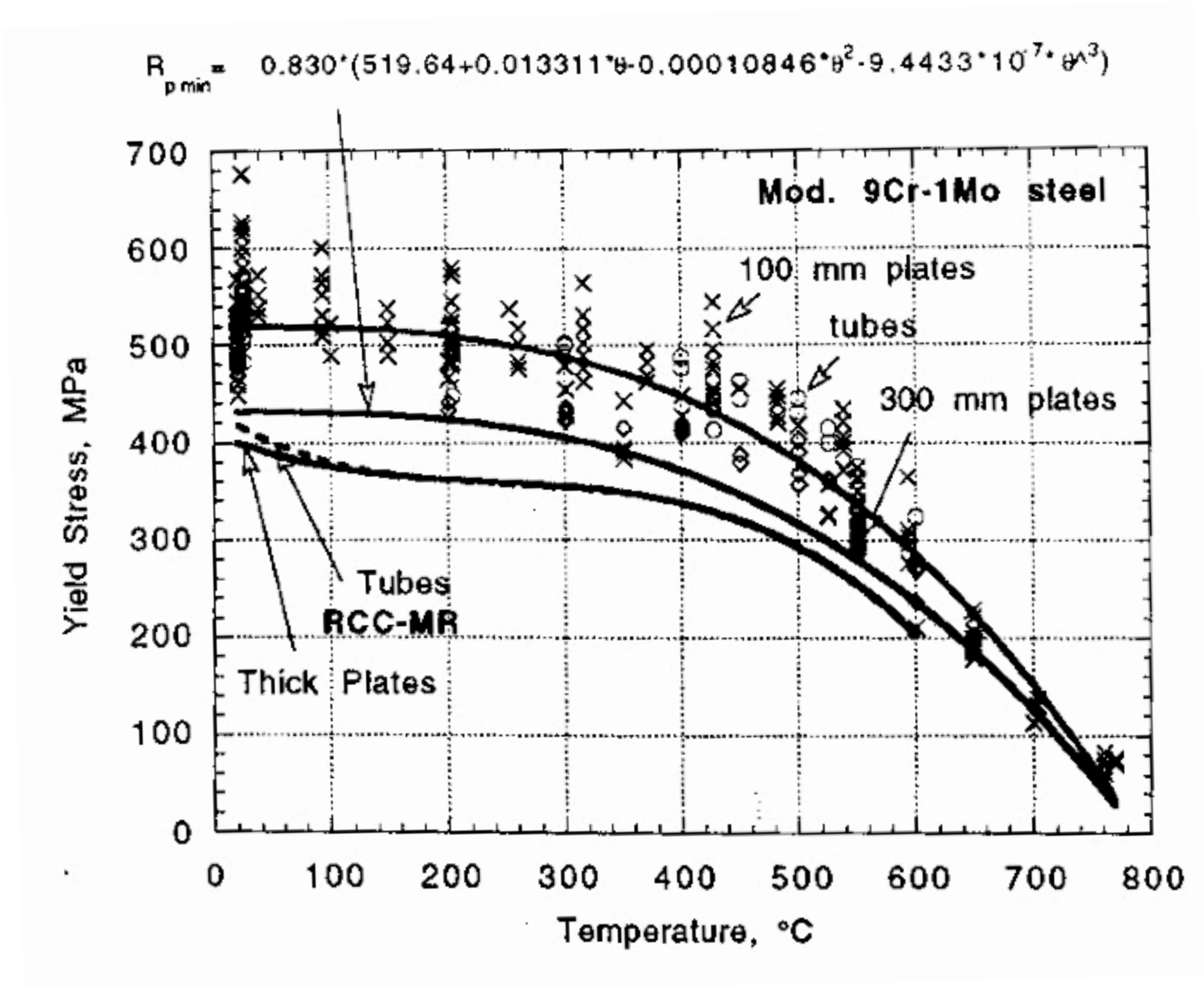


A.A.F. Tavassoli

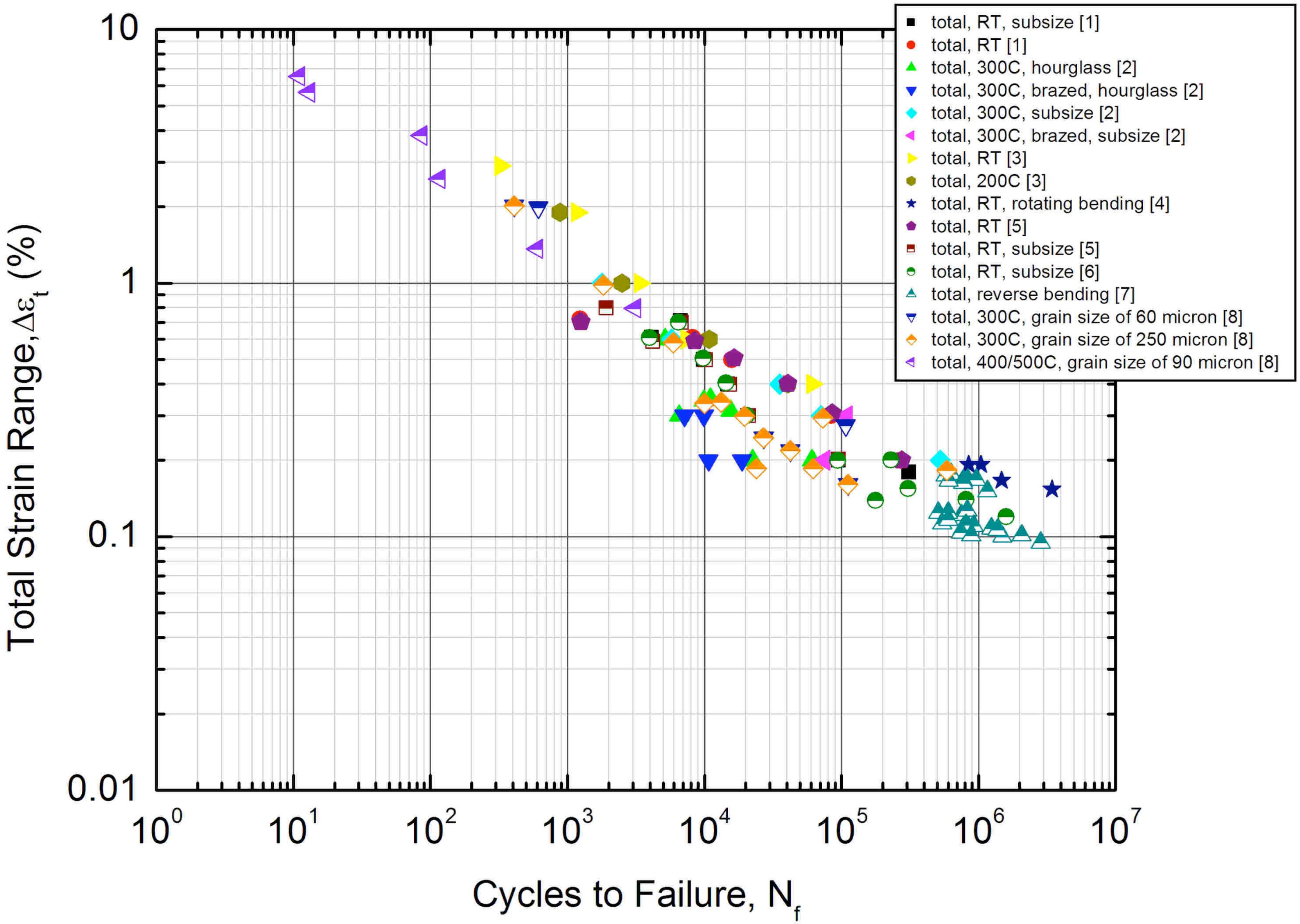
Design yield strength for 9Cr-1Mo ferritic/martensitic steel



A.A.F. Tavassoli

Summary of Pure Unirradiated Cu fatigue data

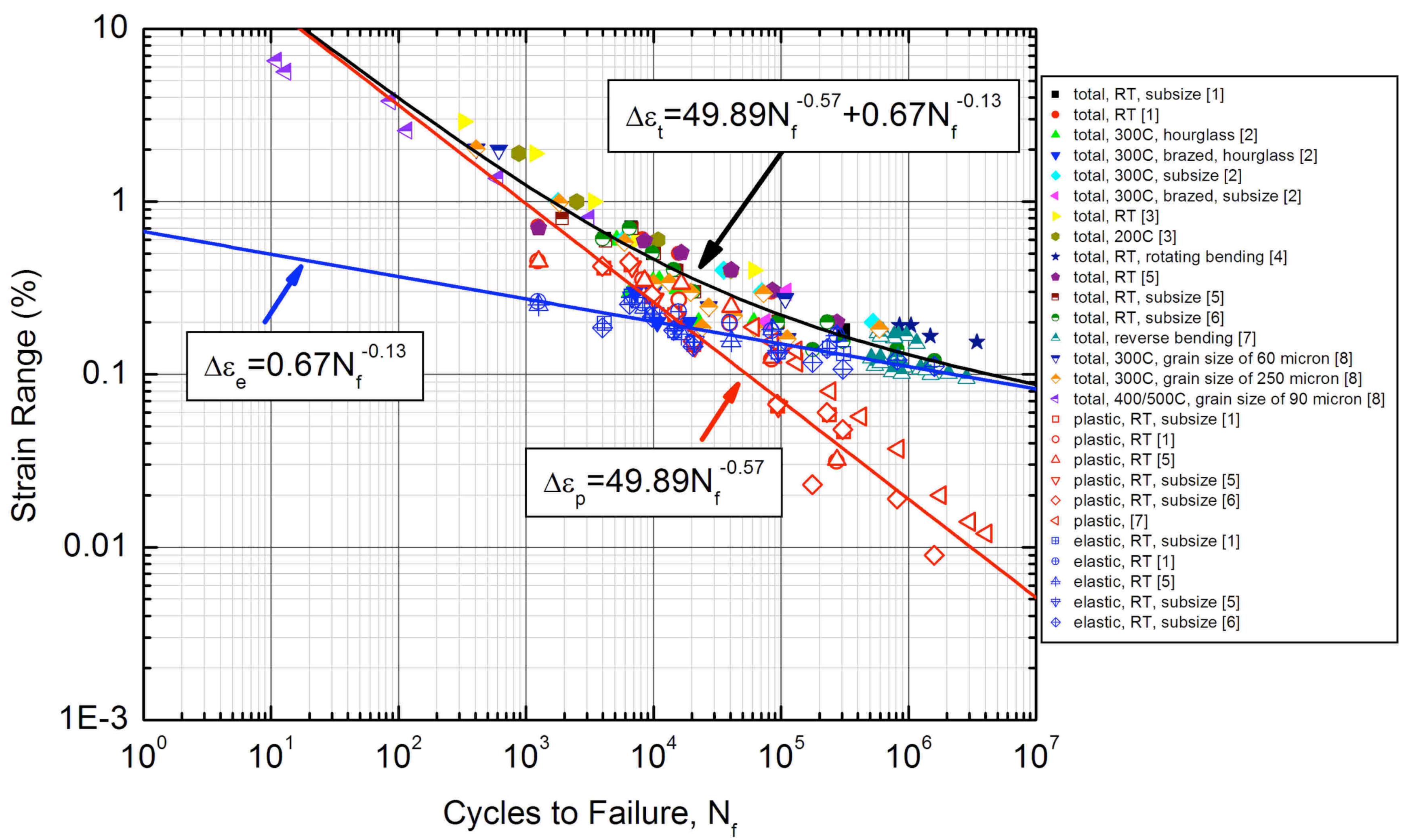
- 8 studies; 214 data points
  - Most data obtained under fullv reversed (R=-1) strain controlled conditions



Coffin-Manson fitted equations for pure Cu

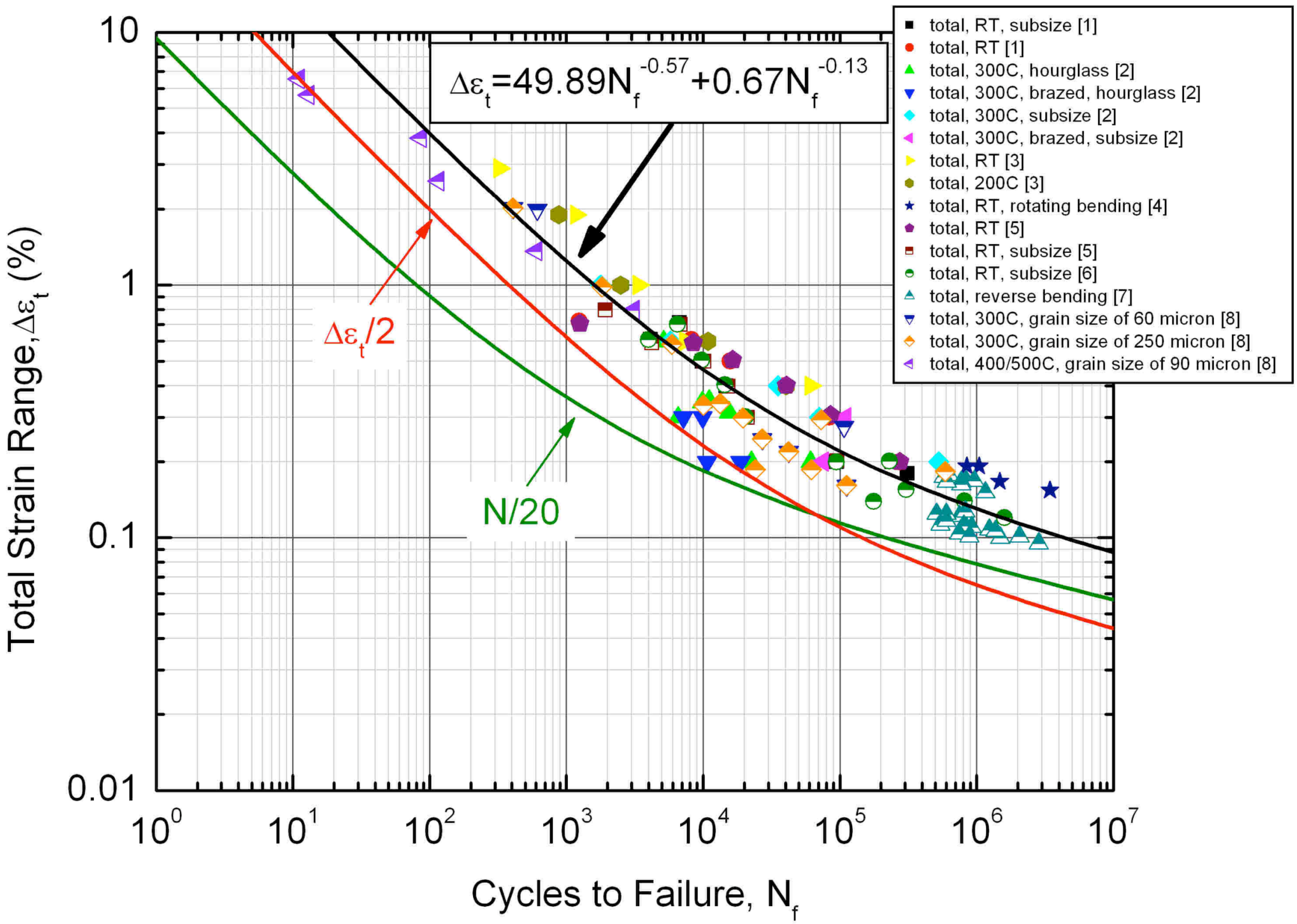
- Unirradiated Cu

$$\Delta\varepsilon_t = A(N_f)^a + B(N_f)^b$$



Summary of design and mean fitted fatigue curve

- Unirradiated Cu



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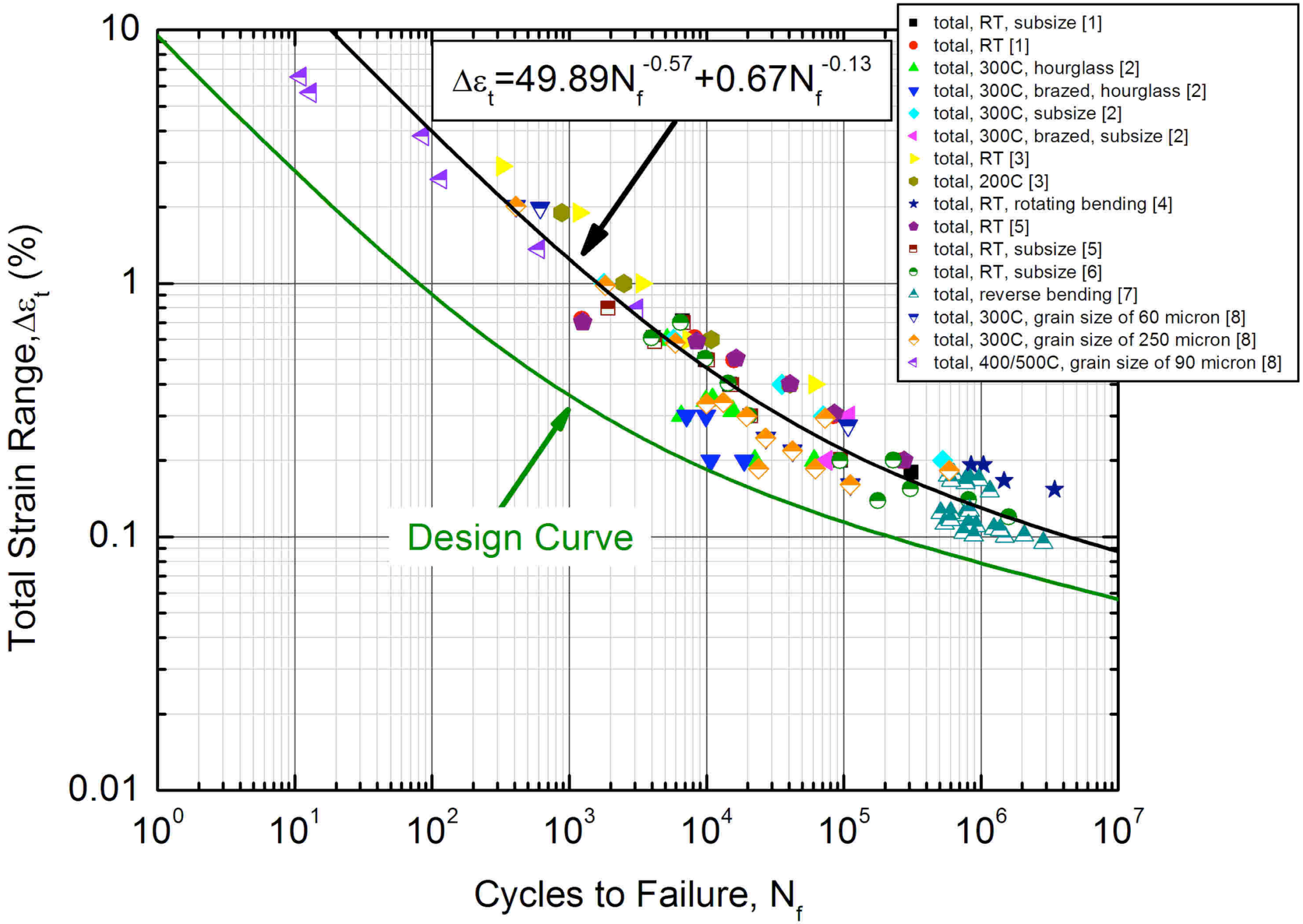
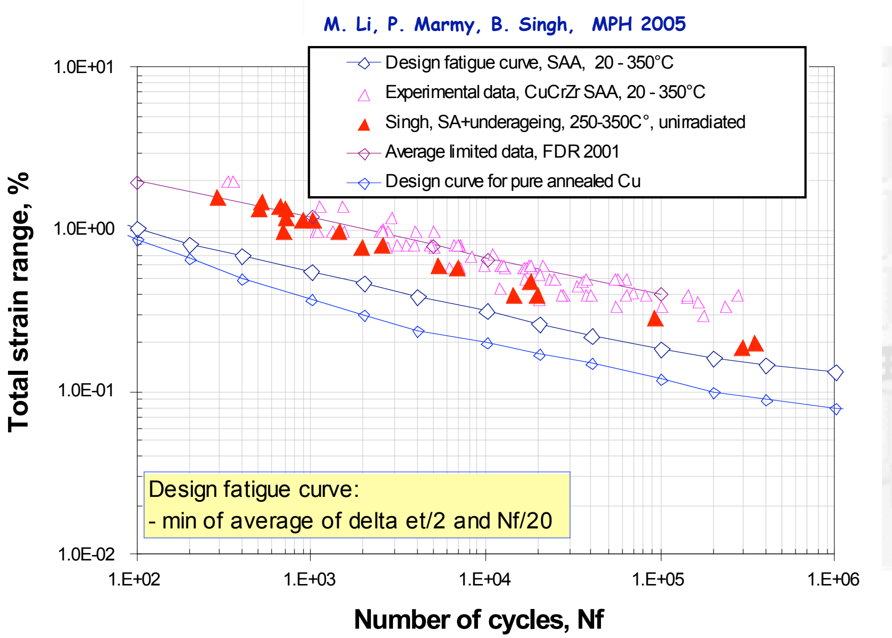


Fatigue and creep-fatigue for CuCrZr alloy

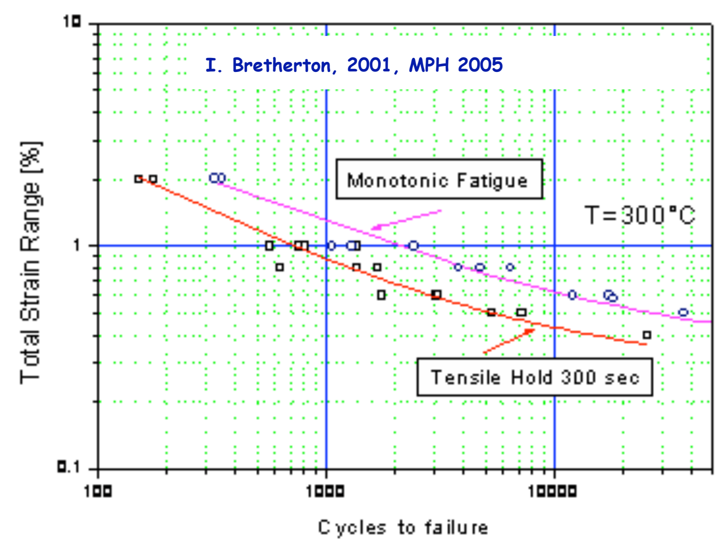
Fatigue:
- Fatigue performance is similar for CuCrZr with different processing treatments

Creep – fatigue interaction:
- very complicated performance:
  => Generally there is reduction of lifetime with hold time; => Higher Sy – higher fatigue lifetime

Fatigue



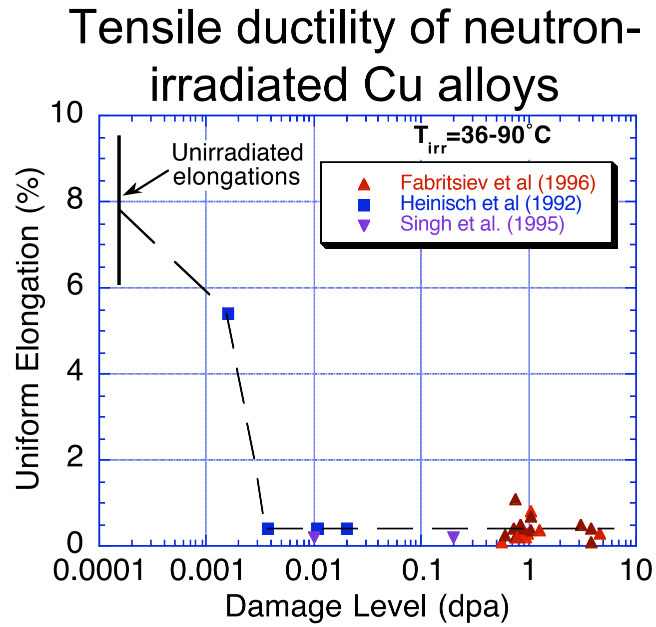
Creep-fatigue



V.R. Barabash

V.Barabash et al., ITER Materials, ICFRM-12, December 4 - 9, 2005, Santa Barbara, USA

Low tensile ductility in FCC and BCC metals after irradiation at low temperature is due to formation of nanoscale defect clusters



Outstanding questions to be resolved include:

- Can the defect cluster formation be modified by appropriate use of nanoscale 2nd phase features or solute additions?
- Can the poor ductility of the irradiated materials be mitigated by altering the predominant deformation mode? (e.g., twinning vs. dislocation glide)

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Stainless steel 316L(N)-IG

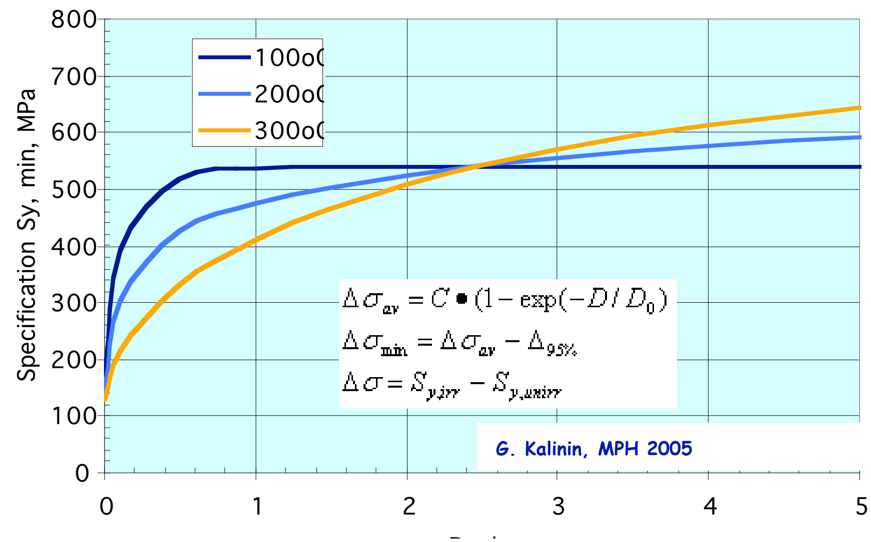
ITER
The Way to Fusion Energy

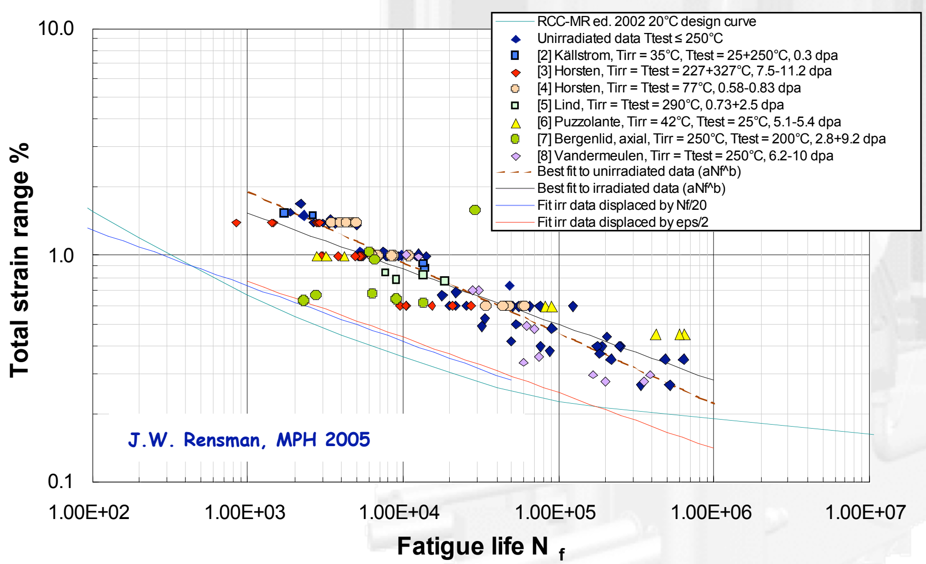
Neutron effect:

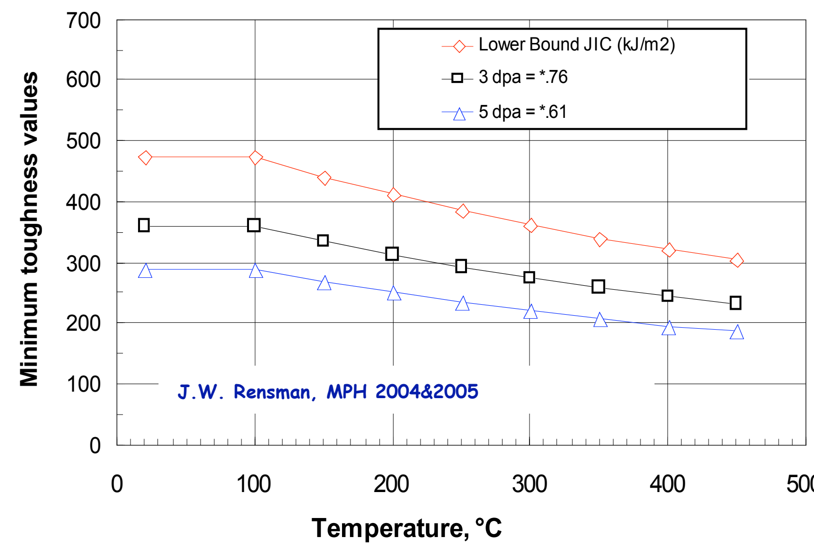
Tensile properties:
-Strengthening and loss of ductility (loss of strain hardening)

Fatigue:
- no effect on fatigue

Fracture toughness:
- reduction, but material still ductile







V.R. Barabash

Vladimir Barabash, ITER Team, presentation at CEA-Cadarache, Jan 27, 20065

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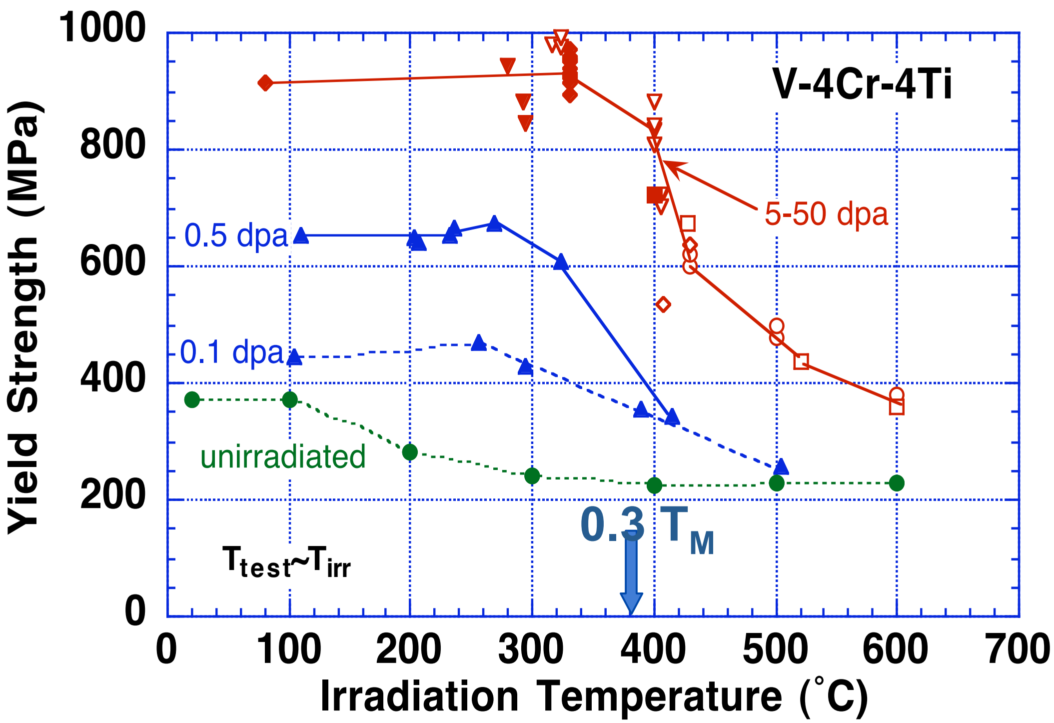
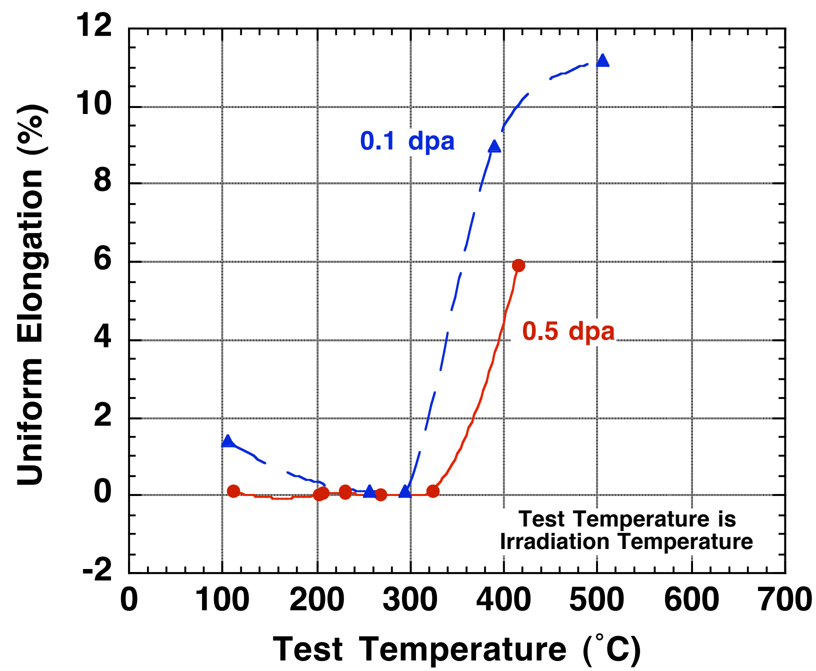
Radiation hardening in V-4Cr-4Ti

High hardening and loss of uniform elongation occurs for irradiation and test temperatures <0.3 T_M





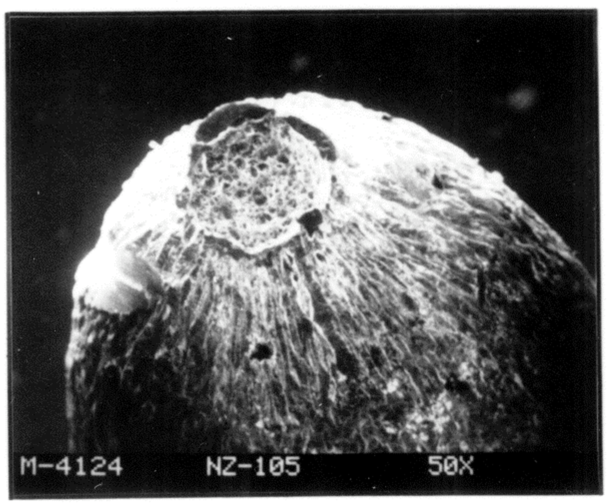
Fracture surface of Irradiated Nb-1Zr shows ductile behavior, despite low uniform elongation value

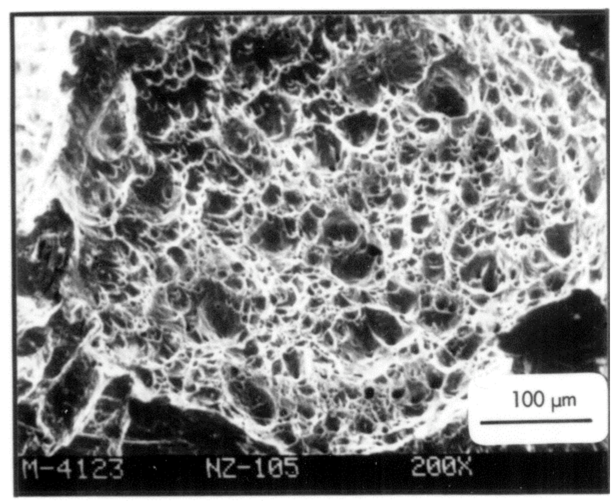
Nb-1 Zr

0.22 dpa at ~70 °C
[4.5×10^{20} n/cm^2 (>0.1 MeV)]

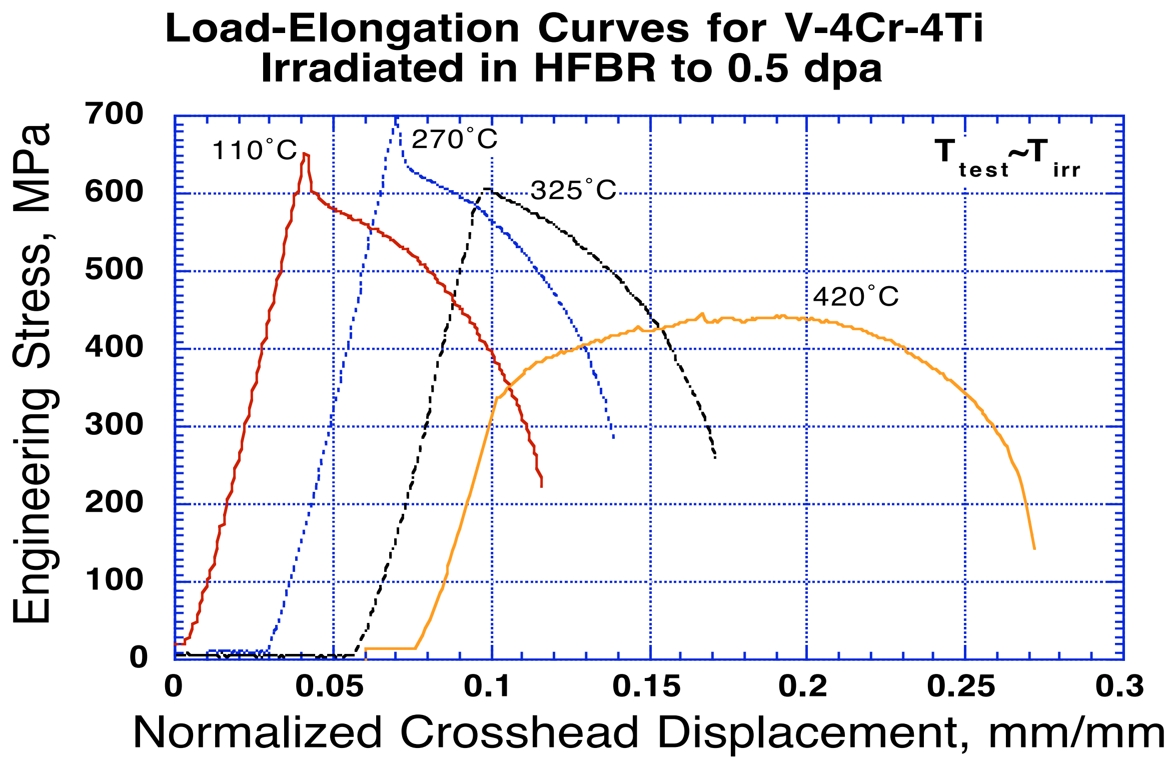
Tensile Test at ~35 °C

0.2 % Uniform Elongation
9.6% Total Elongation

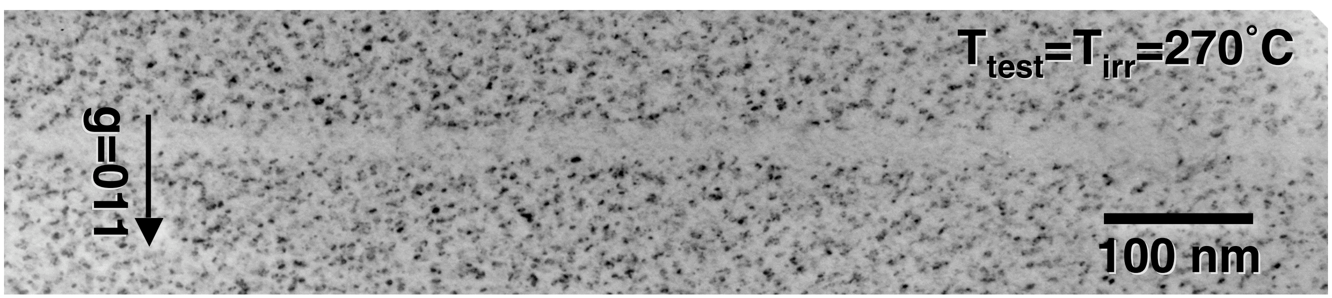




F.W. Wiffen, unpublished results

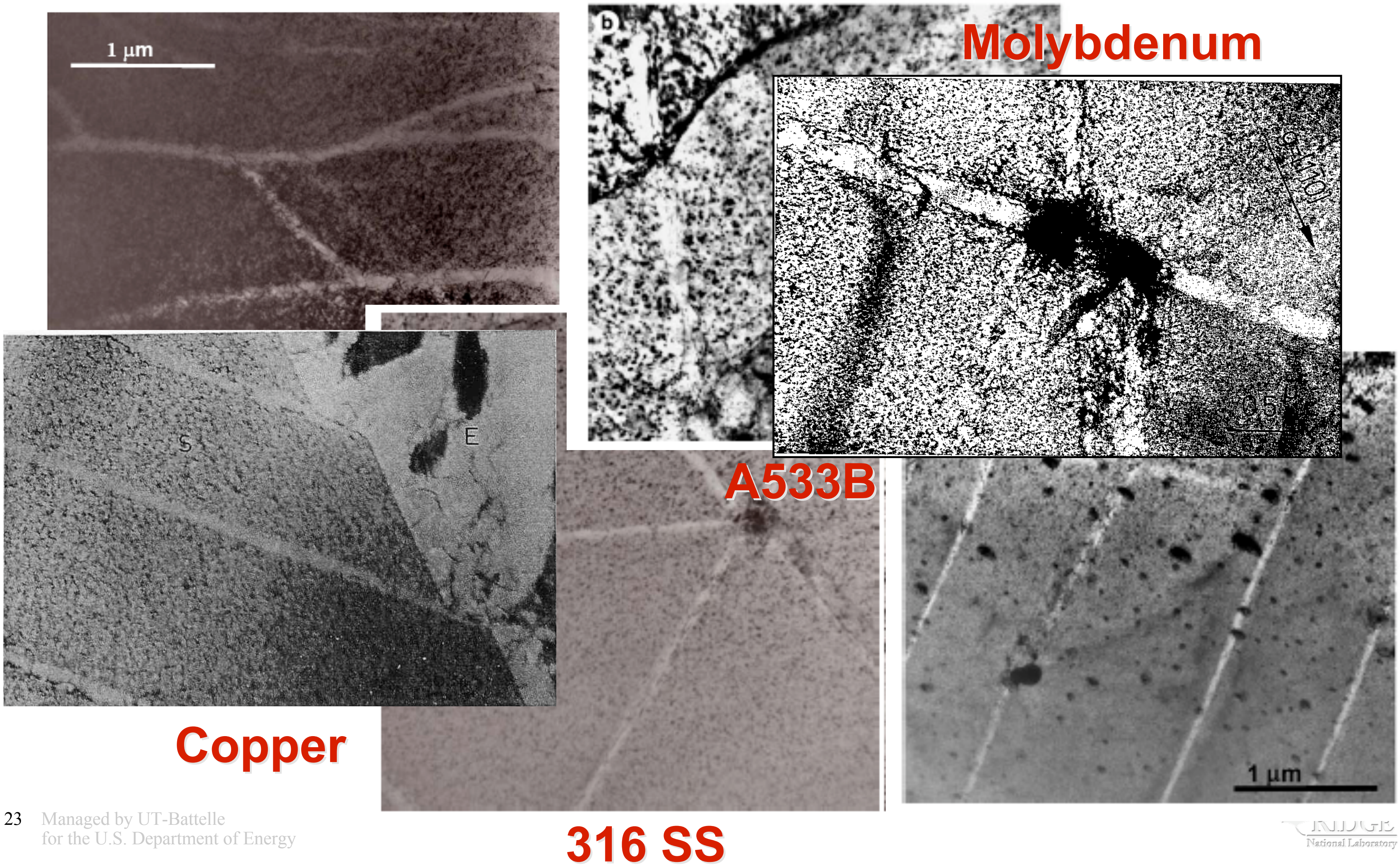


Load-Elongation Curves for V-4Cr-4Ti
Irradiated in HFBR to 0.5 dpa
Engineering Stress, MPa
Normalized Crosshead Displacement, mm/mm
110˚C
270˚C
325˚C
420˚C
T_{test}~T_{irr}
0
100
200
300
400
500
600
700
0.05
0.1
0.15
0.2
0.25
0.3



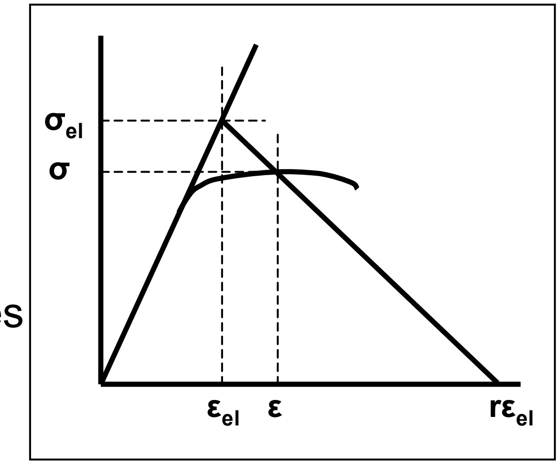
T_{test}=T_{irr}=270˚C
g=011
100 nm

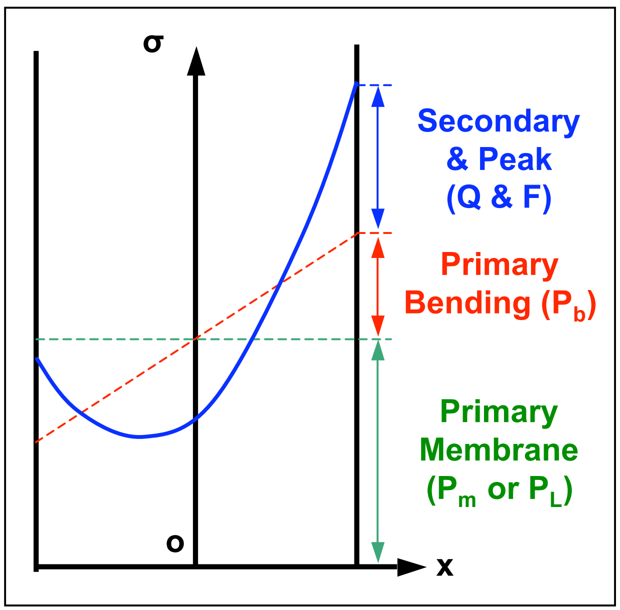
Localized deformation (and dislocation channeling) occurs in many irradiated material systems



ITER Structural Design Criteria (ISDC)

- Based on French Fast Breeder Reactor Code (RCC-MR) and ASME B&PV Code
- RCC-MR and ASME assume materials are sufficiently ductile
- ISDC considers brittle materials by placing limits on the elastic follow-up factor ($r = \infty$ for $\varepsilon_u < 2\%$) to reduce stress allowables
- ISDC adds 2 new rules for embrittled material and establishes limits for negligible creep and swelling





Rule #1 – Prevent Plastic Flow Localization

Combines primary and secondary membrane stresses across structure thickness

Limits (P_L + Q) < 1/3 $S_{ult,min}$ for $\varepsilon_u < 2\%$

Rule #2 – Prevent Exhaustion of Ductility

Combines primary membrane and bending with peak secondary stresses at a critical points

Limits (P_L + P_b + Q + F) < 2/3 $S_{ult,min}$ for $\varepsilon_u < 2\%$

Creep and Swelling Limits

Thermal Creep < 0.05% at yield stress
Irradiation Creep < 0.05% at yield stress
Irradiation Swelling < 0.05% by volume

The Operating Window for BCC metals can be Divided into Four Regimes (red values are relevant for Nb1Zr)

I, II: Low Temperature Radiation Embrittlement Regimes

- Fracture toughness (K_J) embrittlement: high radiation hardening causes low resistance to crack propagation (occurs when S_U>500-700 MPa)
  - Regimes which cause K_J<30 MPa-m$^{1/2}$ should be avoided (T_{irr}< ~600 K ?)
- Loss of ductility: localized plastic deformation requires use of more conservative engineering design rules for primary+secondary stress (S_e)

$$S_e = \begin{cases} \frac{1}{3} S_u & \varepsilon_U < 0.02 \\ \frac{1}{3}\left[S_u + \frac{E(\varepsilon_U - 0.02)}{8} \right] & \varepsilon_U > 0.02 \end{cases}$$

(T_{irr}< ~900-1270 K)

where ε_U is uniform elongation, S_U is ultimate tensile strength, E is elastic modulus (additional design rules also need to be considered)

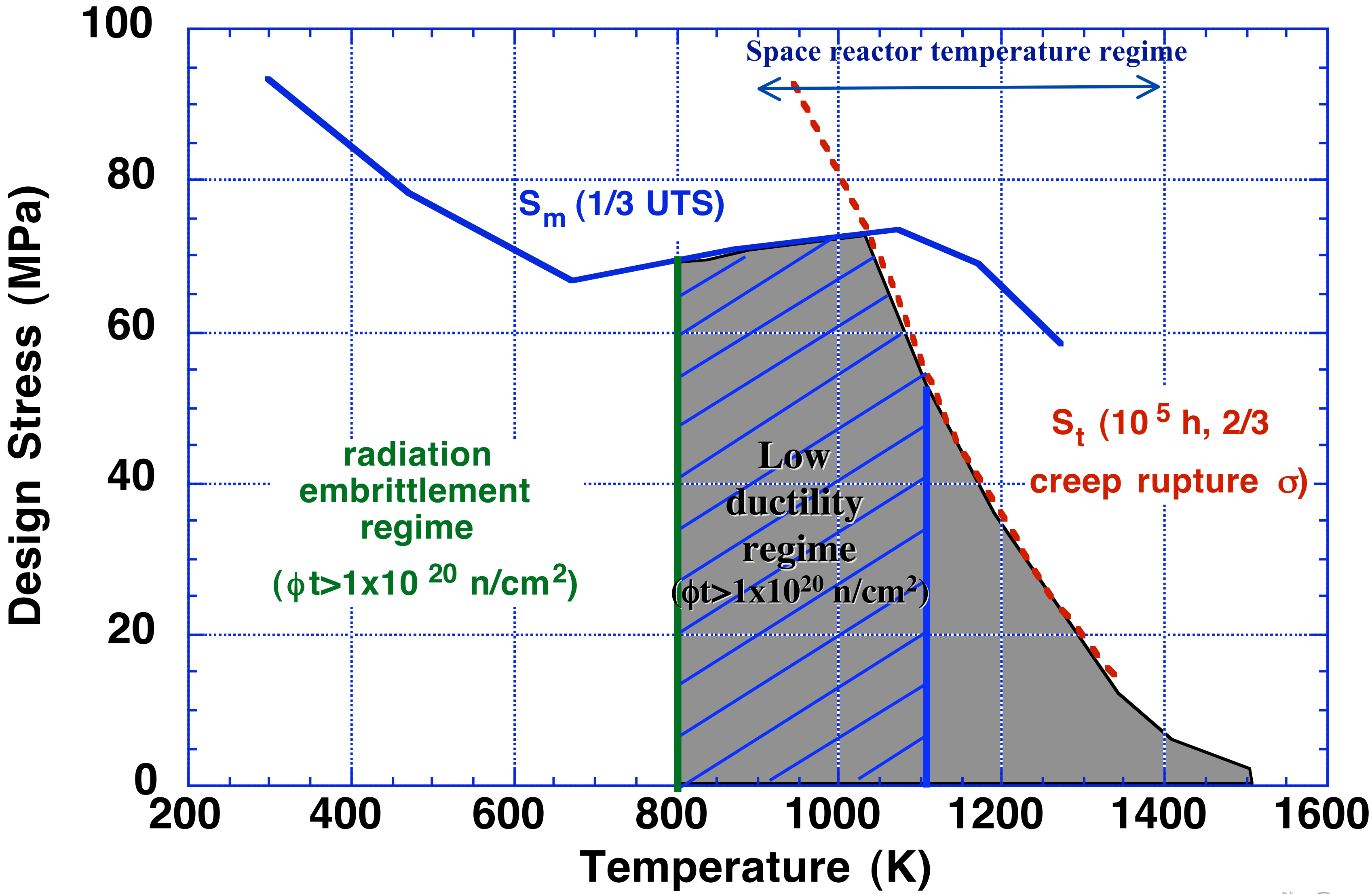
III: Ductile Yield and Ultimate Tensile Strength Regime (e_U>0.02)

- Sets allowable stress at intermediate temperature (very small regime for Nb-1Zr)

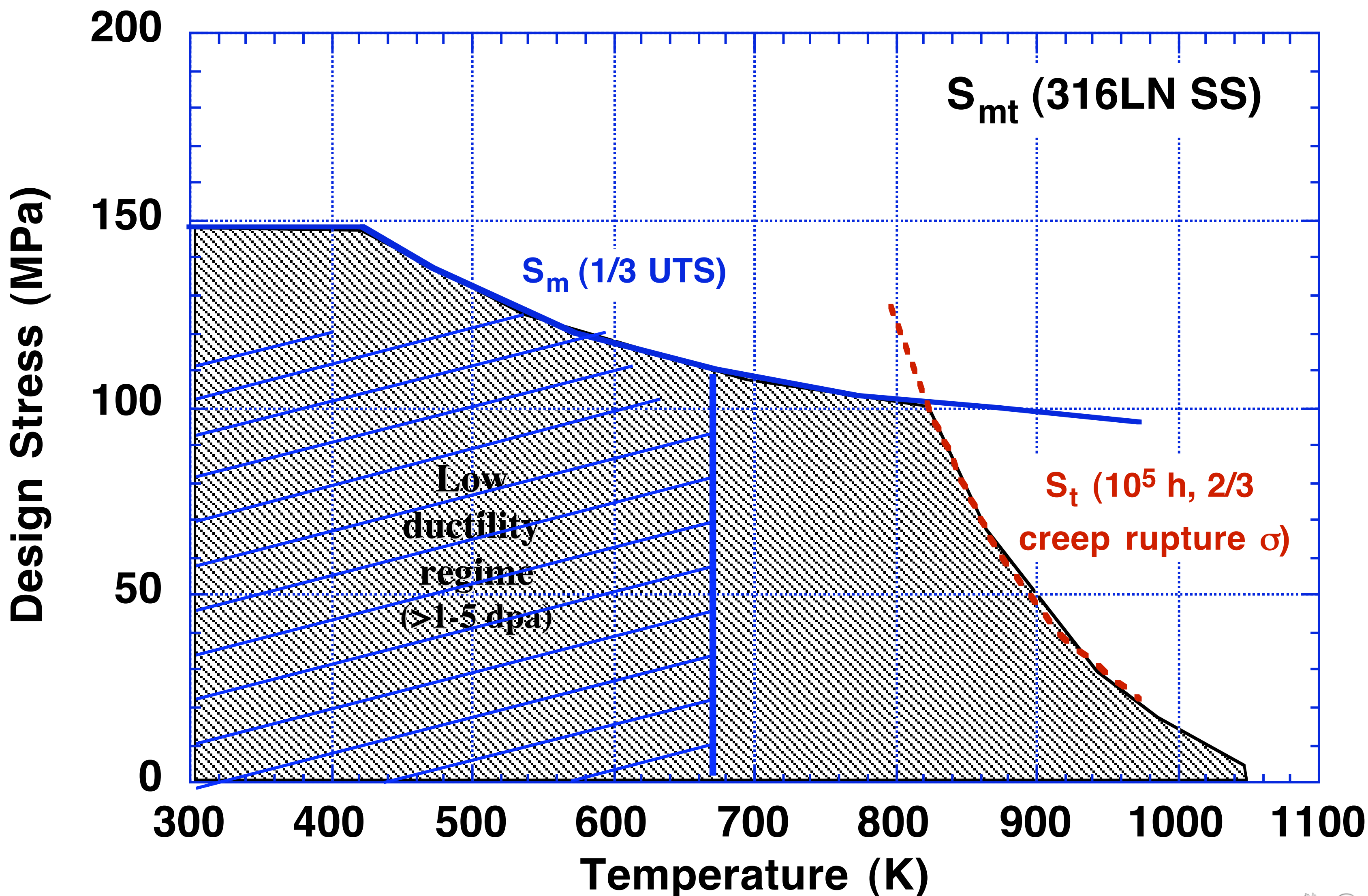
IV: High Temperature Thermal Creep Regime (T>~1050 K)

- Deformation limit depends on engineering application (common metrics are 1% deformation and complete rupture)

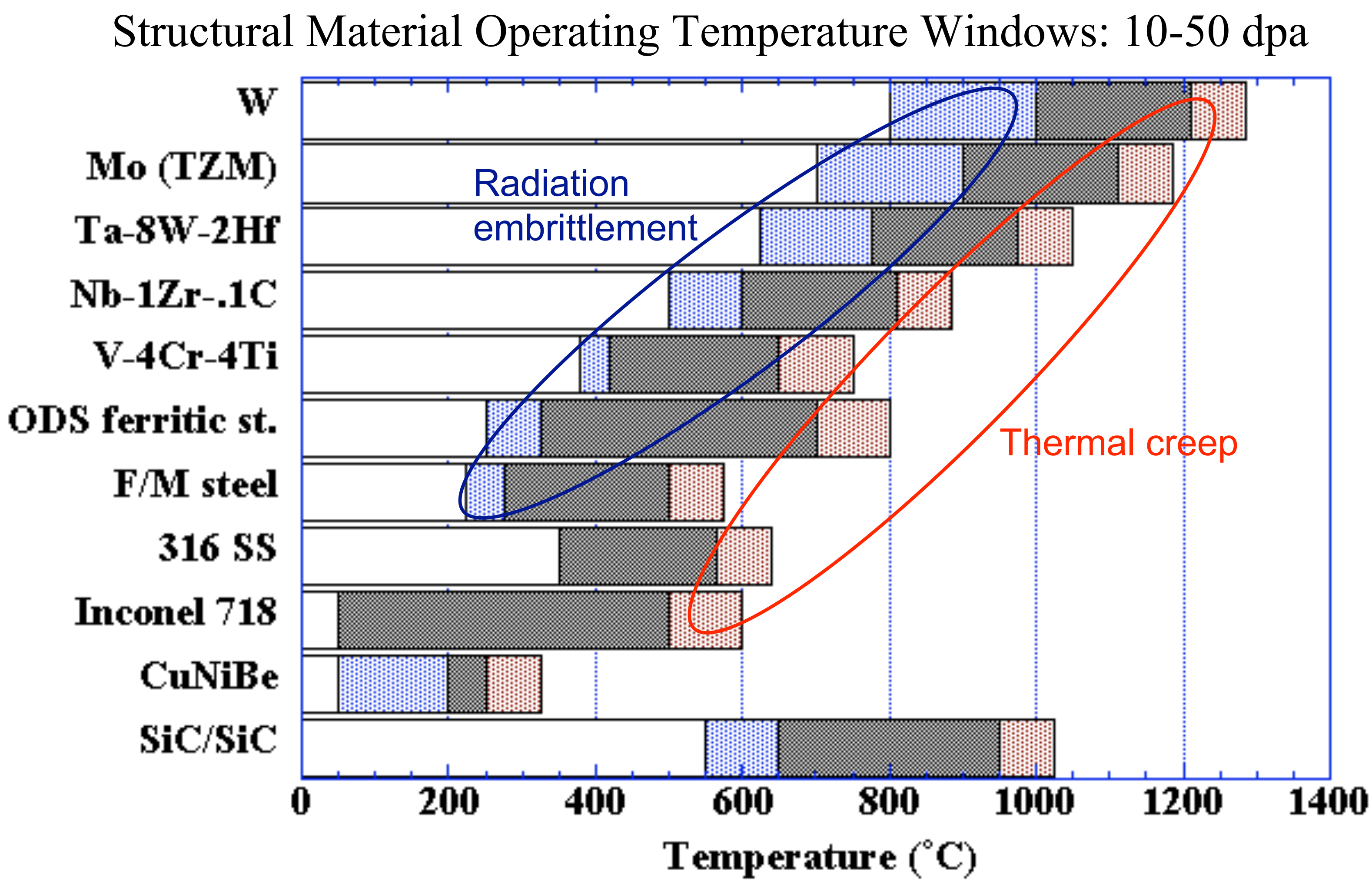
Stress-Temperature Design Window for Nb-1Zr



Stress-Temperature Design Window for Unirradiated Type 316 Stainless Steel



Conventional structural materials are capable of operation within ~300°C temperature window



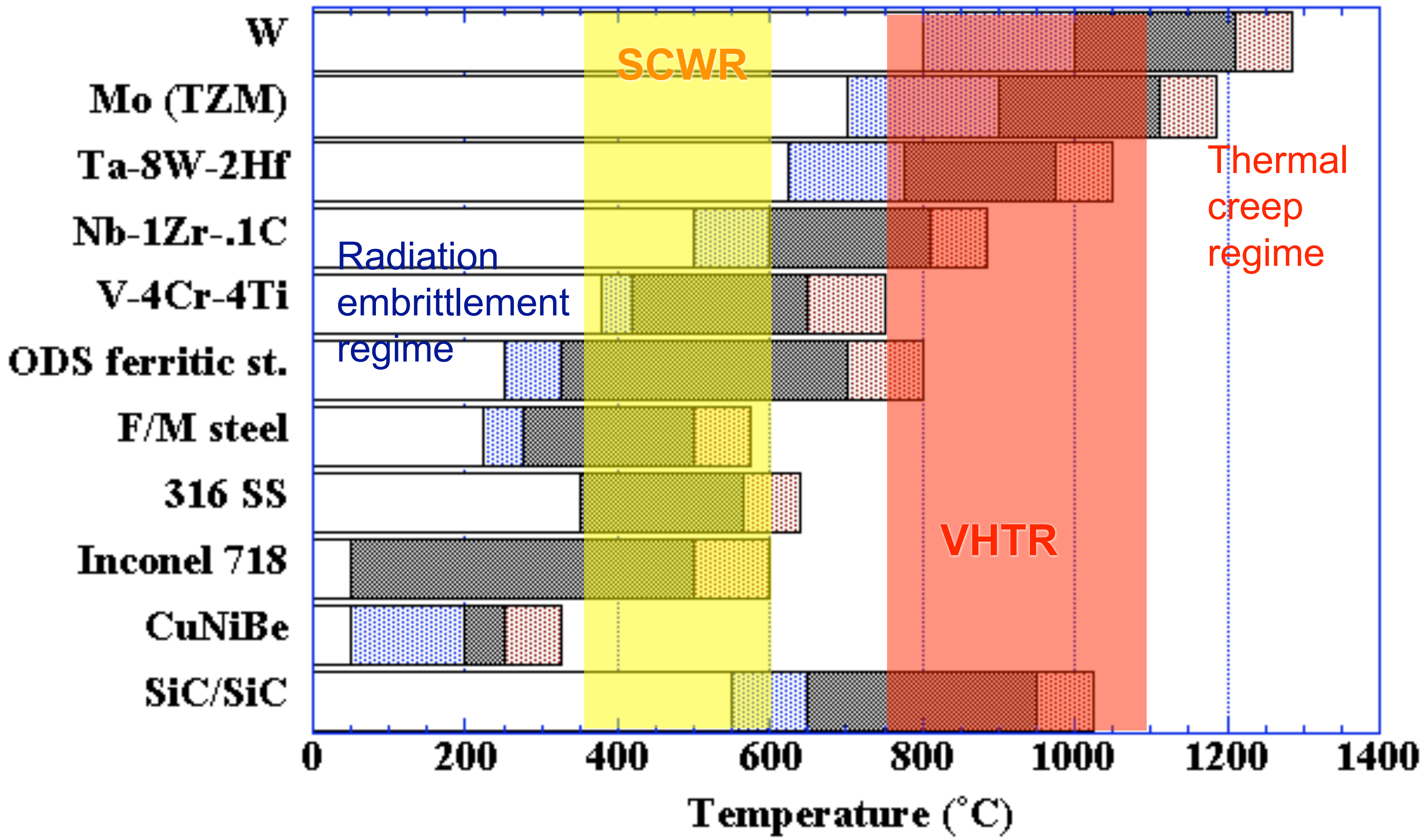
$\eta_{Carnot}=1-T_{reject}/T_{high}$

Low temperature radiation embrittlement typically occurs for damage levels ~0.1 dpa (0.01 MW-yr/m²)

Zinkle and Ghoniem, Fusion Engr. Des. 51-52 *(2000) 55*

The restricted operating temperature window for conventional structural materials means each new reactor design requires development of a new structural materal

Structural Material Operating Temperature Windows: 10-50 dpa



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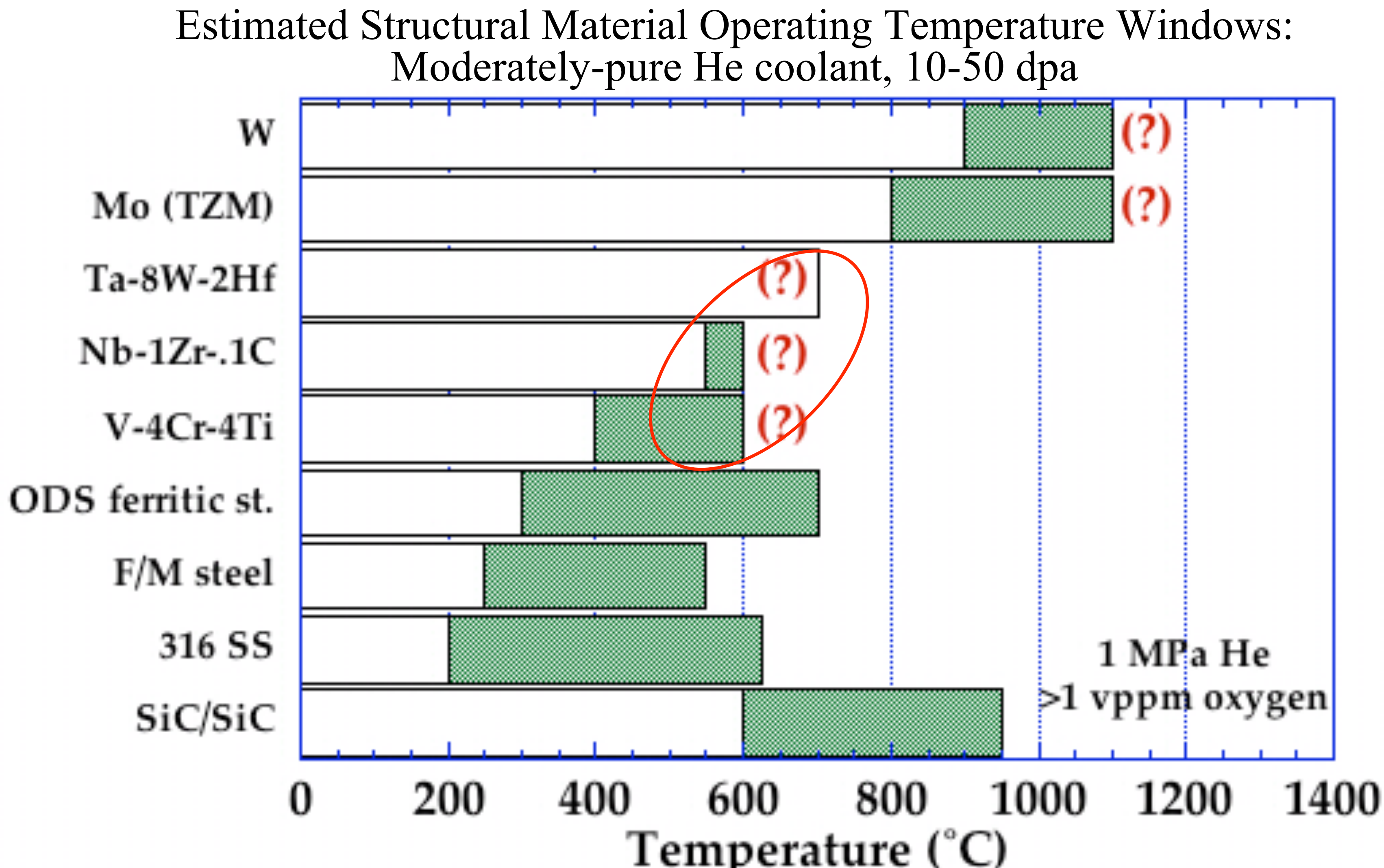
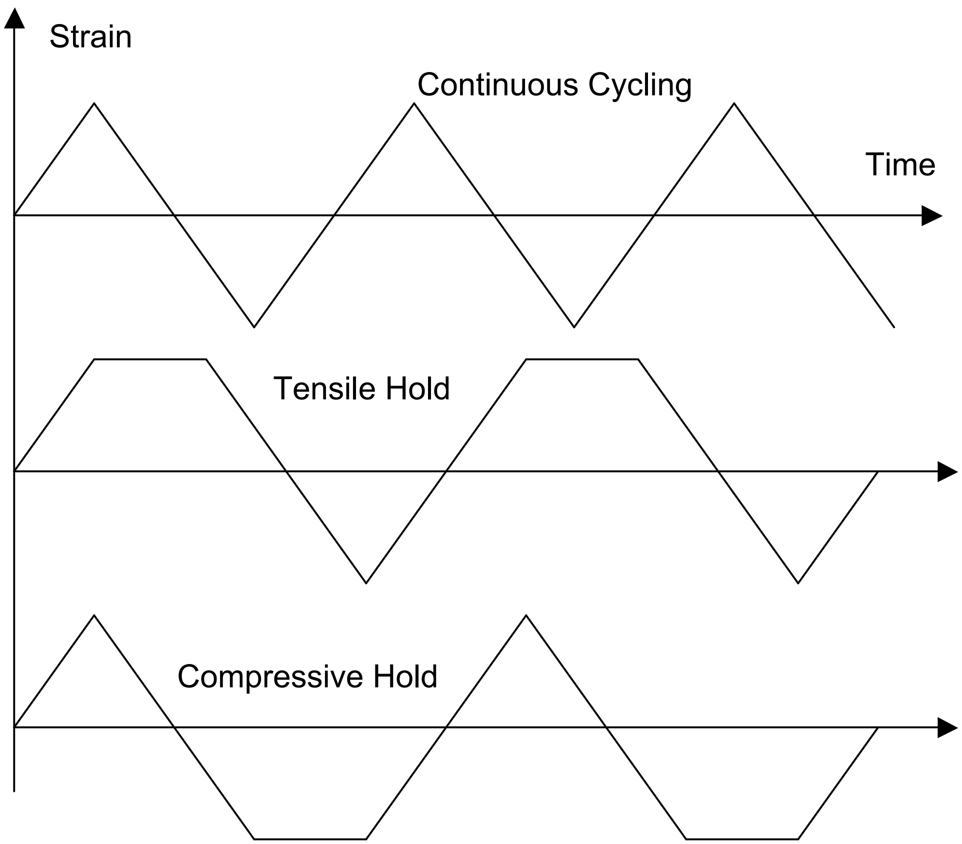
Consideration of Chemical Compatibility can Result in Dramatic Reductions in Temperature Window



Group V refractory alloys are particularly sensitive to embrittlement from O,C,N solute

Zinkle and Ghoniem, Fusion Engr. Des. 51-52 *(2000) 55*

Creep-Fatigue Phenomenology



Number of cycles to failure in cyclic tests at elevated temperatures is reduced progressively with increasing hold time

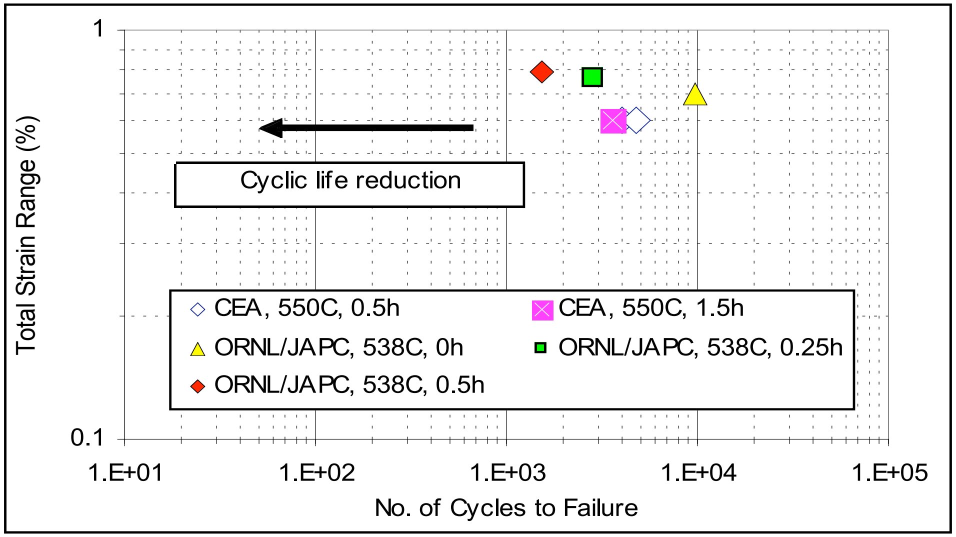
Failure mode often changes from trangranular to intergranular when hold periods are introduced

Depending on material:

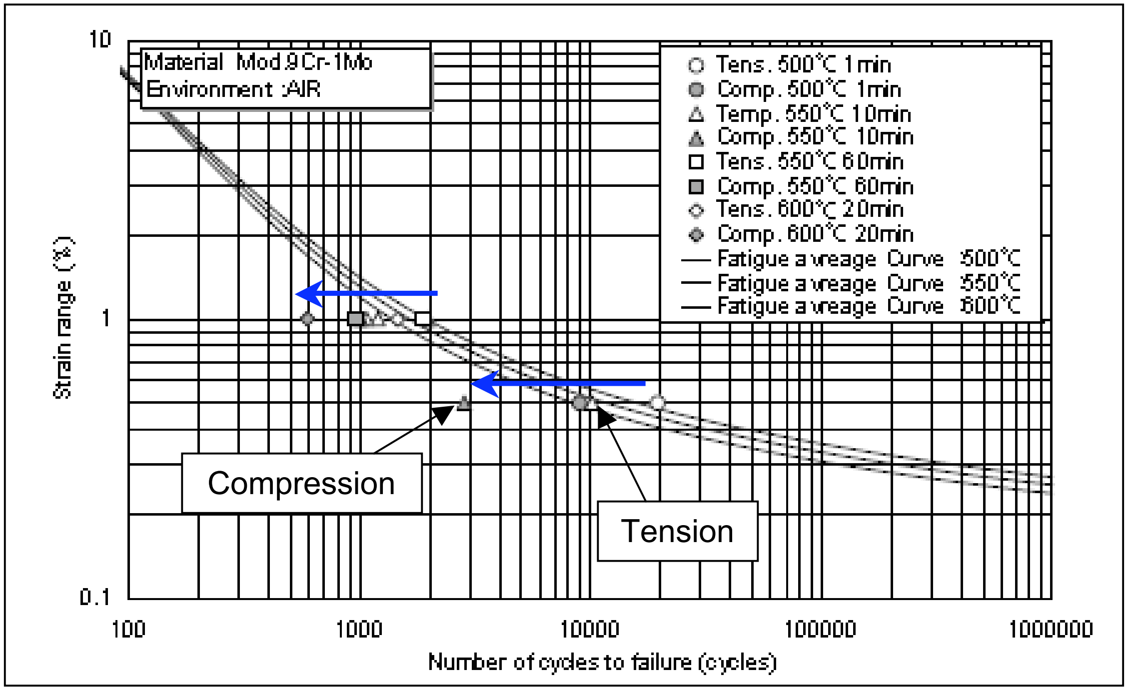
- Hold time effect may or may not saturate
- Tensile strain hold could be more damaging than compressive hold, e.g., 304, 316SS
- Compressive strain hold could be more damaging than tensile hold, e.g., mod. 9Cr 1Mo

Current approach is highly reliant on materials-specific empirical data

Creep-Fatigue Air Data – mod. 9Cr-1Mo steel



Hold time reduces cyclic life



Compressive strain hold more damaging than tensile strain hold

Asayama and Tachibana, 2008

ASME Section III Creep-Fatigue Code Design Rules

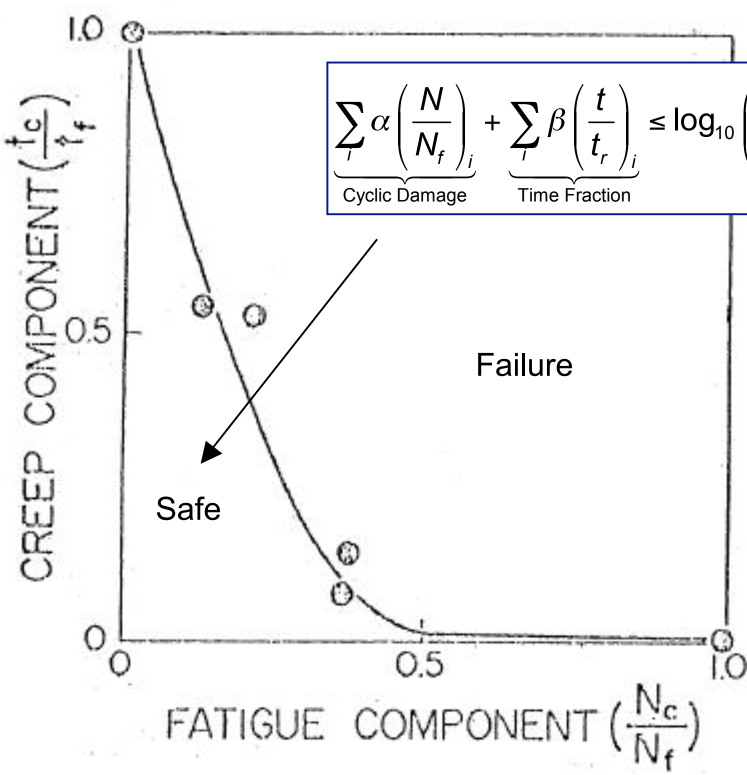
- Various Section III code cases => Code Case N47 => Subsection NH

1960s → 2008

- Creep-fatigue damage was recognized very early on as a very severe structural failure mode under elevated temperature service

- Work of D.S. Wood (1966) led ASME to adopt in 1970 the creep-fatigue failure criterion:

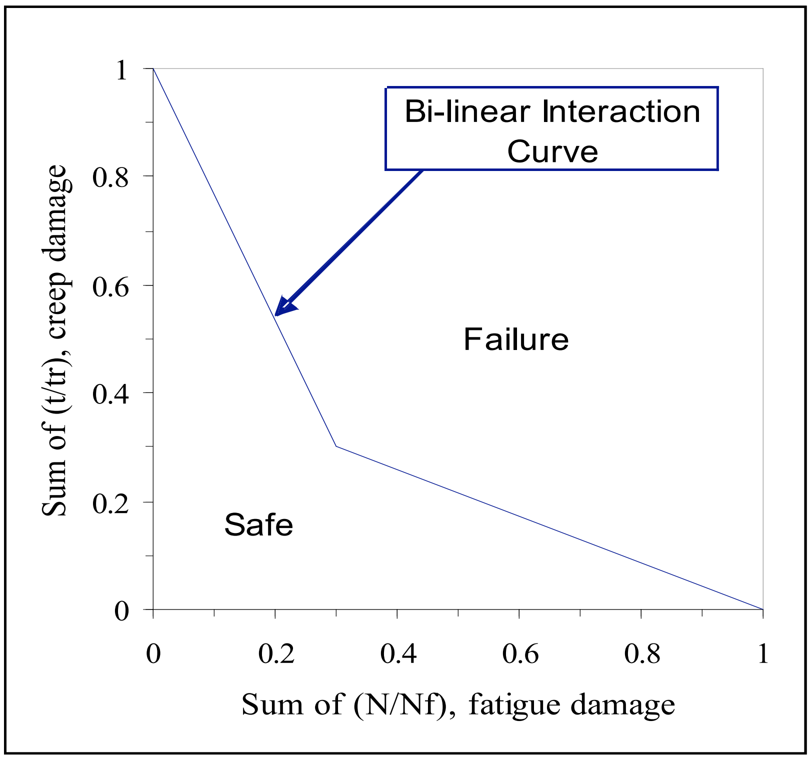
$$\underbrace{\sum_i \alpha \left(\frac{N}{N_f}\right)_i}_{\text{Cyclic Damage}} + \underbrace{\sum_i \beta \left(\frac{t}{t_r}\right)_i}_{\text{Time Fraction}} \le \log_{10}\left(\frac{t}{t_r}\right) + 4.25\left[\left(\frac{N}{N_f}\right)^2 + 0.257\left(\frac{N}{N_f}\right)\right]$$



- Cyclic damage => By cycle fraction
- Creep damage => By time fraction
- Creep-fatigue interaction => By creep-fatigue interaction diagram
- Extensive debates on whether to include creep damage due to hold time in the cyclic-fraction term or the time-fraction term

ASME Section III Creep-Fatigue Code Design Rules, Cont'd

- Work of Severud (1991) incorporated into Code Case N47, and subsequently Subsection NH:
  - Time-fraction accounts for all time-dependent creep damage (including hold time)
  - Cyclic damage does not involve creep damage
- Current Subsection NH creep-fatigue failure criterion:



$$\underbrace{\sum_i \left(\frac{N}{N_f}\right)_i}_{\text{Cyclic Damage}} + \underbrace{\sum_i \left(\frac{t}{t_r}\right)_i}_{\text{Time Fraction}} \leq D$$

D = Bi-linear curve in interaction diagram (determined empirically)

N_f = Cycle to failure

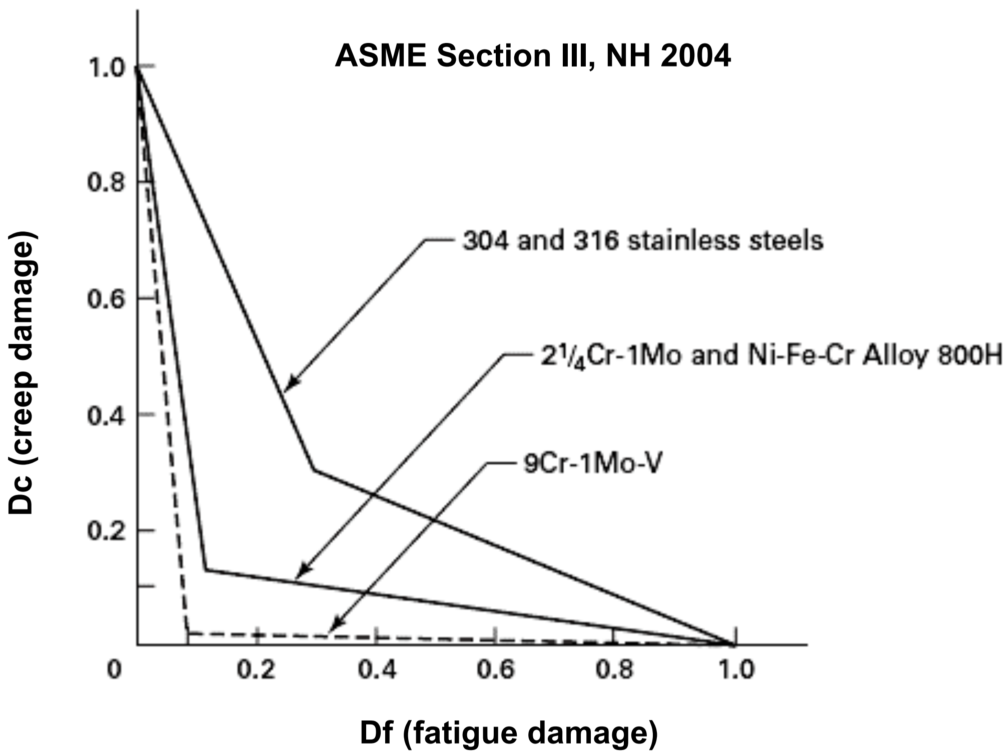
t_r = Stress-rupture time

Creep and cyclic damage evaluated independently

Creep-fatigue interaction accounted for through the D-diagram

Creep – Fatigue Interactions in fusion reactors

- Fusion reactor materials will experience both creep and fatigue
  - Elevated temperature design traditionally starts above 370/426°C (for steels)
    - DEMO structural temperatures may reach 700°C
  - Plasma disruptions produce electromagnetic (EM) and thermal load cycles
    - 3,000 major (50,000 minor) disruptions likely for ITER; number of disruptions unknown for Demo
- Interaction between creep and fatigue damage not well understood
  - Large variations in creep-fatigue damage envelope exist for various materials



- Is linear damage rule appropriate?
- Material testing required
  - interaction mechanisms
  - damage evolution
  - cyclic wave shape & hold time
  - cold work and heat treatments
  - irradiation spectrum & dose
  - environmental effects
  - base vs. welded material
  - estimate component lifetimes

Overall Adequacy of ASME Creep-Fatigue Code Rules

- Use conservative fatigue and stress-rupture curves
- Use conservative safety factor (1/K')
- Accounts for 3D and notch effect

- Use of isochronous stress-strain curves to estimate stress relaxation
- Use of monotonic stress-strain curves to estimate stresses from strains in materials that exhibit either cyclic hardening or cyclic softening behavior

Essential that Code procedure leads to adequately conservative designs when used as an integral package

Riou (2008) and Asayama and Tachibana (2008) showed:

- Current Subsection NH creep-fatigue Code procedure does give rise to conservative, and often overly conservative, designs for mod. 9Cr 1Mo
- Predicted cyclic lives are sometimes non-conservative relative to creep-fatigue data when the safety factor (1/K') from the Code procedure is not used (i.e., set to one)

Alternative (Ductility Exhaustion) Approach to Characterize Creep Damage

- A strain-based approach (versus stress-based approach in time fraction method)
- Fractional creep damage in a cycle with hold period is determined by

Creep rate

$$\left(D_c\right)_i = \int_0^{t_i} \frac{\dot{\varepsilon}_c}{\varepsilon_f\left(\dot{\varepsilon}_c\right)} \mathrm{d}t$$

Creep ductility

- Creep rate can change over many decades during hold period when stress relaxes
- R5 (British structural integrity assessment guideline) has an option to use ductility exhaustion to determine creep damage in creep-fatigue assessment
    - No safety factor used

Time Fraction Approach Versus Ductility Exhaustion Approach

- Conclusions from ASME Subgroup on Elevated Temperature Design
  - Advantages of time-fraction approach
    - Conceptually straight forward
    - Drew on existing data (fatigue and creep-rupture)
    - Fairly easy to implement
  - Disadvantages of ductility exhaustion approach
    - Did not demonstrate a clear superiority in correlating experimental data
    - Required additional new testing
    - More difficult to implement in design rules
- The time-fraction approach has been retained in the Subsection NH creep-fatigue Code rules to this day
  - Also used in French RRC-MR and Japanese DDS codes
- However, effort to improve the creep-fatigue procedure in Subsection NH has continued

Tasks 3 & 5: Creep-fatigue rules for mod. 9Cr 1Mo (Led by AREVA and JAEA investigators)

Major conclusion:

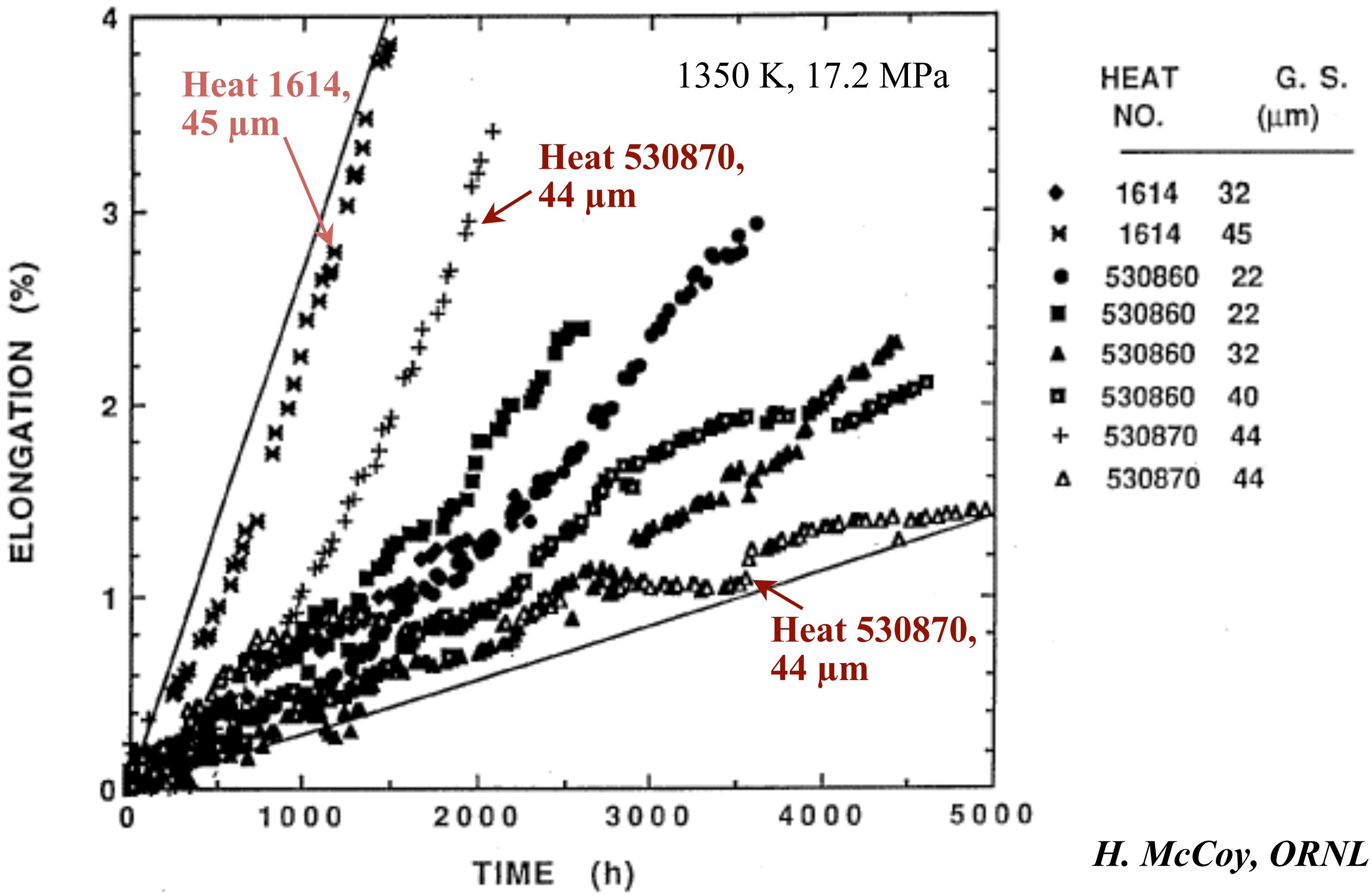
- Subsection NH creep-fatigue procedure for mod. 9Cr 1Mo is overly conservative

Recommendations:

- Modify the procedure for calculating the stress at the beginning of the hold time by accounting for cyclic softening and symmetrization effects (cyclic stress-strain curve)
- Provide additional creep-strain laws in Subsection NH (in conjunction with strain hardening law)
- Provide guidance in Subsection NH to address elastic follow-up effects
- Change the factor from 0.67 to 0.9 for the elastic analysis route in the Subsection NH procedure, as employed in RCC-MR
- Change the intersection point in the creep-fatigue damage envelope from (0.1, 0.01) to (0.3, 0.3), as employed in RCC-MR for mod. 9Cr-1Mo

These recommended changes are driven largely by additional empirical data, rather than improvements in modeling creep-fatigue interaction mechanisms

Large variability in thermal creep behavior for three heats of nominally identical Nb-1Zr

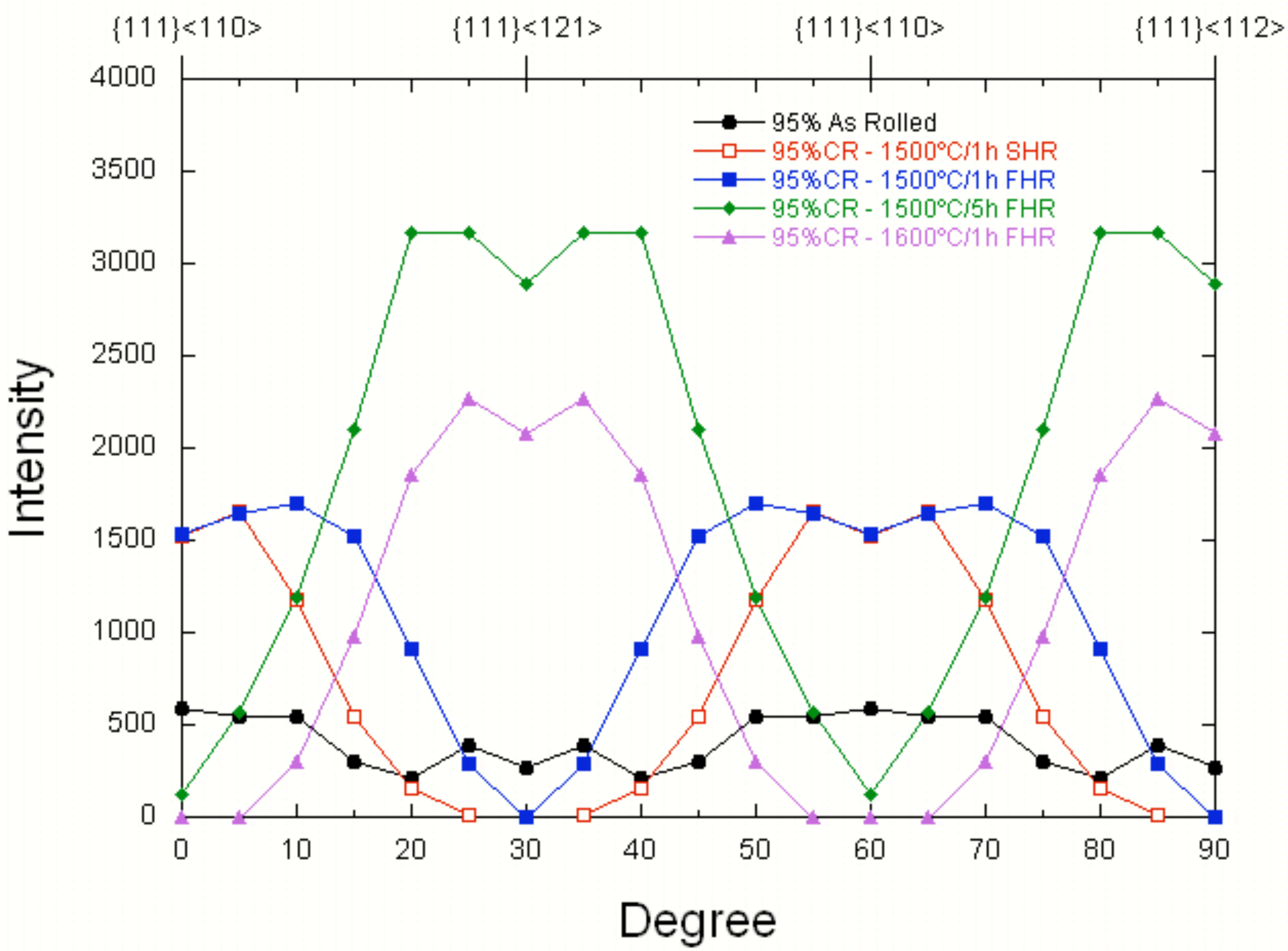


H. McCoy, ORNL

- *In addition to grain size, these results show that **other microstructural inhomogeneities** can also affect the thermal creep behavior of Nb-1Zr*

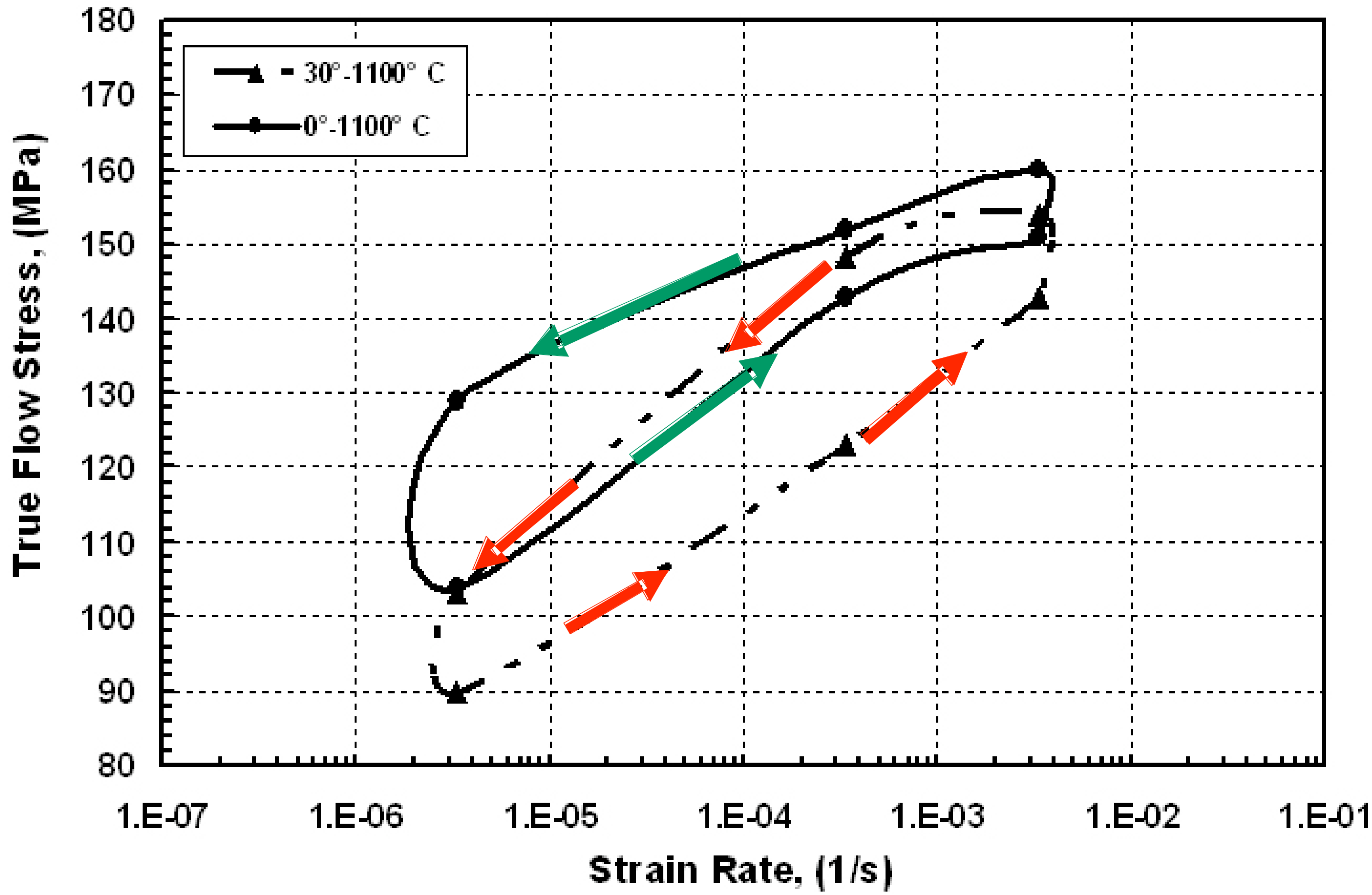
Development of Texture in Annealed Nb-1Zr

- Texture pattern in recrystallized Nb-1Zr is strongly dependent on annealing conditions



FHR: fast heating rate (>1000˚C/min)
SFR: slow heating rate (10˚C/min)

Comparison of effect of strain rate change on the tensile behavior of Nb-1Zr with different textures



Alignment along <110> (0°) provides higher strength than <112> alignment (30°)

Nieh et al., Scripta Mater. 55 (2006) 719

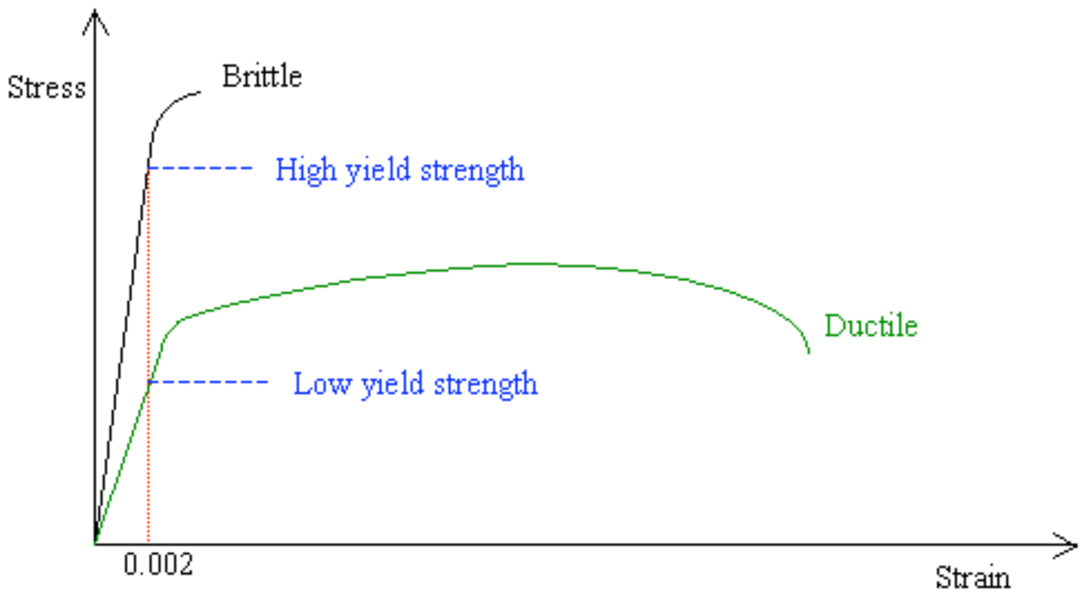
High Temperature Design Criteria: Materials Research for Fusion and Gen IV Fission Energy Applications

- Develop an understanding of the operative damage mechanisms when both creep and fatigue deformation are occurring
- Understand the reasons for the very significant variation in C-F damage from one alloy to another
- Develop alloys/microstructures that have improved C-F performance
- Incorporate all irradiation damage in a high-temperature design methodology and code
  - Swelling, irradiation creep, high temperature He embrittlement, etc.
  - Need increased utilization of flexible in-situ mechanical testing
  - Flow localization and ductility exhaustion concerns may extend to high temperatures due to He embrittlement phenomena
- Understand deformation and failure mechanisms and irradiation damage in SiC/SiC composites and develop a design methodology appropriate for fission and fusion applications

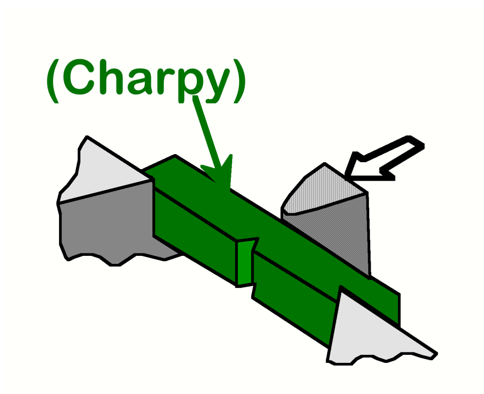
Conclusions

- Qualifying structural materials for nuclear energy applications is a time-consuming process, in part due to the heavy reliance in engineering design codes on empirical data collection
- Development of improved fundamental understanding of physical parameters controlling mechanical property behavior (e.g., texture) may reduce the data scatter and lead to shortened qualification times
- Need improved fundamental understanding of creep-fatigue interaction mechanisms in a broad range of structural materials
  - Current ASME Subsection NH approach in establishing creep-fatigue design rules is empirical, replying on data correlation
  - E.g., role of material crystallography and microstructure on bilinear creep-fatigue interaction curve
    - This understanding will be critical for timely evaluation of proposed new materials for fission and fusion applications

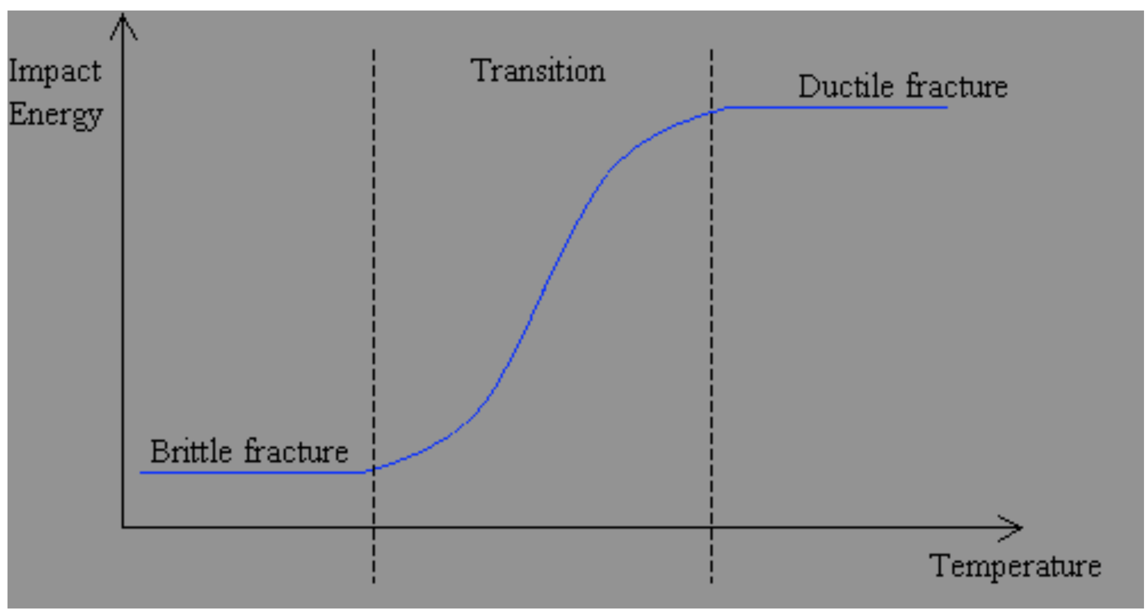
Structural materials involve compromise between strength and ductility



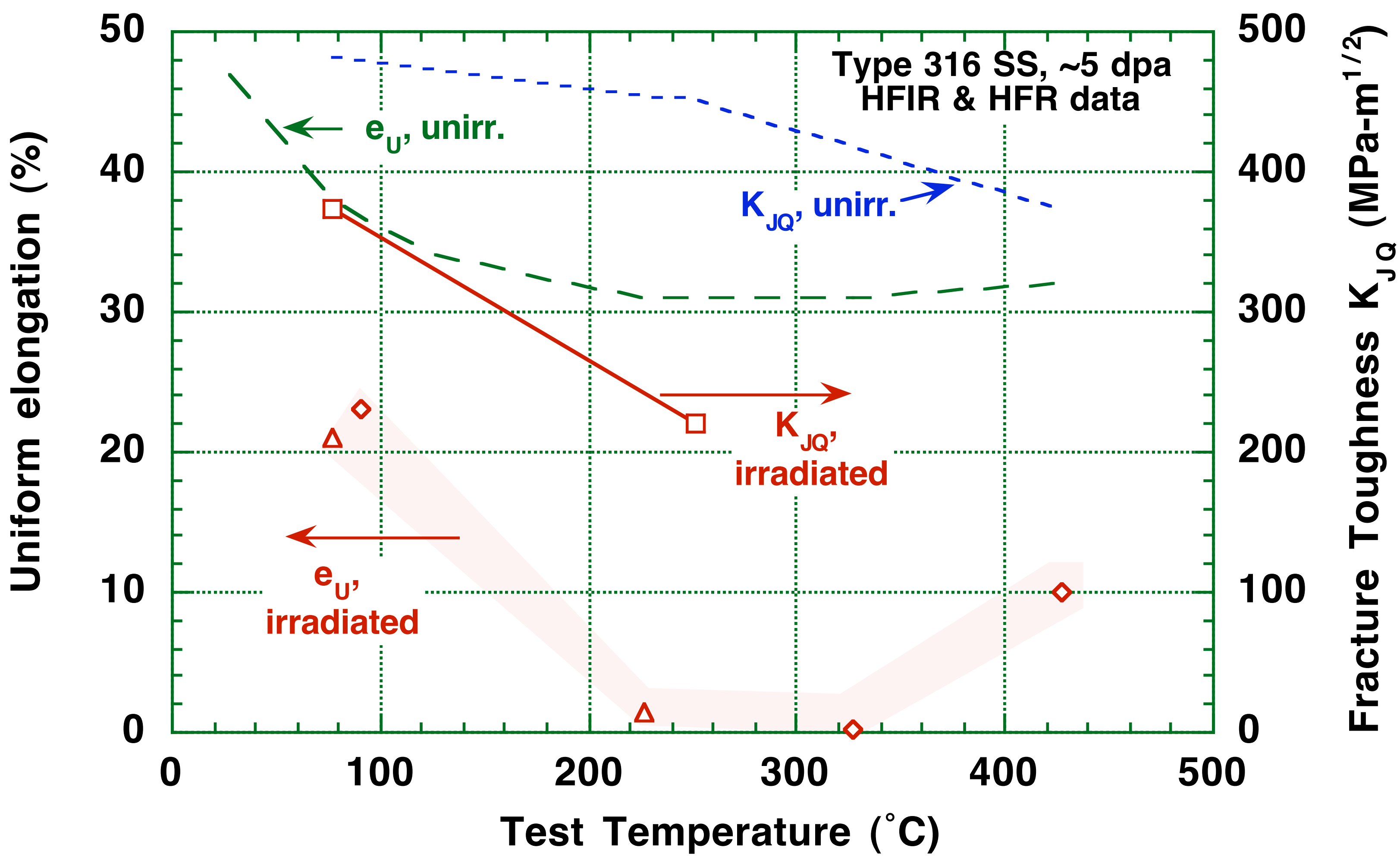
Schenectady Liberty ship, 1943



A simple measure of the resistance to brittle cleavage failure is the Charpy notched impact test



Irradiation of Austenitic Stainless Steel in Mixed Spectrum Reactors causes Pronounced Loss in Elongation and Significant Reduction in Fracture Toughness

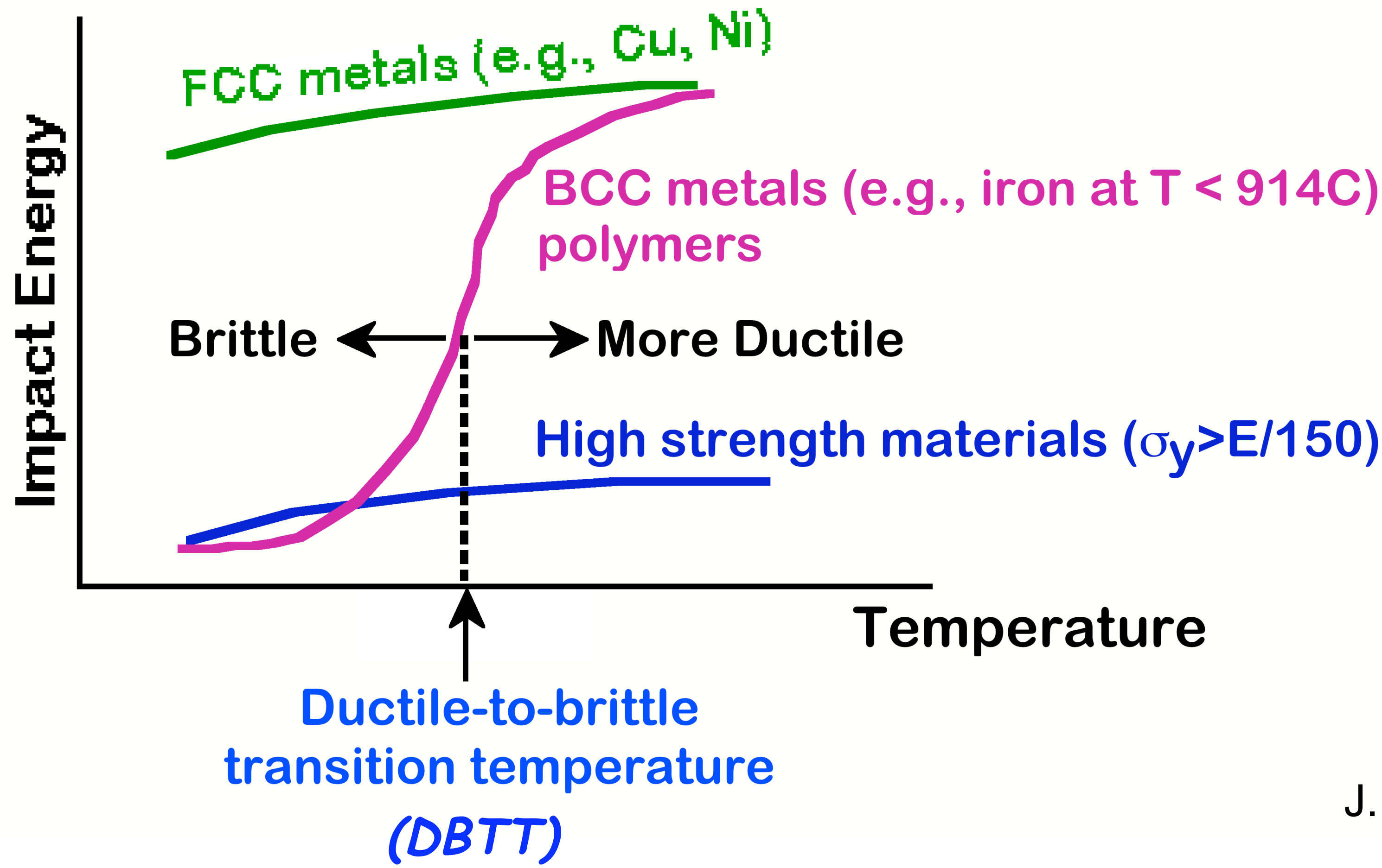


 Managed by UT-Battelle
for the U.S. Department of Energy

Oak Ridge National Laboratory

Brittle behavior at low temperature is of greatest concern for BCC metals (due to Peierls barriers)

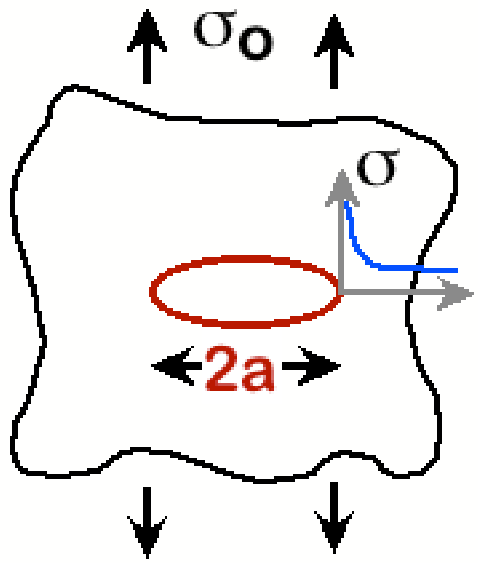
Design strategy: Stay above the DBTT whenever stress is applied



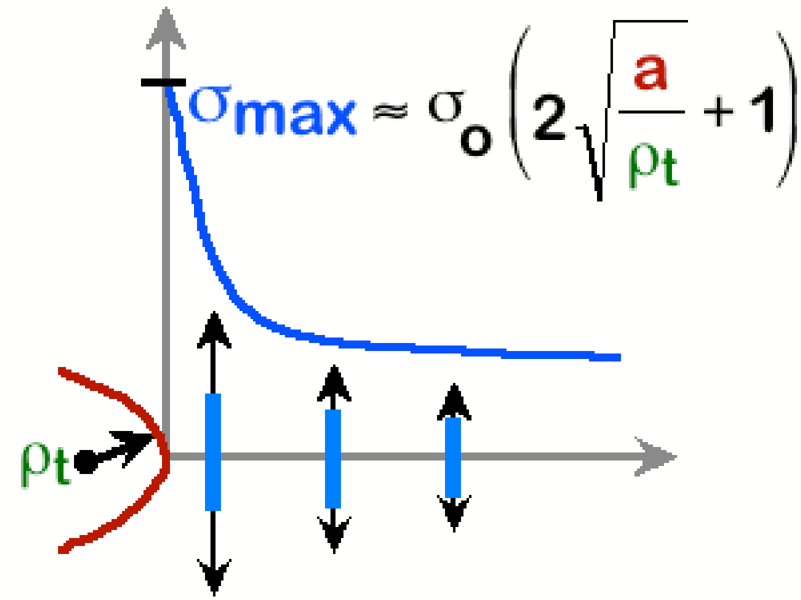
J. Hayton

FLAWS ARE STRESS CONCENTRATORS!

- Ellip
  a pl



- Str



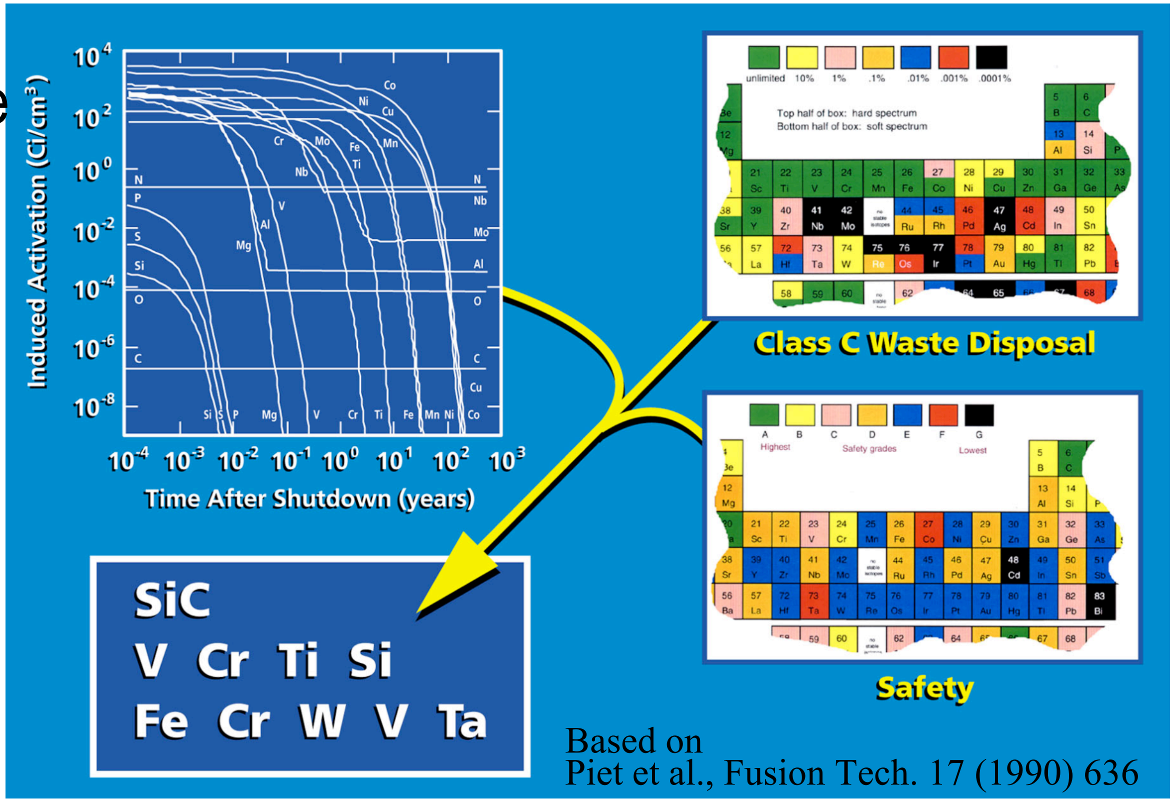
- Stress conc. factor: $K_t = \sigma_{max} / \sigma_o$

- Large K_t promotes failure:

J. Hayton

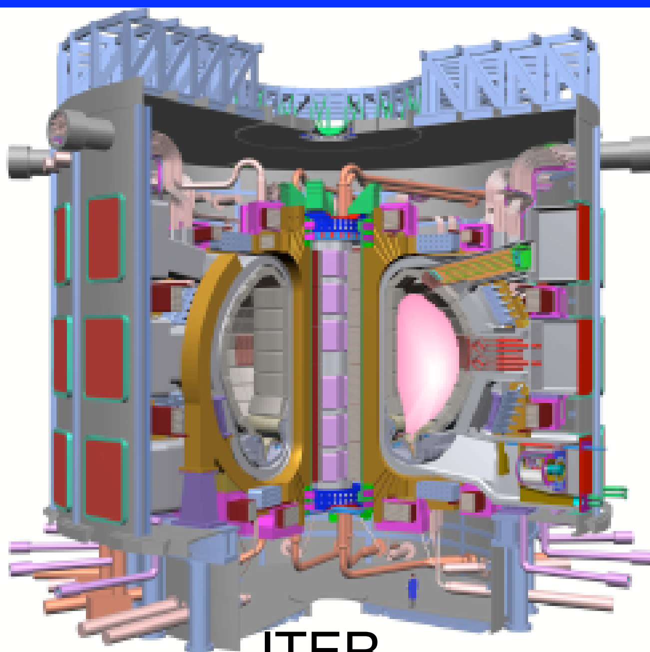
The Overarching Goals for Fusion Power Systems Narrow the Choices and Place Significant Demands for Performance of Structural Materials

- Safety
- Minimization of Rad. Waste
- Economically Competitive
  - High thermal efficiency (high temperatures)
  - Acceptable lifetime
  - Reliability

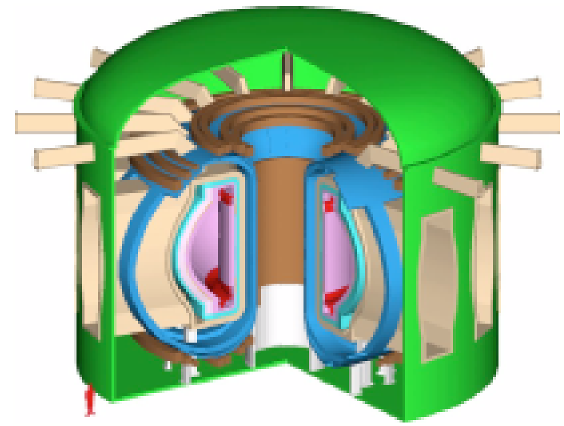


Fe-9Cr steels: builds upon 9Cr-1Mo industrial experience and materials database (9-12 Cr ODS steels are a higher temperature future option)
V-4Cr-4Ti: Higher temperature capability, targeted for Li self-cooled blanket designs
SiC/SiC: High risk, high performance option (early in its development path)

ITER will provide plasma physics basis for Demo, but many key technology issues must be separately addressed



ITER



ARIES-RS
(conceptual Demo design)

	ITER	ARIES-RS
Fusion Gain	10(H), 5(AT)	25 (AT)
Fusion Power (MW)	500 - 350	2170
Power Density(MWm^{-3})	0.6	6.2
Wall Loading Γ_n(MWm^{-2})	0.6	4
Pulse Duration (s)	500 - 3000	20,000,000
Mass of Fusion Core (tonnes)	23,000	13,000
1st wall/ blanket structural material and operating temperature	316 SS, <300°C	F/M steel, ODS steel, V alloy or SiC/SiC, 350-1000°C

Materials Research and the Path to Fusion Power

Plasma Physics Confinement

Fusion Reactor Concept Definition

ITER

Demo Conceptual Designs

Demo Final Design and Construction

- **Materials research to identify candidate FW/B structural materials**
  - **Thermal conductivity and expansion**
  - **Activation**
  - **High temperature mechanical properties**
  - **Compatibility with coolants and T breeders**
  - **Irradiation damage issues**
  - **Joining and fabrication issues**

- **Identify and demonstrate approaches to improve material performance**
- **Identify concept specific issues and demonstrate proof-of-principle solutions, e.g.**
  - **design with ferromagnetic material**
  - **MHD-insulator for V-Li concept**
  - **methods for design of large thermal**
  - **mechanically loaded composite structures**

- Development of materials with acceptable performance and demonstrate to goal life (dpa and He)
- Demonstrate solution to concept specific issues on actual structural materials and prototype components
- Develop design data base, constitutive equations, and models to describe all aspects of material behavior required for design and licensing

Ion Accelerators

RTNS 14MeV point neutron source

Fission Reactors

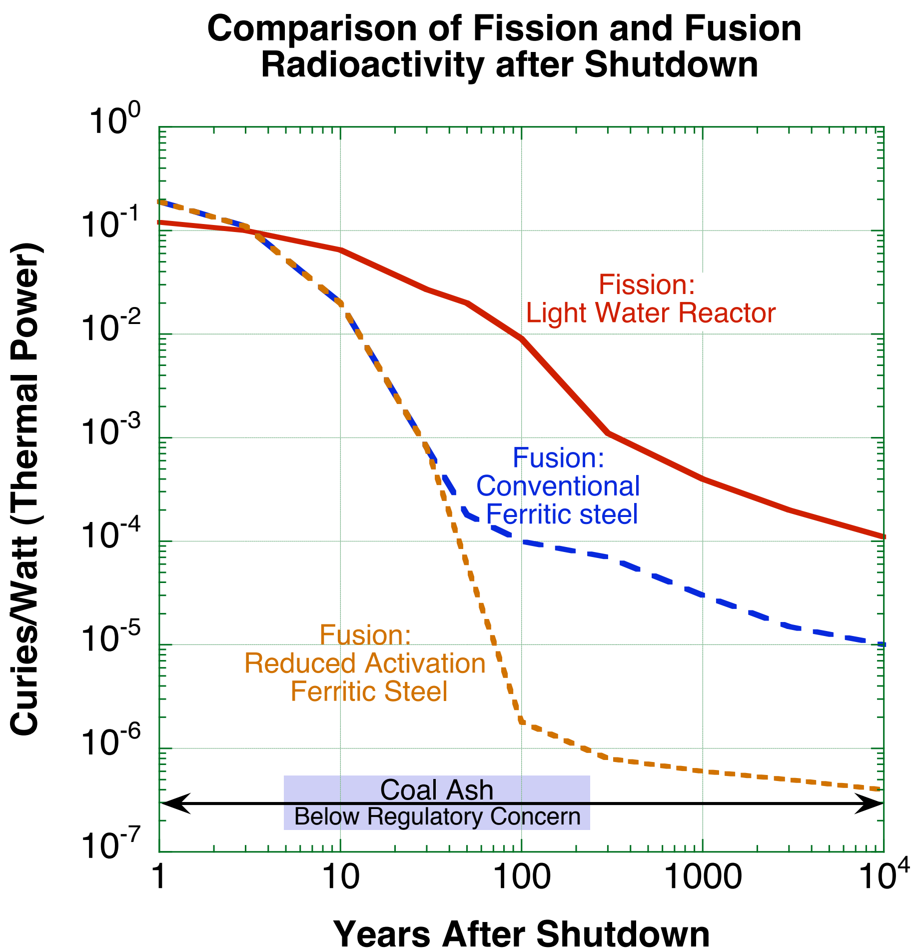
IFMIF

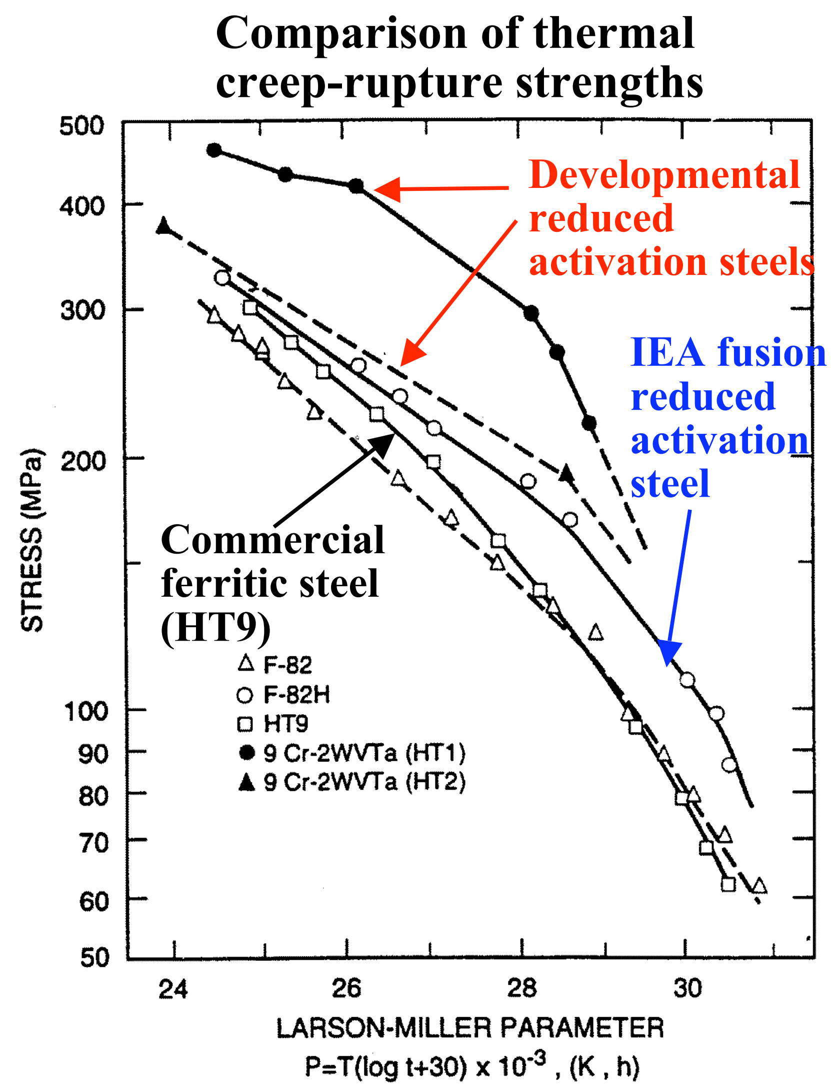
Component Test Facility

Confirmation & Modification of Performance With Actual Fusion Environment Tests

Fusion Power

Ferritic/martensitic Steels with Reduced Radioactivity and Superior Properties Compared to Commercial Steels have been Developed by Fusion

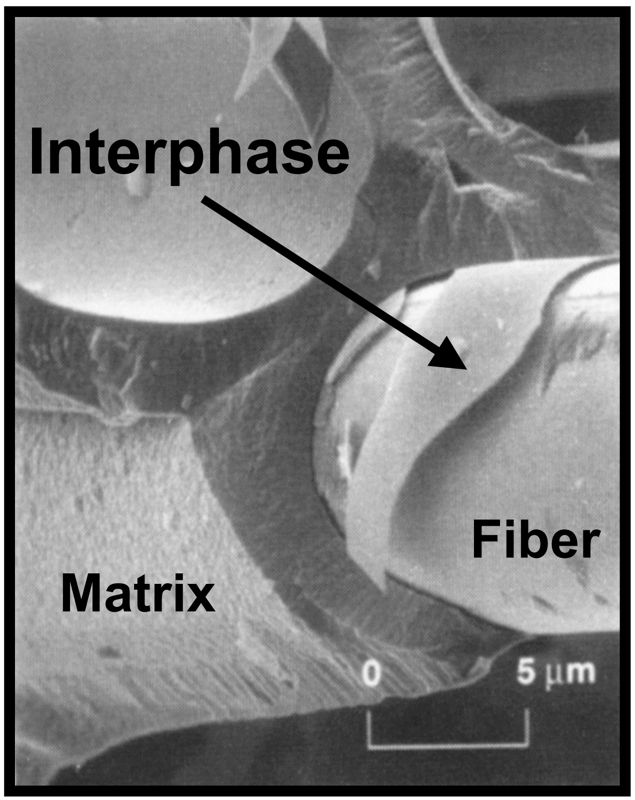


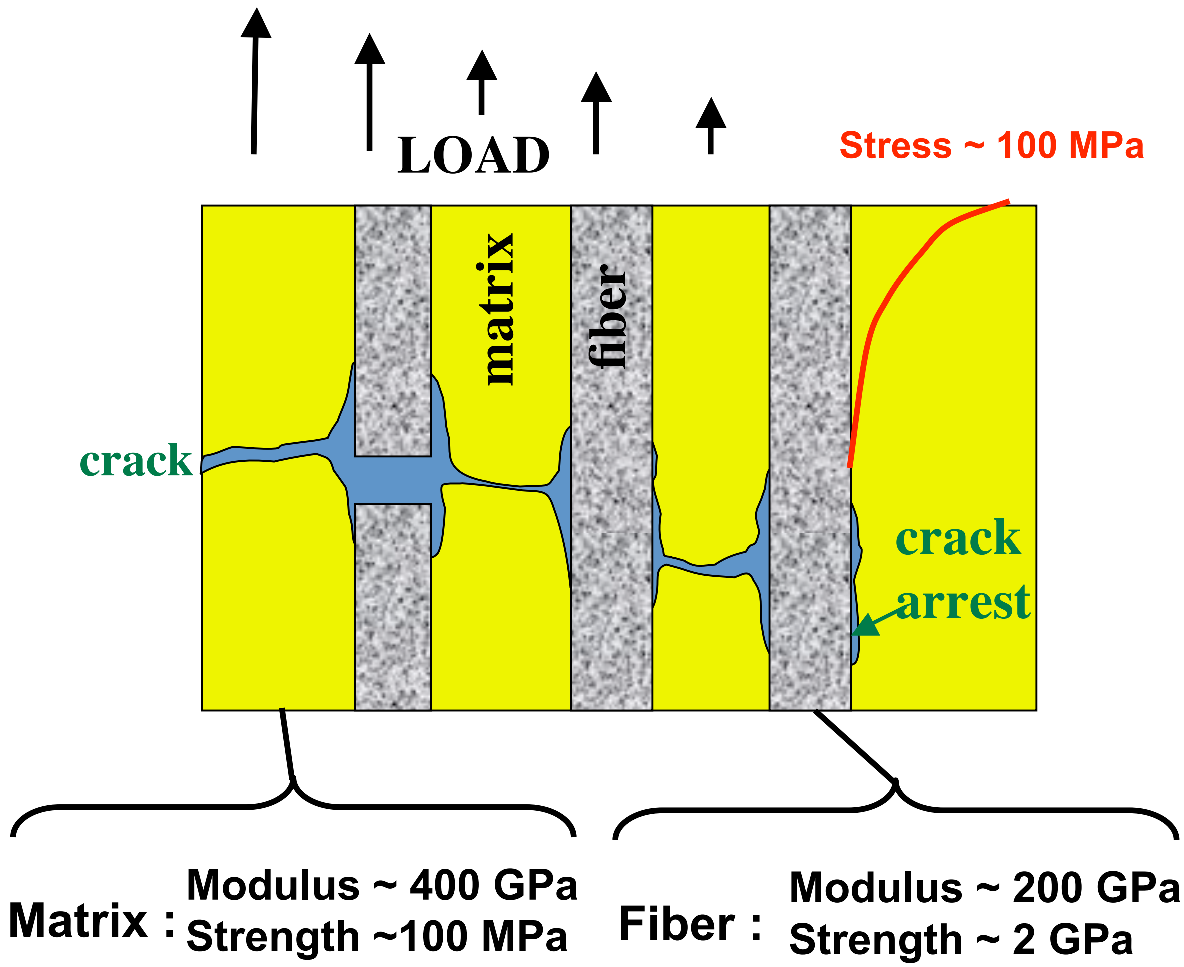


Fusion-developed steels also have superior tensile strength, irradiated fracture toughness, and thermal conductivity

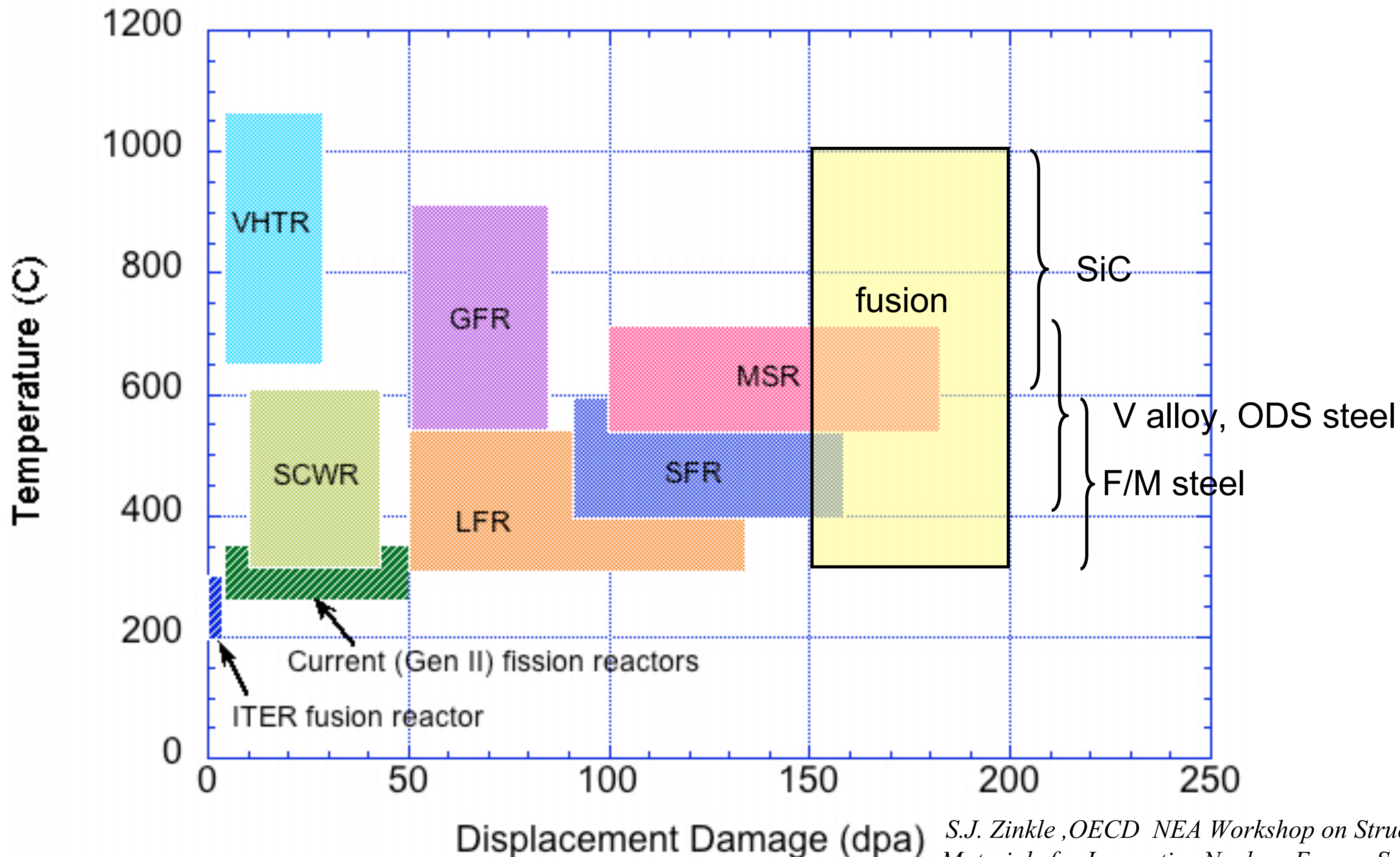
SiC Fiber Composites for Structural Applications

SiC/SiC offers attractive potential (radiation resistance, high temperature capability), but numerous practical issues need to be resolved (structural design rules, etc.)





Comparison of Gen IV and Fusion Structural Materials Environments



S.J. Zinkle ,OECD NEA Workshop on Structural Materials for Innovative Nuclear Energy Systems, Karlsruhe, Germany, June 2007

All Gen IV and Fusion concepts pose severe materials challenges

Fusion is particularly challenging due to high transmutant He levels that promote swelling and He embrittlement