Karlsruher Institut für Technologie

Engineering perspective on material property alterations by fusion neutron irradiation

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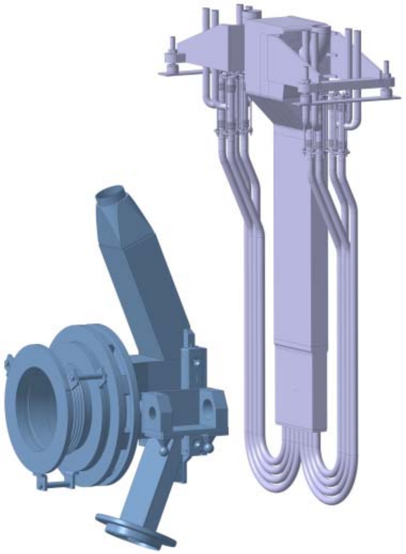

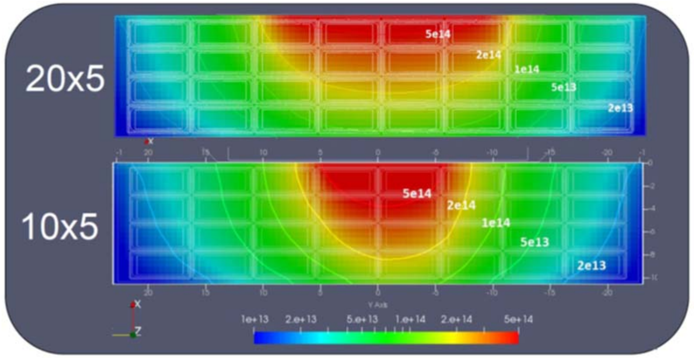

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Contents

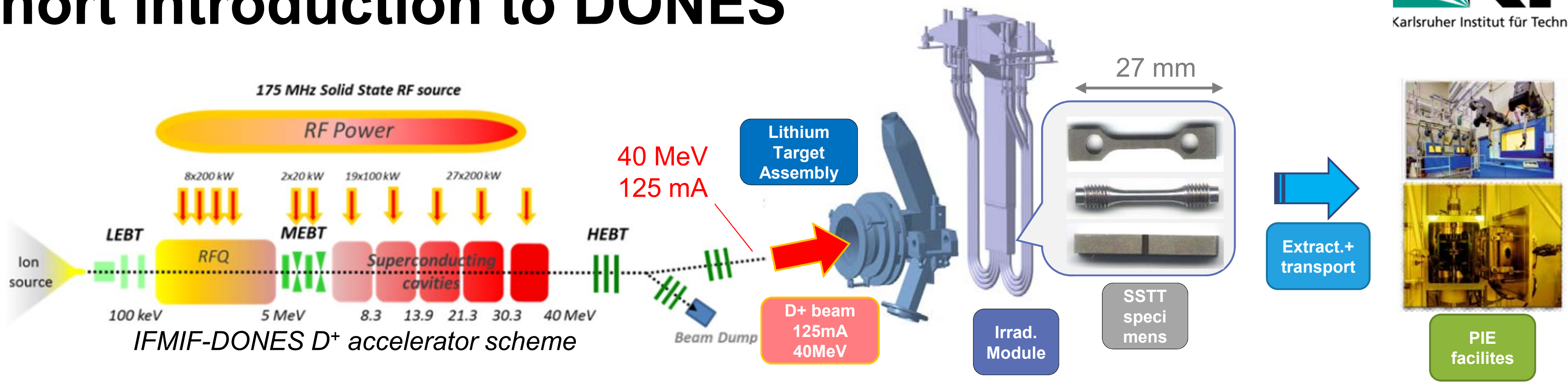
Lecture objective:

To *review* various effects of neutron radiation on materials
at the example of a specific engineering design, the DONES High Flux Test Module

Contents:

- (Very short) Introduction to the IFMIF-DONES facility
- Introduction to the DONES High Flux Test Module
- Review of (selected) material property alterations with *fission* neutrons
  - Swelling
  - Hardening, embrittlement
  - Degradation of insulators
  - Transport properties
- Anticipated differences of 14MeV neutrons vs. existing irradiation sources

Short introduction to DONES



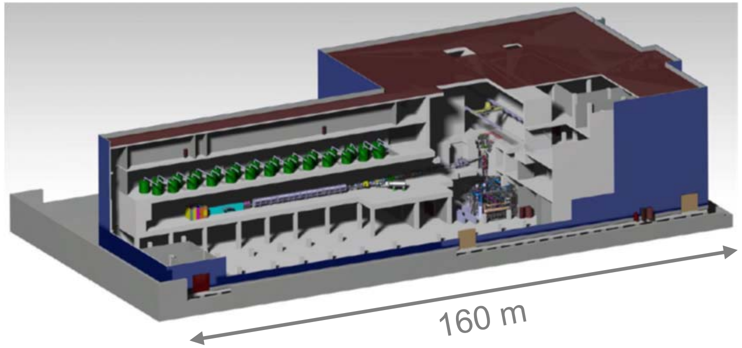
IFMIF-DONES D^+ accelerator scheme

Facility Objectives:

- Provide intensive neutron flux ($5 \cdot 10^{14}$ /cm²/s) with a **fusion-relevant spectrum**
- **Produce irradiated samples** for further (PIE) testing or in-situ experiment

Technical realization:

- Neutrons from D-Li reactions, 5MW deuteron beam on liquid lithium target
- Irradiation experiments placed in the high flux region behind neutron source



IFMIF-DONES facility

The DONES facility is presented in detail by the following talk by D. Bernardi

The DONES Neutron Source

- Broad peak around 14 MeV

  from several Li(d,xn) reactions

- Standard beam footprint 20 cm x 5 cm

- > 5×10^{14} neutrons/cm²/s

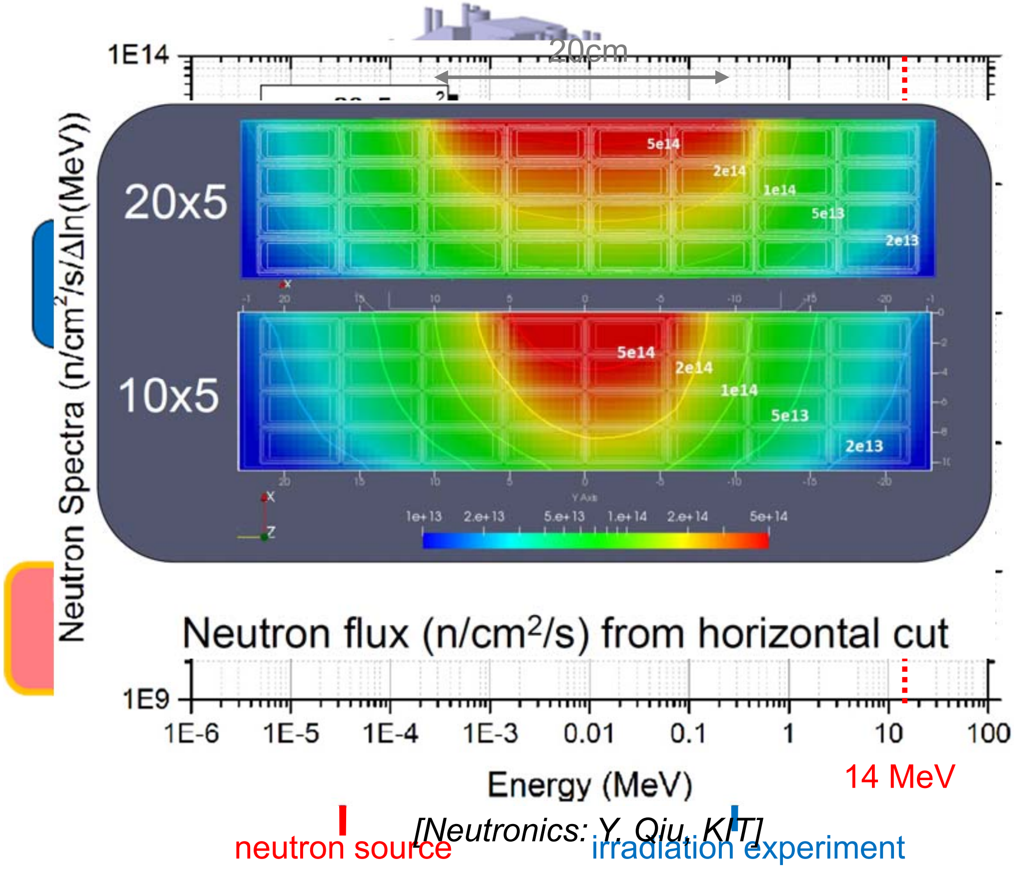
  But: significant spatial gradients!

- > 20 $\mathrm{dpa}_{\mathrm{NRT}}$ per full power year

- Approx. 13-15 appm(He)/dpa,

  50-60 appm(H)/dpa

Well matched to DEMO FW

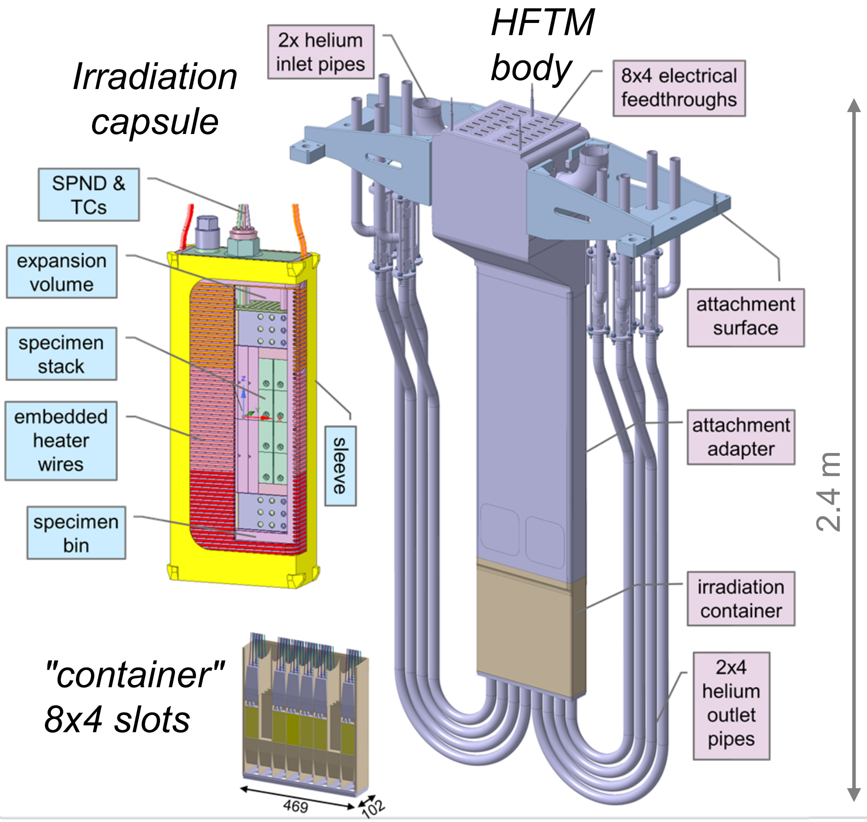


[Neutronics: Y. Qiu, KIT]

The DONES High Flux Test Module

Objective: to irradiate a large volume/number of SSTT samples in the high flux region of the DONES neutron source

- Irradiation temperatures **250 – 550 °C**, homogeneity +/-3%
- ~ **850 specimens** can be irradiated to **12 – 25 dpa/fpy**
- Masses: Total 680 kg, 40 kg irradiation capsules with specimens
- Heating: Nuclear **2.3 W/g peak**, 17 kW tot., 1.5 kW electr. per capsule
- Cooled by **low pressure helium** gas (0.3MPa), 50°C)
- Lifetime: 1year / 2.5 years
- Instrumentation: thermocouples (6 per capsule), activation foils (1...n sets per capsule), SPND, MFC



HFTM prototypes & tests

Testing of HFTM prototypes (HELOKA-LP helium loop, BR2 reactor)

Requested properties for the HFTM

- **High number/density of irradiated specimens** placed in the irradiation footprint

- **Low uncertainty in specimen irradiation conditions**
  - Measured temperatures, limited temperature spread
  - Measured doses/spectra ➔ radiation instrumentation

- **Reliable operation** during irradiation
  - avoiding time-consuming replacement procedure

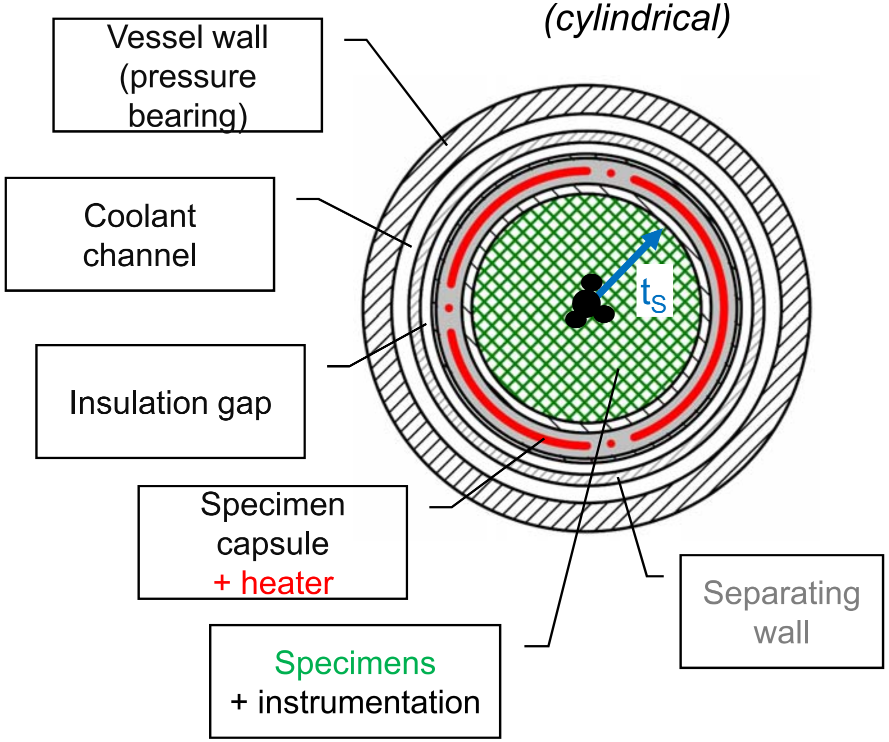
$$\textit{Value generated} \sim \underbrace{\textit{specimen volume x dpa}}_{\textit{product quantity}} \textit{ x } \overbrace{\textit{dpa}}^{\textit{"rarity value weight factor"}}$$

Design driving parameters

$$\Delta T \propto q''' \cdot \frac{t_S^2}{\lambda_S}$$

- Volumetric heating q''' & imposed limits on temperature spread
  ➔ maximum “thickness” of specimen stack t_S
- Ratio of electric heating vs. nuclear (gamma) heating
  ➔ possibility of temperature control
  ➔ heater lifetime limited by electric field
- Requested irradiation temperature level
  ➔ thickness of insulation (stagnant gas) gap
- Total released power, maximum allowed vessel temperature
  ➔ coolant flow rate (translates to coolant pressure head)
- Coolant pressure
  ➔ wall thickness of “body” / vessel

Generic temperature-controlled Irradiation setup (cylindrical)



HFTM 2021 characteristic features

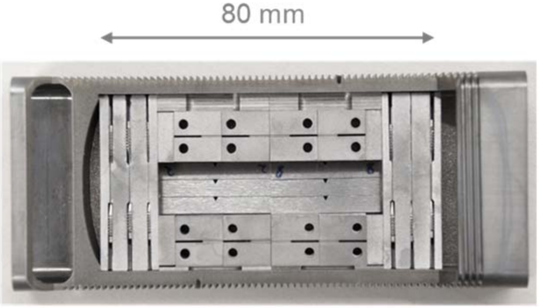
- Box-like structures (cuboid)
  ➔ increased specimen payload
  ➔ bending stresses

- Long slender shape
  ➔ move interfaces away from radiation footprint
  ➔ prone to deformations/deflections

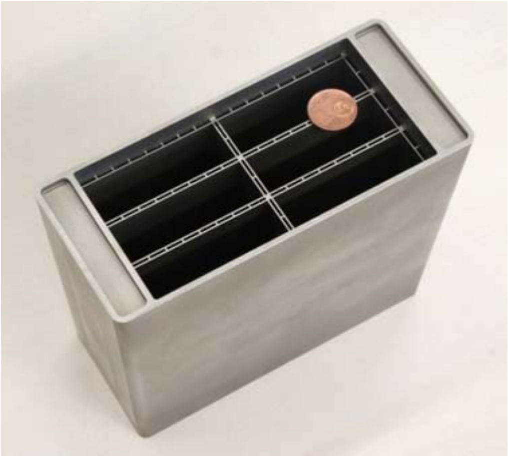
- Electric heating > 100% nuclear heating
  - allows full temperature control
  - consumes irradiation space, complicates manufacturing
  - may be a life-time limiting component

- Highly densified cross section
  ➔ Increased specimen payload
  ➔ challenging fabrication technology, assembly & QA

Balance between increasing raw specimen payload vs. effective irradiation capacity, which includes availability.



Cuboid specimen capsule:
80 SSTT specimens



HFTM body prototype
with integrated minichannels

Materials in the HFTM

- **Austenitic steels** (i.e. "316LN" X2 CrNiMo 17 12 2 - N)
  - included in the RCC-MRx nuclear pressure vessel code (up to 53 dpa)
  - mechanical properties acceptable up to ~ 650 °C
  - used ***for the pressure bearing shell*** (which contributes to the "containment" safety function)

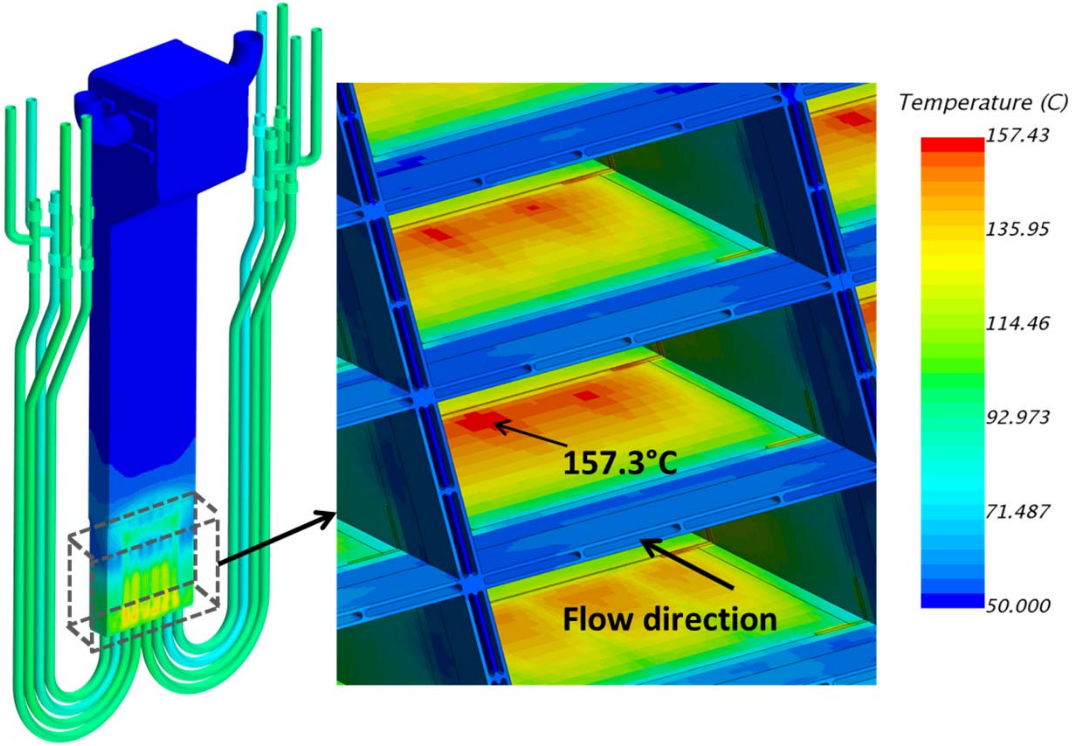
- **Ferritic/martensitic "9%-Cr" steels** (i.e. "Eurofer" X10 CrWVTa 9-1)
  - to be introduced to the RCC-MRx code, no irradiated properties included
  - mechanical properties acceptable up to 550 °C
  - used ***for the specimen capsules*** (which hold and heat the specimens)

structural

- **Sodium (Na)** (liquid) as ***heat transfer medium***
- **Cu-** based brazing (***to join*** parts of the specimen capsules + heater wires)
- **NiCr 80 20 alloy** as ***electrical resistance heater*** wires
- **MgO** ceramic compressed powder as ***electrical insulator***
- Others (as in sensors, i.e. type-K/N thermocouples, SPND, MFC, el. connectors, gaskets)

functional

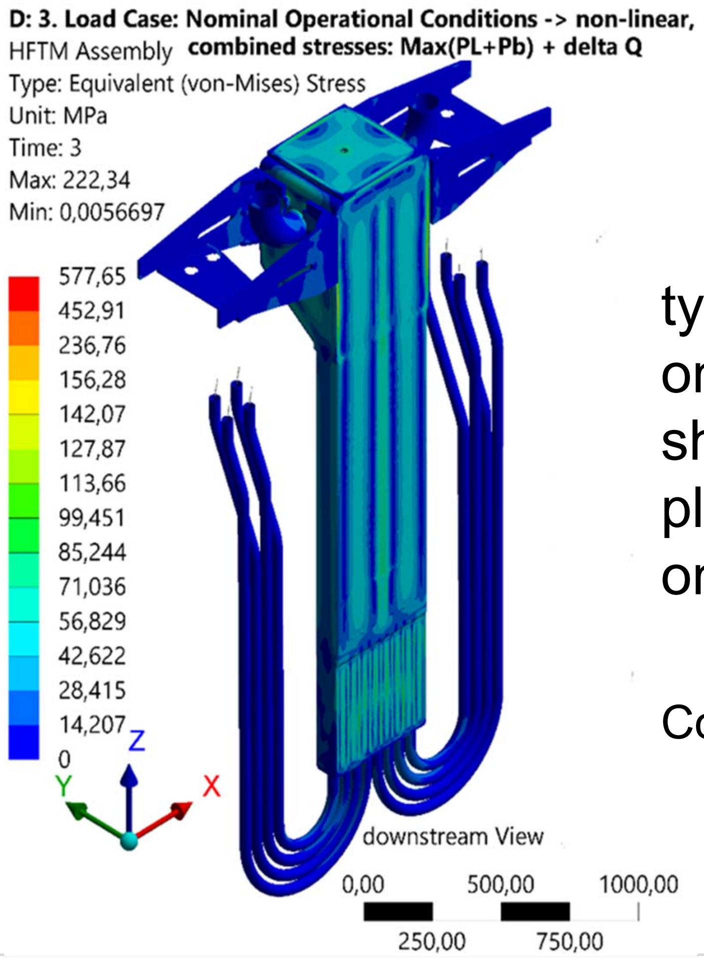
Simulated temperature field during irradiation



Temperatures on body, 316LN
50 – 140 °C typ, 157°C peak

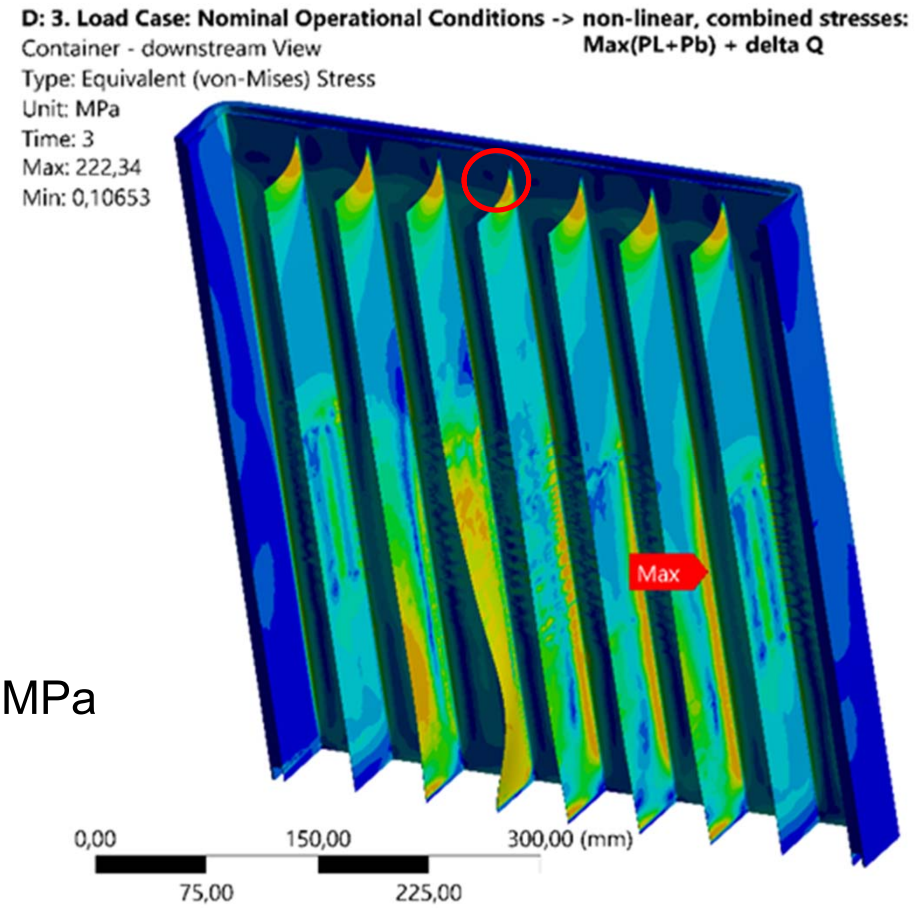
Temperatures in capsules Eurofer
250 / 350 / 450 / 550 °C

Simulated stresses during irradiation



typically ~80 MPa
on pressure bearing
shell, peaks (with
plastification)
on internal baffles.

Compare S_m(150°C)=141MPa



Overview on neutron irradiation damage modes

- Of interest for the structural components
  - Swelling
  - Irradiation hardening, loss of ductility
  - Embrittlement
  - irradiation enhanced creep
  - change of transport prop. (thermal conduct., permeation (i.e. hydrogen)
  - …

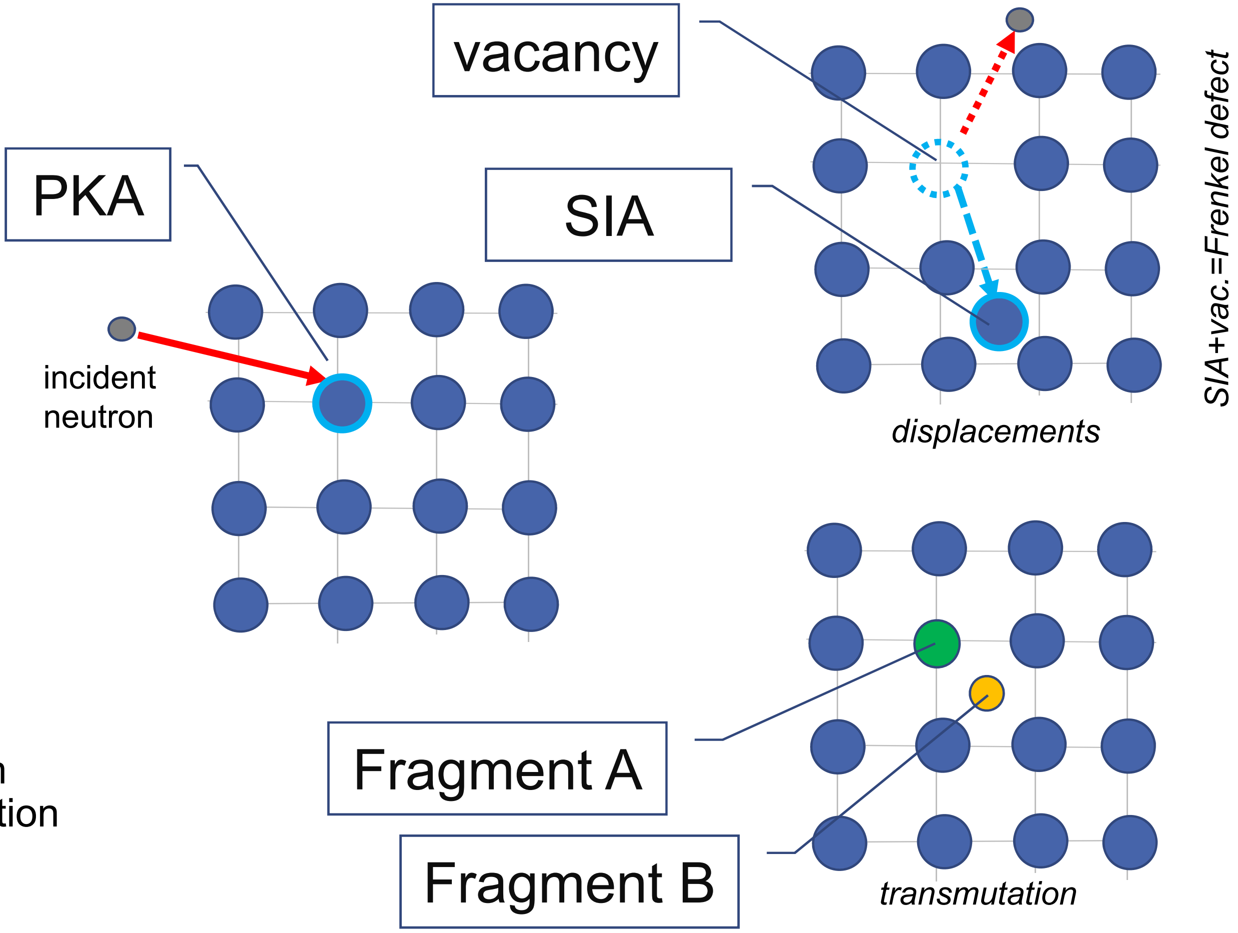
- Of aditional interest for functional materials
  - Radiation induced electrical degradation (of insulators)
  - electr. conductivity, optical transmission, reflectivity,
  - ...

Primary irradiation damage

$$dpa_{NRT}\left(T_{deposit}\right) \propto \frac{0.8\,T_{deposit}}{2\,E_d}$$

for large enough $T_{deposit}$

- **Displacement damage:**
  - incident neutron hits primary knock-on atom (PKA)
  - PKA can dislocate more lattice atoms ➔ **damage cascade**
  - After the energy is distributed below the displacement energy E_d (i.e. 40eV for Fe), the lattice is left with **vacancies** and **self interstitial atoms (SIA)**
  - **dpa : displacements per atom**, NRT formula or MD simulations ≠ *surviving* defects !
  - Relevance of **PKA energy spectrum**

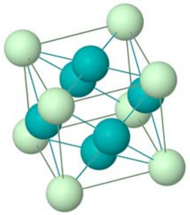


- **Transmutation:**
  - nuclear reaction of neutron and lattice atom according to incident energy and cross section

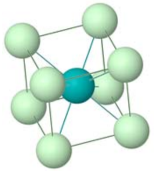
  ➔ **new alloying elements** are introduced !
  - Example: W → W-18Re-3Os @ 50 dpa !

Void Swelling

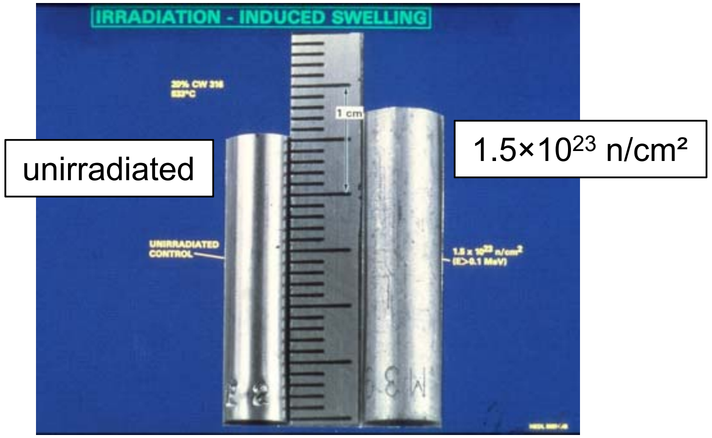
- Phenomenon: Increase of volume (at significant levels!), $\Delta V/V$ [%]
- occurs also in absence of stress
- more intensive in **fcc** lattice than **bcc** lattice
- incubation phase, followed by "linear regime"
- most pronounced at $T/T_m = 0.4 – 0.55$
- condensation of excess vacancies left behind in lattice into voids



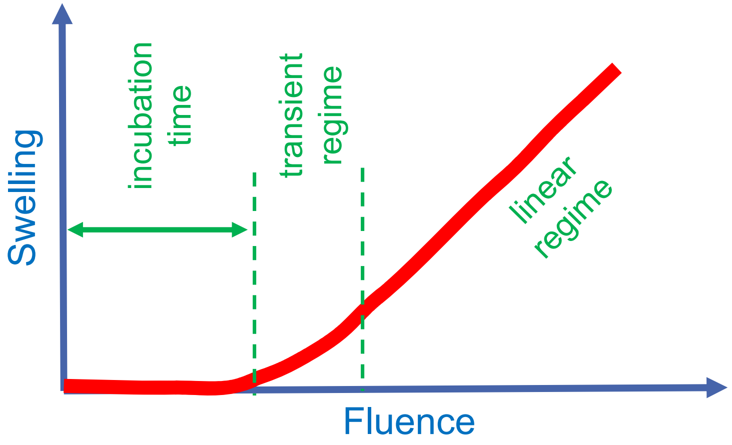
face centered cubic
fcc
→ austenitic steels



body centered cubic
bcc
→ ferritic steels

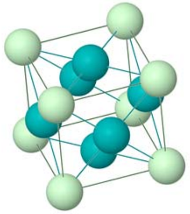


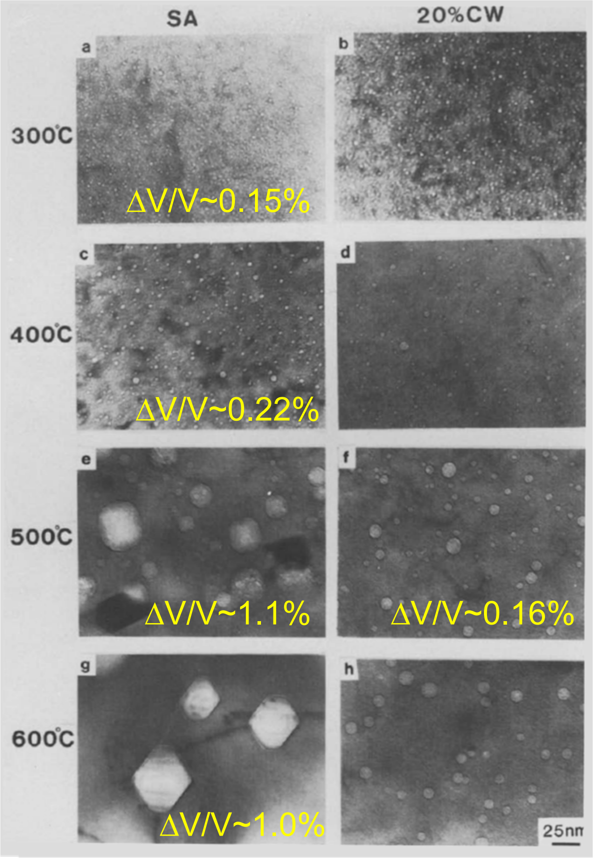
316L (cw) @ 533°C, [Straalsund 1982]



Austenitic steels, 300-series, fcc lattice

Microstructure evolution during swelling



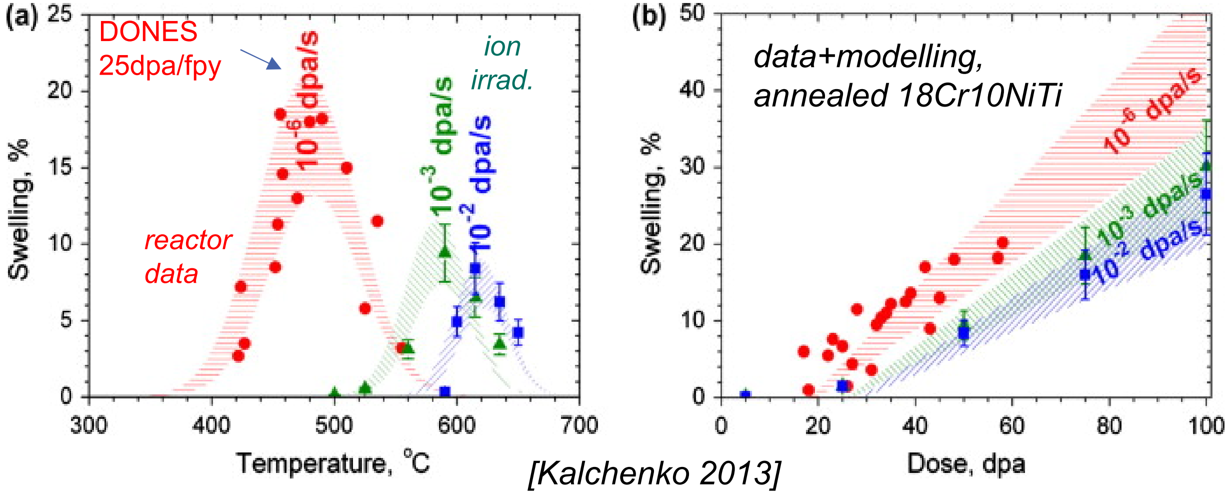


316 steel, 30-36dpa, 2327appm(He) in HFIR
[Hamada 1988]

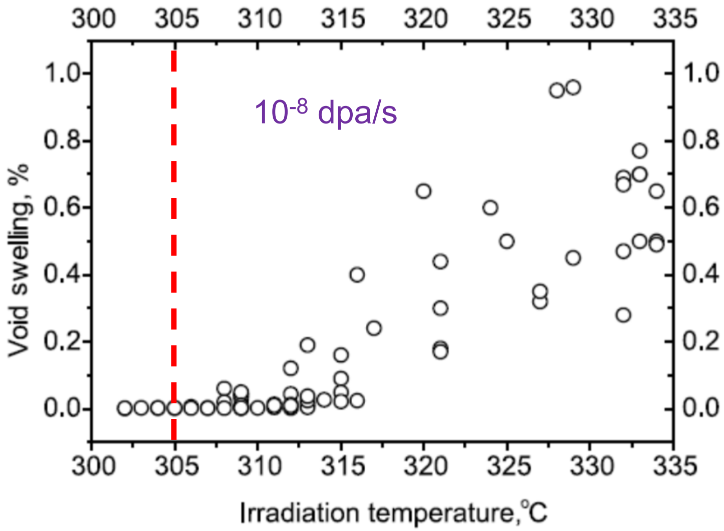
SA : solution annealed
CW : cold worked (20% deformation)

- At low temperatures:
  - high number of small voids
- At high temperatures:
  - coalescence to low number of large voids
  - high dislocation density in cold-worked (cw) steel provides more nucleation sites for bubbles

Sensitivities on irradiation conditions



[Kalchenko 2013]

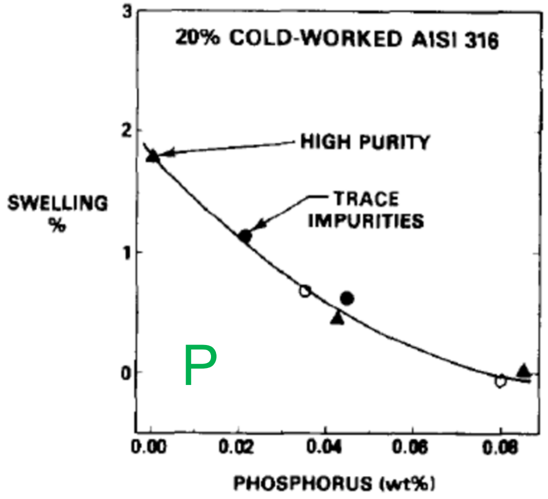


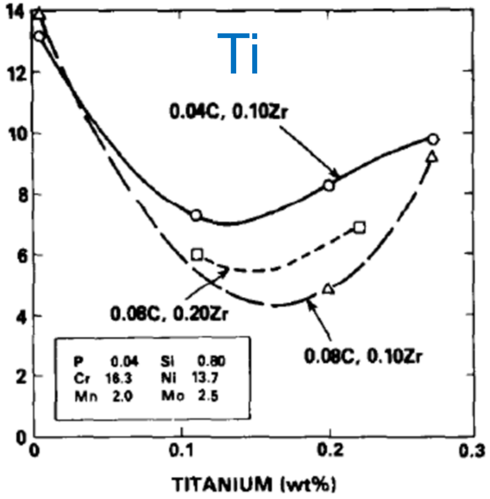
12X18H10T (comp. AISI321), 4-56 dpa in BN-350
[Porollo 2002]

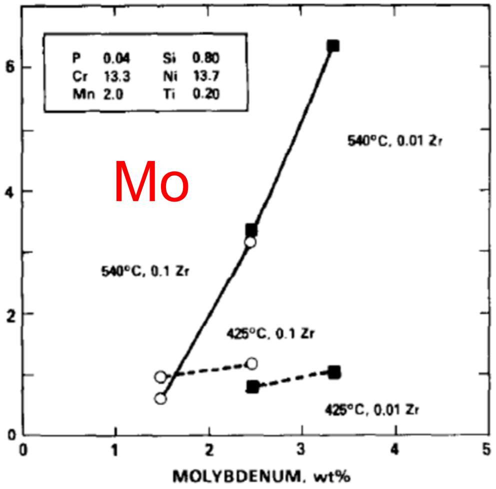
- Onset of swelling (incubation phase) depends on
  - Temperature
  - dose **rate** ➔ More swelling ΔV/V(dpa) at lower dose rates
  - Alloy and initial microstructure (cw vs. sa)

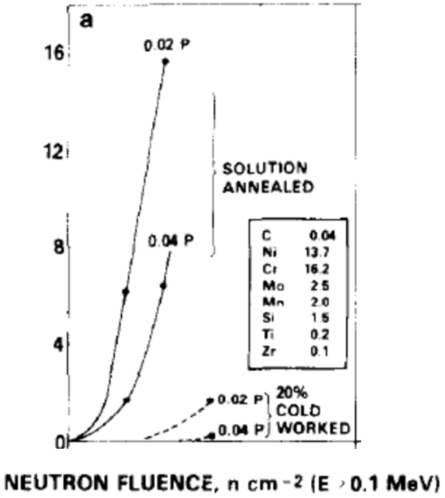
- Less swelling in fast reactors sometimes reported.

Swelling as f(alloy composition)









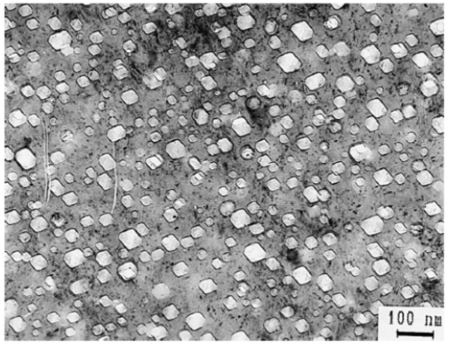
[Garner 1988] systematic alloying study (based on 316L base, 425 – 540 °C, 50-55 dpa)

- Alloy composition {P, Ti, C, Cr, Mo} and
- cold working vs. solution annealed

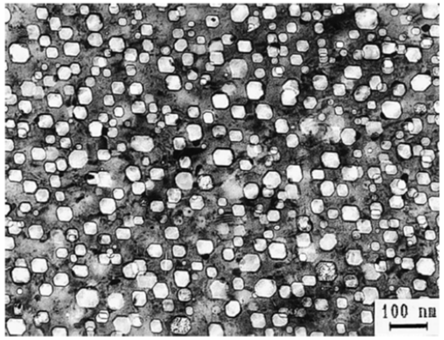
influences *transient regime* (but does not avoid "1% / dpa" in linear regime)

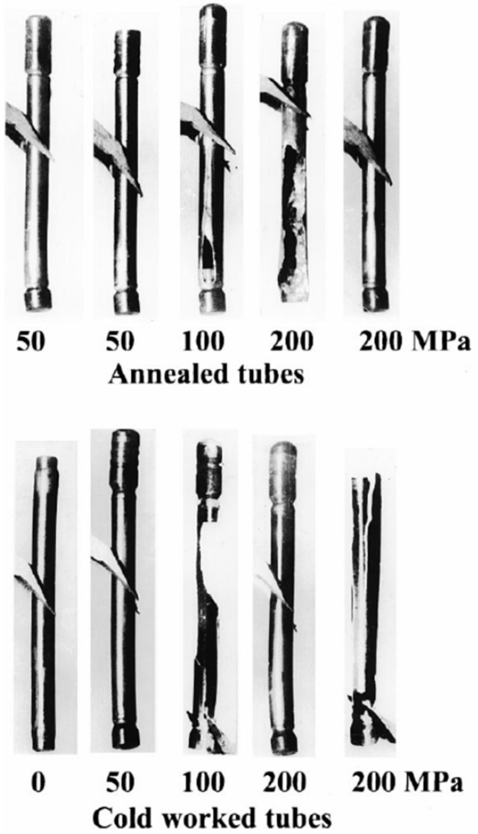
Failure conditions

335°C
73 dpa
(sa)



365°C
82 dpa
(cw)





"It is generally accepted that void swelling of austenitic stainless steels ceases below some temperature in the range 340-360°C, and exhibits relatively low swelling rates up to 420°C."

... but ...

Swelling (~10%) and **brittle failure**
of tubes made of EI-847 16Cr 15Ni 3Mo 0.55Nb austenitic steel irradiated at BN350
324 – 385 °C, **65 - 95 dpa**

[Porollo 1998]

Swelling situation of the 316LN HFTM container

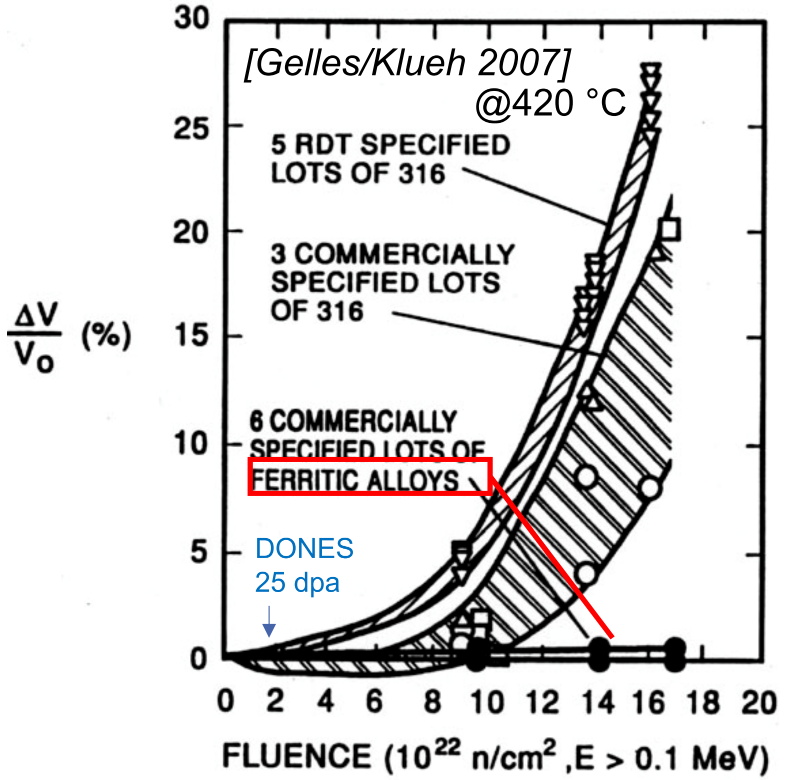
- Asymmetric irradiation:
  - front of container at ~25 dpa/fpy
  - back of container at ~ 3 dpa/fpy
- Outer structure temperatures 50 – 140 °C

swelling would effect bending/buckling of container, deformation of cooling channels !

- RCC-MRx code for 316LN : *"For temperatures less or equal than 400°C and irradiation damages less than 24 dpa, the swelling is negligible."*
- In fast reactors and at high dose rate, swelling was less (compared at the same dpa) BUT: in DONES additionally effect of larger Helium transmutation rate

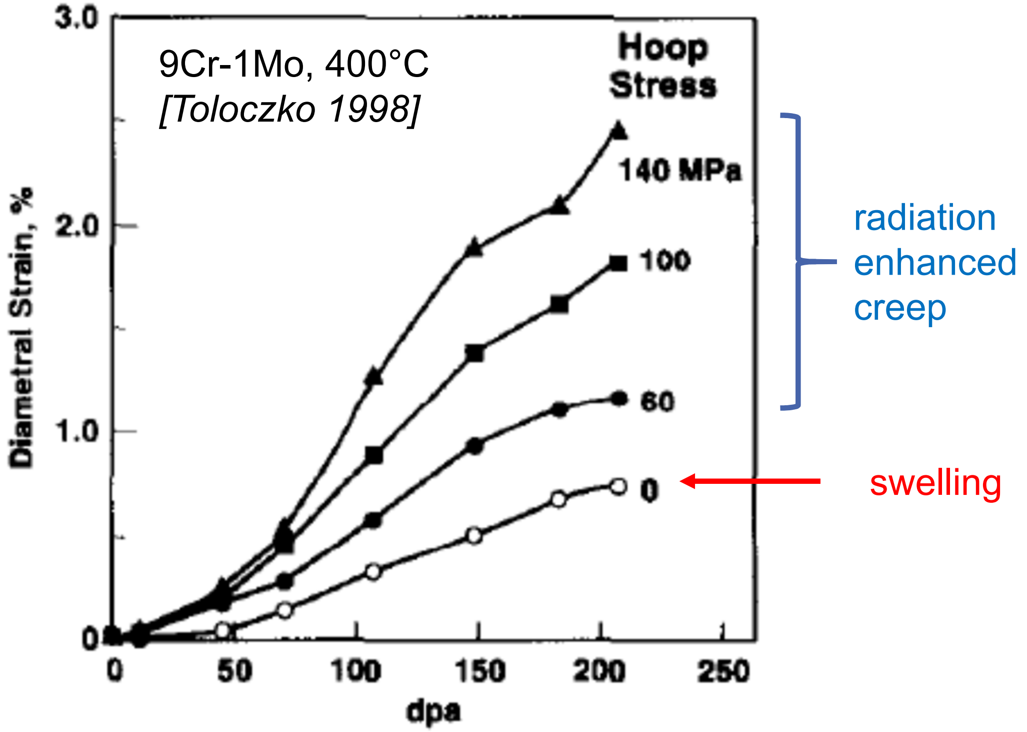
➔Good prospect for ~ 1year operation in DONES based on current knowledge.
316Ti od 321 steels could be attractive alternatives (just from *swelling* perspective).

Swelling of ferritic steels



[Gelles/Klueh 2007]
@420 °C

DONES
25 dpa



9Cr-1Mo, 400°C
[Toloczko 1998]

radiation enhanced creep

swelling

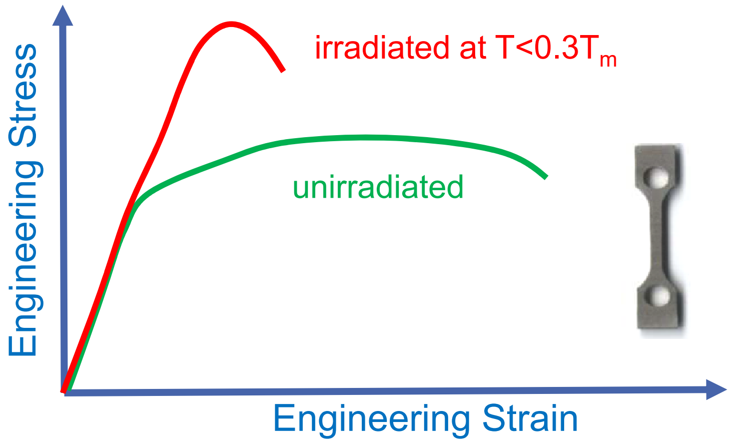
➔ the bcc lattice is much less prone to swelling than the fcc lattice

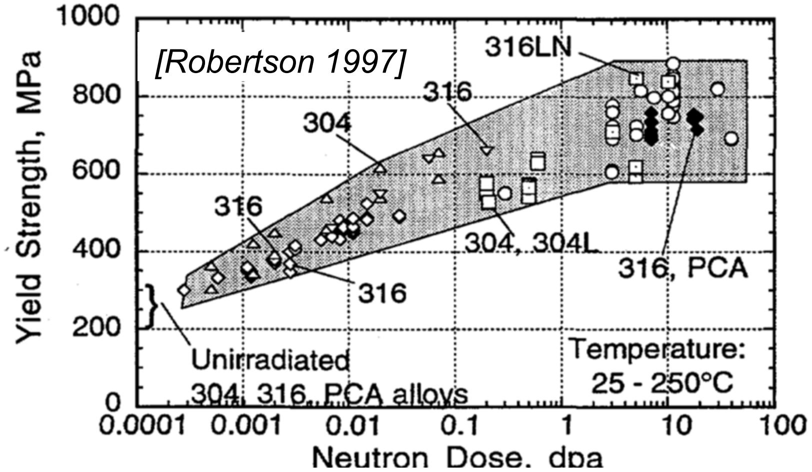
Tensile properties, Irradiation Hardening

Phenomena:

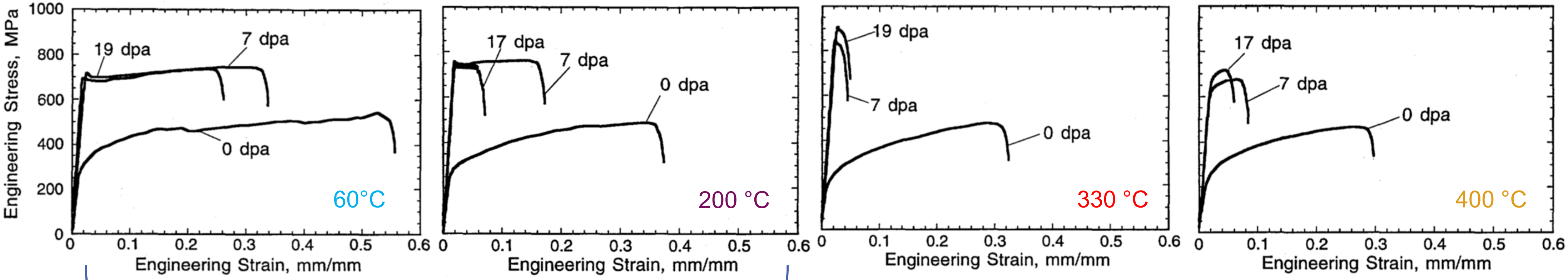
- At low temperatures, $T_{irr} < 0.3\ T_{melt}$
  - Increase of yield strength
  - reduced work hardening capability
  - Decrease of ductility, elongation
- At high temperature:
  - softening

- Effective already at low doses
- Localization of plastification
- Precipitates
- Sessile interstitial loops





Stainless steel (316)

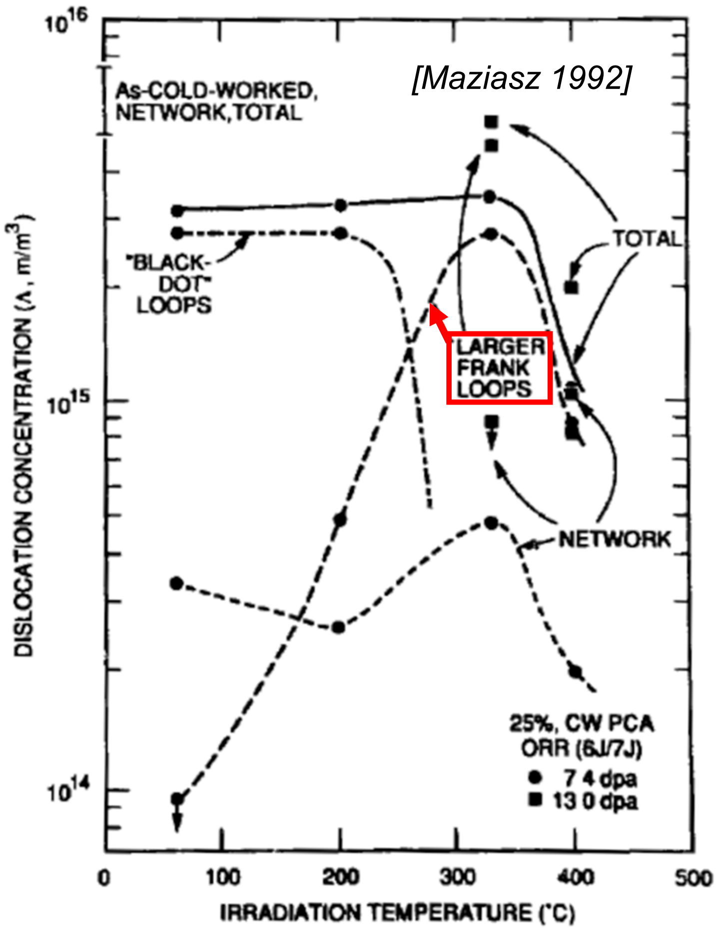


compare HFTM body operation conditions: 50-160 °C, 25 dpa

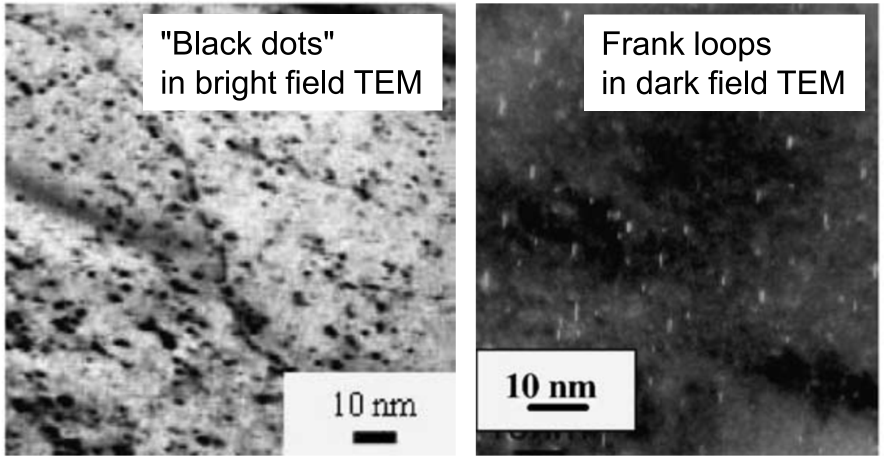
Solution annealed 316 steel, ORR+HFIR irradiation, 10-12 appm(He)/dpa, *[Robertson 1997]* :

- Increase of yield strength up to 300 % (saturates at >3 dpa)
- Loss of uniform elongation / strain to necking / total elongation esp. around 330 °C

Density and size of sessile interstitial loops



[Maziasz 1992]



"Black dots"
in bright field TEM

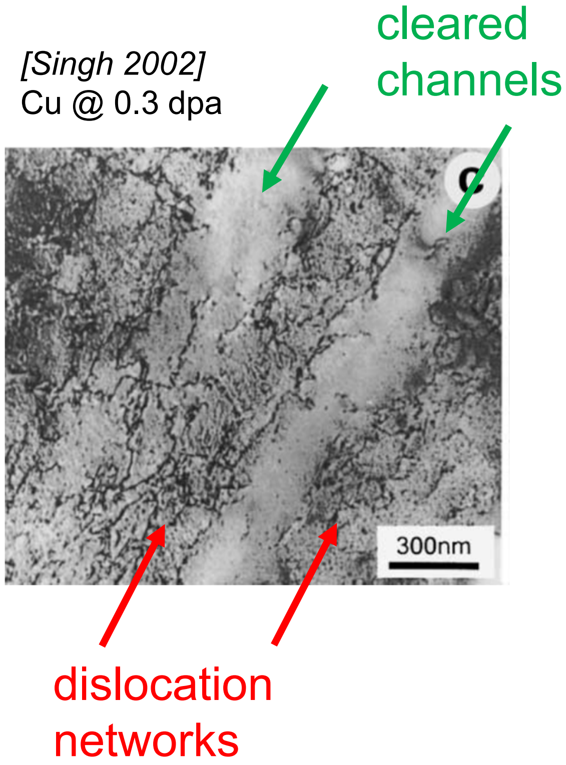
Frank loops
in dark field TEM

[Pokor 2004]

- Density of "Frank Loops" peaks around 330 °C
- Those are interstitial loops with a stacking fault
  ➔ they are sessile / can't glide
  ➔ obstacle for other dislocation movements
  ➔ contribute to the hardening

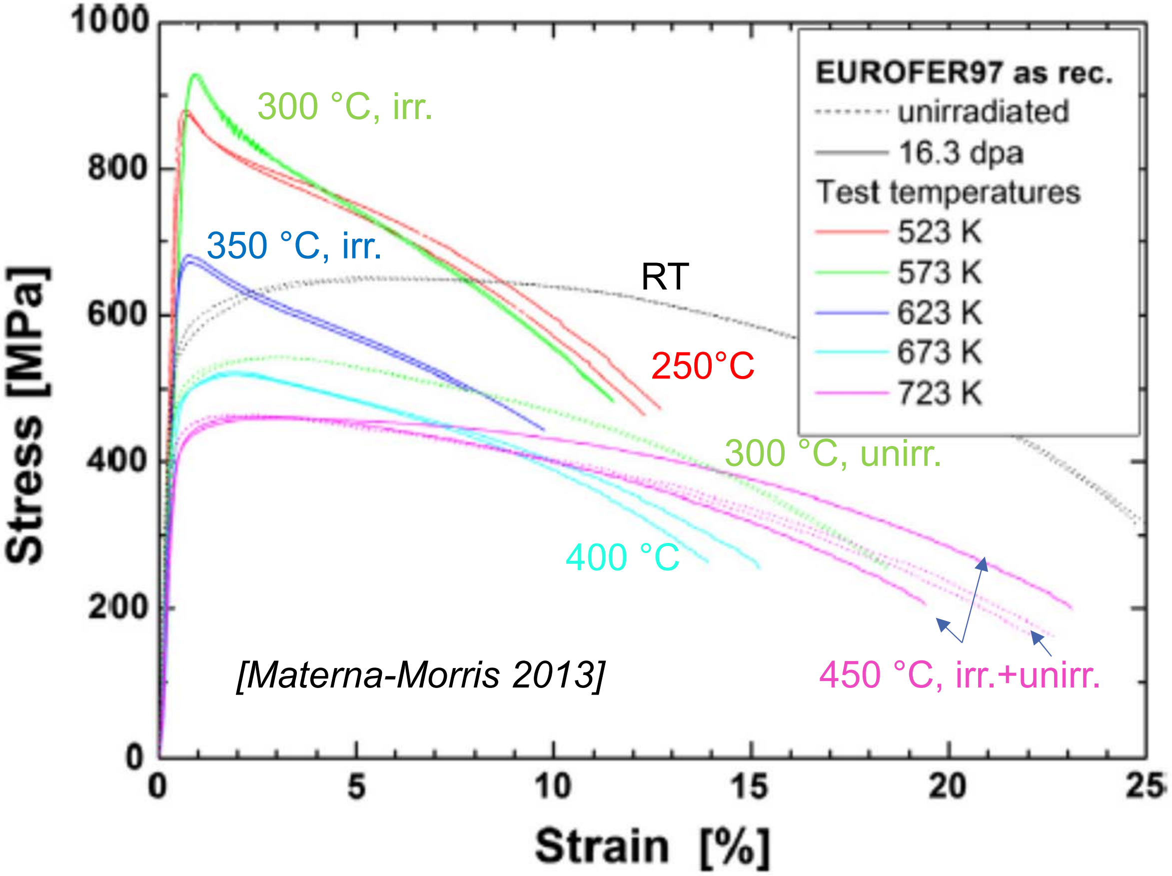
Localized plasticity / flow localization

[Singh 2002]
Cu @ 0.3 dpa



- The plastic flow becomes highly localized
- flow localization results in low ductility
- Relation to observed alternating zones of cleared channels and dislocation networks in the material.

Ferritic steel (Eurofer)



[Materna-Morris 2013]

Below 350 °C :

- hardening
- loss of uniform elongation

Above 350°C :

- balancing of hardening with ageing

Steps towards radiation resistant design of the Eurofer specimen capsules

- The temperatures are **fixed by the mission**: 250 / 350 / 450 / 550 °C
  ➔ embrittlement at 250 °C
  ➔ creep at 550 °C
- After specimens insertion: no more heating > ~ 250 °C allowed:
  ➔ **no post-weld heat treatment possible** !

Approaches:

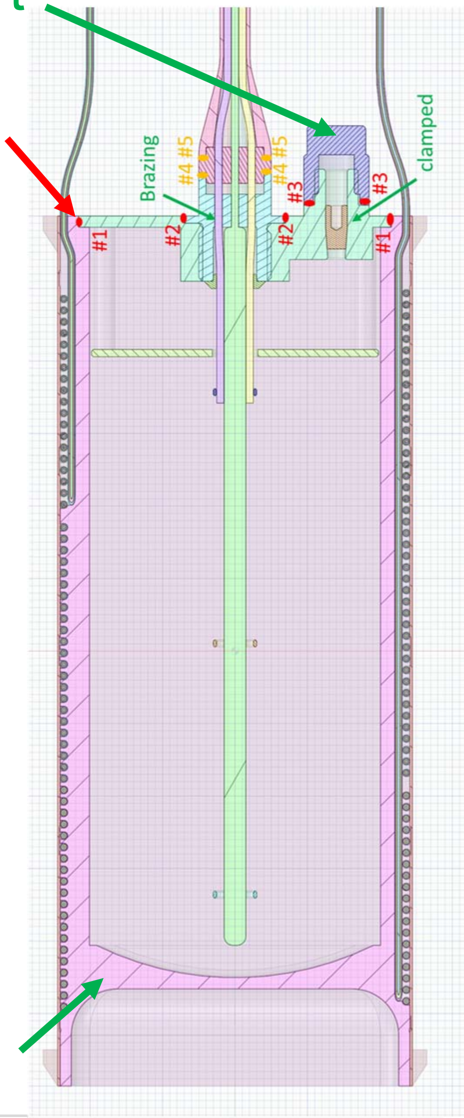
- IFMIF/EVEDA: inert gas filling, up to 6 bar int. pressure at 550 °C

➔ new design : Evacuation after filling to avoid stresses

- IFMIF/EVEDA: prismatic body with welded bottom plug

➔ new design: Avoidance of welds where possible (die sink erosion)

- Favourable swelling properties of the ferritic 9%Cr steel (compared to austenite)



evacuation port

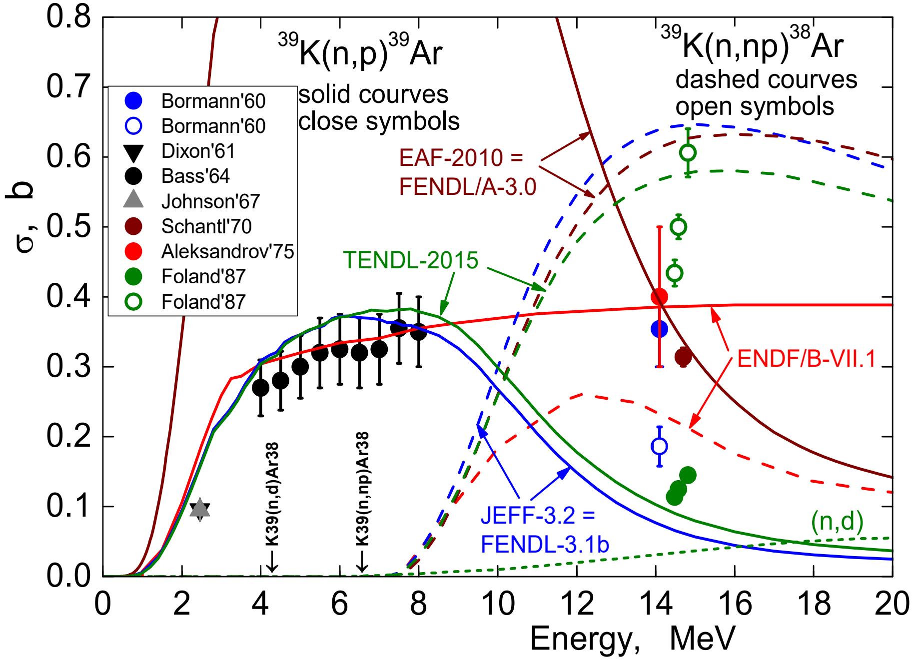
no PWHT !

weld avoided

Transmutation gases

- Up to 2015, the heat transfer medium between the specimens was
  NaK sodium potassium eutectic alloy
- NaK is liquid at room temperature, favourable for filling procedure
- It was observed in the results of activation analysis that a considerable amount of Argon (~24cm³ of Ar gas at STP) would be produced
- Argon solubility in NaK is low. In an expansion volume of 2 cm³ at 550°C, this full amount corresponds to a pressure of **36.27 bar abs**

➔ **NaK was replaced by Na in the current design**



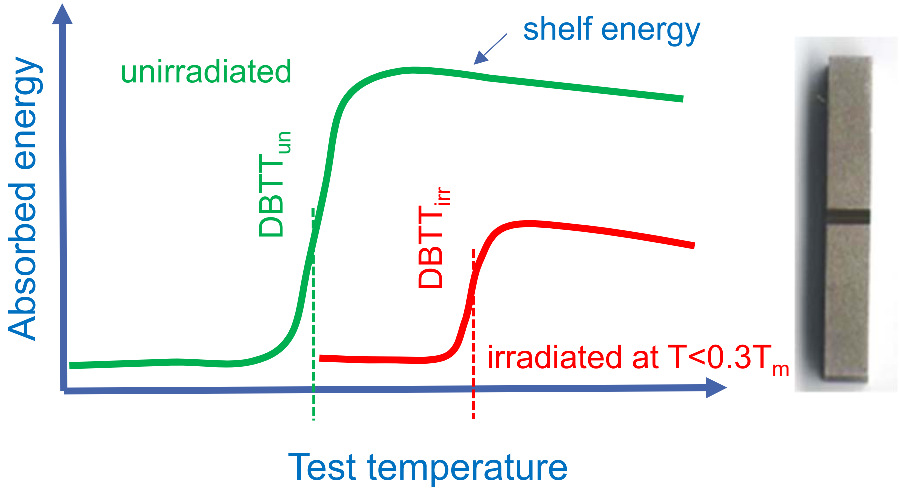
Cross section data for Ar production from K [Qiu,Simakov 2017]

Irradiation embrittlement

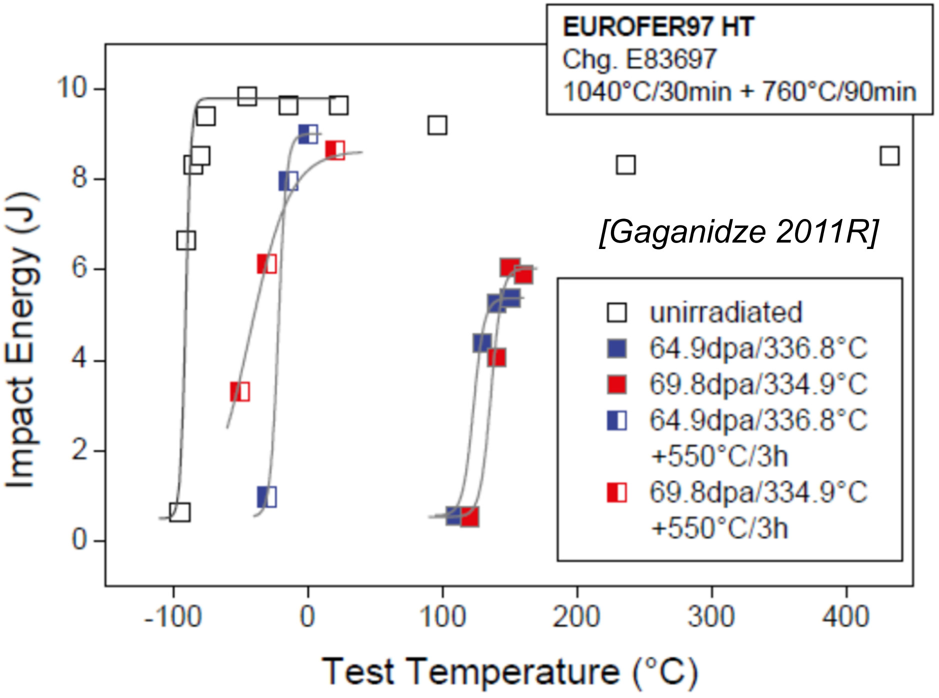
- Results of charpy impact tests, after irradiation at low temperatures:
    - **increase of the ductile-to-brittle transition temperature (DBTT)** during irradiation
    - decrease the shelf energy

    ➔ lower resistance to shock

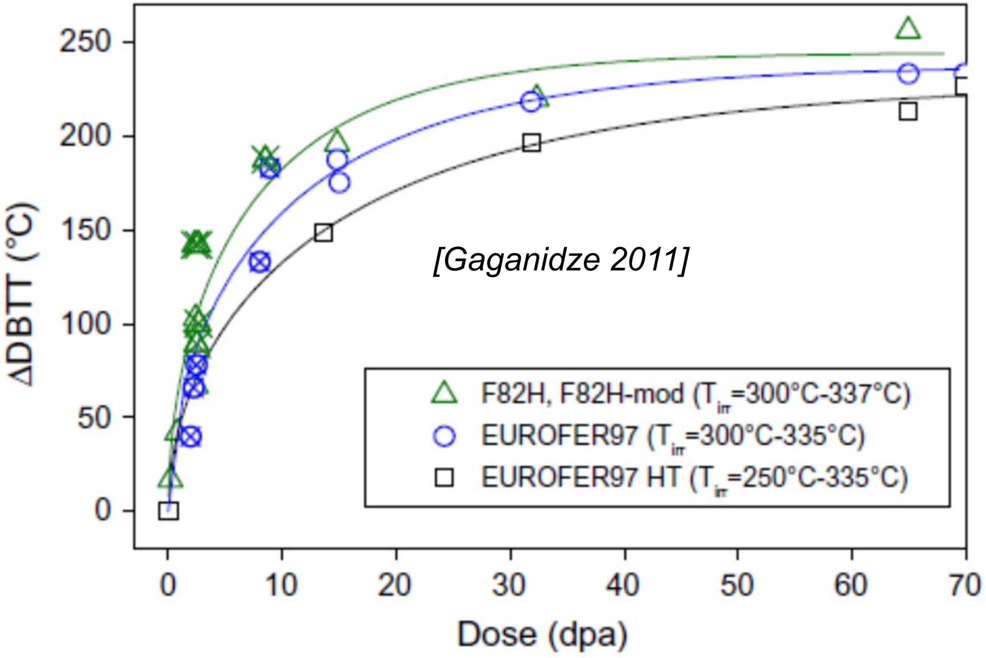
- Related to irradiation hardening
- Steep increase at low dpa, expected to saturate at high doses
- Decohesion of grain boundary by segregation of "tramp" alloying elements
- Evolution of carbides and intermetallic phases
- Helium bubbles



Embrittlement of Eurofer



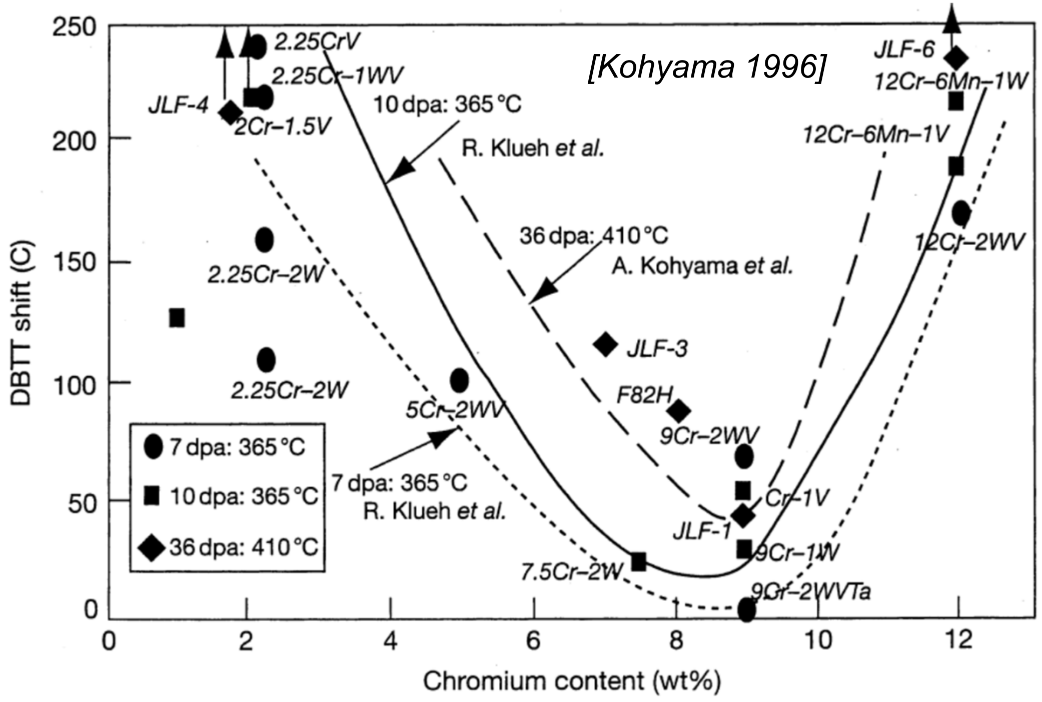
[Gaganidze 2011R]



[Gaganidze 2011]

- Significant for T_{irr}<350 °C : DBTT increases by ~ 200 °C
- For 400°C and higher : less relevance

Alloying dependence of embrittlement


[Kohyama 1996]

V, Nb, Ta "stabilization":
form fine & uniform carbides / carbonitrides

➔ optimum Cr content ~ 9% : F82H, Eurofer97

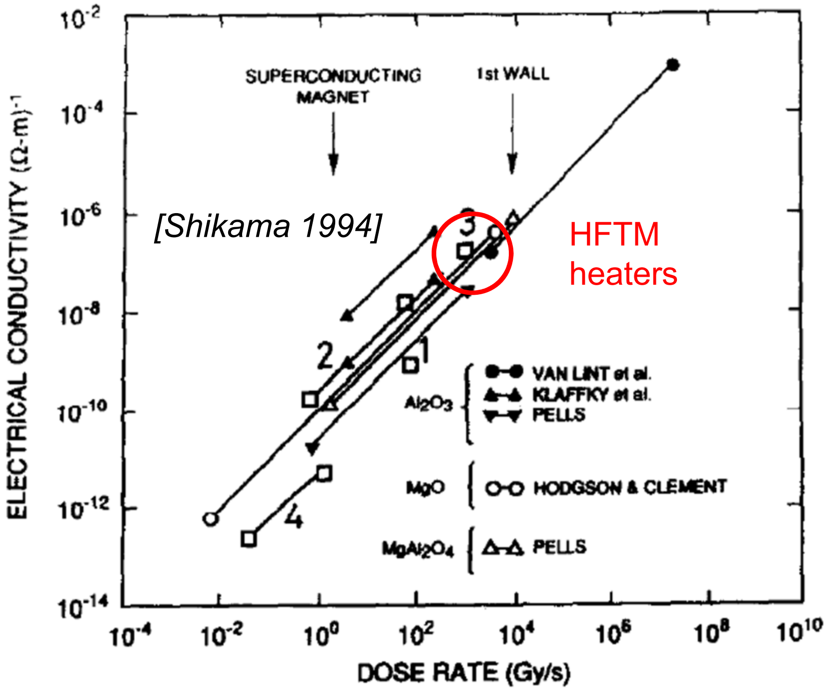
Radiation induced conductivity in insulators

Phenomenon: increase of electrical conductivity σ

- *Instantanous/transient* : Radiation Induced Conductivity RIC
  - excitation of electrons to conduction band
  - **depends primarily on ionization dose *rate***
  - Saturates after initial transient phase.
    Then depends on dose rate DR: $\sigma = \sigma_0 + k \cdot DR^{\delta}$

- *Permanent* : Radiation Induced Electrical Degradation RIED
  - Increase of σ_0
  - **depends on *accumulated* ionization dose**
  - Effective at applied electrical field, 0.6 MV/m
  - becomes effective after offset dose
  - moderate temperatures ~ 400 - 500 °C

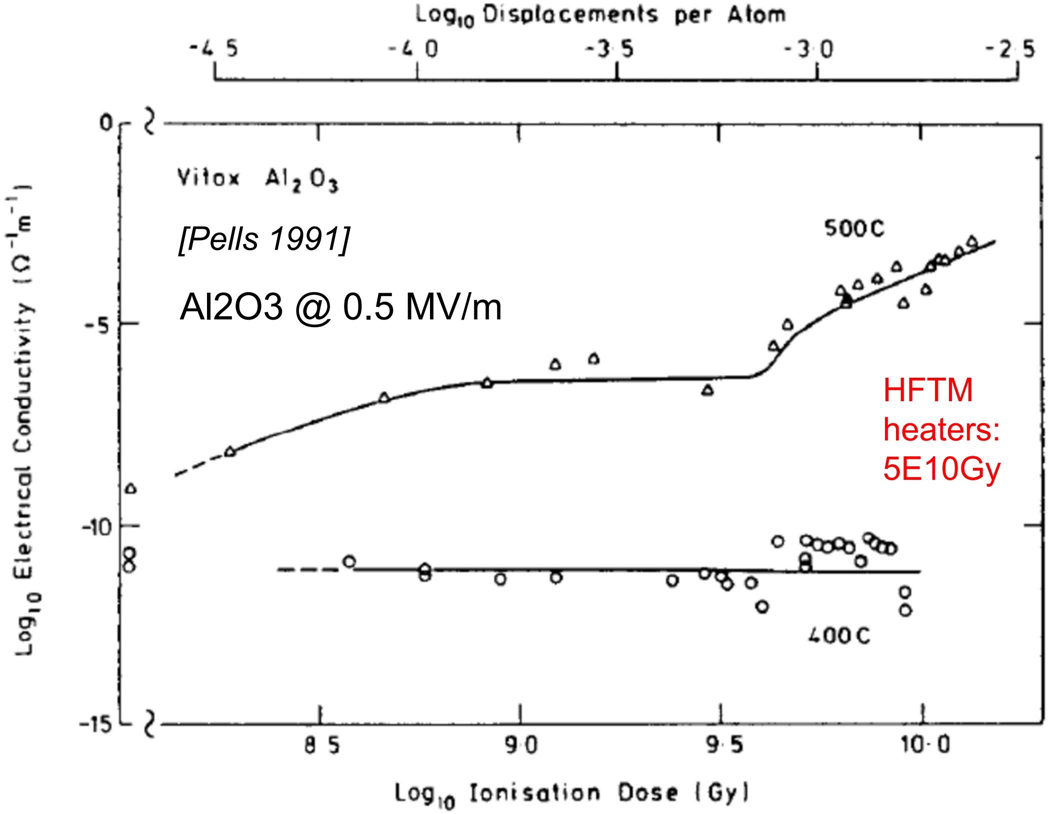
RIC and RIED data



[Shikama 1994]

HFTM heaters

RIC : dose-rate dependent



[Pells 1991]

Al2O3 @ 0.5 MV/m

HFTM heaters: 5E10Gy

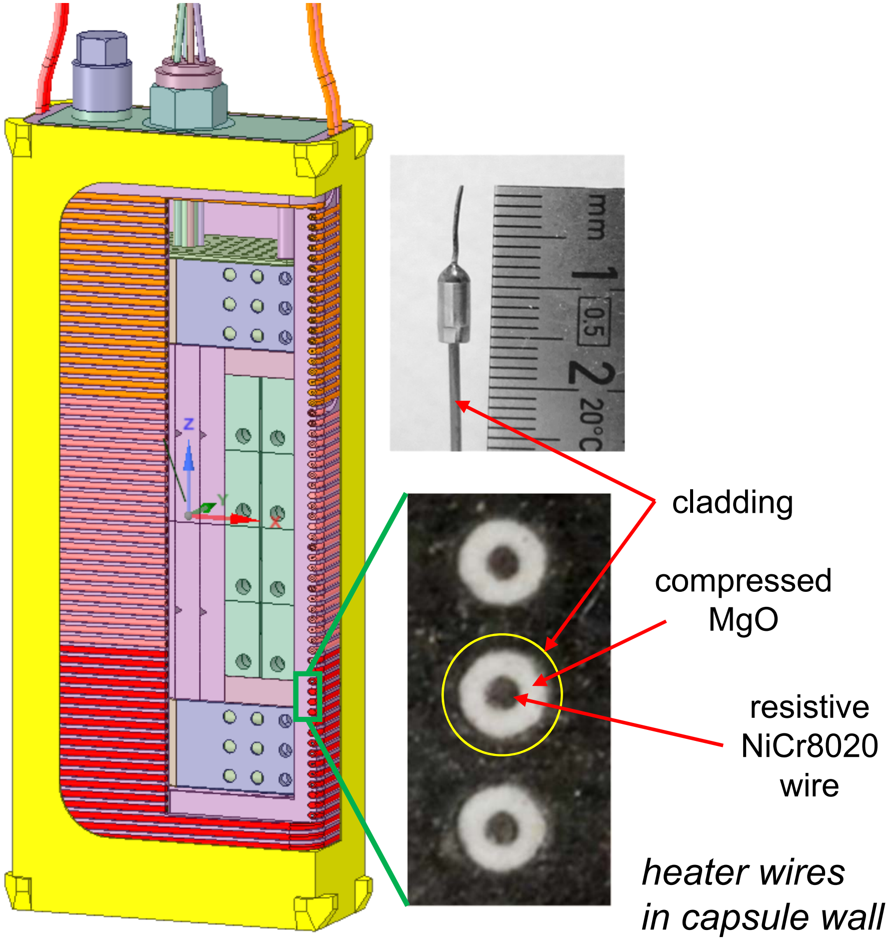
RIED : dose dependent

RIC & RIED in HFTM capsules heaters

- 1mm OD metal-clad mineral-insulated heater wires are used
- Heater wires are wound on machined spiral groves of capsule
- Capsule, heater and "sleeve" are HT-brazed to single component
- ~25% or diameter is compressed MgO powder
- Operation voltage max. 140 V

➔ 0.56 MV/m, O(10^3 Gy/s) :

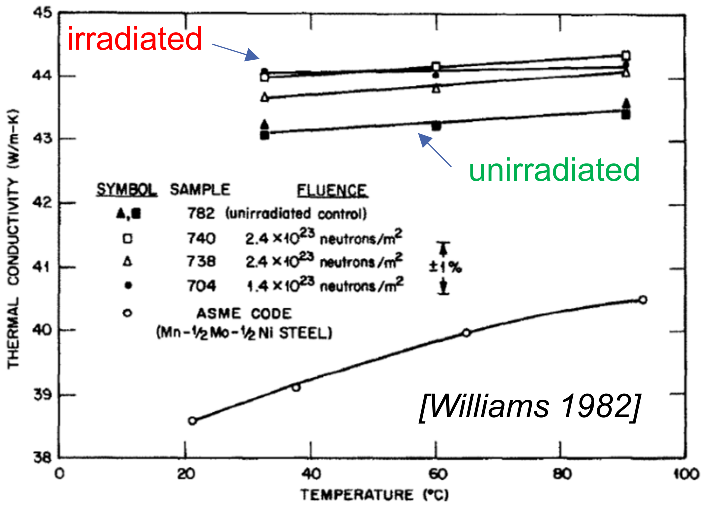
Conditions where RIED can be expected
➔ Dedicated test of heater wires in MARIA reactor, ongoing campaign.



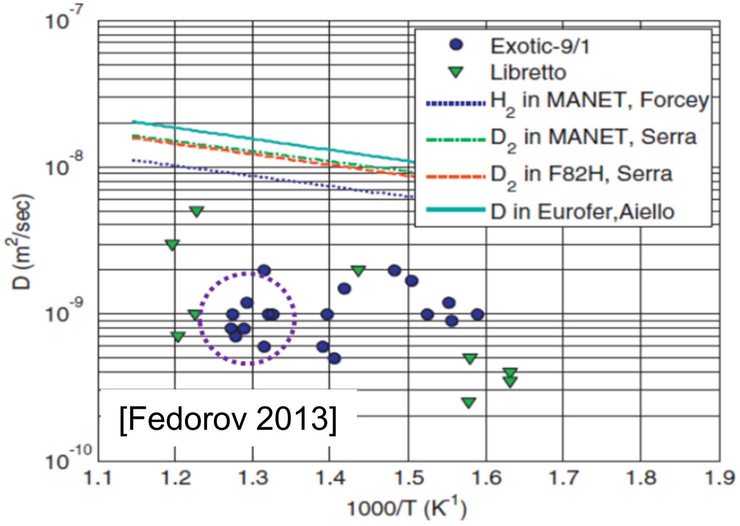
heater wires in capsule wall

Transport properties (exemplary)

- Thermal conductivity ➔ important for first-wall design
  - In uranium fuels : TC decreases with burnup
  - 2 references for steels found, where TC actually increased by neutron irr.
- Diffusivity of hydrogen ➔ important for tritium safety aspects
  - Effective diffusivity reported to be decreased ~ factor 10 during neutron irradiation experiments

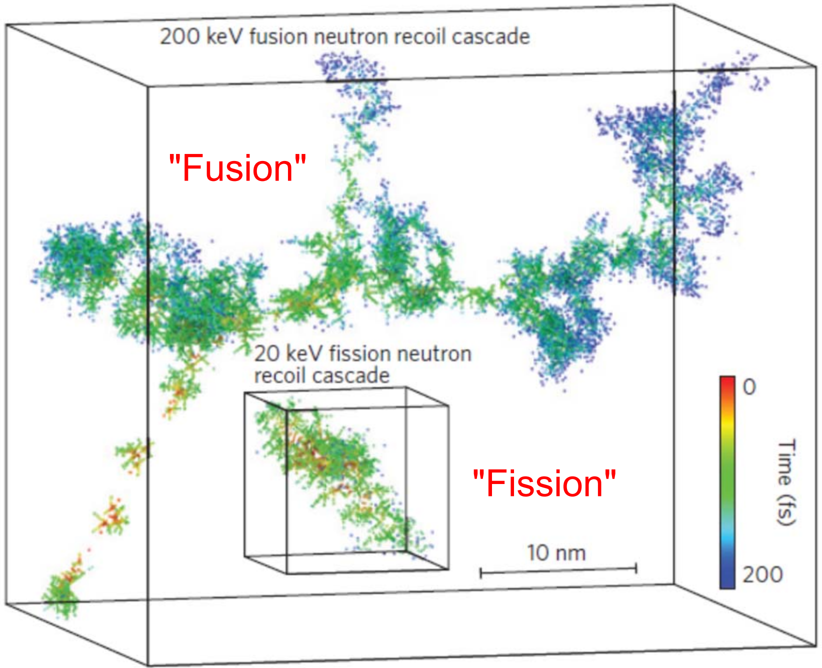


[Williams 1982]

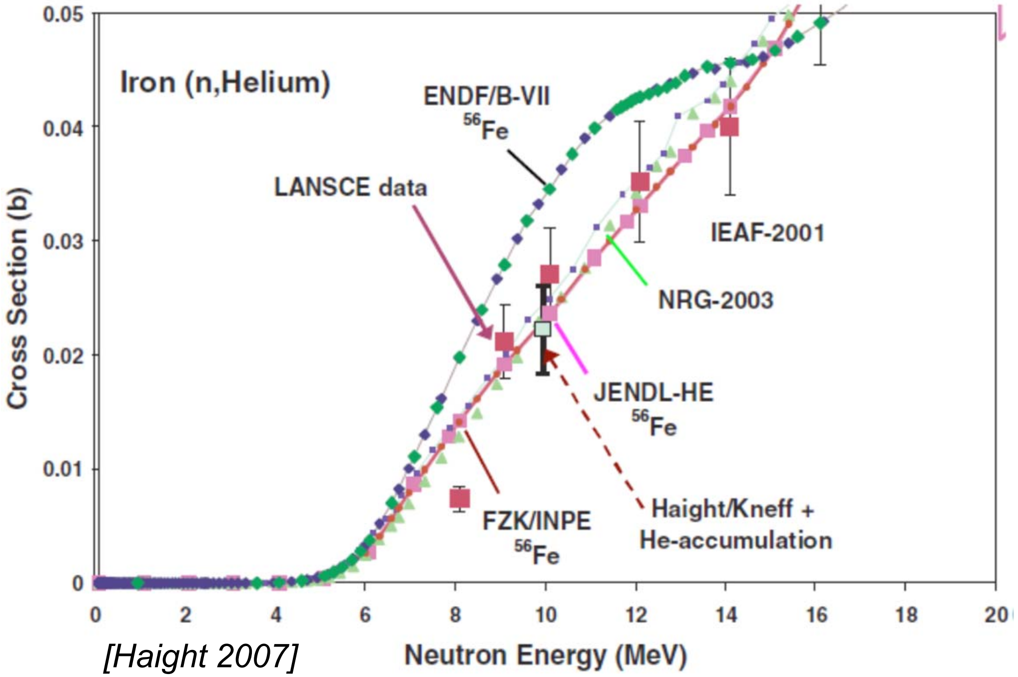


[Fedorov 2013]

What's different with fusion neutrons ?



[Sand, Nordlund in Knaster 2016]



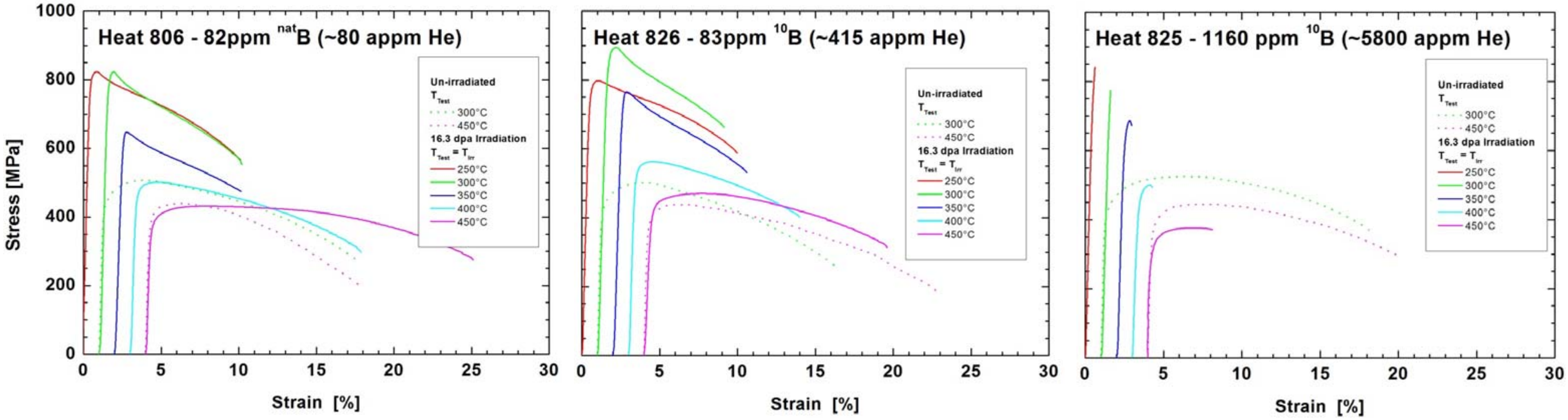
[Haight 2007]

➔ Displacements cascades from fusion neutrons are much larger (however subcascades are similar)
➔ Helium production cross sections of Fe (and Ni, Cr) have a threshold >5 MeV

Effect of He by ^{10}B doping on tensile properties

"Trick" : dope B or Ni to alloy, to increase he production rate with fission neutrons:

^{10}B (n, α)^{7}Li or ^{58}Ni (n, γ)57Ni (n, α)^{56}Fe



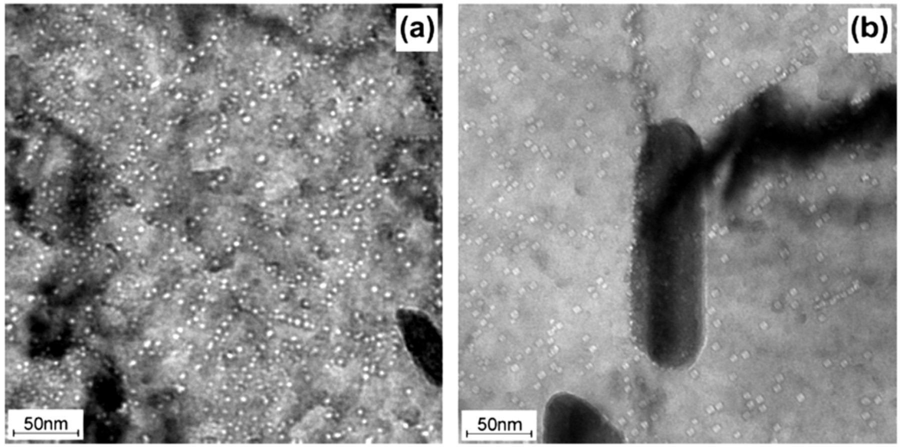
Irradiation of Eurofer to 16 dpa, varying contents of Boron *[Materna-Morris 2009]*

415 appm He : Strength increase but hardly reduction of total elongation

5800 appm He : Entirely brittle fracture; total loss of plasticity

Microstructure in irradiated ^{10}B doped Eurofer

halo region around BN "average" region



Klimenkov 2013, 16dpa

$^{10}B(n,\alpha)^{7}Li$

- B not evenly distributed: BN precipitates ~100nm at grain boundaries
  ➔He **bubbles unevenly concentrated in "halos"** in projectile range"of 2.4µm
- Additional to helium, Lithium is also transmuted
  ➔ involuntary **alloying of "tramp element"**

➔ Simulation of He production by boron doping yields unrealistically pessimistic results

Way forward to a materials database for DEMO

- A broad database on fission-irradiated materials is available, even at very high doses.

But:

- Neglecting the higher He, H transmutaion rates is not conservative
- "Doping trick" for He production (and spallation sources) yield (probably!) overly pessimistic results, margins for engineering an attractive design are too small !

- The DONES neutron source is tailored to match the DEMO Breeding Blanket situation
  - dpa rates 1:1 and higher than in the DEMO Breeder Blanket
  - dpa dose p.a. in DONES (25 dpa) exceeds DEMO starter blanket (20 dpa)
  - well matched He/dpa and H/dpa tranmutation rates
  - well matched PKA energy spectrum

➔ IFMIF-DONES has been integrated into the fusion roadmap to close the gaps.

Conclusions

- There is a large database of radiation effects with fission neutrons, up to 200 dpa
    - For swelling, hardening, creep, (...) a good experimental and theoretical basis is established
    - Other effects, like RIED, transport properties, (...) are still insufficiently understood
- Helium-effects can't be assessed in a satisfactory way with existing irradiation techniques
- The DONES neutron source can fill many knowledge gaps for DEMO design and licensing
- Other issues, like plasma-wall interaction, will depend on other material test facilities
- The DONES-HFTM will be the *first* pressurized device to experience 14MeV neutrons at high dpa:
    - The current knowledge base is used in the design
    - The design aims to avoid / work around radiation issues where possible
    - Some weak spots (i.e. heaters) became evident and will be addressed by load minimization and dedicated testing
    - During the startup of DONES, operational experiences must be integrated to improve the device.