Australian Government

ansto

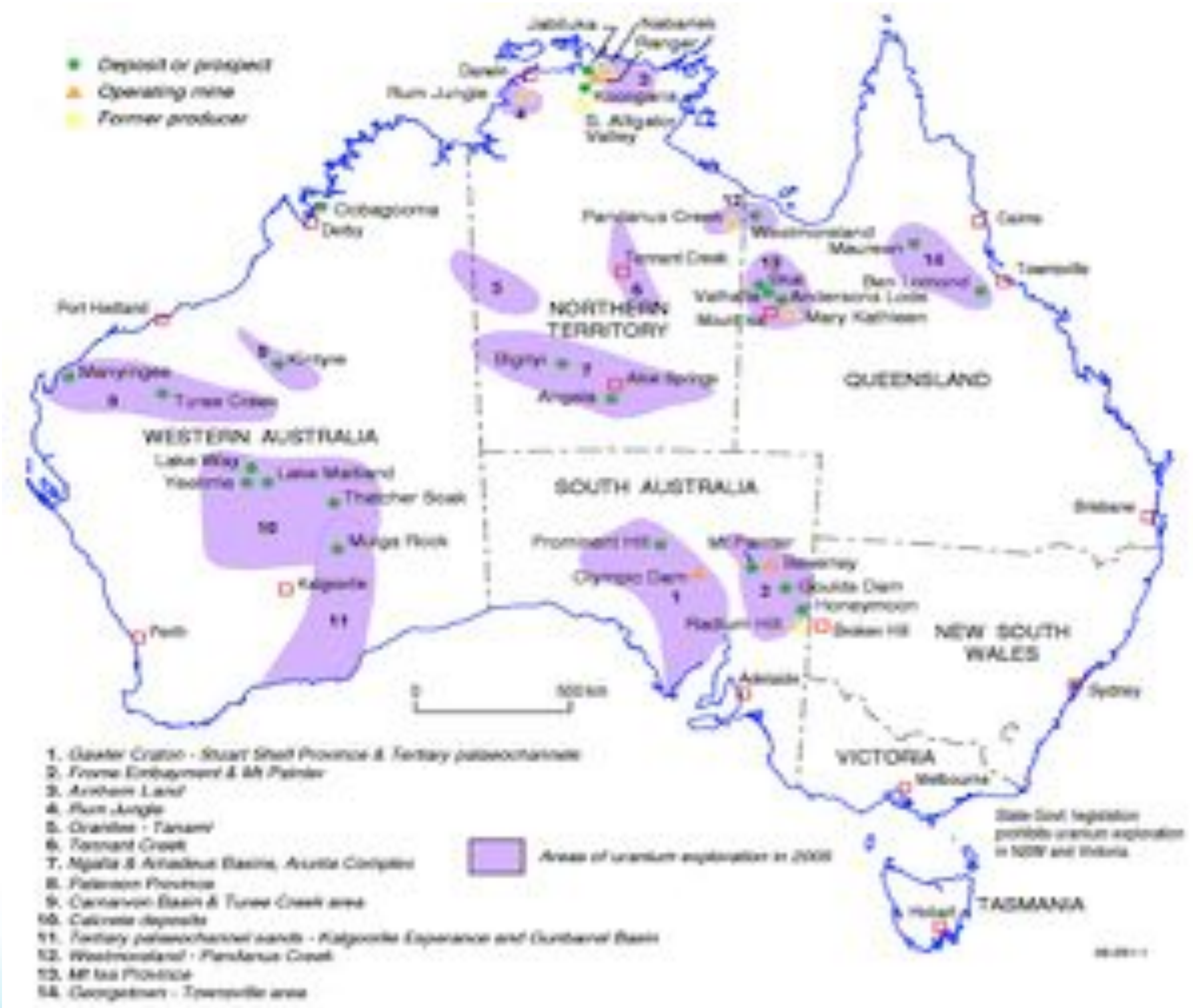
Australian Molten Salt Reactor Materials Research

Prof Lyndon Edwards and colleagues,

Institute of Materials Engineering

Australia and ANSTO

- Has the world’s largest reserves of uranium (23% of the world total)
- Is the world's third-ranking producer behind Kazakhstan and Canada
- In 2010-11, produced over 7000 tonnes of uranium oxide concentrate
- Exports to countries who have signed the Nuclear Non-Proliferation Treaty.
- Has had a national nuclear laboratory: ANSTO, for 60 years.



ANSTO History

- Began as Australian Atomic Energy Commission (AAEC) in 1953
- HIgh Flux Australian Reactor - HIFAR commissioned in 1960 (Dido type)
- 10 MW closed tank heavy water reactor
- Also operated *critical facility* and 100 kW reactor (MOATA)
- Selected design for Jervis Bay 1st Australian power reactor (UK 600 MWe SGHWR) (Cancelled in 1971)
- Focus of AAEC and HIFAR changed
  - radioisotope production
  - neutron-based research
  - materials irradiation
- ANSTO formed 1987
- HIFAR use extended to include
  - silicon irradiation

Decision to fund construction of replacement reactor in 1997

ANSTO TODAY:
Lucas Heights Facility

- 70 hectare campus south of Sydney
- Employs ≈1200 staff

OPAL Reactor

- 20-MW multipurpose open-pool research reactor
- First criticality in 2006
- Provides medical isotopes, irradiation facilities and neutrons for scientific research

Centre for Accelerator Science

- Houses multiple accelerator systems that are used for materials characterisation and ion implantation

Institute of Materials Engineering

- Employs ≈100 staff
- Core responsibilities include undertaking nuclear fuel cycle R&D to maintain Australia’s nuclear capability.

ANSTO as a National Laboratory

- Undertake and facilitate research in the national interest
- Produce and provide state-of the art radiopharmaceuticals for Australia and the world
- Provide trusted advice to Government in all aspects of the Nuclear Fuel Cycle

by

- Operating ANSTO's nuclear facilities including the OPAL nuclear research reactor in a safe and efficient manner

ANSTO and Nuclear Fuel Cycle R&D

- Fuel/cladding interactions
  - Use of atomistic modelling (*e.g.* DFT)
- Structural materials performance under extreme conditions
  - Irradiation, corrosion, high temperatures and/or deformation
- Separation science
  - Synthesis and analysis of titania frameworks for separation of U, Pu
- Wasteform fabrication
  - Construction of a Synroc waste treatment plant to reduce volume of ANSTO nuclear by-products by 99%

ANSTO/SINAP TMSR Materials Research Program

- Shanghai Institute of Applied Physics (SINAP), Chinese Academy of Science (CAS)
  - Centre for Thorium Molten Salt Reactor (TMSR) systems
  - See later presentation.
- Australia/China Science and Research Fund Grant 2013-2015
- ANSTO-SINAP Joint Materials Research Centre
  - Materials technology for Molten Salt Reactors
    - Molten salt corrosion
    - Radiation damage
    - High temperature behaviour

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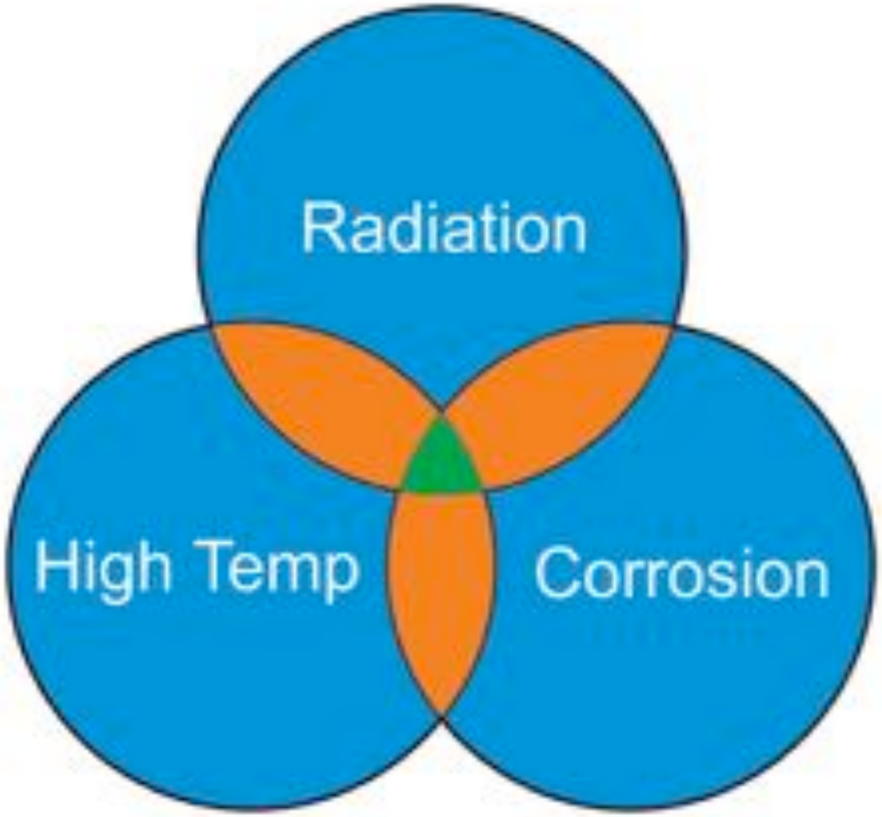
Materials Studied:

1. GH3535, a Chinese variant of Alloy N with the nominal composition of Ni–16Mo–7Cr–4Fe and Si used as an O getter.
2. Various Grades of Nuclear Graphite.

Generation IV Structural Materials R&D

- Irradiation
  - Neutron, ion damage studies
- High temperature
  - Creep testing
- Corrosion
  - Testing in molten salt environments
- Combined environments
  - Irradiation + corrosion (MSR)
  - Corrosion + high temperature creep



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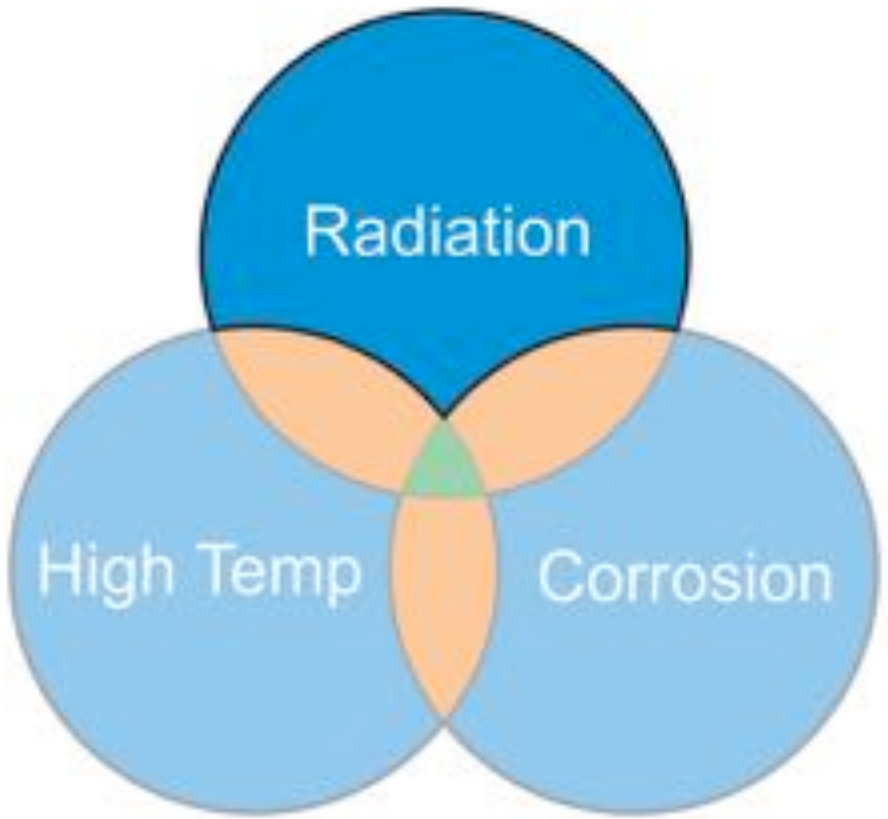


Radiation Damage Test Environments at ANSTO

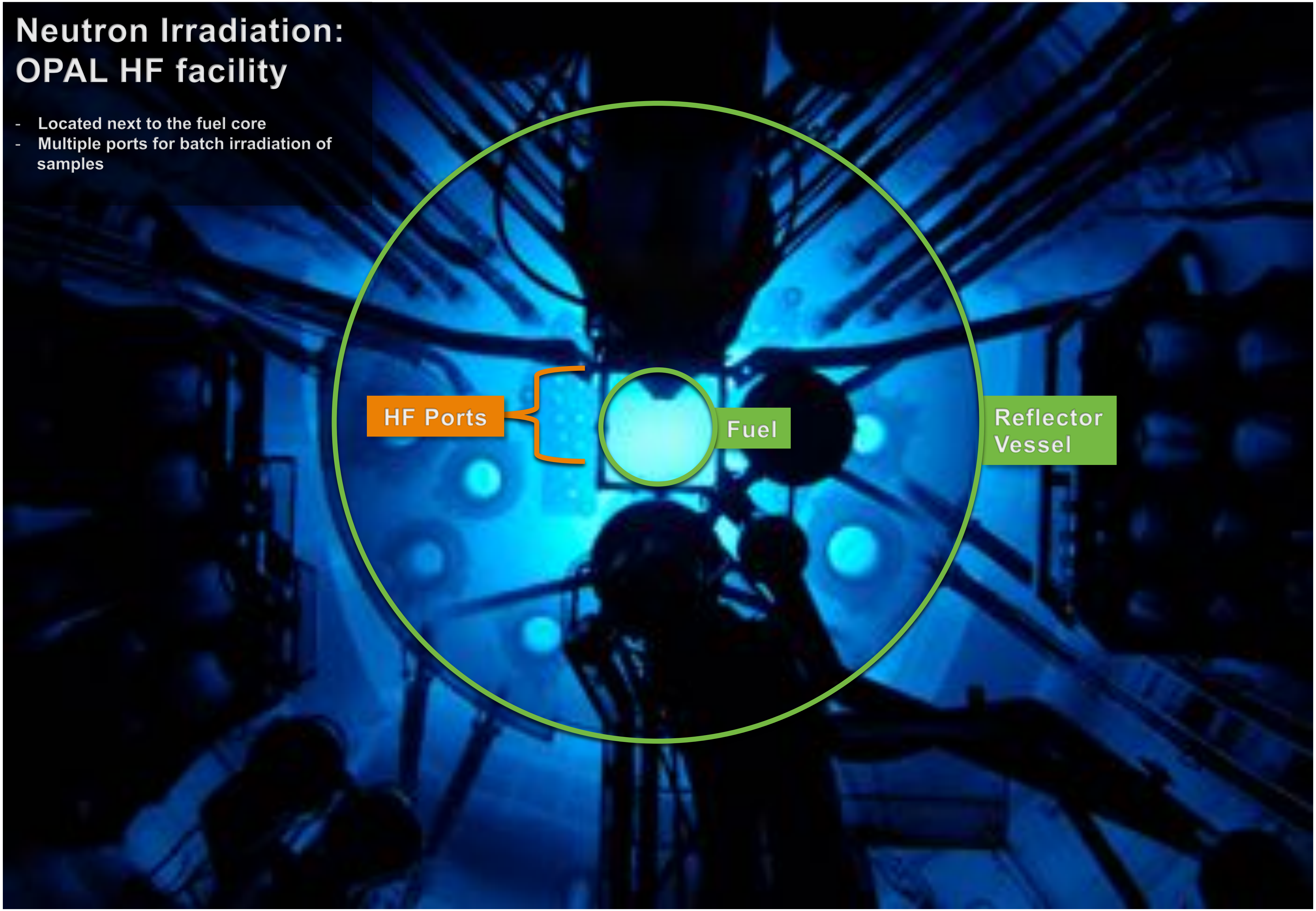

Open Pool Australian Light Water Reactor


Centre for Accelerator Science

- 1MV VEGA accelerator
- 2MV STAR tandetron accelerator
- 6MV SIRIUS tandem accelerator
- 10MV ANTARES tandem accelerator
  - Energies from < 1 MeV to 100 MeV

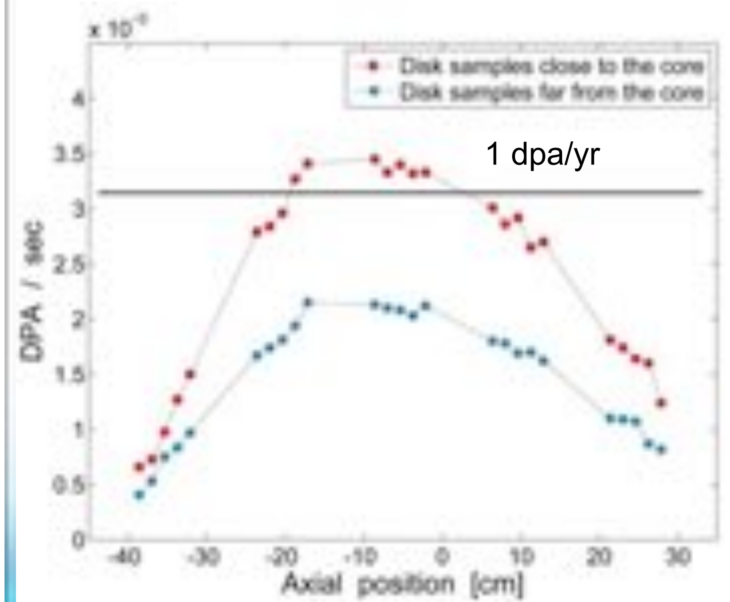
Neutron Irradiation: OPAL HF facility

- Located next to the fuel core
- Multiple ports for batch irradiation of samples



Neutron Irradiation Specimens

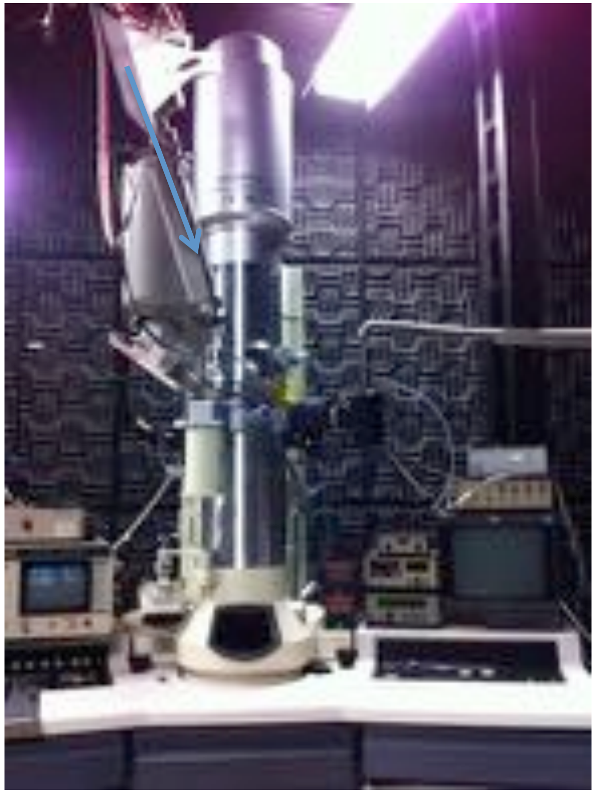
- Miniature dogbone samples
- Small punch discs
- Compact tension discs
- Quarter-size Charpy samples
- Corrosion samples



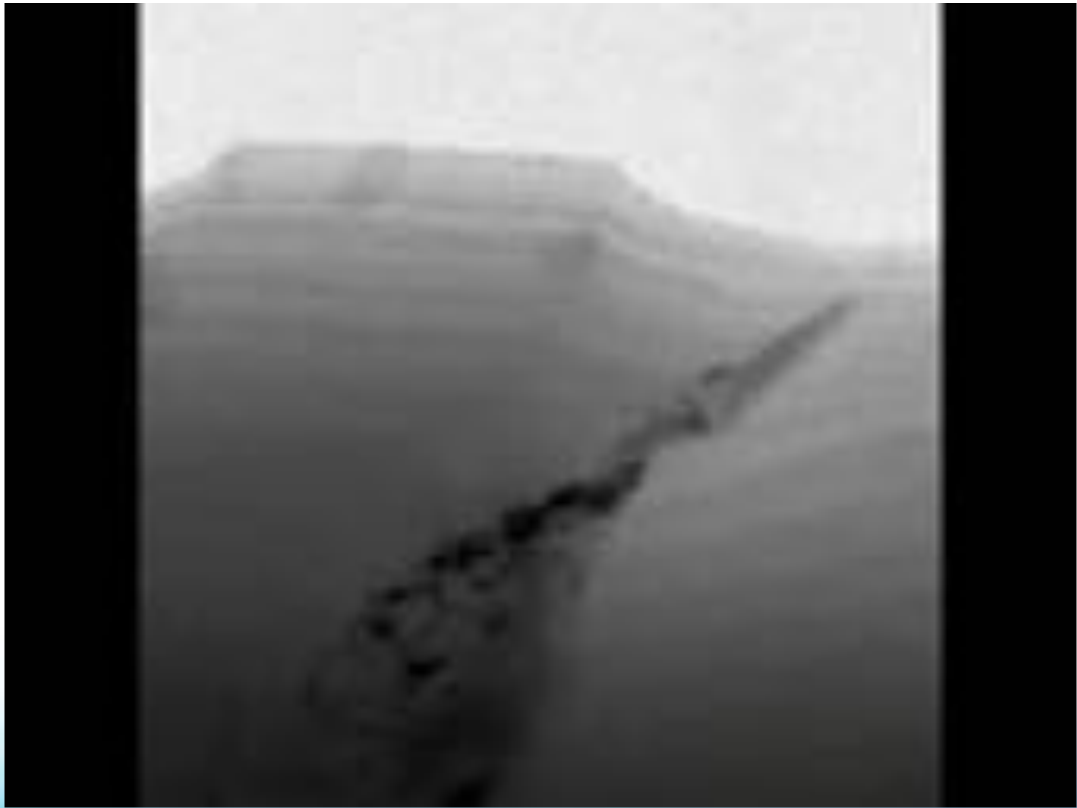
In-situ 1 MeV Kr^+ ion irradiation at RT & 450 ^oC

In-situ irradiation experiments were performed on TEM thinned (~ 100 nm) Ni-Mo-Cr-Fe alloys using 1 MeV Kr^+ ions at room temperature (25 ^oC) and 450 ^oC up to to 100 dpa (1.77×10^{18} ions/cm^2). [110] zone axis.

Post-irradiation analysis undertaken using JEOL 2010F & 2200FS TEMs.



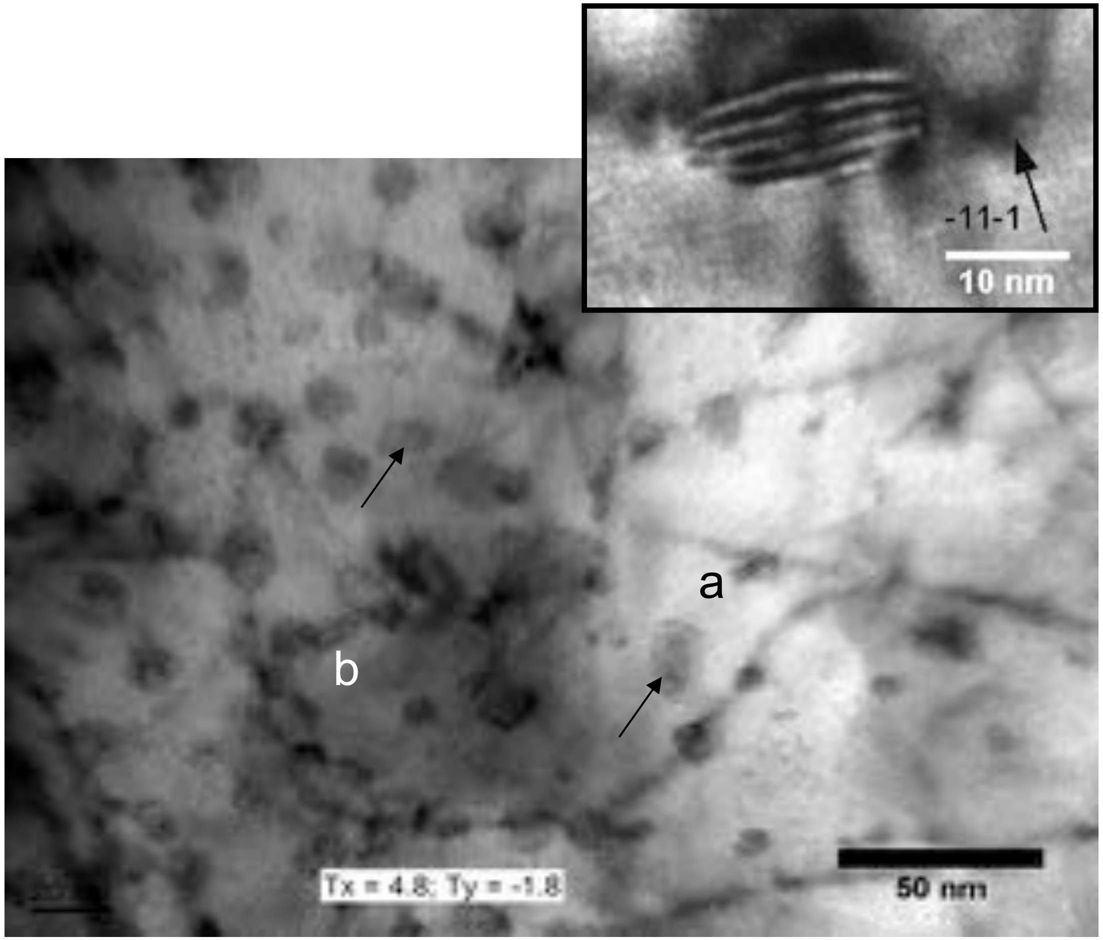
Argonne National Laboratory Hitachi H9000NAR IVEM operated at 200 kV

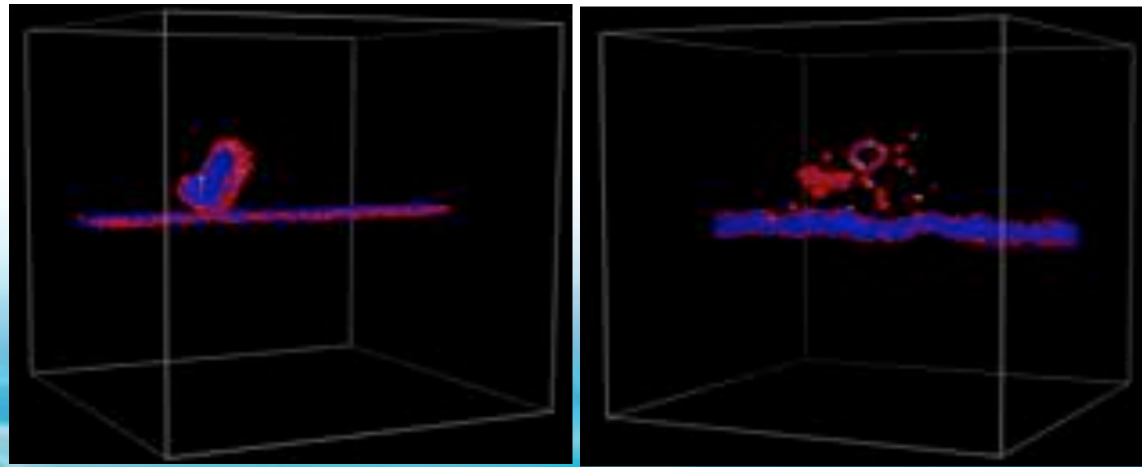


***Microstructural Evolution of an Ion Irradiated Ni-Mo-Cr-Fe Alloy at Elevated Temperatures. Materials Transactions*, 2014, <u>55</u>, pp. 428-433.**

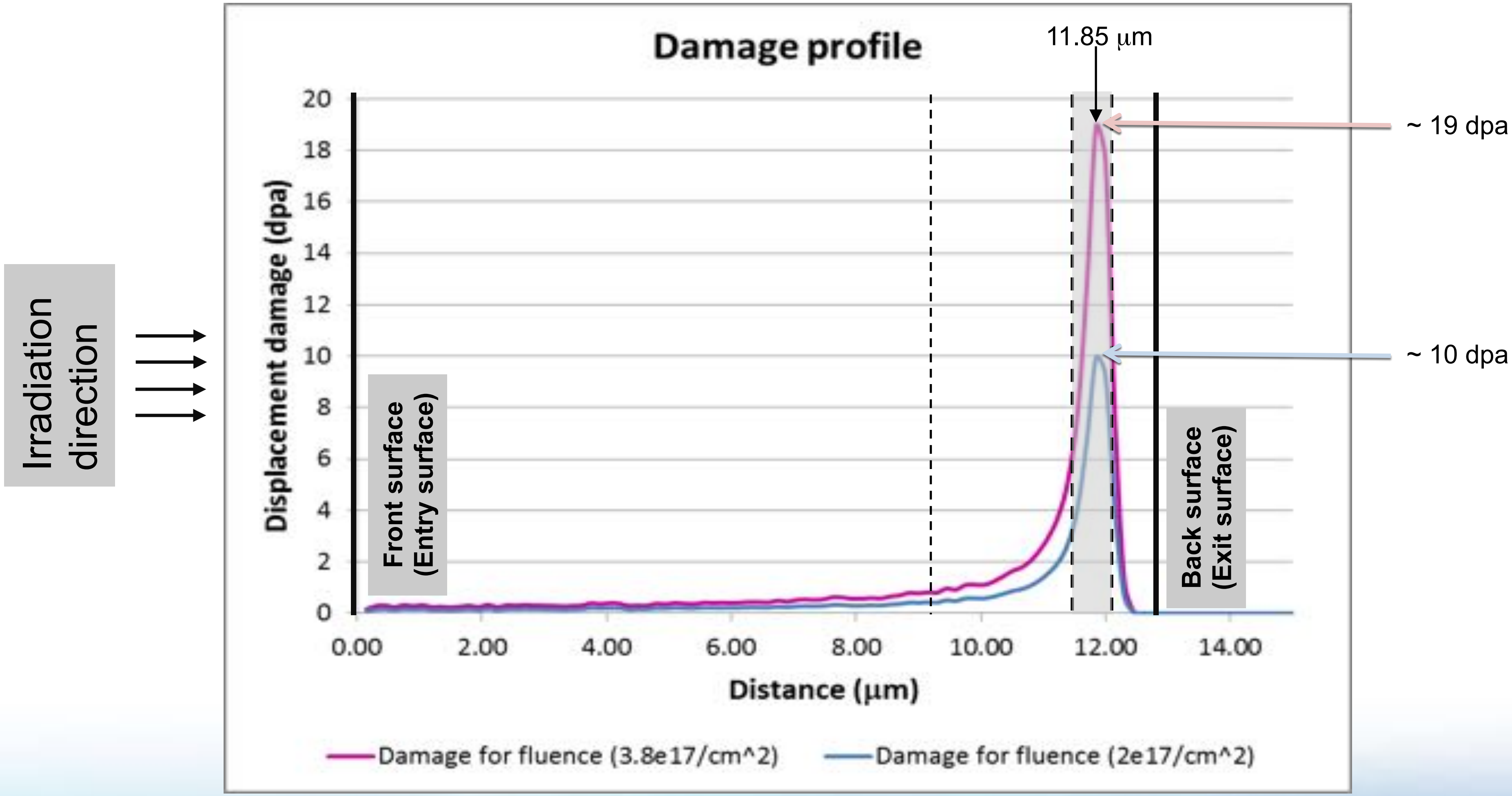
Dislocation Structure

- In-situ 1 MeV Kr^+ ion irradiation at 450°C
- Post Facto TEM analysis
- High densities of both faulted and un-faulted loops observed
- Analysis shows they are un-faulted ½⟨110⟩ SIA loops and faulted 1/3⟨111⟩ vacancy Frank loops
- MD simulations of defects near ½⟨110⟩ edge dislocations suggests that redistribution of displaced atoms from the cascade region towards the dislocation core may result in dislocation climb so remaining vacancies may partially collapse and form 1/3⟨111⟩ faulted vacancy Frank loops nearby.



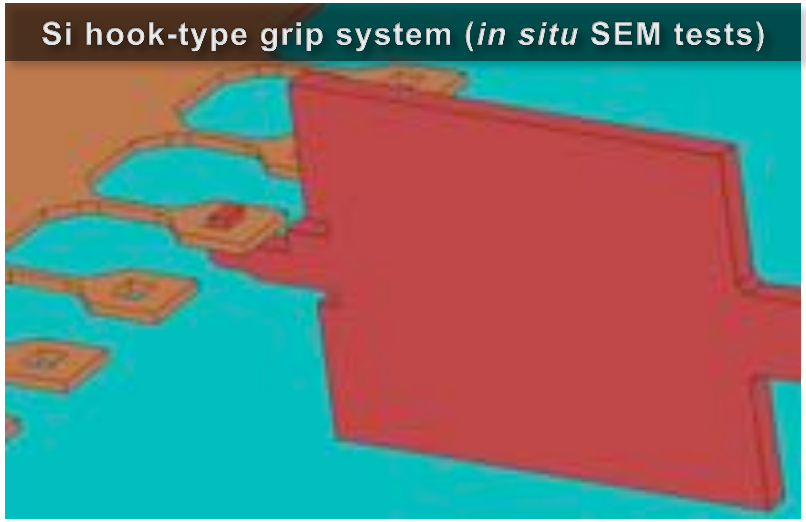


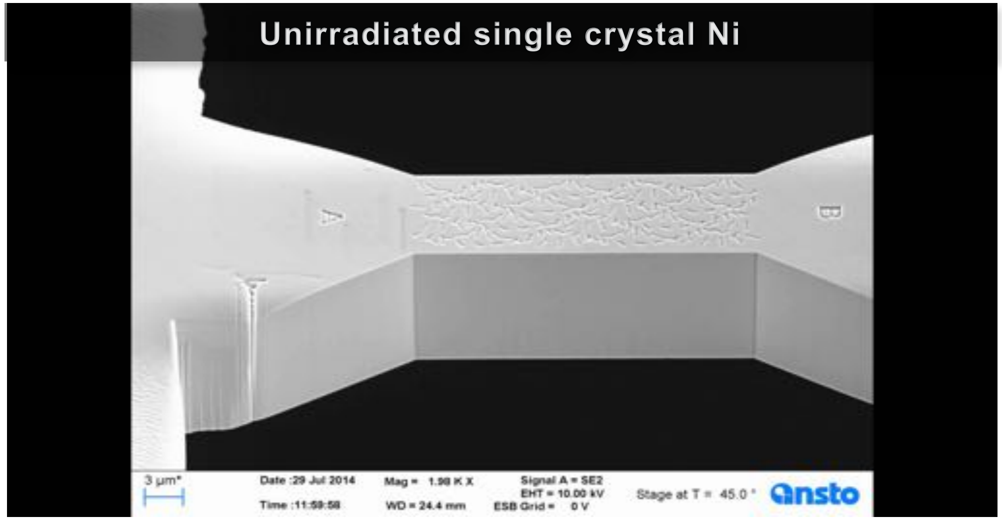
Single Crystal Ni, He+ Ion Irradiation

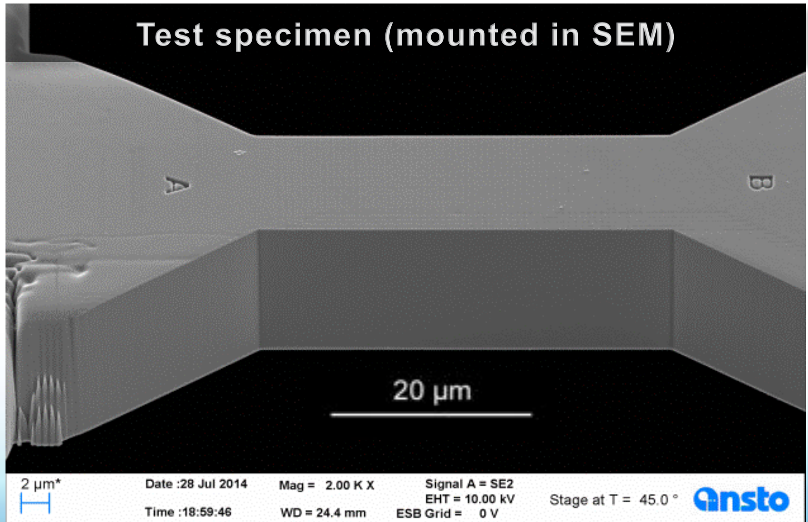


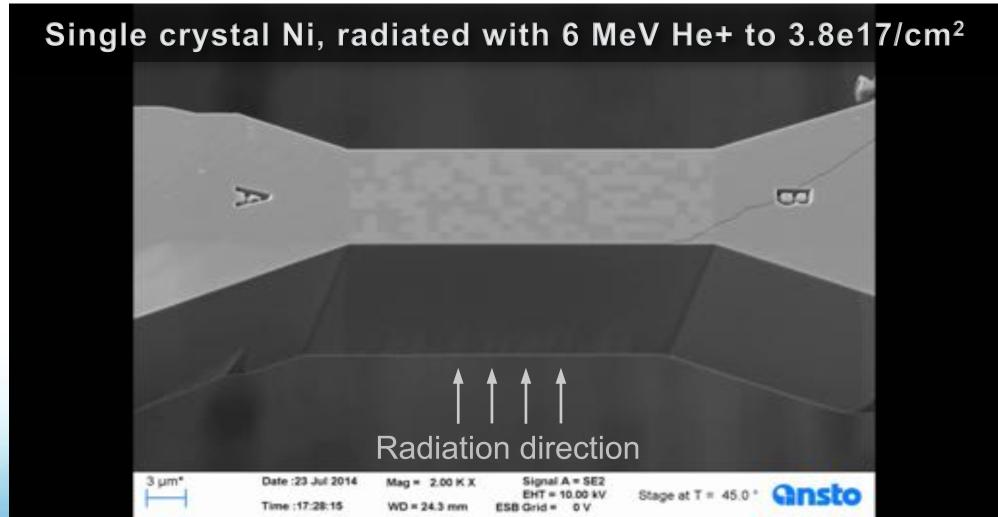
SRIM (The Stopping and Range of Ions in Matter) estimates for He+ irradiation of Ni

Micromechanical Tensile Testing



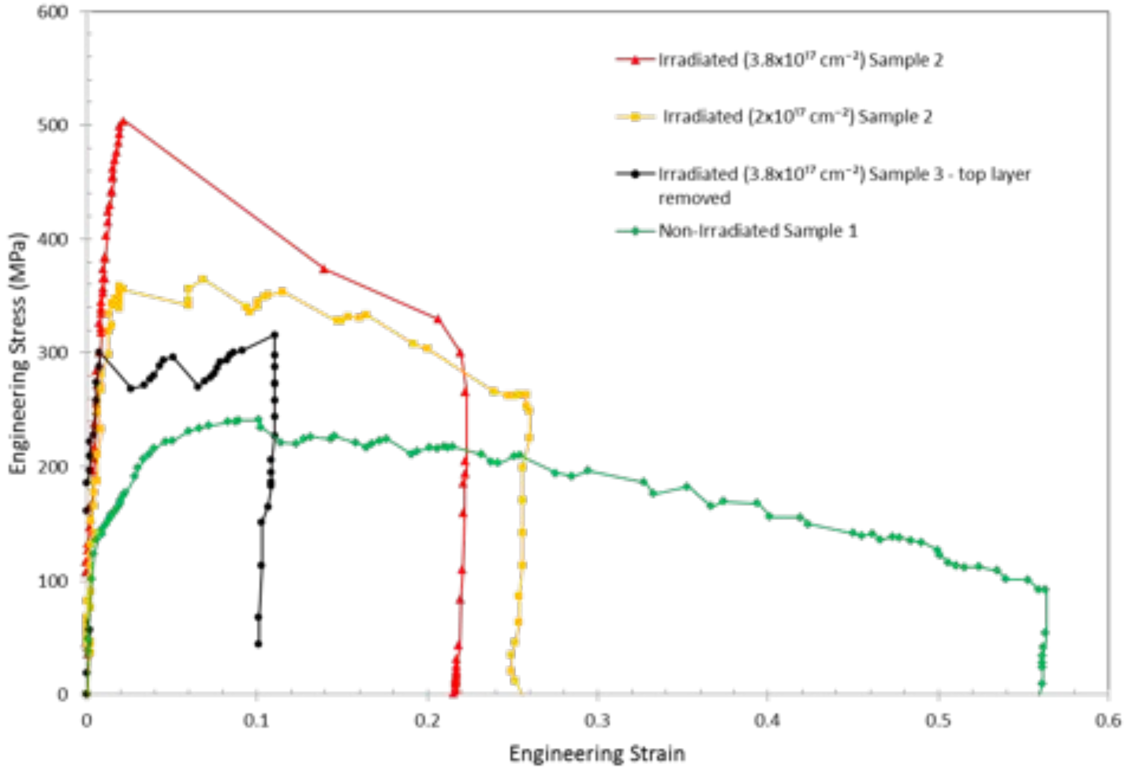


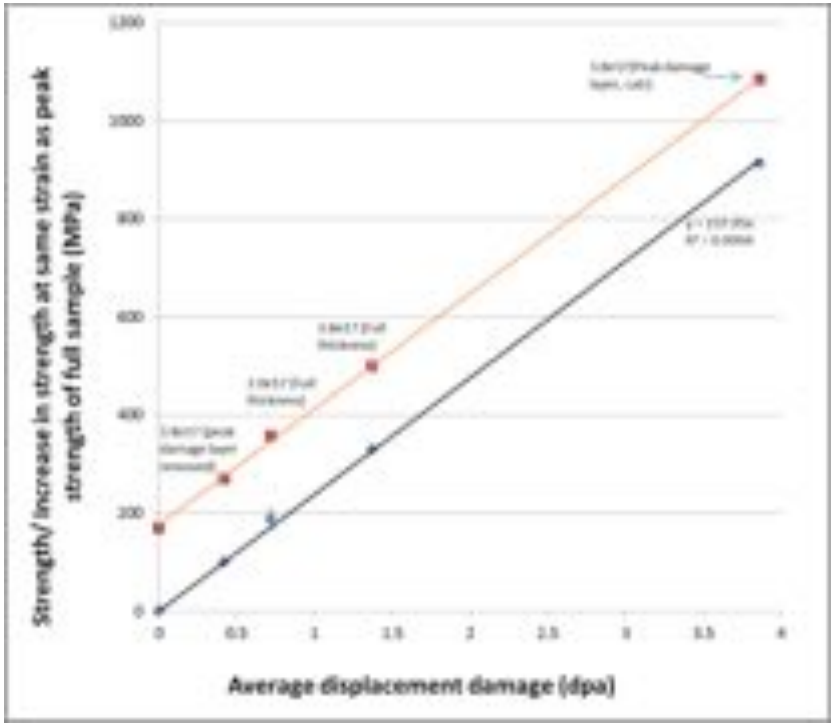




***In situ* micro tensile testing of He^{+2} ion irradiated and implanted single crystal nickel film.**
***Acta Materialia*, 2015, <u>100</u>, pp. 147-154.**

Trends in Radiation Strengthening with Dose





- Ion irradiation provides useful qualitative information
- Full-sample performance does not explicitly consider localised damage

In situ micro tensile testing of He^{+2} ion irradiated and implanted single crystal nickel film. *Acta Materialia*, 2015, 100, pp. 147-154.

Generation IV Structural Materials R&D

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  - Testing in molten salt environments
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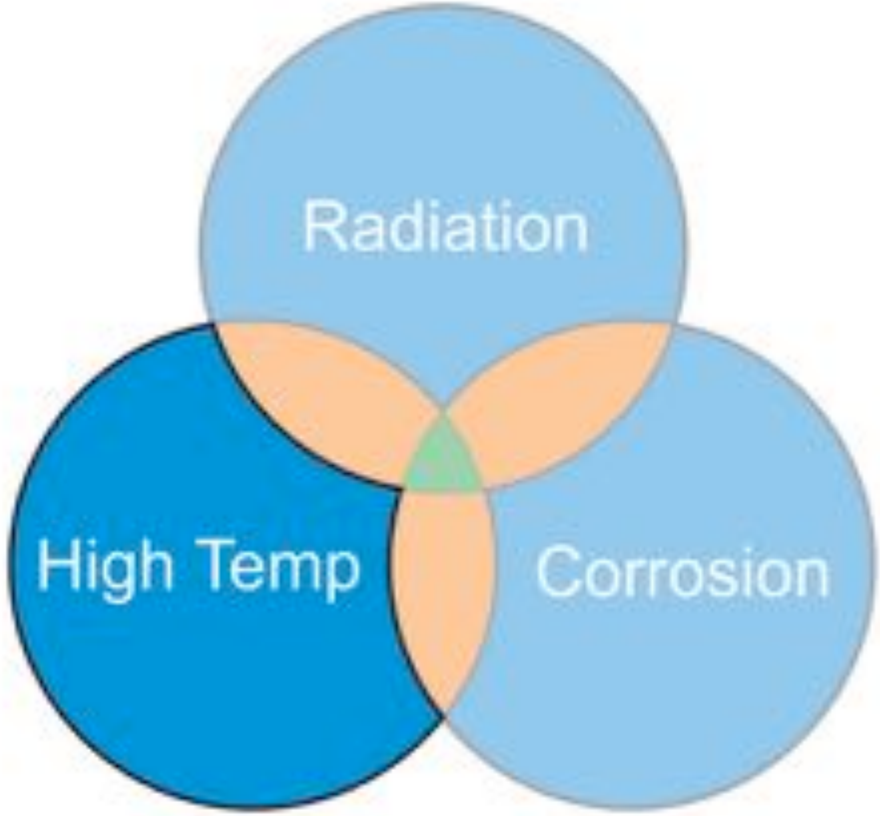
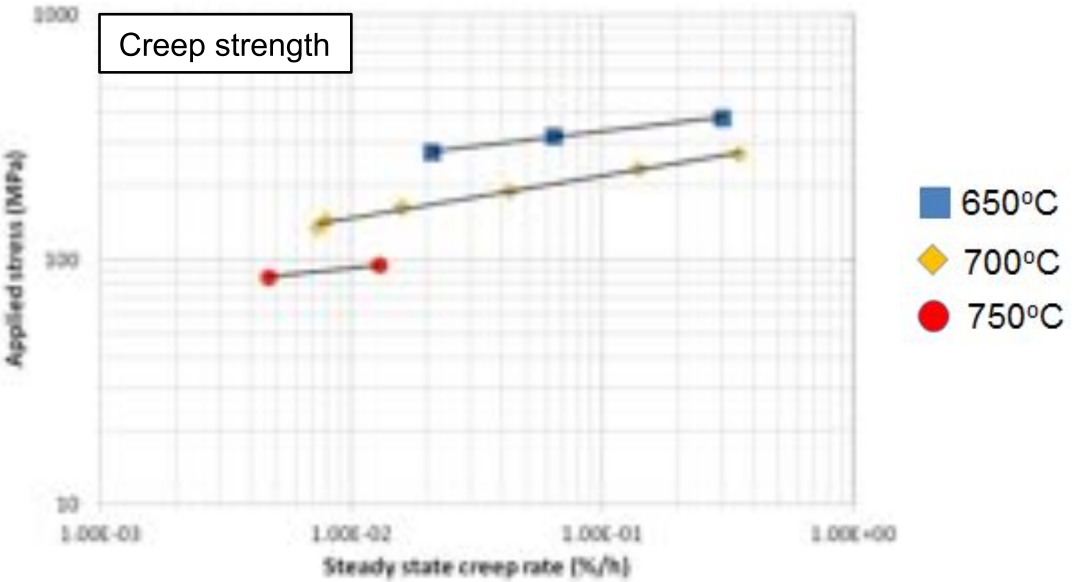
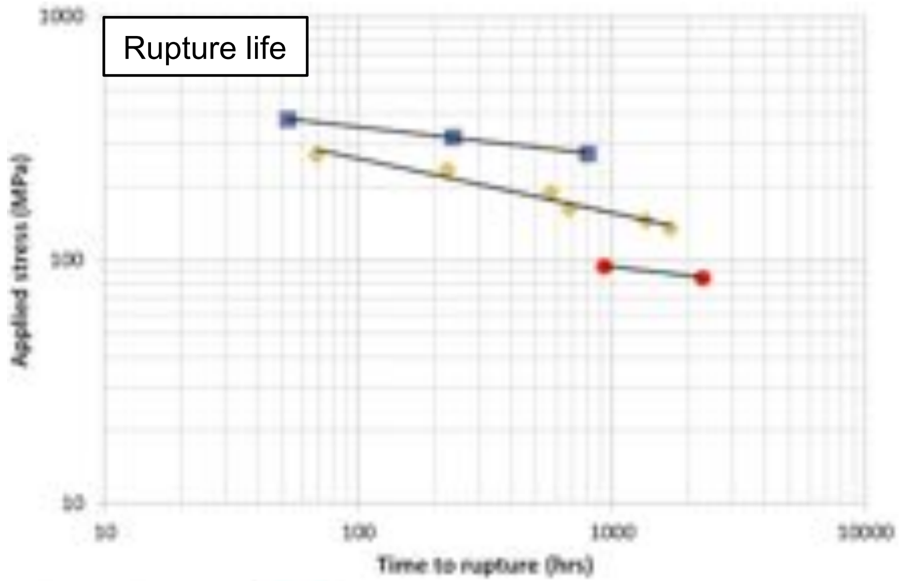
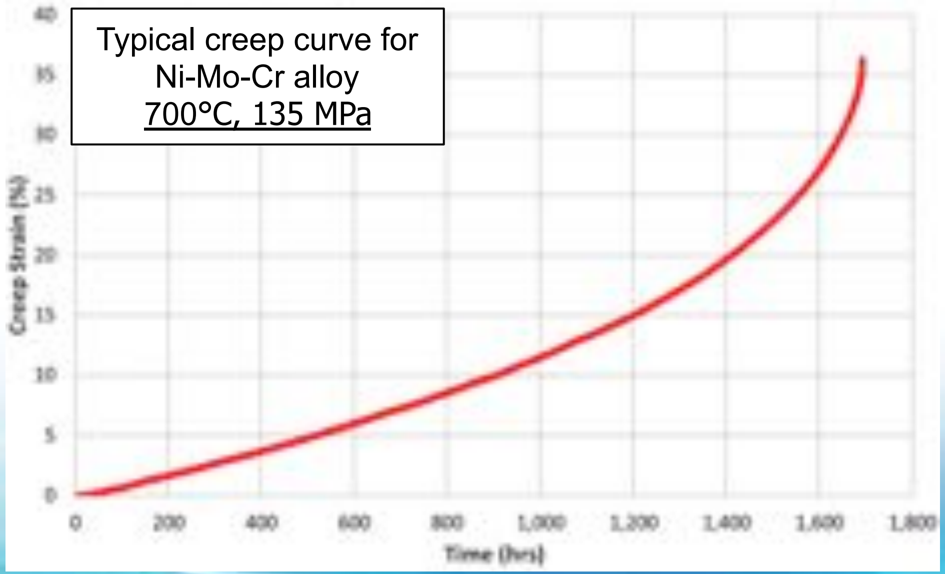
  - Corrosion + high temperature
  - Irradiation under high temperature



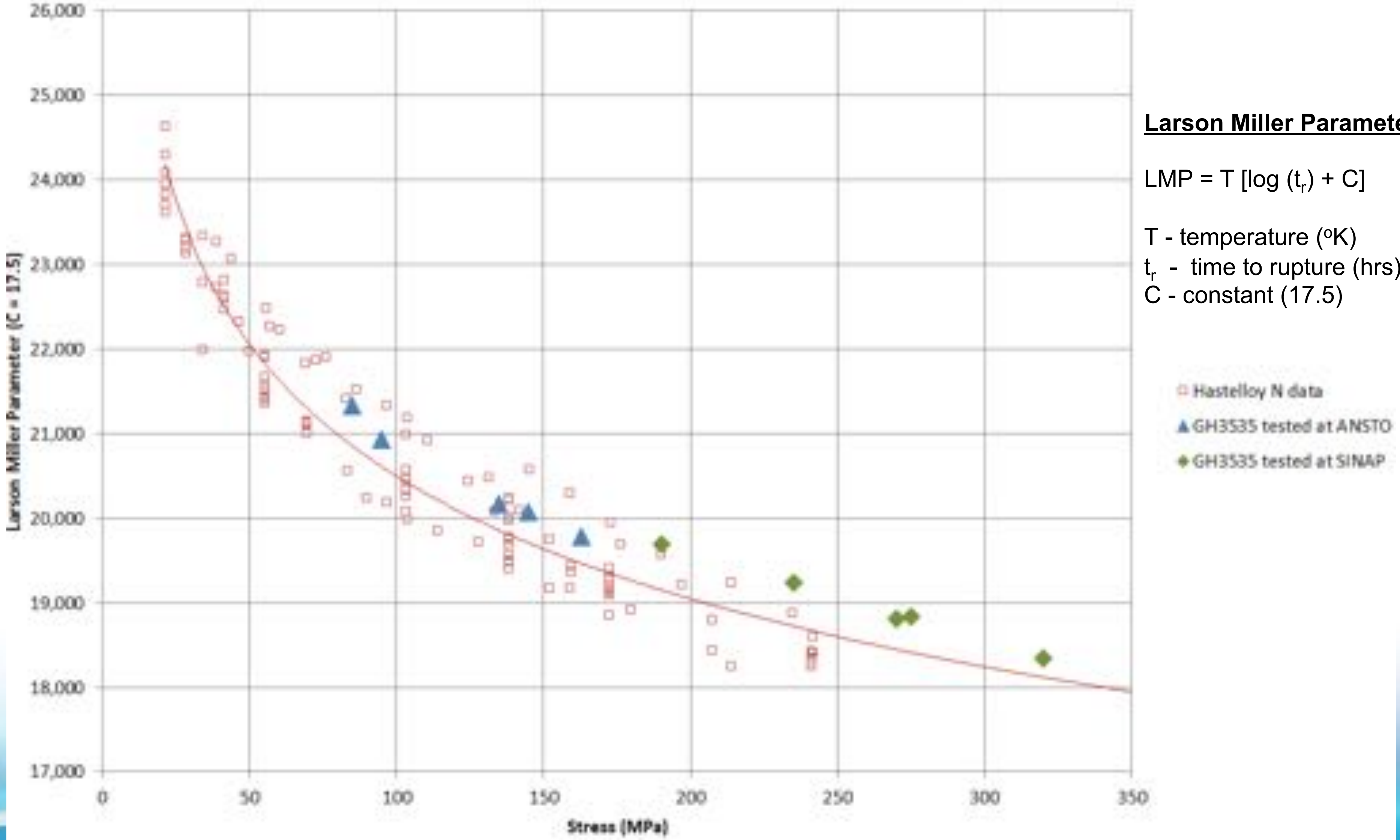
Creep testing GH3535







GH3535 vs Hastelloy N

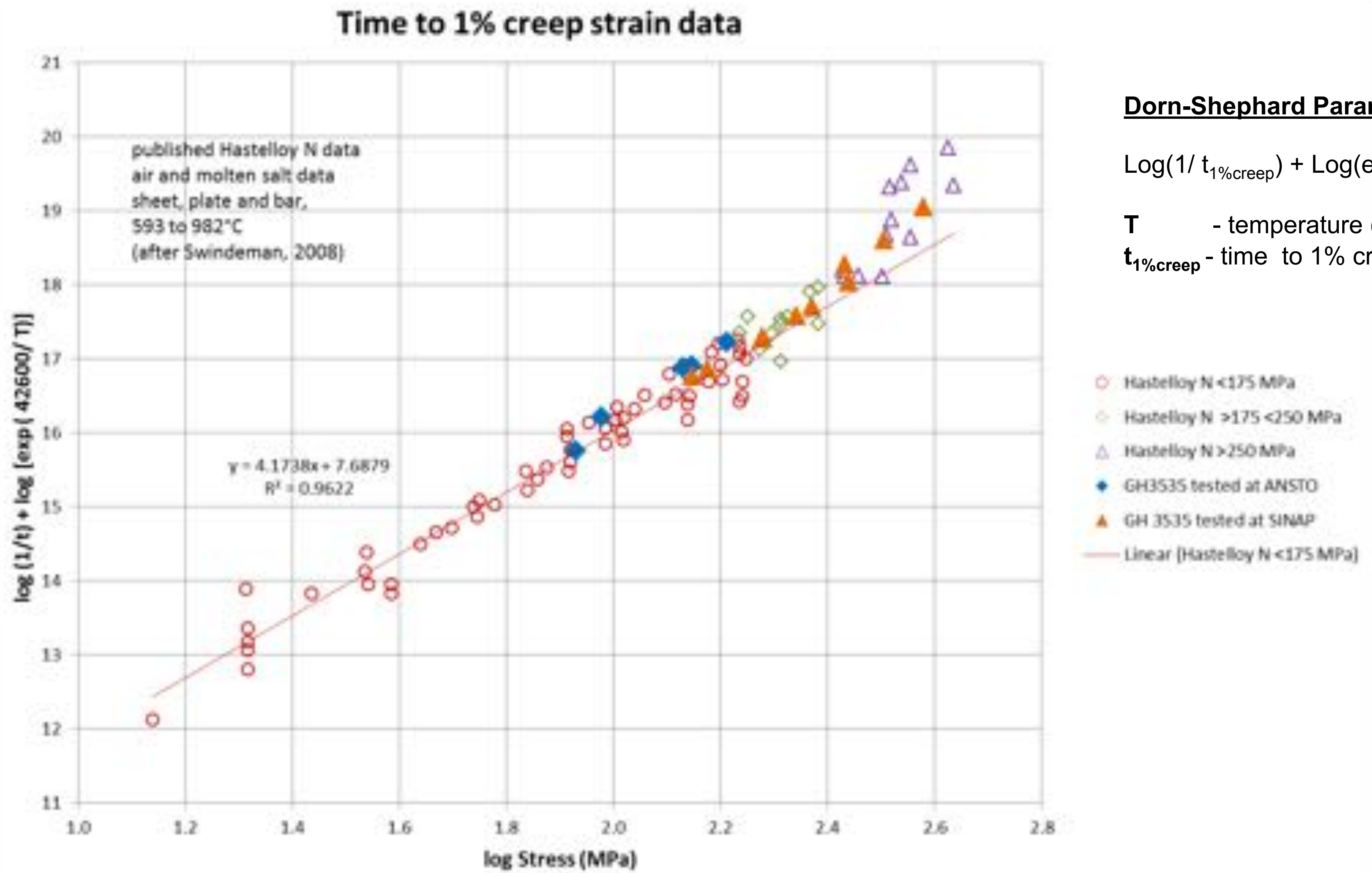


Larson Miller Parameter (LMP)

$LMP = T [\log (t_r) + C]$

T - temperature (°K)
t_r - time to rupture (hrs)
C - constant (17.5)

GH3535 vs Hastelloy N



Dorn-Shephard Parameter for 1% creep

$\mathrm{Log}(1/ t_{1\%creep}) + \mathrm{Log}(\exp(42600/T))$

T - temperature (^{o}K)

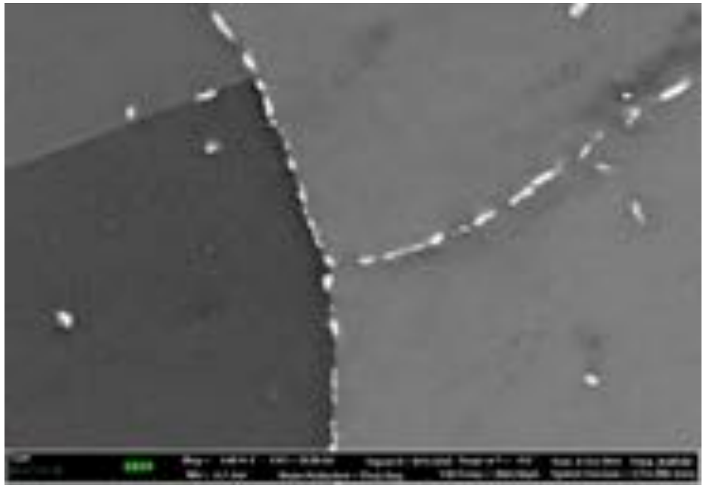
$\mathbf{t_{1\%creep}}$ - time to 1% creep strain (hrs)

GH3535 Creep coclusions

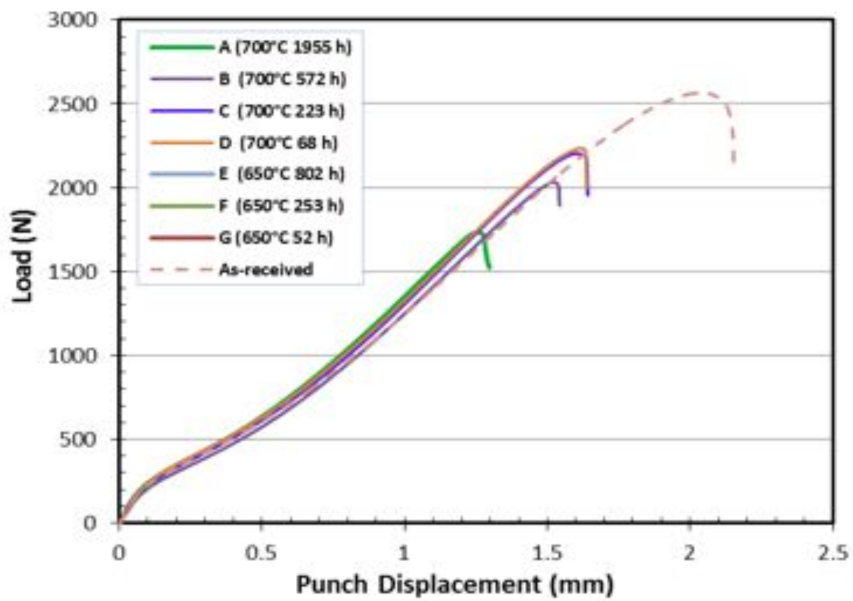
- GH3535 alloy creep tested at 650, 700, and 750 ^{o}C at 85-320 MPa.
- Alloy displayed no primary creep and good creep ductility (above 30%.)
- Using minimum creep rates the calculatde stress exponent is n = 5-6 so dislocation climb is the main creep mechanism
- Formation of dislocation networks and sub-grains confirmed by TEM and EBSD analysis.
- The Dorn Shepard and Larson Miller master curves were derived to 1% creep strain and creep rupture respectively.
- Using ASME BPVC guidelines the maximum allowable design stress at 700 ^{o}C is 35 MPa.
- There is substantial second phase particle precipitation during creep.

Thermal ageing of Ni-based alloy GH3535

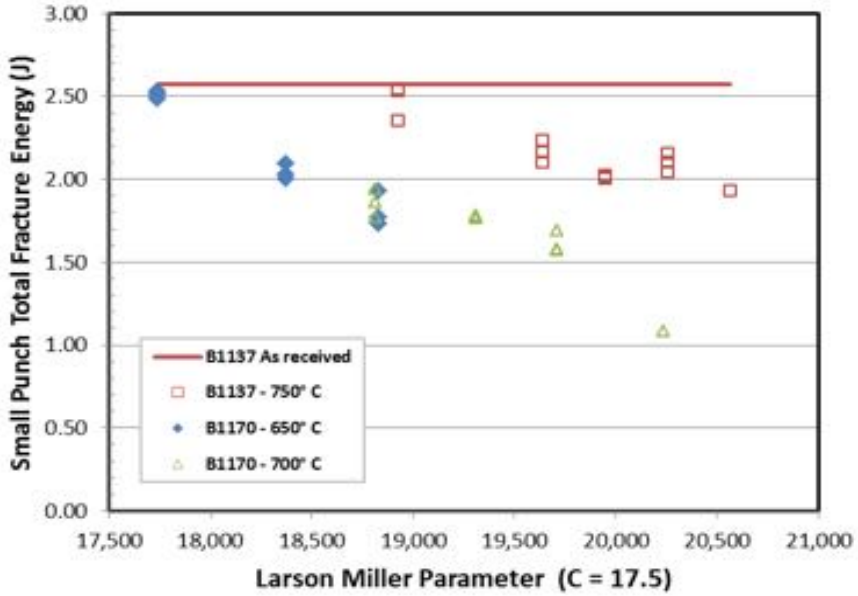
- Thermal ageing of GH3535 is being studied at 650, 700 and 750°C by detailed microstructural characterisation of primary and secondary precipitates.
- Ageing times up to 2,000 hours.
- Mechanical properties are being evaluated by small punch testing due to limited sample size.
- RT small punch fracture energy decreases with longer thermal ageing time.



Secondary precipitation at grain boundaries after 1,366 hours at 700°C in heat B1137



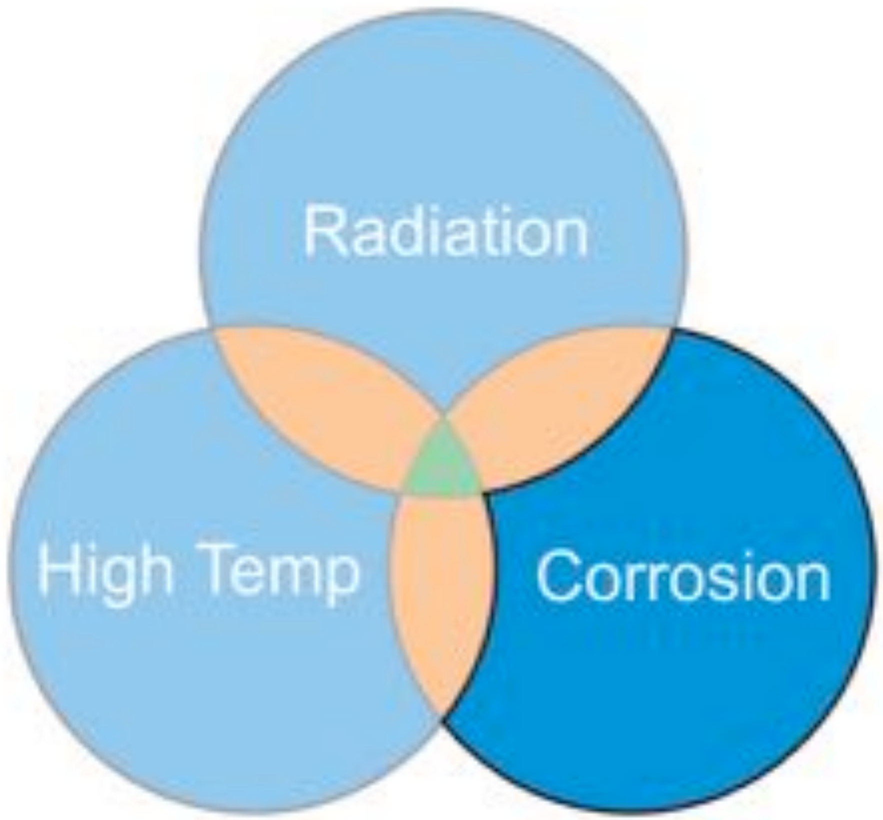
Small punch test results of heat B1170



Small punch test results of heats B1137 and B1170

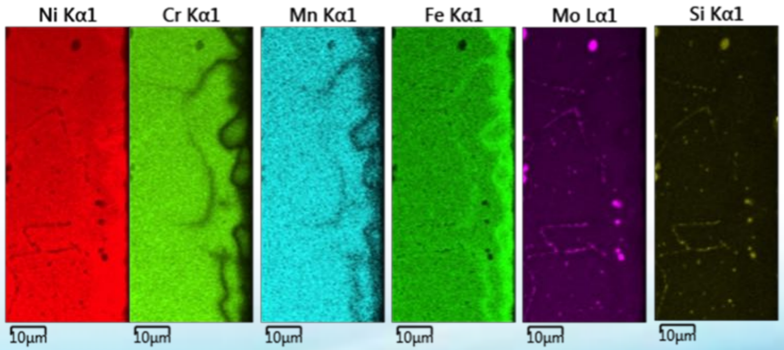
Generation IV Structural Materials R&D

- Irradiation
  - Neutron, ion damage studies
- High temperature
  - Creep testing
- Corrosion
  - Testing in molten salt environments
- Combined environments
  - Irradiation + corrosion (MSR)
  - Corrosion + high temperature
  - Irradiation under high temperature



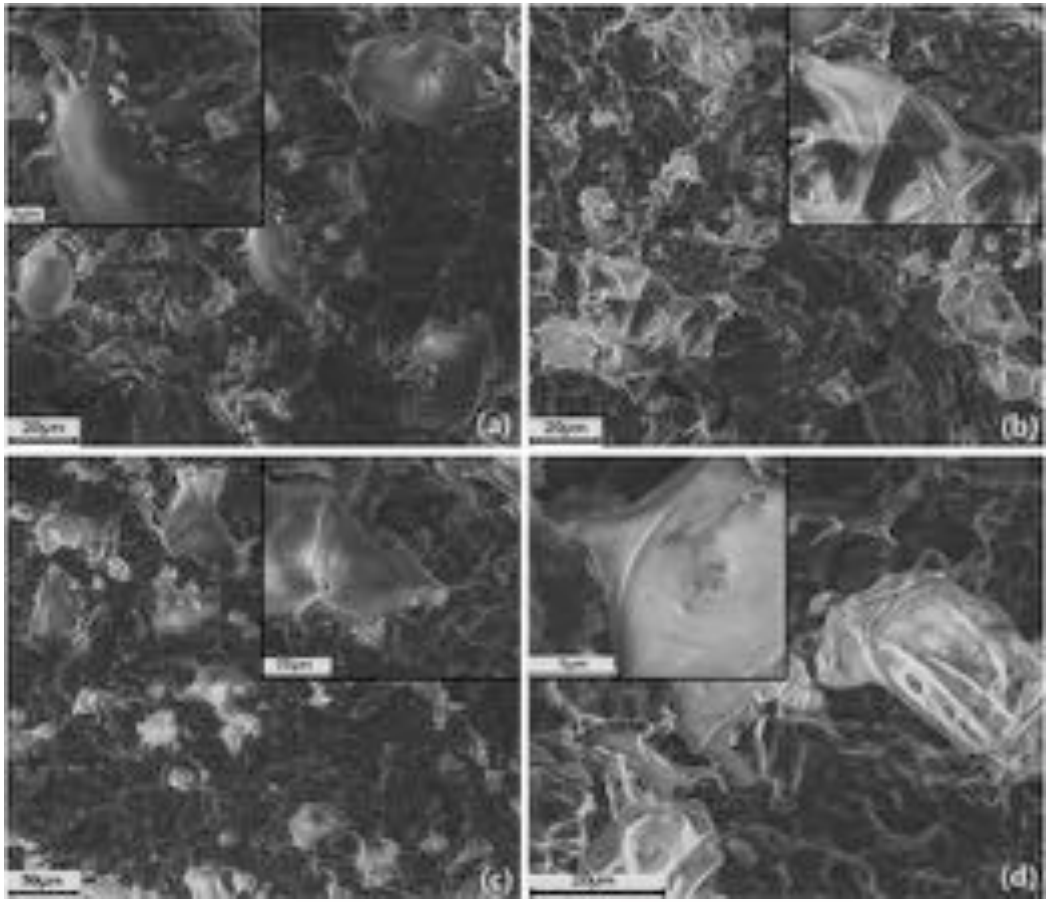
Corrosion Testing

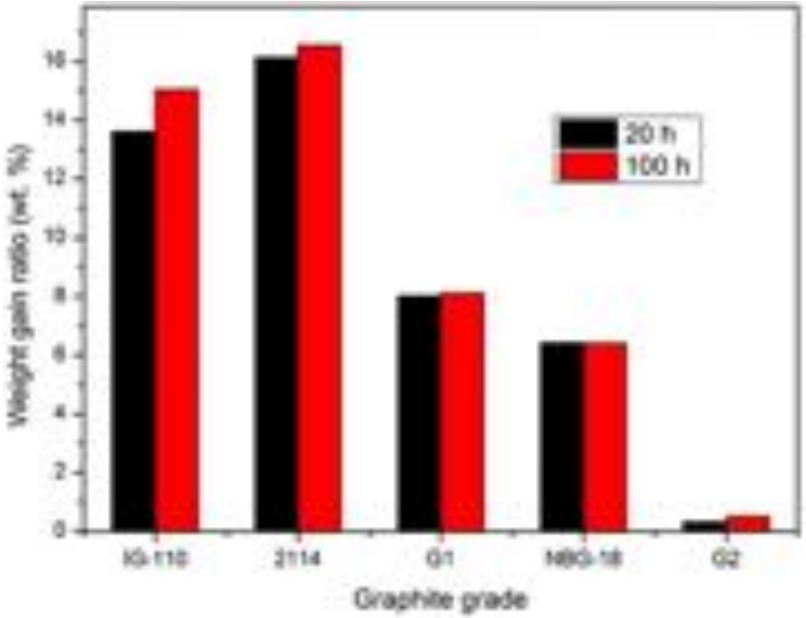
- ANSTO-SINAP JRC (MSR)
  - FLiNaK salt composition
  - Tests conducted at 750ºC for 10, 100 and 200 h
- Materials tested
  - GR 3535
  - Graphite
  - AISI 316

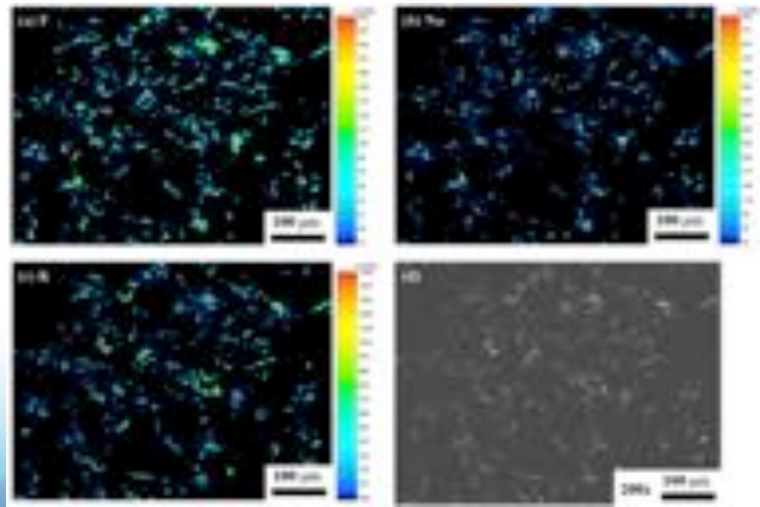


Controlled atmosphere furnace (*above*) with temperature control directly linked to salt temperature; exposed NiCrMoFe is examined via EDS (*left*) to determine surface degradation.

Corrosion/Salt Infiltration into Graphite







- Salt infiltration into various grades of graphite at different pressures (a) 1.0 atm; (b) 1.5 atm; (c) 3.0 atm; and (d) 5.0 atm.
- Location of salt quantified via EDS (*right*) and neutron tomography (DINGO).

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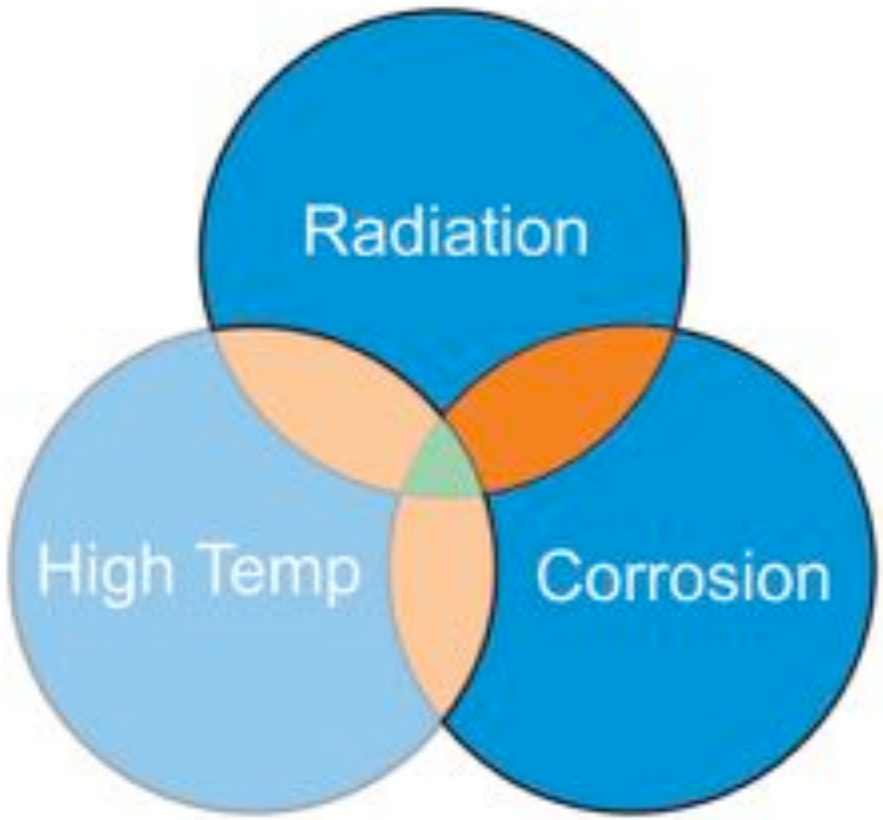
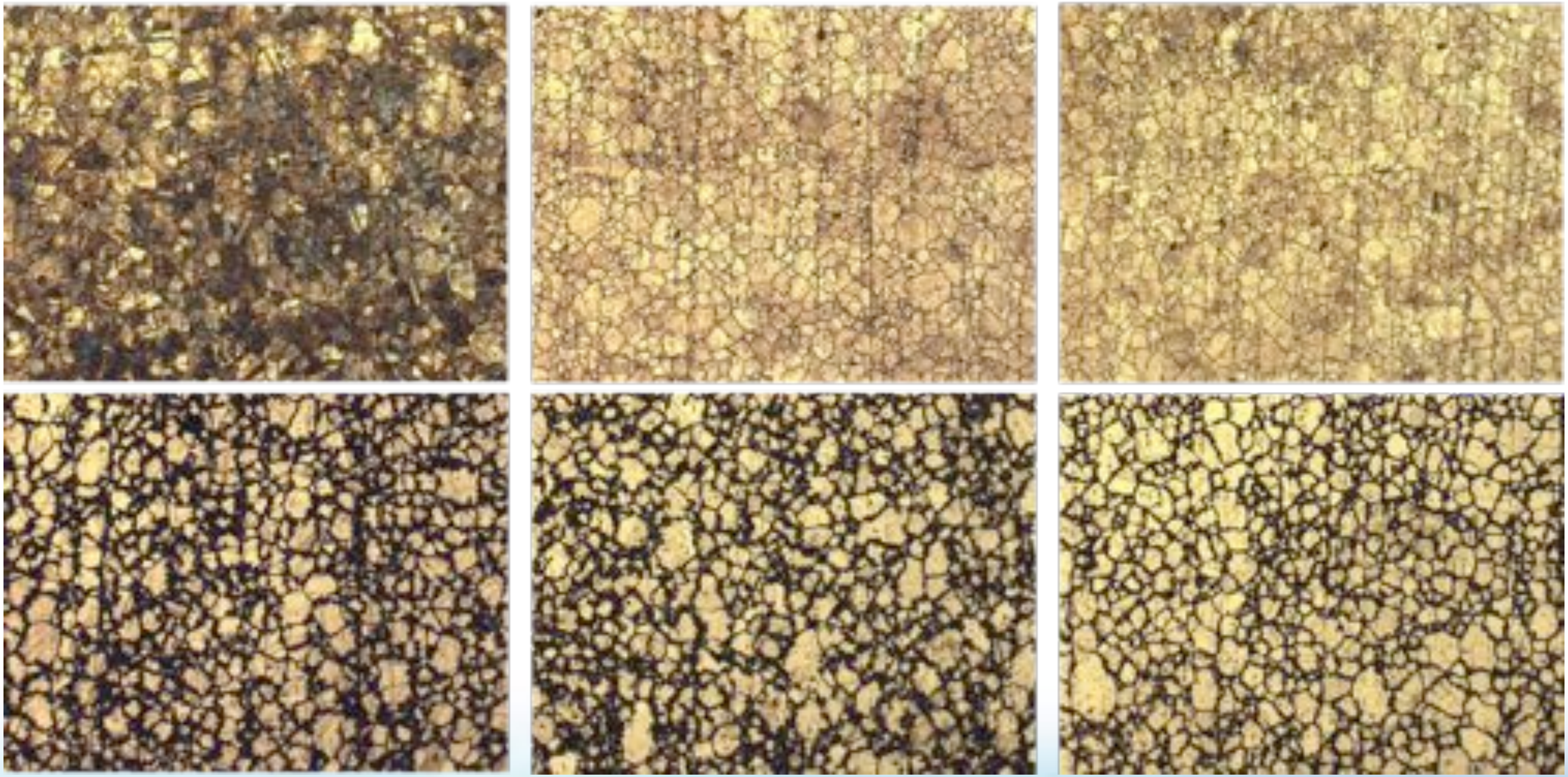


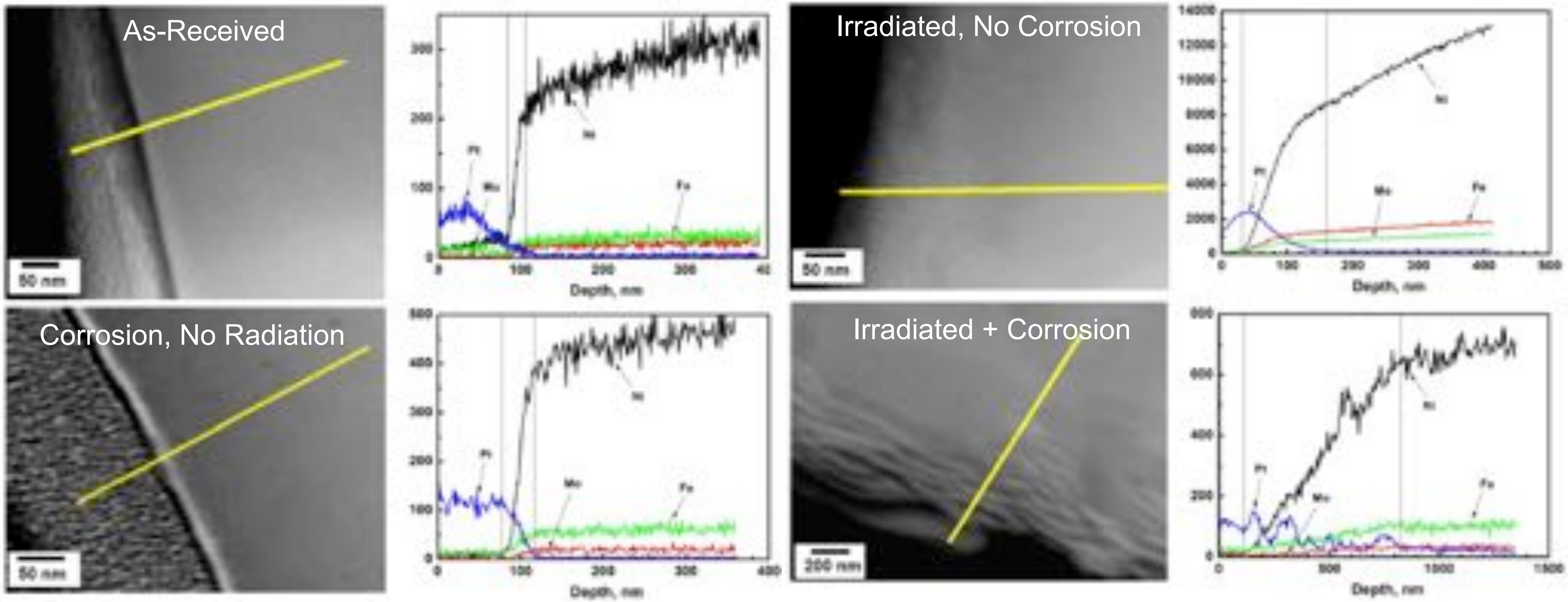
Effect of Ion Irradiation on Corrosion

Salt Only | Salt + 0.5×10^{16} ions/cm^2 He | Salt + 1×10^{17} ions/cm^2 He



GH3535: Vapour etch top figures, salt corrosion bottom figures

Effect of Ion Irradiation on Corrosion

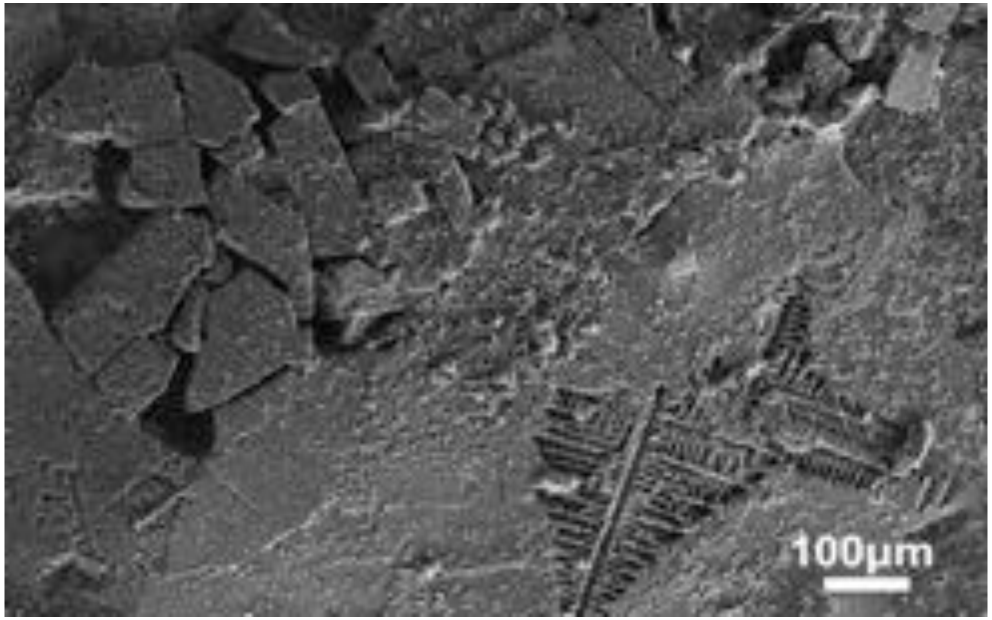


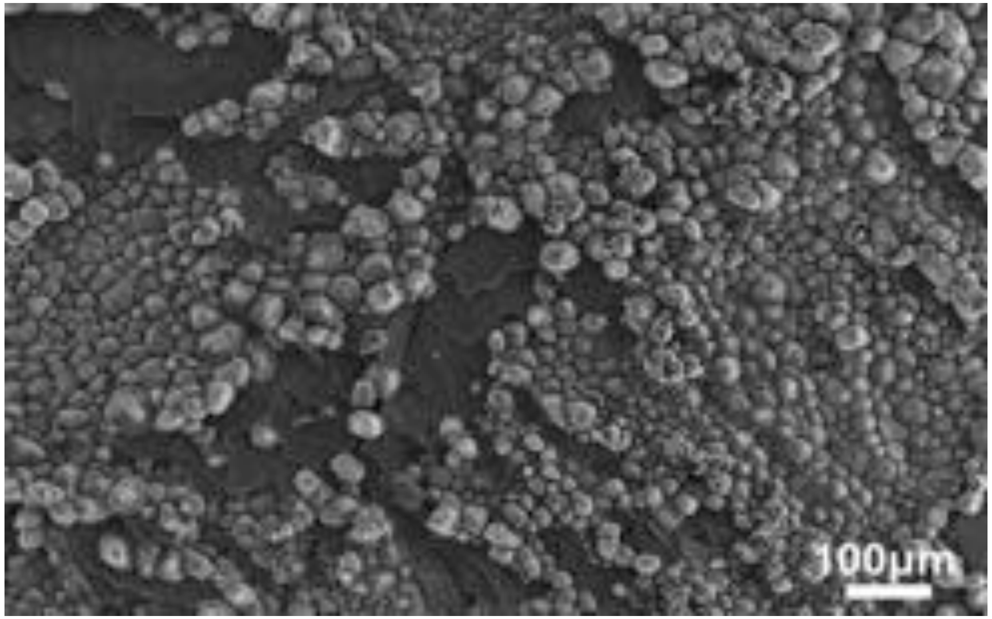
- GR3535 alloy, FLiNaK salt, 10^{17} ions/cm^2 He+
- Helium ion irradiation increases the thickness of the corrosion layer in the irradiated and corroded sample to more 30 times than in the un-irradiated sample.

High-temperature corrosion of helium ion-irradiated Ni-based alloy in fluoride molten salt
***Corrosion Science*, 2015, 91, pp. 1-6.**

Effect of Ion Irradiation on Corrosion

Graphite, FLiNaK salt, 10^{17} ions/cm^2 He+





- Graphite test coupons were irradiated with 30 keV He+ ions
- One section was masked to prevent radiation damage
- The samples exposed to molten FLiNaK salt (150h@750 ºC).
- Clear variations in the corroded structures are visible
- NEXAFS suggests Fluorination of surface

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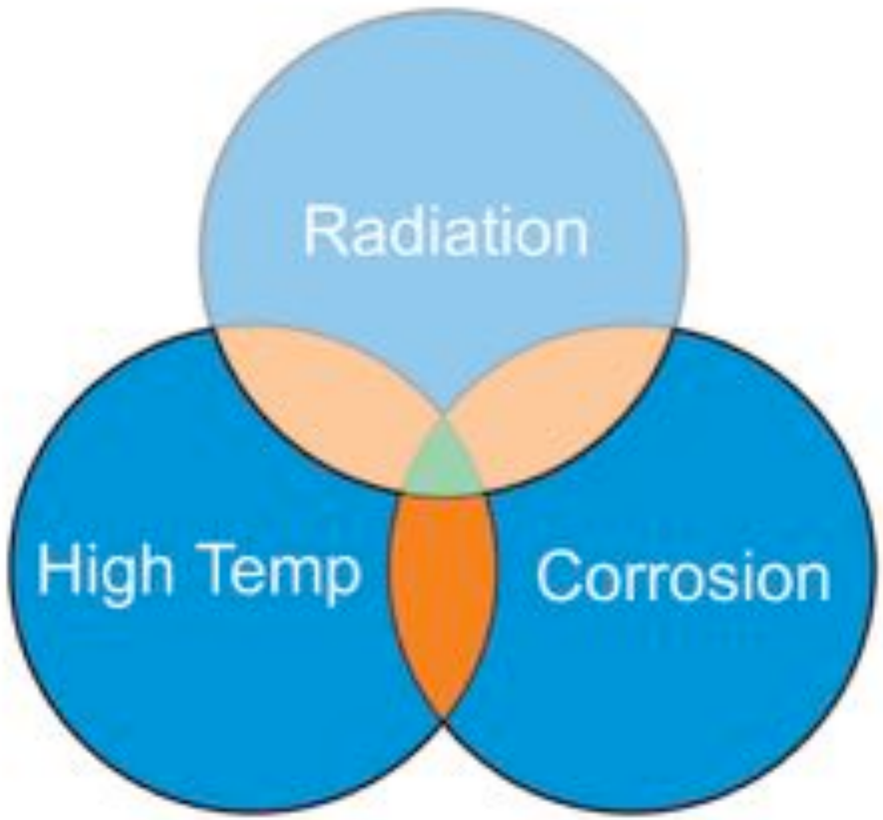
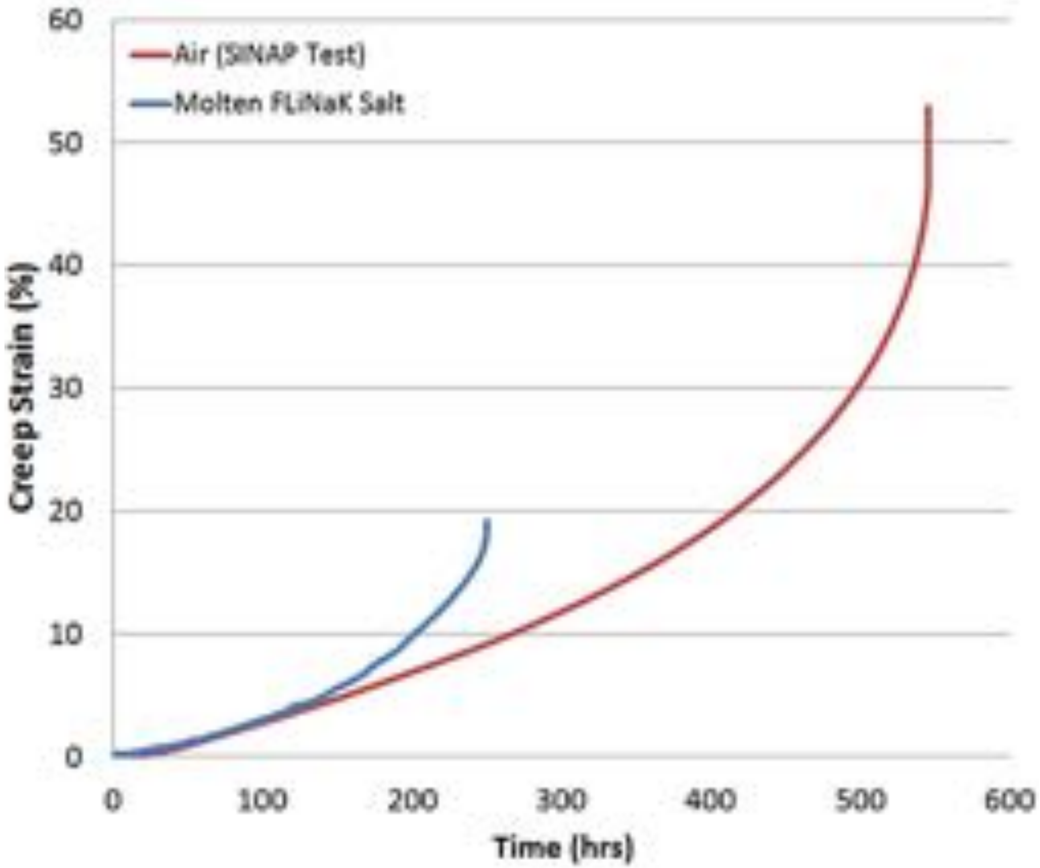
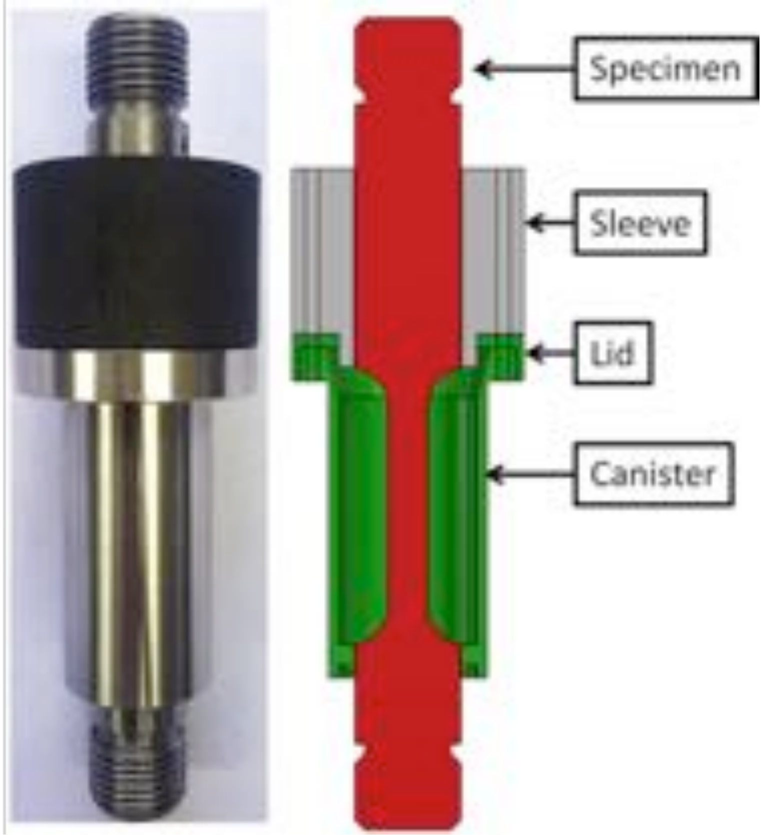


Materials Testing at High Temperatures

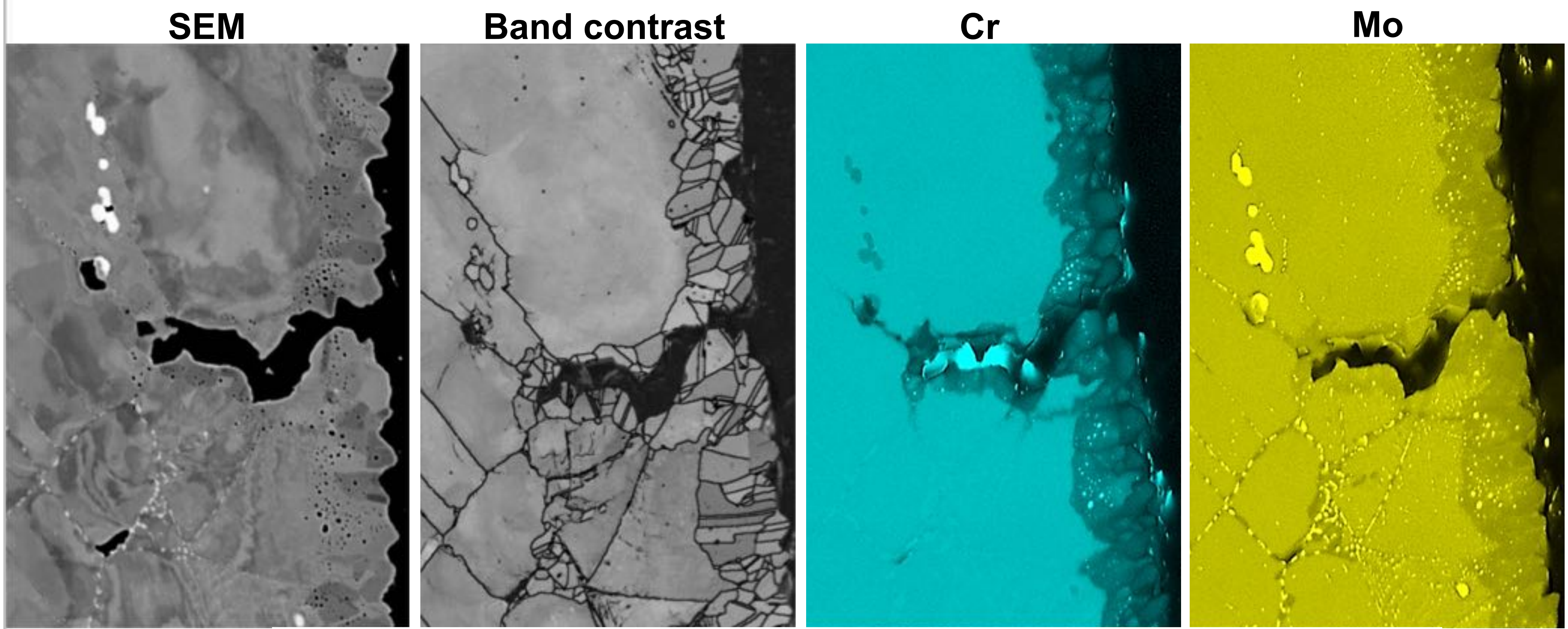




GH3553 creep rupture test (190 MPa @ 700 ºC)

Initial results show significant effect of salt on creep life

GH 3535 Creep testing in molten salt

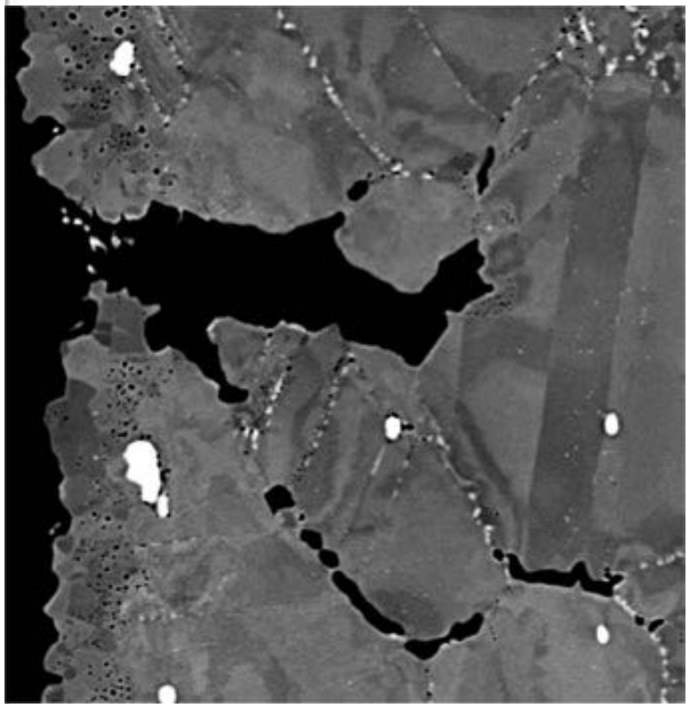


EDS chemical composition (Average wt%)

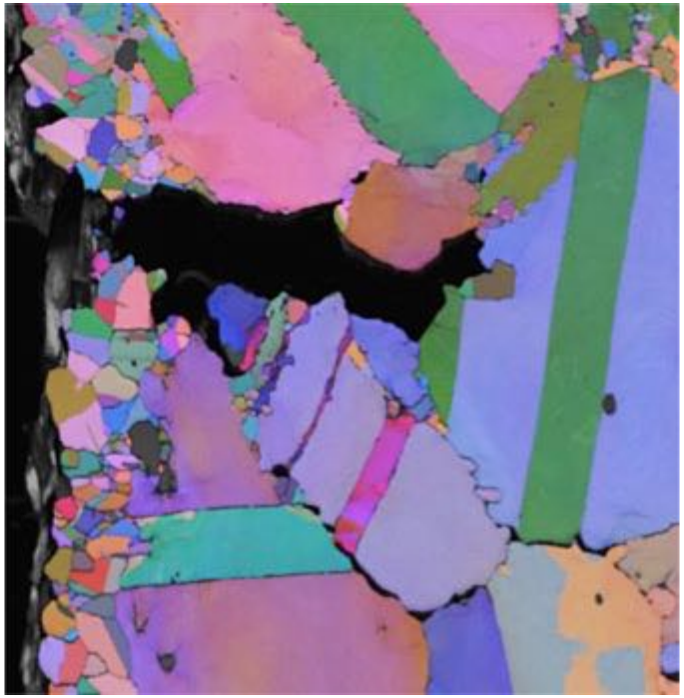
Region	Ni	Mo	Cr	Fe	Mn	Si
Bulk sample	71.1	16.3	7.3	4.1	0.7	0.5
Salt affected regions (st dev)	82.8 (0.5)	12.1 (0.4)	1.5 (0.2)	3.2 (0.2)	0.2 (0)	0.1 (0)

GH 3535 Creep testing in molten salt

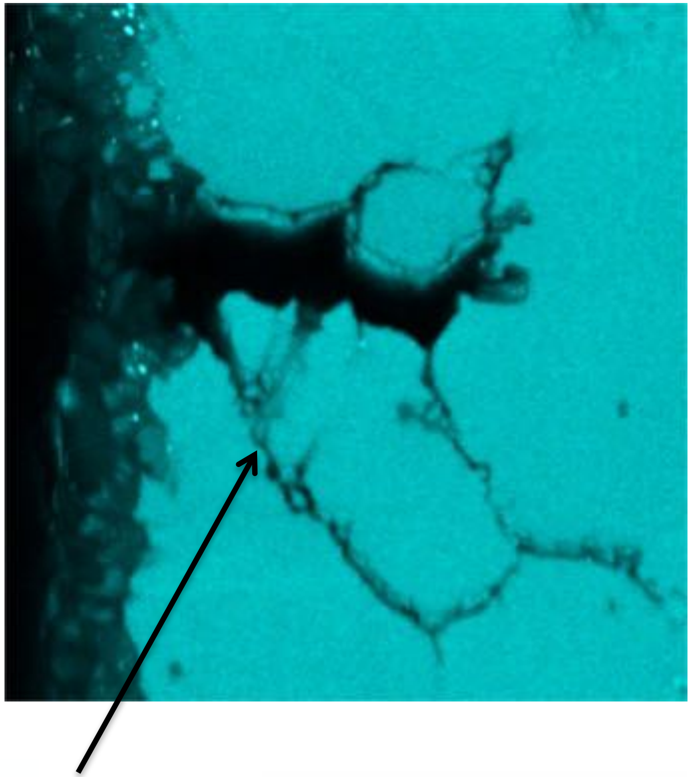
SEM



Euler Map



Cr



20 µm

Evidence of preferential grain boundary attack

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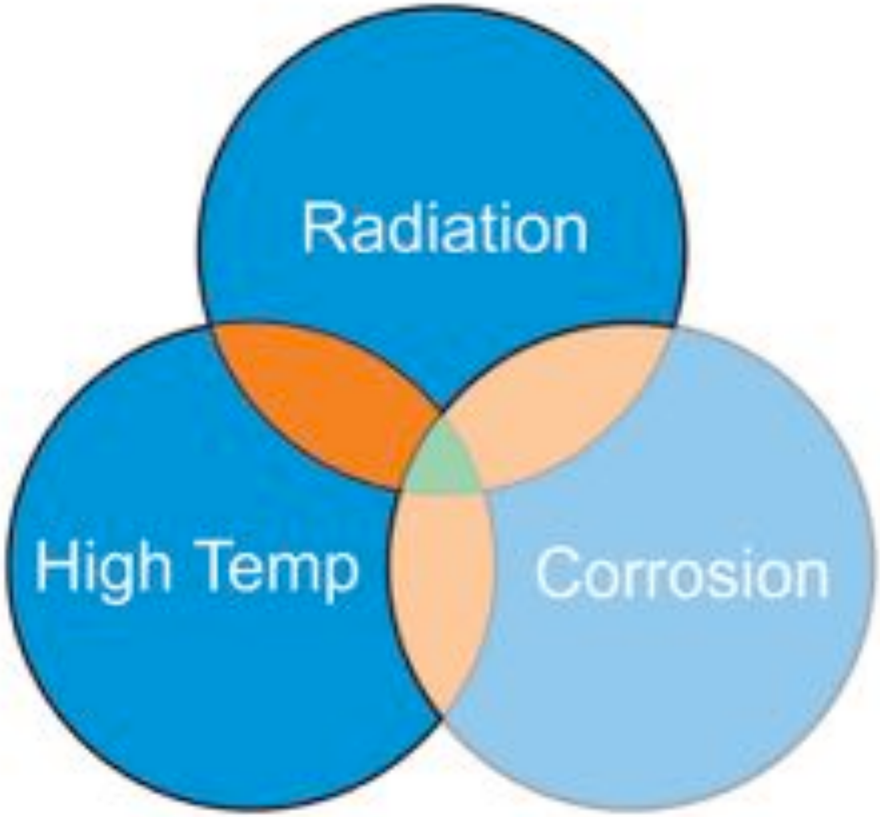
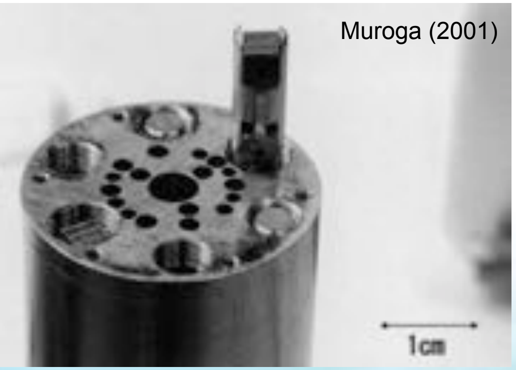
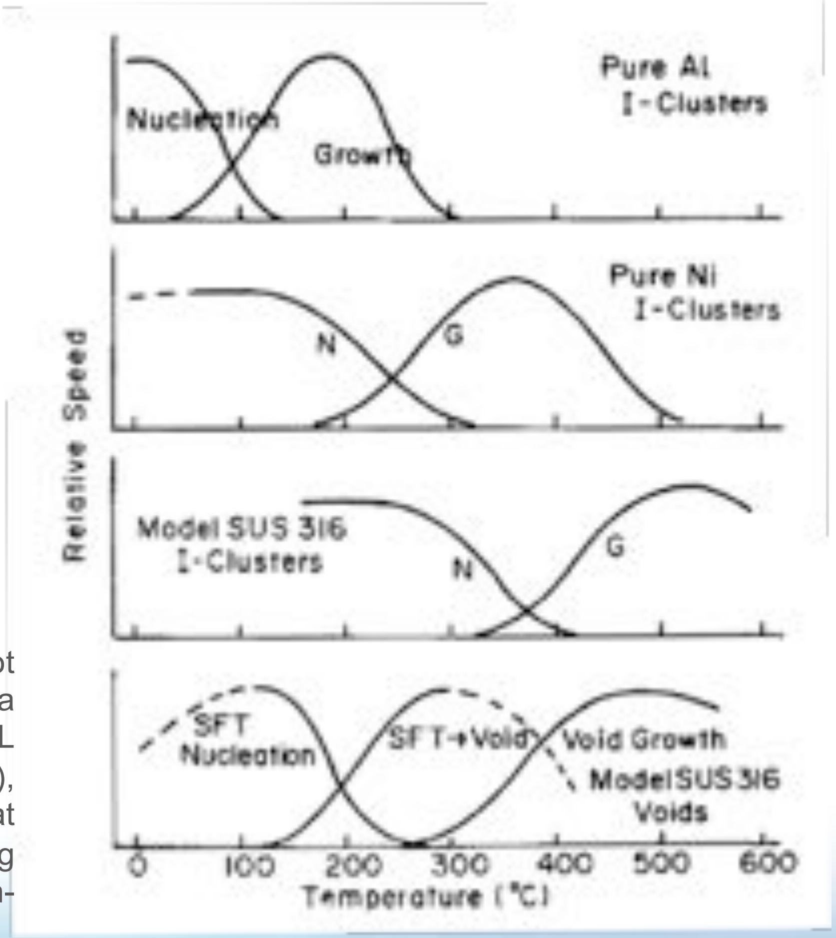


High Temperature Irradiation (Planned)

- Motivation
  - Radiation damage is controlled by a thermally activated processes, so some temperature control is desirable
- Planned Work
  - Neutronics/gamma heating calculations
  - Thermal hydraulics calculations (safety case)
  - Can design and T&C specifications

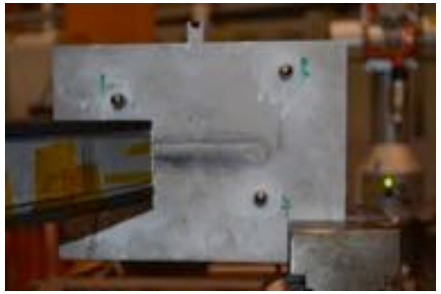


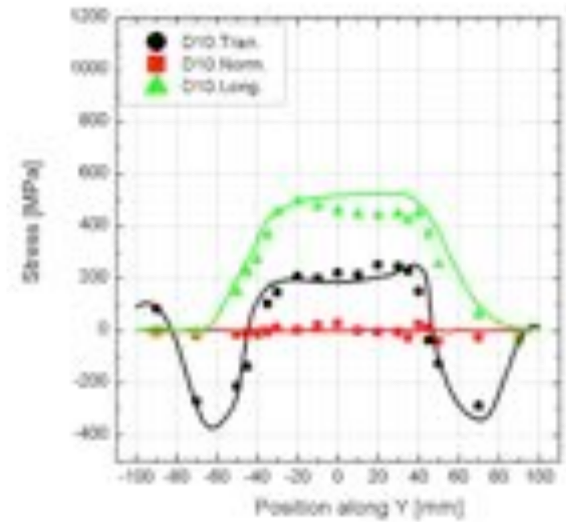
OPAL contains empty space ("hot source") in the RPV. By developing a suitable holder (such as the ORNL HFIR sample heating holder, left), samples may be irradiated in OPAL at higher temperatures, thereby enabling elevated-temperature damage mechanisms to be characterised.

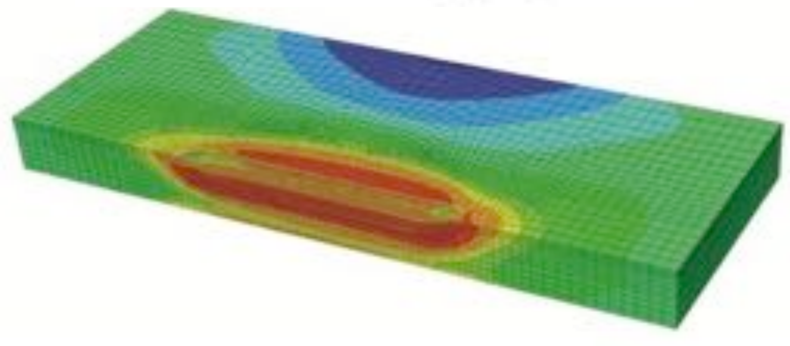


Understanding and Predicting Weld Residual stress

- ANSTO has developed complex models of microstructure and stress state around single- and multi-pass welded joints
- Used to support plant maintenance and design decisions
- Includes high temp Ni alloys







Admiral Hyman Rickover (US Navy)

An *academic* reactor or reactor plant almost always has the following basic characteristics:

(1) It is simple.
(2) It is small.
(3) It is cheap.
(4) It is light.
(5) It can be built very quickly.
(6) It is very flexible in purpose.
(7) Very little development will be required. It will use off-the-shelf components.
(8) The reactor is in the study phase. It is not being built now.

Admiral Hyman Rickover (US Navy)

On the other hand a *practical* reactor can be distinguished by the following characteristics:

(1) It is being built now.
(2) It is behind schedule.
(3) It requires an immense amount of development on apparently trivial items.
(4) It is very expensive.
(5) It takes a long time to build because of its engineering development problems.
(6) It is large.
(7) It is heavy.
(8) It is complicated.

Admiral Hyman Rickover (US Navy)

- The tools of the *academic designer* are a piece of paper and a pencil with an eraser. If a mistake is made, it can always be erased and changed.
- If the *practical-reactor designer* errs, he wears the mistake around his neck; it cannot be erased. Everyone sees it.
- The *academic-reactor designer* is a dilettante. He has not had to assume any real responsibility in connection with his projects. He is free to luxuriate in elegant ideas, the practical shortcomings of which can be relegated to the category of "*mere technical details*."
- The *practical-reactor designer* must live with these same technical details. Although recalcitrant and awkward, they must be solved and cannot be put off until tomorrow. Their solution requires manpower, time and money.

In Nuclear, materials are usually in this category of "*mere technical details*"

Thank you