Ion Irradiation for Radiation Damage Studies

Mimicking Neutron Damage?

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Radiation damage in Materials

The interaction of radiation damage processes.

C.A. English (2011)
## Neutrons

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
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</thead>
<tbody>
<tr>
<td>Usually interested in the response of materials to fluxes of fast neutrons</td>
<td>Very expensive infra-structure required to irradiate and examine radioactive samples.</td>
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<tr>
<td>Able to match dose, irradiation temperature and frequently primary recoil spectra</td>
<td>Not easy to irradiate a large number of samples at a wide variety of irradiation conditions</td>
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<tr>
<td>Accelerated irradiations possible in Material test reactors (well-defined environment)</td>
<td>High level of expertise required to characterise the irradiation environment, design appropriate rigs etc.</td>
</tr>
<tr>
<td>In certain cases can artificially generate high levels of transmutation gas (for fusion)</td>
<td>May not be able to study individual mechanisms. E.g cascades formed from mono-energetic ions</td>
</tr>
<tr>
<td>Bulk Samples</td>
<td>May not be able to reach high doses required for fast reactor or fusion material studies</td>
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J. Hyde (2010)
## Ion beams

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
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<tbody>
<tr>
<td>Non-activating</td>
<td>Don’t get realistic transmutations</td>
</tr>
<tr>
<td>Fast turn-round: High doses up to ~100 dpa achievable in 10’s of hours</td>
<td>Damage rate is $10^3 - 10^5$ times higher than in power reactors</td>
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<tr>
<td>Close control of temperature and dose etc.</td>
<td>Damage and deposited ion concentration is non-uniform with depth</td>
</tr>
<tr>
<td>Multi-beam facilities can independently vary displacement damage and gas accumulation</td>
<td>Multi-beam facilities have very restricted target area (~20mm $\varnothing$)</td>
</tr>
<tr>
<td>TEM in-situ work is possible</td>
<td>Likely free surface effects</td>
</tr>
<tr>
<td>TEM, Atom probe, nanoindentation “easy”</td>
<td>Yield, work-hardening, creep, fatigue, etc. &amp; High T tests difficult</td>
</tr>
</tbody>
</table>
Transmutation
Tungsten transmutation in fusion power reactor

- Pure W under outboard equatorial FW armour flux for 5 fpy
Radiation-induced clustering in alloys

W- 5%Re should be a stable solid solution
… but it’s not when it is irradiated

Under Fusion Power Reactor conditions, pure W transmutes to give 5%Re in ~ 7 years.

Ion irradiation to $2.64 \times 10^{15} \text{ W}^+/\text{cm}^2$ (33dpa)
Dose rate: $3.57 \times 10^{-4} \text{ dpa} / \text{s}$
Temperature: 300°C
Clustering and Hardening in “Transmuted” W

Preliminary analysis indicates that the clusters are very weak obstacles: $\Phi_c \approx 85^\circ$

But there are lots of them, very closely spaced - especially in W-Re-Os

Same would apply to neutron irradiation ???

Transmutations producing He in fusion power reactor
Effects of Implanted He on T91 steel

"Uniform implantation of 23 MeV α-particles up to 5000 appm at 250°C carried out at the compact cyclotron of Forschungszentrum Jülich (FZJ)"
Tungsten – Hardening by Displacement Damage and Helium

<table>
<thead>
<tr>
<th>dpa</th>
<th>appm He</th>
<th>Hardness (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>300</td>
<td>3000</td>
</tr>
</tbody>
</table>

Polycrystalline W 99.99% pure, sequentially ion-implanted with W⁺ and He⁺ at 300°C. W implantation depth 0 - 200nm; He implantation depth range 0 - 2500 nm. Hardness data at 100 nm indenter depth.
Alloy Stability under irradiation
Radiation Damage as it Happens…

IVEM – Argonne National Lab

MIAMI – University of Huddersfield
Oxide – dispersion-strengthened alloys – radiation resistance

Particles stable, and no radiation damage was visible below 1 dpa

ODS PM2000, RT irradiated with 150 keV Fe+, room temperature
Neutron and Ion irradiation-induced clustering in Fe-Cr alloys

Neutron Irradiation: Clustering only seen at > 9% Cr: ~ 85% Cr in clusters


Ion Irradiation:

- Cr clustering observed even in Fe-3%Cr (associated with C)
- Local concentration in clusters:
  - Up to 14% Cr in Fe-3%Cr
  - Up to 35% Cr in Fe-12%Cr
Hardening effects of irradiation damage
# Dose Rates

<table>
<thead>
<tr>
<th>Radiation Source</th>
<th>Dose Rate (dpa / s) in Iron-based Alloys</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fission Reactor Pressure Vessels (RPV)</td>
<td>~ $10^{-12} – 10^{-11}$</td>
</tr>
<tr>
<td>Rotating Target Neutron Source (RNTS-II)</td>
<td>~ $10^{-10}$</td>
</tr>
<tr>
<td>Fast Flux Test Facility (FFTF)/DEMO Fusion Reactor</td>
<td>~ $10^{-8} – 10^{-6}$</td>
</tr>
<tr>
<td>Ion Implantation – Low Dose Rate</td>
<td>$3 \times 10^{-5}$</td>
</tr>
<tr>
<td>Ion Implantation – High Dose Rate</td>
<td>$6 \times 10^{-4}$</td>
</tr>
<tr>
<td>HVEM Irradiation</td>
<td>~ $10^{-3}$</td>
</tr>
</tbody>
</table>
Nanoindentation hardness: Dose rate effects

Hardness (GPa)

0.6dpa, 300°C

Cr content (%)
Nanoindentation hardness: Dose rate effects

0.6 dpa, 400°C

Hardness (GPa)

Cr content (%)
Nanoindentation hardness: Dose rate effects

0.6dpa, 500°C

Cr content (%)
TEM of ion irradiation damage Fe 5%Cr 300°C

Low dose rate

High dose rate
APT data for the Fe 5%Cr alloy irradiated at 400°C with the low dose rate (a) and high dose rate (b). The figure includes an atom map showing Fe atoms and a 0.5 at.% CrN isoconcentration surface (i) and proximity histograms showing the variation in composition from the centre of enrichment outwards into the matrix (ii). Centre of enrichment is defined as the region of highest CrN concentration.
Neutron-irradiated Material: Fe-6%Cr

N-irradiated to 1.7 dpa at 288°C, dose rate ~1 x 10^{-7} dpa/s

- 66 cantilever beams with depths from 0.82μm to 7.3μm
- Also made in:
  - Ion-irradiated Fe-6%Cr, same dpa & temperature
  - Unirradiated

Activity: 37MBq

FIB work at CAES, Idaho
Micromechanical testing Fe-6%Cr – yield stress

Yield Stress (GPa)

Beam depth (μm)

0.00E+00 1.00E+09 2.00E+09 3.00E+09 4.00E+09 5.00E+09 6.00E+09 7.00E+09

Ion-irradiated

Neutron-irradiated

Unirradiated

0.1mm
Micromechanical testing Fe-6%Cr – Size effects

Yield Stress (GPa)

0.0  2.0  4.0  6.0

Beam depth (μm)

0.0  2.0  4.0  6.0  8.0

Strong size Effects in micromechanical tests

Weak size effects in micromechanical tests

Ion-irradiated

Neutron-irradiated

Unirradiated
Micromechanical testing RAFM steels – Size effects

- Weak size effects in micromechanical tests
- Strong size effects in micromechanical tests
Protons ?
Proton irradiation for (micro) mechanics?

SRIM: 2 MeV protons into Fe

Could use specimens 10 – 12 μm thick – well beyond the size-dependent mechanics regime
Proton irradiation for (micro) mechanics?

SRIM: 2 MeV protons into Fe

For 2MeV $^1$H$^+$ into Fe:
- Total damage: $\sim 1 \times 10^{-4}$ events / (Å - ion)
- $\Rightarrow \sim 10^{21}$ ions cm$^{-2}$ for 1 dpa
- Depth $\sim 15$ µm

For 2MeV $^{56}$Fe$^+$ into Fe:
- Total damage: $\sim 4$ events / (Å - ion)
- $\Rightarrow \sim 2 \times 10^{16}$ ions cm$^{-2}$ for 1 dpa
- Depth $\sim 1$ µm
Tensile properties and microstructure of martensitic steel DIN 1.4926 after 800 MeV proton irradiation

Y. Dai\textsuperscript{a,*}, F. Carsugh\textsuperscript{b,1}, W.F. Sommer\textsuperscript{c}, G.S. Bauer\textsuperscript{a}, H. Ullmaier\textsuperscript{b}

Abstract

A double-wall window of martensitic steel DIN 1.4926 (11\% Cr) was irradiated with \textbf{800 MeV protons} in the LANSCE facility of the \textbf{Los Alamos National Laboratory (LANL)} to a total number of about \textbf{6.3 × 10\textsuperscript{22} protons} (2.8 Ah) in a temperature range from 50\textdegree{}C to 230\textdegree{}C. Tensile tests show that irradiation hardening increases with fluence up to the maximum attained dose of about 6.6 dpa. All irradiated specimens show significant embrittlement, ≤1.5\% uniform elongation and 7.5–9\% total elongation as compared to about 11\% uniform elongation and 21\% total elongation for the unirradiated specimens.

Tensile specimen dimensions (mm)

... activated specimens
Summary

- With careful choice of conditions, ion irradiation can be used to mimic effects of neutrons
  - Single heavy-ion beams – displacement damage
  - Dual and triple beams – H, He effects

- BUT Depth is shallow (~1-2µm)
  - OK for TEM, Atom probe
  - Mechanical testing is difficult
    - Hardness
    - Micromechanics: stress-strain curves and fracture
    - Size effects are strong

- Displacement damage from protons?
  - Depth is good (~15-25µm at non-activating energies)
  - Damage rate very low
  - Damage type equivalence to neutrons?
UK: Neutron, Ion & Proton irradiation facilities

Neutron
- Archive specimens
- AWE
  - ASP (14MeV, low current)
  - VIPER (U-235, short pulses)
- JHR – from 2018?

Ion & Proton
- Surrey Ion Beam Centre
  - 2 MeV, wide area
  - high dose rate
- Dalton Cumbria Centre
  - 10-15 MeV, small area
  - H,He high dose rate
  - upgrading to dual-beam (NNUF)
- MIAMI (Huddersfield)
  - In-situ TEM irradiation
  - 100 keV, typically He
- Birmingham
  - Synchotron
    - H,D,\(^3\)He up to 53MeV,
  - Dynamitron
    - 3MeV H, wide area
    - very high dose rate

What we don’t have…
- Neutrons
  - test reactor
- Ion irradiation
  - wide area, high voltage
  - dual or triple-beam
- Proton irradiation
  - high voltage, high current, wide area
UK: Irradiated Materials Characterisation Facilities

£15M NNUF initial capital spend (2013)

£60M NNUF additional funding in 2014 Autumn Statement