HELIUM EFFECTS ON IASCC SUSCEPTIBILITY

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Content of the presentation

- Introduction
- Experimental
- Results & discussion
  - Bubble evolution after post implantation annealing
  - Helium effects on IASCC
- Summary, conclusions & perspectives
Introduction - What is IASCC?

By definition: IASCC is actually inter-granular SCC assisted by irradiation.
Cold worked 316SS after service in PWR conditions

Adapted from Edwards et al., 2003.
Introduction - Helium & IASCC

For given dpa, FBR-irradiated SS show much lower IASCC susceptibility than PWR-irradiated SS in spite of similar irradiation hardening and GB segregation. The He effect might be one of the main reasons for this large difference:

- PWR $\sim 10$ appm He/dpa $>>$ FBR $\sim 0.1$ appm He/dpa

Good correlation between IGSCC susceptibility & He concentration evolution.
**Introduction - Lab Investigations**

**Helium concentration for IGSCC (304 or 316)**

- **Artificially implanted HELIUM**

  **Single GB**
  - >20’000 appm He
  - Bubble spacing < 5 nm
  - GB coverage = 7%
  - 0.95 dpa
  - IG fracture

  **Polycrystal**
  - >10’000 appm He
  - 1 dpa
  - TG-C

- **Irradiated in Halden reactor (BWR) 300°C**

  **Test in HWC**
  - ~few tenths appm He
  - ~1 dpa
  - TG-C (<2%) + TG-D

  **Test in NWC**
  - ~few tenths appm He
  - >0.45-5 dpa
  - IG (3-30%) + TG-D

- **Irradiated in BOR-60 (FBR) 320°C**

  **Test in HWC**
  - ~0 appm He
  - ~48 dpa
  - TG-C (<2%) + TG-D

  **Test in NWC**
  - ~0 appm He
  - ~5 dpa
  - IG (0-50%) + TG-D

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*NO IG → He?*

**References**

- T. Miura et al., 2015.
- Y. Chen et al., 2014.
Approach

- Potential concern for some PWR internals & LTO > 50 years
- SA (baffle formers) and CW SS (baffle bolts)
- He & change in deformation mode at high dose
- Separation of He and displacement damage effects
  -> He implantation (100 to 1000 appm, 0.016 to 0.16 dpa only)
- Simulation of He bubble structure in baffle bolts & variation of He bubble size and GB He bubble coverage
  -> post implantation annealing study
  -> critical He concentrations or GB coverage for IG (IA)SCC
- Characterization of IG (IASCC) susceptibility by SSRT tests in hydrogenated HTW
  -> fracture & deformation mode by SEM & TEM
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316L

<table>
<thead>
<tr>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Si</th>
<th>Co</th>
<th>N</th>
<th>V</th>
<th>C</th>
<th>P</th>
<th>W</th>
<th>Al</th>
<th>Ti</th>
<th>Sn</th>
<th>Nb</th>
<th>S</th>
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<tbody>
<tr>
<td>17.61</td>
<td>12.32</td>
<td>2.379</td>
<td>1.768</td>
<td>0.466</td>
<td>0.164</td>
<td>0.0673</td>
<td>0.036</td>
<td>0.0275</td>
<td>0.024</td>
<td>0.023</td>
<td>0.018</td>
<td>0.007</td>
<td>0.006</td>
<td>0.003</td>
<td>&lt;0.003</td>
</tr>
</tbody>
</table>

SA at 1050°C for 30’ quenched in water
SA or 20 % CW

Average grain size 52 μm

Tensile specimens

45 MeV ≈ 250 μm

\[
\frac{t_c}{s} = 7
\]

\[
\frac{t}{w} \geq 0.2
\]

Miniaturized sample
Standard sample
**Experimental - Helium implantation**

**Irradiation parameters**

$\alpha$-energy = 45 MeV  
$T$-Irradiation = 300 ºC  
Fluence = $2.17 \times 10^{18}$ He cm$^{-2}$  
He-concentrations = 100, 300, 1000 appm  
damage = 0.016, 0.05, 0.16 dpa
Objective: Reproduce BFB microstructure & increase GB coverage

1. 1-2h furnace condition
   - Vacuum tube
   - Sample
   - T<100°C
   - Furnace

2. 1h in furnace at T
   - T<100°C
   - 650°C 750°C
   - 800°C 850°C
   - 900°C 950°C
   - 1000°C
   - Furnace

3. Cooling in vacuum
   - Furnace

Used for He implanted plates and miniaturised samples

10/04/2018
Experimental - Mechanical testing

Tensile test machines and conditions

All tests are SSRT at $10^{-6}$ or $10^{-7}$ s$^{-1}$

- **Water loop.** Standard and miniaturized samples
  - High temperature water tests (HTW) at 288°C
  - Normal water conditions (NWC) with 500ppb O$_2$
  - Hydrogenated water conditions (HWC) with 2.2 ppm H$_2$

- **Roell + Korthaus machine.** Only standard samples
  - **Air tests**
    - Room temperature tests (RTA) at 25°C
    - High temperature tests (HTA) at 288°C

- **SSRT machine.** Only miniaturized samples
  - **Air tests**
    - Room temperature tests (RTA) at 25°C
    - High temperature tests (HTA) at 288°C

*Effective gauge length is needed*
Experimental - Microstructure observations

Metallurgical investigations

Using the “NVision 40 FIB-SEM” with 15 kV

- Characterize the fracture morphology and RA.
- Crack initiation and propagation mechanisms.

Microstructure investigations by TEM

Using JEOL 2020

PIA 850°C
Δf = 500 nm

50 nm
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Results - Bubble distr. PIA (1000 appm)

at 750°C

at 900°C
Results - Bubble average spacing and GB coverage

Limited of GB coverage, hence limited GB weakening
The thermal activation analysis shows that the He bubbles grow according to the dissociative mechanism (Ostwald Ripening) both, for GB and grain interior. This mechanism occurs at least 300°C below the one reported in RT implantation.

<table>
<thead>
<tr>
<th></th>
<th>Cold implant.</th>
<th>Hot implant.</th>
<th>Grain interior</th>
<th>Grain boundary</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_a$ (eV)</td>
<td>0.25</td>
<td>1.03</td>
<td>1.07</td>
<td>1.11</td>
</tr>
<tr>
<td>$Q$ (eV, = $E_a n$)</td>
<td>1.26 - 1.51</td>
<td>3.07 - 4.15</td>
<td>3.21 - 4.28</td>
<td>3.33 - 4.44</td>
</tr>
<tr>
<td>Mechanism</td>
<td>Surf. diffusion</td>
<td>Dissociation</td>
<td>Dissociation</td>
<td>Dissociation</td>
</tr>
</tbody>
</table>
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Results - RIS on Grain Boundary

- GB RIS with FEI TALOS F200X (200 kV)

- Molybdenum

- Iron

- Chromium

- Enrichment
  2.1 — 3 % wt.
  Usually W shape

- Depletion
  64.5 — 63.5 % wt.
  Usually V shape

- No measurable effect
Results - SSRT to SA + 1000 appm samples

1000 appm with/out HT

Transgranular dimple fracture is dominant, some transgranular cleavage in HTW

Hardening produced by He bubbles

<table>
<thead>
<tr>
<th></th>
<th>SA HTW</th>
<th>1000appm Air 288°C</th>
<th>1000appm HTW</th>
<th>1000appm PIA 1000°C HTW</th>
</tr>
</thead>
<tbody>
<tr>
<td>YS</td>
<td>178</td>
<td>383</td>
<td>418</td>
<td>198</td>
</tr>
<tr>
<td>UTS</td>
<td>492</td>
<td>547</td>
<td>594</td>
<td>513</td>
</tr>
<tr>
<td>$\varepsilon_u$ (plastic)</td>
<td>0.28</td>
<td>0.23</td>
<td>0.26</td>
<td>0.27</td>
</tr>
</tbody>
</table>
Results - SSRT to CW + 1000 appm samples

CW with/out 1000 appm

CW samples show tensile response similar to IASCC curves

Transgranular dimple fracture is dominant, some transgranular cleavage in HTW

<table>
<thead>
<tr>
<th></th>
<th>CW HTA</th>
<th>CW HTW</th>
<th>CW-1000appm</th>
<th>CW-1000appm 10^-7</th>
</tr>
</thead>
<tbody>
<tr>
<td>YS</td>
<td>579</td>
<td>576</td>
<td>734</td>
<td>787</td>
</tr>
<tr>
<td>UTS</td>
<td>631</td>
<td>641</td>
<td>740</td>
<td>790</td>
</tr>
<tr>
<td>ε_u (plastic)</td>
<td>0.08</td>
<td>0.08</td>
<td>0.013</td>
<td>0.001</td>
</tr>
</tbody>
</table>
Results - Fracture investigations

- Fracture surface: 1000 appm with/out HT

SA - 1000 appm (Air-288°C)
SA - 1000 appm (HWC)

SA-1000 appm + PIA 1000°C (HWC)
**Results - Microstructure investigation**

- **Deformation microstructure (all HTW)**

  - **As-implanted samples**
    - No apparent 3D deformation cells with random arrangement of dislocations. Microstructure that might be a precursor for SCC.

  - **Post-implantation annealed**
    - PIA +1000 appm
    - Recover 3D deformation microstructure

- **As-implanted CW**
  - CW +1000 appm
  - Prior 3D deformation microstructure still present. No evident increase of more planar deformation

- **SA**
  - 3D deformation cells typical for high Ni contents.
**Why don’t we see IASCC?**

- **Crum depletion/Loop saturation/He concentration/Cluster saturation (%)**
- **Radiation damage (dpa)**

**Main differences**:
- Chromium
- Black dots
- # of loops
- Hydrogen
- Hardening

**Literature data**

**Estimated scenario**
Similar bubble size & distance in grain interior and on GB. PIA increases bubble size, but does only moderately increase GB He bubble coverage.

The activation energy of bubble evolution for GB and Matrix shows that in both cases the bubble grows with dissociative mechanism (OR). This mechanism occurs 300°C below the one reported in RT implantation. The coarsening mechanism might depend on both annealing T and bubble size.

Homogenised He implantation in SA and CW at 300°C up to 1000 appm has little impact on the macroscopic tensile properties and fracture morphology in air and HTW and results in very limited RIS only (only Mo).

The deformation microstructure clearly changes from dislocation cells to random distribution of dislocations in SA & He implanted samples, respectively. The formation of deformation bands is enhanced in as-implanted condition.

Accelerated SSRT (10-6 - 10-7 s-1) in HTW with 2.2 ppm DH at 290 °C did not induce IG (IA)SCC initiation in smooth tensile specimens with homogenized helium implantation at 300°C up to 1000 appm (<0.16 dpa) in SA, CW and PIA (≤ 1000°C) conditions.
However, the mechanically dominated short-term SSRT may be too short to exclude SCC initiation and could overlook other more time-consuming (e.g. corrosion-dominated) precursor and initiation processes.

The absence of IASCC might be related to the lack of a well-marked deformation mode change between the non-implanted and the implanted materials. The number of dislocation channels in the implanted samples and the He enrichment on the GB & GB helium bubble coverage (∼10%) are insufficient to induce IASCC in these conditions.

These results suggest that a helium concentration ≤ 1000 appm alone cannot induce IASCC, therefore there has to be some synergy between dpa damage and helium concentration.

The formation of irradiation-induced dislocation channels (at high dose) with high-stress concentration on grain boundaries, together with the current helium bubbles grain boundary coverage (∼10%), could promote intergranular cracking.

Further evaluations should thus include samples with high displacement damage (besides of high helium concentration) and crack growth experiments with pre-cracked specimens.
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