Microstructural evolution of neutron irradiated T91 ferritic-martensitic steel in the advanced test reactor

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Objectives of my PhD Project

1. Utilise atom probe tomography and nanoindentation to study the microstructure of T91 steel in both unirradiated and irradiated conditions.

2. To understand the nucleation, evolution, and effect of Mn-Ni-Si ppts that are formed by neutron irradiation between 326-377 oC of T91 steel.
1. ASTM Grade 91 steel A213 (Tube 91; T91)
T91 is a candidate for fuel cladding, duct/wrapper and structural material for sodium-cooled and lead-cooled fast fission reactors.
Tube = diameter $3.2 \text{ mm} < d < 127 \text{ mm}$
thickness $0.4 \text{ mm} < t < 12.7 \text{ mm}$

Fuel cladding tubes ~ 10 mm diameter; 0.5 mm thick
T91 Ferritic-Martensitic Steel

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Cr</th>
<th>Mn</th>
<th>Si</th>
<th>Mo</th>
<th>Nb</th>
<th>N</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>V</th>
<th>Ti</th>
<th>Zr</th>
<th>Ni</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>T91</td>
<td>0.07</td>
<td>9.24</td>
<td>0.47</td>
<td>0.28</td>
<td>0.96</td>
<td>t</td>
<td>t</td>
<td>0.02</td>
<td>0.02</td>
<td>t</td>
<td>0.21</td>
<td>t</td>
<td>t</td>
<td>0.16</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
Embrittlement of T91 Ferritic-Martensitic Steel

- Low Temp (50-300°C)
- Medium Temp (300-450°C)
- High Temp (450-600°C)

T91 (Mod 9Cr-1Mo)

- Shift in DBTT (°C)
- Neutron Dose (dpa)

- Unirradiated
- 10 dpa EBR-II
- 23 dpa EBR-II

T91 (Mod 9Cr-1Mo)

- Tensile Stress (MPa)
- Irradiation Temperature (°C)

- Hardening, $\Delta\sigma_y$ (MPa)
- Irradiation temperature (°C)

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Neutron Irradiation of T91 in ATR


<table>
<thead>
<tr>
<th>Steel</th>
<th>Temp (°C)</th>
<th>Dose (dpa)</th>
<th>Flux (n·cm⁻²·s⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T91</td>
<td>326</td>
<td>2.14</td>
<td>2.3E+14</td>
</tr>
<tr>
<td></td>
<td>372</td>
<td>8.82</td>
<td></td>
</tr>
</tbody>
</table>

Advanced Test Reactor
Idaho National Laboratory
First: How does Atom probe work?

- Sharp needle sample; tip radius 50-100nm; 50K temp; high vacuum
- Apply an electric field (2000-10,000V); the field is concentrated at the sharp tip to ~40 V/nm.
- Produces field evaporation at the tip of the needle when a short additional voltage or laser pulse is applied.
- If we know when the ions leave the tip then their time-of-flight can be related to their mass-to-charge ratio:

\[
\frac{\text{mass}}{\text{charge}} = 2eV \left( \frac{\text{time}}{\text{flight distance}} \right)^2
\]
As Received T91 Steel
T91 Steel: As-Received Characterisation

[Image: A diagram showing the distribution of elements in T91 steel, with elements Fe, Cr, Mn, Si, Ni, Mo, P, and Cu. The scale is 50nm.]
T91 Steel: As-Received Characterisation
2.14 dpa 326 °C T91 Steel
Segregation of Si, Mn, Ni, P and Cu

1) Cu forms clusters
2) P forms clusters
3) Si segregates to P and Cu clusters
4) Ni segregates to Si and Cu clusters
5) Mn segregates to Si, Ni and Cu clusters
6) Mn-Ni-Si clusters appear to form adjacent to Cu clusters
7) P and Si segregation to dislocations

Partially forming ‘G-Phase’ Mn-Ni-Si ppts
T91: 2.14 dpa 320C: Mn-Ni-Si cluster

Dislocations decorated by P and Si
T91: 2.2 dpa 320C Mn-Ni-Si clusters

G Phase
$\text{Mn}_6\text{Ni}_{16}\text{Si}_7$

Average Diameter (nm)
Volume Fraction Mn-Ni-Si (%)
Neutron Irradiation Dose (dpa)
Volume Fraction Mn-Ni-Si
Average Diameter Mn-Ni-Si

Average Volume Mn-Ni-Si
Number Density Mn-Ni-Si
Number Density (#/m³)
T91: 2.2 dpa 320C Bonus
T91 8.8 dpa 370 °C
T91: 8.8 dpa; 370C – Mn-Ni-Si clusters
Evolution of the microstructure
Microstructure Evolution of Irradiated T91 Steel

- Neutron Dose (dpa)
  - 0 dpa
  - 2.14 dpa
  - 8.8 dpa

- Cr Si Ni Cu P

- Mn-Ni-Si clusters
- Cu clusters
- Possible alpha'

- Dislocations decorated by P and Si
What does the atom probe data tell us about irradiation of T91 steel?

- Embrittlement (increase in yield stress) is shown below 400 °C. G-Phase ppts inhibit dislocation motion, which contributes majorly to the embrittlement of T91 steel below 400 °C (as shown in figure to the left [3]).

- Observed Mn-Ni-Si clusters at 2.14 dpa and grew in size, volume fraction, average diameter and average volume. However, decreased in number density but this measurement could be affected by localised features in atom probe datasets. No alpha prime observed.

- Future work: nanoidentation and prior austenite grain boundary analysis of neutron irradiated T91 steel

- Overall this research provides an insight into the microstructural evolution of neutron irradiated T91 steel and provides a better understanding of the degradation of nuclear reactor core materials.

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Extra Slides
Challenge for structural materials

Overview of the temperature regimes against expected displacement damage of various current and future reactors [1].

Helium production in advanced steels in fission, fusion and neutron experiments [2].

304 and 316 austenitic stainless steel grades are the work horse material in the nuclear industry however…

Comparison between volumetric swelling of 304L and 9-12Cr ferritic/martensitic steels [3].

Radiation swelling of 316 stainless steel [4]

T91: 8.8 dpa; 370C – Alpha prime? R33_09813

Cr Si Ni Mn Cu P
Mn-Si-Ni precipitates in T91 controlled by G-phase thermodynamics, RIS, dislocation enhanced heterogeneous nucleation sites. Model produced by using CALPHAD, Lattice monte carlo and cluster dynamics.

Microstructure Evolution of Irradiated T91 Steel

- Neutron Dose (dpa):
  - 0 dpa
  - 2.14 dpa
  - 8.8 dpa

- Elements:
  - Cr
  - Si
  - Ni
  - Cu
  - P
  - Mn

- Observations:
  - Mn-Ni-Si clusters
  - Cu clusters
  - Dislocations decorated by P and Si
  - Possible alpha’
Ion Irradiation between 0.1 to 4.0 dpa

<table>
<thead>
<tr>
<th>Steel</th>
<th>Temp (°C)</th>
<th>Fe$^{4+}$ Dose at Bragg Peak (dpa)</th>
<th>Flux (ions·cm$^{-2}$·s$^{-1}$)</th>
<th>Fluence (ions·cm$^{-2}$·s$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T91</td>
<td>300</td>
<td>0.32</td>
<td>4.8×10$^{11}$</td>
<td>3.5×10$^{14}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6.4</td>
<td>4.4×10$^{11}$</td>
<td>6.5×10$^{15}$</td>
</tr>
</tbody>
</table>

0.32 dpa SRIM 2013

6.4 dpa SRIM 2013
DISLOCATIONS

Fe$^{4+}$ Ion Irradiation Dose at 300 °C

@ 300°C

0.075 dpa  0.1 dpa  2.5 dpa  2.5 dpa  4 dpa

P clusters

P/Si clusters

P/Si/Ni clusters

Mn-Si-Ni clusters

25 nm

Ion Irradiation Dose at 300 °C
Nanohardness and Mn-Ni-Si clusters

- Mn-Ni-Si clusters
- To do