IRRADIATION EMBRITTLEMENT CHARACTERIZATION OF THE EUROFER 97 MATERIAL

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Introduction

- The paper summarizes results of irradiation embrittlement study of EUROFER 97 material that has been proposed as one candidate for future GEN IV reactors.
- Test specimens were manufactured from base metal EUROFER 97 as well as from weld metal and tested in initial unirradiated condition and also after irradiation to reach approx. 2.5 dpa.
- Sub-sized TPB (3x4 mm) specimens were tested to determine static and dynamic fracture toughness.
- The main objective: comparison of unirradiated and irradiated properties as well as changes in transition temperature shifts of these material parameters.
- The investigation has been performed in the frame of EFDA project on „Tritium Breeding and Materials“, Task TW2.
Material and Specimens

- Micro-TPB samples (W=4mm, B=3mm) were prepared by machining from plates of 14 mm thickness. Static fracture toughness specimens have been side grooved.

- Materials:
  - BASE METAL: Reduced activation martensitic steel EUROFER 97 (steel plates of 300x300x14mm).
  - WELD METAL: A weld joint was prepared from two plates 14x90x300mm by TIG method in CEA Saclay.

Tab. 1 The chemical composition of EUROFER 97 in w%:

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Ti</th>
<th>Zr</th>
</tr>
</thead>
<tbody>
<tr>
<td>BASE</td>
<td>0.11</td>
<td>0.47</td>
<td>0.040</td>
<td>0.005</td>
<td>0.004</td>
<td>0.0016</td>
<td>0.022</td>
<td>8.82</td>
<td>0.005</td>
<td>&lt;0.005</td>
</tr>
<tr>
<td>WELD</td>
<td>&lt;0.001</td>
<td>&lt;0.005</td>
<td>0.001</td>
<td>0.0034</td>
<td>0.20</td>
<td>1.09</td>
<td>&lt;0.005</td>
<td>&lt;0.005</td>
<td>0.0016</td>
<td>0.006</td>
</tr>
</tbody>
</table>
Material specimens were irradiated in the Chouca rig (irradiation rig with a controlled temperature and inert gas) in the core of the LVR-15 experimental reactor in the Nuclear Research Institute Rez plc. for 8 irradiation cycles (21 days each) at max. 235 °C.

Neutron fluence was equivalent to approx. 2.5 dpa (1.5 x 10^{21} n.m^{-2}, E >1 MeV).
Irradiation 2

Fig. 1 Average neutron fluences (individual blocks of specimens in the irradiation rig)
Procedure 1

Testing end evaluation of the results has been performed in accord with:

- **Dynamic fracture toughness**
  - EN 10045-1 Metallic materials-Charpy impact test-Part 1
  - ISO 14556 Steel-Charpy-V notch pendulum impact test-Instrumented test
  - ESIS TC5 Proposed standard method for instrumented impact testing of sub-sized Charpy-V notch specimen of steels (Draft 10)
  - ASTM E 1921-05 (obtained reference transition temperatures are not in compliance with the requirements of the method)

- **Static fracture toughness**
  - ASTM E 1820-01
  - ASTM E 1921-05 (invalid reference temperature To)
  - Additional evaluation of J-R test in upper shelf in agreement with CSN 420347 (Czech national testing method) and ESIS P2/91D procedure
Procedure 2

Transition temperatures:

- **Static and dynamic fracture toughness**
  - Transition temperature $T_{100}$ at $K_{JC}=100 \text{ MPa.m}^{1/2}$
  - Reference temperature $T_0$ (ASTM E 1921-05)

\[
K_{JC} = K_{\text{min}} + a_0 \cdot e^{a_1 \cdot T}
\]

\[
T_0 = \left( \frac{1}{0.019} \right) \cdot \ln \left( \frac{K_{JC(\text{med})} - 30}{K_{JC(\text{med})} - 30} \right)
\]
RESULTS: Dynamic fracture toughness 1

- Micro TPB specimens (3x4mm) – no side grooves
- Roell Amsler RKP 50 impact tester (nominal capacity of 50 J, max. testing energy 6,29 J)
- Impact velocity : 1,36 m/s
RESULTS: Dynamic fracture toughness 2

- **WELD METAL** - the obtained reference temperatures \( T_o \) are not in compliance with the requirements of ASTM E 1921-05.
RESULTS: Static fracture toughness 1

- Micro TPB specimens (3x4mm) – 20% side grooved
- Instron 1342, load cell capacity 50 kN
- Load point displacement has been measured using LVDT gauges
RESULTS: Static fracture toughness 2

All the obtained reference temperatures $T_0$ are not in compliance with the requirements of ASTM E 1921-05.

EUROFER 97
Base metal - irradiated
$T_0 = +6.1^\circ C$
$\text{Sum } n_i = 0.52$
RESULTS: Static fracture toughness 3

- J-R test in upper shelf have been performed in accord with ASTM E 1820-01.
- Because of small dimension of Micro-TPB samples, ASTM as well as ESIS/CSN qualification of the $J_Q$ validity was not been satisfied.
- From comparison of J-R curves it is seen that $J_Q$ values were reduced by approx. 50% for BM and 30% for WM.
- Low sensitivity of weld metal to neutron irradiation embrittlement in comparison of Eurofer 97 base metal.
SUMMARY
Transition temperature shifts

- Invalid value of $T_0$ (ASTM E1921-05). Maximum $K_{JC}$ capacity ($M=30$) has not been fulfilled + results outside $T_0+/-50$ °C.
- Low sensitivity of weld metal to neutron irradiation embrittlement in comparison of Eurofer 97 base metal.

<table>
<thead>
<tr>
<th>Specimen set</th>
<th>Displ. per atom (dpa)</th>
<th>Dynamic fracture toughness</th>
<th>Static fracture toughness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\Delta T_{100}$ (°C)</td>
<td>$\Delta T_0$ (°C)</td>
</tr>
<tr>
<td>BM</td>
<td>2.43</td>
<td>113.0</td>
<td>122.6</td>
</tr>
<tr>
<td>WM</td>
<td>2.31</td>
<td>61.6</td>
<td>74.7*</td>
</tr>
</tbody>
</table>
SUMMARY
Effect of specimen size and geometry on $T_o$

- Static fracture toughness results Eurofer 97 BM
- $T_o$ determined according to ASTM E1921-05
- Transition temperature $T_o$ appears to be in-dependent on specimen size and geometry.
- Good agreement of NRI INVALID $T_o$ values in comparison with the other data [1, 2]

$$T_o = A_1 + A_2 \left[ 1 - \exp\left(\frac{-Dose}{\tau}\right) \right]$$

$A_1 = -107.4 \degree C$, $A_2 = 297.8 \degree C$, $\tau = 4.23$ dpa [2]

Data from:
[1] Final report for EFDA project Task2
SUMMARY
Shift in transition temperature $T_o$

- Static and dynamic fracture toughness results Eurofer 97 BM
- $T_o$ determined according to ASTM E1921-05
- Good agreement of NRI INVALID $T_o$ values in comparison with the other data [1,2]
- Dynamic fracture toughness shift do not differ significantly in comparison with static fracture temperature shifts

$$To = A \left[ 1 - \exp \left( -\frac{Dose}{\tau} \right) \right]$$

$A_1 = 268.9$ °C, $\tau = 3.58$ dpa [2]

Data from:
[1] Final report for EFDA project Task2
CONCLUSIONS

- Low sensitivity of weld metal to neutron irradiation embrittlement in comparison of Eurofer 97 base metal.
- Transition temperature $T_o$ appears to be in-dependent on specimen size and geometry.
- Dynamic fracture toughness shift do not differ significantly in comparison with static fracture temperature shifts.

**Additional testing**: It will be performed impact testing on un-irradiated and irradiated sub-sized Charpy-V specimens (3x4 mm).
Thank you for your attention

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