Spallation material preparation using the hotcell facility, a new preparation box and focused ion beam for investigations from mechanical testing up to methods using synchrotron light

Hotlab Meeting 2017, 20.5.2017
Introduction

- Nuclear Materials Research at PSI
- STIP irradiation program / Facilities at PSI

Radioactive materials flow (from STIP)

- Flowchart
- New facilities
  - Preparation BOX
  - Focused Ion Beam (FIB)
    (Motivation)

Examples

- Mechanical properties of F/M steels (calls for better understanding)
- Understanding of precipitates
  - APT sample preparation (@ university ETH)
  - Absorption spectroscopy (@ synchrotron)
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Background - Programs

Today in CH
- Generation I
  - Early Prototype Reactors
    - Shippingport
    - Dresden, Fermi I
    - Magnox
- Generation II
  - Commercial Power Reactors
    - LWR-PWR, BWR
    - CANDU
    - VVER/REM
- Generation III
  - Advanced LWRs
    - ABWR
    - System 80+
    - AP600
    - EPR
- Generation III+
  - Evolutionary Designs Offering Improved Economics
  - Generation IV

LWRs
- Near-Term Deployment

High T Fast n
- Generation IV
  - Highly Economical
  - Enhanced Safety
  - Minimal Waste
  - Proliferation Resistant

Spallation
- ADS
  - Lead-Bismuth coolant

Nuclear Fuels

Component Structural Integrity

Advanced Nuclear Materials

STIP program – SINQ Target Irrad. Program Materials in Spallation Target

- Protons and spallation neutrons
- High activation
- High He/dpa ratio (embrittlement)
- Transmutation
  → Challenging material degradation
Irradiation parameters

- $4 \times 10^{14} \text{ n/cm}^2/\text{s}$
- For steel (Fe)
  - 12-15 dpa per year
  - 100-200 mSv/h/cm$^3$
Analysis Tools

Microscopy

- FEG-SEM / EBSD & EDX, SEM/EDX
- TEM, FIB, shielded FIB/SEM
- LM & SM, metallography
- Sample preparation techniques

Corrosion Testing

- 9 HT-water loops with autoclaves with loading systems. Static autoclaves.
- Crack initiation & growth monitoring
- HT electrochemistry (ECN, IS, RE)

ND Diagnostics

- Magnetic methods (EC, 3 MA, GMR, SQUID, Ferromaster)
- Electric & thermoelectric methods ($\rho$, TEP, DCPD)
- PAS, IF, TDS

Mechanical Testing

- Inactive: TMF, HCF, LCF, impact, tensile, creep, hardness, $\mu$-hardness, furnaces, hydrogenation facility, DIC.
- Active: Tensile, LCF, n-intender, small punch, drop tower, in-situ irradiation creep.

Beam Line Techniques

- SLS: EXAFS, XAS, XRD, in-situ testing with $\mu$-LD, …
- SwissFEL
- SINQ: STIP, ND, residual stress, SANS, n-radiography, …

Hot Laboratory (AHL)

- LA ICP-MS, EPMA, SIMS
- Active metallography & sample preparation
- $\gamma$-spectrometry, fission gas analyzer
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Analysis Tools – Connection Through FIB

Spallation Materials

Hot Cells

Preparation box

Hotlab

Mechanical Testing

Hotlab-FIB

Microscopy / APT

Beam Line Techniques

nm - μm - mm
Analysis Tools – Connection Through FIB

Spallation Materials

Hot Cells

Preparation box

Hotlab-FIB

Hotlab

nm - μm - mm

Mechanical Testing

Microscopy / APT

Beam Line Techniques
Preparation box

- Under commissioning
- Ready end of 2017
- Up to 200-300 mSv/h (cumulative)

- Radioactive sample
  - cleaning
  - preparation for mechanical testing
  - preparation for FIB
  - ...

Shielding: 25 t (door 6.5 t)

- Under commissioning
- Ready mid 2018
Shielded FIB

Isolation of small regions for investigation

← • special regions, spatial resolution
• regions addressed by ion irradiation
• special features
• special sample geometry (TEM, pillars, ...)
• activity reduction
→ direct link HOTLAB - Synchrotron / FEL / university / ...
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In situ LBE SSRT testing on HT760

- **TOTAL ELONGATION (%)**
  - **TEST TEMPERATURE (°C)**

  - In Ar
  - In LBE
Embrittlement of FM Steels
(high He production)
Understanding of microstructure development, e.g. formation of precipitates

- APT (at ETH Zürich)
- Synchrotron light (at PSI West)
electric field is induced at the apex of a tip

\[ E \approx 10...30 \frac{V}{nm} \]

mass to charge ratio of the ions allows to determine their chemical nature

\[ \frac{m}{n} = 2 \cdot e(V_0 + V_p) \cdot \left( \frac{t}{L} \right)^2 \]

Number of atoms

![Mass spectrum of Fe-Cr alloy](image)

30 × 30 × 100nm³

few Mega atoms
APT – Sample prep.

Activity for a TEM sample: $10^6$ Bq

Activity for APT sample: $1.4 \cdot 10^{-3}$ Bq
ATP measurement - calculation

Solid spallation products content, APT result

<table>
<thead>
<tr>
<th></th>
<th>F82H</th>
<th>MANET II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dose</td>
<td>11.0 dpa</td>
<td>20.3 dpa</td>
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<tr>
<td>Ti, appm</td>
<td>570 ± 30</td>
<td>800 ± 10</td>
</tr>
<tr>
<td>Sc, appm</td>
<td>50 ± 30</td>
<td>90 ± 20</td>
</tr>
<tr>
<td>Ca, appm</td>
<td>240 ± 40</td>
<td>370 ± 10</td>
</tr>
</tbody>
</table>
APT Results

F82H, 20dpa, 350° C

C-enriched clusters:

- $C_C > 2\text{at}\%$
- $C_T > 5\text{at}\%$

- $C_V > 1\text{at}\%$
- $C_W > 4\text{at}\%$
- $C_{Cr} > 15\text{at}\%$
Experiments at SLS

**New work:** campaign using synchrotron light at Swiss Light Source in November
→ Better understanding or precipitates being formed due to irradiation
  e.g. with EXAFS for near range structure
Research on materials exposed to heavy irradiation (e.g. STIP)
  ... special effects due to He and other spallation products
  ... for basic understanding we need advanced research methods
  ... preparation box and FIB allow
direct transfer to and
usage of more facilities