Ion-irradiation induced degradation of thermo-mechanical properties of carbon-based materials

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Summary

- Materials irradiation facility at GSI
- Irradiation experiments: online and post-irradiation evaluation
- Radiation-induced thermal diffusivity degradation in graphite
- Nanoindentation investigation of mechanical properties of irradiate carbon materials
- Fatigue tests using nanoindentation
- First online creep tests on ion-irradiated carbon materials
Materials irradiation facilities at GSI
Beamlines for material research irradiation at GSI

**UNILAC**
- Ion Sources
- M-Branch
- 100 m

**SIS**
- SIS 18 beam dump
- E up to 1 GeV/u
- Range: cm

**Cave A**
- E 100-300 MeV/u
- Range: mm-cm
- Beam spot: 4 mm² to 25 mm² with scanning

**UNILAC beamlines**
- E: 3.6-11.4 MeV/u
- Range: 40-120 µm
- Beam spot area: 10x10 mm to 50x50 mm

**Ion Sources**
- p, Ar, Au, Pb, U
### UNILAC: beam parameters

#### Typical Energies

<table>
<thead>
<tr>
<th>Energy (MeV/u)</th>
<th>3.6</th>
<th>4.8</th>
<th>5.6</th>
<th>8.6</th>
<th>11.4</th>
</tr>
</thead>
</table>

#### 50 Hz Mode (Penning, ECR)
- **50 Hz**
- **5 ms** length of macropulse

#### High-current Mode (MEVVA source)
- **1-2 Hz**
- **100-200 µs** length of macropulse

#### Waveform Diagrams
- **50 Hz Mode**
  - 5 ms
  - 15 ms

- **High-current Mode**
  - 100 µs
  - 1 s
Thermal camera monitoring of sample temperature

High duty cycle

Low duty cycle
M-branch irradiation facility at GSI

In situ experiments

- energies close to Bragg peak:
  - to maximize energy deposition and damage
  - to avoid activation
- online and in situ monitoring: video camera, fast IR camera, SEM, XRD, IR spectroscopy

\[ \text{dE/dx} \sim Z_{\text{eff}}^2 (\text{ion}) \cdot Z(\text{target}) \]

SRIM code

ion species ..C...Xe...U

flux: up to \(10^{10}\) ions/cm\(^2\) s
Irradiation experiments at M3-branch, UNILAC, GSI

- $^{238}\text{U}$, 1.14 GeV, 0.5 ms, 0.6 Hz, $4 \times 10^9$ ions/cm$^2$ s
- $^{208}\text{Bi}$, 1 GeV, 0.5 ms, 3.4 Hz, $1.2 \times 10^9$ ions/cm$^2$ s
- $^{197}\text{Au}$, 945 MeV, 2 ms, 40 Hz, $4 \times 10^9$ ions/cm$^2$ s
Irradiation experiments
- online
- post-irradiation evaluation
Thermal properties degradation - postirradiation evaluation

fluences: $1 \times 10^{11}, 1 \times 10^{12}, 1 \times 10^{13}, 5 \times 10^{13}/1 \times 10^{14}$ i/cm$^2$ at fluxes $\sim 5 \times 10^9$ i/cm$^2$s

Samples for LFA: Isotropic graphite and flexible graphite
  - classical transmission measuring geometry
  - in-plane measuring geometry
Ion-induced thermal diffusivity degradation of graphite

Comparison U vs Xe irradiation
graphite vs flexible graphite

Flexible graphite

Isotropic graphite

U, 4.8 MeV/u
Xe, 8.6 MeV/u

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Online monitoring of thermal properties degradation

fluences: significant increase of experimental points number due to online capabilities i/cm²
at fluxes \( \sim 5 \times 10^9 \) i/cm²s

• Thermal conductivity degradation monitoring (on-line using thermal camera: estimation of time constant at cooling)
  • Cu-CD, Mo-Gr: 2 orientations, CFC: 2 orientations (U, Bi)

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Thermal camera monitoring of sample temperature during cooling

Temporal evolution of maximum temperature in irradiated samples

Cooling time at the beginning of irradiation

Cooling time at a dose of $1 \times 10^{13}$ ions/cm$^2$
Post-irradiation tests

- Samples for off-line tests: U, Bi, Au, Xe
- Isotropic graphite, low density graphites: foams and flexible graphite grades, CFC: 2 orientations

Microstructural characterization:
- Raman spectroscopy,
- SEM

Mechanical properties:
- Nanoindentation,

Electrical properties:
- 4-point probe resistivity measurements
Mechanical properties degradation-nanoindentation

investigations of hardening and E modulus change of irradiated layers

high temperature

Impact: fatigue damping

Courtesy LOT Quantum Design

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Mechanical behaviour of irradiated isotropic graphite

Evolution with accumulated dose:
- Hardness
- Young modulus

**isotropic graphite, $^{197}$Au, 4.8 MeV/u**

<table>
<thead>
<tr>
<th>Hardness /GPa</th>
<th>Fluence /ions/cm²</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>$1.0\times10^3$</td>
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<tr>
<td>0.5</td>
<td>$2.0\times10^3$</td>
</tr>
<tr>
<td>1.0</td>
<td>$4.0\times10^3$</td>
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<td>$6.0\times10^3$</td>
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<th>Young modulus /GPa</th>
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<tr>
<td>0.0</td>
<td>$1.0\times10^3$</td>
</tr>
<tr>
<td>5.0</td>
<td>$2.0\times10^3$</td>
</tr>
<tr>
<td>10.0</td>
<td>$4.0\times10^3$</td>
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<tr>
<td>25.0</td>
<td>$1.0\times10^4$</td>
</tr>
<tr>
<td>30.0</td>
<td>$2.0\times10^4$</td>
</tr>
</tbody>
</table>

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Mechanical behaviour of irradiated CFC

Evolution with accumulated dose:

Hardness

- **Transversal U**
- **In plane U**
- **In plane Au**
- **Transversal Au**

Red. modulus /GPa

- **Transversal U**
- **In plane U**
- **In plane Au**
- **Transversal Au**
Radiation induced creep measurements on flexible graphite

Au, 4.8 MeV/u

no weight

weight on
Impact nanoindentation study of fatigue behaviour of irradiated isotropic graphite

Cube Corner:
- 5 mN load,
- 28 µm acceleration distance
Failure of graphite exposed to pulsed $^{238}$U beam

**Experiment**

- $5 \times 10^{14}$ i/cm$^2$
- $10^{14}$ i/cm$^2$
- $10^{13}$ i/cm$^2$
- $5 \times 10^{12}$ i/cm$^2$

```markdown
radiation damage $\Rightarrow$ swelling
stress waves $\Rightarrow$ compression
stress concentrators + fatigue $\Rightarrow$ crack
```

**FEM simulations**

<table>
<thead>
<tr>
<th>Graphite target / Pulse structure</th>
<th>Maximum compressive stress (MPa)</th>
<th>Maximum tensile stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>45 µm (single pulse)</td>
<td>-53.3</td>
<td>0.5</td>
</tr>
<tr>
<td>45 µm (double pulse)</td>
<td>-56.4</td>
<td>0.7</td>
</tr>
</tbody>
</table>

$^{238}$U, 1.14 GeV

1.5 $\times 10^{10}$ i/pulse

150 µs, 1 Hz
Conclusions and Outlook

- Ion irradiation induces:
  - early degradation of thermal diffusivity
  - hardening and increase in E modulus
  - fatigue resistance decrease
  - Creep!

dependent on dE/dX

Failure