Status of irradiation tests at BNL of LHC collimator materials

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CollUSM – August 26th 2016
Great news...

All capsules from last irradiation campaign (up to $> 2 \times 10^{20} \text{ p/cm}^2$) have been opened. At a first inspection:

All MoGr samples survived!
All samples MACROSCOPICALLY intact

*Courtesy of N. Simos*

Detailed microstructural analysis will follow in the next months.
Outline

- Introduction
- Review of irradiation campaigns at BNL
- Report on X-ray diffraction tests
- Next steps
Introduction – Radiation hardness

• Radiation-induced effects on collimator materials is main challenge for the design of HL-LHC
  ➔ May compromise the operation of the machine
  ➔ Material properties is of key importance

• Long and fruitful collaboration between CERN and BNL (USA):
  ➔ Exploring resilience/vulnerability of middle-to-high-energy proton beam during long term irradiation of a set of LHC collimator materials
Irradiation campaigns at BNL

- **2014:** 1ˢᵗ irradiation campaign:
  - 200 MeV proton irradiation at BLIP (up to $1.1 \times 10^{21}$ p/cm²)
  - Spallation neutrons from 112 MeV protons at BLIP
  - Tightly focused 28 MeV proton beam at TANDEM

  Materials irradiated:
  - Glidcop AL-15 (SCM Metals, USA)
  - Mo (Plansee, Austria)
  - CuCD (RHP Tech., Austria)
  - MoGr (Brevetti Bizz, Italy), 3ʳᵈ generation grade

- **2016:** 2ⁿᵈ irradiation campaign:
  - 200/160 MeV protons at BLIP (~ $2 \times 10^{20}$ p/cm²)

  Materials irradiated:
  - MoGr, 6ᵗʰ and 7ᵗʰ generation grades
  - CFC (current material of LHC primary and secondary collimators)
Irradiation facilities at BNL

- **BLIP:** irradiation up to 200 MeV proton or spallation neutrons from 112 MeV protons
- **Tandem van de Graaff:** Irradiation with 28 MeV proton for very localized damage
First campaign: 2014

MoGr demonstrated **serious damage** at high fluences ($1.1 \times 10^{21} \text{ p/cm}^2$)

**Damage threshold ?**
Differences from 2014:

- 4 steps in fluence (from $5 \times 10^{19}$ to $> 2 \times 10^{20} \text{ p/cm}^2$)
- 3x MoGr grades (6th-7th generation) + CFC

**Experimental layout**

**Phase 1:**
- 6 capsules MoGr:
  - 3x MG-6530Aa (6th)
  - 3x MG-6403Ga (7th)
- 1 capsule CFC (3 layers)

**Phase 2:**
- 4 capsules MoGr:
  - 2x MG-6530Aa (6th)
  - 1x MG-6403Ga (7th)
  - 1x MG-6403Fa (7th)

Failure of isotope target downstream
Details of 2\textsuperscript{nd} irradiation campaign

<table>
<thead>
<tr>
<th>N. capsule</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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<tbody>
<tr>
<td>Material</td>
<td>MG-6403Ga</td>
<td>MG-6530Aa</td>
<td>MG-6403Ga</td>
<td>MG-6530Aa</td>
<td>MG-6403Ga</td>
<td>MG-6530Aa</td>
<td>CFC</td>
<td>MG-6403Fc</td>
<td>MG-6530Aa</td>
</tr>
<tr>
<td>Sample layers in capsule</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3</td>
<td>1</td>
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<tr>
<td>N. samples L (41x4x4 mm)</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>16</td>
<td>6</td>
<td>6</td>
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<tr>
<td>N. samples L (20x4x4 mm)</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>16</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>N. samples T (20x4x4 mm)</td>
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<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>6</td>
<td>24</td>
<td>6</td>
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Phase 1 = 200 MeV proton irradiation (Part 1) - end April 2016

<table>
<thead>
<tr>
<th>Irradiation time</th>
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<tbody>
<tr>
<td>Status of START</td>
<td>IN IN IN IN IN IN OUT OUT</td>
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<tr>
<td>Achieved fluence [p/cm²]</td>
<td>5.0E+19</td>
</tr>
<tr>
<td>Beam current (peak) [µA]</td>
<td>265</td>
</tr>
<tr>
<td>Integrated beam current [µA h]</td>
<td>20000</td>
</tr>
<tr>
<td>Estimated DPA</td>
<td>1.3E-2</td>
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<td>Status of END</td>
<td>OUT OUT IN IN IN IN OUT OUT</td>
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Phase 1 = 200 MeV proton irradiation (Part 2) - May 2016?

<table>
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<th>Irradiation time</th>
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<tbody>
<tr>
<td>Status of START</td>
<td>OUT OUT IN IN IN IN IN IN</td>
</tr>
<tr>
<td>Achieved fluence [p/cm²]</td>
<td>&gt; 1E+20</td>
</tr>
<tr>
<td>Beam current (peak) [µA]</td>
<td>-</td>
</tr>
<tr>
<td>Integrated beam current [µA h]</td>
<td>40000</td>
</tr>
<tr>
<td>Estimated DPA</td>
<td>-</td>
</tr>
<tr>
<td>Status of END</td>
<td>OUT OUT IN OUT OUT OUT OUT</td>
</tr>
</tbody>
</table>

Phase 2 = 160 MeV proton irradiation - end July 2016

<table>
<thead>
<tr>
<th>Irradiation time</th>
<th>8 days</th>
<th>8 days</th>
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</thead>
<tbody>
<tr>
<td>Status of START</td>
<td>OUT OUT</td>
<td>OUT OUT</td>
</tr>
<tr>
<td>Achieved fluence [p/cm²]</td>
<td>~ 2E+20</td>
<td>~ 2E+20</td>
</tr>
<tr>
<td>Beam current (peak) [µA]</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Integrated beam current [µA h]</td>
<td>40000 +</td>
<td>?</td>
</tr>
<tr>
<td>Estimated DPA</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Status of END</td>
<td>OUT OUT</td>
<td>OUT OUT</td>
</tr>
</tbody>
</table>

Ended earlier than expected due to failure of one isotope production target downstream
Inspection of irradiated capsules

April ‘16
Phase 1
200 MeV

May ‘16
Phase 1
200 MeV

July ‘16
Phase II
160 MeV

August ‘16
Microstructural
tests at NSLS II

First 2 MoGr capsules opened:
(fluence: \(\sim 5 \times 10^{19} \text{ p/cm}^2\))

No macroscopic damage!

All capsules opened:
(max fluence: > \(2 \times 10^{20} \text{ p/cm}^2\))

No macroscopic damage!

Courtesy of N. Simos

Courtesy of N. Simos
National Synchrotron Light Source

NSLS II

Most advanced synchrotron facility in the world.

Mandate: study of material properties and functions with nanoscale resolution.

Tested performed in 2014

NSLS I

- 3 GeV electron storage ring, produces x-rays up to 10000 times brighter than NSLS I
- 16 beamlines currently opened

We used 28-ID beamline for XPD

ColUSM – 26.08.2016
28-ID beamline at NSLS II

- **Monochromatic X-ray beam**
  - Energy = 67 keV

- **2-dimensional X-ray powder diffraction studies** to investigate the effect of the irradiation on the structural integrity of the materials

- Implementation of a **4-point bending load** applied in-situ on sample:
  - More information on structural behaviour
Constructive interference: $n\lambda = 2d \sin \theta$

**Bragg’s law**

**Single crystal** diffracts in discrete directions

**Polycrystalline material** creates series of diffraction cones

Two dimensional (2D) XPD system is a diffraction system which allows:

- 2D x-ray detection
- 2D image processing
- 2D diffraction pattern manipulation

Main advantages w.r.t. 1D-XPD:

- Measurement area no limited to diffraction plane
- Diffraction rings (Debye rings) measured simultaneously
2-dimension XPD at 28-ID

Aquisition 2D diffraction pattern (0.25 s acquisition → FAST)

Integration in 2-theta angle → info on interplanar distance

Gaussian beam
Beam current = 250 nA
Beam spot size: 0.5 x 0.6 mm

Irradiated MG-6530Aa (~5E19 p/cm²)

Metals
Composite materials

beam spot
2-dimension XPD at 28-ID: stress-strain scanning

Understand effects on material microstructure when in-situ stress applied:
→ Change of interplanar distance due to stress
→ Peak shift in XPD spectrum

XDP with increasing in-situ load applied on sample
Summary of material tested

...at NSLS II (2016):

<table>
<thead>
<tr>
<th>Material</th>
<th>XPD</th>
<th>XPD (under load)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>pristine</td>
<td>irradiated</td>
</tr>
<tr>
<td></td>
<td>pristine</td>
<td>irradiated</td>
</tr>
<tr>
<td>from 2016 irradiation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CFC</td>
<td>L + T</td>
<td>(scan 3 pos.)</td>
</tr>
<tr>
<td>MG-6530Aa</td>
<td>L + T</td>
<td>2x</td>
</tr>
<tr>
<td>MG-6403Ga</td>
<td>L + T</td>
<td>hot</td>
</tr>
<tr>
<td>MG-6403Fc</td>
<td>L + T</td>
<td>hot</td>
</tr>
<tr>
<td>from 2014 irradiation</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MG-111E</td>
<td></td>
<td>4x</td>
</tr>
<tr>
<td>CuCD</td>
<td></td>
<td>(short irrad.)</td>
</tr>
<tr>
<td>Mo</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Glidcop</td>
<td></td>
<td></td>
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<tr>
<td>2D-CFC</td>
<td></td>
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</tr>
</tbody>
</table>

Critical handling due to high activation, at the limit to access NSLS II

1x long sample
2x short samples
1x sample from center of capsule (saw p+n irradiation)

For comparison or not done in 2014

...at NSLS (2014):

<table>
<thead>
<tr>
<th>Material</th>
<th>XPD-X17A - monochromatic</th>
<th>XPD-X17B1 - &quot;white&quot; beam</th>
<th>XPD-X17B1 - load</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>pristine</td>
<td>BLIP</td>
<td>TANDEM</td>
</tr>
<tr>
<td>from 2014 irradiation</td>
<td></td>
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<td></td>
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<tr>
<td>MG-111E</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CuCD</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Mo</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Glidcop</td>
<td></td>
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</tbody>
</table>
Conclusions and Outlook

- Second irradiation campaign at BLIP completed in July 2016
- Irradiated: 3 latest grades of MoGr + CFC
- All capsules opened in hot cell:
  → none shows macroscopic damage up to $2 \times 10^{20} \text{ p/cm}^2$
- X-ray Diffraction experiment successfully completed at NSLS II
  → intensive data analysis on-going

What’s next?

- Comprehensive analysis of XPD patterns
- Microstructural characterization of pristine samples at CFN
- Mechanical tests of pristine/irradiates samples
Thank you for your attention

Greetings from 28-ID beamline at NSLS II

Acknowledgements to:

• material supplying company: Brevetti Bizz (Italy)
• the very kind and helpful 28ID beam line staff: E. Dooryhee, S. Ghose and J. Trunk
Backup slides
## Irradiation tests on MoGr

### 3rd generation
- **MG 1110-E**
  - Density: 3.7 g/cm³
  - Status: Not fully molten
  - Composition: 20% Mo, 40% C-fibers (long+short)

### 4th generation
- **MG 3110-P**
  - Density: 2.67 g/cm³
  - Status: Fully molten
  - Composition: 20% Mo, 40% C-fibers (long+short)

### 5th generation
- **MG 5200-S**
  - Density: 2.65 g/cm³
  - Status: Fully molten
  - Composition: 7.2% Mo, 46.4% C-fibers (long+short)
  - Annealing cycles: 1150°C, 1300°C

### 6th generation
- **MG 6400-U**
  - Density: 2.63 g/cm³
  - Status: Fully molten
  - Composition: 4.5% Mo, 5% C-fibers
  - Annealing: 1800°C

- **MG 6530-Aa**
  - Density: 2.5 g/cm³
  - Status: Fully molten
  - Composition: 4.5% Mo, 5% C-fibers (long only)
  - Annealing: 1900°C

### 7th generation
- **MG 6541-Aa**
  - Density: 2.49 g/cm³
  - Status: Fully molten
  - Composition: 4.5% Mo, 0.05% Ti, 5% C-fibers (short only)
  - Annealing: 1900°C

- **MG 6403-Ga**
  - Density: 2.52 g/cm³
  - Status: Fully molten
  - Composition: 4.5% Mo, 0.2% Ti
  - NO C-fibers
  - Annealing: 1900°C
200 MeV proton irradiation (8 weeks):
- **Glidcop AL-15** *(SCM Metals, USA)*
- **Molybdenum** *(Plansee, Austria)*
- **MoGr** *(Brevetti Bizz, Italy)*
- **CuCD** *(RHP Tech., Austria)*

Note: 200 MeV proton irradiation performed at BLIP also on CFC and Glidcop in 2012 (US-LARP collaboration).

**Spallation neutrons from 112 MeV protons:**
- **CuCD** *(RHP Tech., Austria)*
- Graphite (also interesting for collimators)
...and at TANDEM

- Tightly focused 28 MeV proton beam (1.5 x 1.75 mm beam core + tail)
- Primary beam intercepted by Mo sample (high-Z material) to maximize secondary particle spectrum
- Spallation neutron field produced by primary protons used to irradiate MoGR, CuCD and Glidcop (2x2x42 mm)
Energy Dispersive X-Ray Diffraction at X17B1 beamline

- Continuous **white** radiation
- Energy **up to 200 keV** (bulk analysis)
- **Fixed angle** 2\(\theta\) (good for in-situ measurements)
- **Energy distribution** of scattered photons analyzed by a semiconductor detector
- Multichannel analyzer to determine pulse height
EDXRD at X17B1 for simultaneous phase and strain mapping

Load on sample placed in 4-points bending fixture

Discretized scan along sample thickness
- beam spot = 20 μm x 1 mm
- 20 μm step

Discretized scan along sample length
- beam spot = 20 μm x 0.5 mm
- 0.5 mm step