CERN2 Capsule inspection update

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Under the supervision of:
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With the collaboration of STI/BMI

On behalf of the RaDIATE collaboration
1. Irradiations at BLIP: setup and aims
2. The CERN2 capsule: contents and irradiation
3. Global status of the project
4. Opening the capsule and extracting the specimens
5. Results of the visual inspection
6. Next steps: planning the adhesion test
Irradiations at BLIP

- Organized and carried out in the framework of the RaDIATE (Radiation Damage In Accelerator Target Environments)
- CERN has been a key member since 2012, along with FNAL, PNNL, U. of Oxford, ESS and other laboratories
- 5th in-person collaboration meeting held at CERN in Dec. 2018.

**BLIP – Brookhaven Linear Isotope Producer at BNL**

Material irradiation in tandem and upstream of isotope targets:

- Primary proton energy: up to 200 MeV
- RaDIATE Target array needs to be optimized for each run to deliver a beam that is appropriate for isotope production
- POT ~ 1E21
- Peak DPA: 0.88 (Ta2.5W, CERN2 capsule), 1.2 (Ir, HighZ capsule)
- Rastered beam profile to achieve more constant irradiation profile
- Irradiation length O(months)
- DPA and Heappm/DPA levels representative of conditions achieved in BIDs

1. Courtesy K. Ammigan
2. Courtesy J. Canhoto
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BLIP – Brookhaven Linear Isotope Producer at BNL

- Primary proton energy: up to 200 MeV
- Material irradiation in tandem and upstream of isotope targets:
  - BNL has recently modified irradiation setup and aims (stop in production of Sr82)
  - Consequent increase in beam time costs (~3.5x), subject under negotiation with FNAL
  - Next irradiation slot is Jan. 2022

- Rastered beam profile to achieve more constant irradiation profile
- Irradiation length O(months)
- DPA and Heappm/DPA levels representative of conditions achieved in BIDs

1 Courtesy K. Ammigan
2 Courtesy J. Canhoto
Organisation of an irradiation at BLIP

1. **Definition of irradiation run aims and target assembly**

FLUKA Studies

Comparison with the conditions achieved in the BID and BNL proton energy budget

ANSYS Studies

1. Courtesy K. Ammigan
Organisation of an irradiation at BLIP

1. **Definition of irradiation run aims and target assembly**

   [Diagram showing FLUKA and ANSYS studies, with a note on comparison with conditions achieved in BID and BNL proton energy budget]

2. **Capsule and material specimens fabrication**

   - Capsule Cover
   - Filling with specimens
   - EBW

   [Images of capsule cover and filling with specimens]

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\(^1\)Courtesy K. Ammigan

23/04/2021
Organisation of an irradiation at BLIP

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Comparison with the conditions achieved in the BID and BNL proton energy budget

3. Shipment of capsule to BNL and installation in the beamline

Capsule fitted in capsule holder

Basket assembly (2.5 mm water gap)

Target box assembly

Capsule Cover

Filling with specimens

EBW

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1. **Definition of irradiation run aims and target assembly**

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2. **Capsule and material specimens fabrication**

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3. **Shipment of capsule to BNL and installation in the beamline**

4. **Irradiation and subsequent cooldown – O(\text{year})**

   - Capsule fitted in capsule holder
   - Basket assembly (2.5 mm water gap)
   - Target box assembly

CERN2 Capsule ➔
- Design started in second half of 2017
- PIE on some of the specimens was only possible starting 2 years after

\(^1\text{Courtesy K. Ammigan}\)
Stainless steel welded capsule containing:
- 40x Ta2.5W specimens
- 20x Monocrystalline Si specimens
- 4x CFC specimens
- 4x MoGr specimens
- Panasonic graphite foils
- Graphite fillers

4x MoGr specimens:
- Material: Molybdenum carbide grade Na-8304-Gb
- Dimensions: 20x20x1.35 mm (pre-irradiation)
- 8 µm-thick pure molybdenum coating
- Serial number clearly engraved on each specimen
- Central interest of the activity at this stage
Irradiation carried out in Jan-March 2018

Organised in the framework of the RaDIATE collaboration

Rastered beam profile to have a more constant irradiation profile

2.81 x 10^{21} POT in 33d @158 mA average

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### Computed Temperature profile

- **PNG Foil T=290°**: Ta2.5W T=270°C
- **Si T=320°C**: CFC T=330°C
- **MoGr T=250°C**

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### Primary MoGr collimators

<table>
<thead>
<tr>
<th></th>
<th>Left</th>
<th>Right</th>
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</thead>
<tbody>
<tr>
<td>POT</td>
<td>~1E17</td>
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</tr>
<tr>
<td>DPA (peak)</td>
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<tr>
<td>He appm</td>
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<tr>
<td>He appm/DPA</td>
<td>600-1000</td>
<td>600-1000</td>
</tr>
</tbody>
</table>

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Global Status of the project

Project delays mainly due to:
- Coordination of shielding container design
- Shipment acceptance due to COVID19-situation
- Absence of on-site personnel at BNL before Sept. ‘20
- Documentation harmonization for Type A drum in course of US regulation changes

Project has respected (or is ahead of) schedule since capsule arrival at Framatome
Capsule opening procedures

**PNNL opening setup**

- Blade cuts along outer perimeter of window
- **Reliability problems**: machine was misaligned on multiple occasions (specimens inside Si capsule compromised)
- **Cut performed by pressing on capsule** → High stress on specimens, difficult to separate a priori the effects of irradiation and cut during PIE

**Framatome CNC milling**

- **High-precision milling**: performed on outer edge of capsule window, opposite the EBW
- Reduced stresses on specimens, discharged mainly on inner capsule body
Opening the capsule and extracting the specimens

Joint HL-LHC & RaDIATE Collaboration Project

Opening of a BLIP-irradiated capsule (Brookhaven National Laboratory) containing material for HiLumi Project Collimation and advanced refractory materials for Targetry at CERN.

Done 2021 in the Radiochemistry and Hot Cells Laboratory of Framatome GmbH in Erlangen.

Video available at EDMS 2563586
Summary of the extraction

- All specimens have been extracted and, besides two Si specimens, they are all in great conditions.
- Milling process on the outer edge of the window minimized stress on specimens.
- Good behaviour of Panasonic Graphite (PNG) sheets. Promising for further applications.
- Measured dose rates:
  - Mo-coated MoGr M12
    - Contact: 3.4 mSv/h
    - @20 cm: 69 µSv/h
  - Mo-coated CFC, specimen C06
    - Contact: 195 µSv/h
    - @20 cm: 5 µSv/h
**Visual Inspection results**

**Requirements set out in the Technical Specification**

- Pictures of the specimens must be taken to show possible **macroscopic damage** of the 4 Mo-coated MoGr specimens and 4 Mo-coated CFC specimens extracted from the CERN2 irradiated capsule.

  In particular, the investigation must assess:

  - Structural integrity of the specimens
  - Permanent deformation and geometry changes
  - Presence of crack on the coated and uncoated side
  - Macroscopic loss of coating adherence
  - Blistering in correspondence of the coating/uncoated surface

**Inspection Setup**

Pictures of the specimens acquired by means of an optical microscope integrated in a glove box/fume cupboard. The microscope enables acquisition of panoramic images by stitching together pictures from 20x to 2000x magnification.
Visual Inspection results – MoGr specimens

- **M12 Irradiated specimen**
  - Coated face
  - Uncoated face
  - **No macroscopic damage** is visible in any of the pictures
  - This is valid for every MoGr and CFC specimen
  - Uncompressed images available at [EDMS 2539802](#)

- **M13 Unirradiated specimen, extracted from spare capsule**
  - Further inspection possibilities:
    - Taking pictures at an angle to better assess presence of asperities → not possible because of small depth of field of optical microscope
    - **SEM inspection of coating** → Consistent with optional activity 6 (21.5 k€ from bid). Framatome offered to conduct a **partial SEM inspection** on one specimen to have a sample of the results free of charge.
      - Need to provide information on previous SEM-analyses of Mo coatings for analysis setup.
      - Results will be submitted to WP5 to assess interest in option activation
Visual inspection – CFC specimens

- **C06 Irradiated specimen**
  - Coated face
  - Uncoated face
  - Side

- No macroscopic differences in specimen status between irradiated and un-irradiated specimens
- Acquisition of pictures of un-irradiated CFC specimens with the same setup is underway
Next Steps: Adhesion test

- **Purpose of the test** → Evaluation of the adherence of the Mo coating to the substrate after irradiation and comparison with the results observed for the non-irradiated material

- **Subject of the test** → **Two** of the four Mo-coated irradiated MoGr specimens (CERN will communicate which two)

- **Test performed using a PAT handy adhesion tester**

- **Aspects of interest:**
  - Value (MPa) at which the dolly detachment from the specimen occurs
  - Location of the rupture (in the coating-bulk interface or in the bulk?)
  - Pictures of the fracture surface

- A spare Mo-coated MoGr specimen will be supplied by CERN for benchmarking of test procedures and comparison of test results with the irradiated specimens
Preparation of the adhesion test

1. Write and submit to CERN the adhesion test procedure. O(10 days)
2. After CERN’s approval, demonstrating it on spare MoGr specimens
3. After second approval, go ahead with the two adhesion tests (mid-May)

⚠ Potentially offset position of beam impact
   • Relevant for selection of the two MoGr specimens on which the adhesion test will be performed
   
   ▪ Actual position of beam impact investigated by placing a GAFchromic™ film on the capsule cover.
   ▪ Gamma decay develops the film and reveals beam impact position

• Film will be shipped from CERN in the coming week
• A fee may be charged by Framatome (TBD)
• Will update DPA and FEA simulations based on measurement
Baseline and Optional Activities

- **Baseline activities of the contract**
  1. Shipment of the capsule from BNL (USA) to Framatome (DE)
  2. Capsule opening and specimen extraction
  3. Examination on the specimens:
     a) Visual inspection on 4 Mo-coated MoGr and 4 Mo-coated CFC specimens
     b) Test adhesion of the coating on 2 Mo-coated MoGr specimens

- **Optional activities**

<table>
<thead>
<tr>
<th>#</th>
<th>Optional Activity</th>
<th>Destructive?</th>
<th>Cost (k€)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Evaluation of the density</td>
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<tr>
<td>2</td>
<td>Measurement of the electrical conductivity of bulk and coating</td>
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<td>N/Aa)</td>
</tr>
<tr>
<td>3</td>
<td>Measurement of the thermal diffusivity of the bulk material at room temperature</td>
<td>ND</td>
<td>NPb)</td>
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<tr>
<td>4</td>
<td>Microstructural Investigation of the bulk and of the coating with X-ray Diffraction</td>
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<td>5</td>
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<tr>
<td>5</td>
<td>Microstructural Investigation of the bulk with Raman spectroscopy</td>
<td>ND</td>
<td>N/Ac)</td>
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<tr>
<td>6</td>
<td><strong>Microstructural investigation of the bulk/coating interface</strong></td>
<td>S/D</td>
<td>21.5</td>
</tr>
<tr>
<td>7</td>
<td>Measurement by micro-indentation of the elastic modulus of the coating</td>
<td>D</td>
<td>6.3</td>
</tr>
<tr>
<td>8</td>
<td>Measurement of the elastic modulus of the bulk</td>
<td>D</td>
<td>6.3</td>
</tr>
</tbody>
</table>

CERN has up to 1 month after the acceptance of the final report (~end of July) to activate optional activities

a), c) Not available at Framatome’s site
b) Not possible at room temperature
Conclusions

- CERN2 Capsule irradiation carried out in Jan-March of 2018
- 2. $81 \times 10^{21}$ POT. FLUKA-computed values of DPA and He ppm/DPA \textbf{comparable with values achieved in primary MoGr collimators}

- Delay in the project mainly due to Covid-related obstacles in capsule shipment to Framatome
- Project has respected schedule ever since the arrival of the capsule at Framatome’s site

- Capsule extraction carried out by milling on the outer edge of capsule window $\rightarrow$ \textbf{no damage to specimens}

- Visual inspection has \textbf{not revealed any macroscopic difference} between irradiated and un-irradiated specimens, even with the same lighting conditions
- SEM-inspection of the bulk-coating interface could give more detailed information. Cost 21.5 k€. Framatome will perform a partial SEM-inspection as a sample of results free of charge

- \textbf{Adhesion test will be performed in mid-May} on two Mo-coated MoGr specimens
- Test procedure benchmarked on spare MoGr specimen
- Position of beam impact assessed with GAFchromic foil, important for choice of the two specimens

- CERN has up to ~end of July to activate optional activities
BACKUP SLIDES
Microstructural investigation of the bulk/coating interface

This measurement shall be performed on the two MoGr irradiated specimens that are left after the adhesion test is performed. The test shall follow the following steps:

1) Investigation of the coating morphology at microscopic level with Scanning Electron Microscopy (SEM) at different magnifications (25X, 100X, 400X, 1600X, 6400X, 25600X). These measurements shall take place in the centres of each quarter of the specimens’ main surfaces, as identified in Figure 3;

2) Focused Ion Beam (FIB) shall be used to produce cross sectional millings in the areas investigated in the previous step;

3) The SEM shall take pictures of the cross-sectional area at different magnifications (1600X, 3200X, 6400X);

4) Energy Dispersive X-rays (EDX) mapping (minimum 5 points) shall be performed on each of the cross section cuts to determine the elemental composition of the coating and the cross section. SEM images shall be attached with indications of every point in which the EDX spectrum was measured.

For this investigation, CERN suggests the use of the following settings:

a) For the FIB milling, an accelerating voltage of 30 keV and a current of 15 nA, followed by a fine polishing at 3 nA;

b) For the EDX mapping, a voltage of 20 kV and beam current of 3.9 nA.