Microscopy investigation of the surface behaviour of different materials after $\text{H}^-$ irradiation in different conditions

A. T. Pérez Fontenla (EN-MME-MM) on behalf of the spare RFQ Project team
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| 2. | Additional study on collimator |
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Linac4 RFQ in-situ inspection

L4 tunnel

Optical microscopy at vane front face

Endoscopy at front face and vacuum port

Worm-like features

BKV craters
Linac4 RFQ in-situ inspection

- L4 tunnel
- Optical microscopy at vane front face
- Endoscopy at front face and vacuum port

CLIC TD24_R05 (tested in XBox_1)
CLIC Crab Cavity (tested in XBox_2)

Image presented in HG2016 courtesy of Enrique Rodriguez
Additional study on Cu-OFE collimator

Collimator/mark fabricated from cold rolled Cu-OFE plate (3 mm thickness) was installed and tested at $1.7 \times 10^{19}$ H ions at 45 keV (number of total impacting ions calculated from the electrical signal).

Microscope inspection of the Cu surface around the square was performed before and after testing.

Halo perceptible during visual inspection
Approximate diameter $\approx 20$ mm
Estimated area $\approx 2.5$ cm²
Additional study on Cu-OFE collimator

Comparison before and after testing pointed out the presence of blisters.

Before testing

Micrometric protrusions/blister?
Only appreciable at SEM with >1k×
Additional study on Cu-OFE collimator

Removed volume: $10 \times 8 \times 5 \, \mu m^3$

Superficial layer of Cu (at $\approx 0.5 \, \mu m$ depth from the surface) heavily affected by pores

Similar features are reported in the bibliography due to penetration of relatively insoluble gases (hydrogen)
Proposed studies

- A list of candidate materials was carefully defined as alternative to Cu-OFE for manufacturing of a future RFQ with better performance.

<table>
<thead>
<tr>
<th>Material</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>Cu-OFE</td>
<td>Forged and annealed (current base material)</td>
</tr>
<tr>
<td>CuCr₁Zr</td>
<td>Forged, solution annealed and aged</td>
</tr>
<tr>
<td>Cu₉₈Be₂</td>
<td>Hard temper (precipitation hardening)</td>
</tr>
<tr>
<td>Nb</td>
<td>RF purity (RRR300)</td>
</tr>
<tr>
<td>Ta</td>
<td>Melted quality Annealed</td>
</tr>
<tr>
<td>Ti₆Al₄V</td>
<td>Premium grade β-forged</td>
</tr>
</tbody>
</table>

- The materials were selected based on:
  - Their usability for meter-long high gradient RF cavities and,
  - Their potential resistance to blistering and breakdown phenomena;

Relevant testing parameters:
- Radiation dose
- Ion energy
- Angle of incidence
- Temperature of the “target”

Relevant metallurgical aspects:
- Purity
- Grain size
- Pre-existing defects
- Mechanical properties
- Crystal orientation at surface
Proposed studies

- Combined H\(^-\) source test stand + pulsed DC:
  - Particle type H\(^-\) ions at 45 keV
  - Different incidence angles are possible
  - Electrodes of Ø80 mm and Ø60 mm

- Helsinki’s system:
  - Particle type H\(_2\)\(^+\) at 90 keV
  - Different incidence angles are possible
  - Sample holder ~ Ø100 mm → simultaneously 4 samples (30 mm x 30 mm)

<table>
<thead>
<tr>
<th>Combined H(^-) source + DC testing at CERN</th>
<th>Helsinki’s system</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu-OFE</td>
<td>Cu(_{10})Be(_2)</td>
</tr>
<tr>
<td>Tested</td>
<td>Manufacturing completed</td>
</tr>
<tr>
<td>~ Dose 1.0 x 10(^{19}) p/cm(^2)</td>
<td>1</td>
</tr>
</tbody>
</table>
Results: Cu-OFE electrode

After thermal treatment in vacuum at 650 °C for 2 hours
Cathode: After irradiation test with an estimated total dose of 1.2E19 p delivered during 40 hours (data courtesy of Giulia Bellodi).

Previous affected surface estimation on collimators test was 2.5 cm² and in the electrode 0.5 cm²

Central region

“C-shaped”

Outer ring

Image courtesy of Sebastien Bertolo
Results: Cu-OFE electrode

In the transition from irradiated spot to non-irradiated zones, some grains are more affected than others.

All the C-shaped area presents blisters.

Why C-shaped? Investigations are ongoing.
Cu-OFE electrode

Transition area

When observed at high magnification faceting of the Cu is noticeable.

The blisters present small variations in size diameter and morphology depending on the grain orientation.
Results: Cu-OFE electrode

Blistering during the growth and in many sites coalescence of two or three blisters is observable. Some of the blisters present openings (holes).

Opening most probably throw the cap thickness

Coalescence

Initial phase

EHT = 7.00 kV
WD = 6.4 mm
Signal A = SE2

Sample ID = Cu-OFE_
Anite Perez Fontenla
Date: 16 Oct 2020
Mag = 5.00 K X
Results: Cu-OFE electrode

The relation between the blisters location and crystallographic orientation was evaluated experimentally using the non-destructive technique Electron Back-Scattered Diffraction (EBSD) into the SEM.

Using the common color-codes for cubic materials, it can be observed that in general the blue and green grains (corresponding to crystal directions [111] and [101] respectively) were less affected by blisters than the red grains (corresponding to crystal direction [001]).
Results: Cu-OFE specimen (Helsinki)

Presence of blisters randomly distributed on the Cu-OFE surface was confirmed with both used doses in Helsinki (is not possible to correlate with grain orientations at the transition areas due to the surface roughness)

Size, density and shape of the blisters on the collimator are similar to the ones observed at the lower dose test region

In the case of higher dose, different features appeared (larger blisters?) in addition to the smaller blisters

Some blisters present holes/openings at the cap
Results: Cu-OFE specimen (Helsinki)

Nanopores observed in the collimator are only visible punctually in the affected layer on the Cu-OFE irradiated samples in Helsinki.

The blister cap thickness on the collimator is similar to the lower dose test in Helsinki and similar to some features in the higher dose (initiation/nucleation sites?).

Presence of larger blisters containing smaller events is confirmed and a carbon layer is visible;
Results: Cu-OFE specimen (Helsinki)

Cu-OFE Helsinki (4E^{18}p/cm^2)

The cap is thicker at the central part

Thinning due to plastic deformation
Results: CuCr$_1$Zr specimen (Helsinki)
Results: CuCr$_1$Zr specimen (Helsinki)

CuCr$_1$Zr Helsinki ($4\times 10^{18}$ cm$^{-2}$)

Thin layer

Aspect of fragile fracture at the blister base

~ 35 wt. % C

~ 10 wt. % C
Results: CuCr$_1$Zr specimen (Helsinki)

- Microstructure is visible
- The cap is thicker at the central part
- Thinning due to plastic deformation
- Pt deposit

CuCr$_1$Zr Helsinki ($4E^{18}p/cm^2$)

CERN Engineering Department
Results: Nb and Ti$_6$Al$_4$V (Helsinki)

**Nb Helsinki** \((4E^{18} p/cm^2)\)

**Ti$_6$Al$_4$V Helsinki** \((4E^{18} p/cm^2)\)
The in-situ test in Linac4 RFQ by optical means pointed out the presence of local damage on the Cu surface (BKD craters and “worm-like features”);

Further investigations with collimators/masks confirmed the presence of blisters induced by the interaction with the beam;

The size of those events differs but they could be connected → a deeper look to better understand the blister-BKD location relation is needed;

Combined testing (irradiation + pulsed DC system) was performed on RFQ base material (Cu-OFE). The presence of blister was confirmed and their aspect and location were characterized by SEM and EBSD;

In addition, other candidate materials for RFQ production were identified and are being studied in similar way in order to compare their performance and resistance to blistering and BKD phenomena;

The cost and leading time to fabricate and test at CERN large electrodes oriented us to look for testing alternatives and the preliminary results obtained with Helsinki’s system compared well with CERN studies in collimators/masks;

This flexible and reproducible system allowed us to test simultaneously Cu-OFE, CuCr$_1$Zr, Nb and Ti$_6$Al$_4$V specimens with two different doses. Nevertheless, some questions raised up (C layer, bigger blisters at higher doses/time, surface preparation…) → more tests needs to be performed;

So far, both cuprous materials tested presented blisters (in the case of the CuCr1Zr only at higher dose or on deformed areas) and Nb and Ti$_6$Al$_4$V were free of features even at higher doses;

It’s being a continuous learning process and it is important to mention the implication of a wide range of experts at CERN and Helsinki to evaluate the findings until now and the project support to hire a PhD student.
Thank you for your attention!
Extra slides
EDS comparison at the central region (SOI-5) and external region (SOI-7)
Extra slides
Extra slides
Faceting on the Cu surface is visible independently of the region. That is a consequence of the thermal treatment performed on the electrode before testing. Faceting is related with selective evaporation of atoms in non-energetically favourable crystal position thus the appearance varies depending on the grain orientation.

Mo rich inclusions are visible all over the Cu surface.
Very superficial features and are visible all over the Cu surface.
When observed at higher magnification:
When observed at higher magnification:

SOI-5: Central region

SOI-7: Outer ring region

SOI-2: C-shaped region
Comparison of Nb sample same location before and after irradiation (dose 4E18 H/cm^2)

Top view
Before irradiation

Top view
After irradiation

Nb surface present pitting after etching
Top view
After irradiation

Cross section
After irradiation
Extra slides

Lowers the cohesive strength of interfaces (e.g. “cortical layer”)