Recap of MoGr production: powder changes and their vacuum validation

F. Carra

With contributions from C. Accettura, A. Bertarelli, J. Guardia, G. Cattenoz, G. Bregliozzi.

ColUSM
CERN, Geneva
January 25th, 2021
Outlook

- Recap of graphite powders used during production
- Vacuum results per batch
- Lessons learnt and future studies
Graphite powders

- Nanoker adopted **Asbury 93002 powders** until batch 13 (one graphite lot could cover 70% of production needs)
- **Early September 2019**: new lot of 93002 (in principle identical!) procured by Nanoker, enough to complete production.
- **End of September**: 6 plates cracked during annealing. High vacuum pressure noticed in the chamber, bad sulphur smell.
- Nanoker powder analysis showed nonconformity in the 93002 powders, high level of impurity (>5% vs specification <4%). Message from Asbury: we cannot guarantee good reproducibility for 93002 powders.
- Proposal from Asbury to Nanoker: use 93004 powders: finer, higher purity (>99%), more reproducible → more expensive. Nanoker asked to replace the 93002 lot, and in parallel also bought enough 93004 powders to complete the production
- 93004 is a much more controlled and high-quality material, and together with Nanoker we decided to validate it by producing 4 taperings and measure density, electrical conductivity and UHV properties.
New graphite powders

<table>
<thead>
<tr>
<th></th>
<th>93004</th>
<th>93002</th>
<th>3260</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean (um)</td>
<td>5.23</td>
<td>20.12</td>
<td>35.0</td>
</tr>
<tr>
<td>SD (um)</td>
<td>2.53</td>
<td>11.91</td>
<td>27.2</td>
</tr>
</tbody>
</table>
Validation of 93004 powders – Batch 14

- **17th October 2019**: reception of the 4 taperings at CERN and standard approval process.

1. **Density** slightly higher than previous batches (2.57 g/cm³ vs 2.54-2.56 g/cm³), likely better compaction (*Action: analyse UHV*).

2. **Electrical conductivity** slightly lower (5-10%: expected, because more grain boundaries with smaller powders. Acceptable since we are producing TCSPM material, coating! *After coating → electrical conductivity is the same*).

3. **UHV (28th October)**: factor of 2 higher than previous uncoated batches. Still similar or lower than previous **coated** batches: before coating, tolerable, but close to the limits (EDMS 2315082). Higher air content.

4. **Thermomechanically and dimensional stability, springback**: material is significantly better!

- **Nanoker**: decision taken to continue with the production in parallel with validation, to avoid delays with planning → thanks!

### Specification

<table>
<thead>
<tr>
<th>Property</th>
<th>Specification</th>
<th>NB-8304Ng</th>
<th>NB-8404Ng</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density at 20°C</td>
<td>2.40 – 2.60</td>
<td>2.55</td>
<td>2.57</td>
</tr>
<tr>
<td>Specific heat at 20°C</td>
<td>&gt;0.6</td>
<td>0.63</td>
<td>0.65</td>
</tr>
<tr>
<td>Electrical conductivity at 20°C</td>
<td>&gt;0.90</td>
<td>0.80-0.87</td>
<td>0.75-0.80</td>
</tr>
<tr>
<td>Thermal Diffusivity 20°C/at 300°C</td>
<td>&gt;390/120</td>
<td>340/95</td>
<td>29/8</td>
</tr>
<tr>
<td>Thermal conductivity at 20°C/at 300°C</td>
<td>&gt;500/300</td>
<td>527/306</td>
<td>47/25</td>
</tr>
<tr>
<td>Volumetric CTE 20-1000°C</td>
<td>&lt;7</td>
<td></td>
<td>5.8</td>
</tr>
<tr>
<td>Coefficient of thermal expansion 20-1000°C</td>
<td>&lt;2.9 &lt;15</td>
<td>2.5</td>
<td>12.4</td>
</tr>
<tr>
<td>Young’s Modulus at 20°C</td>
<td>35&lt;E&lt;70</td>
<td>64.9</td>
<td>5.0</td>
</tr>
<tr>
<td>Flexural strength at 20°C</td>
<td>&gt;60</td>
<td>70.3</td>
<td>10.7</td>
</tr>
<tr>
<td>Flexural strain to rupture at 20°C</td>
<td>&gt;2500</td>
<td>2000</td>
<td>4100</td>
</tr>
<tr>
<td>Dimensional stability</td>
<td>&lt;0.05 &lt;0.25</td>
<td>0.02</td>
<td>0.2</td>
</tr>
</tbody>
</table>
UHV behaviour

MoGr Batch total outgassing

Rejected

Lower sintering temperature

93002 powder blocks used in
• TCPPMs#1÷5
• TCSPMs #1÷4

93004 powders

93004 powder blocks used in
• TCSPMs#5÷10

Improved vacuum firing (1200 vs 950 °C, Ne injection at 400 °C)

UHV data: courtesy of G. Cattenoz, G. Bregliozzi

Batch number

MoGr acceptance threshold (in collimators with NEG)

UHV data: courtesy of G. Cattenoz, G. Bregliozzi
Future studies and improvements

- MoGr end of production review held on October 2\textsuperscript{nd} 2020 (see report \url{https://edms.cern.ch/document/2422552})

- One of the lessons learnt in view of similar future productions (also acknowledged by the supplier): add as one of the initial milestones the verification of the \textbf{availability of raw material at the supplier to cover the full production.}

- Ongoing and future studies on the UHV behaviour include:
  - Understanding of the increase of air and methane outgassing after Mo coating on MoGr
  - Time constant for air reabsorption after thermal treatment, once blocks are out of vacuum envelopes (93002 and 93004 powders, can we see a difference?)
  - Temperature monitoring of jaws during bakeout

- Possible additional improvements to the pumping system to be discussed together
Thanks for your attention!
Backup slides
### UHV behaviour – Summary

#### UHV performance before coating
(for 1 collimator)

<table>
<thead>
<tr>
<th>Powders</th>
<th>N\textsubscript{2} (A)</th>
<th>CH\textsubscript{4} (A)</th>
<th>Qtot (mbar*l/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>93002</td>
<td>(1÷1.5)E-9</td>
<td>(1÷1.5)E-9</td>
<td>(2÷4)E-8</td>
</tr>
<tr>
<td>93004 b.14-15</td>
<td>(3÷3.5)E-9</td>
<td>(1÷1.5)E-9</td>
<td>(5÷7)E-8</td>
</tr>
<tr>
<td>93004 b.16</td>
<td>1.5E-9</td>
<td>1E-10</td>
<td>2E-8</td>
</tr>
<tr>
<td>Ratio new/old</td>
<td>2-3</td>
<td>~1</td>
<td>1-3</td>
</tr>
</tbody>
</table>

#### UHV performance after coating
(for 1 collimator)

<table>
<thead>
<tr>
<th>Powders</th>
<th>N\textsubscript{2} (A)</th>
<th>CH\textsubscript{4} (A)</th>
<th>Qtot (mbar*l/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>93002</td>
<td>(1.5÷3)E-9</td>
<td>(6÷10)E-9</td>
<td>(4÷8)E-8</td>
</tr>
<tr>
<td>93004 b.14-15</td>
<td>15E-9</td>
<td>2E-9</td>
<td>(14)E-8</td>
</tr>
<tr>
<td>93004 b.16</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ratio new/old</td>
<td>5-10</td>
<td>0.2-0.3</td>
<td>2-3</td>
</tr>
</tbody>
</table>
UHV behaviour – 93004 powders

Batch 15 before coating (18 blocks+4 taperings)

- After coating: methane higher by factor ~1.5, air higher by a factor of 5.
- **Powders 93002 vs 93004 after coating**: air content up by factor 5-10, total outgassing factor of 2 (both before and after coating)
UHV behaviour – 93004 powders

Batch 16 before coating (20 blocks+4 taperings)

Batch 15 (22 blocks uncoated)
Batch 16 (24 blocks uncoated)
Test bench reference
UHV behaviour

MoGr Batch outgassing

Total outgassing rate (mbar*$/s$)

Lower T

Uncoated

Coated

93004 powders

Batch number

F. Carra (CERN), 25 January 2021
UHV behaviour

MoGr Batch outgassing

93004 powders

Total outgassing rate (mbar*l/s)

Batch number

Uncoated
Coated

F. Carra (CERN), 25 January 2021
Methane up by a factor ~4-10 (10 here). After coating, air/(CO) goes up by a factor of ~1-2.
Same methane as 93002. Air higher by a factor of ~2 (in average over the batches. wrt previous slide, actually almost identical…) → total outgassing higher by a factor of 2
UHV behaviour – 93004 powders

Batch 14 after coating (18 blocks)

- After coating: methane higher by factor ~1.5, air higher by a factor of 5.
- **Powders 93004 vs 93002 after coating**: air content up by factor 5-10, methane lower by factor 3-5, total outgassing higher by factor of 2
UHV behaviour – Summary

- No dramatic difference between 93002 and 93004 powders before coating (factor of ~2 higher air and total outgassing), but air significantly goes up in 93004 powders after coating (up to a factor of 10!).
- A significant air outgassing can saturate NEG quickly.
- Air increase during coating: intrinsic of the new material, or depending on issues at DTI? (or both)
- DTI confirmed that an air leak was detected for batches 12-13-14

**Actions:**

- **Batch 14**: currently on hold, waiting to re-assess the situation once more statistics on next batches comes. In the meantime, thinking about treatments to improve the UHV behaviour of the batch. No straightforward solution (we are limited in T).
- **All next batches will be UHV tested before and after coating**
- **Batch 15**: pre-coating a bit better than uncoated batch 14, shipped to DTI for coating. Procedure for coating (N2 venting, leak repair) agreed with the company.
- **Batch 16 (and following ones):**
  - “Carlotta+” (1200°C, 72h, Ne venting) instead of “Carlotta” treatment (950°C, 72h, Ne venting) → already successfully tested in 2019, we gained a factor 2 in total outgassing (EDMS 2038883).
  - Currently under UHV test pre-coating
Dimensional tolerances

- In primary collimators, difficulties arose due to the springback of the blocks (worsening of block flatness with time and change of thickness which affects the total flatness of the active surface CZ)

- Measures put in place:
  - 2-stage machining
  - Metrology as the last step before coating
  - Block sorting before shipping to CINEL
  - Monitoring of long-term springback

- Additionally, the use of graphite powders 93004 provides a more stable and easier to machine material
Flatness single blocks

Batch 3

Batch 14: effect of 93004 powders

Batch 11: effect of 2-stage machining
Flatness jaw common zone

- Sorting tests are done on the marble in the metrology before shipping, to make sure that no further springback occurs (~3 months after machining), and to have a measurement more representative to the real application.

- In spite of having 1000 mm of CZ instead of 600 mm, significantly better results obtained with secondaries thanks to the actions implemented (also, only type 1 blocks: more regular behaviour).

- Everything traced in MTF (EDMS 2141159 and EDMS 2155433).

<table>
<thead>
<tr>
<th>Jaw</th>
<th>Straightness central line (μm)</th>
<th>Total flatness (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TCPPM</td>
<td>38±5</td>
<td>44±4</td>
</tr>
<tr>
<td>TCSPM (93002)</td>
<td>30±4</td>
<td>35±6</td>
</tr>
<tr>
<td>TCSPM (93004)</td>
<td>16±1</td>
<td>22±1</td>
</tr>
</tbody>
</table>

At the level of the best metallic blocks!
Conclusions

- The production of MoGr for CINEL is completed, and only the material for TCSPM#10 is still to be received (it is currently under machining). An additional batch will cover spare blocks (not to be coated).

- Validated blocks for 5 TCPPM and 5 TCSPM are available or have already been shipped to CINEL.

- The current production schedule at the company foresees 4 TCSPM, then 1 TCPPM and 1 TCSPM → all this material is validated and available.

- Material for last 5 TCSPMs has been produced with new 93004 powders. Overall better performance, but UHV behaviour is worse, especially after coating: implementing corrective actions.

- Dimensional parameters of the blocks are significantly better for TCSPM collimators, we hope this will ease the assembling process at CINEL!