Status and plan for vacuum validation

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Outline

• Vacuum specifications for collimators

• Results overview of TCSPM

• Raw material studies

• Future plan
Vacuum specification

- Vacuum requirements for LHC collimators: EDMS 428155

- Performance:
  - Bakeable to 250°C for 24h00
  - Total outgassing rate \( \sim 1.10^{-7} \text{ mbar.l/s} \)
  - **Clean**: no traces of hydrocarbons, organic and inorganic residues (Residual Gas Analysis)
  - Externaly **leak tight**: global helium leak rate < \(10^{-10}\) mbar.l/s
  - Material outgassing rate < \(10^{-12}\) mbar.l/(s.cm\(^2\)) *i.e.* furnace treatment at 950°C under vacuum (carbon, SS, ferrites ...etc)
  - Bellows should be bakeable and leak tight after 4000 cycles

- Construction and design:
  - Bake-out system: Permanent, remotely controlled and easily disconnected (reduce dose to personnel, ALARA)
  - Avoid: trapped volumes + contact between large surfaces (internal virtual leakage)
  - Welds: Number and length minimised, no crossing, fully penetrating

- In-situ access:
  - Easy access to potentially leaking components (reduce dose to personnel) *e.g.* bellows, feedtroughs
  - Quick release flange with Conflat knife design
Vacuum requirements for collimators

- LHC Long Straight Sections vacuum requirement: EDMS 339088

Achieve and maintain the required static pressure of $5 \cdot 10^{-9}$ mbar

Effective pumping speed of 20 l/s (limited space/conductance of beam chambers)

**Total outgassing rate of one collimator should not exceed** $\approx 1 \cdot 10^{-7}$ mbar·l/s

In LHC:

1. Rely on NEG pumping for H₂, CO and CO₂
2. CH₄ pumping $\rightarrow$ Ion Pumps

For collimators:

Increased local pumping speed with ion pump and NEG cartridge

Advantage:
- Passive
- Remotely controlled
- Re-activable
Vacuum requirements for collimators and test criteria

- Measure of residual gases partial pressures to control the presence of critical gases such as $\text{CH}_4$, CO and $\text{CO}_2$.

Affect the saturation level of NEG coating.
Total outgassing rate measured per type

Since LS1: 1 out of 8 is over acceptance limit

Tuesday, 2 May 2017
Special CoIUSM: Material and design readiness for LS2 production
Gas analysis and outgassing of TCSPM prototype with NEG

Test | Outgassing [mbar.l/s]
--- | ---
Without NEG cartridges | $1.8 \times 10^{-6}$
With NEG cartridges | $1.2 \times 10^{-7}$

Acceptance limit for collimator

- Q [mbar*l/s] | $1 \div 2 \times 10^{-7}$ ✔
- Dominant gas | $H_2$ ✗

All data are taken after 48h at RT after a full bake out cycle
TE-VSC vacuum acceptance test laboratory of b.113

Today’s layout

- Complete jaw assembly test
- Complete TCDIL jaw assembly
- Collimator final validation
- 7 test benches out of 13 dedicated to collimation
- Dedicated new material/development measurement
- Dedicated MoGr measurement
- Jaw block/beam test
- Materials outgassing

Tuesday, 2 May 2017

Special ColUSM: Material and design readiness for LS2 production
Raw Materials Characterization
UHV characterization of Molybdenum Graphite - Production

• 2 different grades were tested:

<table>
<thead>
<tr>
<th></th>
<th>MG6403Fc</th>
<th>MG6403He</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder compaction</td>
<td>P= 300Mpa T=RT Atmosphere: air</td>
<td>P= 300Mpa T=250°C Atmosphere: vacuum</td>
</tr>
<tr>
<td>Sintering</td>
<td>T=2300°C Duration= 2400s Atmosphere: vacuum Venting gas: air</td>
<td>T=2300°C Duration= 2400s Atmosphere: vacuum Venting gas: Ar</td>
</tr>
<tr>
<td>Post sintering</td>
<td>T=2400°C Duration= 3000s Atmosphere: vacuum Venting gas: air</td>
<td>T=2400°C Duration= 3000s Atmosphere: vacuum Venting gas: Ar</td>
</tr>
</tbody>
</table>

Same initial composition of powders:
%vol Mo=4.5
%vol Graphite=95.3
%vol Ti=0.2

Tuesday, 2 May 2017
Special CoUSM: Material and design readiness for LS2 production
UHV characterization of MG6403Fc - as received samples

- Outgassing test of different size samples:

<table>
<thead>
<tr>
<th>Size [mm]</th>
<th>Qspecific [mbar<em>l/s</em>cm²]</th>
<th>Qsetofjaws [mbar*l/s]</th>
<th>Dominant gas</th>
</tr>
</thead>
<tbody>
<tr>
<td>70x25x6</td>
<td>$6 \times 10^{-12}$</td>
<td>$2.3 \times 10^{-8}$</td>
<td>H₂</td>
</tr>
<tr>
<td>125x45x25</td>
<td>$1.5 \times 10^{-10}$</td>
<td>$7.5 \times 10^{-7}$</td>
<td>CO/N₂</td>
</tr>
</tbody>
</table>

- Different thickness
- Different orientation
- Different machining
- Different production plate
UHV characterization of MG6403Fc - SEM observations

- FIB (Focused Ion Beam) images of the 2 samples:

  - Compact bulk for both samples at 2±5 μm from the surface
  - Difficult to see clear trend from the surface
  - Different outgassing behavior seems to be surface related

70x25x6 mm sample

125x45x25 mm sample
UHV characterization of MG6403Fc - Treated sample

• Different vacuum tests are planned to check effect of all the treatments done on the prototype blocks:
  - Vacuum firing 2h, 950°C (standard procedure)
  - CO\(_2\) blasting
  - Mo/TiN coating
**UHV characterization of MG6403Fc - Block Vacuum firing**

**Test** | **Estimated outgassing for a set of jaws**
---|---
Before VF | $7.5 \times 10^{-7}$
After VF | $2.9 \times 10^{-7}$

Vacuum firing effect:
- Important CH4 content reduction
- Decreasing of outgassing mainly related to CH4 reduction
- No strong effect on air content
UHV characterization of MG6403Fc - Vacuum firing

- Vacuum firing is needed to remove hydrocarbons, as confirmed by TDS:

  - The sequence of hydrocarbons mass found can be related to the oil of the pump used in post-sintering treatment.
  - TDS of a sample after vacuum firing shows a decreasing of H₂ content, not seen in the block: possibly need to increase VF duration to increase diffusion length → next test.
UHV characterization of MG6403Fc - CO$_2$ blasting

<table>
<thead>
<tr>
<th></th>
<th>Before CO$_2$ blasting</th>
<th>After CO$_2$ blasting*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Qs [mbar·l/s·cm$^2$]</td>
<td>1.2 * 10$^{-10}$</td>
<td>3.6 * 10$^{-9}$</td>
</tr>
<tr>
<td>Q set of jaws [mbar·l/s]</td>
<td>4.9 * 10$^{-7}$</td>
<td>1.4 * 10$^{-5}$</td>
</tr>
<tr>
<td>I$<em>{28}$/I$</em>{14}$</td>
<td>6.94</td>
<td>8.65</td>
</tr>
<tr>
<td>I$<em>{28}$/I$</em>{12}$</td>
<td>190</td>
<td>189</td>
</tr>
<tr>
<td>I$<em>{44}$/I$</em>{2}$</td>
<td>0.017</td>
<td>1.65</td>
</tr>
</tbody>
</table>

- Total outgassing rate increased: need to check microscopic structure to check if it is related to roughness or crack formation
- Clear indication of CO$_2$ implantation
- Air content increased

*Value for CO2 blasting test after 10h at TR
UHV characterization of MG6403 Machined: Fc vs He

Sample tested | Qset of jaws [mbar*l/s] | $I_{Ar}/I_{N2}$
--- | --- | ---
MG6403Fc-machined | $7.5 \times 10^{-7}$ | 0.021
MG6403He-machined | $2 \times 10^{-6}$ | 0.018

- No influence of the gas used to vent during production steps
- Higher outgassing rate. But no methane contamination
UHV characterization of MG6403He: Machined vs Rectified

<table>
<thead>
<tr>
<th>Sample tested</th>
<th>Qsetofjaws [mbar*l/s]</th>
<th>I_{Ar}/I_{N2}</th>
</tr>
</thead>
<tbody>
<tr>
<td>MG6403He -rectified</td>
<td>5.4 * 10^{-6}</td>
<td>0.018</td>
</tr>
<tr>
<td>MG6403He - machined</td>
<td>2 * 10^{-6}</td>
<td>0.018</td>
</tr>
</tbody>
</table>

- Block machined like the collimator block as a lower outgassing (factor \sim 3)
- Rectified block has higher air and CO₂ content
FIB (Focused Ion Beam) images of He sample with different machining

- No evident differences in the bulk (both compact)

Rectified block vs Machined block

Rectified: More surface damage?
Outlook and future plan

- **MG6403Fc block**
  - Outgassing over limit, air content dominant

- **Vacuum firing**
  - Outgassing decreased, CH$_4$ decreased but no benefit on air

- **CO$_2$ blasting**
  - Outgassing increased, CO$_2$ implantation

- **FIB observation**
  - To check outgassing-roughness relation

- **Longer vacuum firing (48h)**
  - Check effects of the total outgassing and H$_2$ content

- **Coating**
  - To check contribution of coating on a prototype block
Outlook and future plan

MG6403He block machined

US cleaning + VF

Find out other possible NOT aggressive treatment (Ion polishing ?, ..)

Outgassing over limit, air content dominant

To check other possible surface treatment
Thank you for the attention!
TDS sample observation

2mm sample

1mm sample
TDS after vacuum firing

TDS test with 10°C/h ramp: hydrogen content comparison in a vacuum fired vs as received sample
Outgassing trend in function of time after bake out

- After firing
- Before firing
- Fired Batch 1 with coating
- Outgassing limit for collimator jaws

Mathematical equations:
- $y = 9E-06x^{0.317}$, $R^2 = 0.9425$
- $y = 0.0755x^{-1.482}$, $R^2 = 0.9909$
- $y = 0.0129x^{-0.374}$, $R^2 = 0.963$