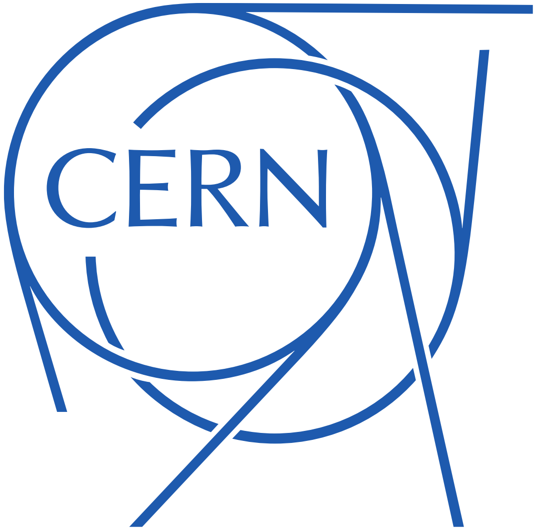

CERN

Vacuum Systems
Slot 4

V. Baglin

CERN TE-VSC, Geneva

juas

Joint Universities Accelerator School

https://indico.cern.ch/event/1470062/timetable/?view=standard#day-2025-02-17

Outline

1. Hydrogen reduction
2. Other materials
3. Qualification of materials

1. Hydrogen reduction

Vacuum firing

- A method to reduce hydrogen content in stainless steel (316 series)
- Degassing of the material is performed in an oven at 950°C under vacuum ($<10^{-5}$ mbar) for 2 h
- The high temperature allows to enhance hydrogen diffusion

Courtesy P. Chiggiato, TE-VSC

CERN large furnace

Length: 6 m

Diameter: 1 m

Maximum charge weight: 1000 Kg

Ultimate pressure: 10^{-9} mbar

P at the end of the treatment:
high 10^{-6} mbar range

Impact on structural properties

- Under the heat surface treatment 304 series stainless steel are recrystallized due to carbide precipitation at the grain boundaries
  - ➔ cannot be used as material for flanges (leak at the level of the knife)!
  - ➔ low carbon and low carbon nitrogen alloys stainless steel are used (316LN)
- The hardness of the material is not modified
- The surface is enriched in Fe due to the Cr evaporation during the heat treatment
- When the material is brought back to atmospheric pressure, it keeps the memory of the treatment since the hydrogen diffusion at room temperature is small
  - ➔ A single treatment is needed for the full life of the material

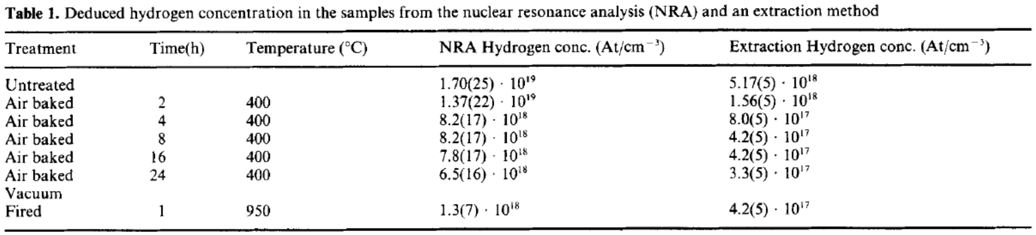


Table 1. Deduced hydrogen concentration in the samples from the nuclear resonance analysis (NRA) and an extraction method

Treatment	Time(h)	Temperature (°C)	NRA Hydrogen conc. (At/cm^{-3})	Extraction Hydrogen conc. (At/cm^{-3})
Untreated			$1.70(25) \cdot 10^{19}$	$5.17(5) \cdot 10^{18}$
Air baked	2	400	$1.37(22) \cdot 10^{19}$	$1.56(5) \cdot 10^{18}$
Air baked	4	400	$8.2(17) \cdot 10^{18}$	$8.0(5) \cdot 10^{17}$
Air baked	8	400	$8.2(17) \cdot 10^{18}$	$4.2(5) \cdot 10^{17}$
Air baked	16	400	$7.8(17) \cdot 10^{18}$	$4.2(5) \cdot 10^{17}$
Air baked	24	400	$6.5(16) \cdot 10^{18}$	$3.3(5) \cdot 10^{17}$
Vacuum Fired	1	950	$1.3(7) \cdot 10^{18}$	$4.2(5) \cdot 10^{17}$

c ~ 2 10^{19} H/cm^3 reduced to 10^{18} H/cm^3

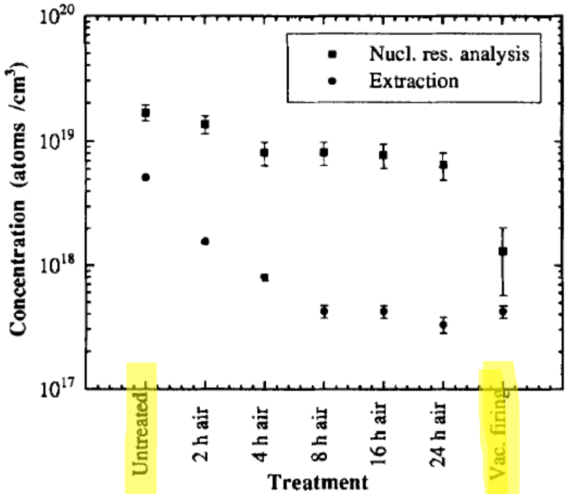


Figure 1. The average hydrogen concentration in the 0.05–0.7 µm depth range of the virgin, air baked and the vacuum fired samples. The results from the extraction method are also included.

L. Westerberg *et al.*, Vacuum 48 (1997) 771-773

Subsequent bakeouts following vacuum firing

- As in previous lecture (slide 32), considering the achieved hydrogen surface concentration following the vacuum firing, the hydrogen outgassing rate after n successive bakeout at temperature T_{BO} and duration t_{BO} is obtained from the 1st Fick Law:

$$q(t) = D\left(\frac{\partial c(x,t)}{\partial x}\right)_{x=0}$$

$$\boxed{q_{n,F}(t) = \left[c_F + (c_o - c_F)\, e^{-\left(\frac{\pi}{L}\right)^2 D(T_F)t_F}\right] \frac{4\,D(T_{RT})}{L}\, e^{-n\left(\frac{\pi}{L}\right)^2 D(T_{BO})t_{BO}}}$$

R. Calder, G. Lewin, Br J Appl. Phys, 18, 1967, 1459

- The diffusion terms dominates:

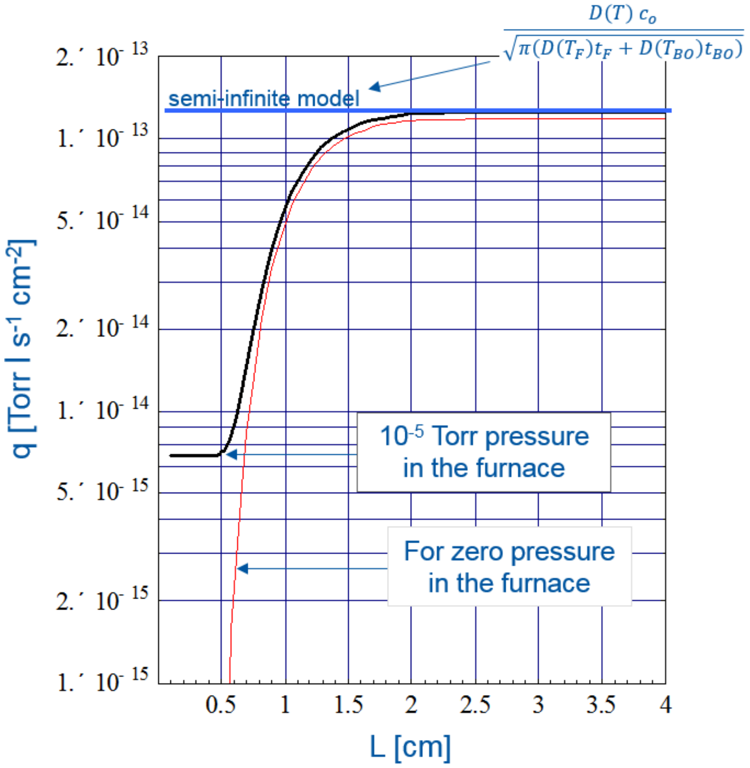
$$D(T) = D_o e^{-E_{diff}/kT}$$

- For thin sheets (tubes), the initial content of the hydrogen is fully removed. The final outgassing is defined by the H_2 pressure in the furnace:

  q ~ 5 10^{-15} mbar.ℓ/(s.cm^2)

- For thick slab (flanges), the pressure in the furnace as limited influence:

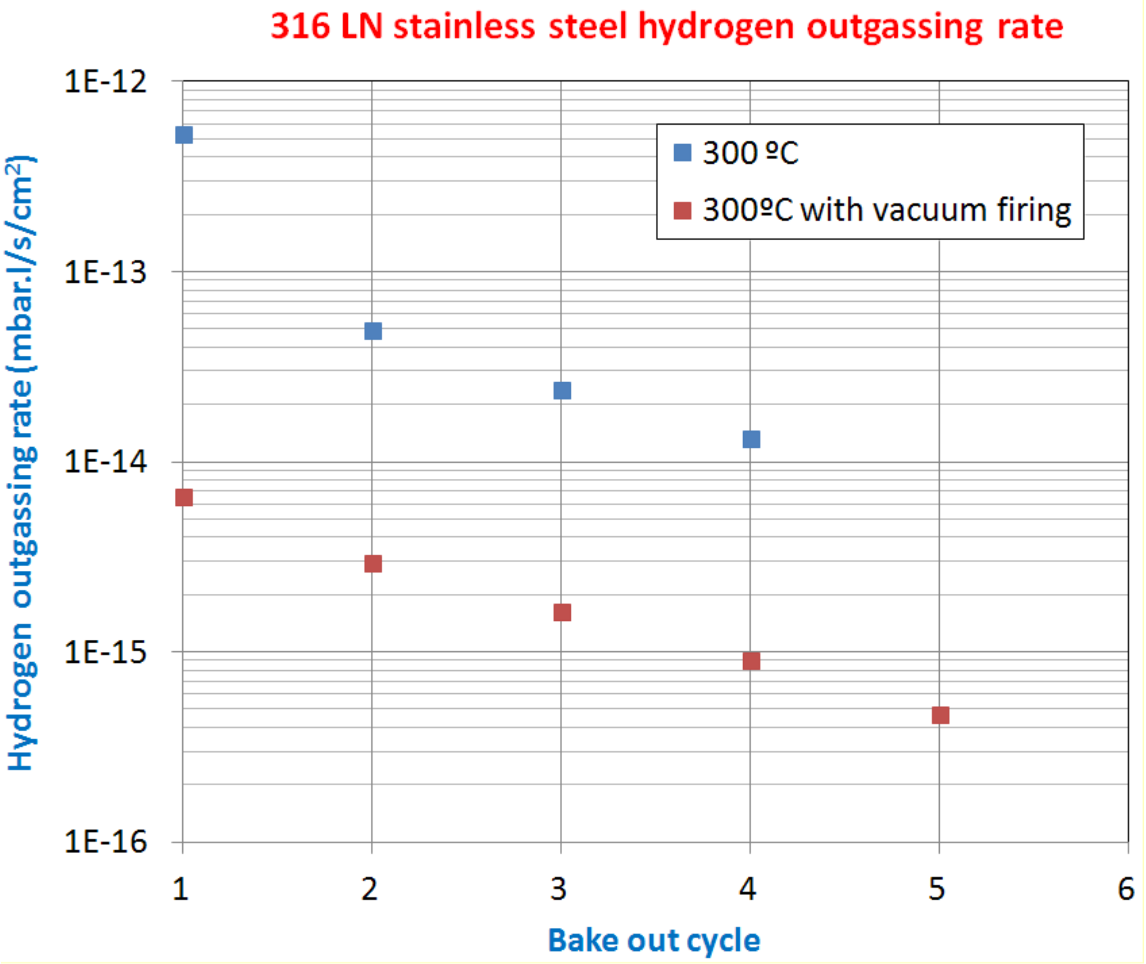
  q ~ 10^{-13} mbar.ℓ/(s.cm^2)



Courtesy P. Chiggiato, TE-VSC

Stainless steel 316 LN

- 1.5 mm thick sheet held at 300°C for 24 h, rate measured 120 h after the end of bake-out
- As expected from diffusion theory:
  - H_2 outgassing rate of 6 10^{-15} mbar.ℓ/(s.cm^2) and a reduction of ~ 1.8 between each cycle



B. Versolatto, N. Hilleret, CERN Vacuum Technical Note 2002

Diffusion barrier: air baking

- The hydrogen diffusion is reduced by a diffusion barrier created during the air bake-out

- Stainless steel tube:
  8 m length, 1.2 m diameter, 2mm thick
- Air fired at 400 deg for 38h
- Then baked at 150 deg for 7 days
- Oxide thickness x 10
- q = 10^{-15} mbar.ℓ/s/cm^2

- Diffusion energy increased from 0.5 to 0.6 eV

- Low cost!

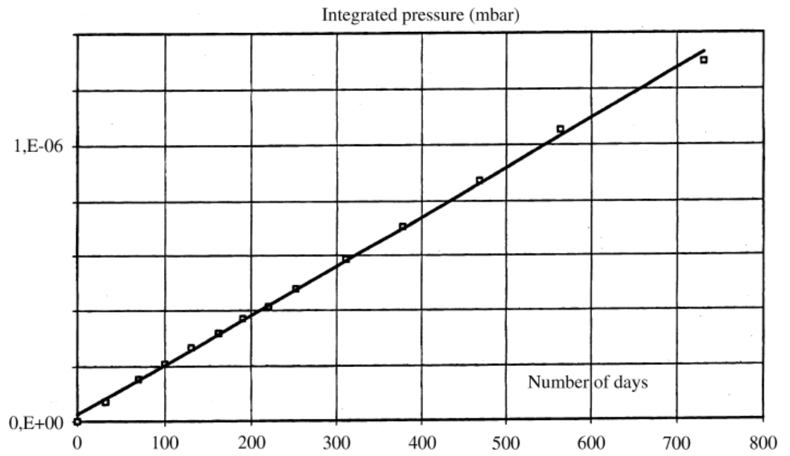


Fig. 1. Hydrogen accumulation over a long period of time in the Orsay corrugated prototype tube for VIRGO.

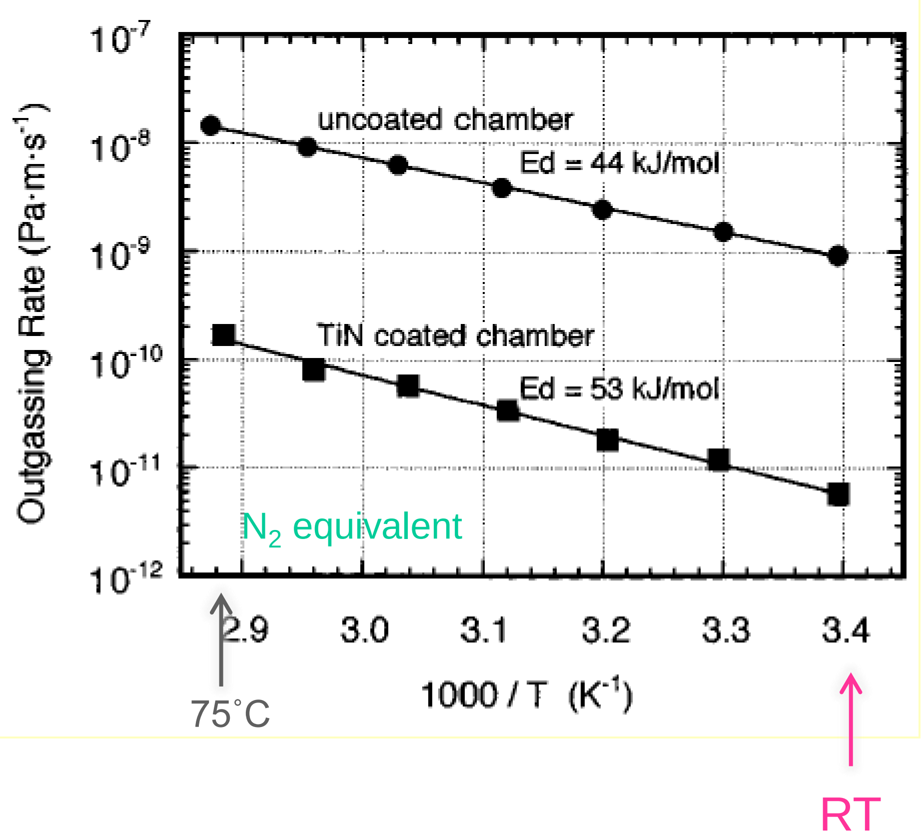
Outgassing performance of an industrial prototype tube for the Virgo antenna, P. Marin *et al.* , Vacuum 49 (1998) 309

2x3 km arms

VIRGO Gravitational Waves detector at Pisa, Italy

Diffusion barrier: TiN coating

- The hydrogen diffusion is reduced by a diffusion barrier created by a coating e.g. TiN
- At least 1 μm film thickness
- The film reduce the hydrogen permeation
- Extrapolation from coupons measurements predicts 10^{-14} Pa.m s^{-1} (10^{-17} mbar.l/s/cm^2)
- 3D objects: difficulties to realise a uniform coating without pinholes which compromised the observed performance on a tube or vacuum chamber
- Reduction of 2 orders of magnitude of the hydrogen outgassing rate:
  - Uncoated chamber: 10^{-12} mbar.l/s/cm^2
  - TiN coated chamber: 7 10^{-15} mbar.l/s/cm^2



TiN thin film on stainless steel for extremely high vacuum material, K. Saito *et al.* , J. Vac. Sci. Technol. A 13(3) May/Jun 1995, 556

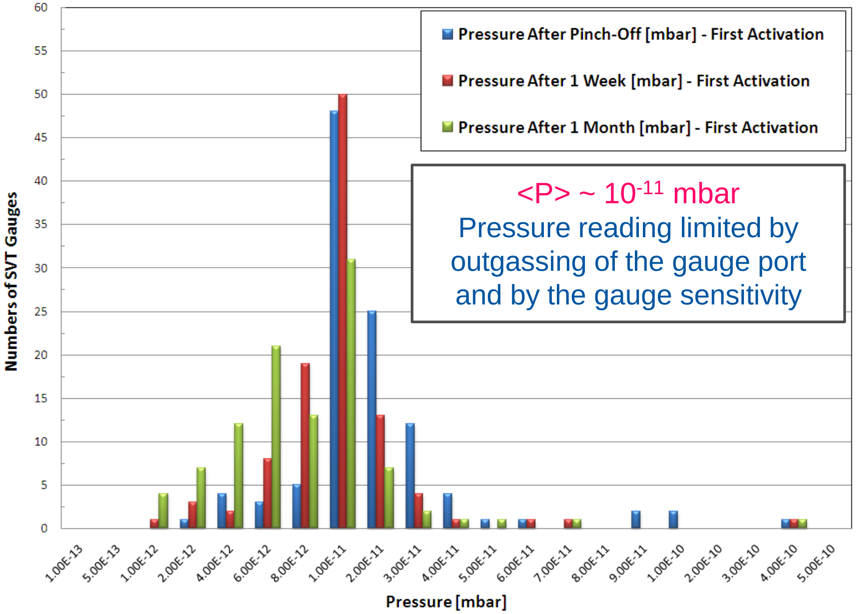
Today: no application in accelerator yet (some attempts for SNS ring, P. He et al., PAC 2003)

Dissolving hydrogen: NEG (TiZrV) coating

- Ti-Zr-V is coated by magnetron sputtering using Kr gas
- ~ 1 µm thick
- Owing to the high solubility and diffusivity of hydrogen once activated to 180-200˚C, the coating provides at room temperature extremely:
  - Low outgassing rate: $<10^{-17}$ Torr.ℓ/cm^2 that means < 300 CH_4/cm^2
  - Large pumping speed: 0.3-2.2 ℓ/s/cm^2 for H2 and 5-9 ℓ/s/cm^2 for CO

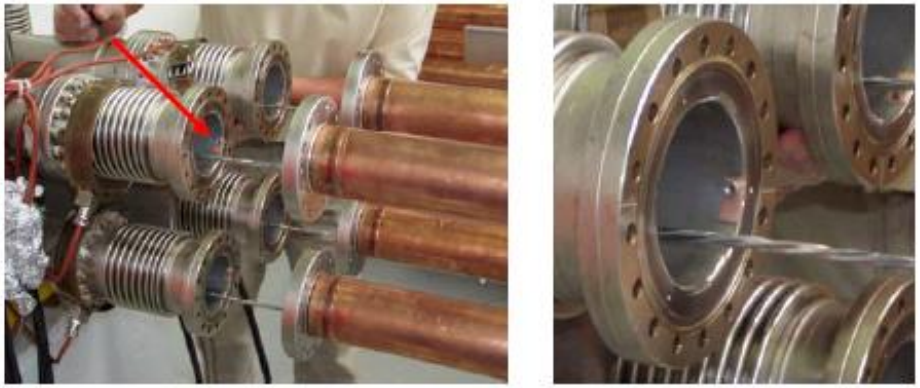
➔ ~ 2 000 & 15 000 ℓ /s per m of LHC vacuum chamber for H_2 and CO resp. !

Pressure distribution in the LHC RT vacuum sectors



$<P> \sim 10^{-11}$ mbar
Pressure reading limited by outgassing of the gauge port and by the gauge sensitivity

G. Bregliozzi *et al.* EPAC'08, Genoa 2008



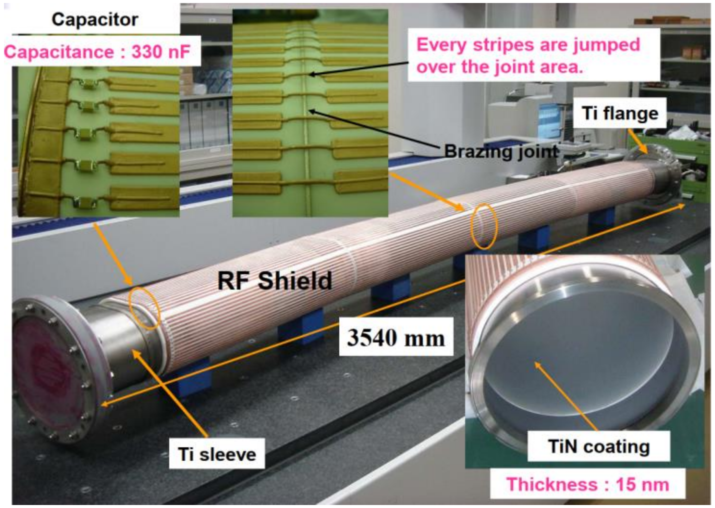
C. Benvenuti et al. Vacuum 60 (2001) 57-65
P. Costa Pinto, P. Chiggiato / Thin Solid Films 515 (2006) 382-388

2. Other materials

Outgassing of ceramics

- Ceramics are used as insulators for feedthrough, as RF windows for their transparency to EM fields or to minimise Foucault's currents induced in the vacuum chambers of fast ramping magnets (kickers) or fast cycling accelerators.
- Outgassing rates are function of the porosity

for quadrupole magnet



J-Parc Vacuum chambers, M. Kinsho, Nov. 2005

- Alumina (Al_2O_3) is sintered at high temperature (1600˚C) and chemically stable
- TiN coated alumina chamber has q = $1\ 10^{-11}$ mbar.ℓ/s/cm^2 after 50 h pumping

Y. Saito et al. Vacuum 86 (2012) 817-821

- Macor and nitrides ceramics requires in-situ bakeout at 200˚C to reach ~ 10^{-11} mbar.ℓ/s/cm^2 at RT

K. Battes et al. J. Vac. Sci. Technol. B39, 034202 (2021)

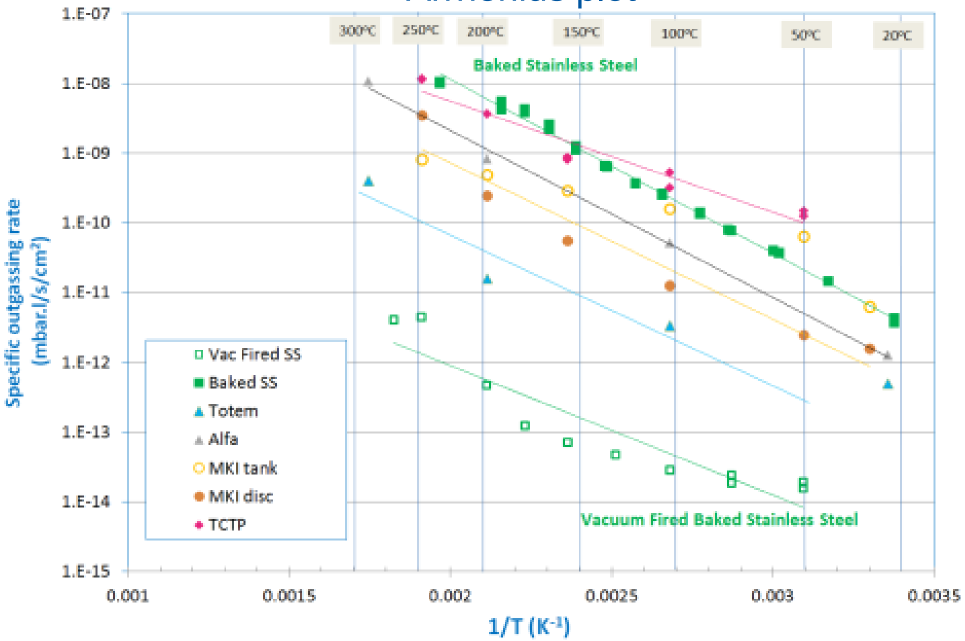
Outgassing of ferrites

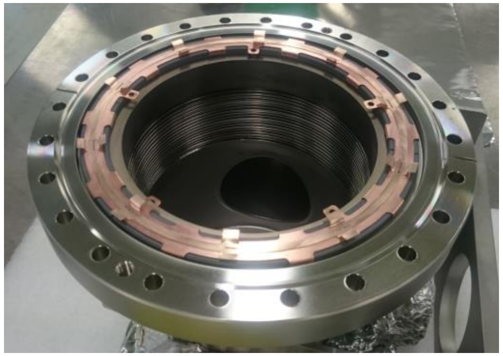
- Ferrites inserted in devices (XRP, TCSP, TCTP, MKI, TDI …) have low outgassing rate 10^{-11} mbar.ℓ/s/cm^2 (after bakeout)
- TT2-111R, CMD5005 and CMD10
- Treated at 400˚C – 1000˚C

- But can heat up during operation => increase of outgassing rate

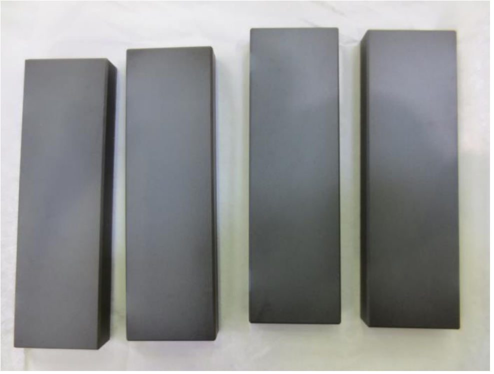
°C	50	100	150	200
$\frac{q}{q_{RT}}$	5	40	150	600

Arrhenius plot





LHC Roman Pots ferrite

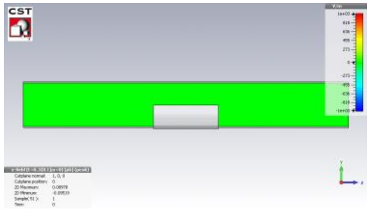


Example TDI during RUN1 and 2

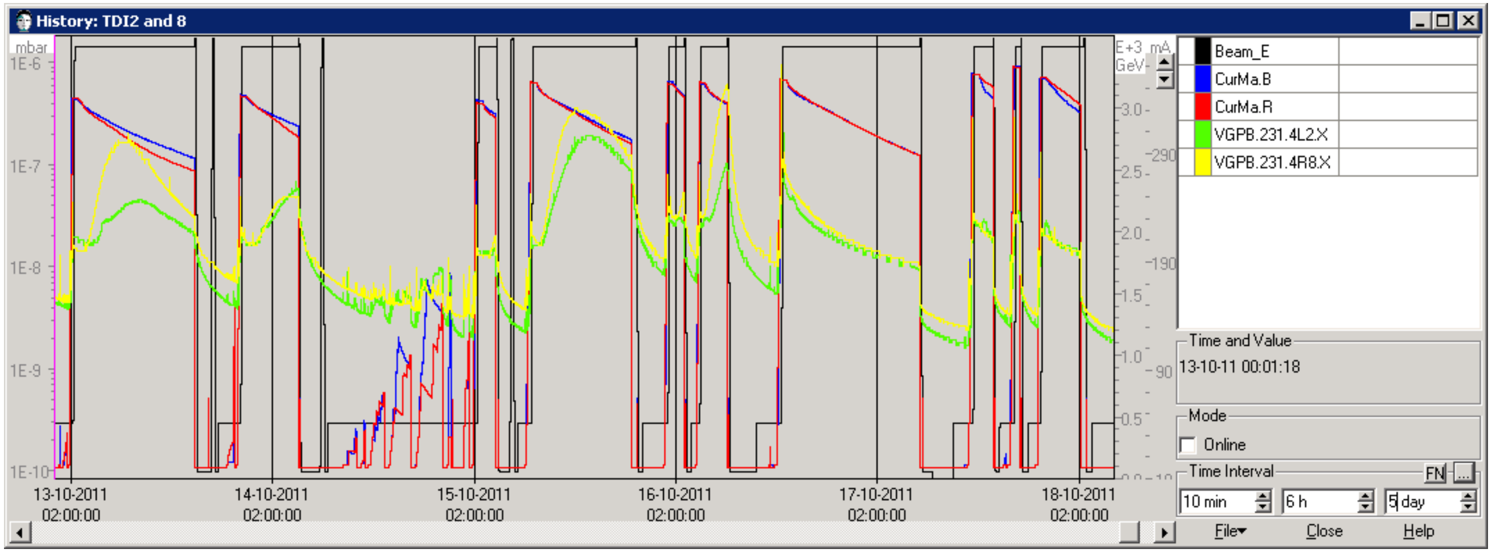
- A Target Dump for Injection device to protect downstream elements
- Circulating beams increase the temperature of the BN jaws by impedance effects
- Any beampipe component generates EM wakefields and may behave like a resonator or a damper

Beam induced heating

$$Q = Q_o e^{-\frac{E}{k\,T}}$$



X. Buffat, CAS, 2022



History: TDI2 and 8

■	Beam_E
🟦	CurMa.B
🟥	CurMa.R
🟩	VGPB.231.4L2.X
🟨	VGPB.231.4R8.X

Time and Value
13-10-11 00:01:18

Mode
☐ Online

Time Interval
10 min
6 h
5 day

B. Salvant et al. EM simulations of the impedance of the LHC TDI. Proc. of IPAC 2012, New Orleans, USA
B. Salvant et al. Update on beam induced RF heating in the LHC. Proc. of IPAC 2013, Shangai, China

Outgassing of additive manufactured materials

- Recent technique, only few applications yet
- Additive manufactured stainless steel tube outgassing are as low as conventional ones.

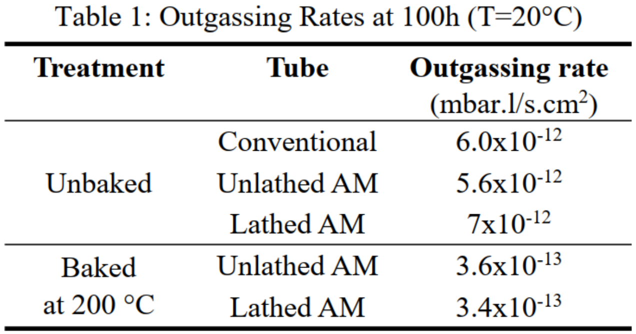


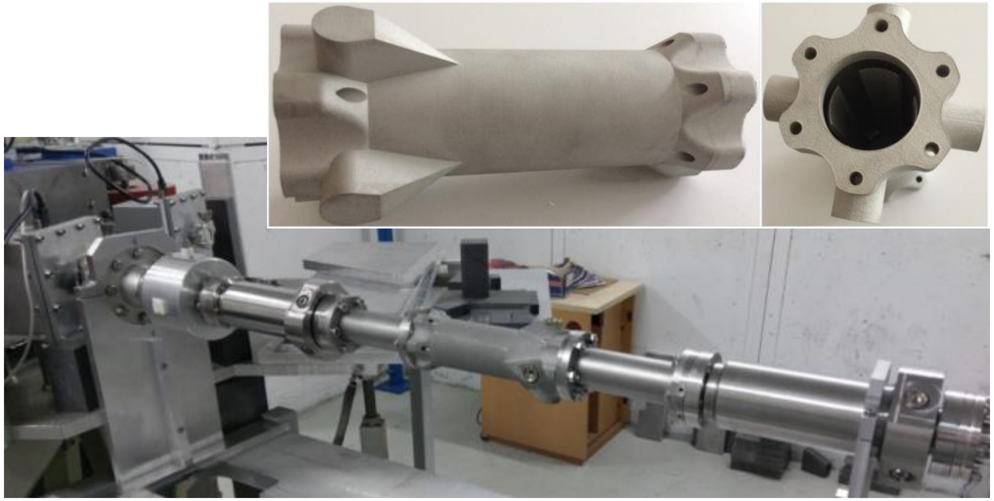
Table 1: Outgassing Rates at 100h (T=20°C)

Treatment	**Tube**	**Outgassing rate** (mbar.l/s.cm^2)
Unbaked	Conventional	6.0×10^{-12}
	Unlathed AM	5.6×10^{-12}
	Lathed AM	7×10^{-12}
Baked at 200 °C	Unlathed AM	3.6×10^{-13}
	Lathed AM	3.4×10^{-13}

G. Sattonnay et al. Proc. of IPAC 2019, Melbourne, Australia

- Construction of an AM BPM
- Test on a photo-injector show results as good as conventional BPMs

➔ AM BPM became THOM-X baseline



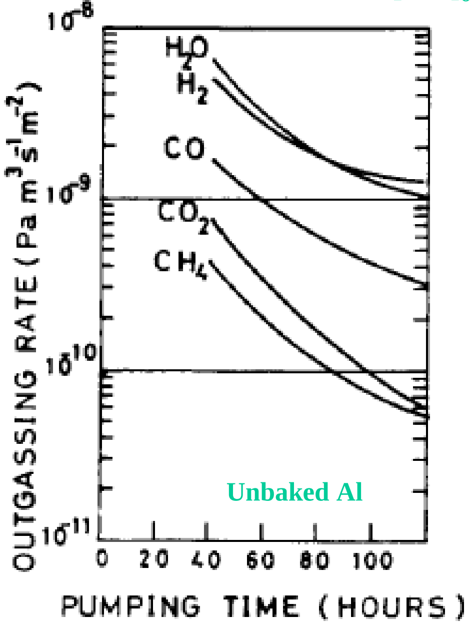
N. Delerue et al. Proc. of IPAC 2019, Melbourne, Australia

Outgassing of polymers & elastomers

- Plastic materials are highly porous and contains much more water than metallic surfaces
- Their outgassing rate is limited by a diffusion process with a = 1/2

$$q(t) = q_0\, t^{-a}$$

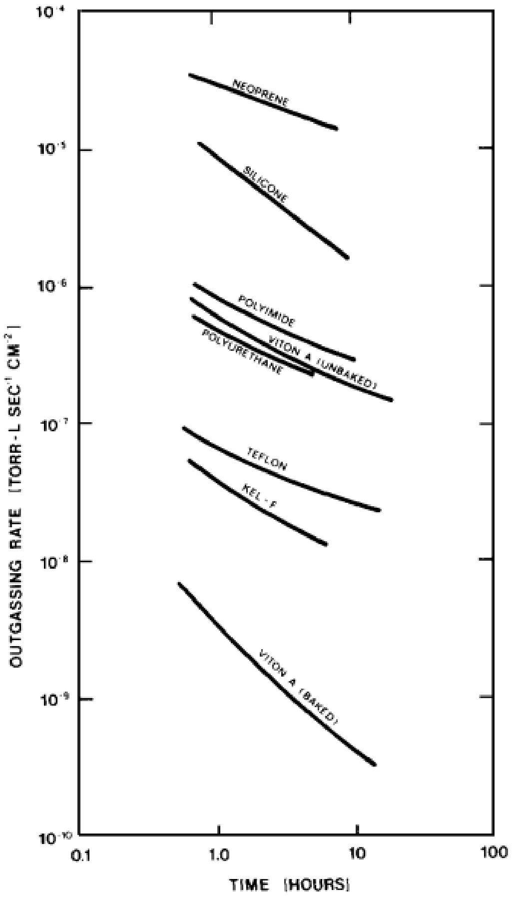
Metallic surfaces $q \sim q_0/t$



A.G. Mathewson *et al.*
J.Vac.Sci. 7(1), Jan/Fev 1989, 77-82

x 10^2 - 10^5 →

Plastic surfaces **$q \sim q_0/\sqrt{t}$**



R.N. Peacock
J.Vac.Sci. 17(1), Jan/Fev 1980, 330-336

Good Vacuum Design :

Use ONLY metallic surfaces and reduce to ZERO the amount of plastics

Outgassing of thin polymers

- The thicker the material, the more moisture is embedded

- q decreases from 10^{-5} after 1h to 10^{-10}-10^{-7} after 100h

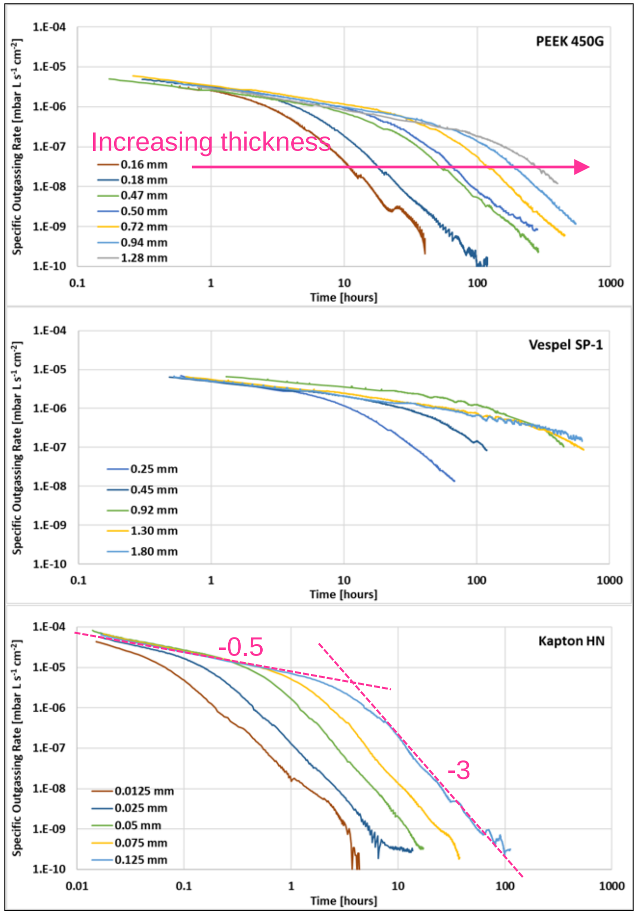
- Compared to Vespel® & Kapton®, PEEK has:
    - larger diffusion coefficient hence faster q reduction
    - lower moisture content hence lower q

- Diffusion process in $1/\sqrt{t}$ then $1/t^3$ after extended time (>100 h)

- Polymers thicker than ~ 1 mm requires more than 2 weeks of pumping to reduce q significantly

- A bake-out to 200˚C strongly reduce outgassing in the 10^{-11} – 10^{-10} mbar.ℓ/s/cm^2 range

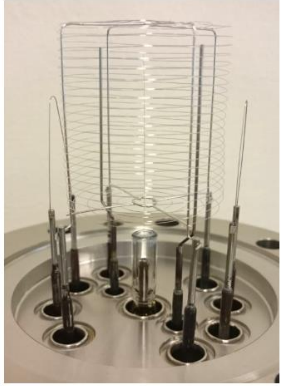
- Venting with N_2 or dry air preserve the bake-out benefit



S. Sammartano et al. CERN-ACC-Note-2020-0039

Outgassing of gauges filaments

- During operation, filaments are heated to ~ 2 000˚C consequently desorbing gas and provoke also Electron Stimulated Desorption (ESD)



$Q = 2\ 10^{-9}$ mbar.ℓ/s for W filament

$= 1\ 10^{-10}$ mbar.ℓ/s for thoriated W filament

RGA	Ion source	Filament	Q (mbar.ℓ/s)
Faraday/SEM	Vac. fired grid	W	$2\ 10^{-9}$
Faraday/Channeltron	Vac. fired grid	W	$4\ 10^{-9}$
	grid	W	$4\ 10^{-8}$
	Vac. fired open source	Y	$8\ 10^{-9}$
	Open source	Y/W	$2\ 10^{-8}$ $5\ 10^{-7}$

S. Meunier, CERN, 2019

3. Qualification of materials

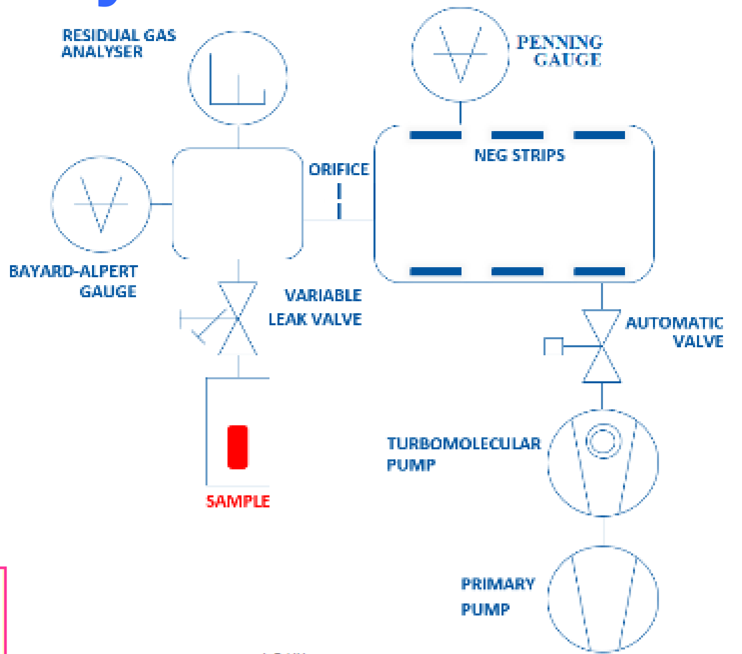
Outgassing measurement by Accumulation

- A sample is placed in an evacuated vessel and the leak valve closed

- Desorbed gas accumulates into the sample chamber

- After some accumulation time t_{acc}, the leak valve is opened and the mass spectra recorded

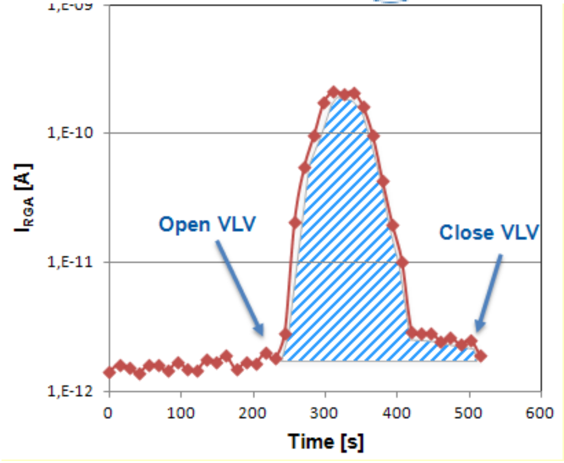
- The leak valve is closed again and the procedure repeated every 1 to 72 h



$$\boxed{Q(\Delta t_{ac}) = \frac{S_i \int_0^{\Delta t} \alpha_i \, I_{RGA}(t)\, dt}{\Delta t_{ac}}}$$

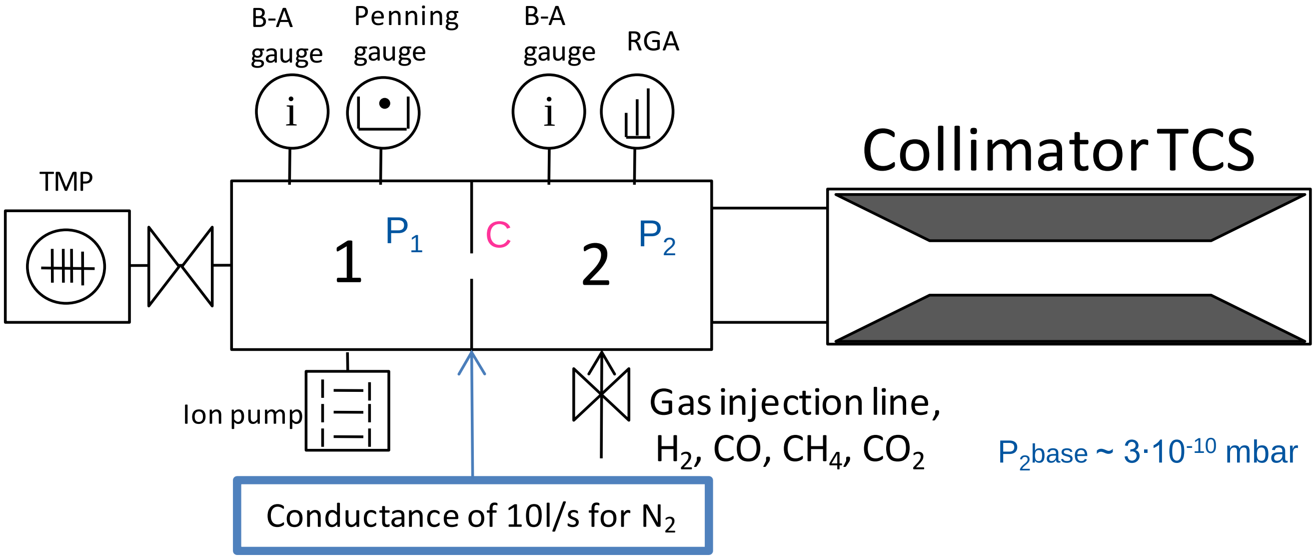
- With:
  - S_i the pumping speed for gas, i
  - α the RGA calibration factor for gas, i
  - I_{RGA} the current recorded during the leak valve opening
  - Δt the RGA recording duration
  - Δt_{ac} the accumulation time

- Sensitive measurement but gas re-adsorption may matters



Courtesy I. Wevers, TE-VSC

Outgassing measurement: throughput method



P_2base ~ $3 \cdot 10^{-10}$ mbar

J. Kamiya *et al.*, Vacuum 85 (2011) 1178-1181

- The component is connected to a pumping system via a conductance, C
- Background is determined by a dry run
- The outgassing rate is

$$\boxed{Q_{N2eq} = C\,(P_2 - P_1)}$$

In N_2 equivalent no RGA is needed!

$$Q_i = S_{eff} P_{2,i} = C_i\,(P_{2,i} - P_{1,i})$$

$$S_{eff} = \frac{C_i\,(P_{2,i} - P_{1,i})}{P_{2,i}} = C_i\,\left(1 - {}^{P_1}/_{P_2}\right)$$

$$\boxed{Q_i = C_i \alpha_i I_i\,\left(1 - {}^{P_1}/_{P_2}\right)}$$

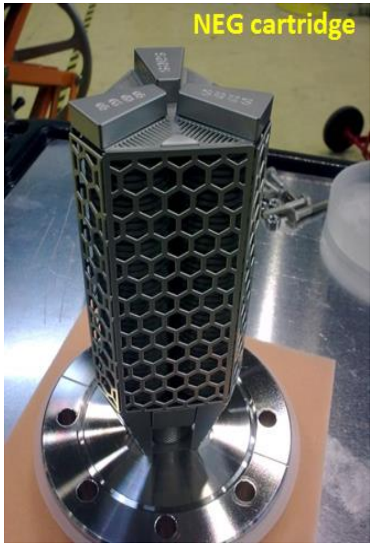
α the RGA calibration factor for gas, i
I_i the RGA current for gas I
c_i the conductance for gas i

Vacuum Acceptance Test Laboratory

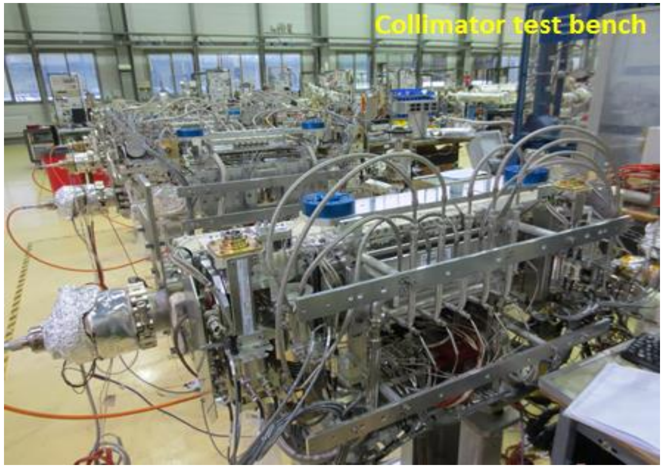
LHC vacuum laboratory during LS1 (2013-2014)

Example of tested parts

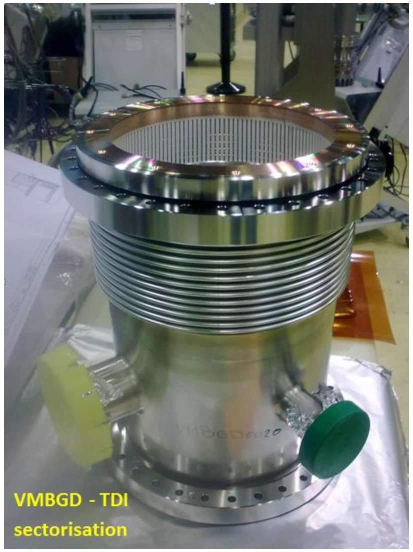

VPIAN test


NEG cartridge


BTVSS.6R8

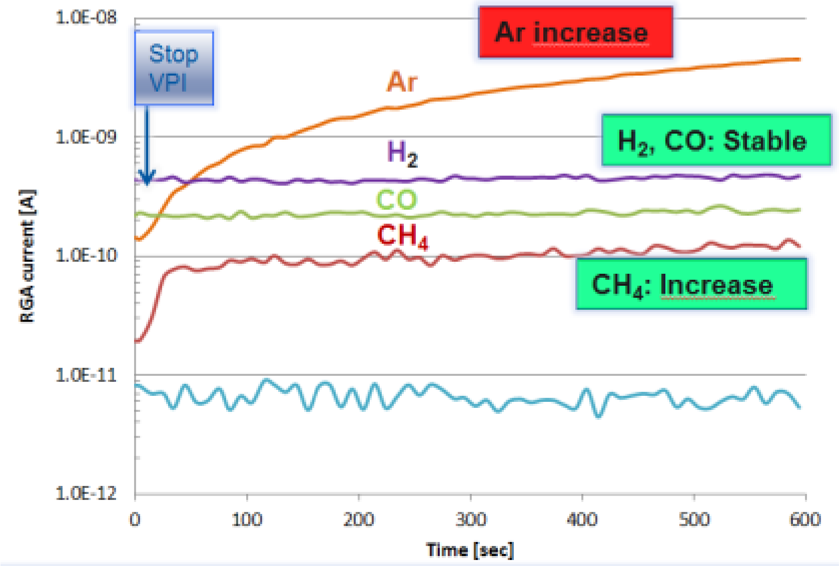

Collimator test bench


VM NEG copper inserts

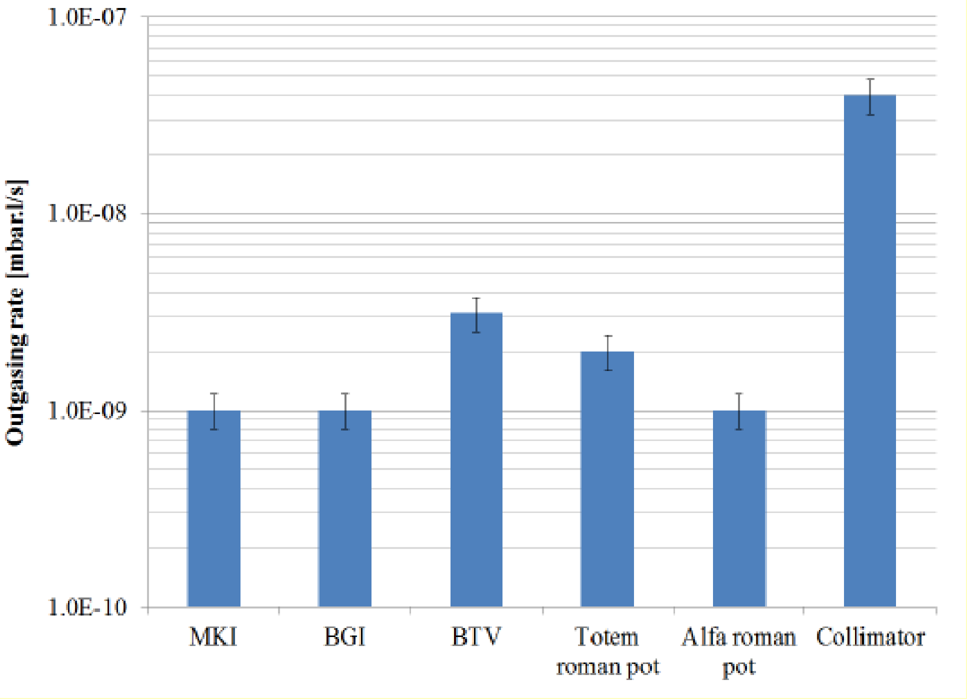

VMBGD - TDI sectorisation

Vacuum Acceptance Tests

- Prior to LS1 installation ~1200 LSS's equipments have been baked and validated at the surface :
  - functional test
  - pump down
  - leak detection
  - residual gas composition
  - total outgassing rate



Identification of virtual leaks by accumulation test whilst pumping with NEG system



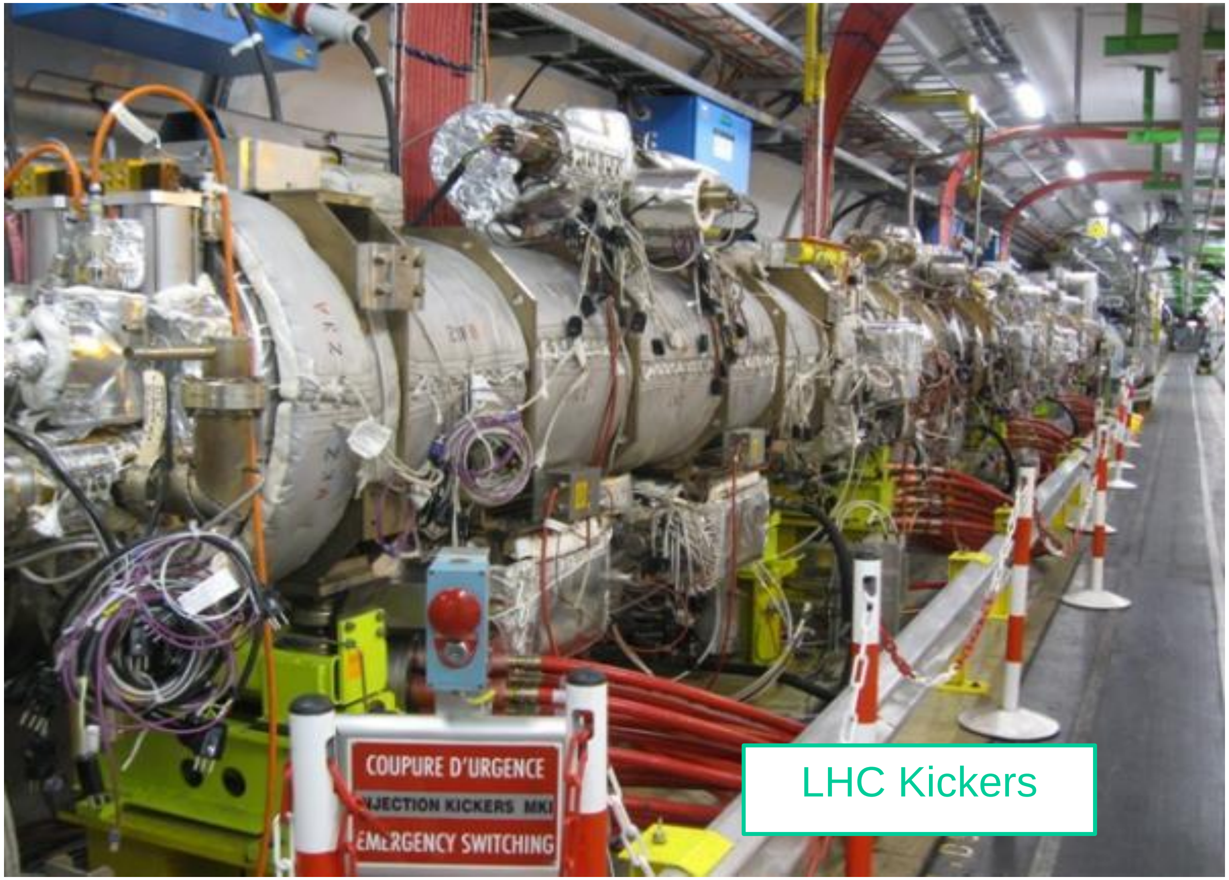
Outgassing rate of some LHC components

G. Cattenoz *et al.* , Proceeding of IPAC'14, Dresden, Germany

Magnet Kicker for Injection (MKI)

- Total outgassing rate of this chain of 4 MKIs is ~ 5 10^{-9} mbar.ℓ/s

  ➔ that corresponds to the outgassing of 2 VITON seals after 1 year of pumping



LHC Kickers

=

+

Cleaning Methods

- Chemical cleaning is used to remove gross contamination such as grease, oil, fingerprints.
- It can be needed to attack the surface with acids to etch the oxide layer
- Passivation can be helpful to produce a “stable” oxide layer on the surface

- Example of CERN LHC beam screens :
  - Degreasing with an alkaline detergent at 50°C in an ultrasonic bath
  - Running tap water rinse
  - Cold demineralised water rinse by immersion
  - Rinse with alcohol
  - Dry with ambient air

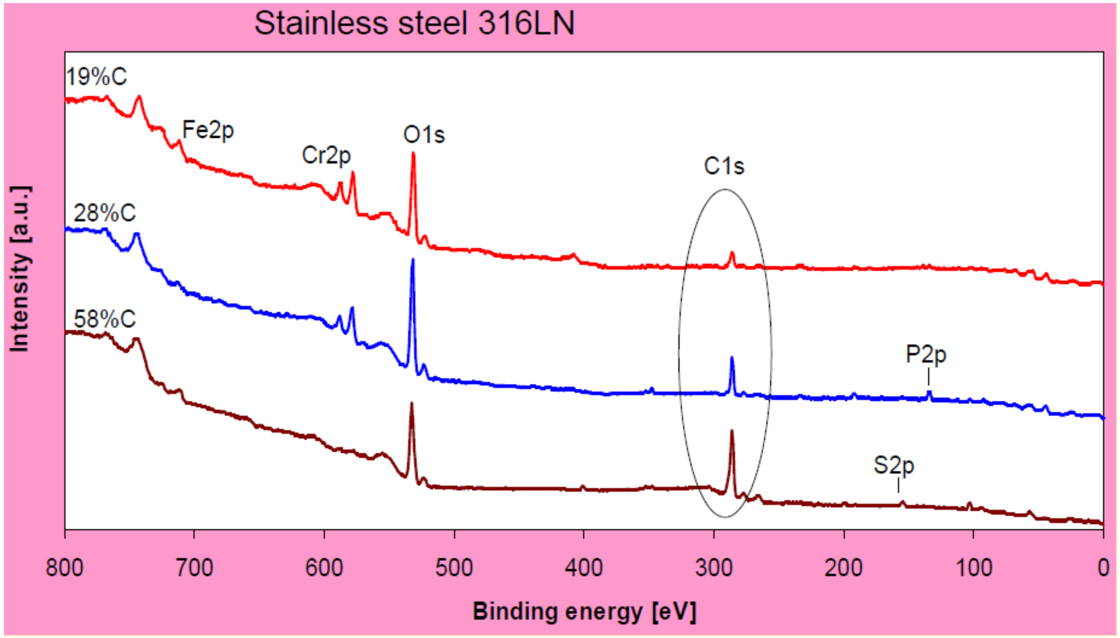
L. Ferreira, CERN TE-VSC

Surface analysis methods

- Surface analysis is used to evaluate the cleanliness of a material, identify species, monitor a process etc.

- Auger electron Spectroscopy

- X-ray Photoelectron Spectroscopy



The assessment of metal surface cleanliness by XPS, C. Scheuerlein and M. Taborelli, Appl. Surf. Sci 252 (2006) 4279

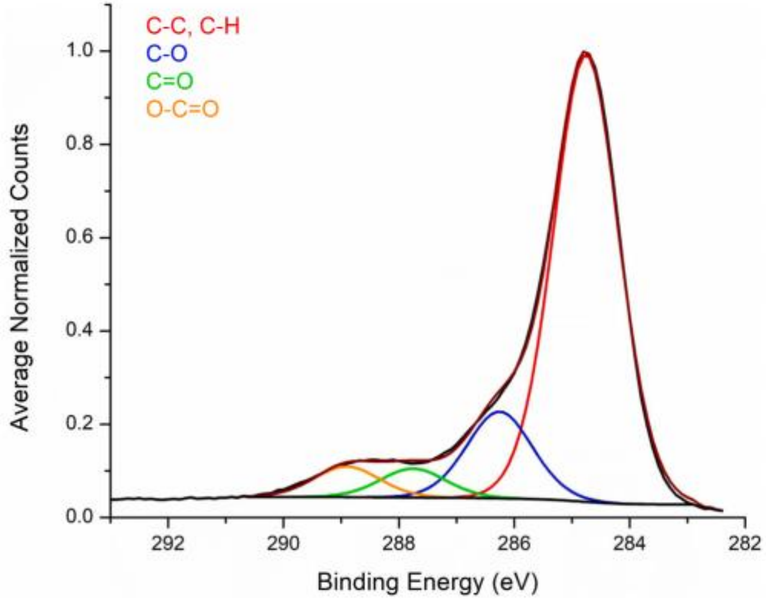
Example of adventitious carbon

- Adventitious carbon = a thin layer of carbonaceous material that deposits on air-exposed surfaces

- XPS analysis of adventitious carbon around C1s at 284.8 eV

- Adventitious carbon layer is aliphatic (hydrocarbons C, H, having an open chain structure) in nature and not graphitic, with functional groups of C, O

- Presence of CO and CO_2 molecules bounded on the very near surface (nm)



LH Grey et al., Appl. Surf. Sci 635 (2024) 159319

Slot 4 summary

- Vacuum firing is used to reduce by 2 orders of magnitude the hydrogen outgassing of baked material.

- Other material than metals have their outgassing rate spread over several orders or magnitude with plastics at the high extreme (x 10^5 metallic surface)

- Vacuum instruments outgas!

- Components must be characterised in the laboratory with appropriate tools to guarantee a good performance in an accelerator

Thank you for your attention !!!

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