

# Cold / sticky system

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# Cold / sticky system ??



# Vacuum ??



# Eureka ??



# Cold / sticky system

## Outline

1. Cryopumping
2. Adsorption isotherms
3. Cryosorbers in cold systems
4. He leaks in cold systems

# 1. Cryopumping

# Desorption probability

- The desorption probability is a function of the **binding energy**,  $E$  and the **temperature**,  $T$  (first order desorption, Frenkel 1924). The surface coverage,  $\theta$ , varies like :

$$\frac{d\theta}{dt} = -\theta \nu_0 e^{-\frac{E}{kT}}$$

$(\nu_0 \sim 10^{13} \text{ Hz}, k = 86.17 \cdot 10^{-6} \text{ eV/K})$

- The desorption process is characterised by the **sojourn time**,  $\tau$  :

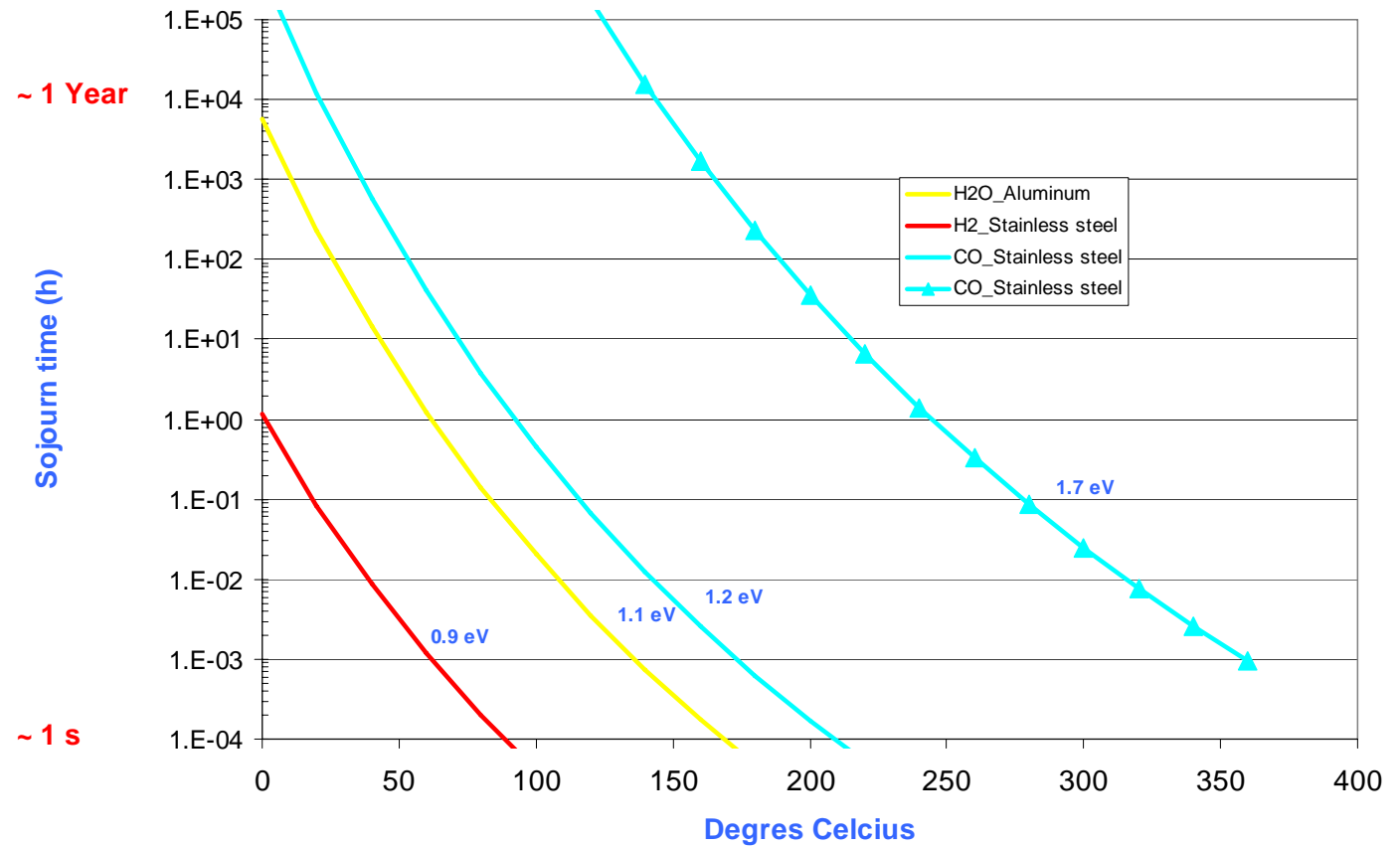
$$\tau = \frac{e^{\frac{E}{kT}}}{\nu_0}$$

- For large  $E$  and small  $T$ , molecules remains onto the surface : **CRYOPUMPING**
- For some combination of  $E$  and  $T$ , the molecule is desorbed (bake out)

# Sojourn time

## Chemisorbed molecules

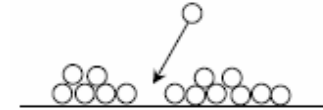
- At room temperature :  
H<sub>2</sub>O dominates  
> 100 °C to remove H<sub>2</sub>O
- A bake out up to 300 °C  
removes molecules with large  
binding energies which could  
be desorbed by stimulated  
desorption



# Cryopumping regimes

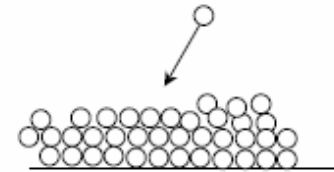
## Physisorption

- **Sub-monolayer** coverage : attractive force (van der Waals) between a gas molecules and a material
- Binding energy for physical adsorption
- $H_2$  from 20 to 85 meV for smooth and porous materials resp.
- 1 h sojourn time at 5.2 K and 26 K for smooth and porous materials resp.



## Condensation

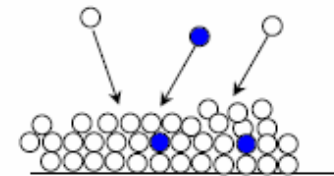
- For **thick gas coverage**, only forces between gas molecules
- Energy of vaporisation 9 to 175 meV for  $H_2$  and  $CO_2$  resp.
- 1 h sojourn time at 2.8 K and 53.4 K for  $H_2$  and  $CO_2$  resp.



➔ sub-monolayers quantities of gas can be *physisorbed* at their boiling temperature  
(ex :  $H_2$  boils at 20.3 K and a bake-out above 100 °C removes water)

## Cryotrapping

- Use of a easily condensable carrier (e.g. Ar) to trap molecules with high vapor pressure (e.g. He,  $H_2$ )





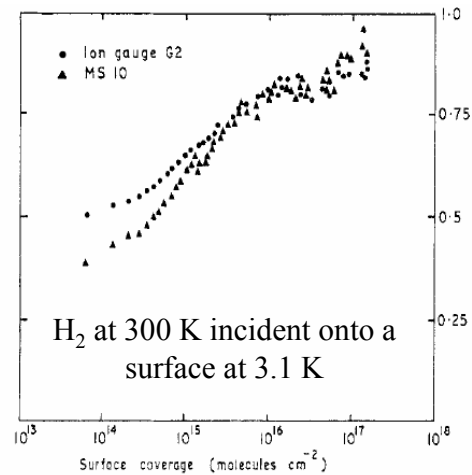
# Sticking probability/coefficient

- Probability :  $0 < \sigma < 1$

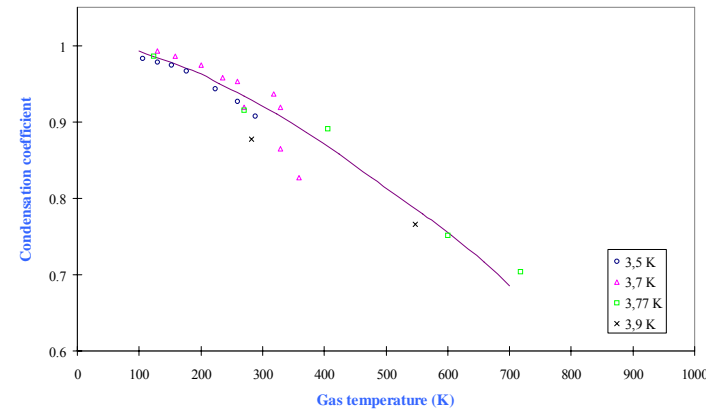
$\nu$  collision rate (molecules.s<sup>-1</sup>.cm<sup>-2</sup>)

$$\sigma = \frac{V_{\text{incident}} - V_{\text{departing}}}{V_{\text{incident}}} = \frac{V_{\text{sticking}}}{V_{\text{incident}}}$$

- Function of gas, surface, surface coverage, temperature of gas and surface temperature



J.N. Chubb *et al.* J. Phys. D, 1968, vol 1, 361



J.N. Chubb *et al.* Vacuum/vol 15/number 10/491-496

- Pumping speed

$$S = \frac{1}{4} \sigma \left( 1 - \frac{P}{P_{\text{sat}}} \right) A \bar{v} \approx \frac{1}{4} \sigma A \bar{v}$$

*i.e* :  $\sigma$  times the conductance of a surface

$$S [\text{l.s}^{-1}.\text{cm}^{-2}] = 3.63 \sigma \sqrt{\frac{T}{M}}$$

- H<sub>2</sub> and CO at 4.2 K :

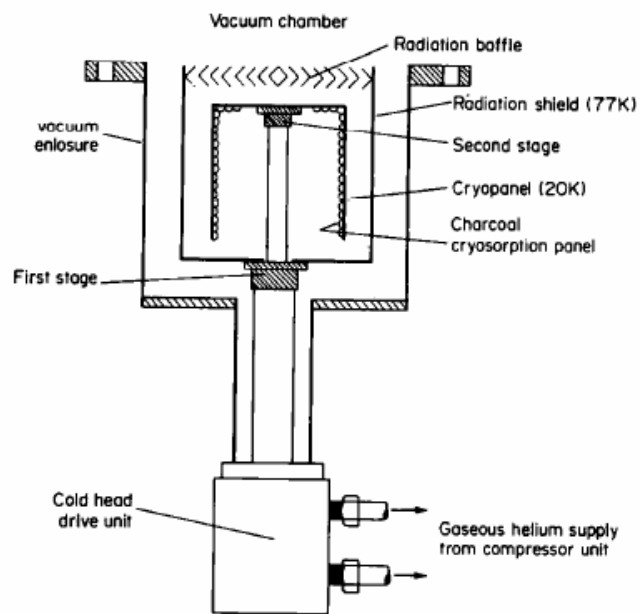
$$S_{\text{H}_2} = 5.3 \text{ l.s}^{-1}.\text{cm}^{-2}$$

$$S_{\text{CO}} = 1.4 \text{ l.s}^{-1}.\text{cm}^{-2}$$

# Capture factor, $C_f$

- Takes into account the geometry of the system :

## Baffle in a cryopump



$$C_f \sim 0.3$$

R. Haefer. J. Phys. E : Sci. Instrum., Vol 14, 1981, 273-288

## Holes in the electron shield of the LHC beam screen

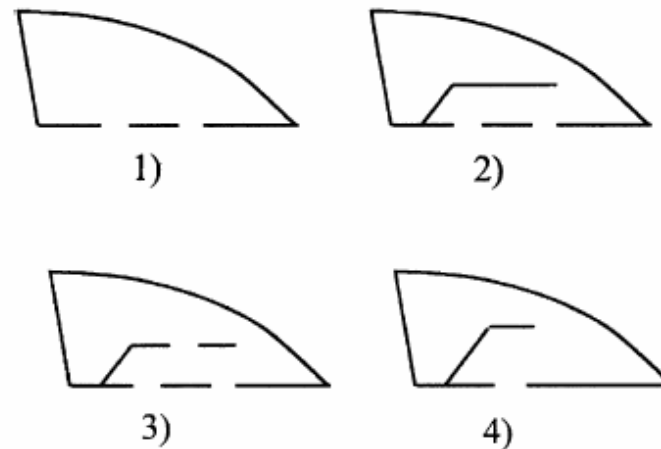


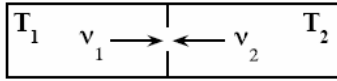
Fig. (1) Two slots in the beam screen, without electron shield, (2) two slots in the beam screen, electron shield without slot, (3) two slots in the beam screen, electron shield with slot, (4) only one slot in the beam screen, electron shield without slot.

$\sigma$	1	2	3	4
0.1	0.48	0.26	0.39	0.43
1	0.68	0.36	0.51	0.57

A.A. Krasnov. Vacuum 73 (2004) 195-199

# Thermal transpiration

- Vacuum gauges are located at room temperature to reduce heat load
- For small aperture, the collision rate,  $\nu$ , is conserved at the cold / warm transition



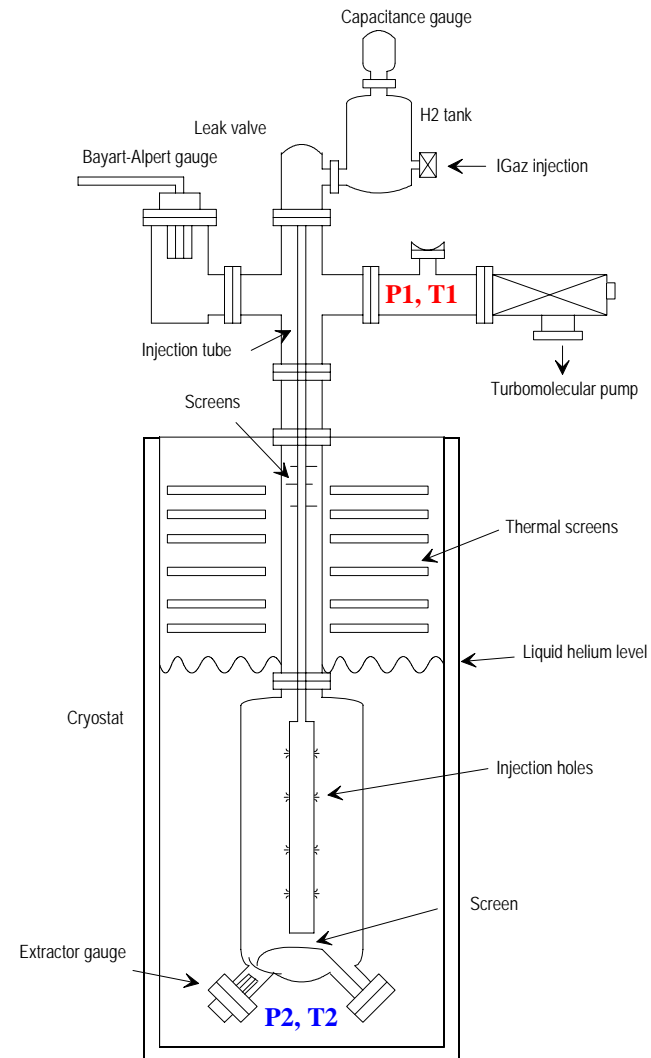
$$\nu = \frac{1}{4} n \bar{v}$$

- Since the average velocity scales like  $\sqrt{T}$

$$\frac{P_1}{P_2} = \sqrt{\frac{T_1}{T_2}}$$

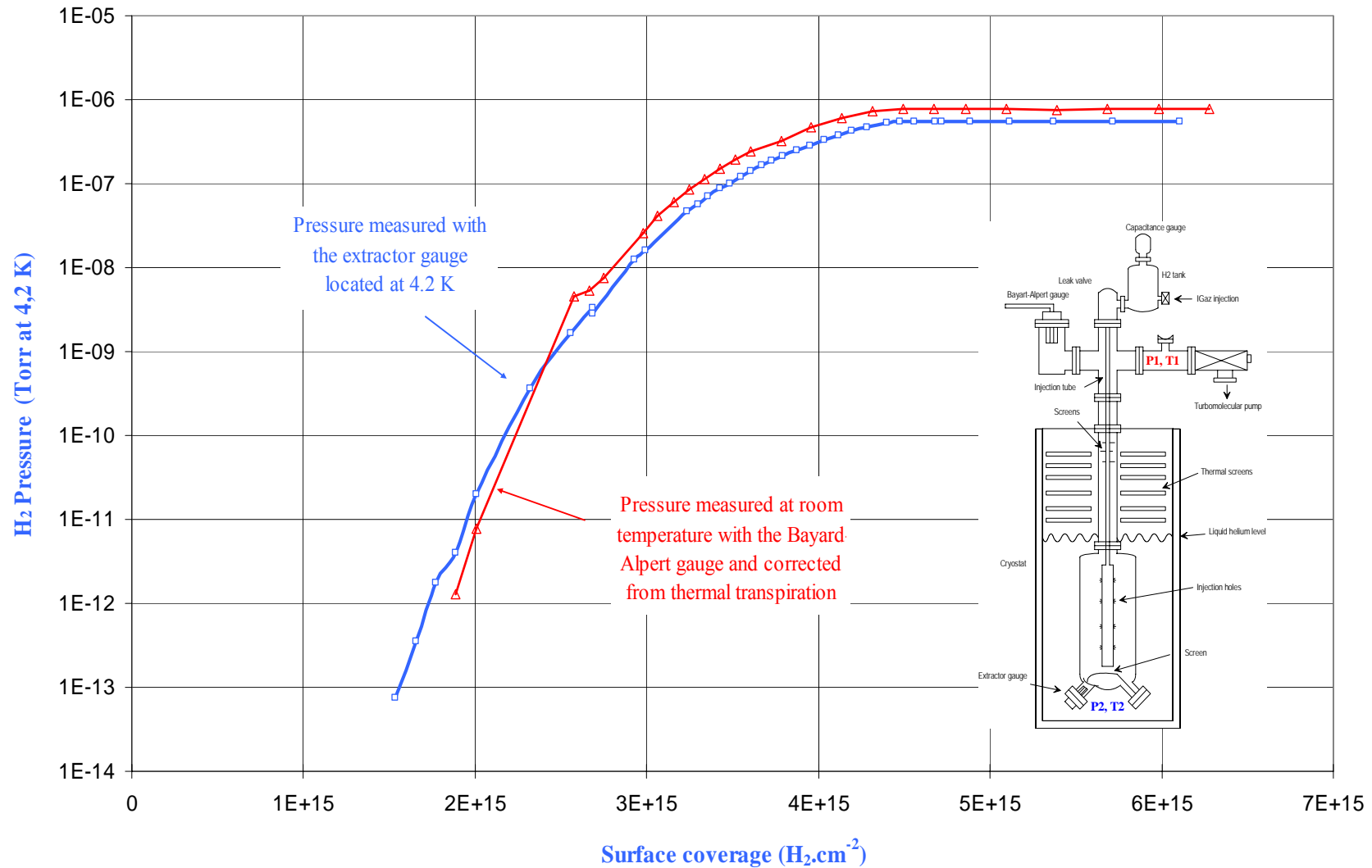
$$\frac{n_1}{n_2} = \sqrt{\frac{T_2}{T_1}}$$

T (K)	4.2	77
$P_1/P_2$	8	2



# Experimental evidence of thermal transpiration

## Static conditions



V. Baglin *et al.* CERN Vacuum Technical Note 1995

## 2. Adsorption isotherms

# Adsorption isotherm

- Measurement, at constant temperature, of the equilibrium pressure for a given gas coverage,  $\theta$
- Varies with:
  - molecular species
  - surface temperature (under 20 K only H<sub>2</sub> and He)
  - surface nature
  - gas composition inside the chamber
  - ...
- Models :
  - Henry's law for low surface coverage

$$\theta = c P$$

DRK (Dubinin, Radushkevich and Kaganer) for metallic, glass and porous substrate. Valid at low pressure. Good prediction with temperature variation

$$\ln(\theta) = \ln(\theta_m) - D \left( kT \ln \left( \frac{P_{\text{Sat}}}{P} \right) \right)^2$$

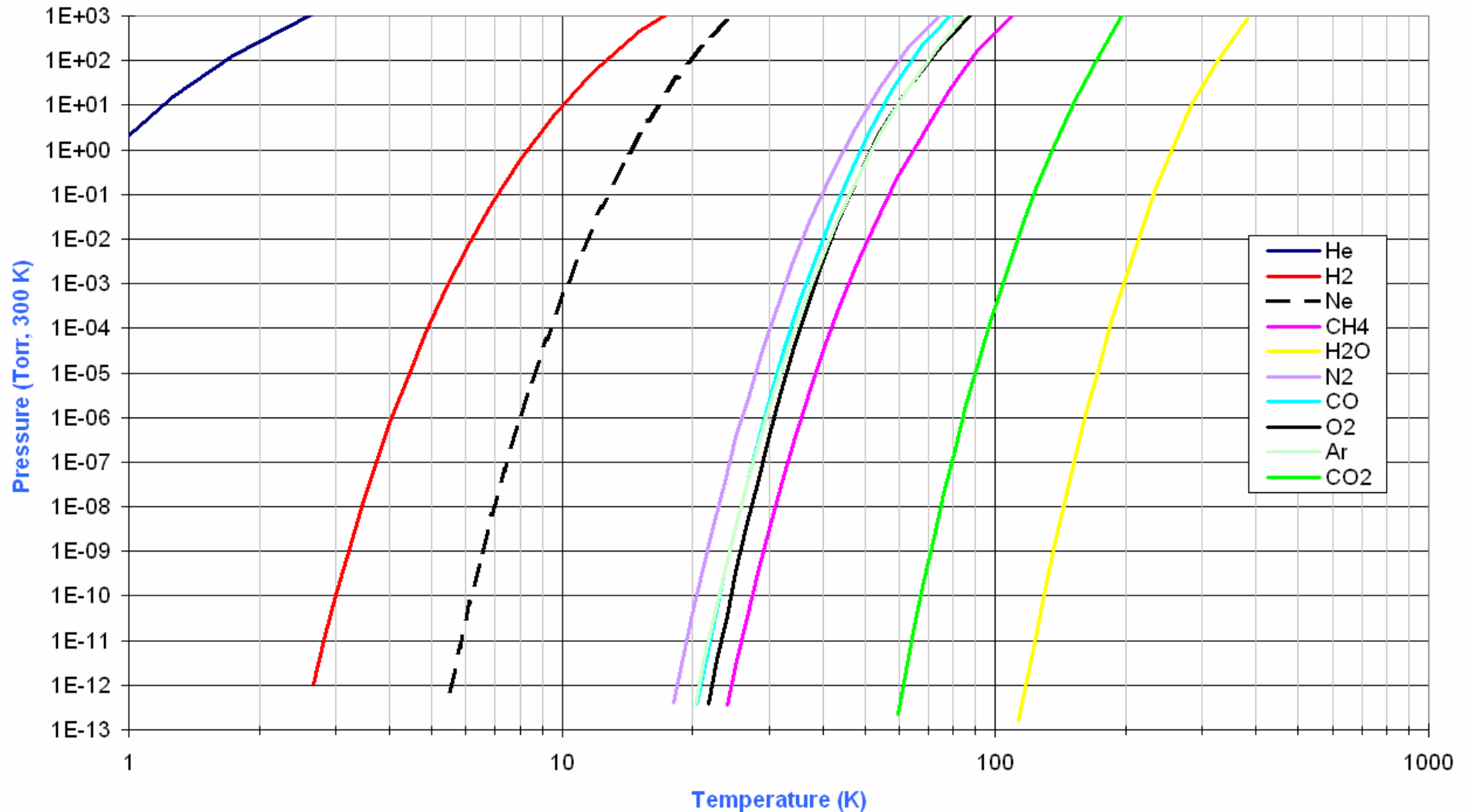
BET (Brunauer, Emmet and Teller). Multi-monolayer description

$$\frac{P}{\theta (P - P_{\text{Sat}})} = \frac{1}{\alpha \theta_m} + \frac{(\alpha - 1)}{\alpha \theta_m} \frac{P}{P_{\text{Sat}}}$$

# Saturated vapour pressure

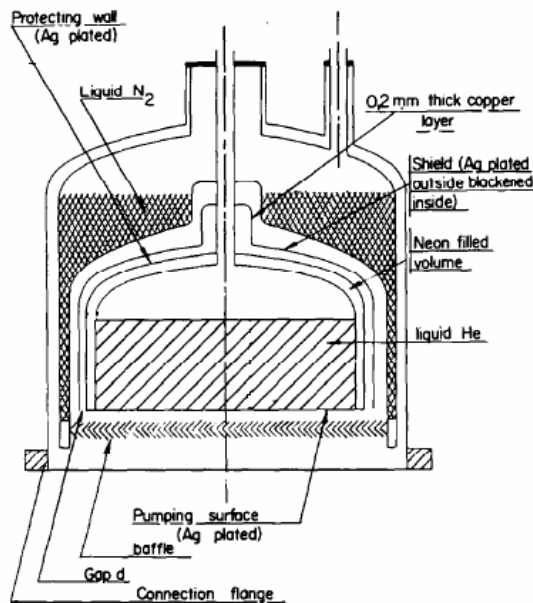
- Pressure over liquid or gas phase (many monolayers condensed)
- Clausius-Clapeyron equation :  $\text{Log } P_{\text{sat}} = A - B/T$

Saturated vapour pressure from Honig and Hook (1960)

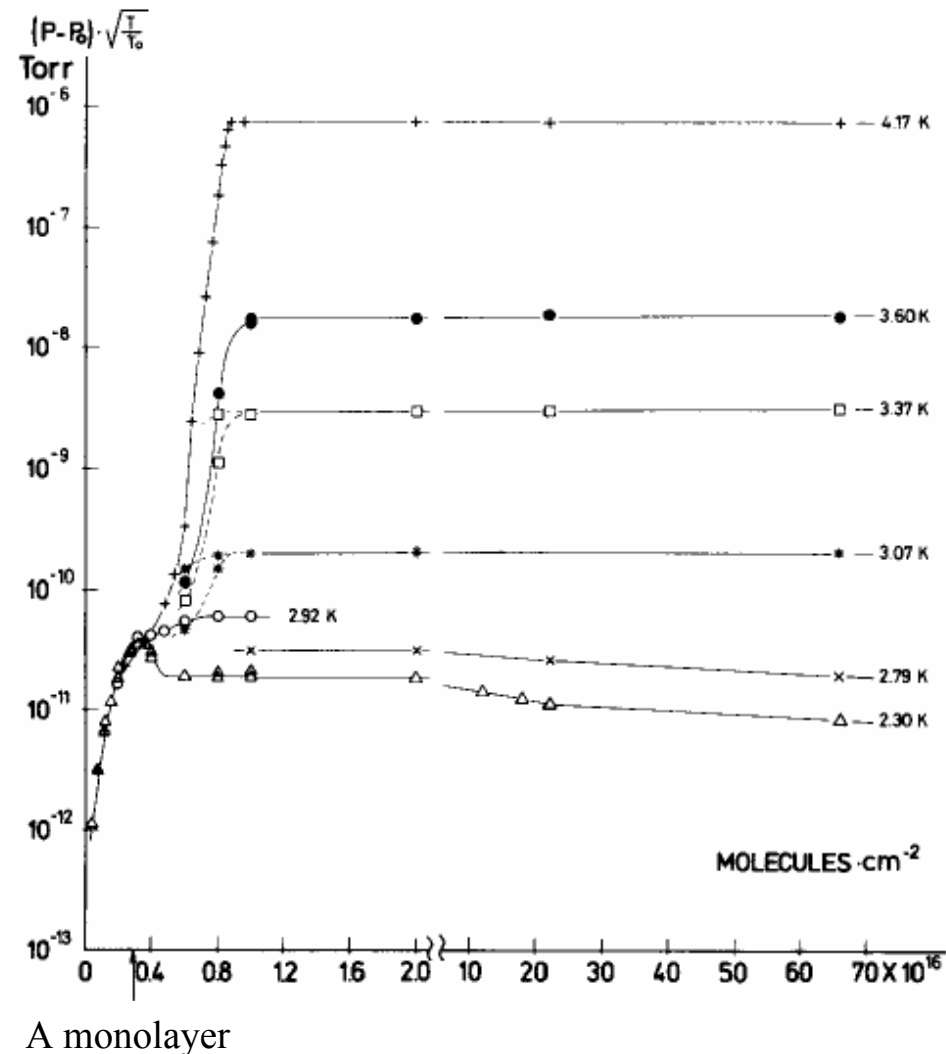


# H<sub>2</sub> adsorption isotherm on stainless steel

- Condensation cryopumps allows to pump large quantities of H<sub>2</sub>
- CERN ISR condensation cryopump operated with liquid He at 2.3 K (50 Torr on the He bath)



C. Benvenuti *et al.* Vacuum, 29, 11-12, (1974) 591



C. Benvenuti *et al.* J.Vac.Sci. 13(6), Nov/Dec 1976, 1172-1182



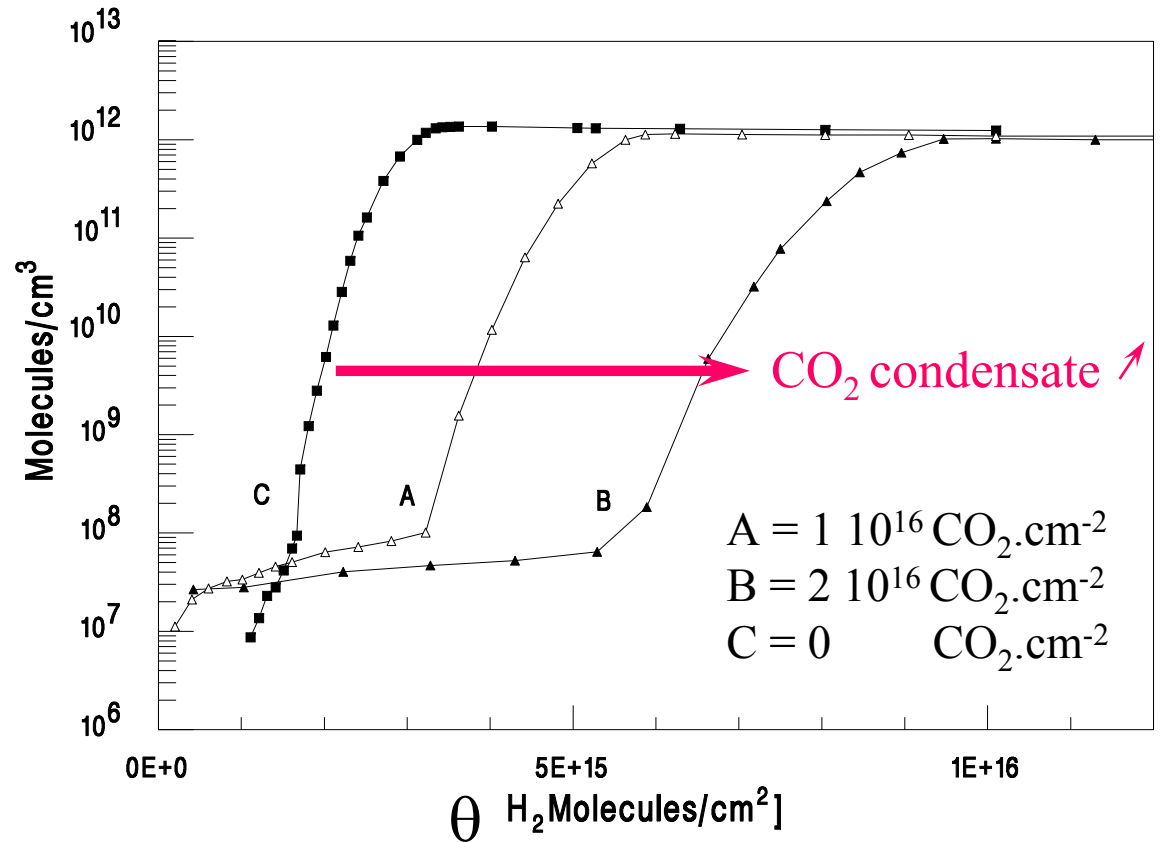
# H<sub>2</sub> adsorption isotherm at 4.2 K on CO<sub>2</sub> condensat

- Packing growth for CO<sub>2</sub> films

⇒ Porous layer

- DRK adsorption capacity :  
0.3 H<sub>2</sub>/CO<sub>2</sub>

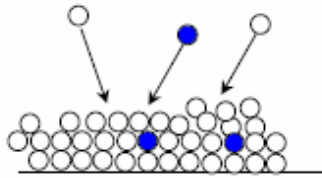
- Electroplated Cu



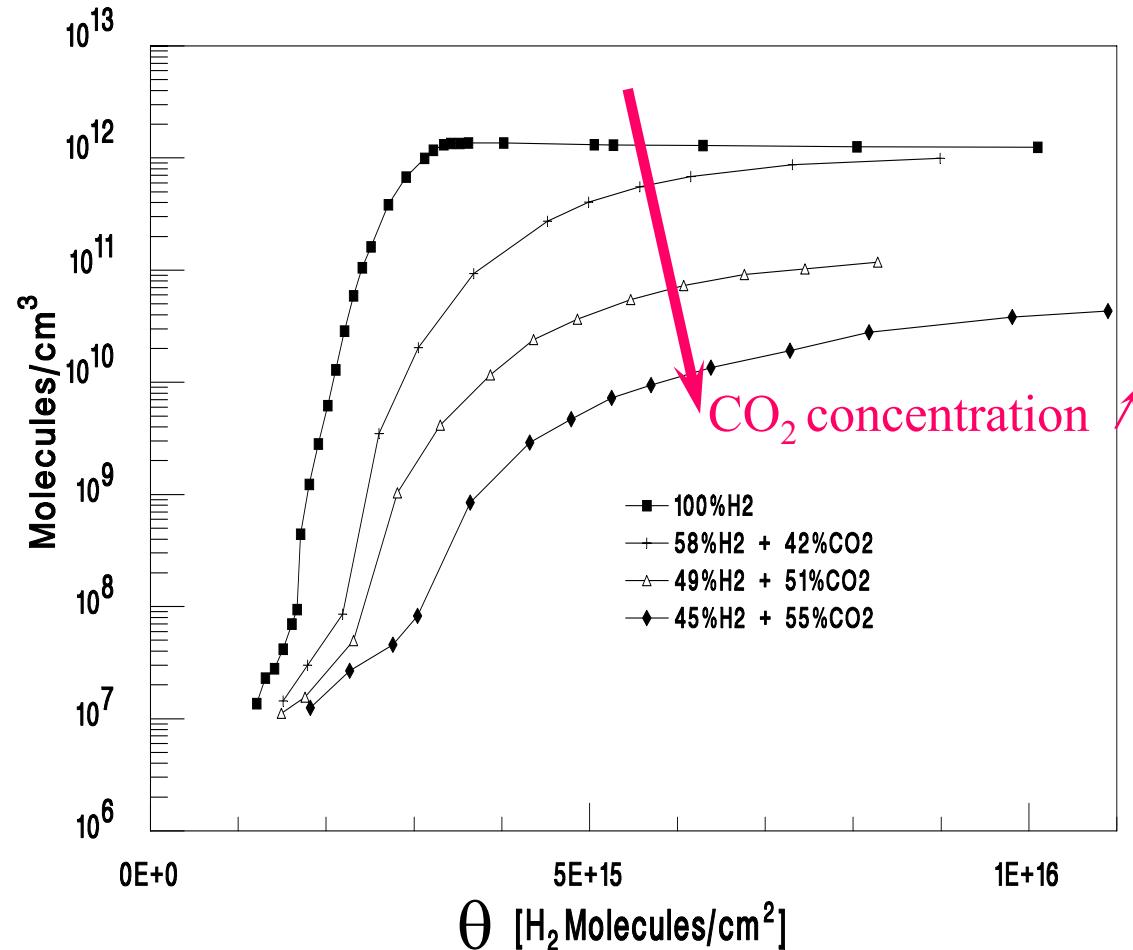
# H<sub>2</sub> adsorption isotherm at 4.2 K in co-adsorption with CO<sub>2</sub>

- Reduction of the saturated vapour pressure by orders of magnitude

⇒ Cryotrapping

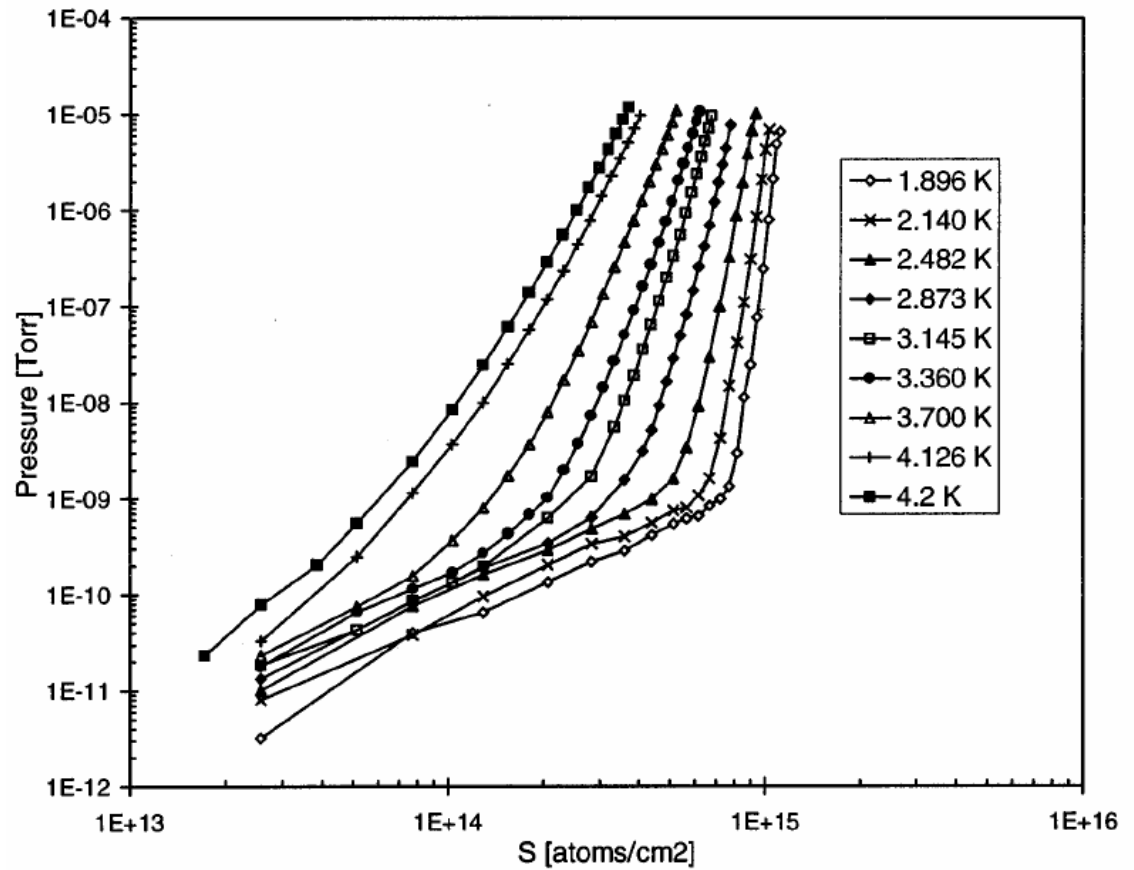


- Electroplated Cu
- In cryopumps CO<sub>2</sub> is admitted to enhance the pumping of H<sub>2</sub> and He



# He adsorption isotherm from 1.9 to 4.2 K

- Sub-monolayer range
- Approach of Henry's law at low coverage
- The isotherms are well described by the DRK model
- $\theta_m \sim 1.3 \cdot 10^{15} \text{ H}_2/\text{cm}^2$
- Stainless steel



E. Wallén. J.Vac.Sci.A 15(2), Mar/Apr 1997, 265-274.

# Cryosorbing materials

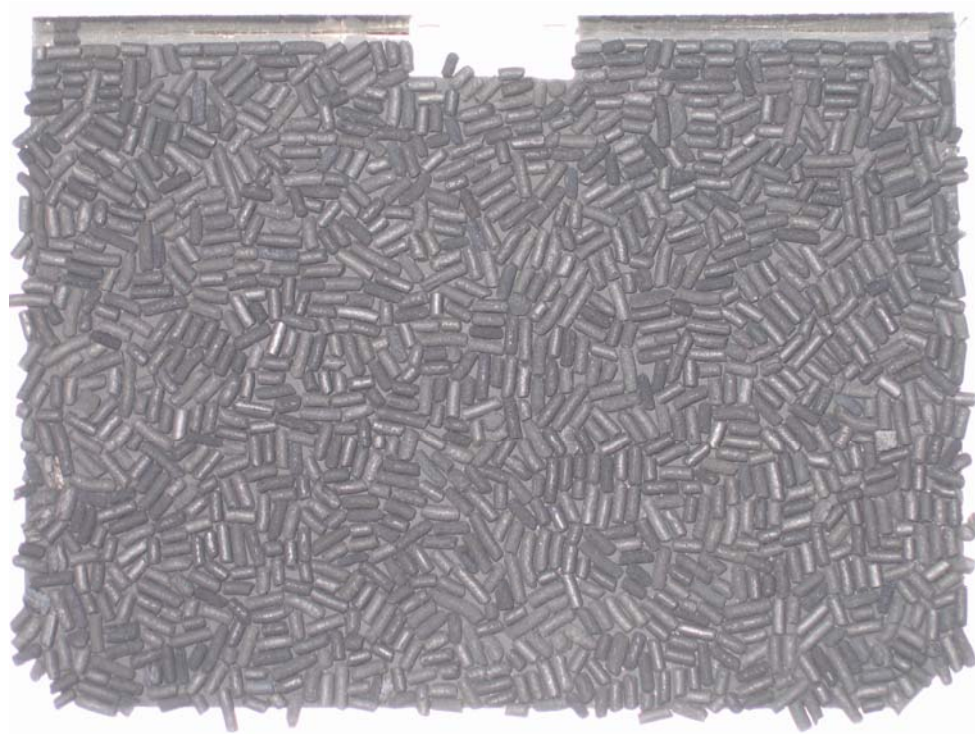
- Large capacity
- Large pumping speed
- Large temperature working range (up to  $\sim 30$  K)

*e.g.* **Activated Charcoal** used for cryopumps (see cryopumps talk)

Capacity  $\sim 10^{22}$  H<sub>2</sub>/g *i.e.*  $10^{21}$  monolayers (P. Redhead, Physical basis of UHV, 1968)

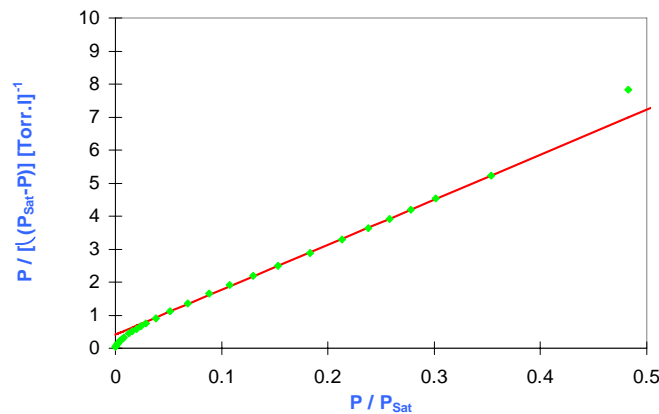
Sticking coefficient  $\sim 30$  % at 30 K (T. Satake, Fus. Tech. Vol 6., Sept. 1984)

20 K cryopanel



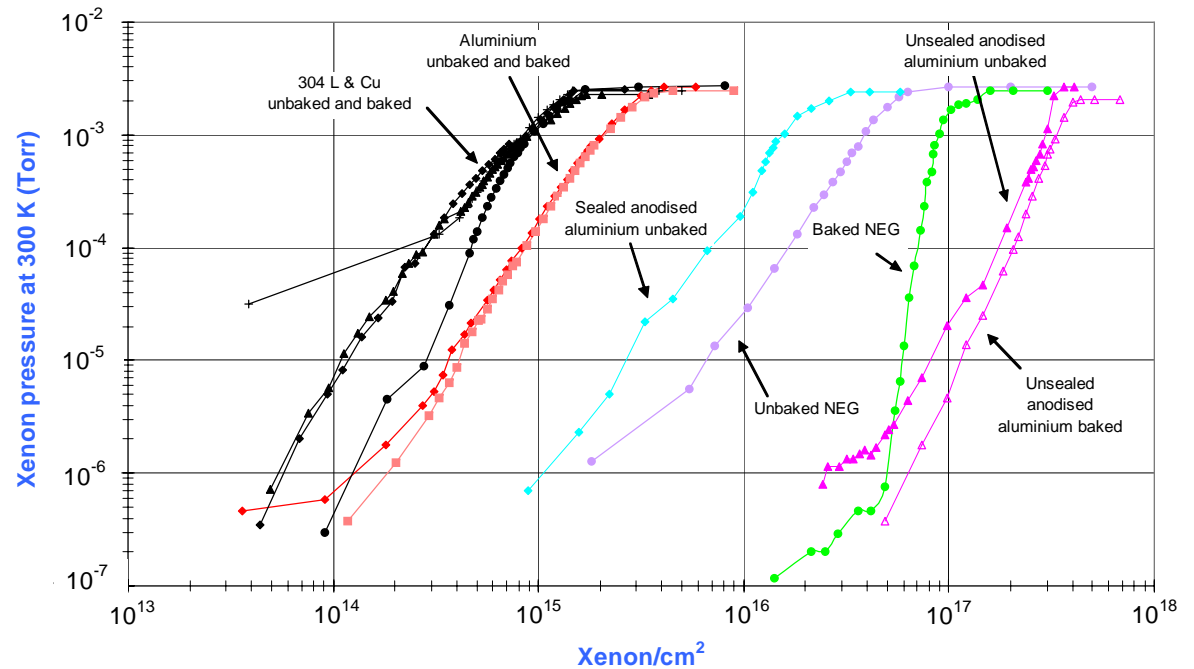
# B.E.T surface area – Roughness factor

- Multi-monolayers theory
- Valid for  $0.01 < P/P_{\text{sat}} < 0.3$
- BET monolayer =  $\theta_m$
- $\alpha = \exp(\Delta E/kT) \gg 1$



$$\frac{P}{\theta (P - P_{\text{Sat}})} = \frac{1}{\alpha \theta_m} + \frac{(\alpha - 1)}{\alpha \theta_m} \frac{P}{P_{\text{Sat}}} \approx \frac{1}{\theta_m} \frac{P}{P_{\text{Sat}}}$$

$$R = \frac{A_R}{A_G} = \frac{A \times \theta_m}{A_G}$$

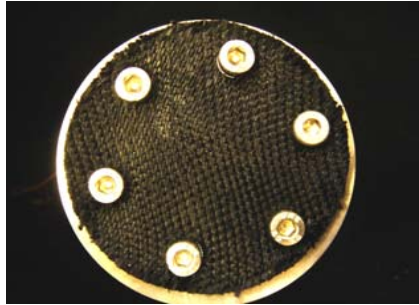


V. Baglin, CERN Vacuum Technical Note 1997

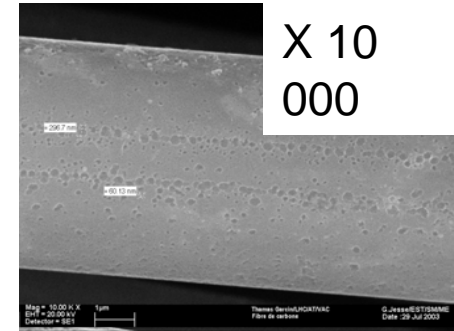
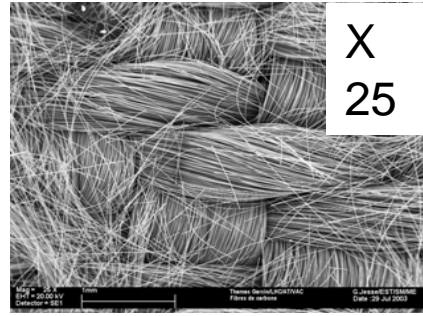
Technical surface	Unbaked	Baked at 150 °C
Copper Cu-DHP acid etched	1,4	1,9
Stainless steel 304 L vacuum fired	1,3	1,5 (at 300 °C)
Aluminium degreased	3,5	3,5
Sealed anodised aluminium 12 V	24,9	not measured
Unsealed anodised aluminium 12 V	537,5	556,0
NEG St 707	70,3	156,3

# H<sub>2</sub> adsorption isotherm on cryosorbers

Woven carbon fiber developed by BINP



V. Anashin *et al.* Vacuum 75 (2004) 293-299

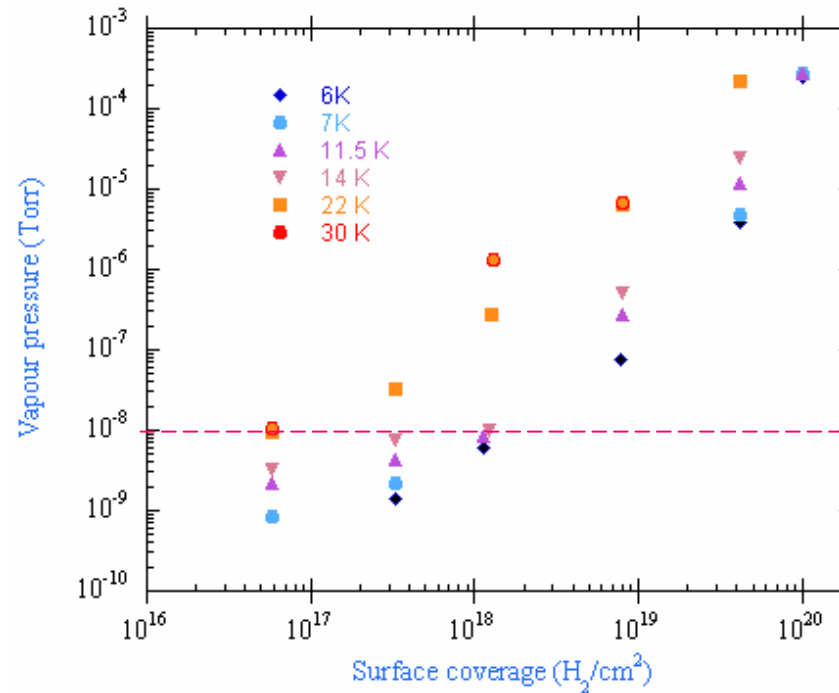


- Capacity :

$10^{18}$  H<sub>2</sub>/cm<sup>2</sup> at 6 K

$10^{17}$  H<sub>2</sub>/cm<sup>2</sup> at 30 K

$$R \sim 10^3 R_{\text{Cu}}$$



V. Baglin *et al.* EPAC'04, Luzern 2004.

### 3. Cryosorbers in cold systems.

Case of the LHC superconducting magnets  
operating at 4.5 K

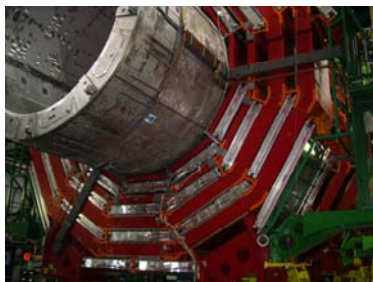


# The CERN Large Hadron Collider (LHC)

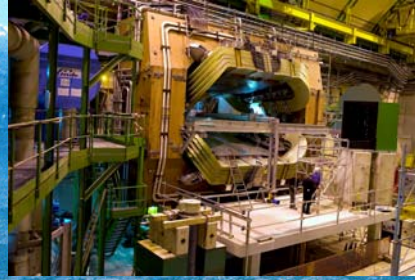


- 26.7 km circumference
- 8 arcs of 2.8 km
- 8 long straight sections of 575 m
- 4 experiments
- 7 TeV
- 1<sup>st</sup> beam in summer 2007

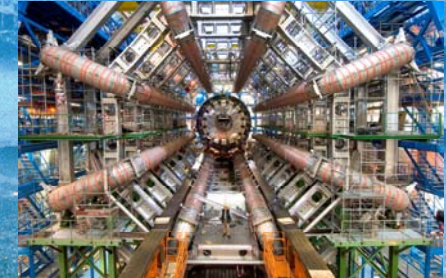
CMS



LHCb



ATLAS



ALICE



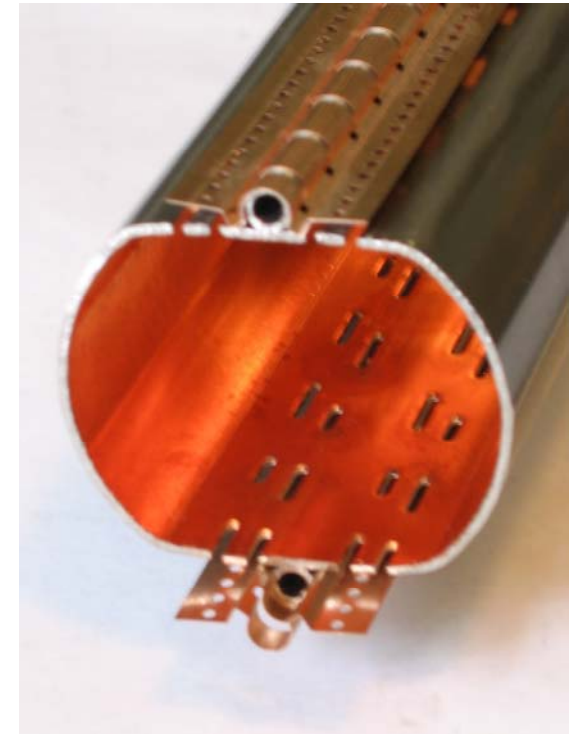
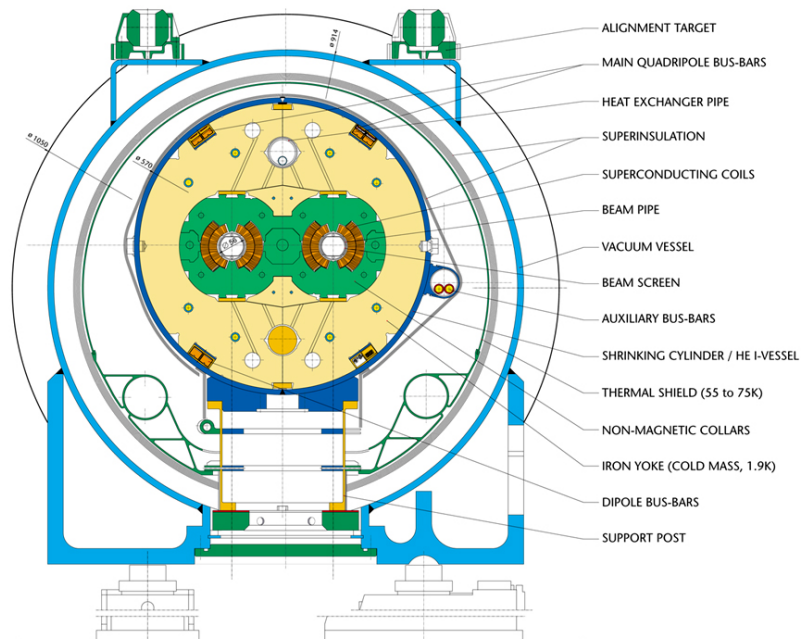


# LHC dipole vacuum system



- Cold bore (CB) at 1.9 K
- Beam screen (BS) at 5-20 K (intercept thermal loads)

**LHC DIPOLE : STANDARD CROSS-SECTION**



CERN AC/DI/MM - HE107 - 30 04 1999

# LHC vacuum system principle

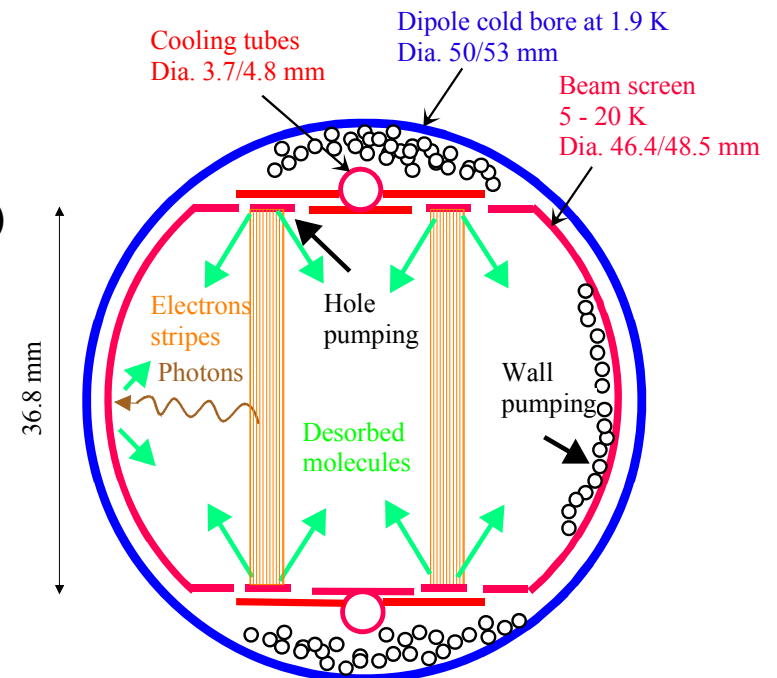
- Molecular desorption stimulated by photon, electron and ion bombardment (see beam-vacuum talks)
- Desorbed molecules are pumped on the beam vacuum chamber
- 100 h beam life time (nuclear scattering) equivalent to  $\sim 10^{-8}$  Torr  $H_2$  at 300 K

## In room temperature elements

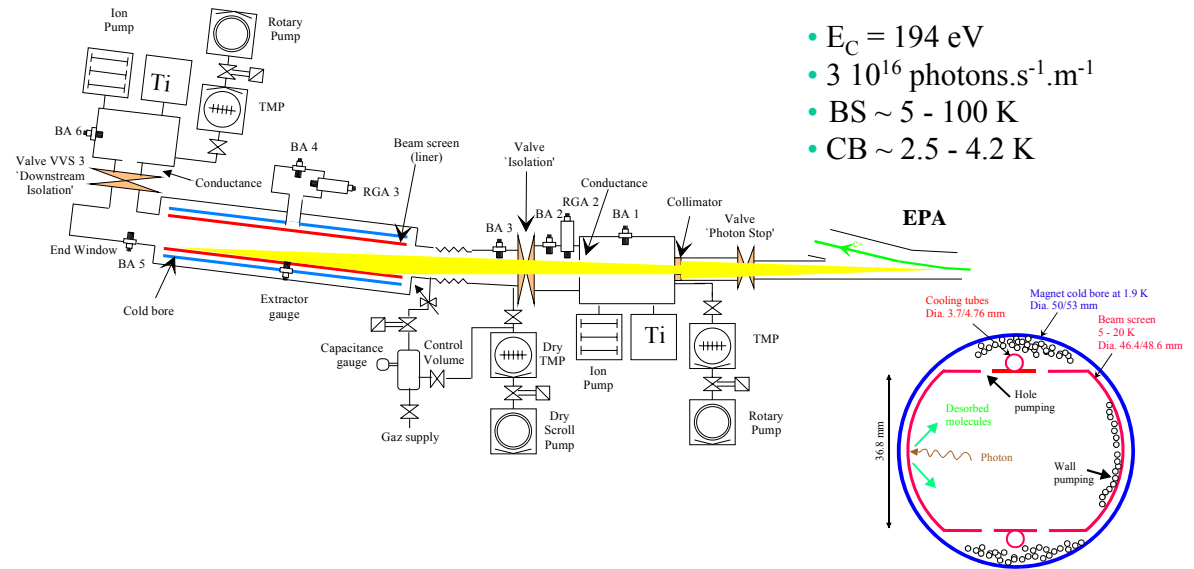
- Molecular **chemisorption** on NEG coating and in sputter ion pump (see getter talks)

## In cryogenic elements

- Molecular **physisorption** onto cryogenic surfaces (weak binding energy)
- Molecules with a low recycling yield are **first physisorbed onto the beam screen** ( $CH_4$ ,  $H_2O$ ,  $CO$ ,  $CO_2$ ) and **then onto the cold bore**
- $H_2$  is physisorbed onto the cold bore



# LHC dynamic pressure with perforated beam screen

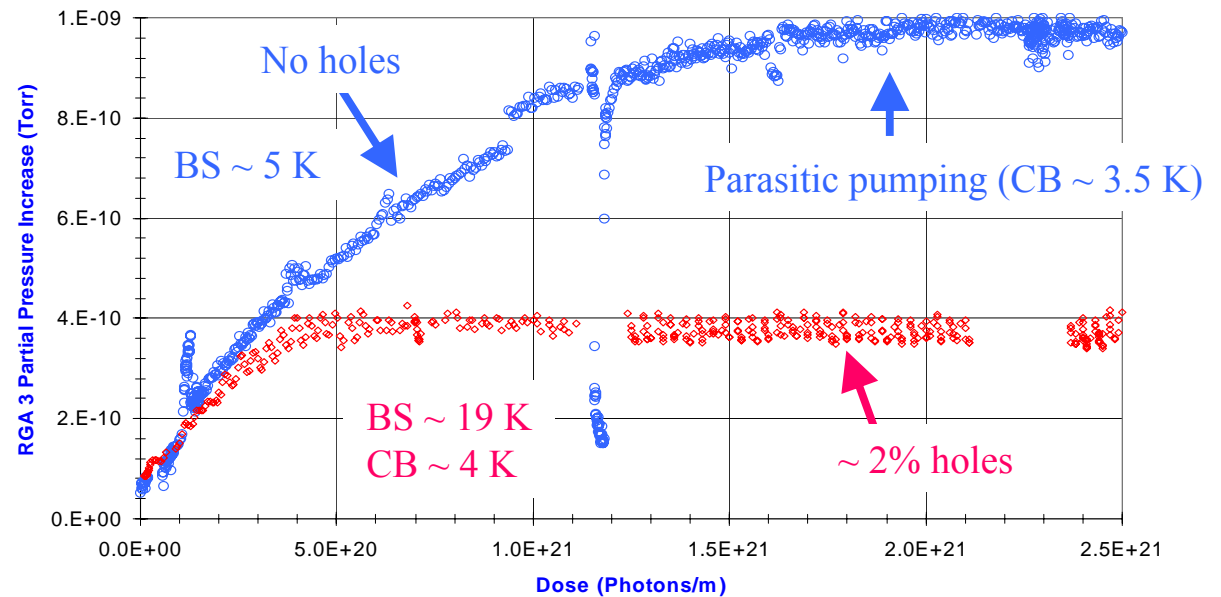


- Equilibrium pressure

$$n_{eq} = \frac{\eta \dot{\Gamma}}{C}$$

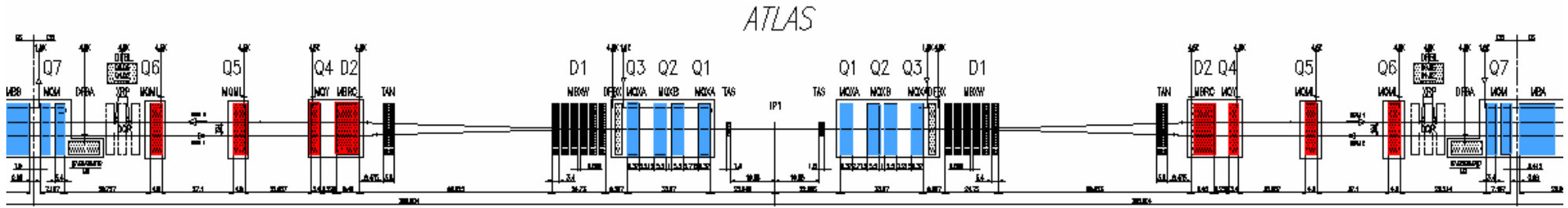
- Equilibrium coverage

$$\theta_{eq} = \left( \frac{\sigma S}{C} \frac{\eta}{\eta_0} \right) \theta_m$$

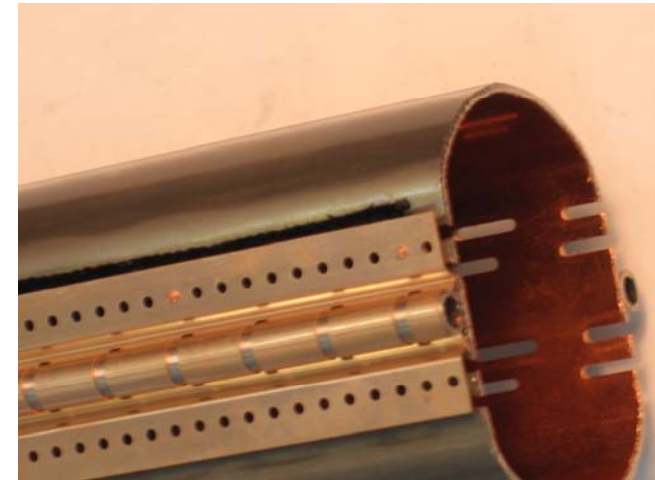


V. Baglin *et al.* EPAC'00, Vienna 2000.

# LHC Long straight section vacuum system

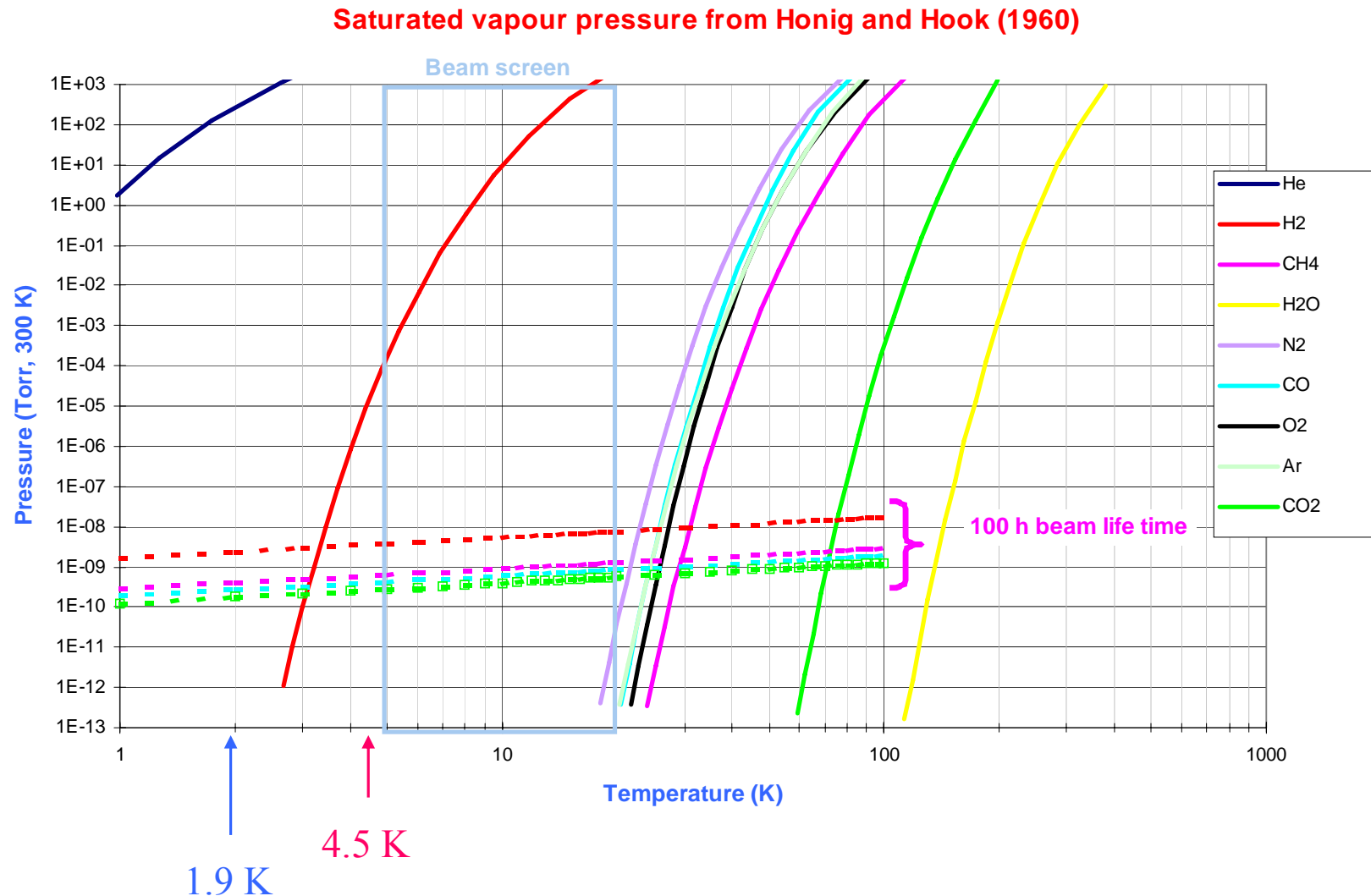


- Dynamic vacuum  $\Rightarrow$  perforated beam screens
- 1.9 K cold bore ( $\sim 660$  m, arc beam screen technology)
- $\sim 4.5$  K cold bore ( $\sim 740$  m)  $\Rightarrow$  cryosorbers
- Required performances (for installation of  $200 \text{ cm}^2/\text{m}$ ):
  - Operates from 5 to 20 K
  - Capacity larger than  $10^{18} \text{ H}_2/\text{cm}^2$
  - Capture coefficient larger than 15 %



# Why cryosorbers ?

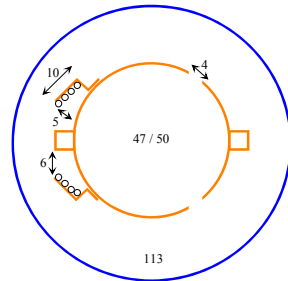
Design requires  $> 100$  h life time with 4.5 K cold bore and thick surface coverage  
 $\Rightarrow$  porous surface



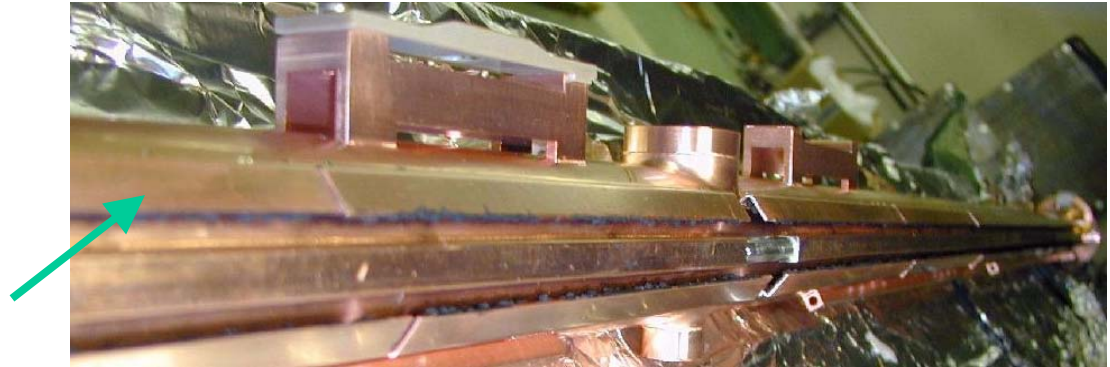


# Performance with perforated cryosorbing screens

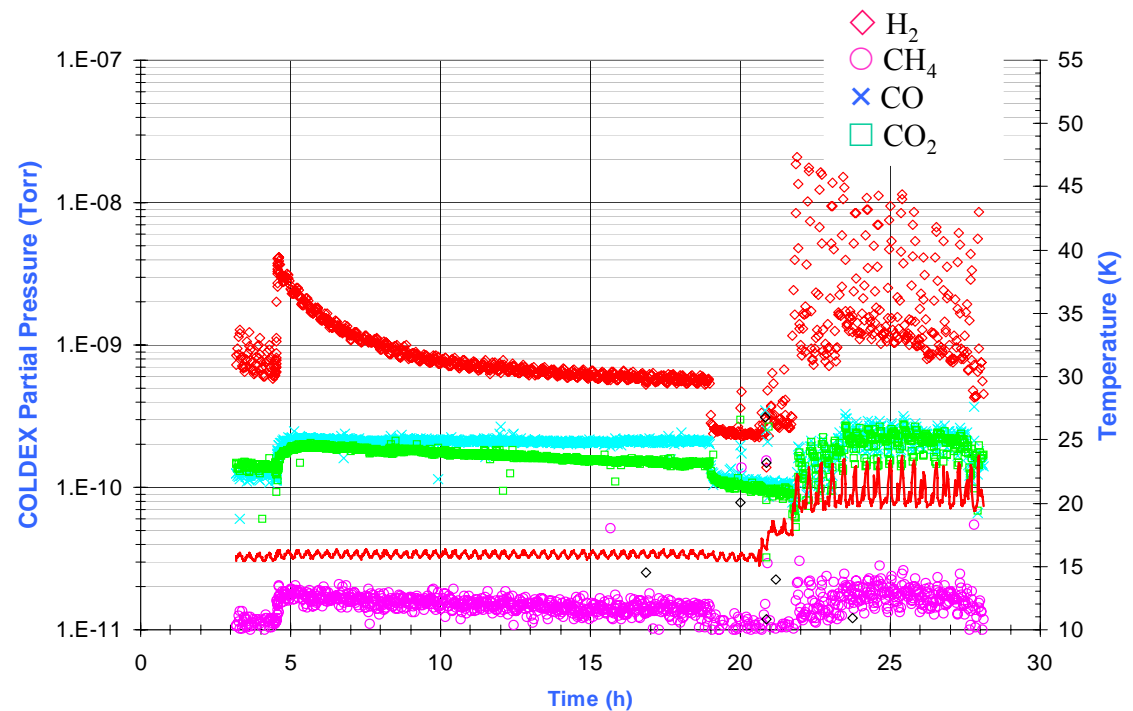
- ~ 2 % holes
- Charcoal cryosorber
- $206 \text{ cm}^2 \cdot \text{m}^{-1}$
- OFE Cu
- 2.2 m
- ID 47 mm



Strips with charcoal



- 100 monolayers of  $\text{H}_2$  condensed onto the beam screen prior SR irradiation
- $2 \cdot 10^{19} \text{ H}_2/\text{cm}^2$  condensed onto the cryosorber (20 x required capacity)
- $\text{CB} > 70 \text{ K}$
- BS ~15 and 20 K
- Below design pressure ( $10^{-8} \text{ Torr}$ )



V. Baglin *et al.* EPAC'02, Paris 2002.

# LSS cryosorbers regeneration

## Principle of operation

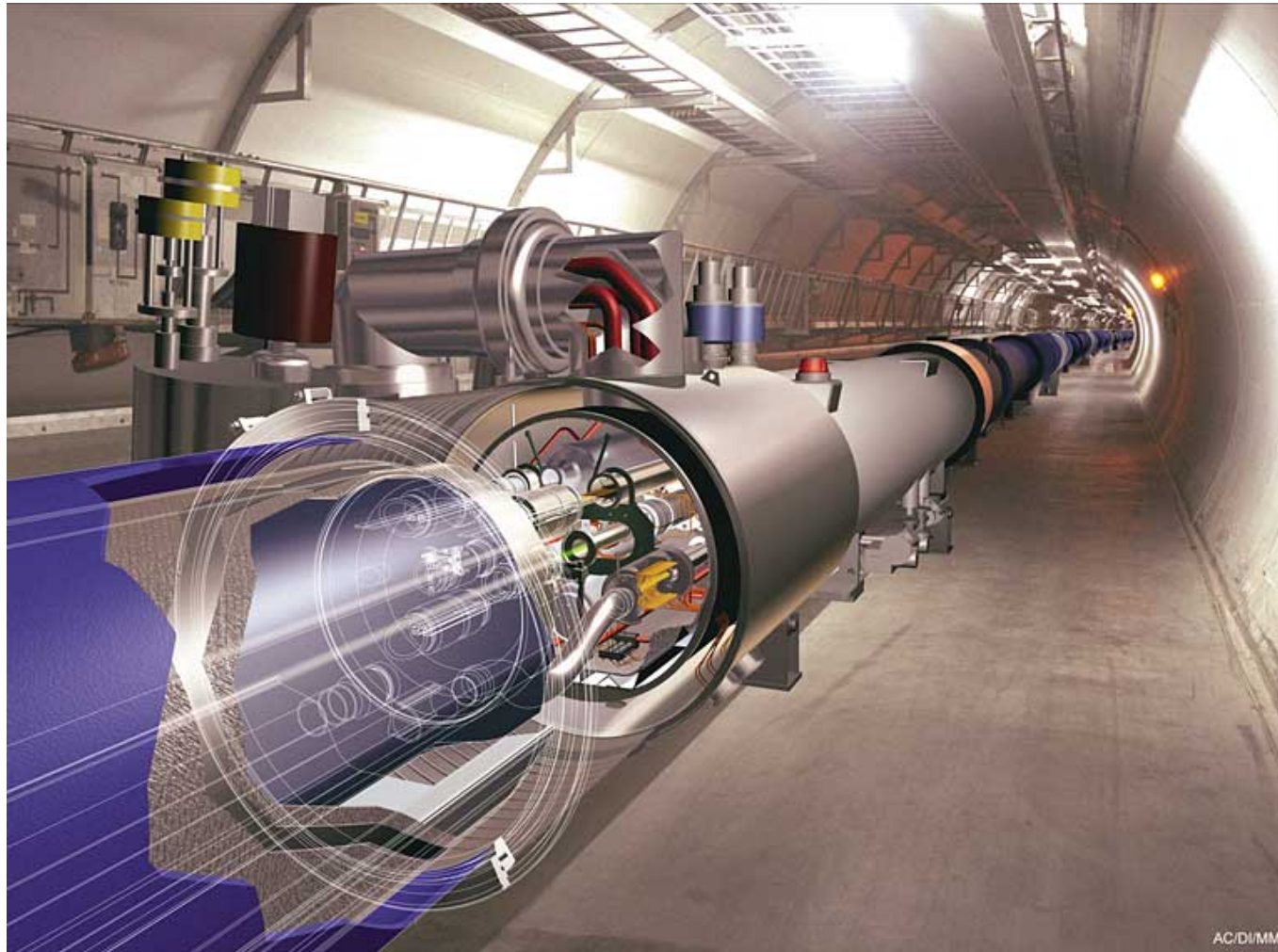
- The cryosorbers installed onto the back of the BS provide the required capacity and pumping speed for H<sub>2</sub>. They are located in cryoelements operating with 4.5 K cold bores
- A regeneration during the annual shutdown for removing the H<sub>2</sub> is required
- The active charcoal is regenerated at ~ 80 K
- While regenerating, the beam is OFF and the BS should be warmed up to more than 80 K and the CB held at more than 20 K (emptying cold mass)
- While the H<sub>2</sub> is desorbed from the cryosorbers, it is pumped by an external pumped system.

## 4. He leaks in cold systems.

LHC beam tube case

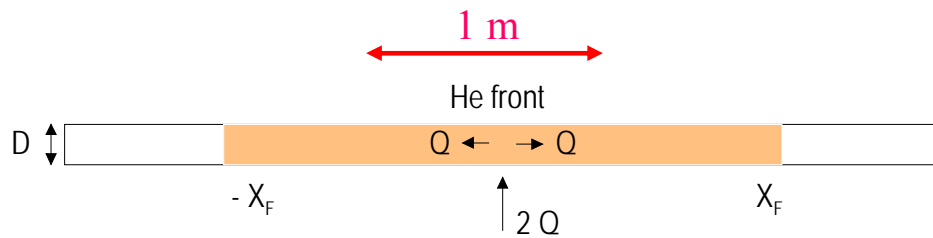


# LHC : Superconducting technology

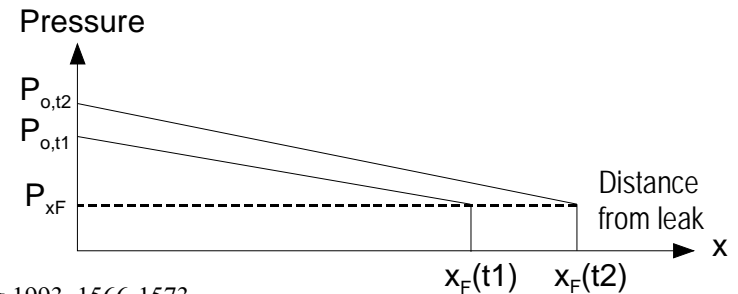


- Air leak or He leaks could appear in the beam tube during operation : the consequences are risk of **magnet quench, pressure bump and radiation dose**

# Description of a He leak



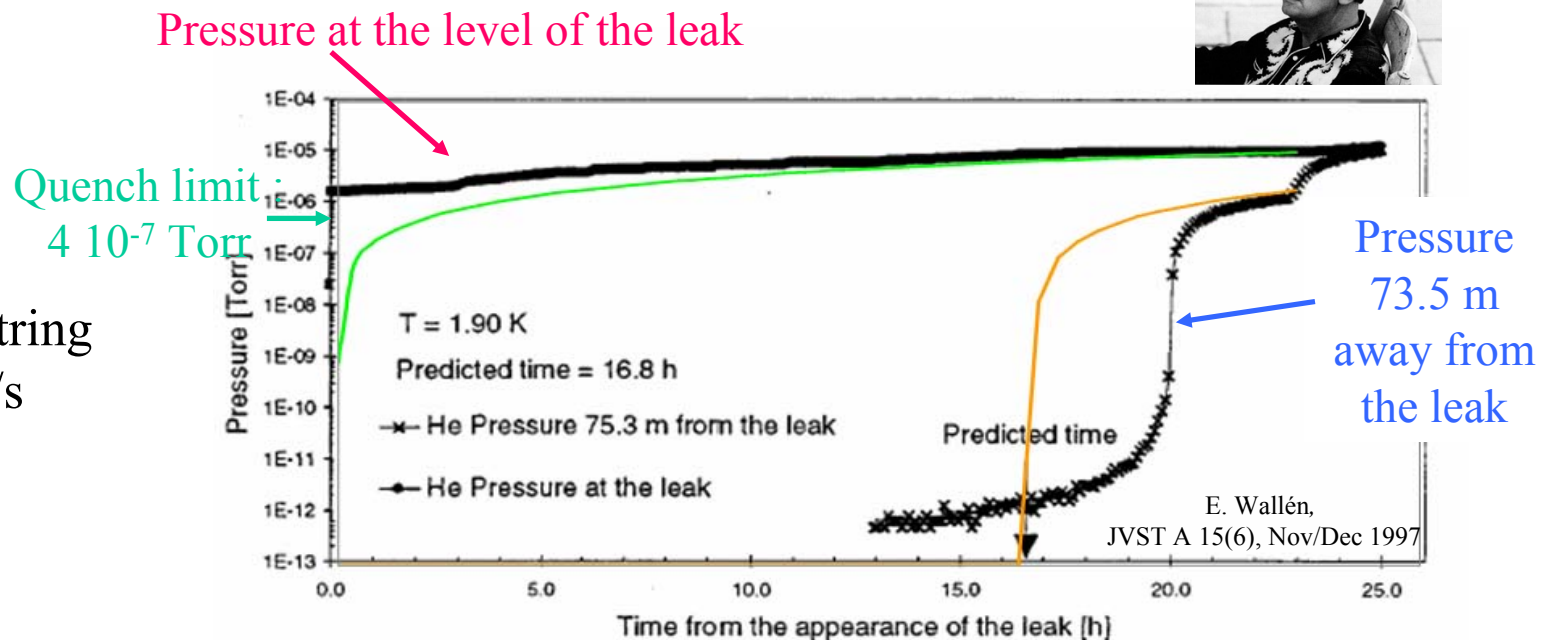
P. Hobson *et al.* J.Vac.Sci. A. 11(4), Jul/Aug 1993, 1566-1573



- A **He pressure wave** is developed with time along the beam vacuum chamber
- The He wave can **span over several tens of meter** without being detected
- The **local pressure bump** gives a local proton loss (risk of quench)



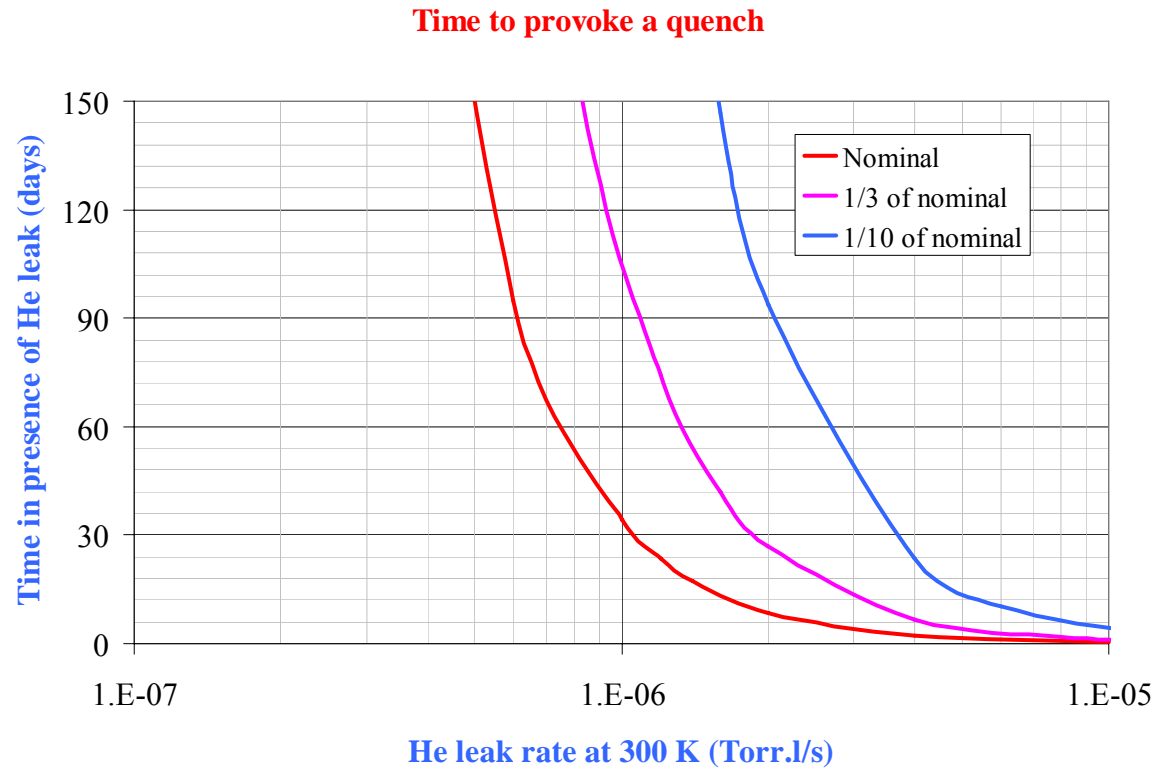
Example : LHC Test string  
Leak rate  $6 \cdot 10^{-5}$  Torr.l/s  
Distance 75.3 m



# He leak rate with risk of quench

- 1 year of operation  $\sim 150$  days

Helium leak rate above  $5 \cdot 10^{-7}$  Torr.l/s shall be detected to avoid the risk of a quench



- Lower leak rate :  
Require a pumping of the beam tube on the yearly basis (cold bore  $> \sim 4$ K)
- Larger leak rate will provoke a magnet quench within :  
30 to 100 days beam operation for He leak rate of  $10^{-6}$  Torr.l/s  
A day of beam operation for He leak rate of  $10^{-5}$  Torr.l/s

# Vacuum gauge diagnostic

- By design, a vacuum gauge is placed every 3 or 4 cells (320-428 m) i.e. 6 to 8 gauges per arc
- Small probability to detect a He leak in the arcs
- At nominal beam current, leak rates **below**  $2 \cdot 10^{-7}$  Torr.l/s are systematically detected by the arc vacuum gauges before a quench occurs

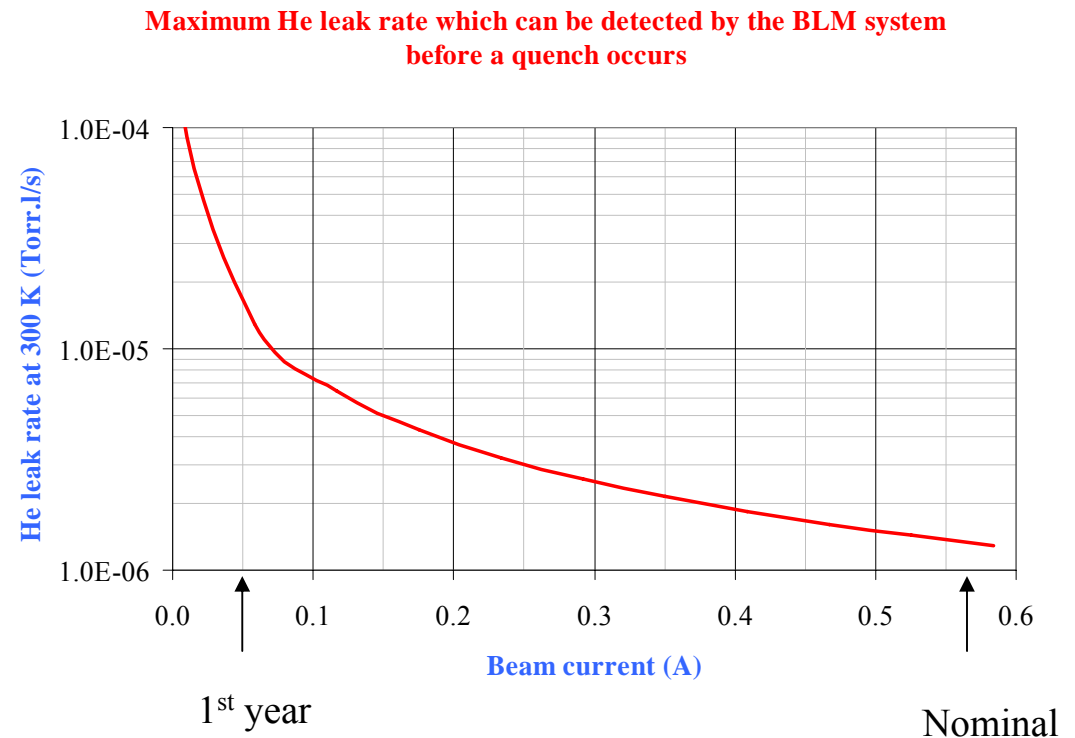
The vacuum gauges does not allow a systematic on line detection of the He leaks in the LHC arc !

- So, one needs to find other diagnostic means

# Beam loss monitor based diagnostic

- BLM provide a measurement every 53 m at each arc quadrupole
- All quench due a He wave half-length larger than 26 m are detected by the BLM

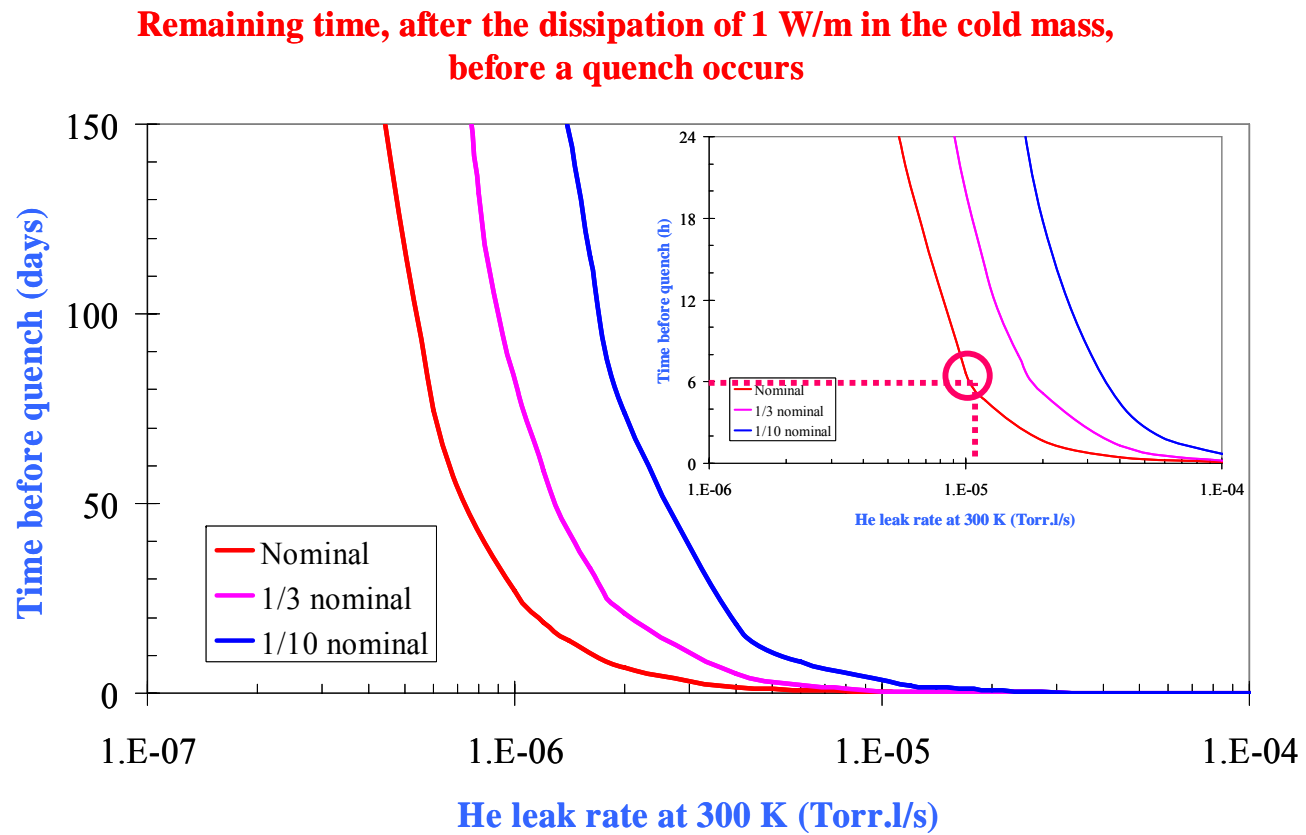
At nominal beam current, leak rates below  $1.3 \cdot 10^{-6}$  Torr.l/s are systematically detected by the BLM system before a quench



# Cryogenic based diagnostic

- Per cell (102 m), a **power** of  $\sim 1$  W/m can be measured in the cold masses
- Requires stable operation to integrate over a long time ( $\sim 6$ h)

At nominal beam current, **leak rates below  $1 \cdot 10^{-5}$  Torr.l/s** are systematically detected by the cryogenic system before a quench



# Suspected leak in a given area

(1)

- If possible, the cold mass temperature shall be temporarily increased to 4 K to **move the He front** towards the nearest short straight section
- **Vacuum gauge or residual gas analyser** can be **locally** installed, **at this short straight section**, in the tunnel for a leak detection

(2)

- **Mobile radiation monitors** can be installed to monitor a “radiation front” while the beam is circulating

## In case of a quench

- After a quench, the faulty magnet is identified by the triggered diode.
- The cold bore is warmed up to more than 30-40 K during the quench
- The He is flushed to the nearest unquenched magnet and condensed over  $\sim 10$  m
- The He shall be flush to the nearest SSS to perform a leak detection with a RGA



# Repair

- Warm up of the cold bore to  $> 4$  K and pump out of the He every month allows to operate with leak rates up to:
  - $2 \cdot 10^{-6}$  Torr.l/s and 1/3 of nominal beam current
  - $4 \cdot 10^{-6}$  Torr.l/s and 1/10 of nominal beam current
- Time estimate  $\sim 1$  day
- Leak rates larger than  $4 \cdot 10^{-6}$  Torr.l/s require an exchange of the magnet

# Exchange of a magnet



Time estimate ~ few weeks !

**BE SURE** that the **OBSERVED QUENCH** is due to a **He LEAK** at **THE GIVEN MAGNET**  
... otherwise ...

# Exchange of a magnet

Lyn Evans

BE SURE that the OBSERVED QUENCH HAD A LEAK at THE GIVEN MAGNET  
... otherwise ...



**The guy responsible of the  
magnet exchange will be ...**



**Thank you for your attention !!!**