



# **Task 4.4 (INFN-CERN): Study vacuum stability at cryogenic temperature**

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LNF-INFN, Frascati (Italy)

&

Presently CERN, Geneva, CH

# Agreed Deliverables.

INFN Frascati will determine vacuum stability and adsorption isotherms at different cryogenic beam-screen operating temperature ranges (**D-4.1**).

## Agreed Objectives.

- LNF-INFN will perform complementary studies on beam-induced stimulated desorption phenomena by photons, electrons and ions.
- These studies rely mainly on experimental samples and require beam-screen prototypes supplied by CERN.
- Description of samples and existing prototypes used as baseline.
- Documentation of vacuum stability and adsorption isotherms at different beam screen operating temperature ranges from laboratory tests.

# Study vacuum stability at cryogenic temperature

- Still a number of interesting open questions
- Study @ LNF on small samples
- Strategy and future plans
- resources

# Study vacuum stability at cryogenic temperature

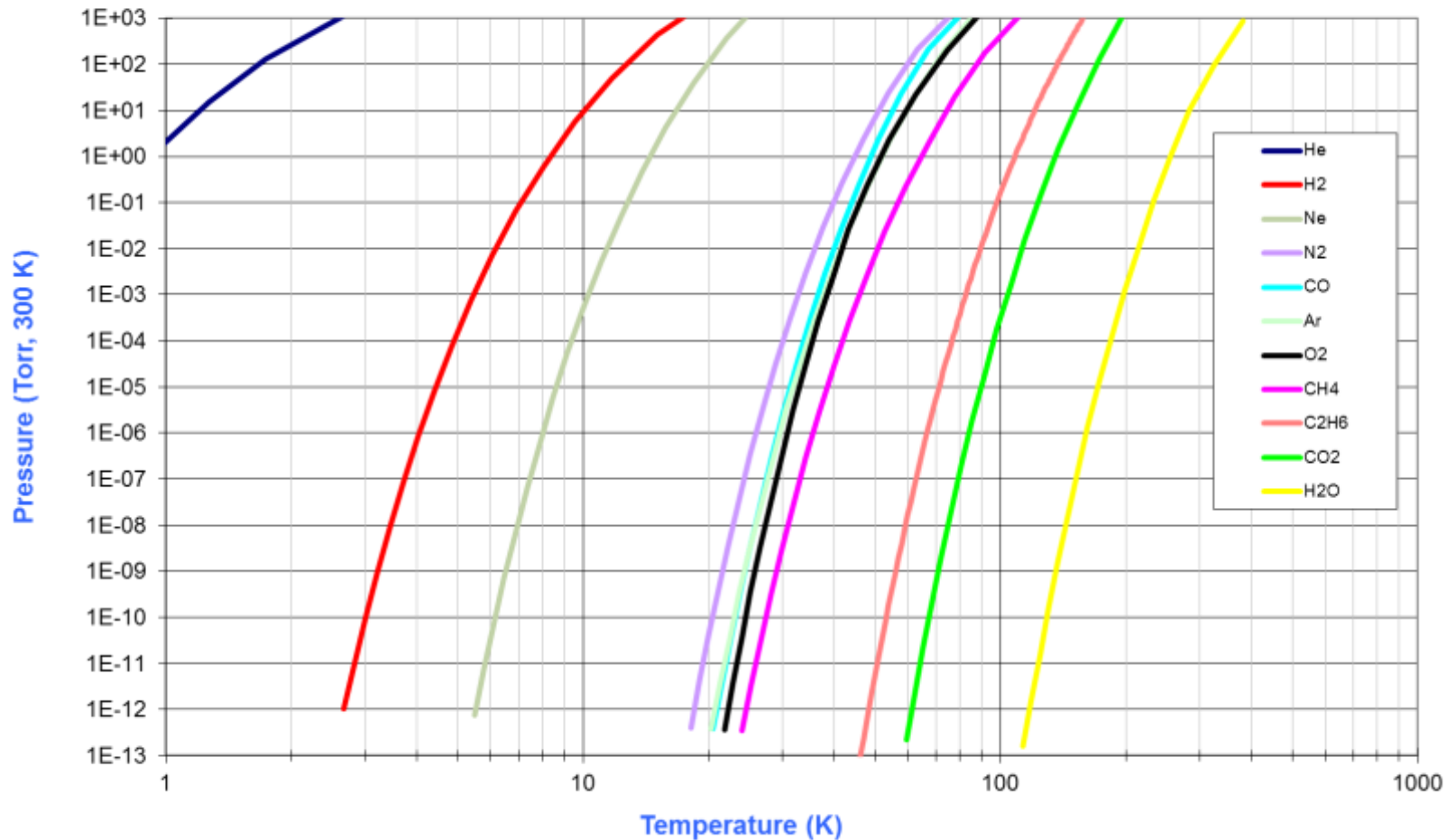
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**From Vincent Baglin talk  
in Washington:**

# Saturated Vapor Pressure

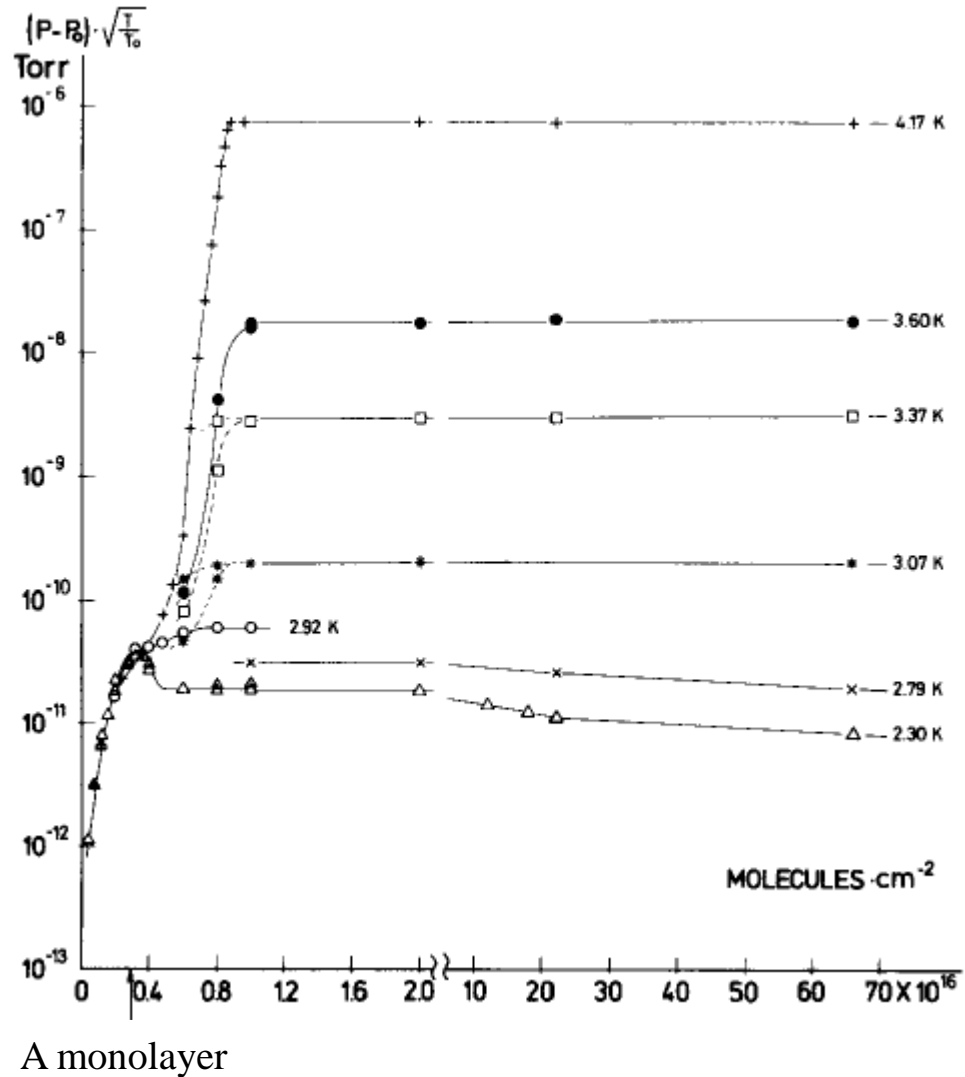
- Pressure over liquid or gas phase (**many** monolayers condensed)
- Follows the Clausius-Clapeyron equation:  $\text{Log } P_{\text{sat}} = A - B/T$

Saturated vapour pressure from Honig and Hook (1960) (C2H6 Thibault et al.)



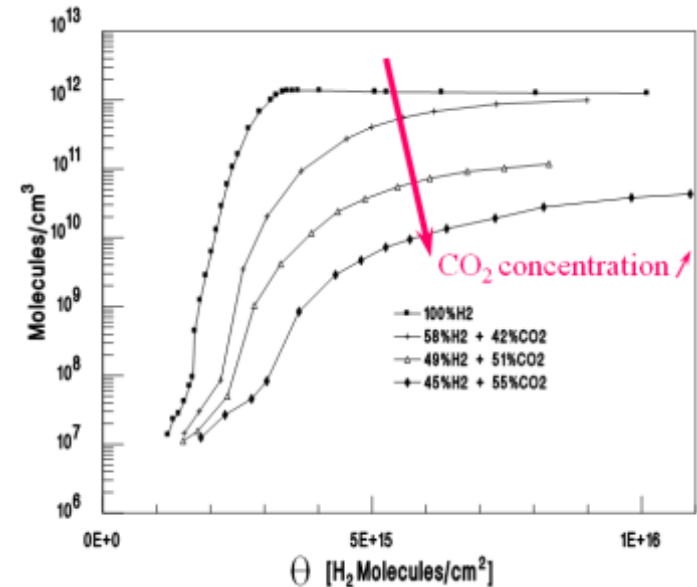
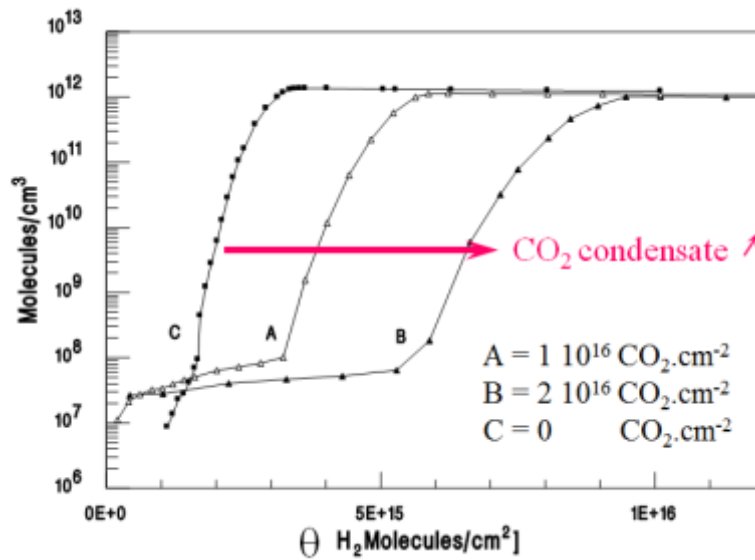
# H<sub>2</sub> adsorption isotherm on stainless steel

- The **vapor pressure** increases when increasing the adsorption of gas up to a few monolayers ( $\sim 10^{15}$  molecules/cm<sup>2</sup>)
- The **vapor pressure saturates** when several monolayers of gas are adsorbed
- The pressure level of the saturation is a function of the **temperature**



# Vapor Pressure in a Machine

- Several types of molecules are present in machine vacuum systems
- The adsorption isotherm is affected by the presence of these molecules
- Condensed CO<sub>2</sub> forms a **porous layer** increasing the hydrogen capacity
- Co-adsorption of CH<sub>4</sub>, CO and CO<sub>2</sub> reduce the vapor pressure of H<sub>2</sub> by **cryotrapping**



E. Wallén, JVSTA 14(5), 2916, Sep./Oct. 1996

→ Studies with real machine environments are mandatory

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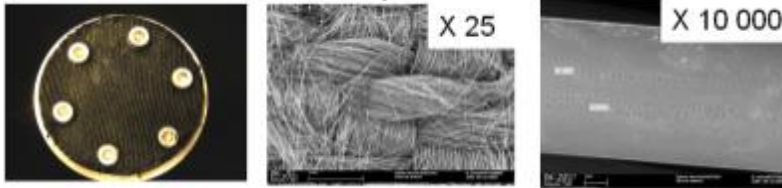
Vincent Baglin talk @ fcc meeting in Washington

# BET surface area – Roughness factor - Cryosorbers

- Xe adsorption isotherms are used to derive the roughness factor of surface using the BET multi-monolayer theory

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

- Woven carbon fibers are used in LHC as cryosorbers in 4.5 K magnets

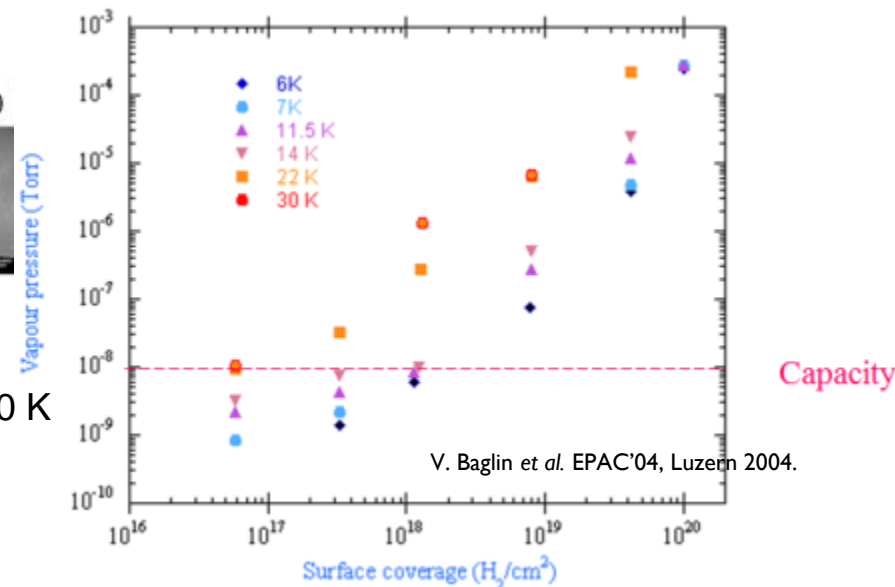
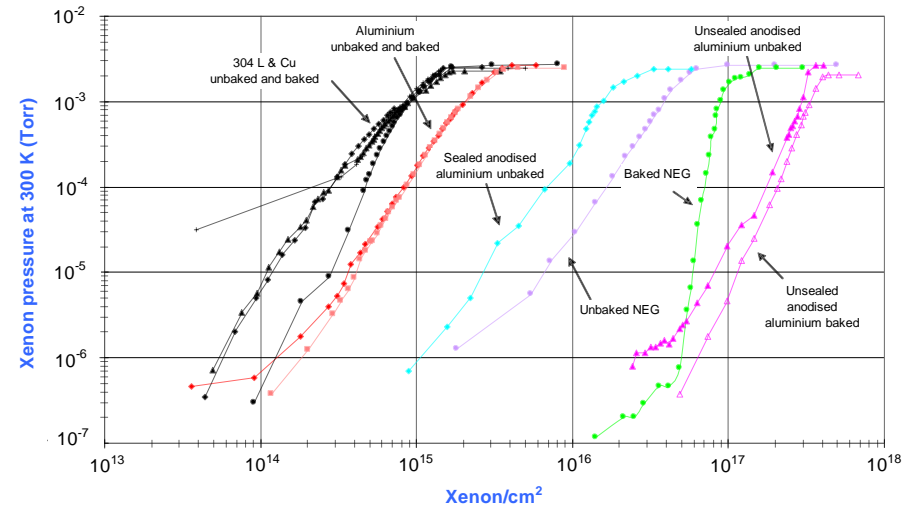


V. Anashin et al. Vacuum 75 (2004) 293-299

- Capacity:  $10^{18}$  H<sub>2</sub>/cm<sup>2</sup> at 6K,  $10^{17}$  H<sub>2</sub>/cm<sup>2</sup> at 30 K

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

V. Baglin. CERN Vacuum Technical Note 1997



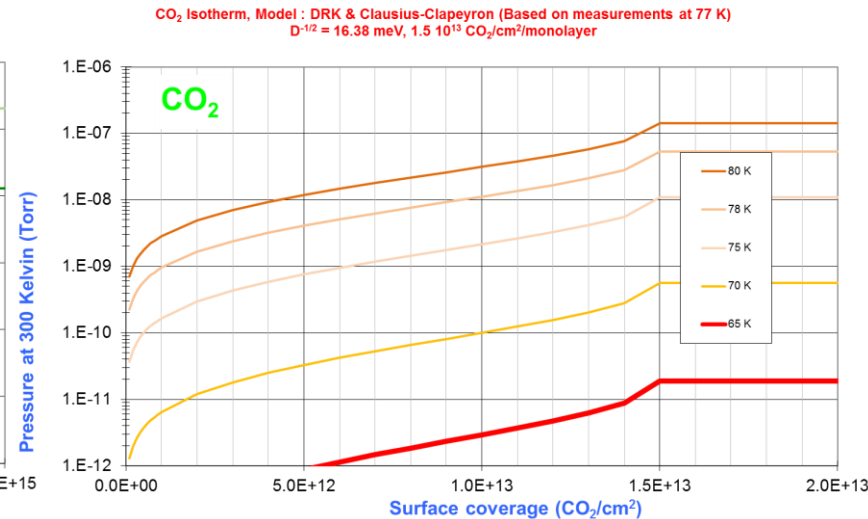
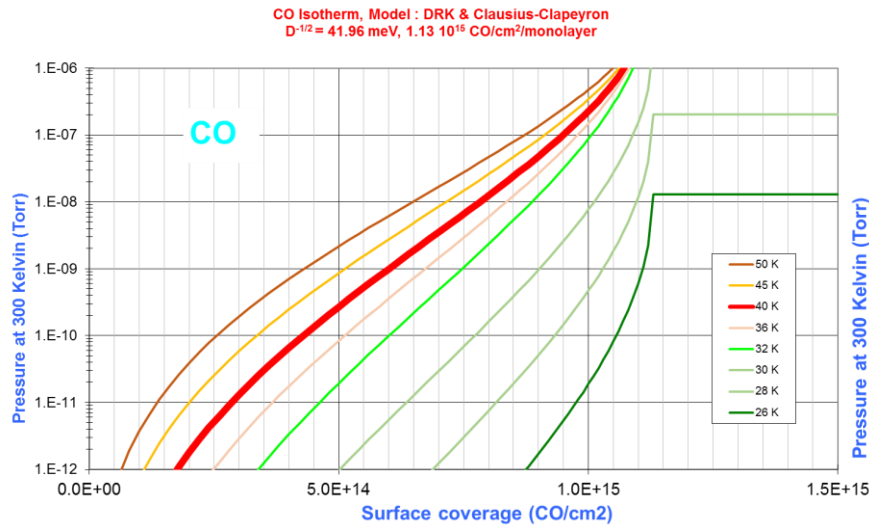
V. Baglin et al. EPAC'04, Luzern 2004.

Vincent Baglin talk @ fcc meeting in Wasington



# Temperature Window

- Temperature excursions of the beam screens must not lead to vacuum transients.
- Above ~ 40 K, CO physisorbed with a sub-monolayer capacity, will be thermally desorbed / condensed
- Above ~ 60 K, CO<sub>2</sub> physisorbed with a sub-monolayer capacity, will be thermally desorbed / condensed

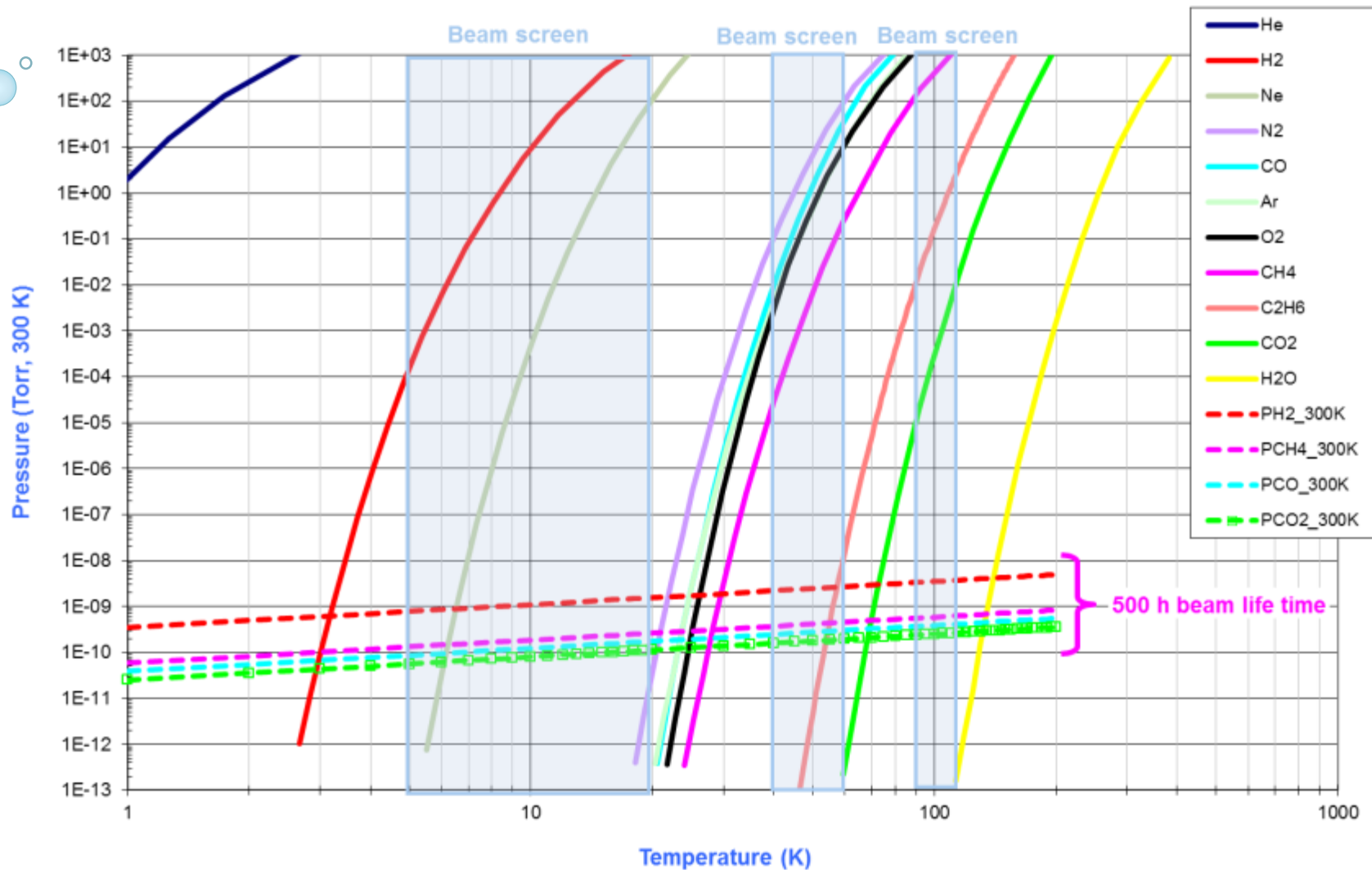


Based on measurements by V.V Anashin *et al.*

Qualification of the proposed temperature window is mandatory

# Beam Screens Operating Temperature

Saturated vapour pressure from Honig and Hook (1960) (C2H6 Thibault *et al.*)

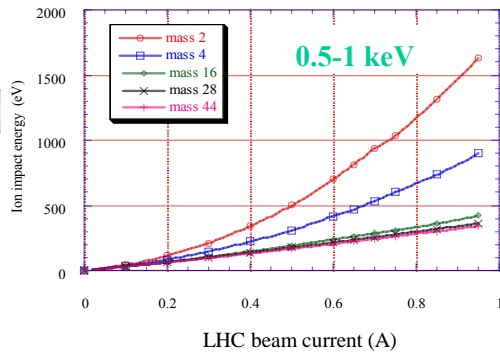


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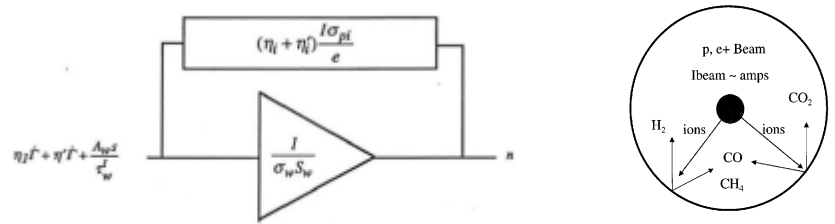
Vincent Baglin talk @ fcc meeting in Washington

# Vacuum Instability

- ions, produced by **beam ionisation**, desorb molecules which are subsequently ionised
- Ion impact energy in the keV range



O. Gröbner, CERN 99-05

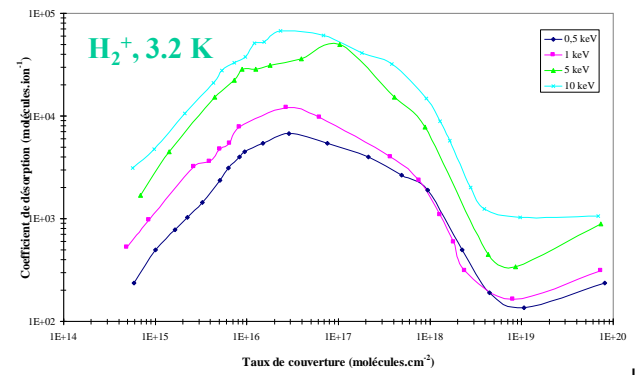


W.C. Turner. J. Vac. Sci. Technol. A. 14(4), Jul/Aug 1996  
O. Grobner, R. Calder, IEE Trans. Nucl. Sci. NS-20, 760 (1976)

- Simple beam tube without beam screen

$$I_{\text{crit, tube}} = \frac{\sigma_w S_w}{(\eta_{\text{ion}} + \eta'_{\text{ion}}) \frac{\sigma}{e}}$$

$\eta'_{\text{H}_2} \sim 1000$   
 $\eta'_{\text{CO}_2} \sim 2$   
@ 1 keV and 1 monolayer

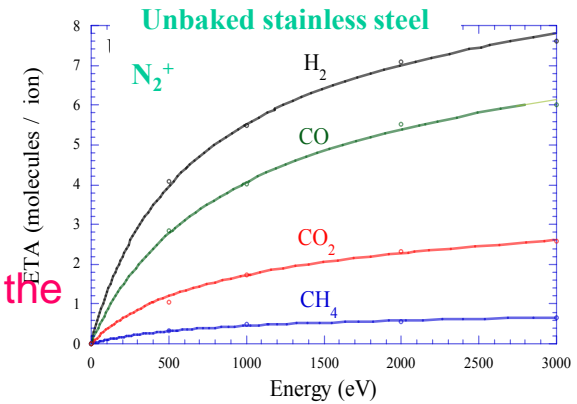


(N. Hilleret, R. Calder, IVC, 1977)

- With a perforated beam screen, C

$$I_{\text{crit, BS}} = \frac{1}{\eta_{\text{ion}}} \frac{C}{\sigma/e}$$

$\eta_{\text{H}_2} \sim 5$   
 $\eta_{\text{CO}_2} \sim 1$   
@ 1 keV and 1 monolayer



A.G. Mathewson, CERN ISR-VA/76-5

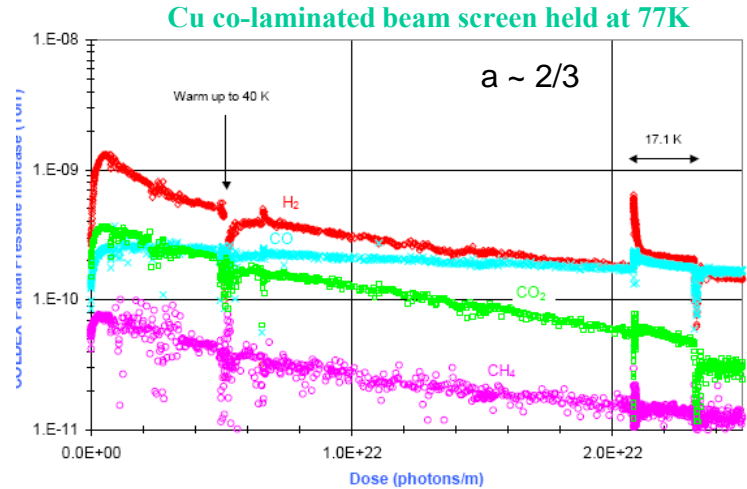
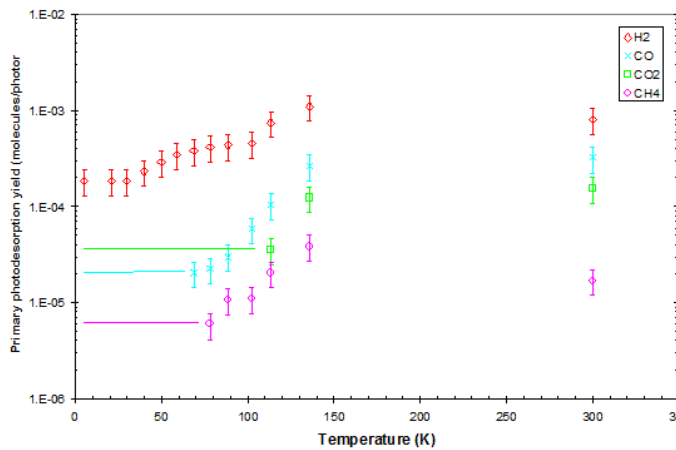
- In **both** cases, when the beam current **approach the critical current**, the pressure increases to infinity

Vincent Baglin talk @ fcc meeting in Washington

# Photodesorption at Cryogenic Temperature

- Initial yield,  $\eta_0$ , and conditioning rate,  $a$ , are smaller than at room temperature

V. Baglin et al., Vacuum 67 (2002) 421-428

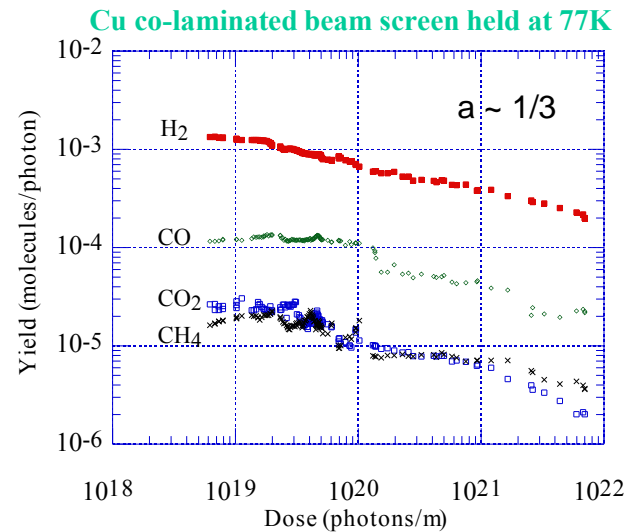


$$\eta(D) = \eta_0 \left( \frac{D}{D_0} \right)^{-a}$$

Table 1 : Primary, recycling photodesorption yield measured at  $10^{22}$  photons/m and cleaning rate

	H <sub>2</sub>	CH <sub>4</sub>	CO	CO <sub>2</sub>
$\eta$	$2 \cdot 10^{-4}$	$> 6 \cdot 10^{-6}$	$> 3 \cdot 10^{-5}$	$> 2 \cdot 10^{-5}$
$(\eta + \eta') / \sigma$	$2 \cdot 10^{-2}$	$6 \cdot 10^{-4}$	$3 \cdot 10^{-3}$	$2 \cdot 10^{-3}$
$a$	0.6	0.6	0.2	0.8

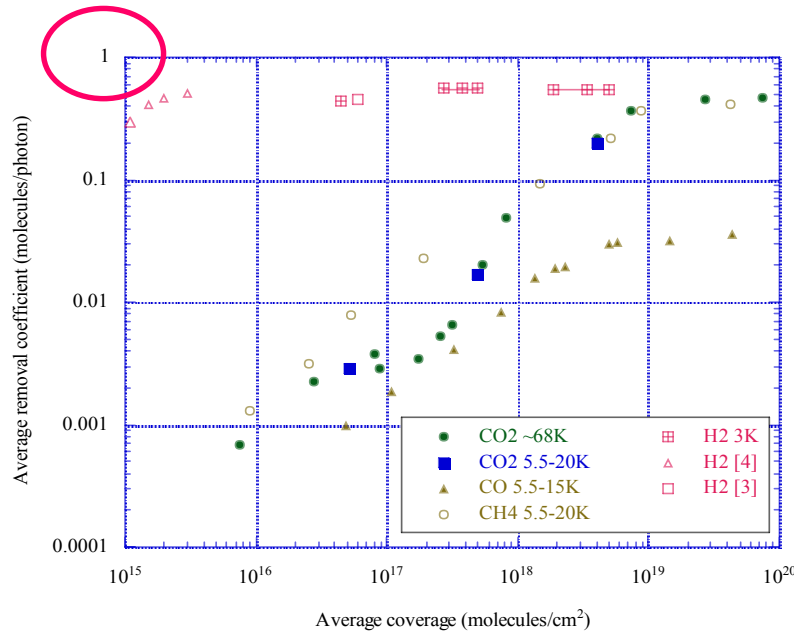
Cu co-laminated beam screen held at 7K



V. Baglin et al. EPAC 2002, Paris, France.

# Photodesorption of Physisorbed Gases

- Desorption of physisorbed molecules:  
Large recycling yields  $\eta'$



Stainless steel, 250-300 eV. Perpendicular incidence

V. Anashin *et al.*, Vacuum 53 (1-2), 269, (1999)

- Photo-cracking of molecules:  
CH<sub>4</sub> into H<sub>2</sub>  
CO<sub>2</sub> into CO and O<sub>2</sub>

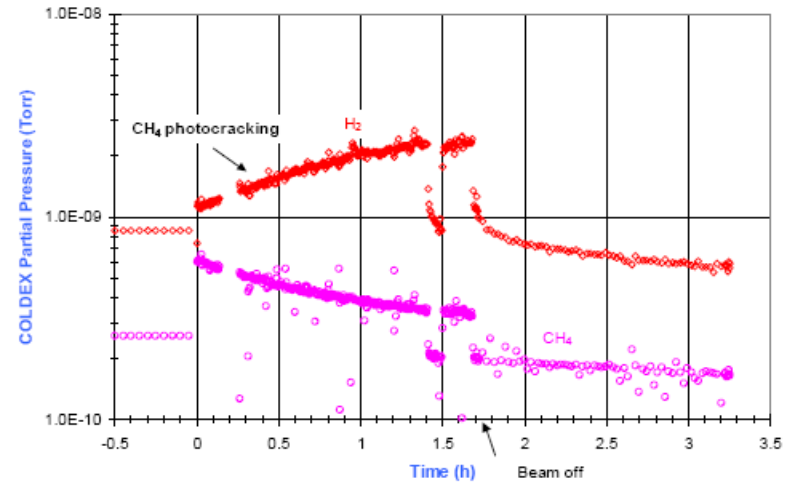
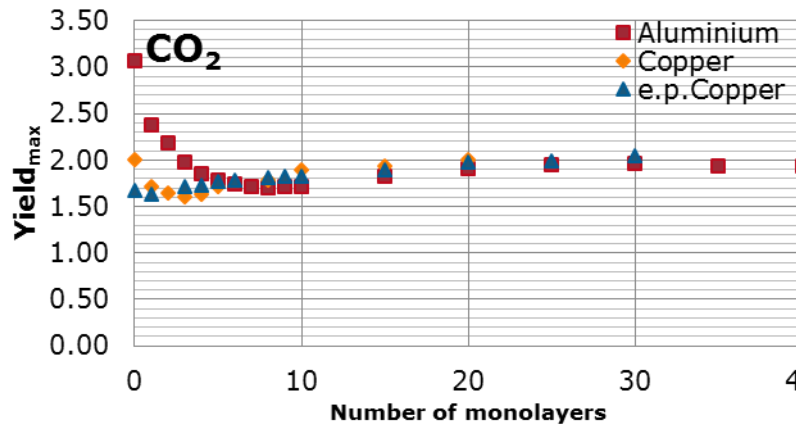


Figure 5 : ~ 10 monolayers of CH<sub>4</sub> condensed onto a BS without hole prior to irradiation at 6 K

V. Baglin *et al.* EPAC 2002, Paris, France.

# Electron Interaction with Physisorbed Gases

- Electron stimulated molecular gas desorption **increase** with surface coverage: 0.5 CO<sub>2</sub>/e at one monolayer
- Condensed gas have **large** secondary electron yields ( $\Delta_{max\_CO_2} > 1.6$ )



A. Kuzucan *et al.* J.Vac.Sci. A. 30, 051401 (2012)

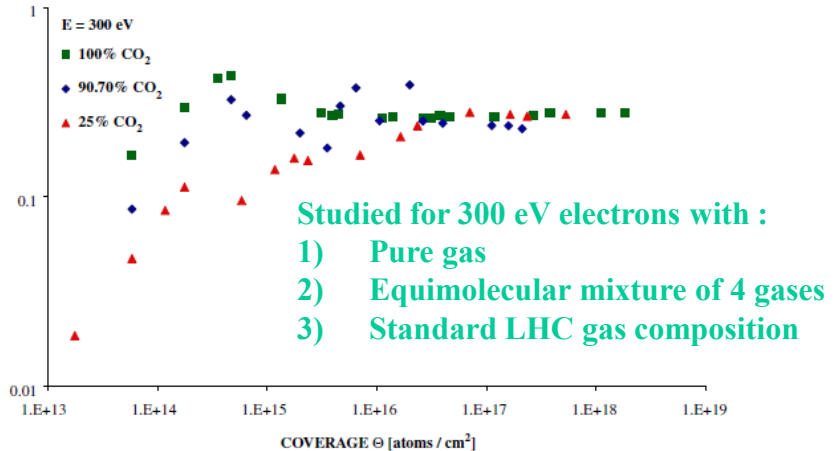


Fig. 8. The CO<sub>2</sub> desorption yield as a function of CO<sub>2</sub> coverage for different condensed gas composition (electron energy 300 eV).

H. Tratnik *et al.*, Vacuum 81, 731,(2007)

Inputs parameters need to be known and optimised against the design

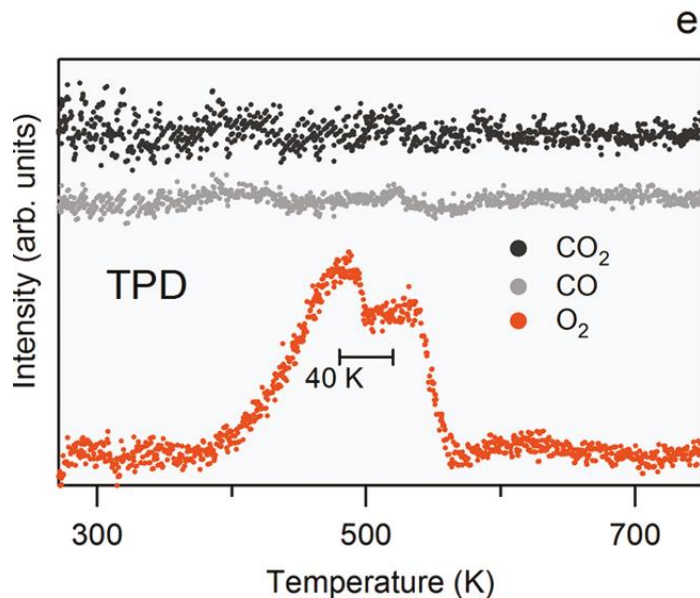
➔ The beam screen surface must remain as “clean” as possible

# Study vacuum stability at cryogenic temperature

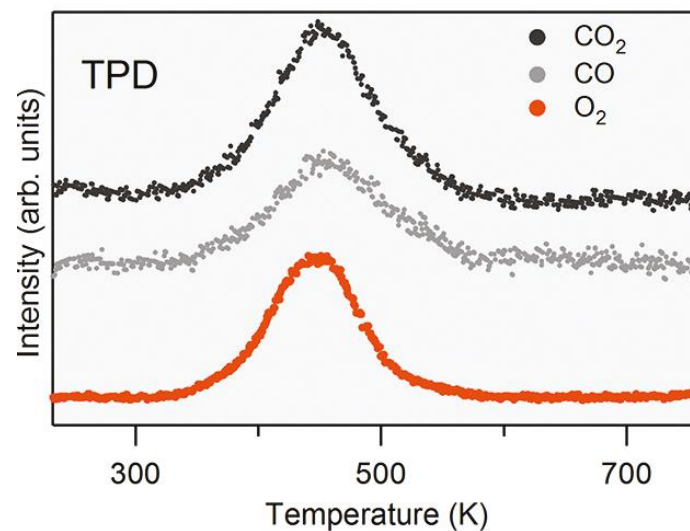
- Still a number of interesting open questions
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# Study @ LNF on small samples: TPD.

- The TPD (Temperature Programmed Desorption) can be recorded with a quadrupole mass spectrometer with a “Feulner cup” with a sample-size opening.



O<sub>2</sub>  $\theta$  = 0.03 ML on Graphene /Ir



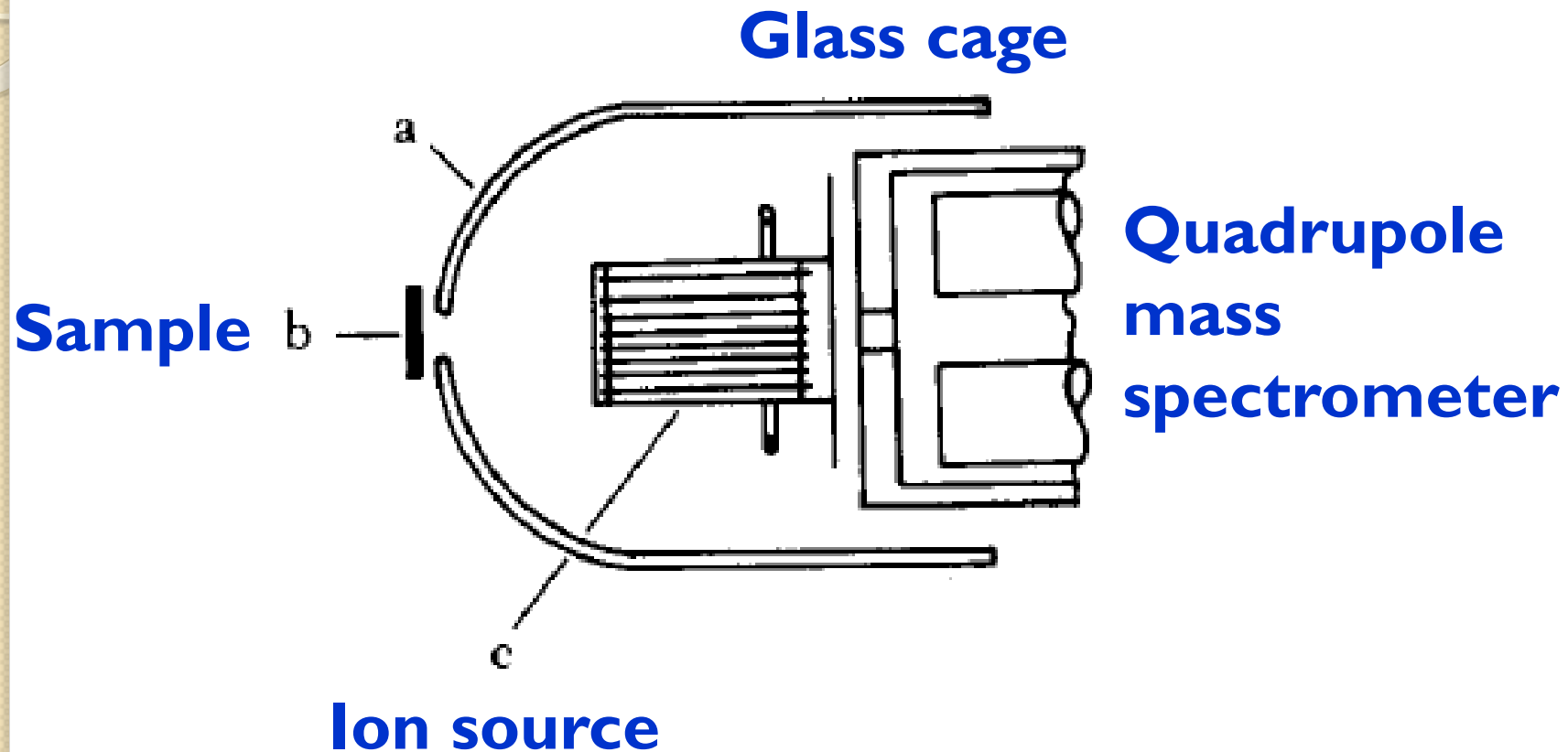
O<sub>2</sub>  $\theta$  = 0.25 ML on Graphene /Ir

Dual Path Mechanism in the Thermal Reduction of Graphene Oxide Rosanna Larciprete, Stefano Fabris, Tao Sun, Paolo Lacovig, Alessandro Baraldi, and Silvano Lizzit Journal of the American Chemical Society 2011 133 (43), 17315-17321



# Study @ LNF on small samples: TPD.

- a “Feulner cup”

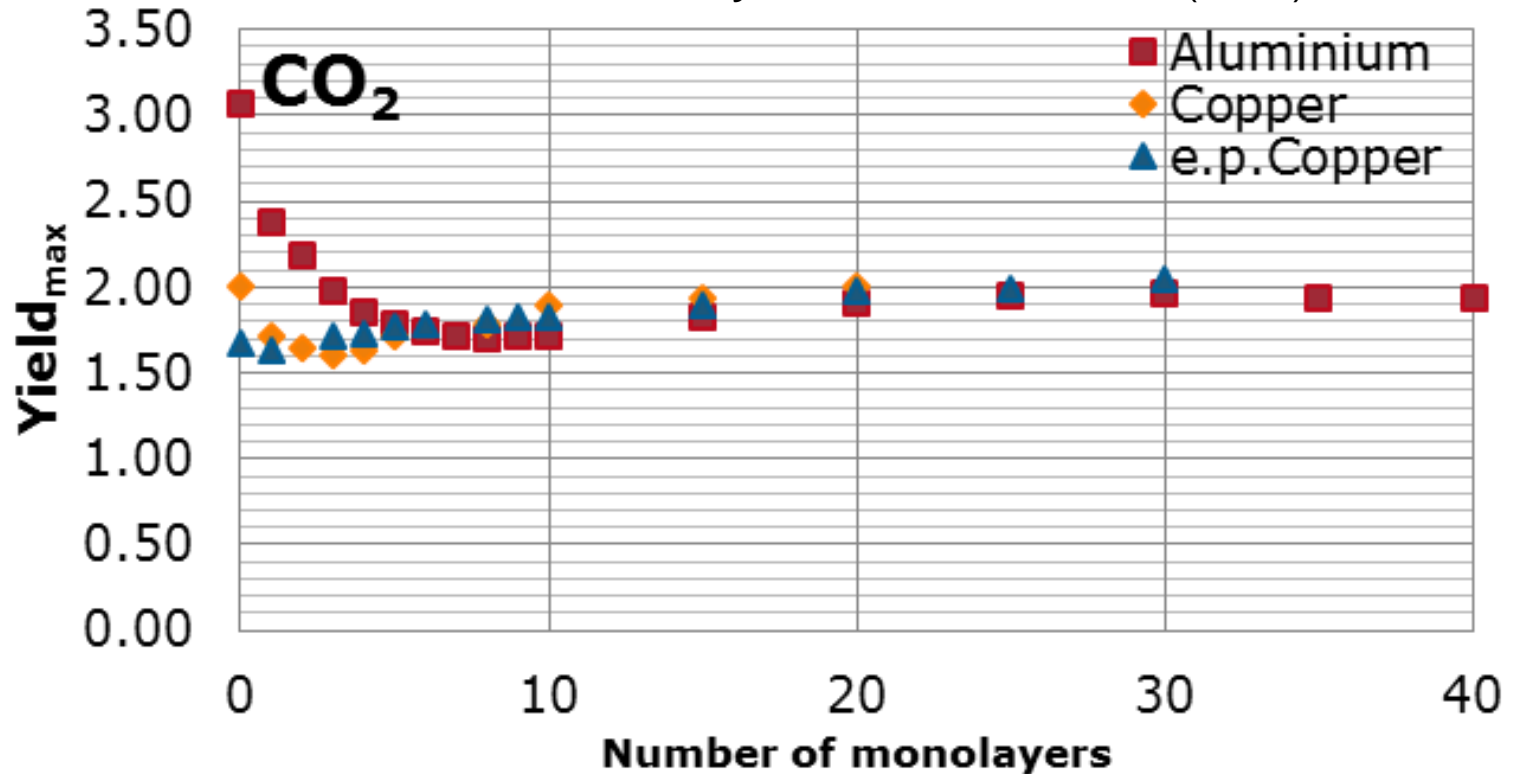


Feulner, P.; Menzel, D. J. Vac. Sci. Technol. 1980, 17, 662–663.

# Study @ LNF Vacuum desorption with SEY.

- Assume (very low current) SEY does not perturb the physisorbed gas:

A. Kuzucan *et al.* J.Vac.Sci.A. 30, 051401 (2012)



## SEY can monitor Vacuum desorption

# Study @ LNF: Vacuum desorption with SEY and TPS.

- At high Electron current, the electron beam can induce gas desorption: SEY and TPD to contemporarily study physisorbed gas.
- Ions?
- Photons?
- Benchmarking results with “large surface” CERN approach

Different samples, surface coatings and roughness as produced by CERN and the collaboration.

# Study vacuum stability at cryogenic temperature

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# As all experimental studies it needs:

- Instrumentation**
- maintenance**
- upgrades.**

- LNF-INFN is ready to:
- Make partially available the existing laboratory for FCC-hh experiments
- Request further grants for maintenance and upgrade (for 2016-2019)

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# The available laboratory: (~3M€)

- Our laboratory is devoted to the growth and study of chemical, structural and electronic properties of thin and ultrathin
- **Two ultra-high vacuum systems** equipped with several growth facilities (Chemical Vapour Deposition, physical evaporation, magnetron sputtering) and with **in-situ, UHV, electronic diagnostics (Low Energy Electron Diffraction, X-ray and UV Photoelectron Spectroscopy, Secondary Electron Yield and Raman Spectroscopy)** and scanning probe microscopy (**variable temperature Scanning Tunneling Microscopy also recently implemented for UHV use**).
- **One of the systems is equipped with a low temperature (~ 7 K) manipulator.**

# further grants for maintenance and upgrade

- We will apply (July 2015) and know (October 2015) at SCV of INFN to (among other WPs) :
- Maintain the existing laboratory
- Upgrade the system with a second LT manipulator (to be located on the XPS chamber)
- Dote the system with a “Foulner” cup and an optimized (highest sensitivity) Mass spectrometer for TPD studies.
- Support collaboration with CERN and EuroCircol.

**The project will include other Italian partners and other research topics and WPs (as Hi-Lumi LHC) to strengthen the request.**

- **Official Co-funding are very important ACEs to play!**

# FYI: in agreement with CERN – FCC-hh

ERC Advanced Grant 2015 by R. Cimino

(Submitted the 2-6-15).

## GECO

### Green Circular Colliders

GECO aims to bring to maturity the possibility offered by a **highly X ray reflecting beam screen**, to control and dissipate the SR induced heat load in the warm part of any future hadron accelerator at the energy frontiers. This will result in a much more cost effective accelerator. The proposed solution has to be solid, stable and validated to be compliant to all the functionalities required to a BS for optimum machine performance.



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ERC Advanced Grant 2015 by R. Cimino

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## GECO

### Green Circular Colliders

GECO aims to bring to market a new type of collider offered by a **highly** **x** to control **en**, **in**, **e**, **st**, **nas** to be **compliant** to all the **to a BS** for optimum machine

If rejected; (very low acceptance rate!) FCC-hh management should decide what to do with it!  
It is not within EuroCirCol WP objectives, deliverables and reach!

# Last but not Least: RESOURCES for WP4

@ LNF:	(Month)
Roberto Cimino	24
Antonio Grilli	4.8
M. Pietropaoli	4.8
A. Raco	4.8
Vittorio Sciarra	4.8
Vinicio Tullio	4.8
Giacomo Viviani	4.8
Tot.:	52.8
Estimated value:	256k€

From EuroCirCol:	
Total	<b>208 k€</b>
Overhead* (<25%)	- 52 k€
Travel* (<15%)	- 30 k€
	<b>=126 k€</b>

\* Under negotiation and evaluation

**At the lowest Italian rates  
(if candidates are available)  
4 y < Post DOC < 5y (\*)**

**We plan to call for: (PhD are extremely rare!)  
1 Post DOC for ~2/3 y asap (September?)  
1 Post DOC for ~1/2 y (Nov/Dic? 15)**

# Tentative Plan:

- ✓ **Post DOC's will start asap (following the Italian recruitment Procedures: Open call)**
- ✓ **October 2015: the economic resources from INFN to the project will be known. (and become available from 2016).**
- ✓ **Then, either just maintenance or upgrade will start.**
- ✓ **Now: identify and prepare samples to be studied (with CERN)**
- ✓ **Early 2016: first results with existing set up on available samples.**
- ✓ **Late 2016: also first results with new set ups (if available).**

# Conclusion

- ✓ **Being a significant experimental campaign EuroCirCol just boost its start!!!**
- ✓ **A collaborative effort will be beneficial to the project and to availability of additional resources (On equal basis funding on similar issues, with documented letter of interest).**
- ✓ **Scientific data will start flowing soon.**