Vacuum stability at cryogenic temperature

WP4 - Activity at LNF

Karlsruhe, 17/10/2018

Luisa Spallino, Marco Angelucci, Rosanna Larciprete and Roberto Cimino

Outline

EuroCircol Meeting in Karlsruhe

- **Summary of the main activities**
  - Dose calibration
  - Temperature calibration
  - Ar TPD measurements: data analysis and results
- **CH_4 and CO TPD measurements**

Vacuum stability at cryogenic temperature

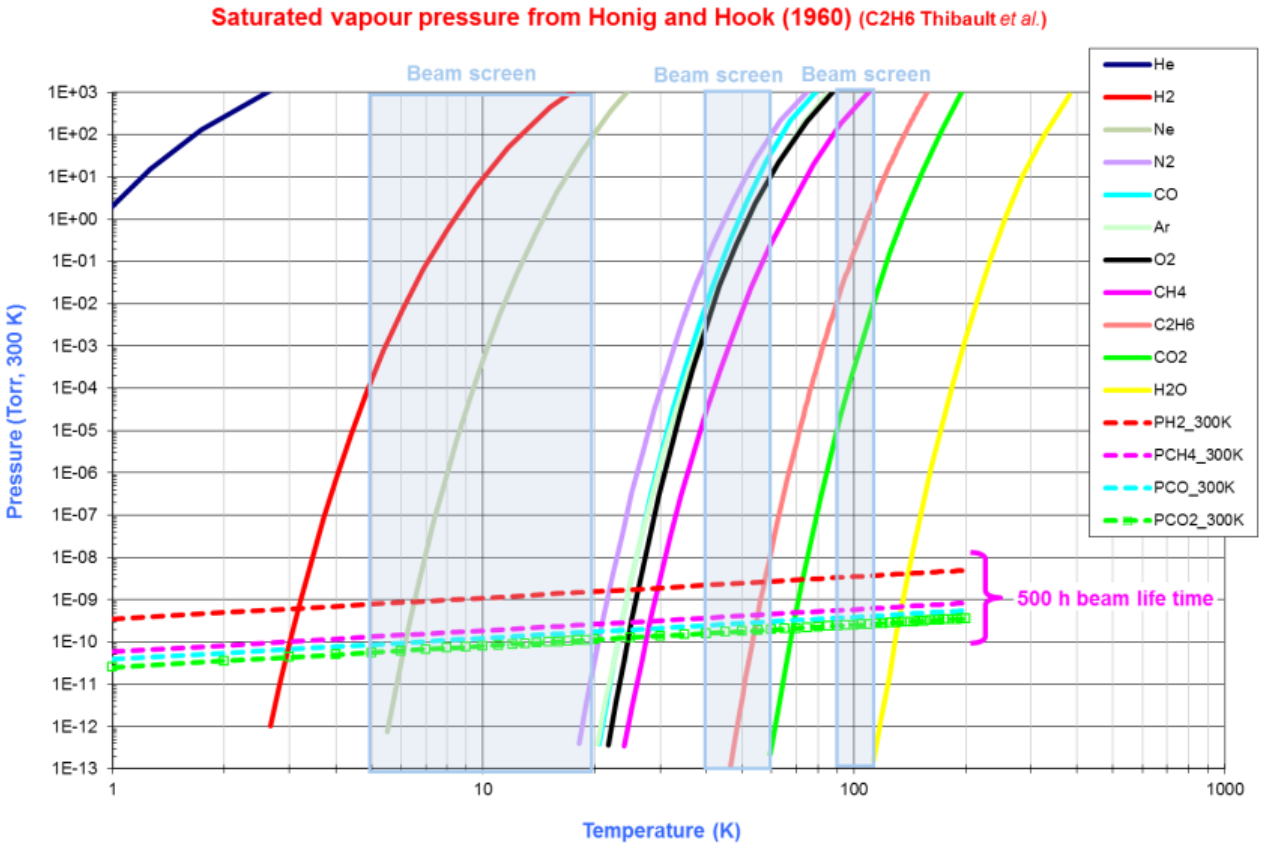
Working Pressure (<10^{-11} mbar) ⟺ **Beam screen Temperature Range**

LHC
Synchrotron Radiation
Power = 0.13 W/m

FCC
Synchrotron Radiation
Power = 40 W/m

Temperature/Pressure Variation

Beam life Time



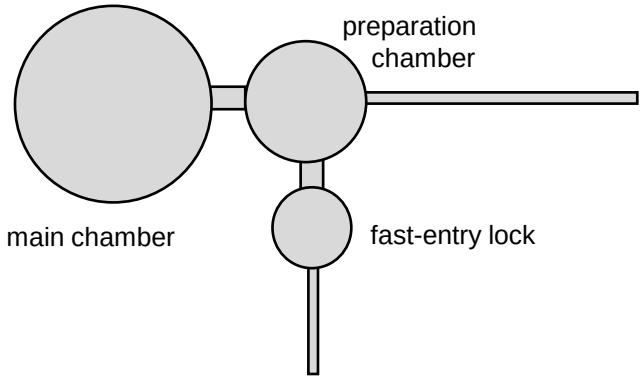
<u>Independently on the substrate treatment</u>, the vacuum stability due to the desorption of residual contaminant gases has to be guaranteed

Our task

Study of adsorption/desorption behaviour of typical contaminant gases in accelerators near their critical temperatures as a function of the surface morphology

Set-up and Strategy at LNF

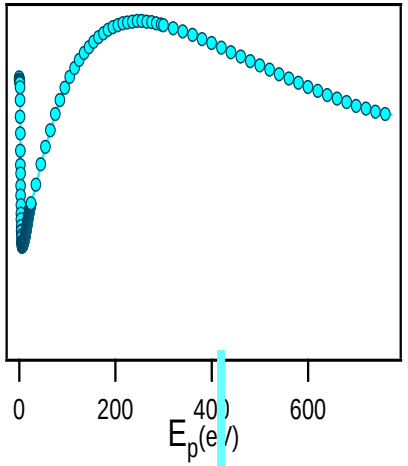
Ultra high vacuum systems

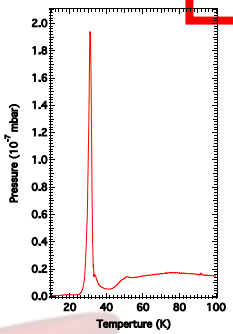


- LNF-cryogenic manipulator
- Sample at 15-300 K

Secondary Electron Yield (**SEY**) measurements

Equipment : Electron gun, Faraday cup



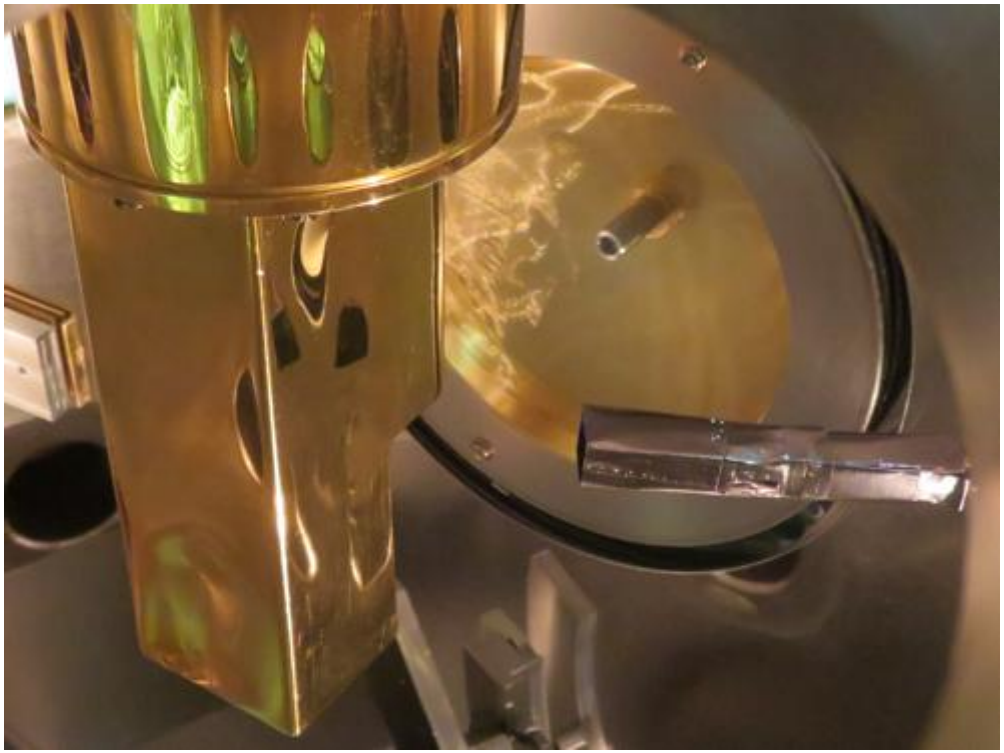


Temperature Programmed Desorption (**TPD**) measurements

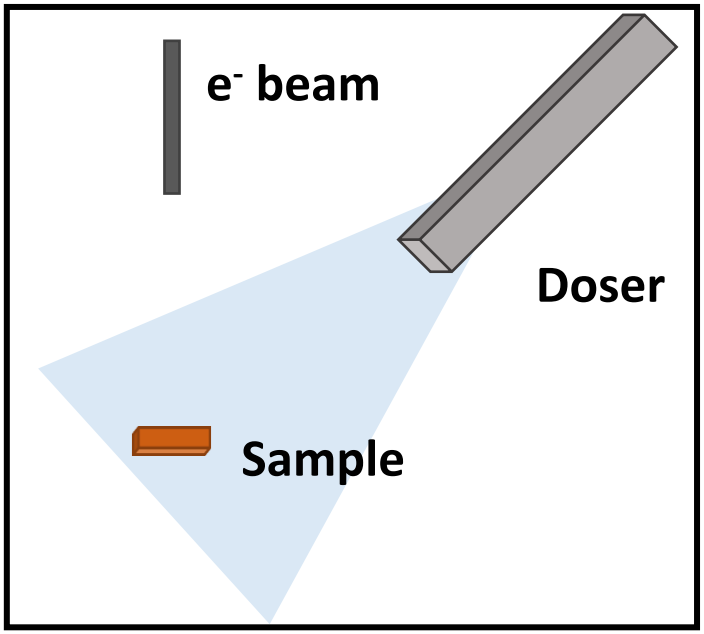
Equipment : QMS (Hiden HAL 101 Pic)

Dose calibration

Gas dosing

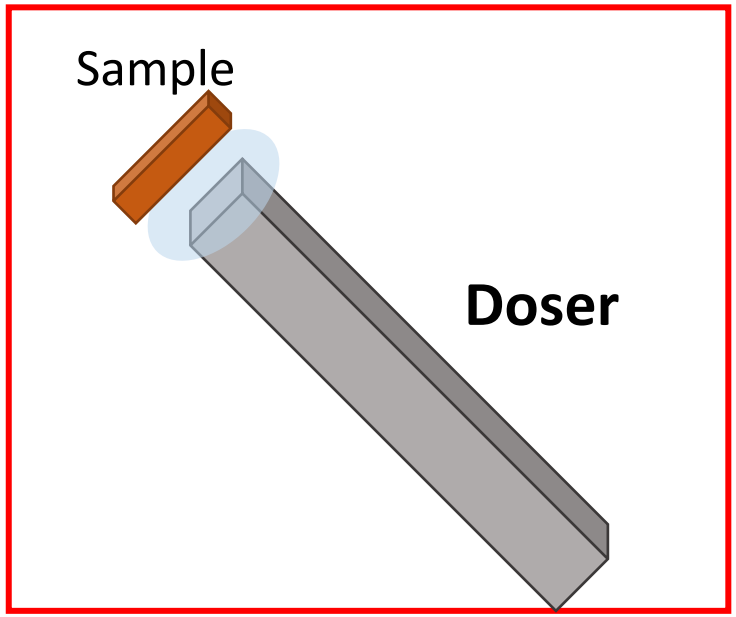


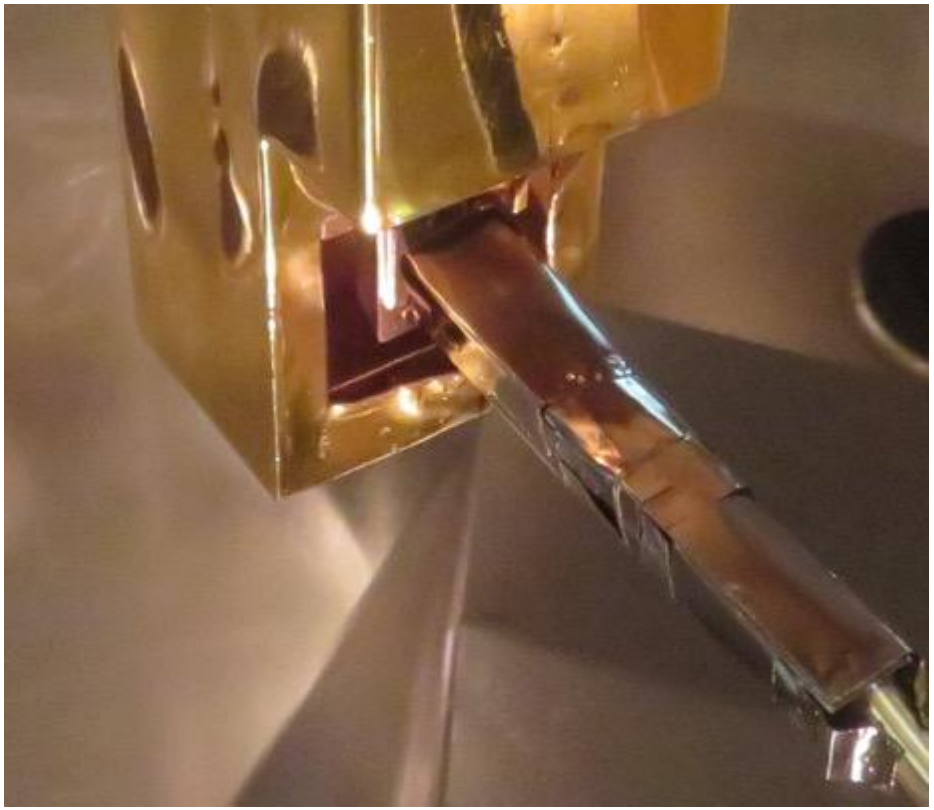
Far from the sample



Dose calibration

Near to the sample



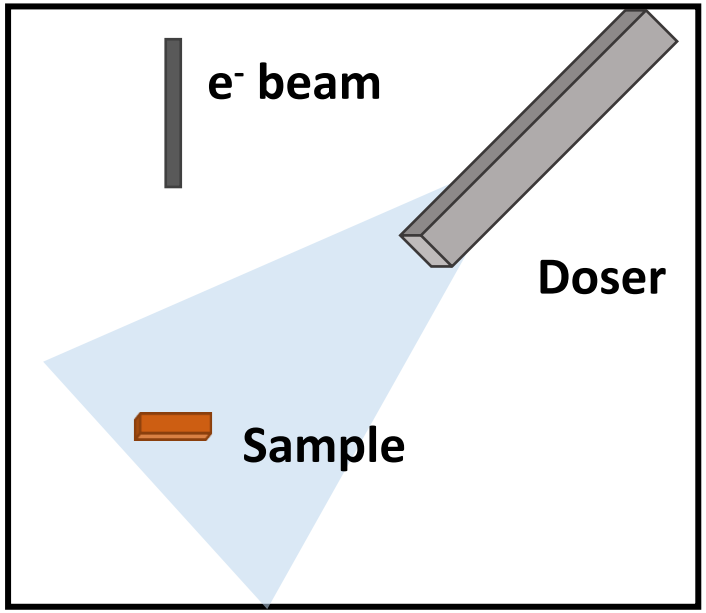


Dose calibration

Different local pressure on the sample

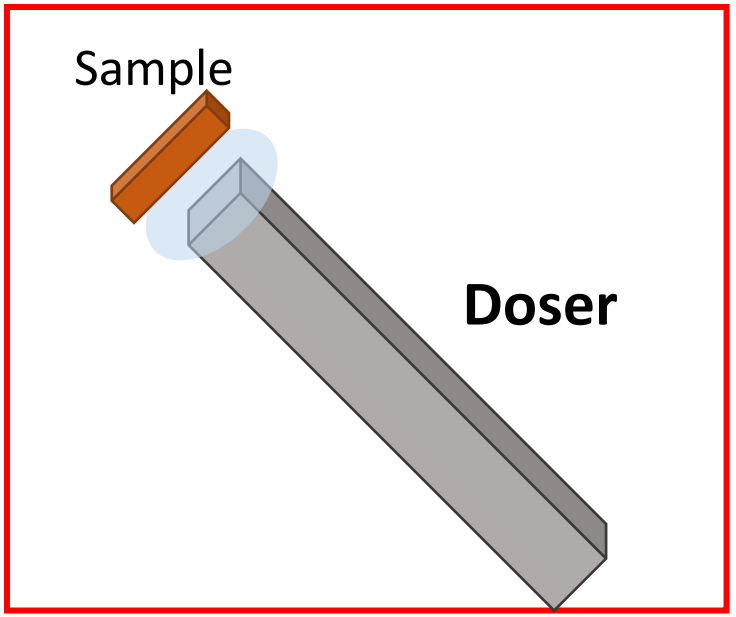
1s@1.33x10^{-6} mbar corresponds to

Far from the sample



1 Langmuir

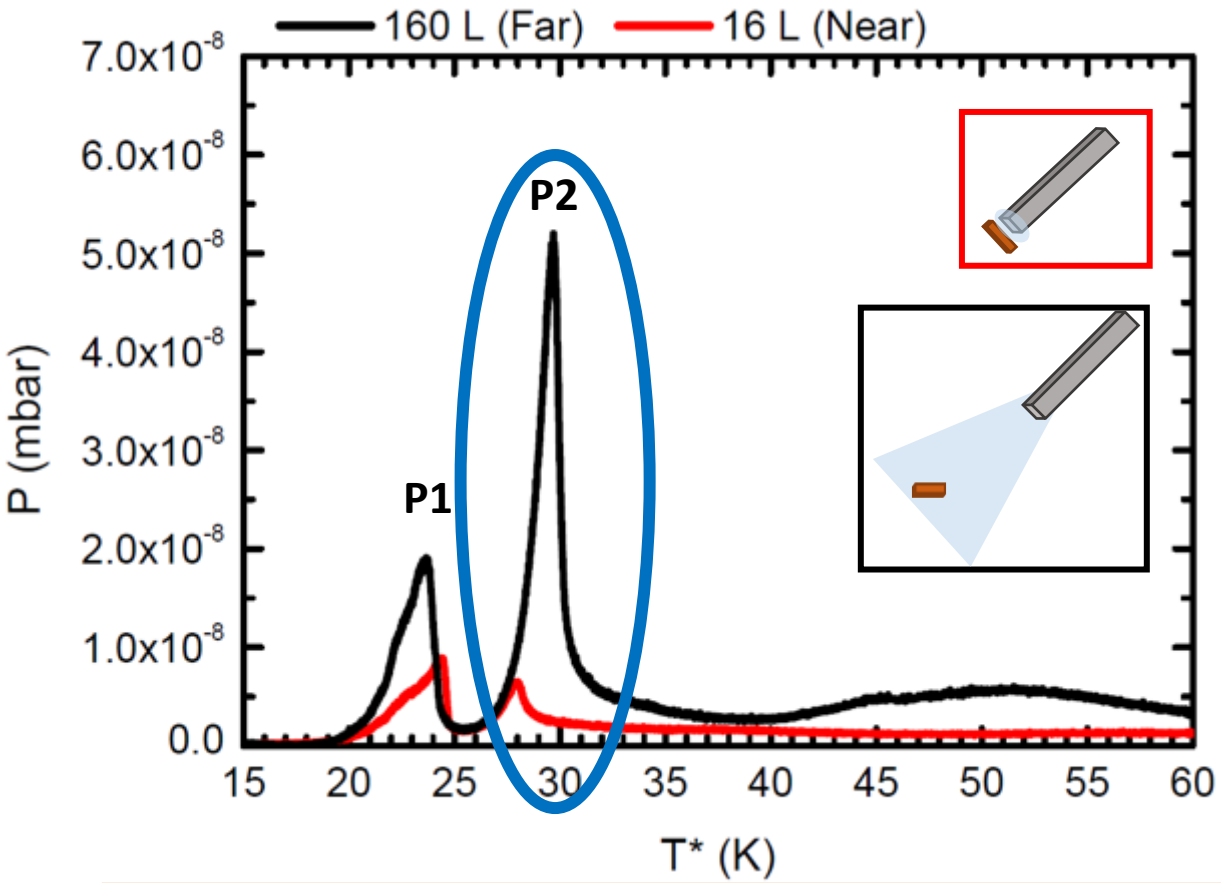
Near to the sample



≈ 5-10 Langmuir

Accurate Calibration in progress

Importance of dosing near the sample

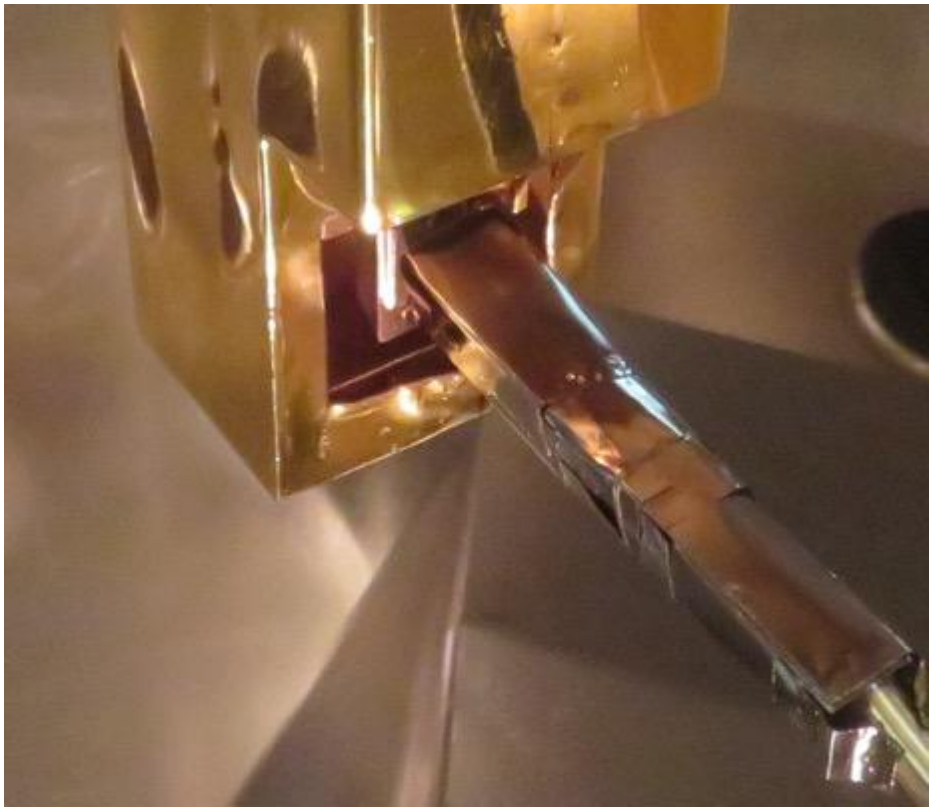


✓Putting the doser near the sample is effective for the reduction of the desorption related to the manipulator

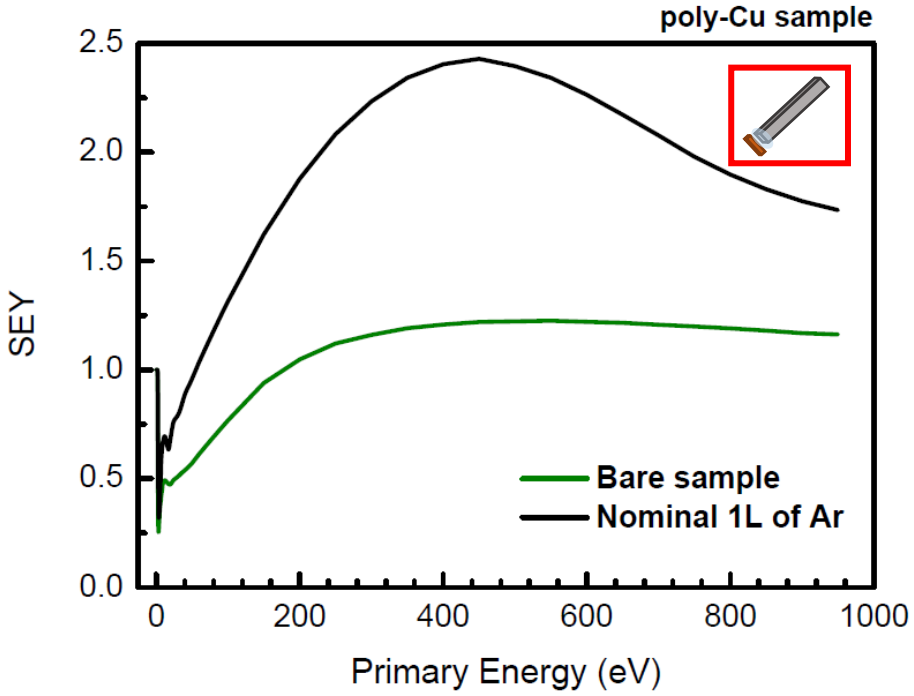
Coverage calibration by SEY

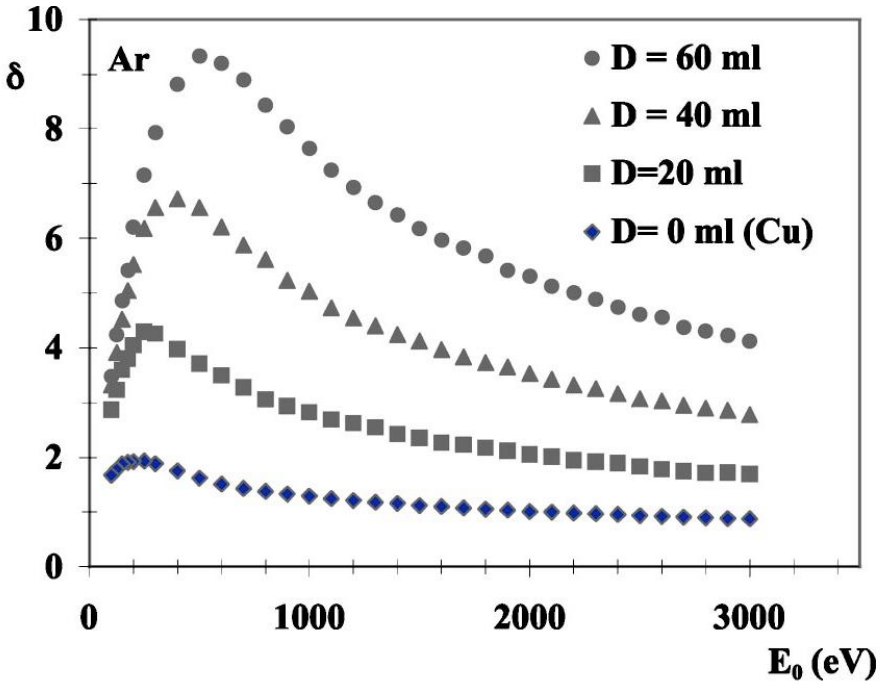
Coverage calibration by SEY

Accurate Calibration in progress



Coverage calibration by SEY

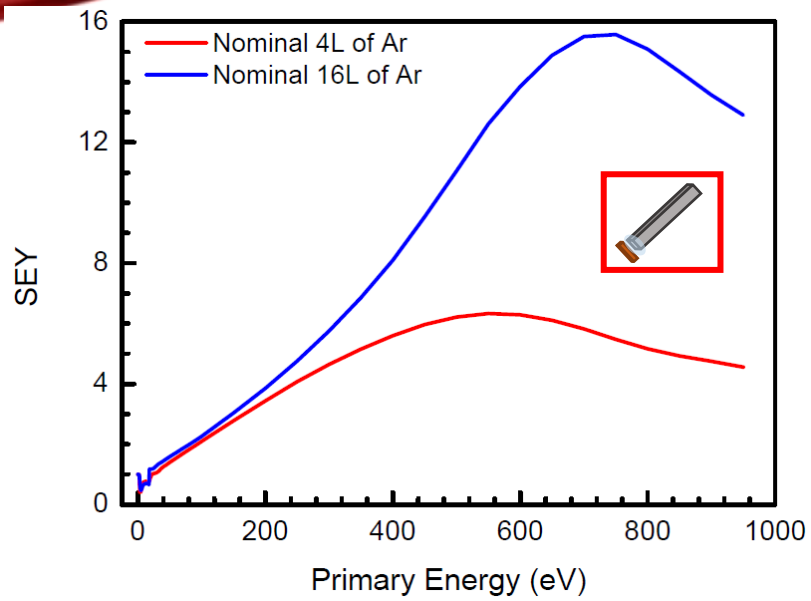




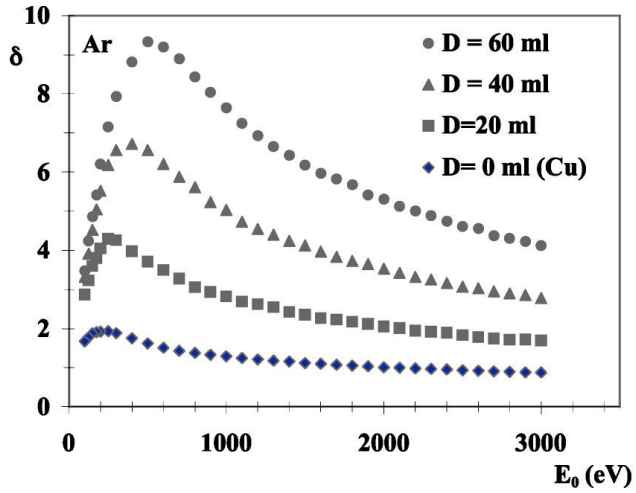
✓Nominal 1L of Ar dosed in the vacuum chamber corresponds to a coverage of ~10L on the sample surface

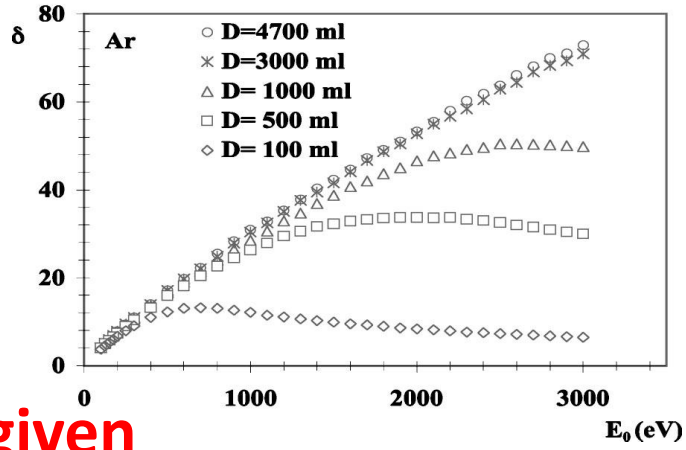
J. Cazaux et al.; Phys. Rev. B, 71 (2005)

Coverage calibration by SEY



J. Cazaux et al.; Phys. Rev. B, 71 (2005)



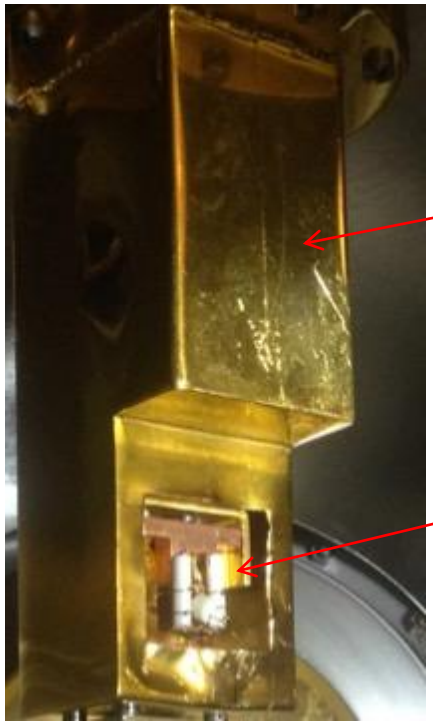


✓Nominal 4L and 16L of Ar dosed in chamber correspond to a coverage of ~25L and ~ 100L on the sample surface

From here on, calibrated coverages are given

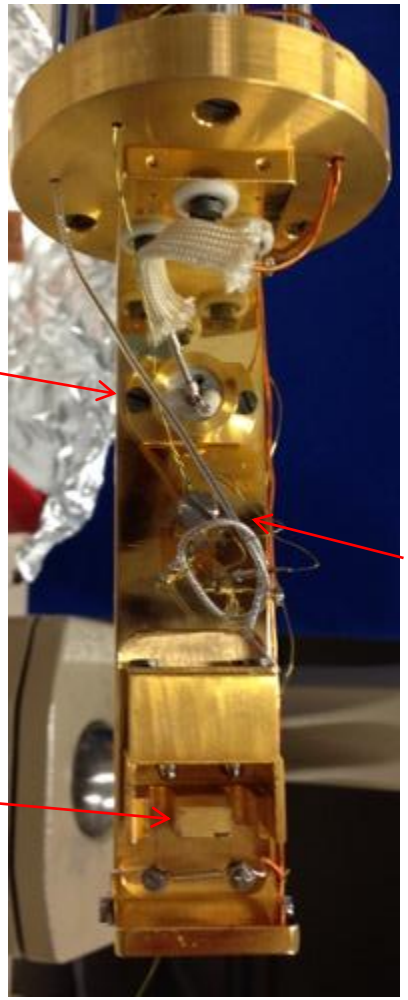
Temperature calibration

LNF-Cryogenic Manipulator

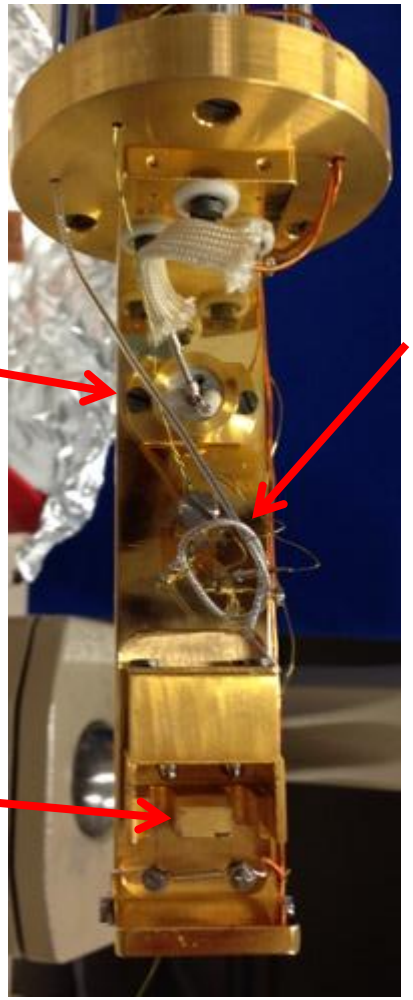


Screen

Sample



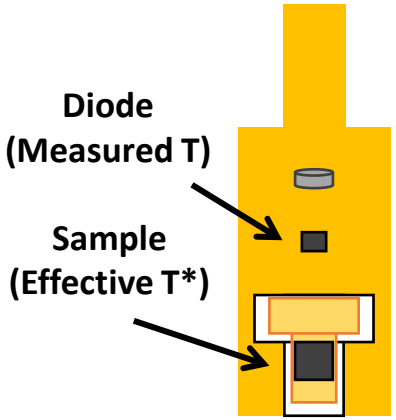
Temperature calibration



Measured Temperature (T*)

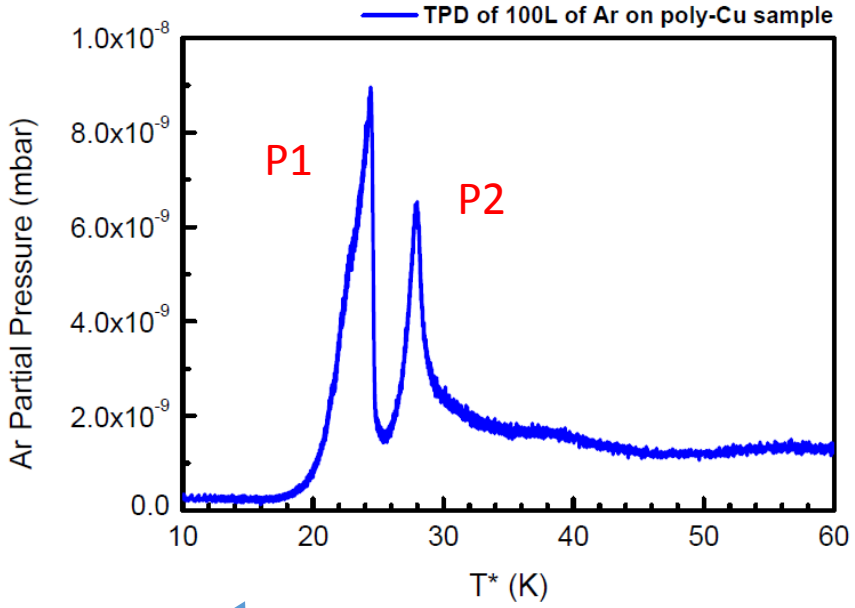
≠

Sample Real Temperature (T)



Temperature calibration

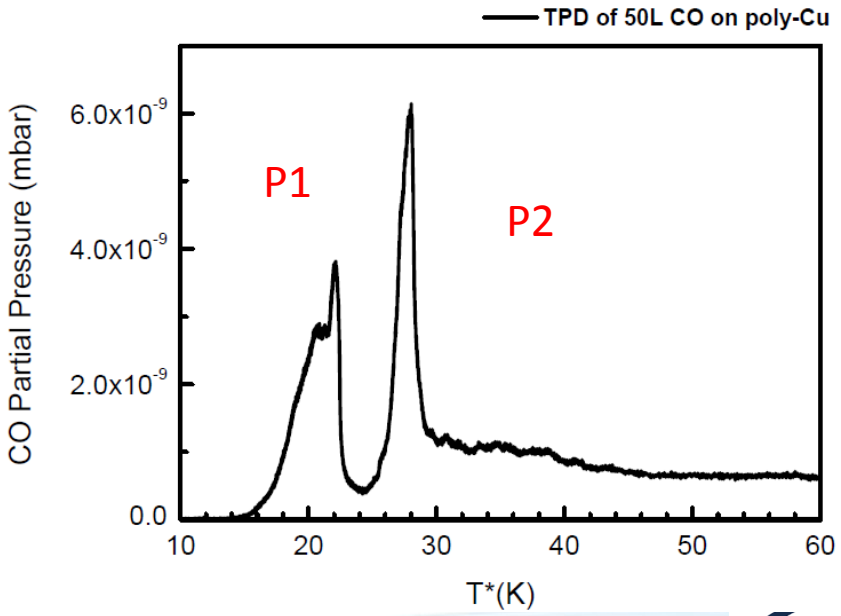
Temperature Programmed Desorption



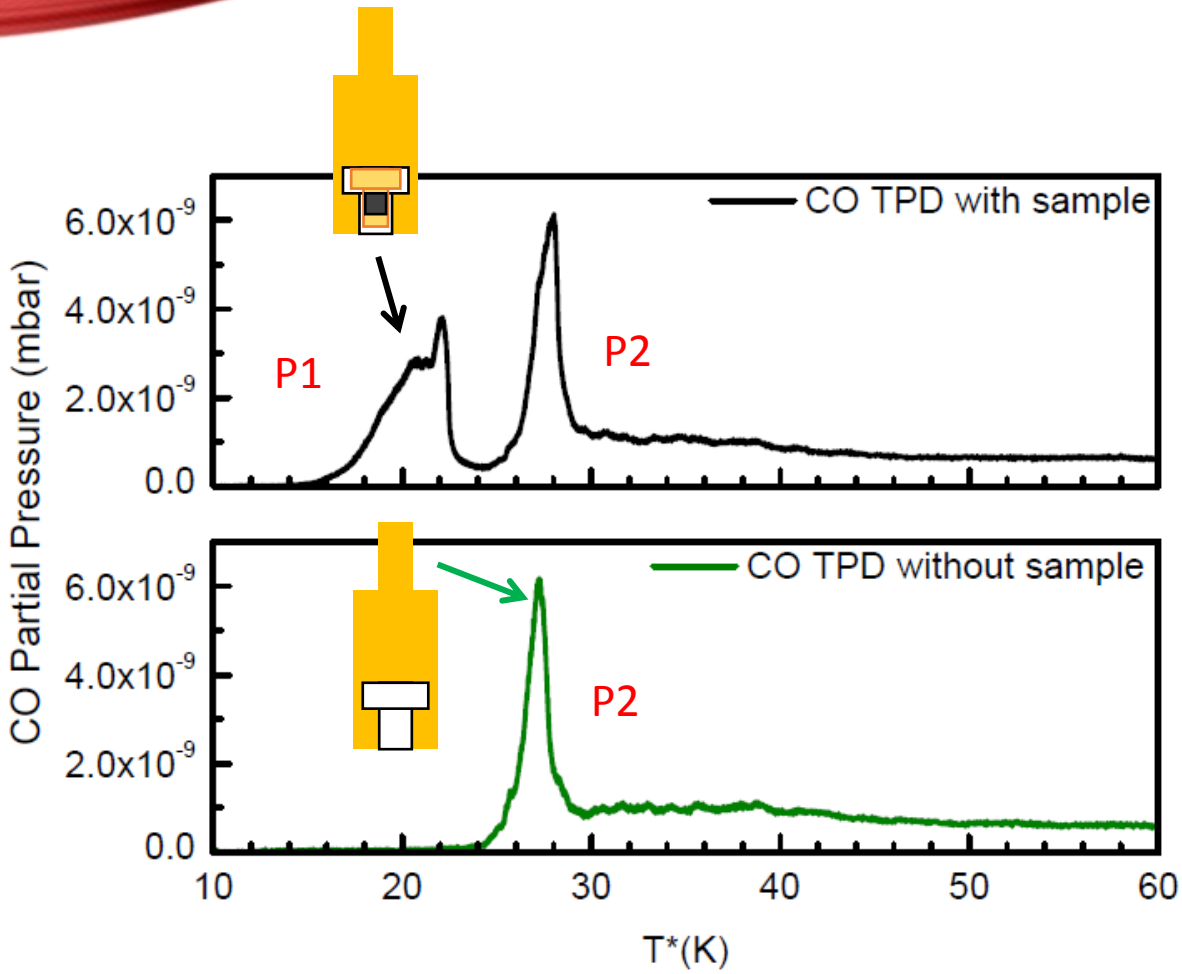
Example of as acquired Ar TPD curve

The **different desorption peaks** are experimental artefact

Example of as acquired CO TPD curve

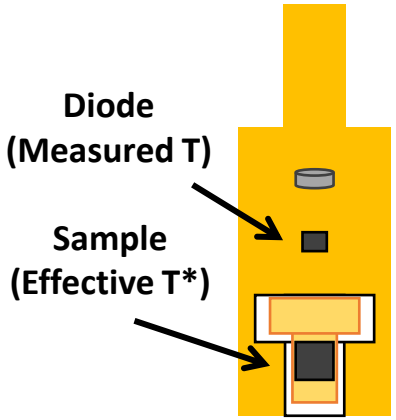


Temperature calibration



Peak 1: Desorption from sample ("hotter part" at T*)

Peak 2: Desorption from Manipulator (at T)



Temperature calibration

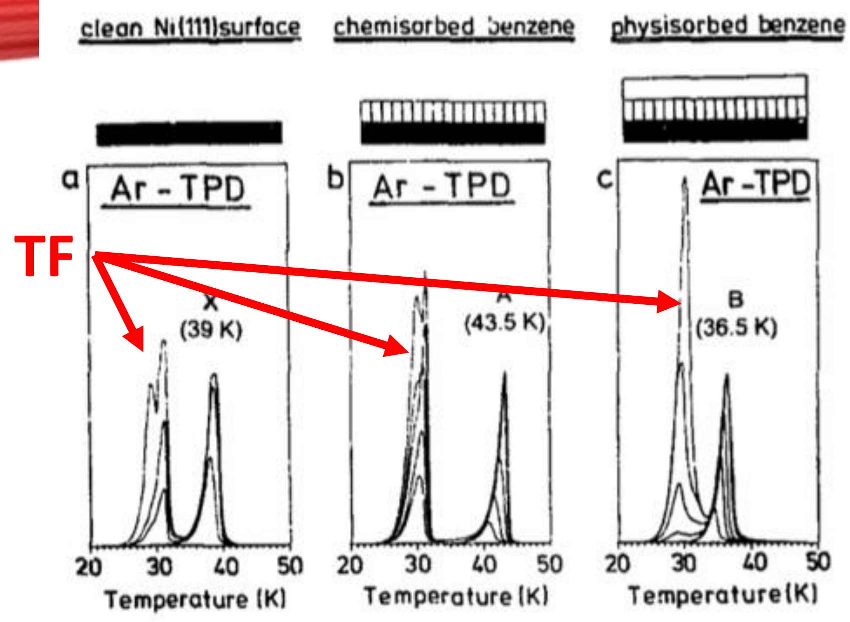


Fig. 1. Argon desorption spectra for increasing argon exposures onto various underlying "substrates": (a) clean Ni(111); (b) saturated chemisorbed ($\sqrt{7} \times \sqrt{7}$)R19.1° benzene layer on Ni(111); (c) saturated first physisorbed benzene layer on top of the chemisorbed layer. Adsorption temperature 22 K; heating rate 1 K/s. The "substrates" are schematically indicated above the corresponding TPD spectra.

M. Stichler et al.; Surface Science 348 (1996)

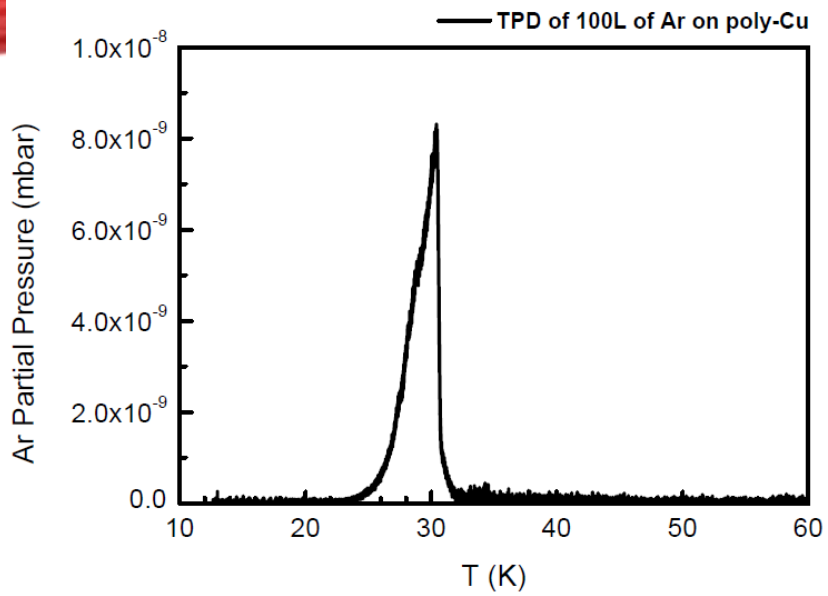
Same Desorption temperature of Argon Thick Film (TF) on different substrates

Ar TF desorbs at a unique T~30 K

From literature, CO and CH_4TF desorb at T~30 K a T~37 K respectively

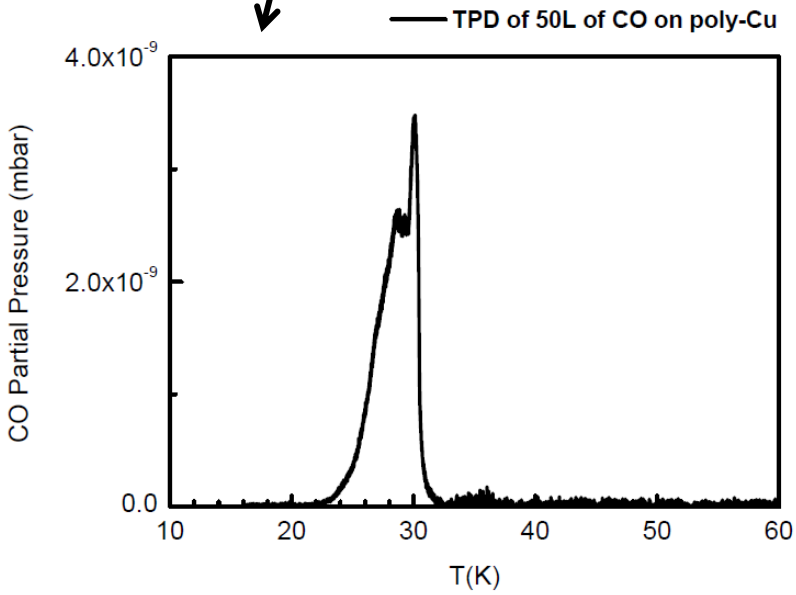
J. A. Noble et al., Mon. Not. R. Astron. Soc., 421 (2012)
R. S. Smith et al., J. Phys. Chem. B, 120 (2016)
T. Suhasaria et al., Mon. Not. R. Astron. Soc., 472 (2017)

Temperature calibration

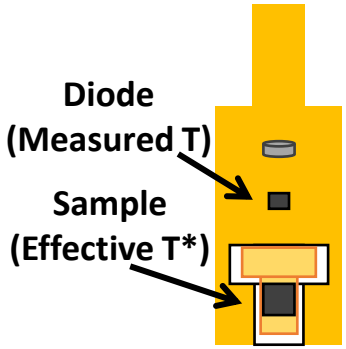


Example of Ar TPD curve after data analysis

Example of CO TPD curve after data analysis

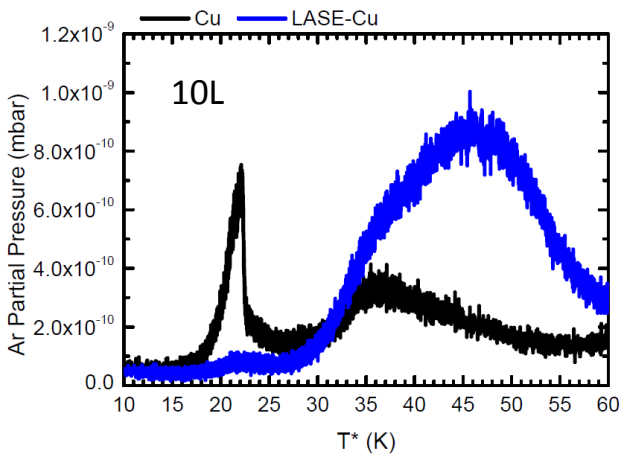


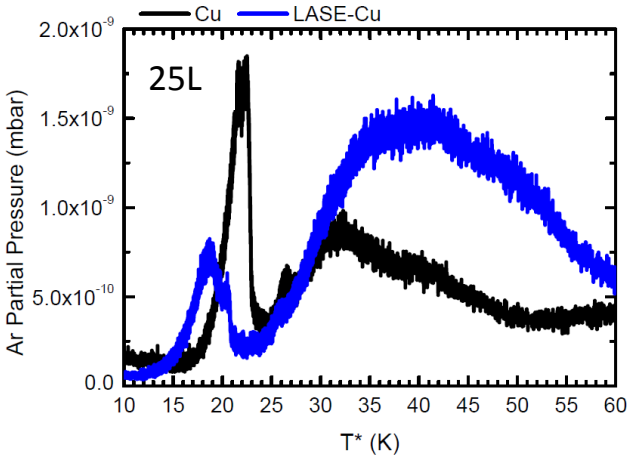
Temperature correction from T* to real T

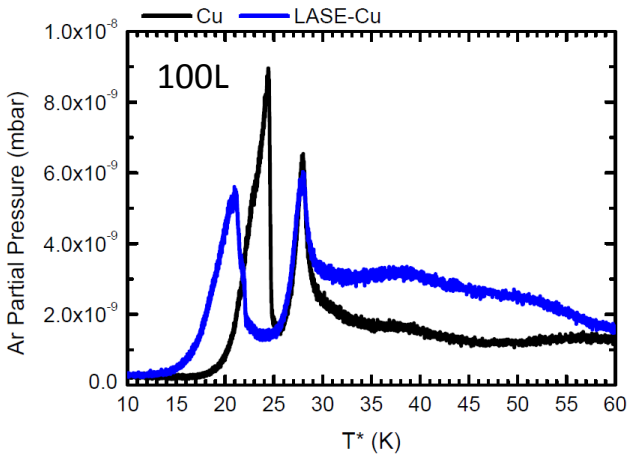


Ar TPD measurements: data analysis and results

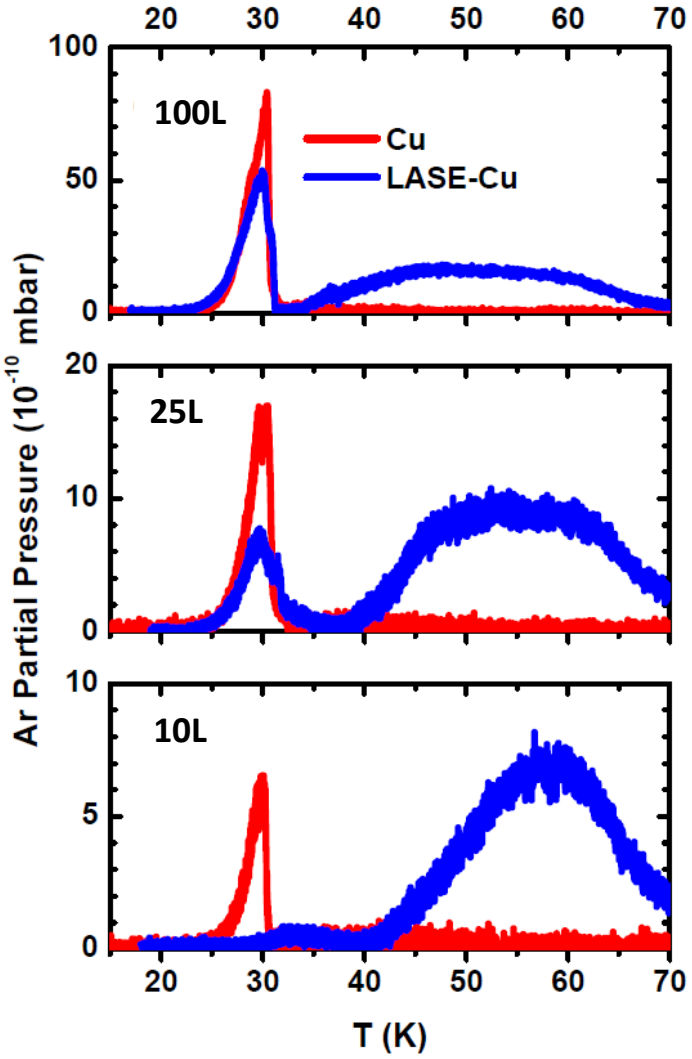
Synopsys of the raw data







Final Ar TPD results

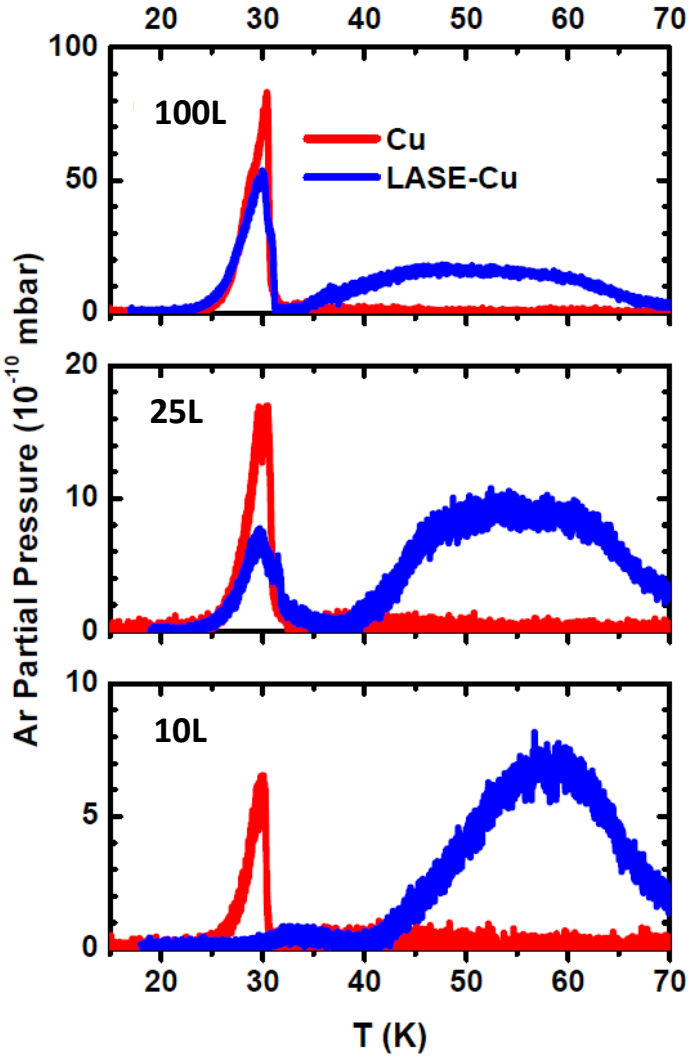


Poster presentation at the FCC Week 2018 and talk presentation at EuroCirCol Meeting in Amsterdam

Oral presentation at the e-Cloud 18 in Isola d'Elba

2 publications in preparation: one regular article and the e-Cloud 18 proceeding

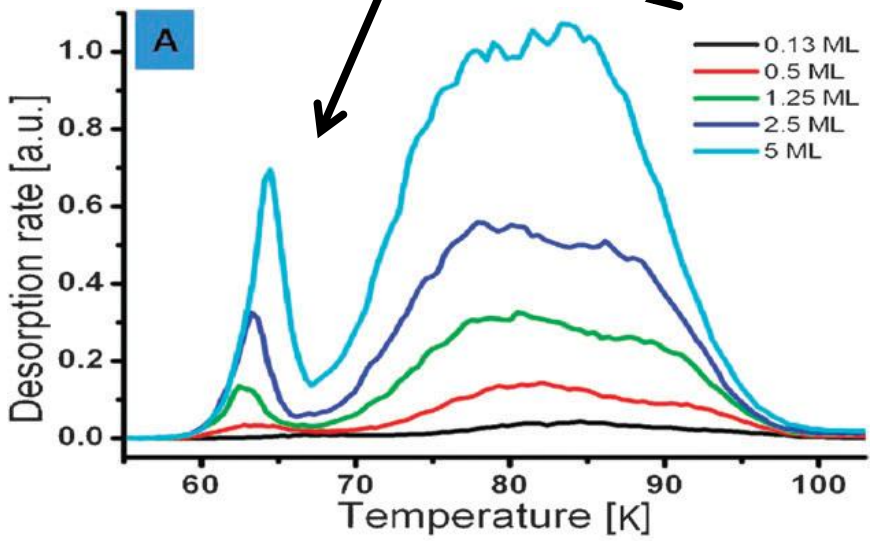
Final Ar TPD results



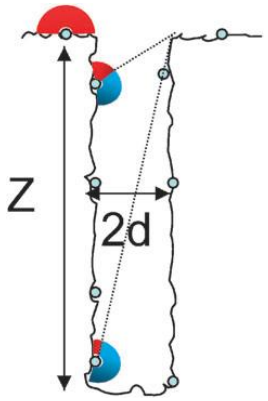
- On flat Cu Ar adsorbs due to the weak Ar-Cu and Ar-Ar Van der Waals interactions and the desorption curve consists of the sharp peak at T~30 K.
- For the LASE-Cu substrate the Ar adsorption energy at the undercoordinated surface defect sites increases and desorption occurs at higher T. However, at high coverage, multilayer desorption at T~30 K is also observed.

From literature

Desorption from the top surface and from the topmost part of the pore



Desorption from pore depth



High temperature TPD peak

Due to the wide distribution of the binding energy of the adsorption sites and multiple desorption–readsorption cycles on the inner pore walls

PAPER www.rsc.org/pccp | Physical Chemistry Chemical Physics

Xe interacting with porous silicon

Assaf Paldor,[a] Gil Toker,[a] Yigal Lilach[b] and Micha Asscher*[a]

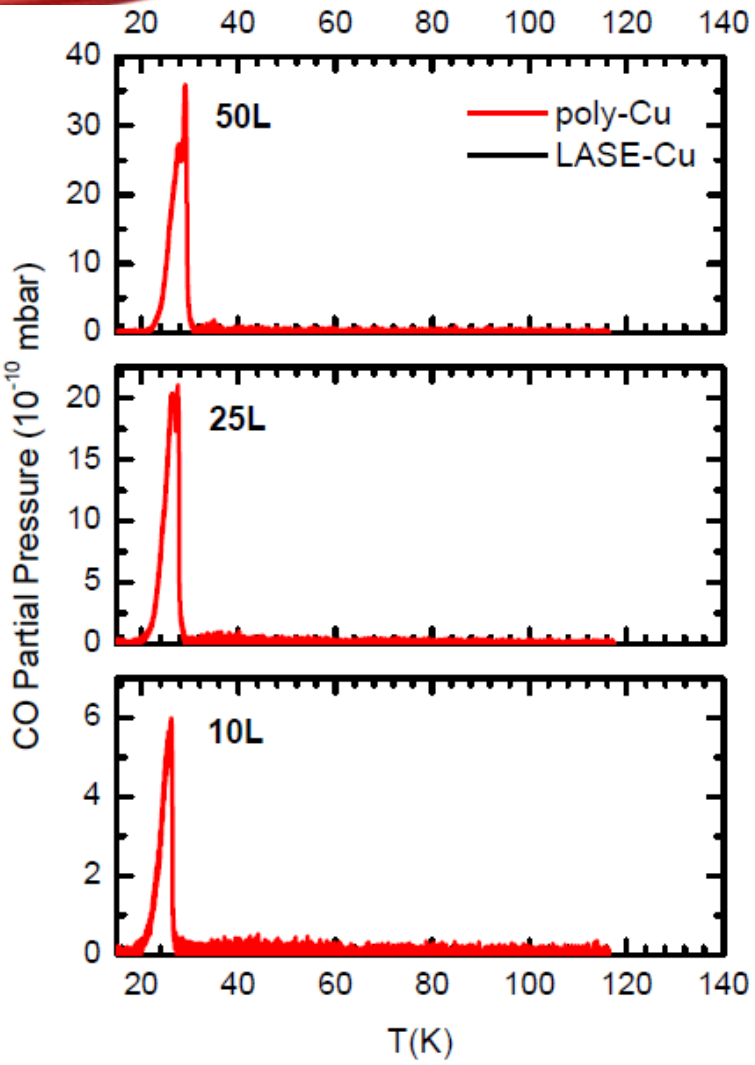
Received 17th December 2009, Accepted 29th March 2010
First published as an Advance Article on the web 30th April 2010
DOI: 10.1039/b926692e

EuroCircol Meeting in Karlsruhe

✓Temperature calibration
✓Coverage calibration

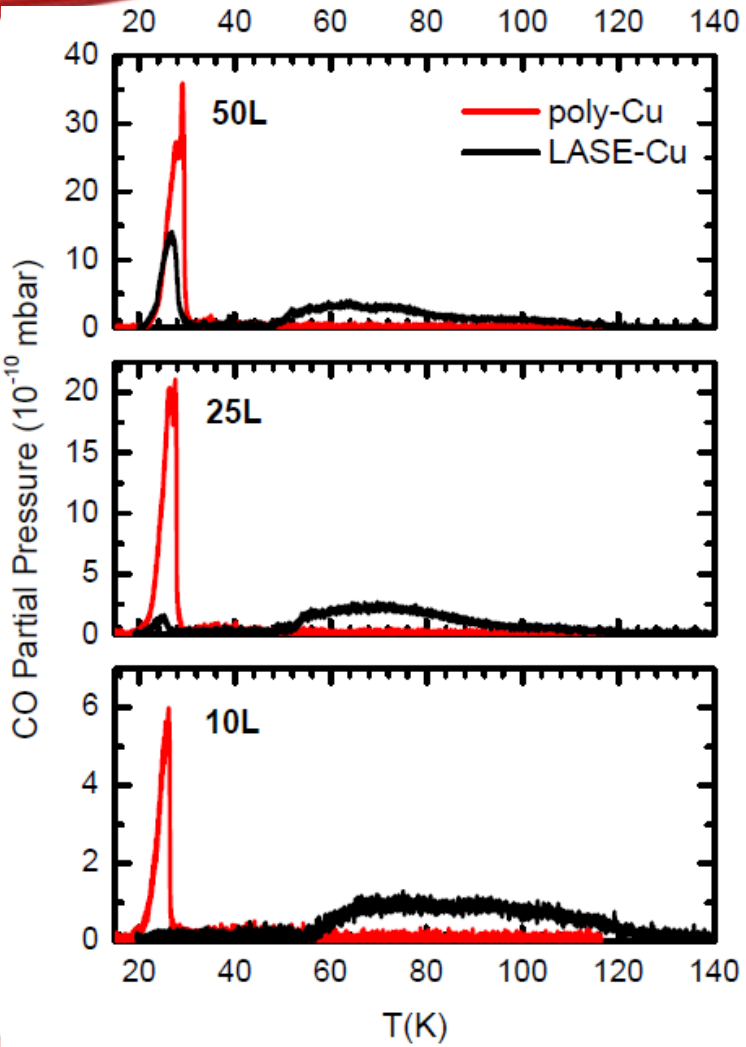
CO and CH_4 thermal desorption measurements: preliminary results

CO TPD Measurements



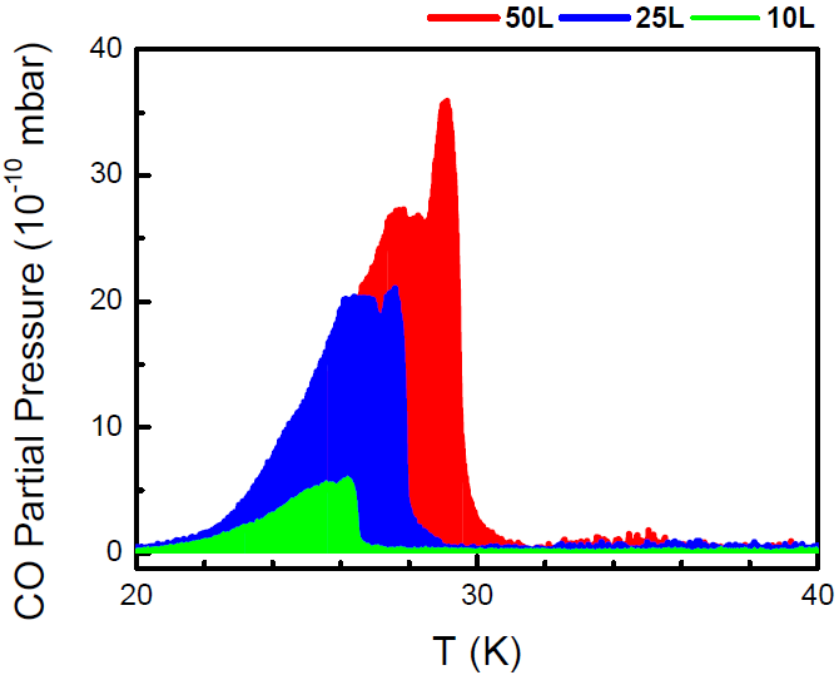
- On flat Cu CO adsorbs due to the weak CO-Cu and CO-CO Van der Waals interactions and the desorption curve consists of the sharp peak at T~30 K.

CO TPD Measurements



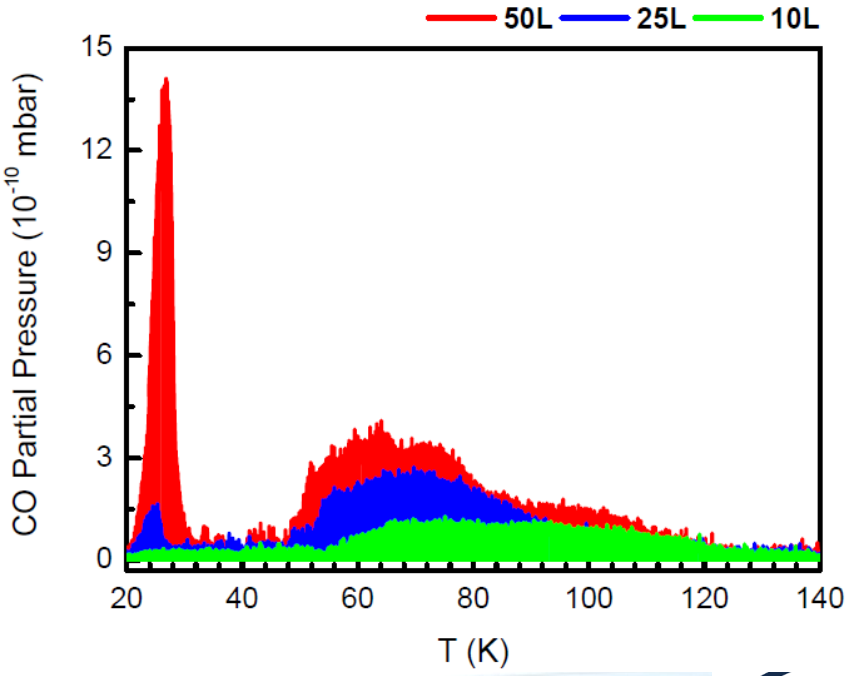
- On flat Cu CO adsorbs due to the weak CO-Cu and CO-CO Van der Waals interactions and the desorption curve consists of the sharp peak at T~30 K.
- For the LASE-Cu substrate the CO adsorption energy at the undercoordinated surface defect sites increases and desorption occurs at higher T. However, at high coverage, multilayer desorption at T~30 K is also observed.

CO TPD Measurements

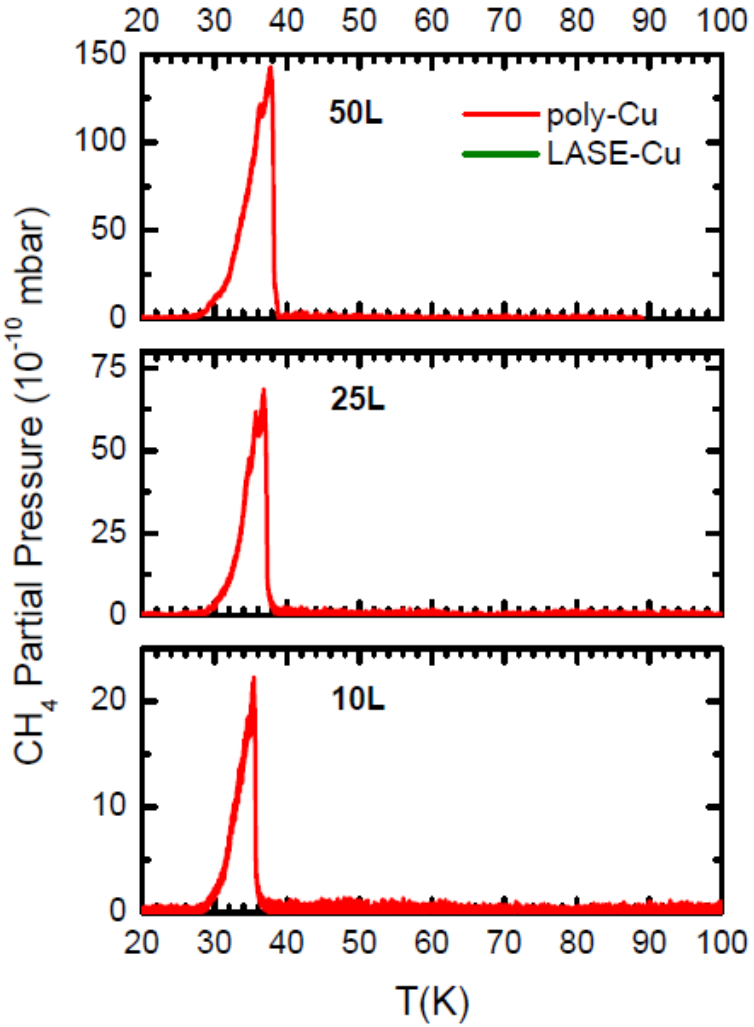


CO TPD curve from poly-Cu

CO TPD curve from LASE-Cu

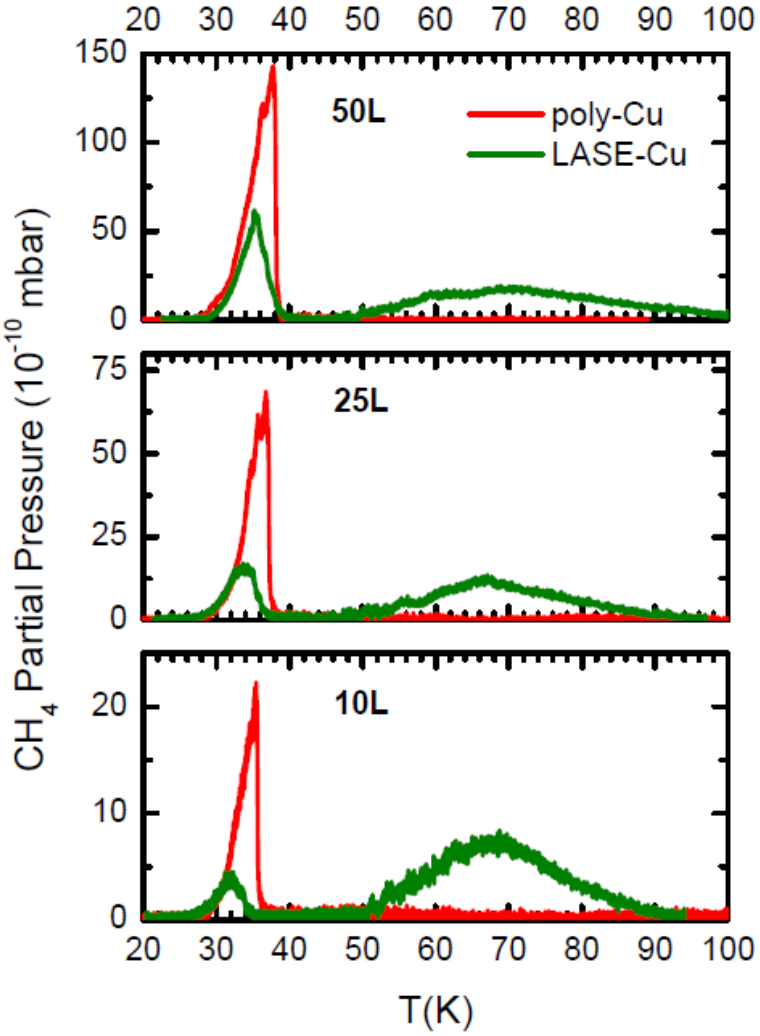


CH_4 TPD Measurements



- On flat Cu CH_4 adsorbs due to the weak CH_4-Cu and CH_4-CH_4 Van der Waals interactions and the desorption curve consists of the sharp peak at T~39 K.

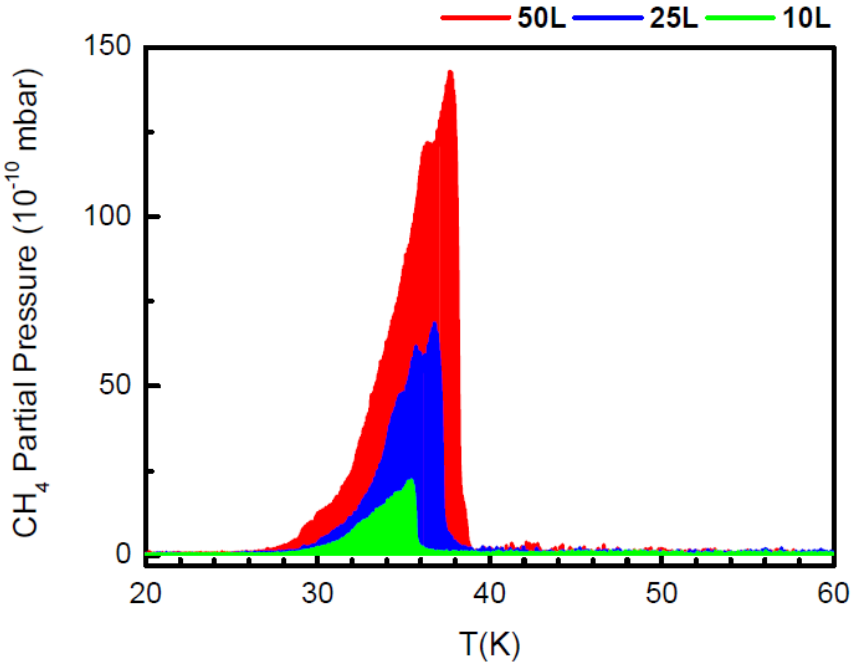
CH_4 TPD Measurements



- On flat Cu CH_4 adsorbs due to the weak CH_4-Cu and CH_4-CH_4 Van der Waals interactions and the desorption curve consists of the sharp peak at T~39 K.

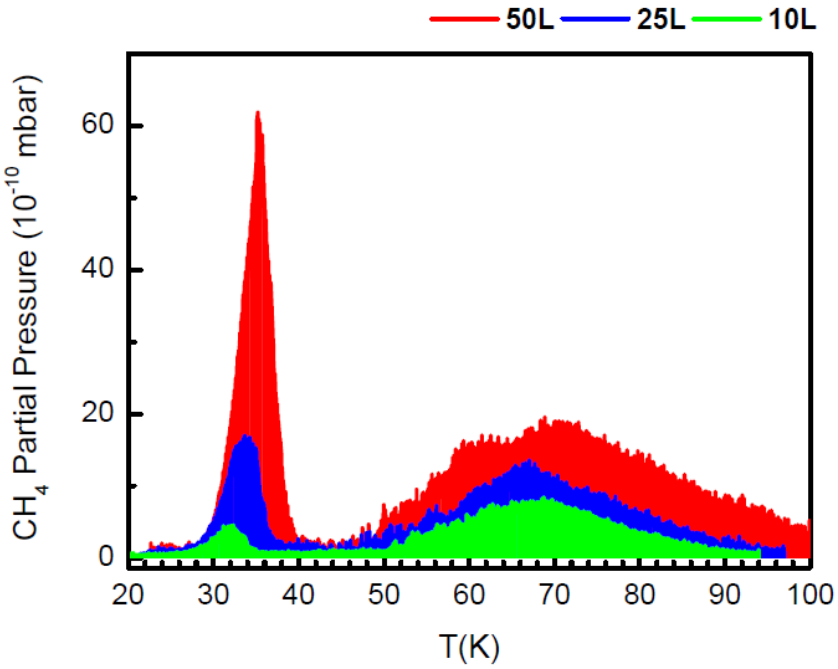
- For the LASE-Cu substrate the CH_4 adsorption energy at the undercoordinated surface defect sites increases and desorption occurs mainly at higher T. However, on increasing the coverage, the multilayer desorption at T~39 K also increases.

CH_4 TPD Measurements



CH_4 TPD curve from poly-Cu

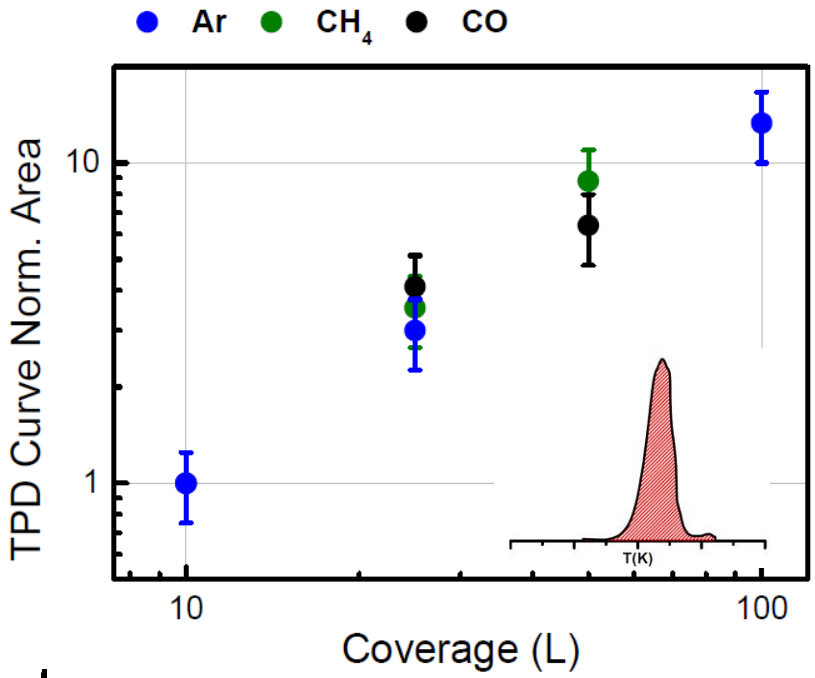
CH_4 TPD curve from LASE-Cu



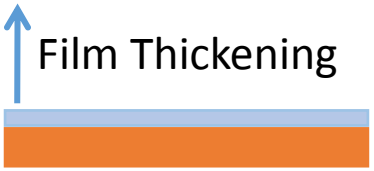
General Considerations

✓**The TPD results here reported confirm that what has been observed for Ar is a general trend common to some specific gases present as contaminant in accelerators**

General Considerations



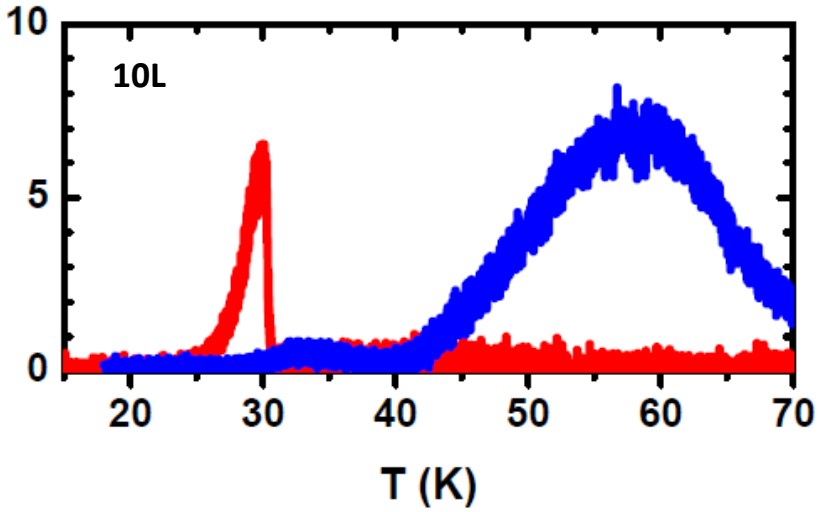
Gases dosed on poly-Cu substrate



Normalization to the TPD curve area at the lower coverage

General Considerations

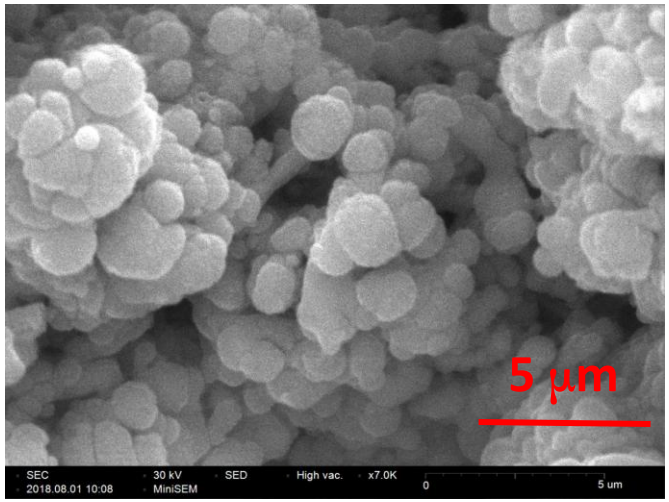
Ar TPD curve from poly-Cu and LASE-Cu



LASE-Cu can accommodate a larger quantity of gas so as expected by its morphology

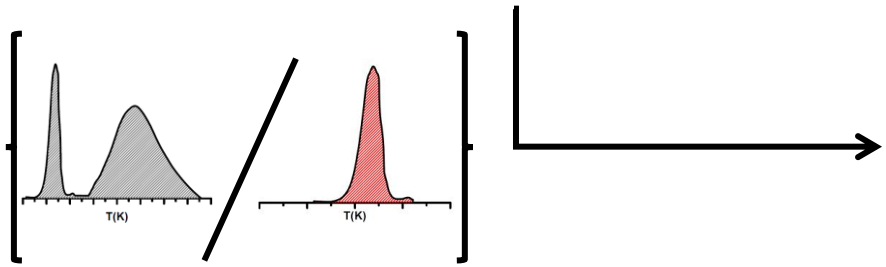
Highly porous and inhomogeneous surface with nanometric features (undercoordinated surface defect sites)

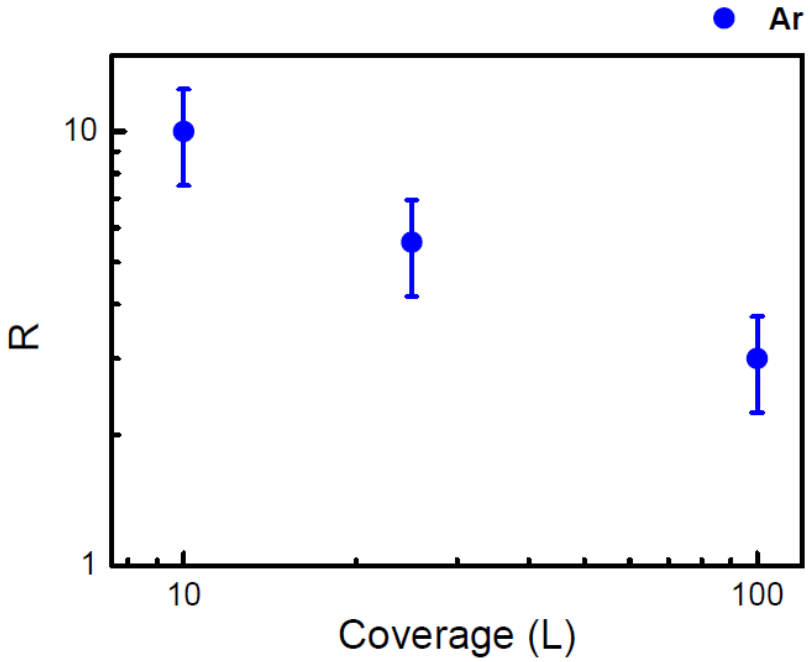
Morphology of LASE-Cu by SEM
(G. Viviani @ LNF-INFN)



General Considerations

R=(Ar on LASE-Cu TPD Curve Area)/(Ar on poly-Cu TPD Curve Area)



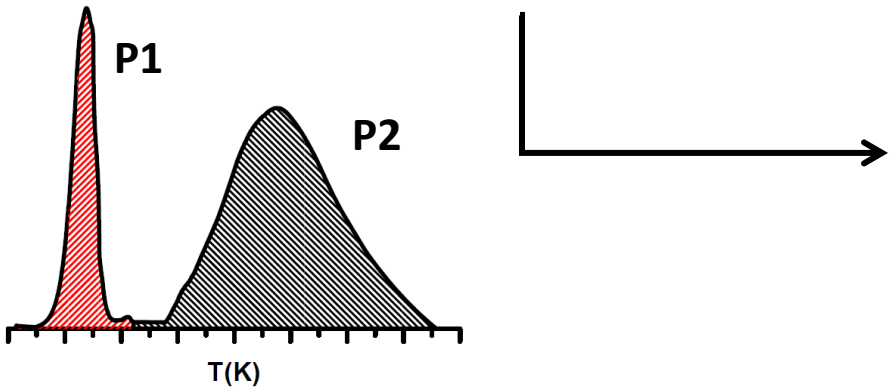


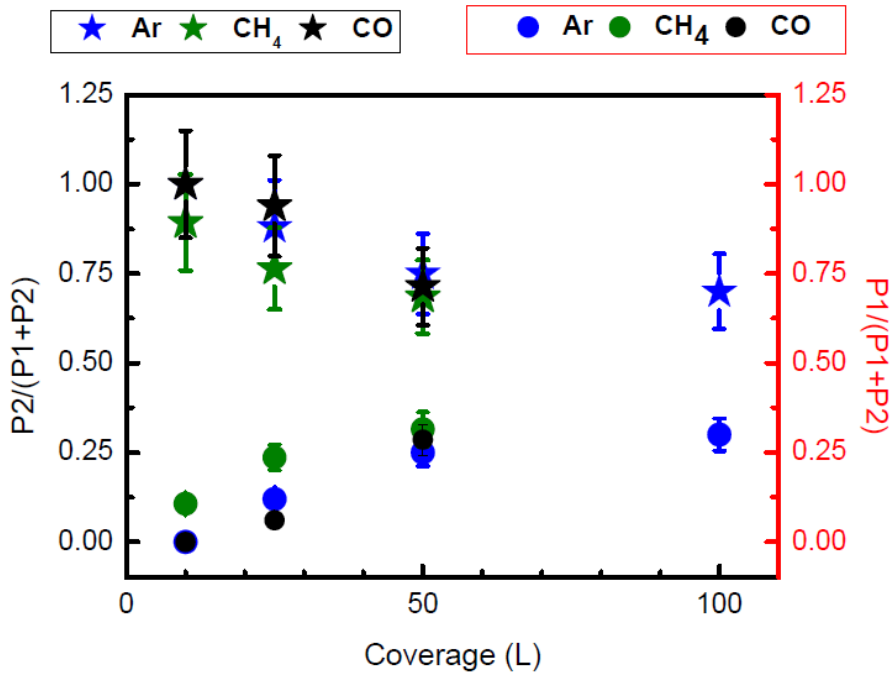
Accurate measurement of the specific surface is lacking. In this covering range, it's at least about 10 times larger than poly-Cu surface

This trend accounts for the desorption (and adsorption) kinetics of the gas in the LASE-Cu substrate determined by its morphology

General Considerations

P2/(P1+P2)=(TPD Curve Area under P2)/ (Total TPD Curve Area)





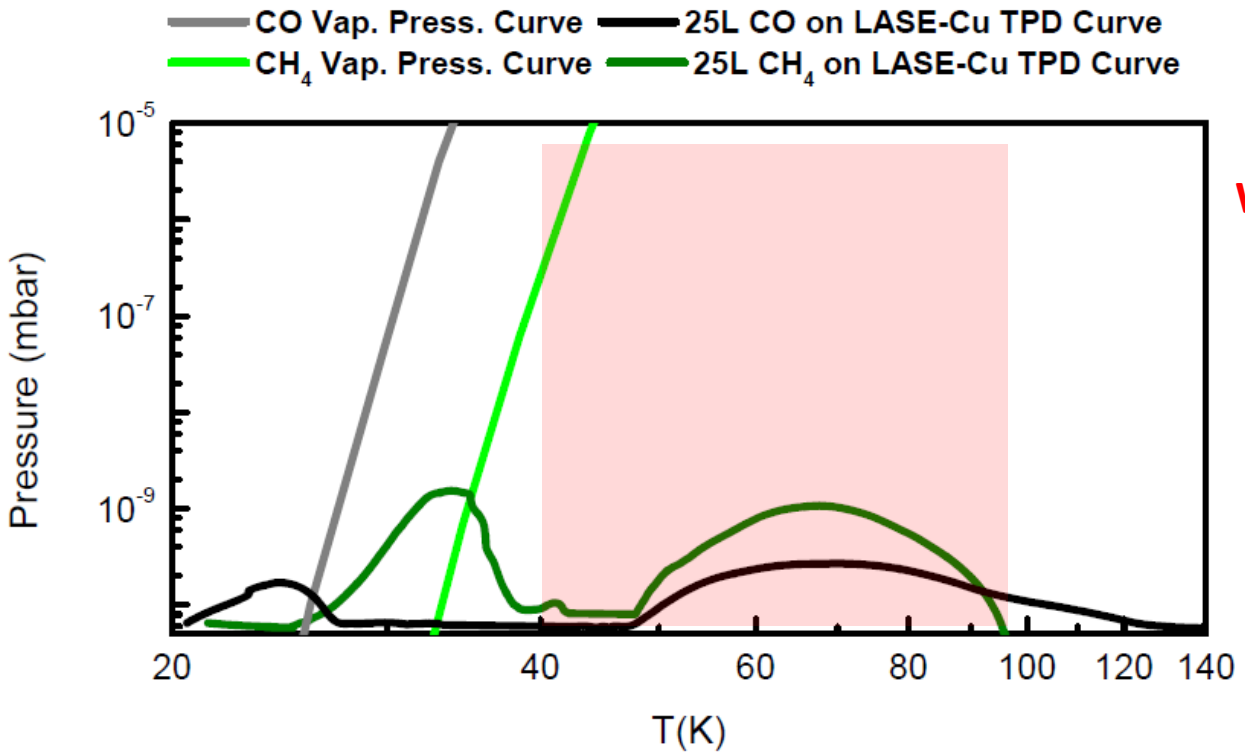
Gas desorption kinetics on LASE-Cu

Ubiquitous P2 contribution spreading in a broad temperature range

Gradually occupation of all available adsorption sites (pores wall included), up to saturation and ice thickening also on top surface

Implication for FCC-hh Vacuum Stability

Saturated vapour pressure from Honig and Hook (1960)



Wide desorption contribution over 40 K due to the morphological structuring of the material (intrinsic broad distribution of adsorbing defective sites and pores)

Implication for FCC-hh Vacuum Stability

WARNING!

This could render very difficult to find a temperature interval for the LASE-type sample here studied where vacuum stability could be granted for all the molecular species composing the residual gas in the beam pipe components

Outlook and future work

Conclusion

- **The use of this typo of LASE-substrate could be a problem for vacuum stability issues**

Outlook

- **<u>Electron and photo-desorption investigations</u>**
- **Better estimate of the consequences of such distributed temperature desorption via gas dynamic studies in real machines**
- **Optimization of LASE and SEY mitigation studies to optimize a material which is compliant both for SEY reduction and for vacuum stability**
- **Improve synergies between the different studies**
- **Long work: to be performed during EUROCIRCOL2?**

THANKS TO….

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