Vacuum stability at cryogenic temperature

WP4 - Activity at LNF

Amsterdam, 12/04/2018

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In March, during EuroCircol Meeting in Frascati

Thermal desorption measurements: preliminary results

Desorption processes of Ar from clean Cu and Laser Treated (LASE)-Cu

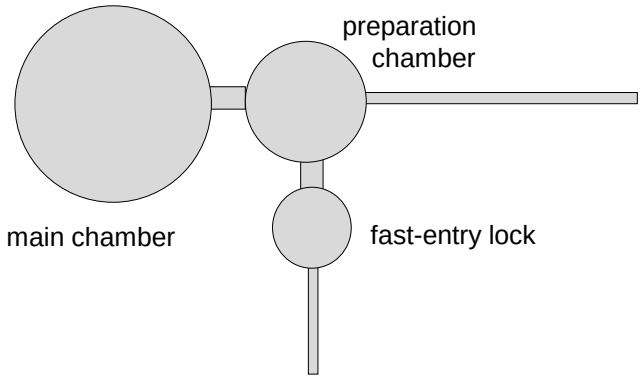
Istituto Nazionale di Fisica Nucleare

Today, during the EuroCircol Meeting in Amsterdam

- **Summary of the main activities**
  - Dose calibration
  - Temperature calibration
  - Desorption calibration
- **Thermal desorption measurements: data analysis**

Set-up and Strategy at LNF

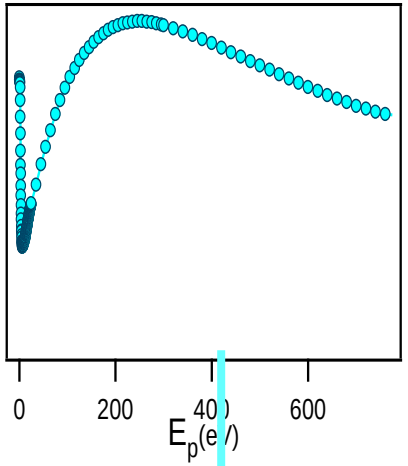
Ultra high vacuum systems

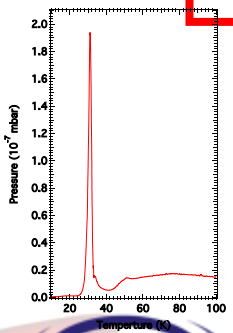


- LNF-cryogenic manipulator
- Sample at 15-300 K

Secondary Electron Yield (**SEY**) measurements

Equipment : Electron gun, Faraday cup



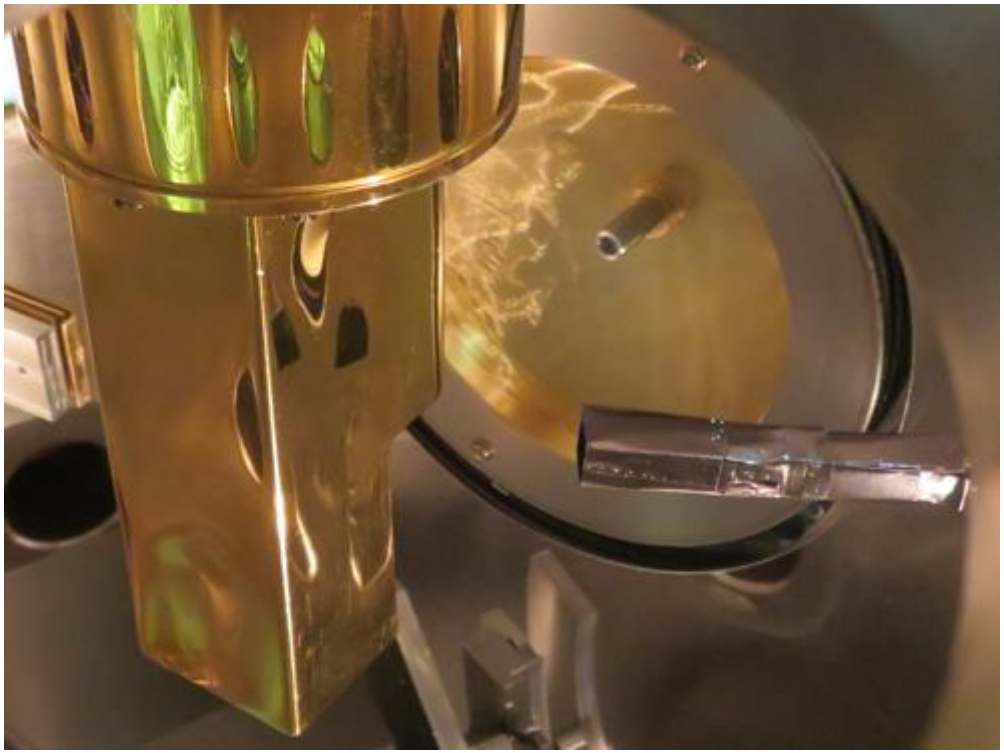


Temperature Programmed Desorption (**TPD**) measurements

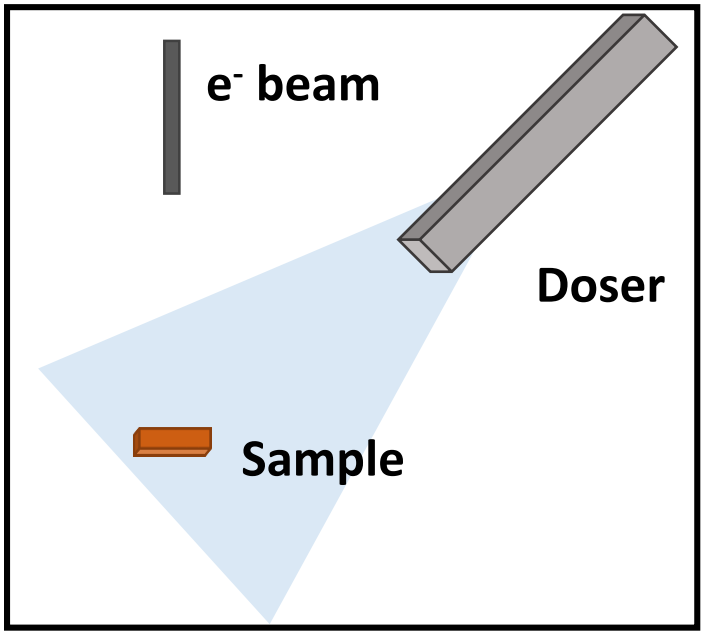
Equipment : QMS (Hiden HAL 101 Pic)

Dose calibration

Gas dosing OLD set-up

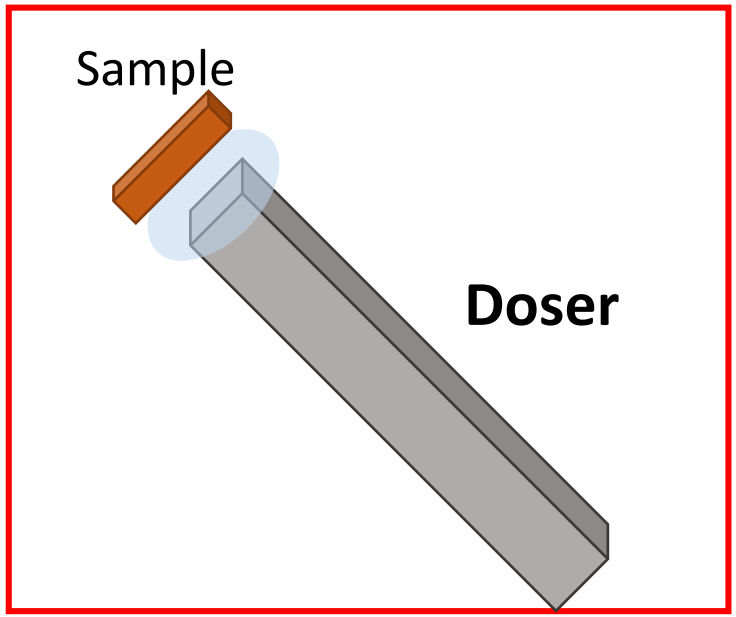


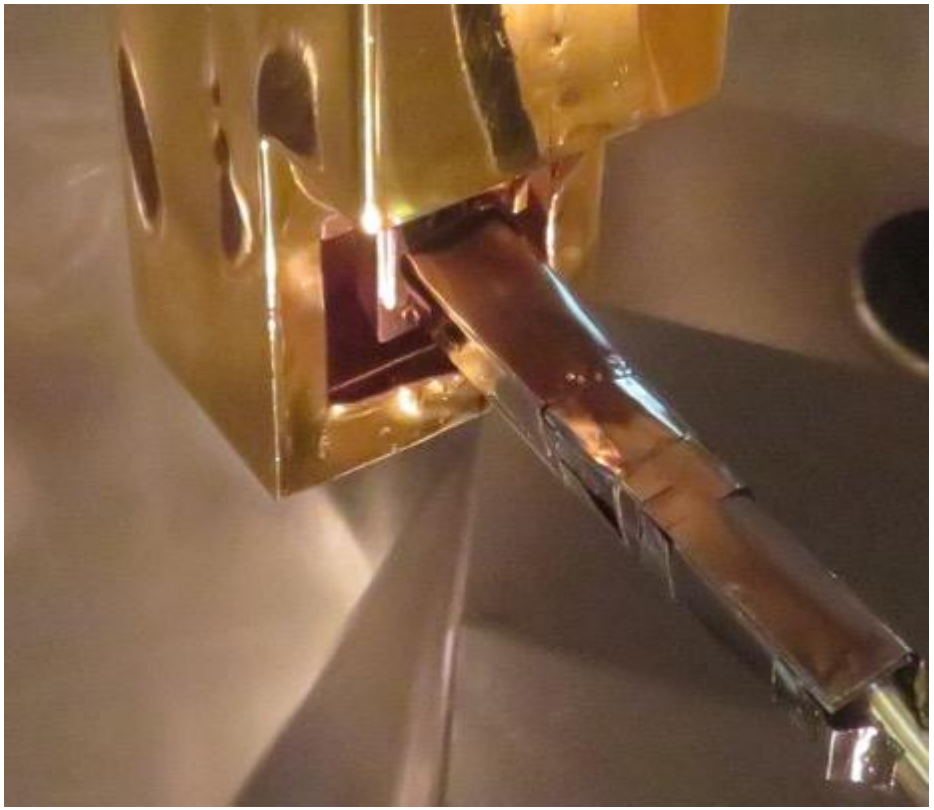
Far from the sample



Gas dosing NEW set-up

Near to the sample

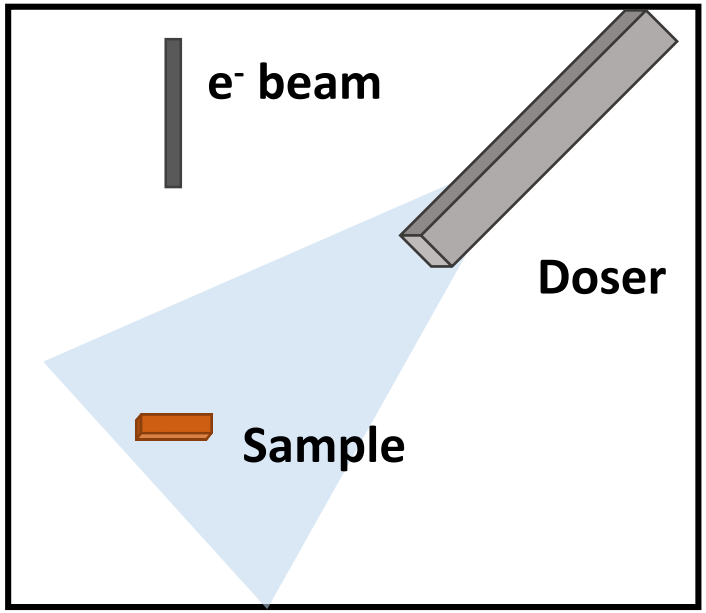




Different local pressure on the sample

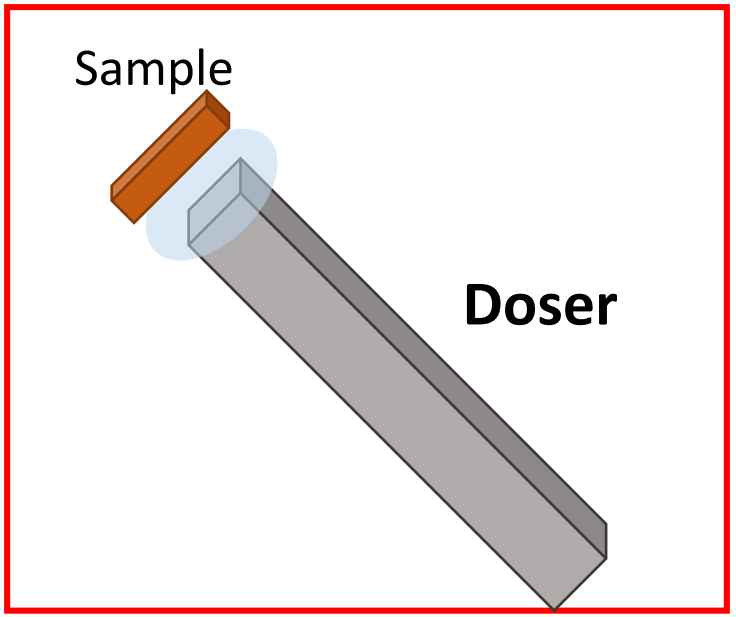
1s@1.33x10^{-6} mbar corresponds to

Far from the sample



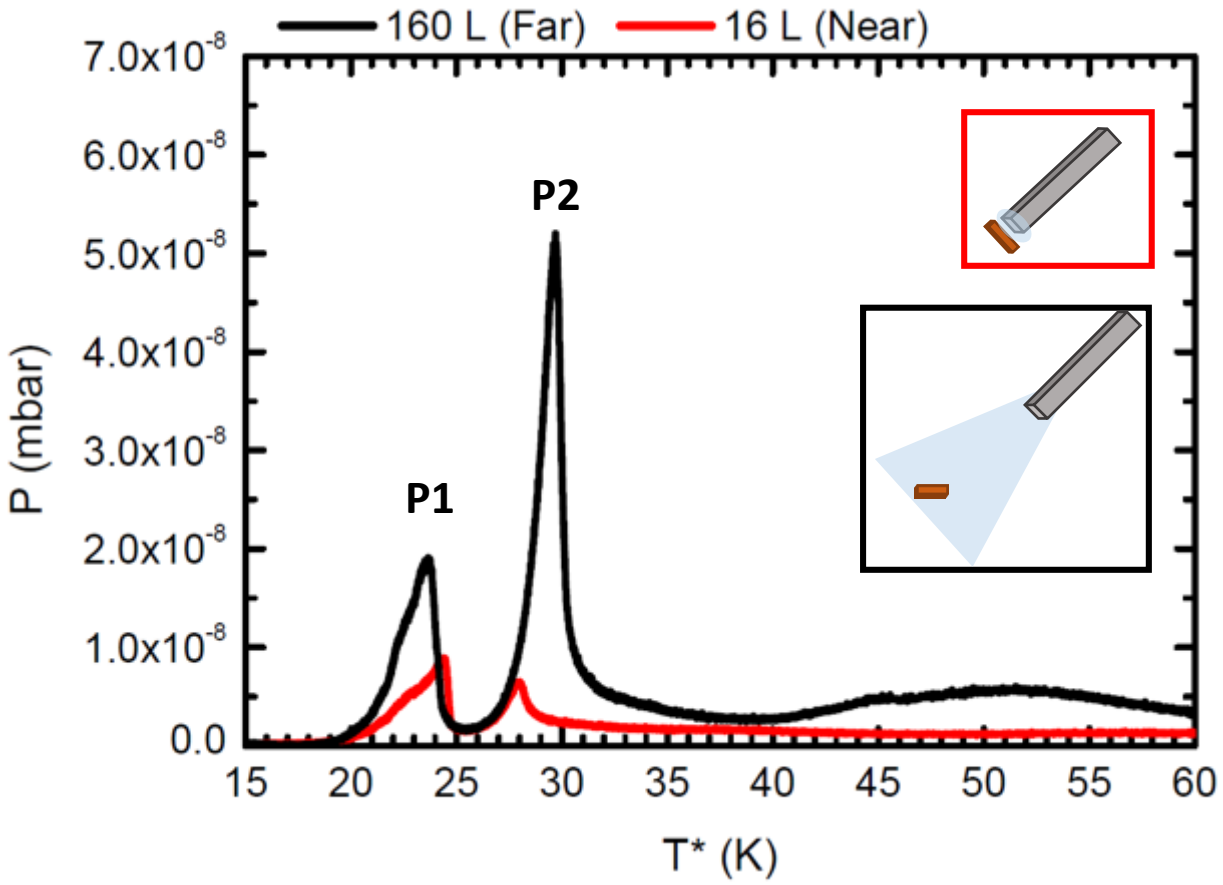
1 Langmuir

Near to the sample



≈ 8 Langmuir

Accurate Calibration in progress



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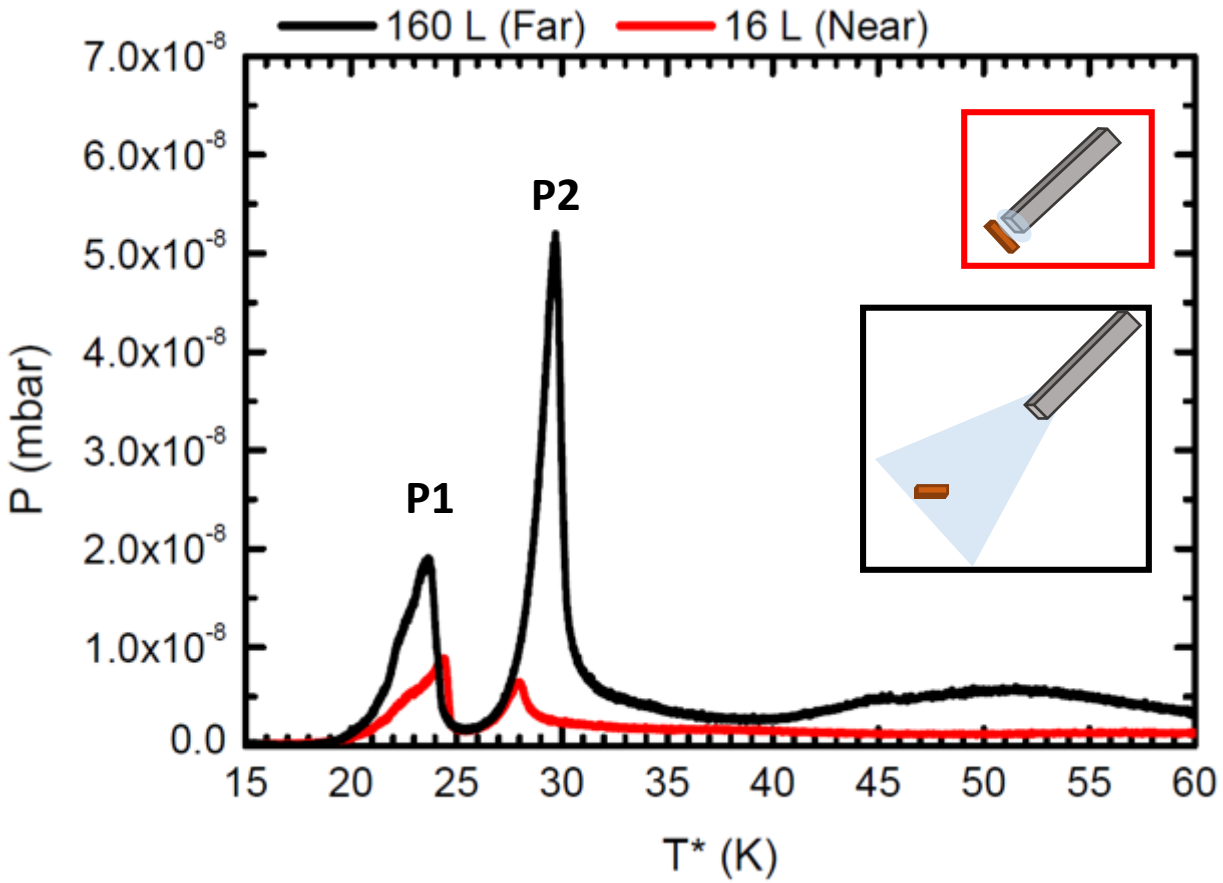
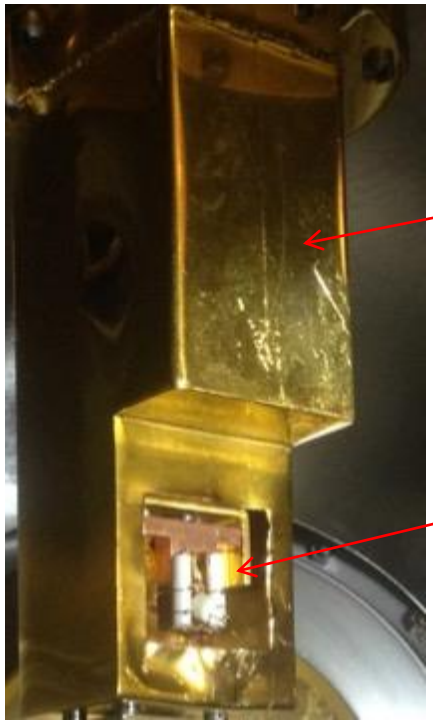


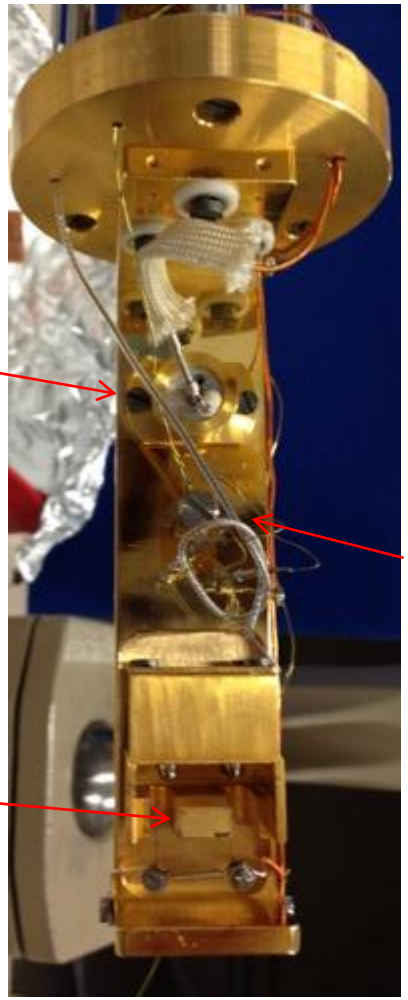
Accurate Calibration in progress

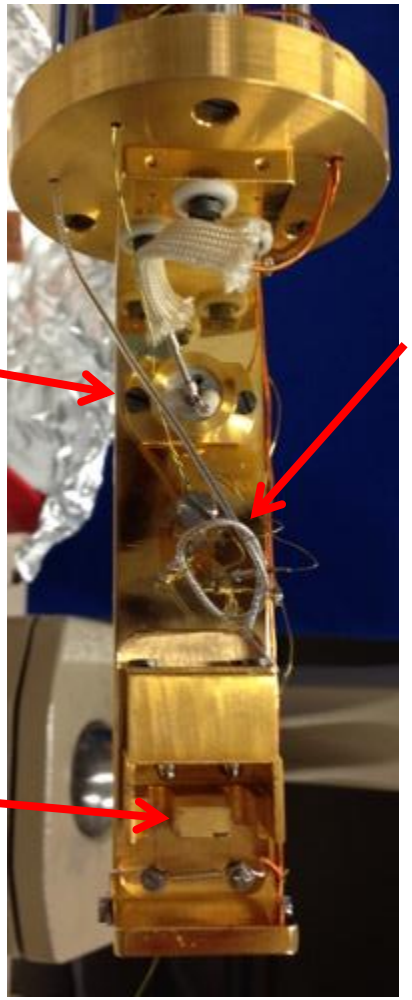
✓Putting the doser near the sample is effective for the reduction of the desorption related to the manipulator

Temperature calibration

LNF-Cryogenic Manipulator



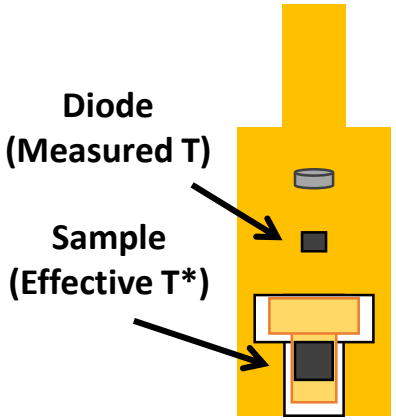


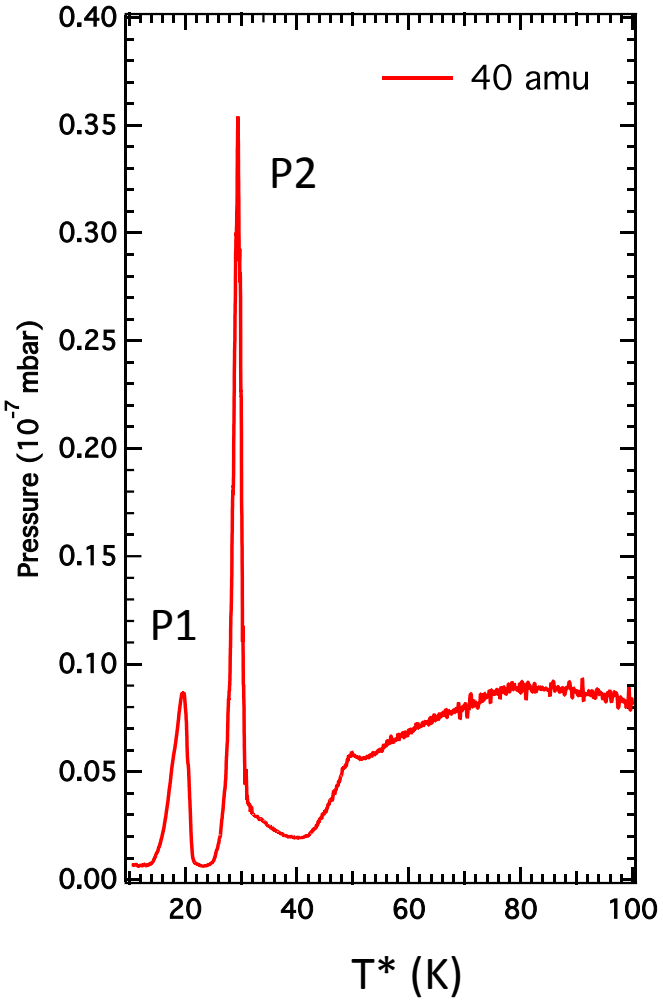


Measured Temperature (T*)

≠

Sample Real Temperature (T)





Ar Temperature Programmed Desorption

The **different desorption peaks** are experimental artefact

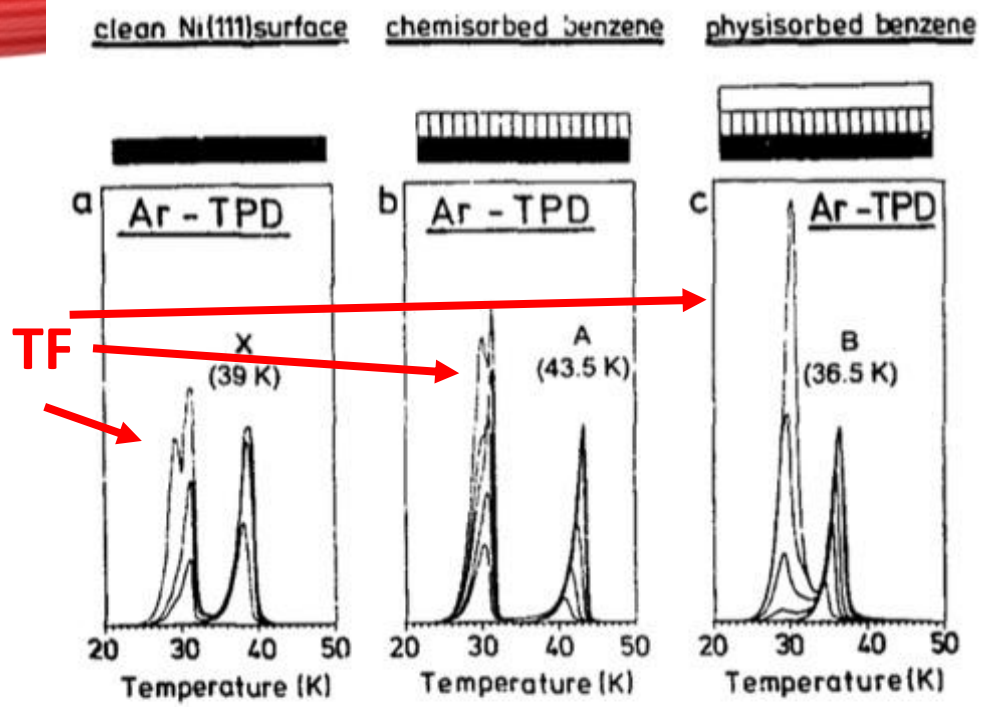


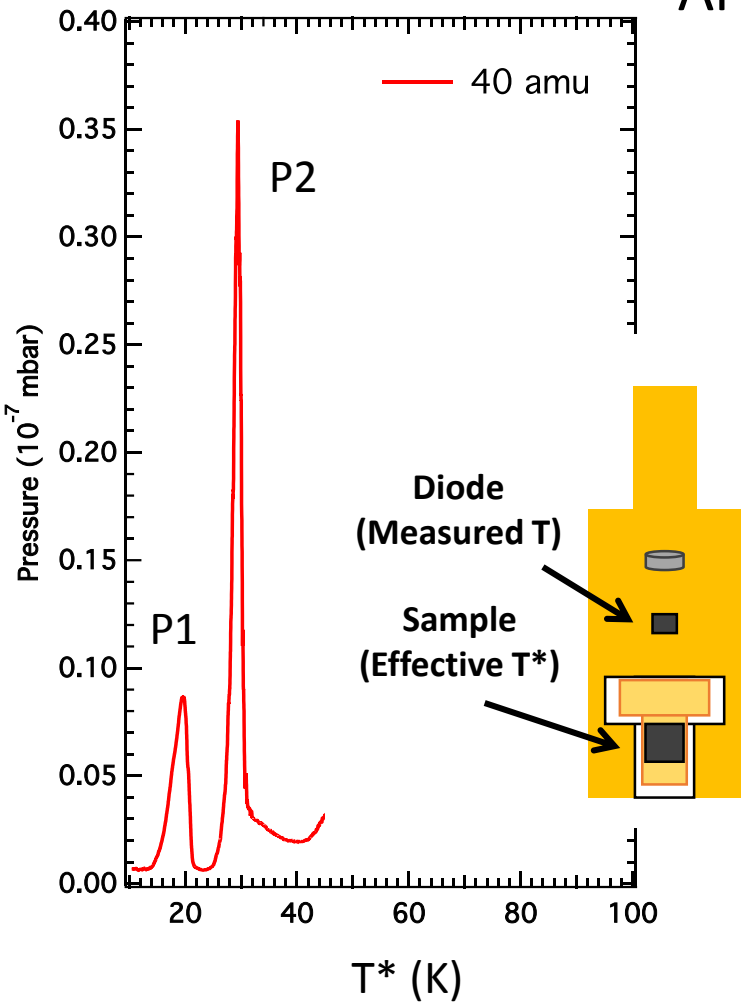
Fig. 1. Argon desorption spectra for increasing argon exposures onto various underlying "substrates": (a) clean Ni(111); (b) saturated chemisorbed $(\sqrt{7} \times \sqrt{7})R19.1°$ benzene layer on Ni(111); (c) saturated first physisorbed benzene layer on top of the chemisorbed layer. Adsorption temperature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

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

Ar TF desorbs at a unique T~30 K

M. Stichler et al.; Surface Science348 (1996) 370-378

Ar Temperature Programmed Desorption

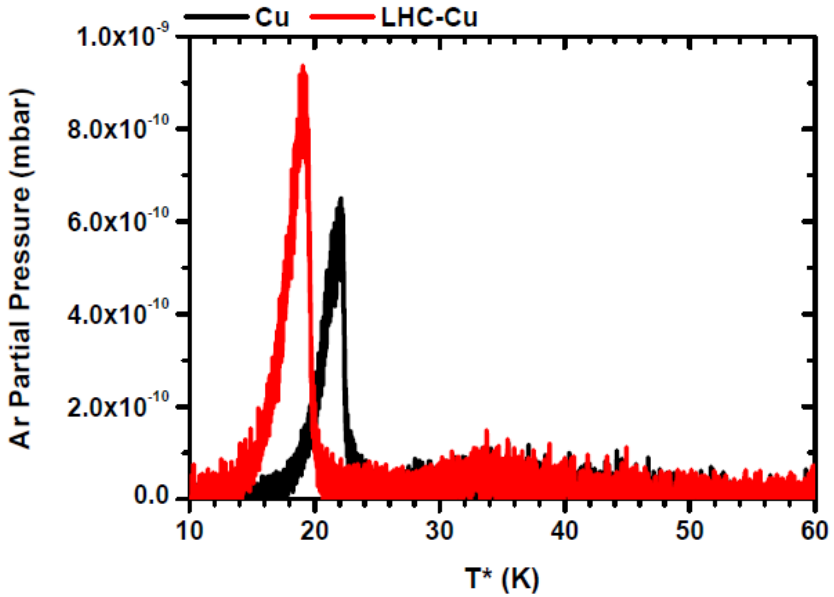


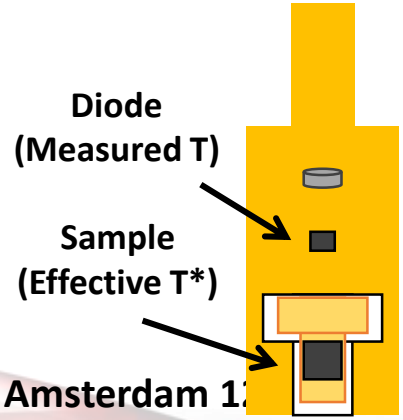
Peak 1: Desorption from sample ("hotter part" at T*)

Peak 2: Desorption from Manipulator (at T)

The different desorption peaks are experimental artefact, not real...but **advantageous for us**!!!

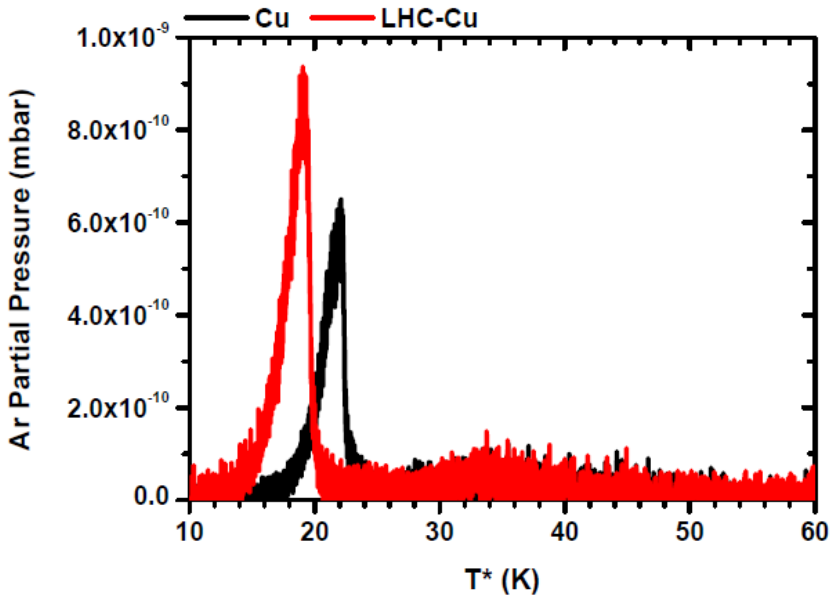
T* shift

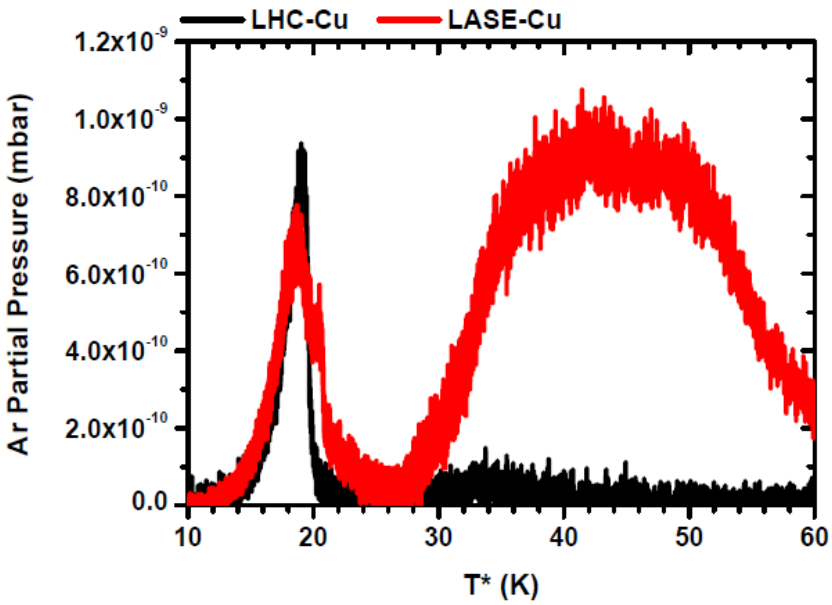


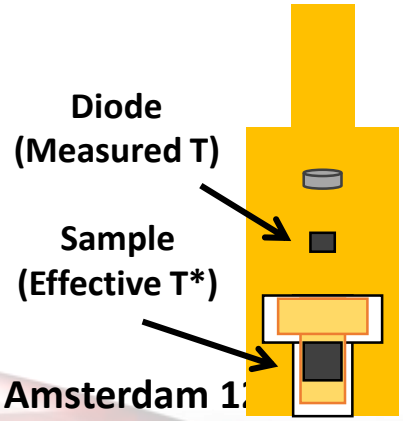


The effective temperature of LCH sample is higher due to stainless steel core, so its desorption peak appears at a lower T* respect to Cu!

T* shift







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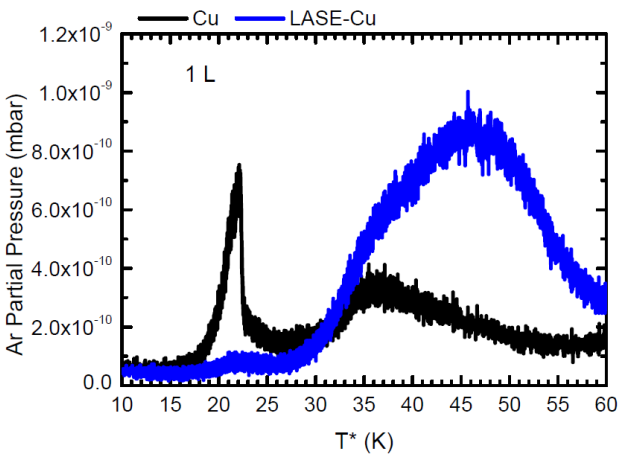
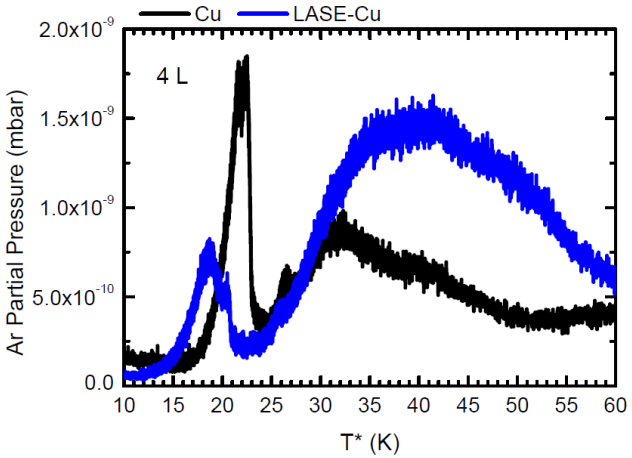
In March, during EuroCircol Meeting in Frascati

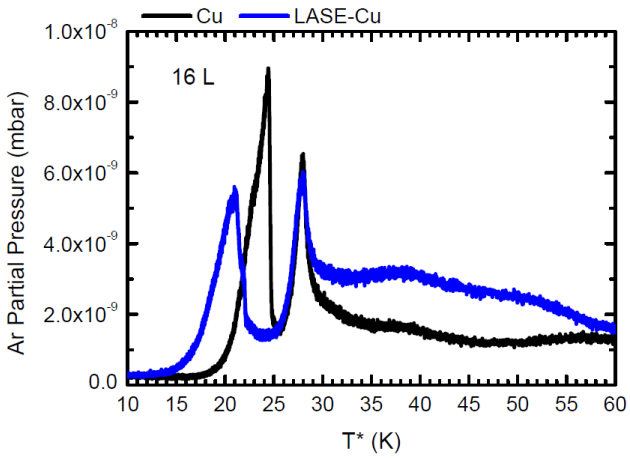
Thermal desorption measurements: preliminary results

Desorption processes of Ar from clean Cu and Laser Treated (LASE)-Cu

Synopsys of the raw data

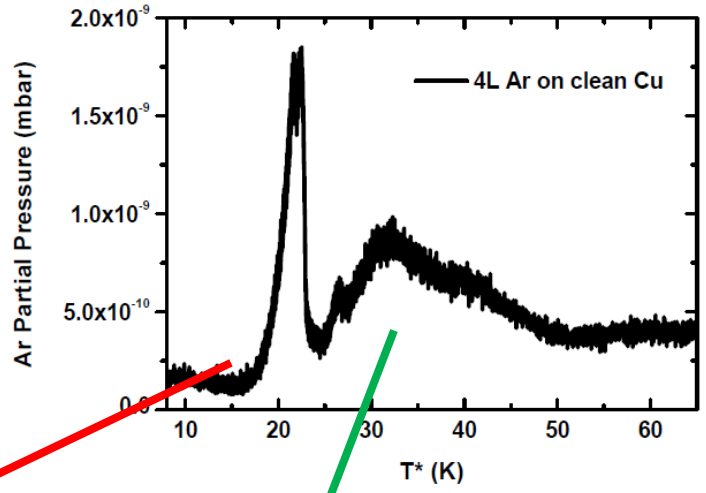




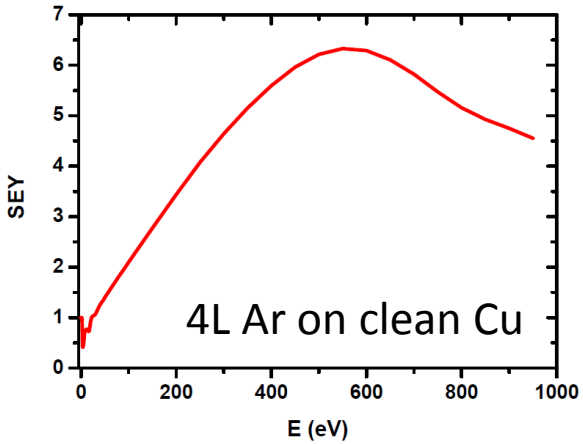


Thermal desorption measurements: data analysis

SEY vs T as cross-check for interpreting the thermal desorption curves

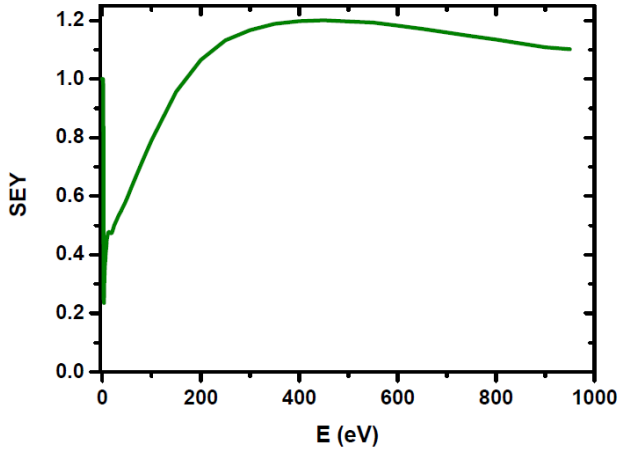


Below T=20 K

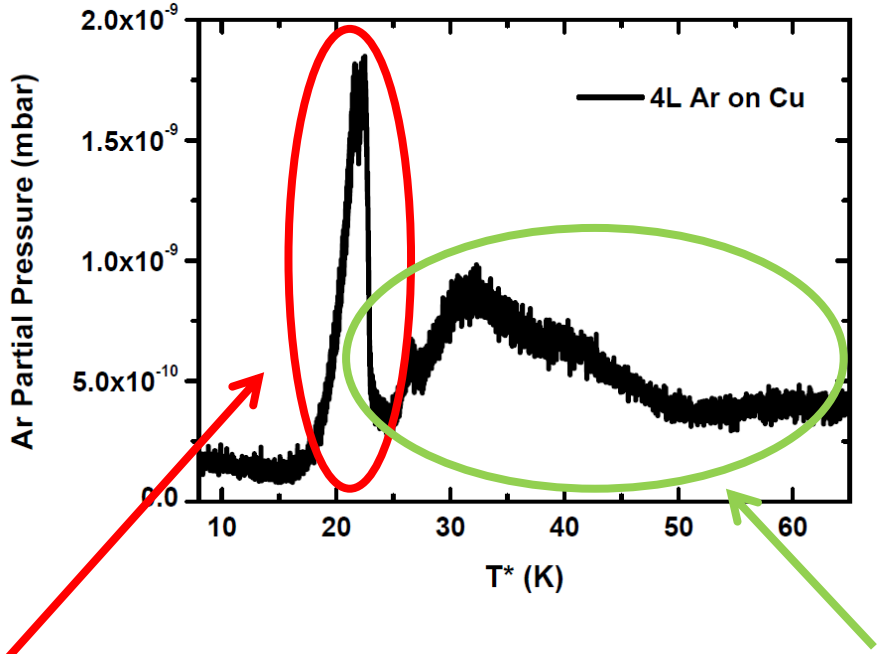


Typical SEY of 1, 4, 16 L of Ar thick film on clean Cu

Above T=30 K



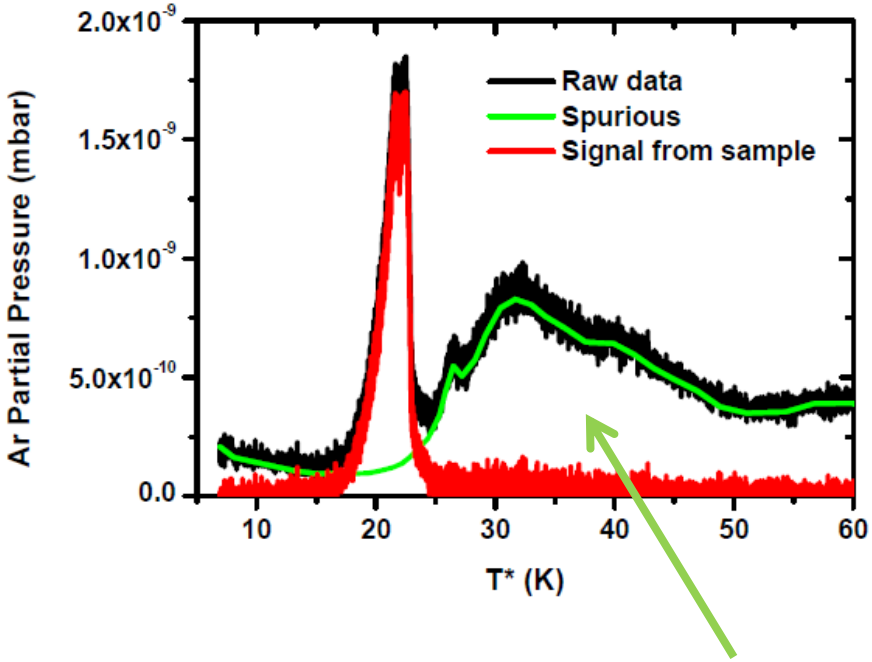
Typical SEY of clean Cu



Ar desorption from other part of the system

Ar desorption from sample

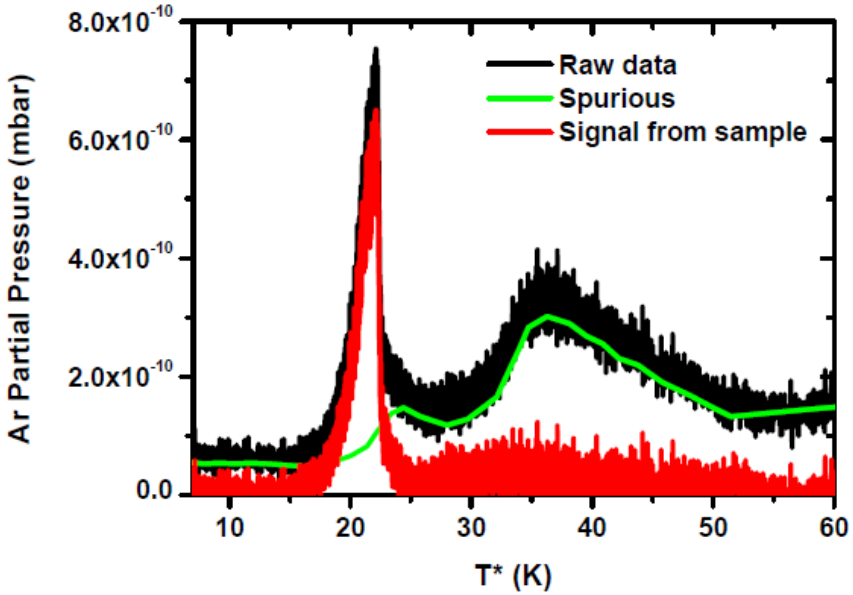
Subtraction of the contribution accounting for the desorption from other part of the system



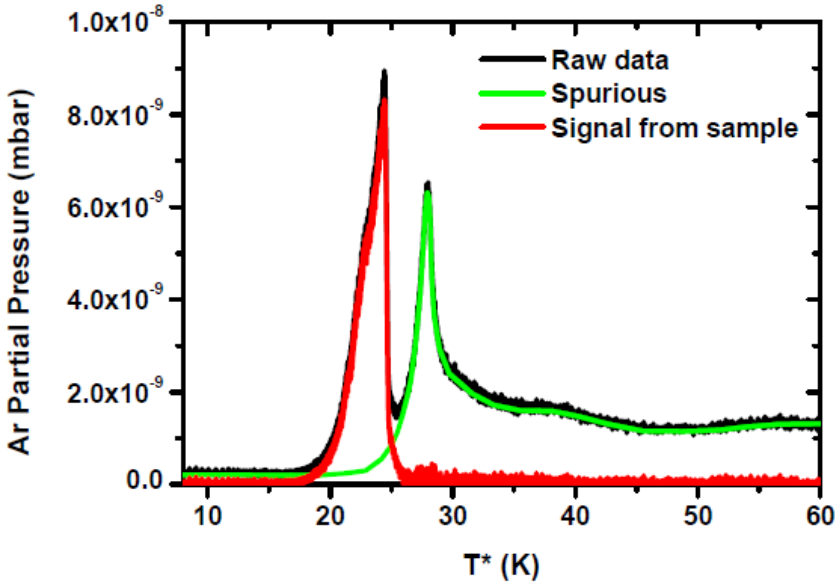
4 L on clean Cu

Spurious contribution is independent from the sample!!!
It depends on the dose only.

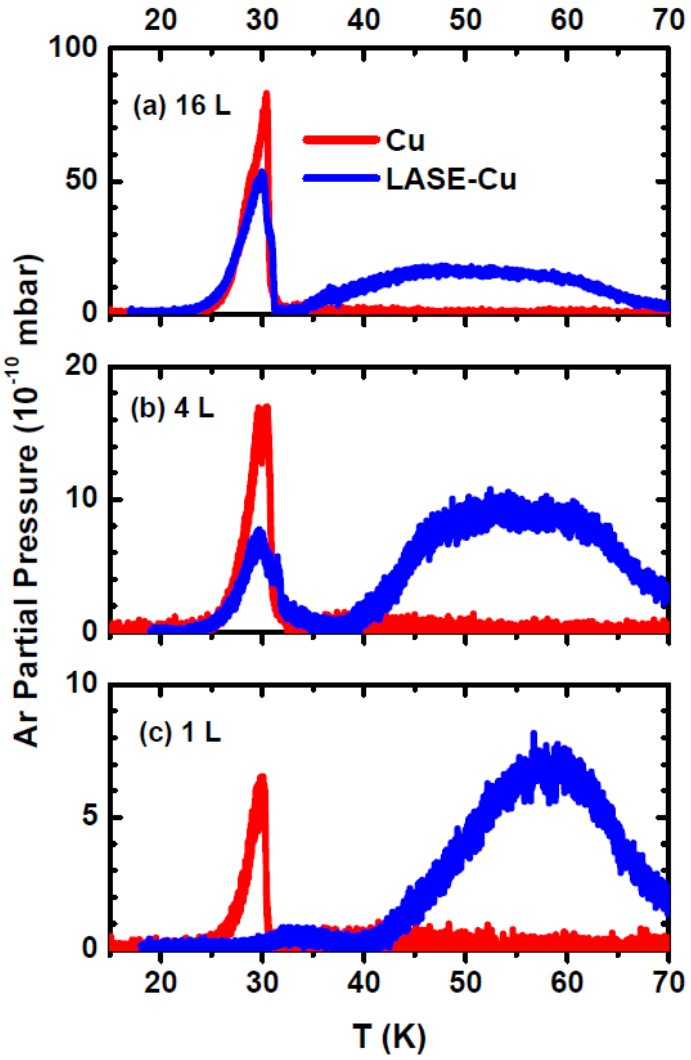
1 L on clean Cu



16 L on clean Cu



At a fixed gas dose, the desorption of Ar on flat substrate is decisive to single out both the sample and the spurious contribution at that dose



- 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.

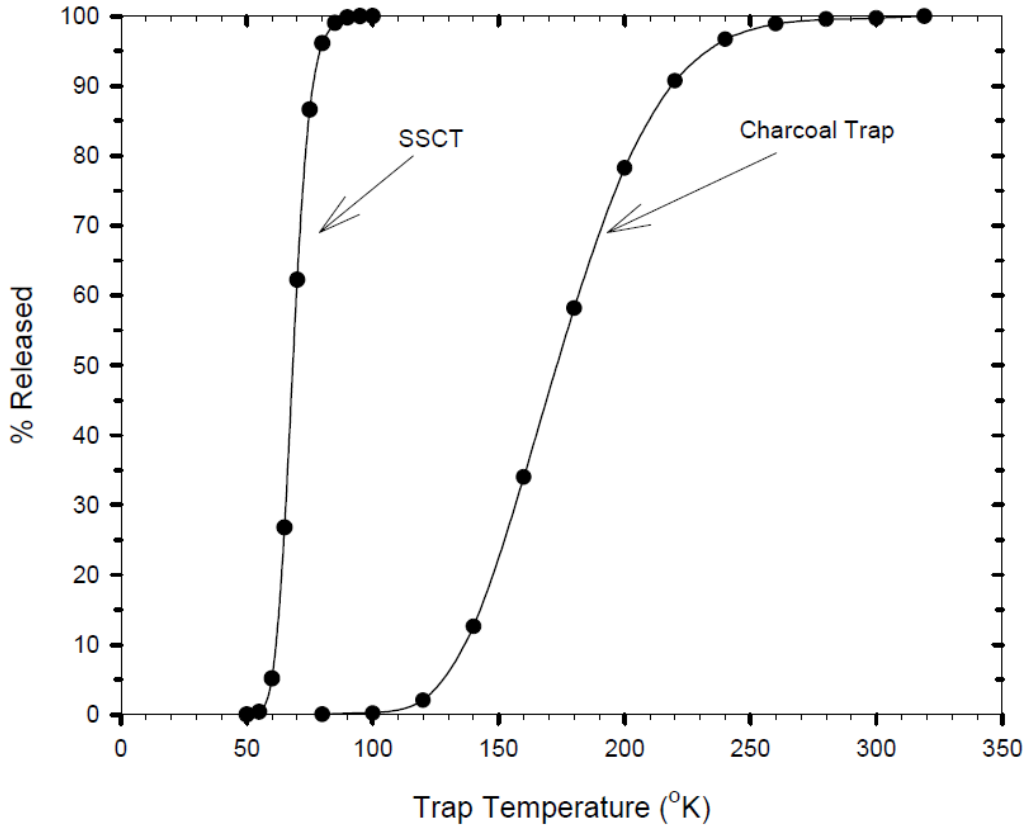


Figure 5. A comparison of the SSCT and charcoal cryogenic trap release characteristics for argon as a function of temperature.

Geochemistry Geophysics Geosystems G3
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Volume 2
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Paper number 2001GC000202
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Improvements in noble gas separation methodology: A nude cryogenic trap

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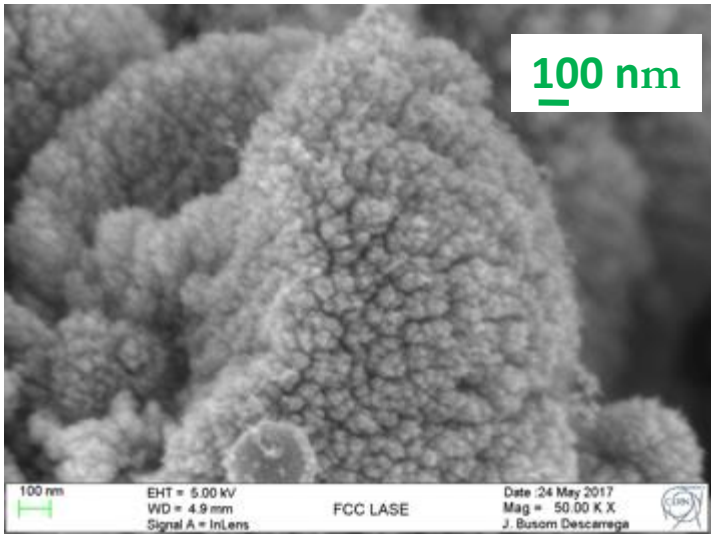
Department of Marine Chemistry and Geochemistry, Woods Hole Oceanographic Institution, Woods Hole, Massachusetts 02543, USA (dlott@whoi.edu)

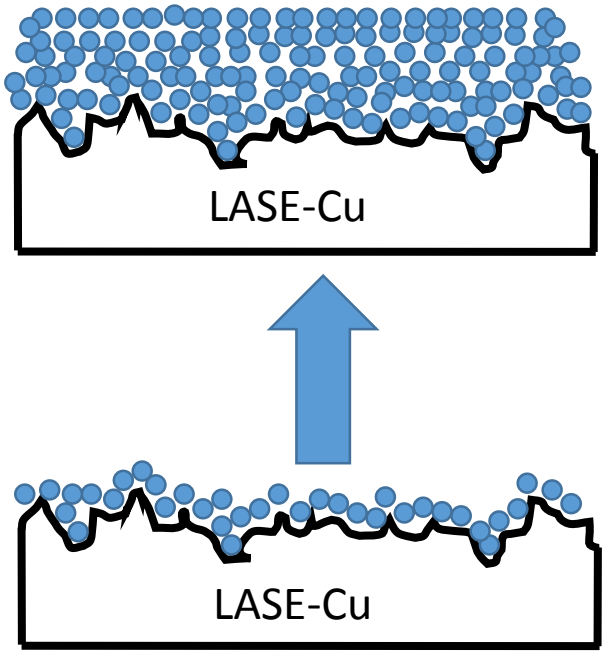
Desorption processes in charcoal and other cryotraps



Morphology of LASE-Cu by SEM

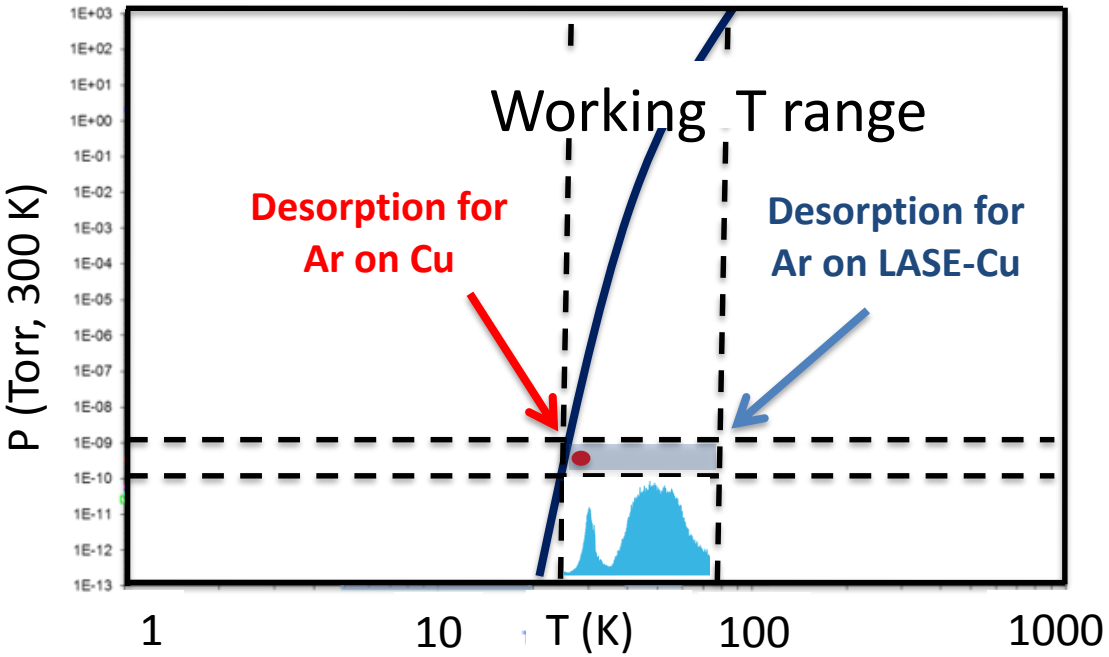
Highly rough and inhomogeneous surface with nanometric features (undercoordinated surface defect sites)

At higher coverages the desorption is dominated by usual Ar/Ar Van-der-Waals interaction



At low coverages the desorption is dominated by Ar/LASE interaction

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



For ices dominated by Ar-LASE, Ar desorbs both at T~ 25-30 K and in a much wider range

WARNING: If confirmed, the use of highly porous materials at LT must be considered with great care!

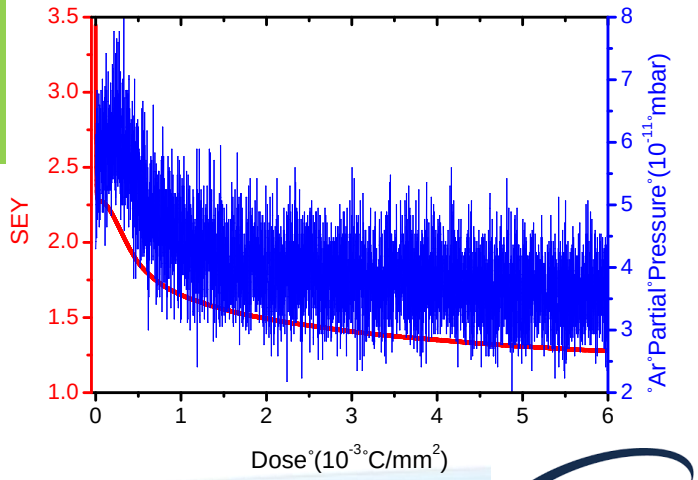
Outlook and future work

Technical work

- **Gas-line assembly**
- **Assembly and test of the new heater**

Outlook

- **CO, CO_2, CH_4 desorption studies**
- **Electron and photo-desorption investigations**



THANKS TO….

E. La Francesca

R. Cimino

R. Larciprete

A. Liedl

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The team at LNF

DAΦNE-L

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