Contamination par le carbone, décontamination et prévention dans un environnement UHV

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Journées Thématiques « Molécules Organiques et UHV »

12-13/10/2015

« Carbon Cleaning » à SOLEIL: le début

- LURE: Optics Group performs off-situ Plasma cleaning as standard
- 2005 first tests with UV-lamps at LURE (SB7:Chauvet/Kubsky/Sirotti)
- 2005- first meetings with beamline scientists at SOLEIL
- 2005- development of a test bench at the Surface Laboratory (LaSu)
- 2006-2008 development of UV-lamps and a UHV device for TEMPO in collaboration with LCPMR (Paris6)
- 2008- First tests of EVACTRON and subsequently GV10x on DESIRS
- 2008- several carbon cleaning campaigns for beamlines: LUCIA, DESIRS, TEMPO, DISCO
- 2009- proof of concept for the UV-UHV and *in-situ device* on TEMPO
- 2009- quantitative measurements on decapping speed at LaSu
- 2010- Start of engineering for an integrated concept of UV-based Carbon-Cleaning for HERMES and NANOSCOPIUM: Purgeable UV-lamps

Carbon Cleaning: Goals for SOLEIL

- A practical cleaning procedure is necessary due to rapid Carbon deposit with respect to second generation sources on timescales of only a few weeks
- Cleaning must be possible *in-situ* since many beamlines employ cryogenically cooled optics: difficulty to dis- and remount and delicate adjustment procedures needed each time (~1 week downtime)

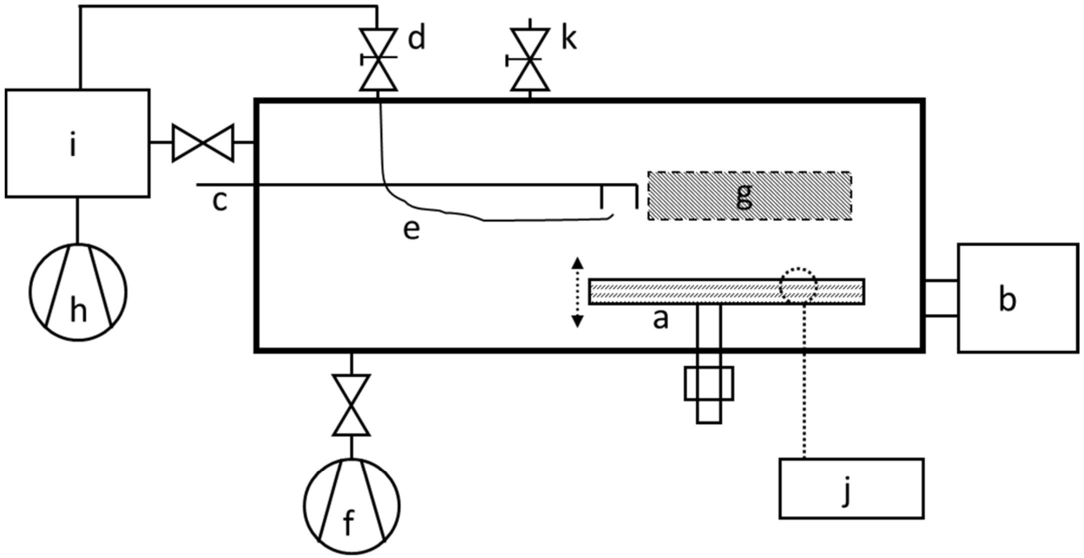
- Difficult to quantify results on decapping speed and quality
- Problematic to test real-world samples
- How to understand the underlying physical principles

- In-situ cleaning can save time and money, but needs a multilateral approach:
  - Cleaning device and procedure: Make one !
  - Measures to prevent contamination in order to minimize re-cleaning: Chamber quality
  - Understanding of the mecanisms: See Pellegrin-presentation and paper

Carbon Cleaning at SOLEIL: How it started

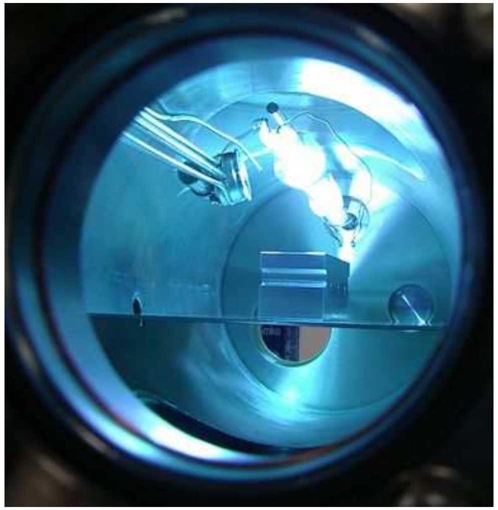
First test in 2005 at SuperACO

Drew Bertwhistle (CLS) on visit at LaSu



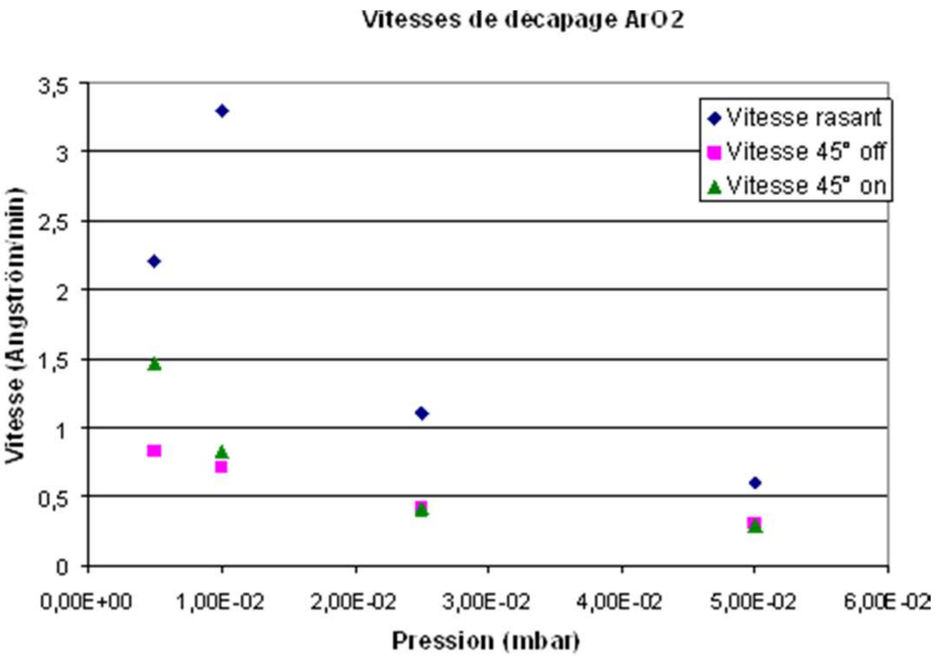
a : UV Lamp with translation
b : Plasma Generator GV10x
c : Quartz Balance
k : gas inlet valve
d, e : valve and capillary to RGA
f,h : pumping
i : RGA chamber
g : sample
j : optical spectrometer

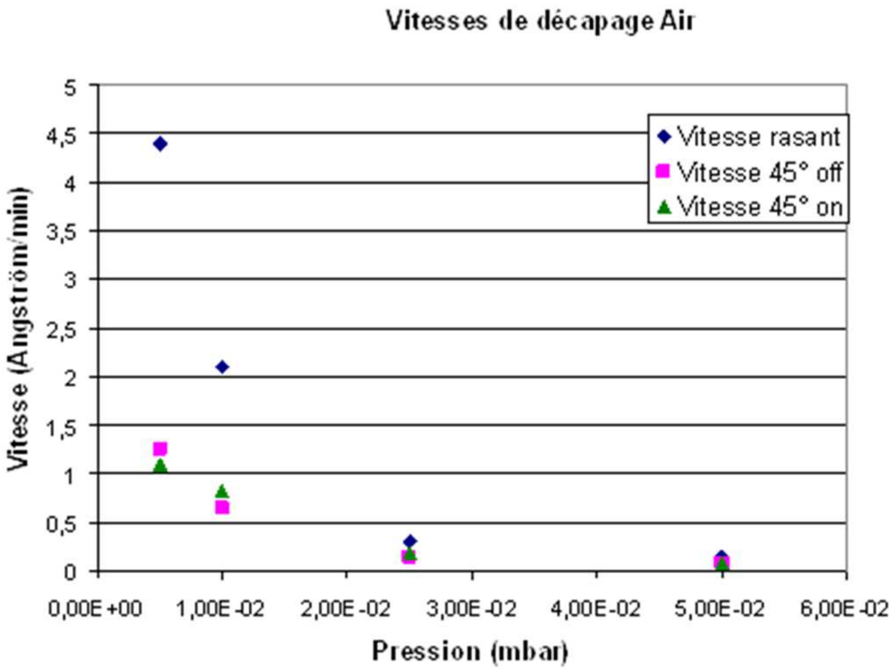
GV10X
plasma asher



Actual setup in operation at LaSu

Plasma-source GV10X: results

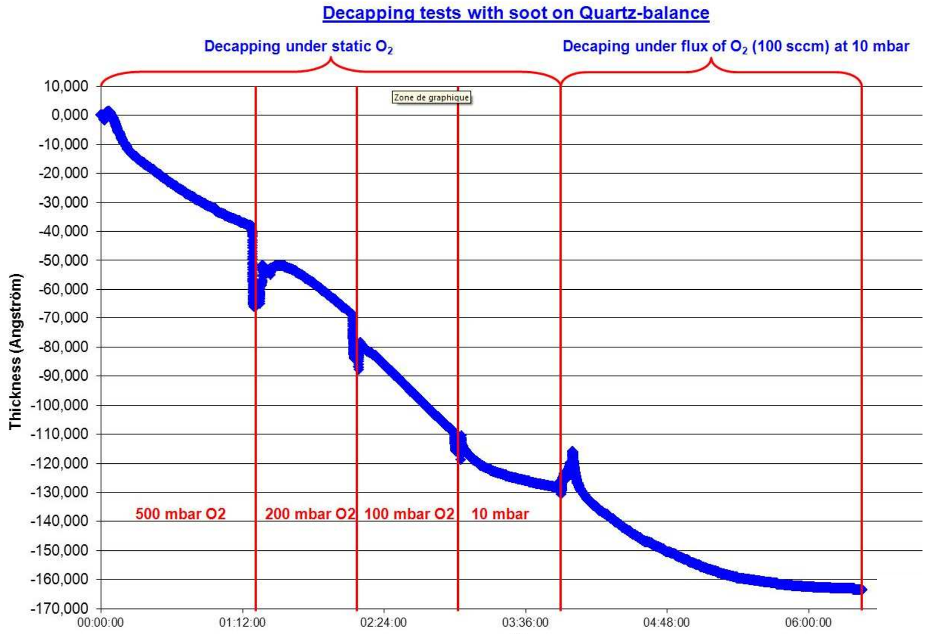




Observations :

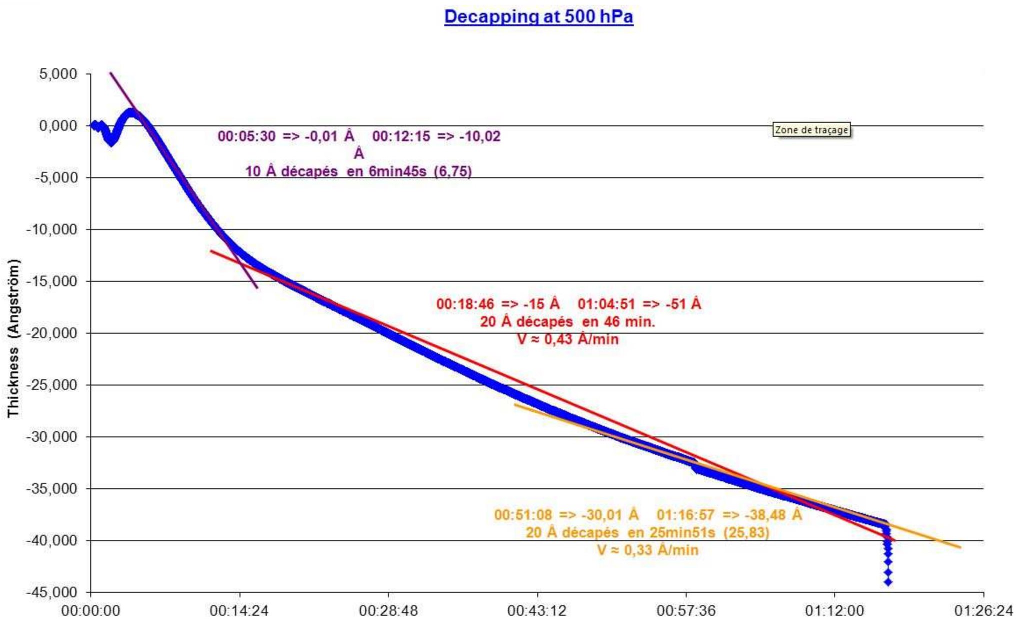
- **Decapping is systematically more efficient in grazing incidence**
- **Low pressure (~1*10^{-2} hPa) is better**
- **No large difference between Ar/O_2 et Air as reactants**
- **Decapping speed up to ~4,5 Ang/min**

UV-lamp cleaning : results



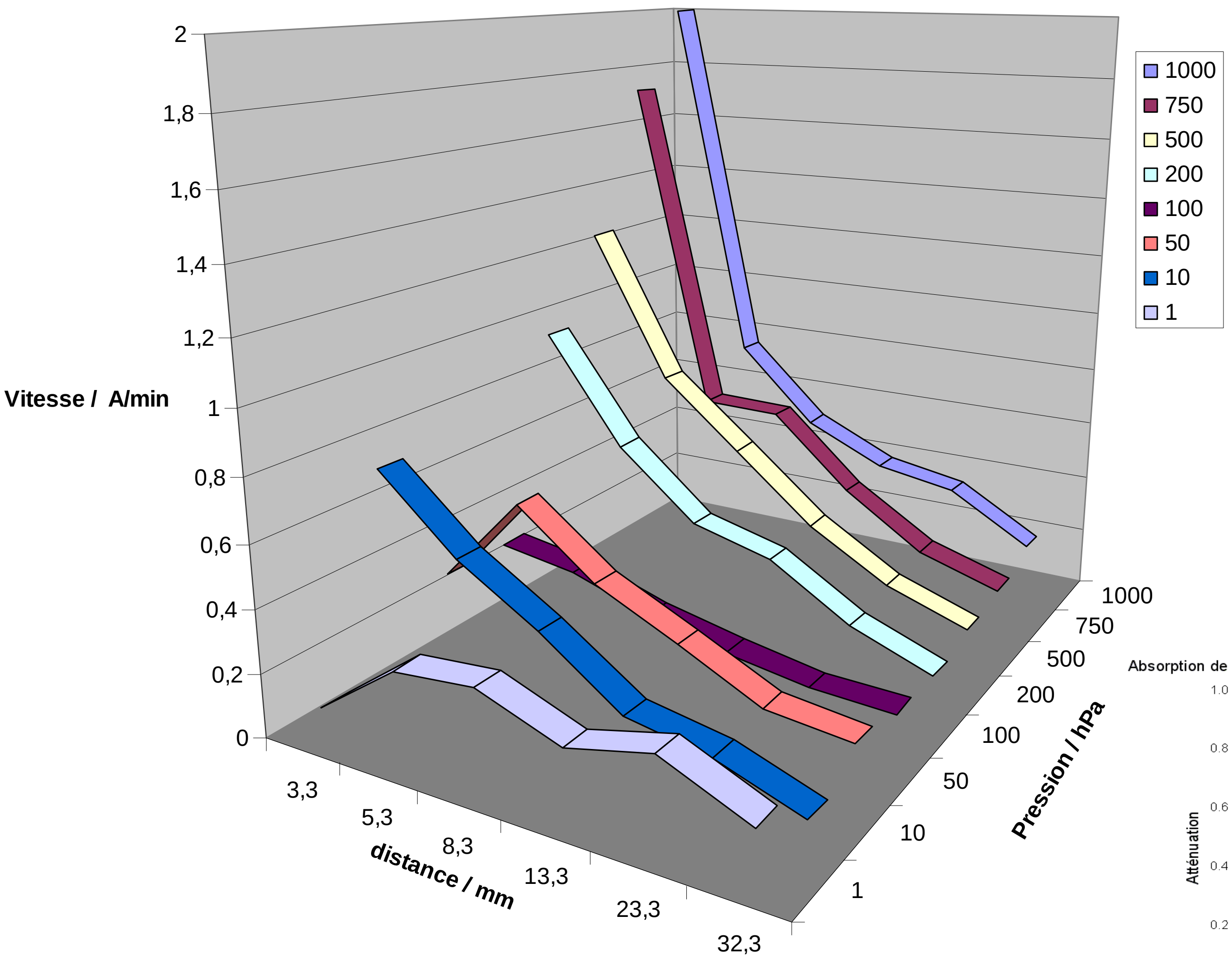
Quartz-balance measurements raw data :

- At the beginning of each measurement a *thickness increase* is observed
- the decapping speed is not constant : take average in order to obtain realistic values



UV-lamp cleaning : results

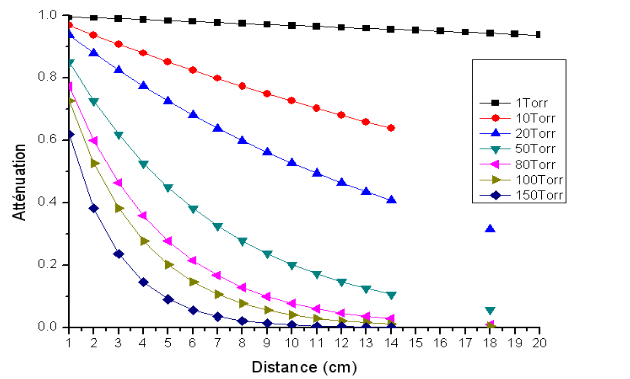
Vitesse de décapage en fonction de la pression et de la distance



Decapping speed as a function of pressure *and* lamp to sample distance:

- ✓ The smaller the distance, the better, 5mm or less is recommended
- ✓ Macroscopic pressure close to atmosphere gives 10X better results

Absorption de la raie à 185 nm en fonction de la pression d'oxygène O2 et de la distance

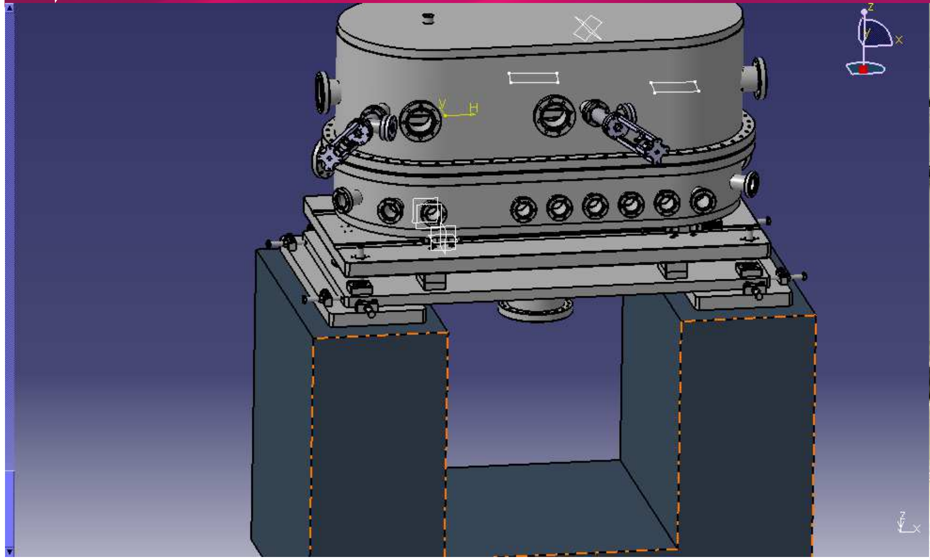


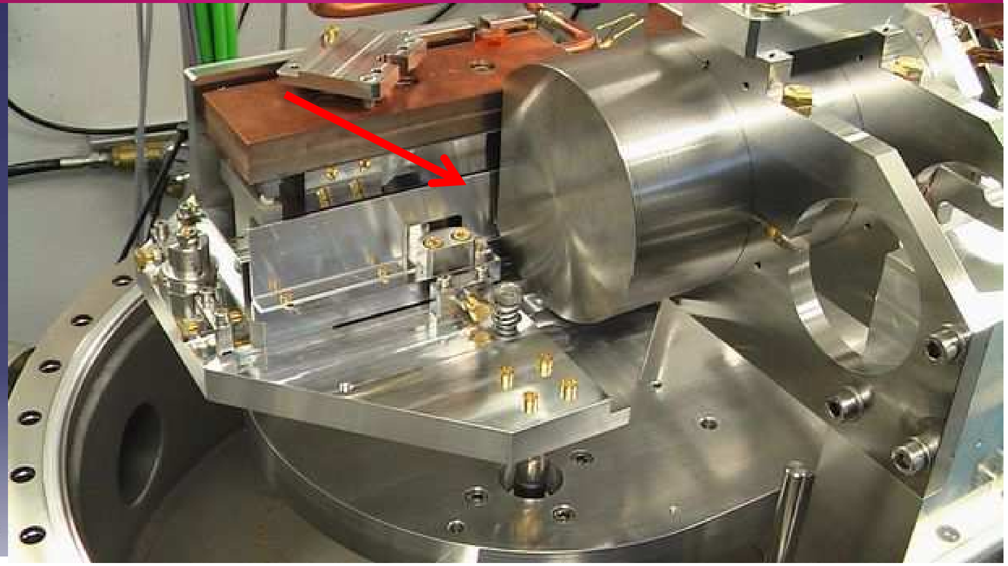
UV-lamp and Plasma cleaning : results

- **Several materials have been tested/treated**
  - **UV-cleaning and Plasma is OK for all optical components except CaF_2 : no degradation of surface quality has been observed (roughness).**
  - **Typical materials used in UHV-optics chamber are compatible with both processes, except: Ag, Cu, certain alloys of Al.**

- **Geometry / accessibility of the optics in the respective UHV-chambers plays the crucial role to be able to clean in-situ.**

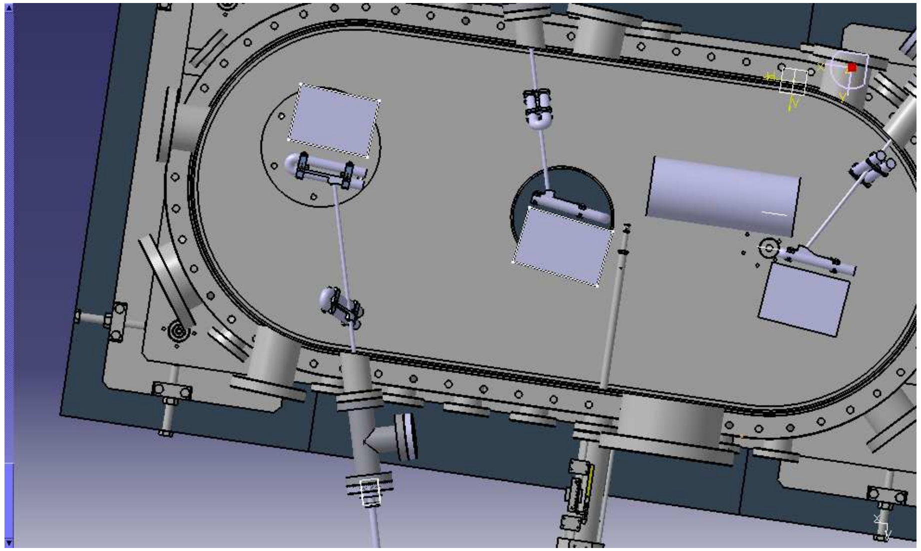
In-situ System for TEMPO

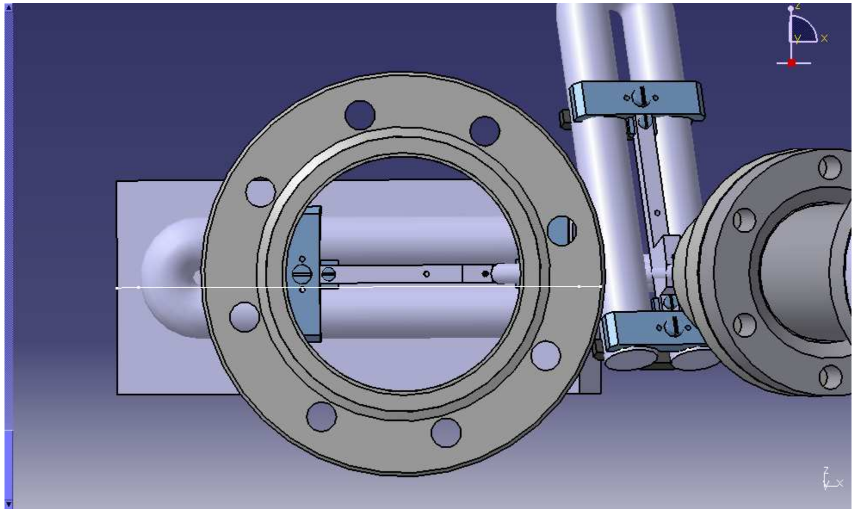




Tempo-M1 Chamber opened: Carbon trace on optic

M1 Chamber of TEMPO: Front and top-view

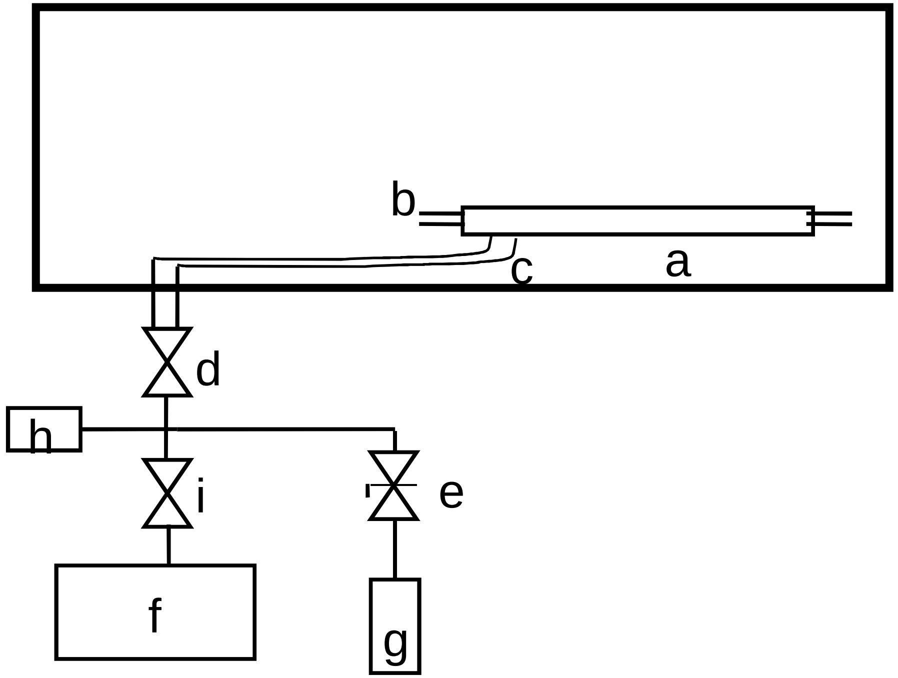




View through viewport: system in place / retracted

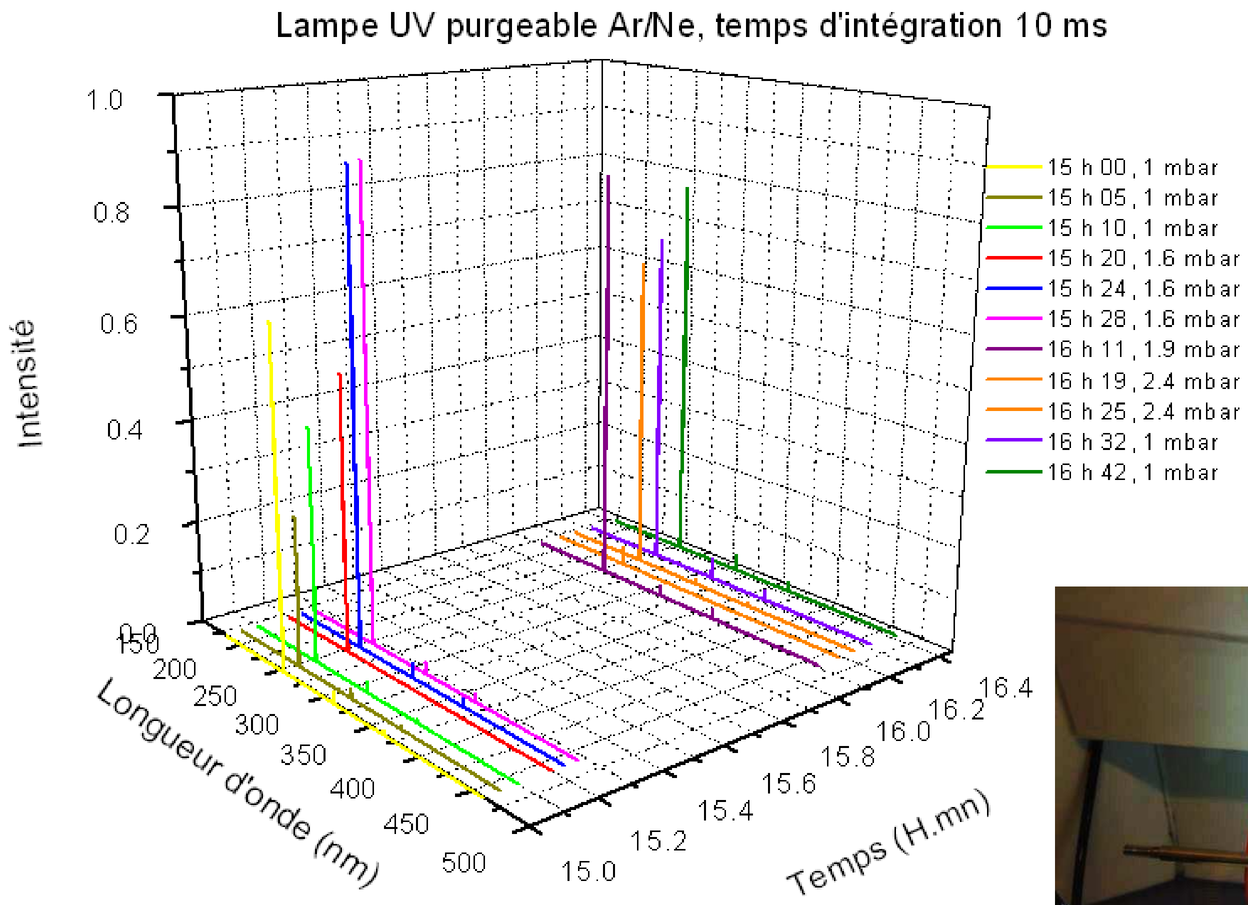
Backdraw of the TEMPO-system:

- ❖ UV-lamp use on windowless synchrotron beamlines such as TEMPO and on other soft x-ray lines is incompatible with ring operation safety due to the fact that noble gases are contained in the lamps and are not pumped by the rings' pumping systems: A month-long downtime of the complete synchrotron could be the consequence of a broken lamp.
- ✓ Christian Herbeaux (SOLEIL) introduced the **idea** to evacuate the UV-lamps when not in operation
- ✓ Goal: show that this lamp can be filled and refilled several tens of times before failure



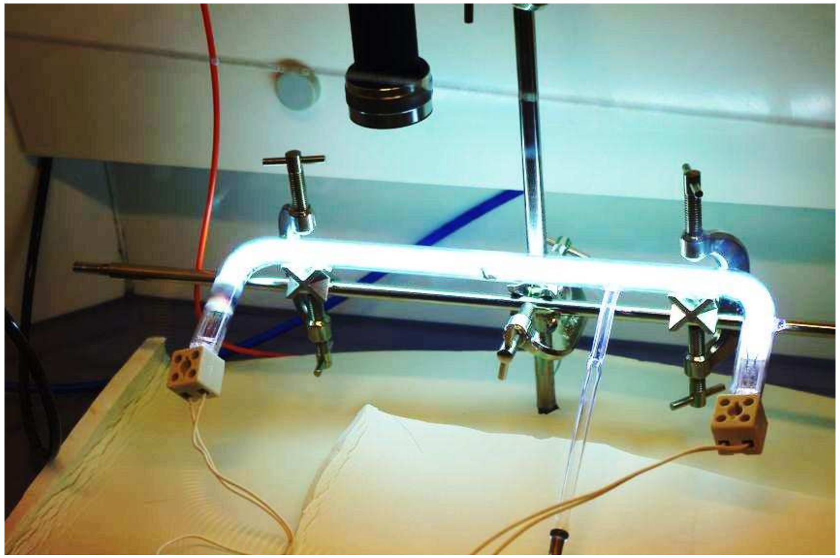
a : UV-lamp
b : eletrical feedthrough
c : capillary with quartz-metal transition
d : all metal valve with lock
e : Leakvalve
f : Pumping : primary and turbo
g : gas reserve for lamp refill
h : gauge
i : isolation valve for pumping unit

Purgeable UV-lamps project



Experimental results:

- Difficult to ignite the lamp
- Lamp life is much too short (a few times of refill before failure)



Méthode Plasma ex-situ

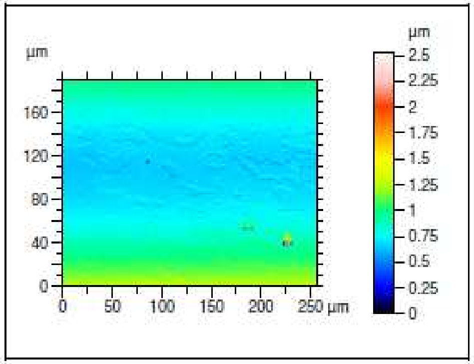
La Réacteur Plasma *DIENER* (Atto) effectue un traitement sur un mirroir cylindrique avec une couche métallique au Laboratoire de Surfaces de SOLEIL le 7 Octobre 2015.

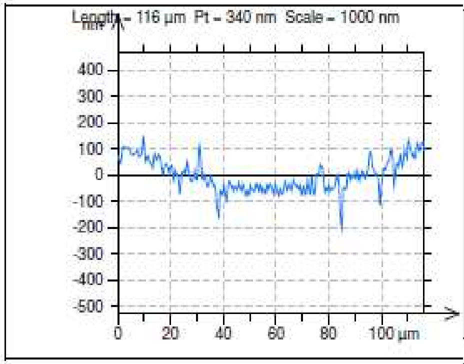
La procédure dure ~10 min.

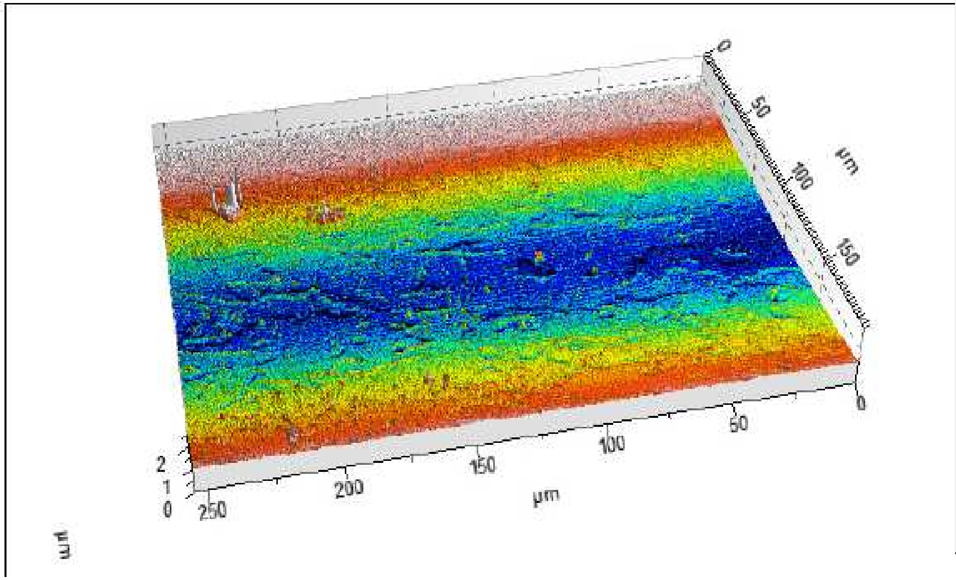
Le gaz est un melange 80% O_2 et 20%Ar, @p=0,44hPa

Analyse Méthode Plasma ex-situ

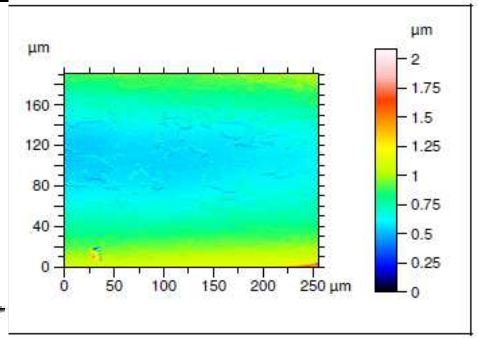
Avant traitement 10 min.

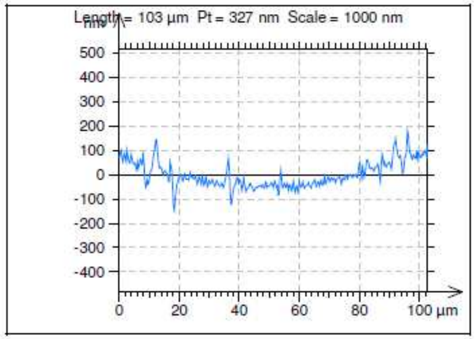


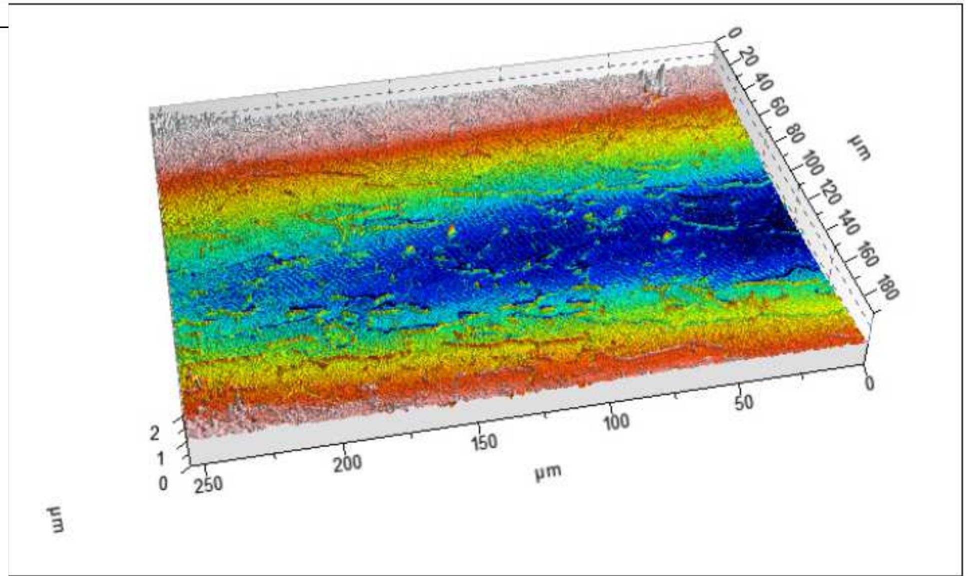




Après traitement 10 min.







Observation: L'interférométrie à lumière blanche (WLI) montre une ruguosité pareille, le métal n'a pas été modifié.

Conclusions and Perspectives

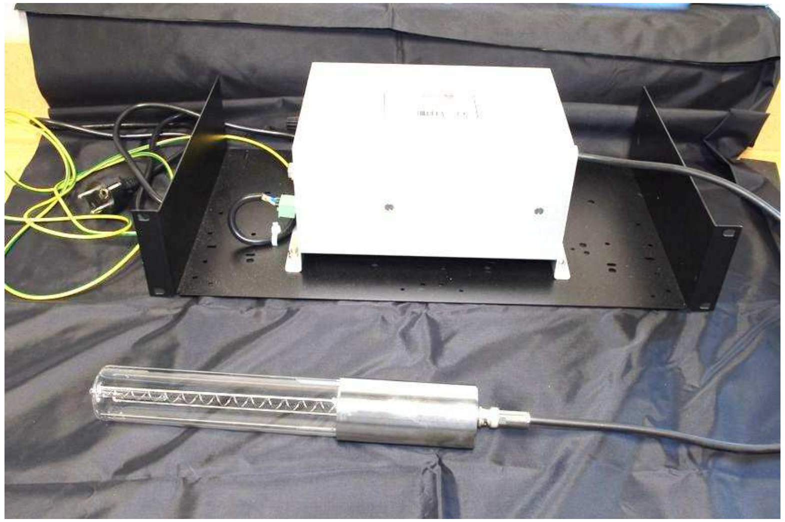
- **Two independent ways to clean optics used in UHV**
  - Plasma-cleaning is standard for certain beamlines
  - UV-cleaning speed as a function of all relevant parameters can be measured and employed semi in-situ on soft x-ray lines
  - + Plasma: ex-situ with commercial device

- *Complementarily*, TEMPO found a way to **keep optics clean** by permanent O_2 treatment (10^{-8}mbar).

- Purgeable UV-lamps do not provide the necessary rerliability for an all *in-situ* solution to SOLEIL beamlines

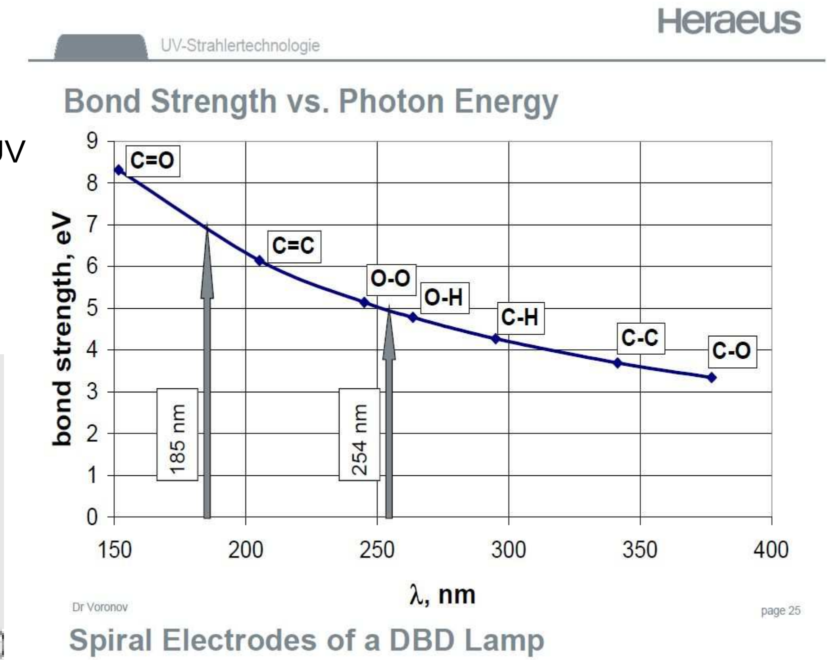
MERCI !

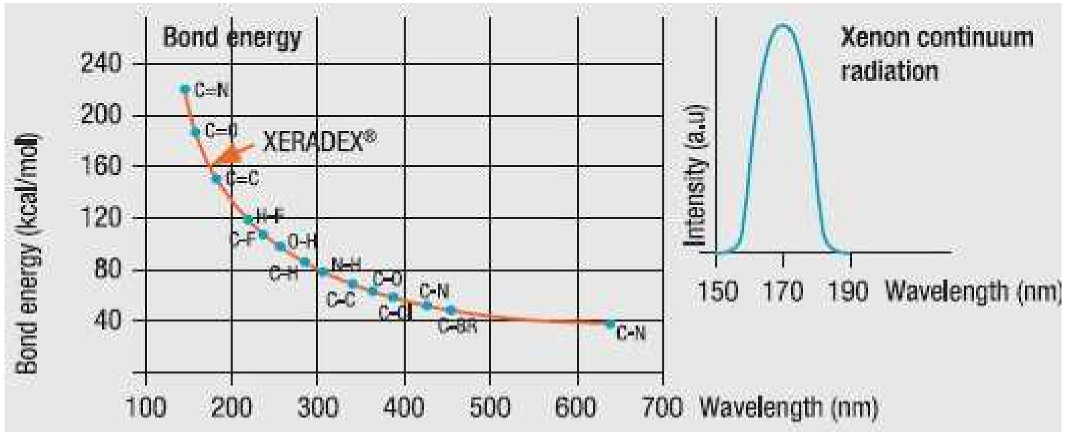
New Approach at SOLEIL: Excimer Lamp



If **Xenon** is the fill gas – as in the XERADEX® lamps – and if a special pulsed voltage is applied across the electrodes, unstable xenon excimer molecules (Xe2*) are formed from the xenon atoms. These molecules dissociate by emitting **VUV radiation at 172 nm**.

Right: LP Mercury UV Lamp





New EXCIMER Lamp at the SOLEIL Surface Laboratory: next steps (2013):

- **test lamp under air**
- **test lamp in vacuum test chamber with QB.**