



Optimization of non-evaporable getter coating for accelerator beam pipe

O.B. Malyshev, R. Valizadeh,
B.T. Hogan, R.M.A. Jones, C. Naran,
M. Pendleton and A. Hannah

*ASTeC Vacuum Science Group,
STFC Daresbury Laboratory, UK*





Outlook

- Introduction
 - What is NEG coatings
 - Sticking probability and capacity for different NEG coatings
- Pumping property
 - Film deposition
 - Surface analysis
 - NEG activation procedure
 - Pumping properties measurements
 - Activation temperature
- Desorption properties
 - How to reduce ESD
 - What film is needed
 - What achieved
- Conclusions



What are usual considerations for vacuum

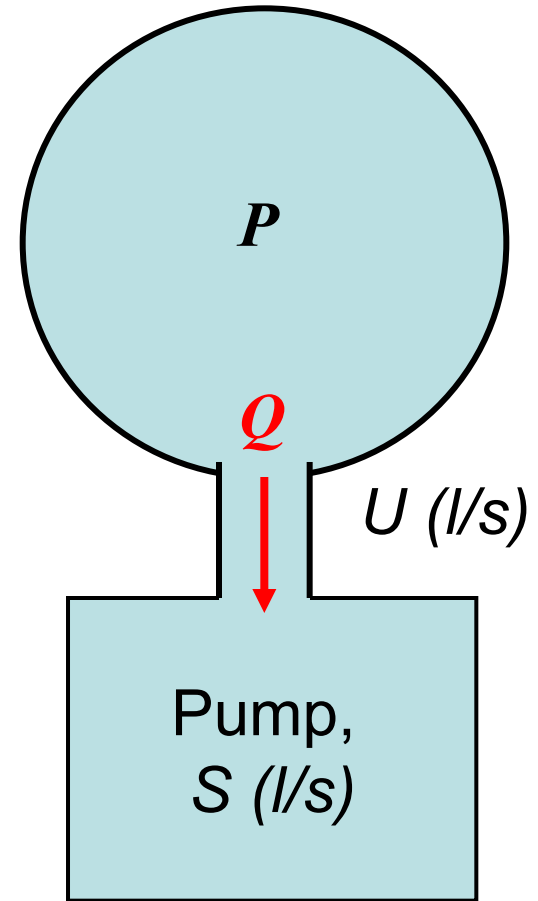
Required pressure P is defined by gas desorption Q in the vessel and effective pumping speed S_{eff}

In a simple case it is

$$P = \frac{Q}{S_{eff}} = Q \left(\frac{1}{S} + \frac{1}{U} \right)$$

$$Q = qA + \eta_{\gamma}\Gamma + \eta_e I_e + \eta_{ion} I_{ion}$$

Thermal, photon, electron and ion
stimulated desorption

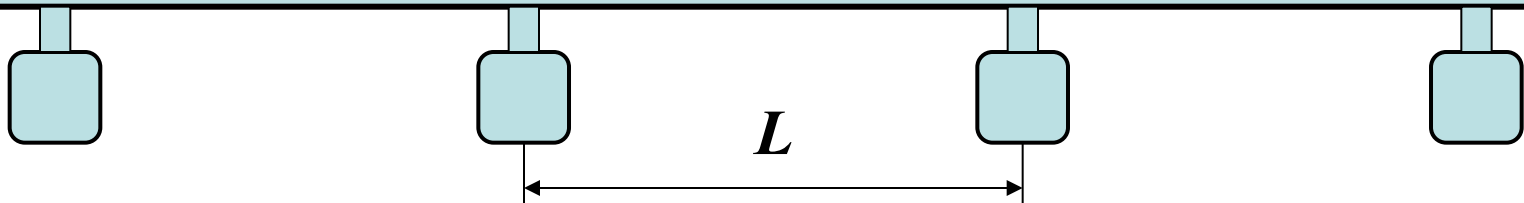
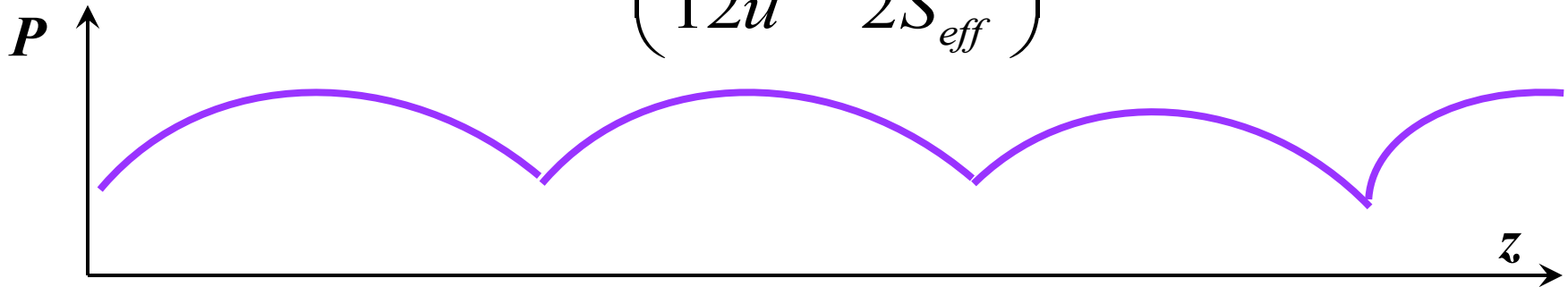




Usual accelerator vacuum chamber

- Long tube with length $L \gg a$, where a - transversal dimension
- Average pressure depends on vacuum conductance $u(L, a)$ of the beam vacuum chamber

$$\langle P \rangle = qL \left(\frac{L}{12u} + \frac{1}{2S_{eff}} \right) k_B T$$



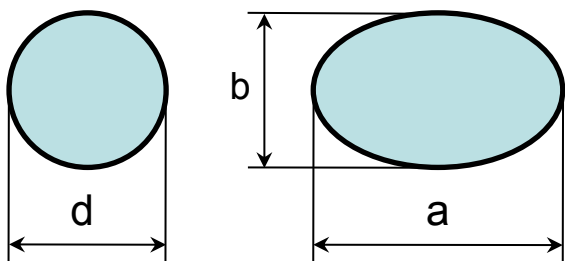


Vacuum chamber cross sections

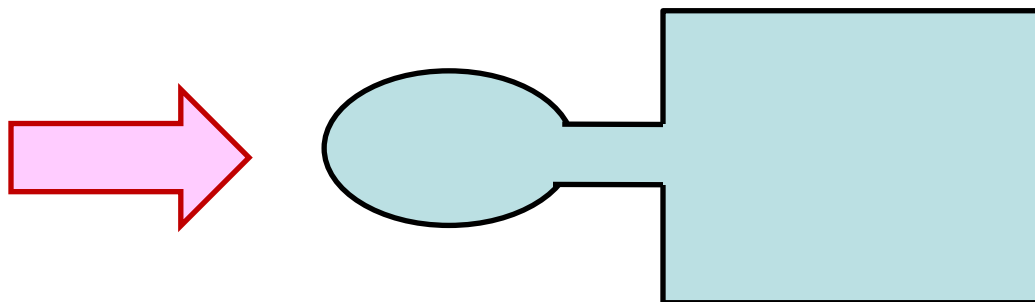
Beam pipe

Circular or elliptical

$$4 \text{ mm} \leq d, a, b \leq 200 \text{ mm}$$

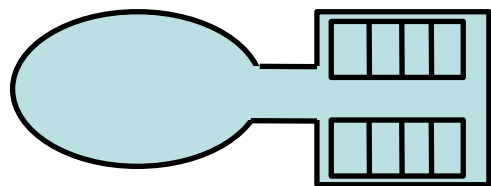


Vacuum chamber with an antechamber
for larger vacuum conductance, U

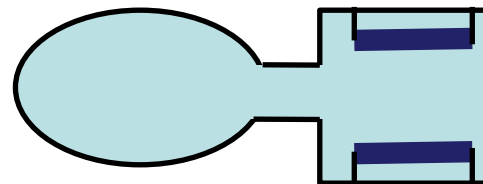
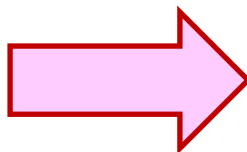


Distributed pumping

In dipole magnetic field



With NEG strips
(LEP in CERN)





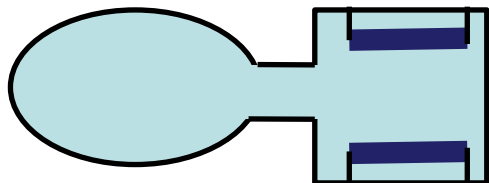
Two concepts of the ideal vacuum chamber

Traditional:

- surface which outgasses as little as possible ('nil' ideally)
- surface which **does not pump** otherwise that surface is contaminated over time

Results in

- Surface cleaning, conditioning, coatings
- Vacuum firing, *ex-situ* baling
- Baking *in-situ* to up to **300°C**
- Separate pumps

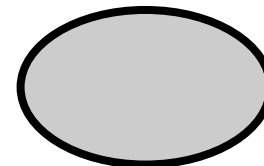


'New' (C. Benvenuti, CERN, ~1998):

- surface which outgasses as little as possible ('nil' ideally)
- a surface which **does pump**, however, will not be contaminated due to a very low outgassing rate

Results in

- NEG coated surface
- There should be no un-coated parts
- Activating (baking) *in-situ* at **150-180°C**
- Small pumps for C_xH_y and noble gases



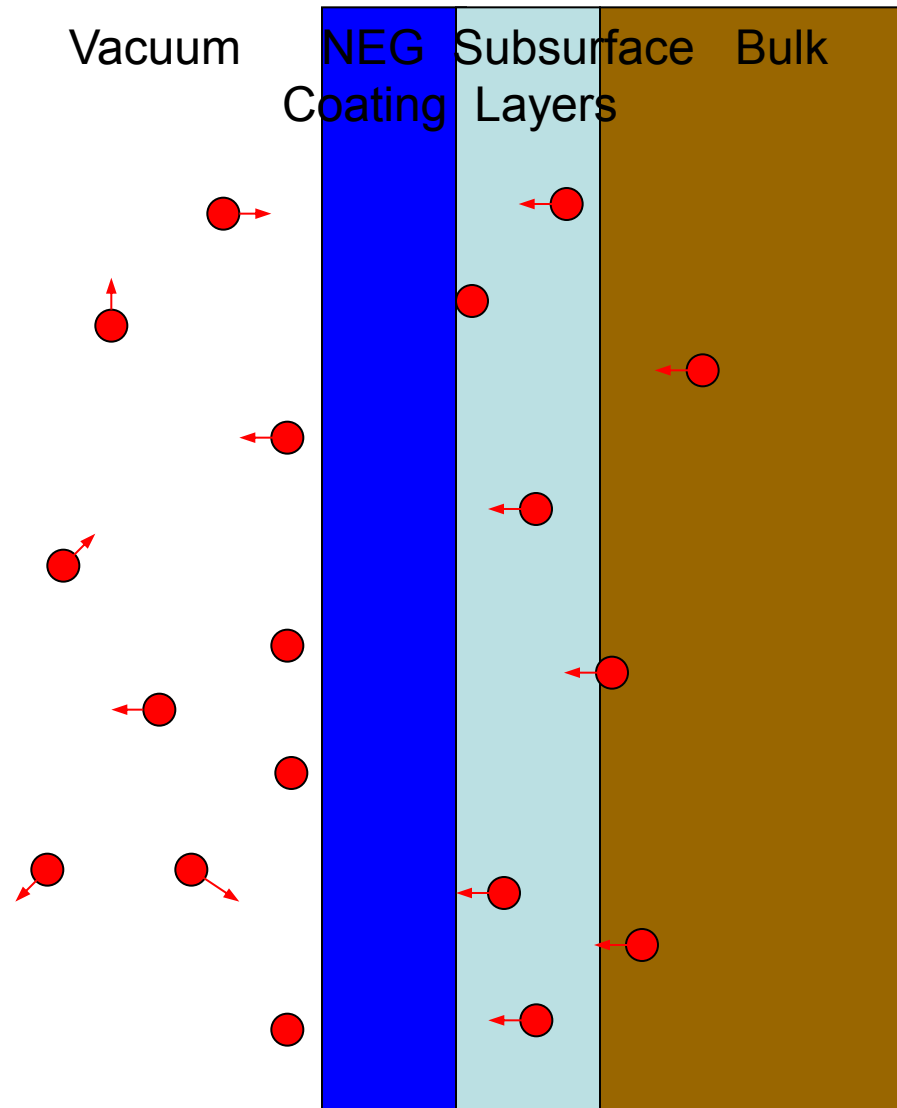


What the NEG coating does

- **Reduces gas desorption:**
 - **A pure metal film ~1- μm thick without contaminants.**
 - **A barrier for molecules from the bulk of vacuum chamber.**
- **Increases distributed pumping speed, S :**
 - **A sorbing surface on whole vacuum chamber surface**

$$S = \alpha \cdot A \cdot v / 4;$$

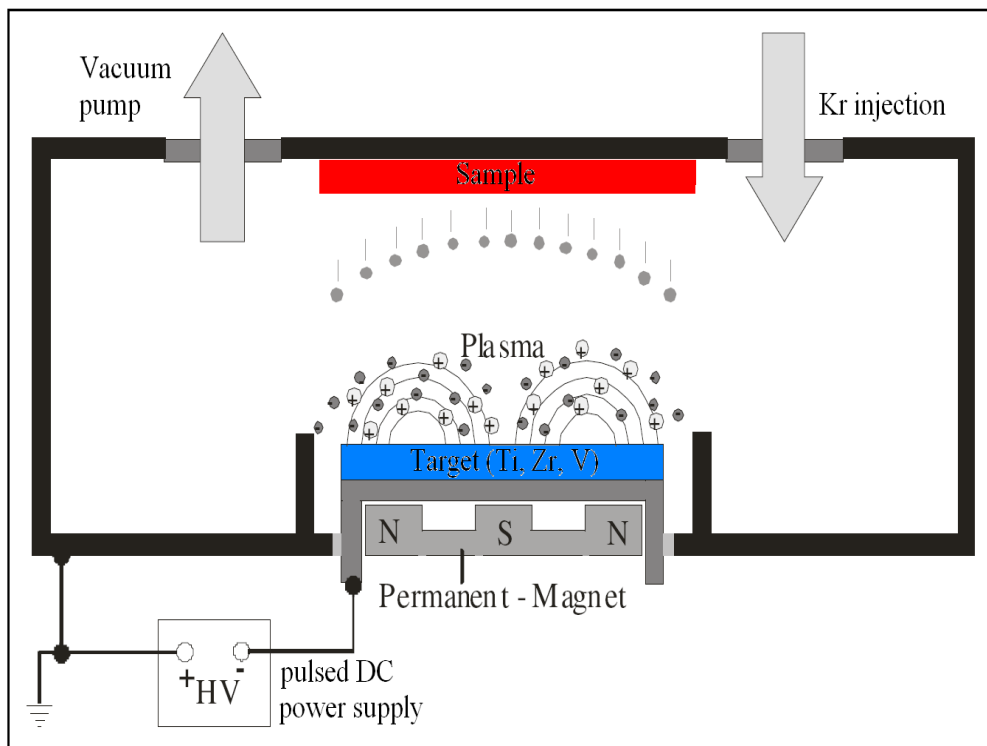
where α – sticking probability,
 A – surface area,
 v – mean molecular velocity



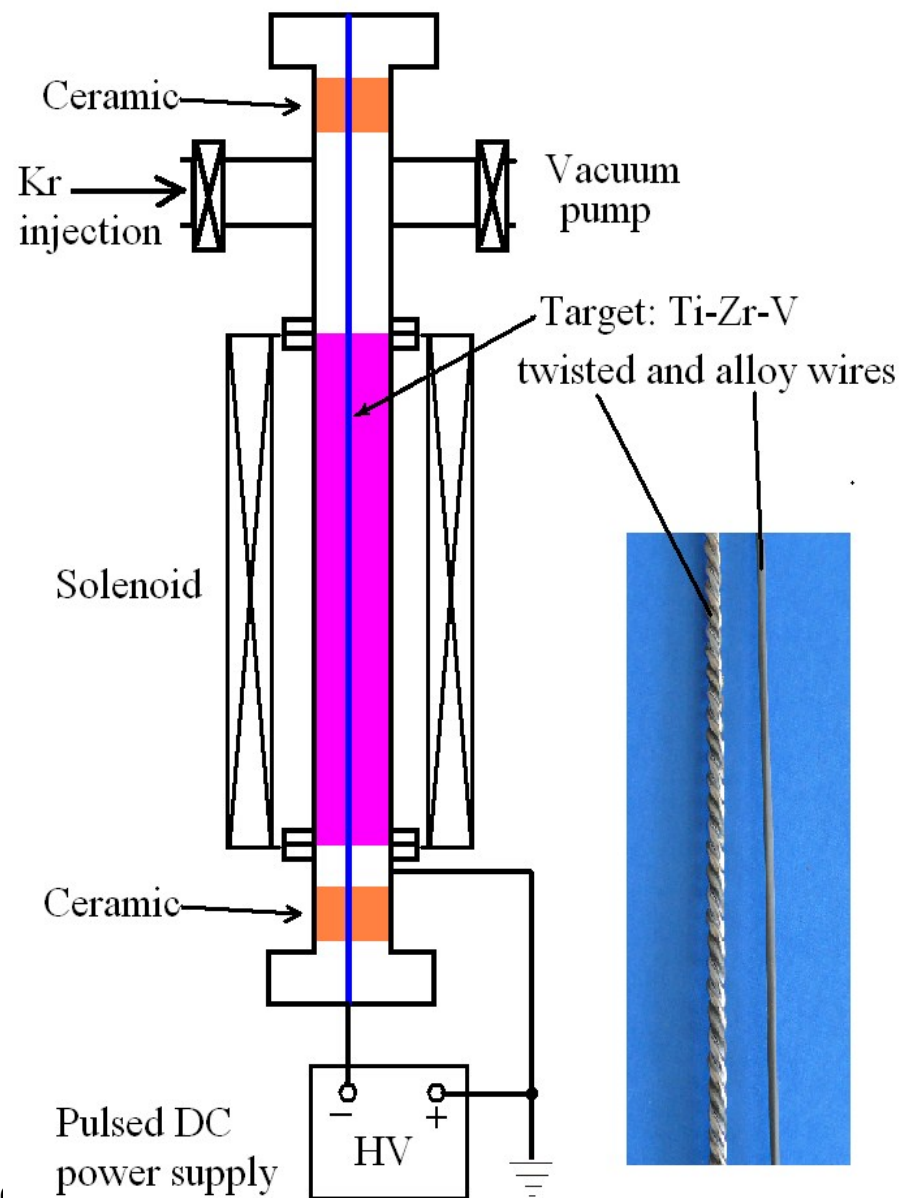


Deposition method

Planar magnetron deposition

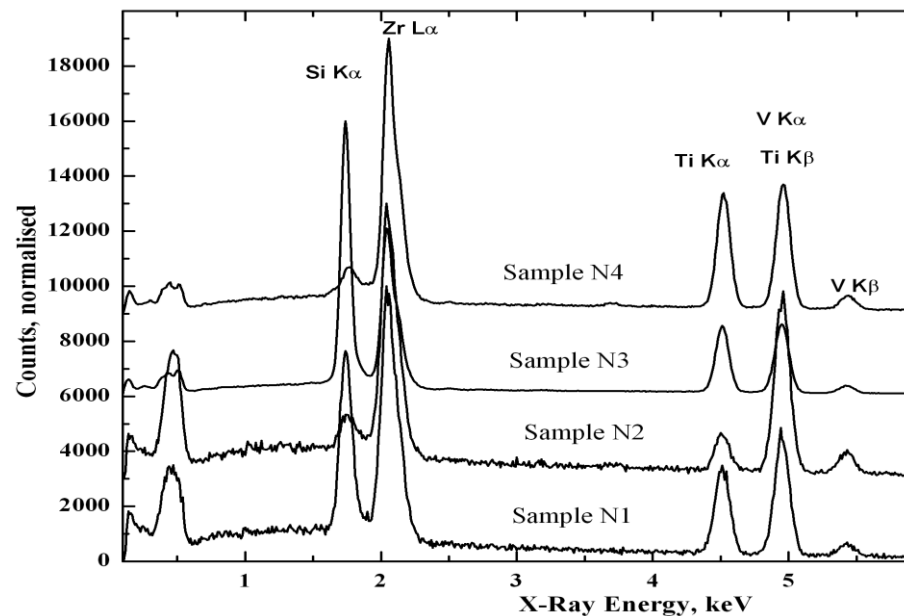
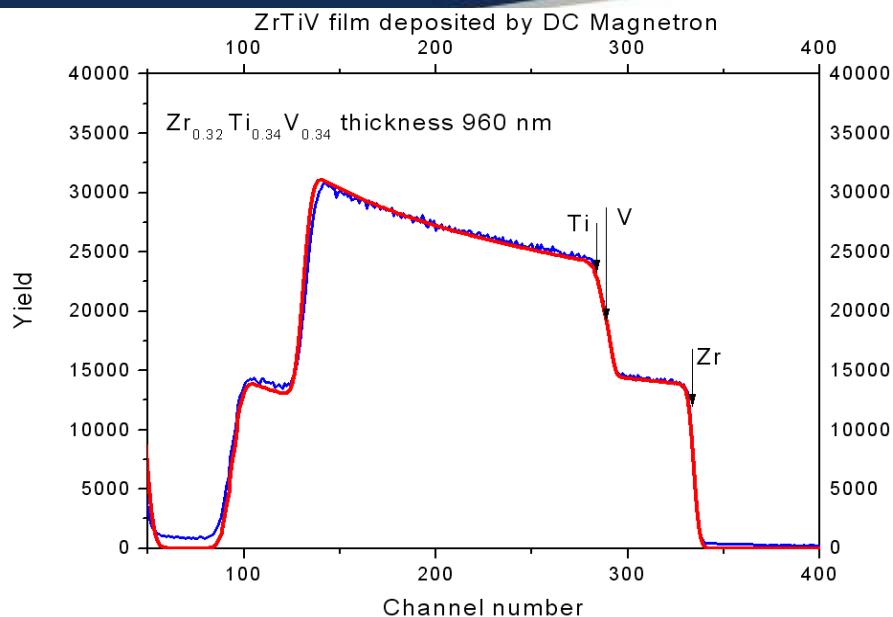


Cylindrical magnetron deposition





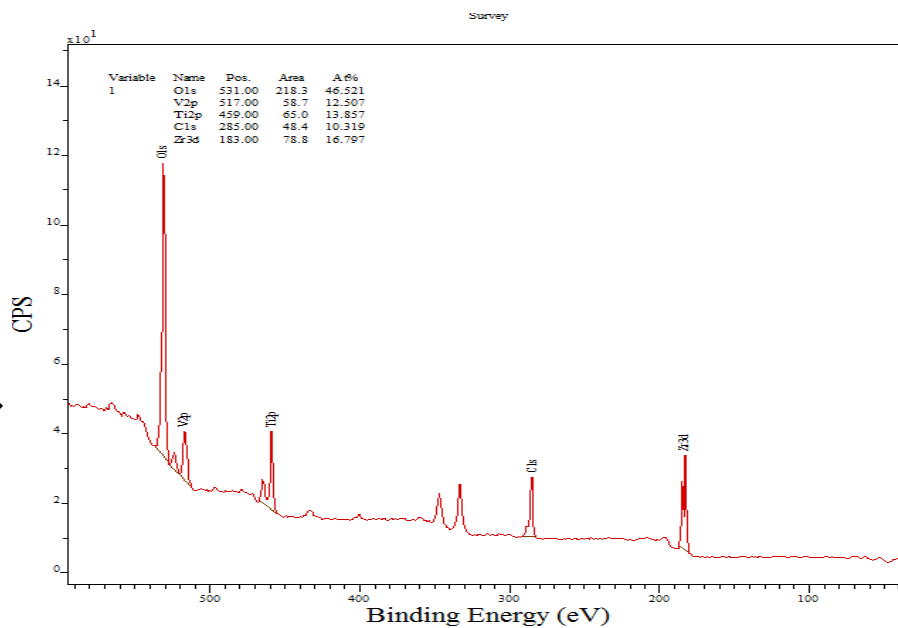
Bulk and Surface Composition



RBS (film
composition in bulk)

XPS (surface
composition
and chemical
bonding)

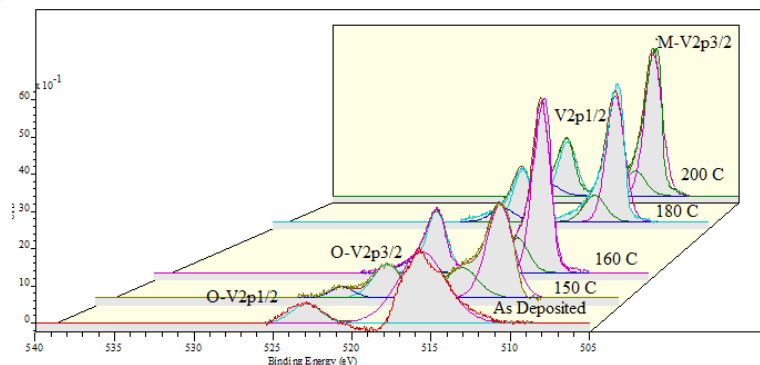
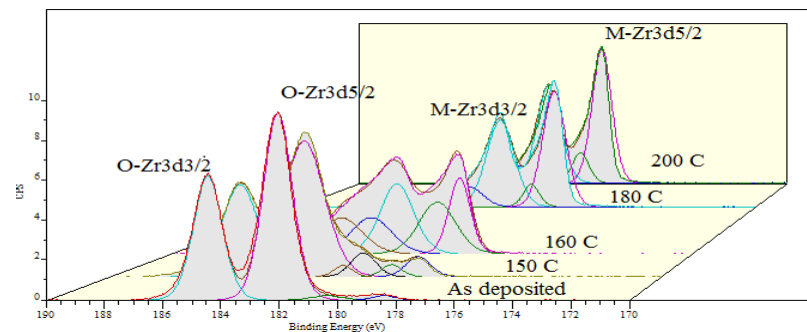
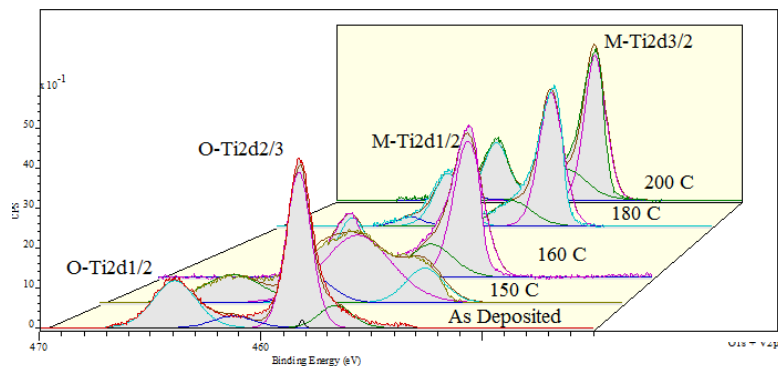
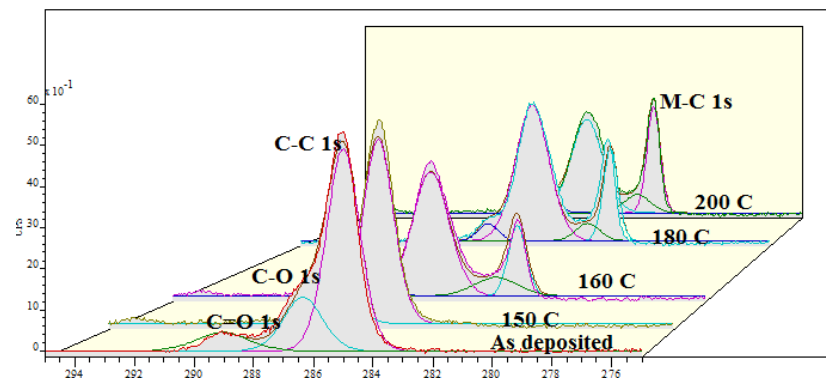
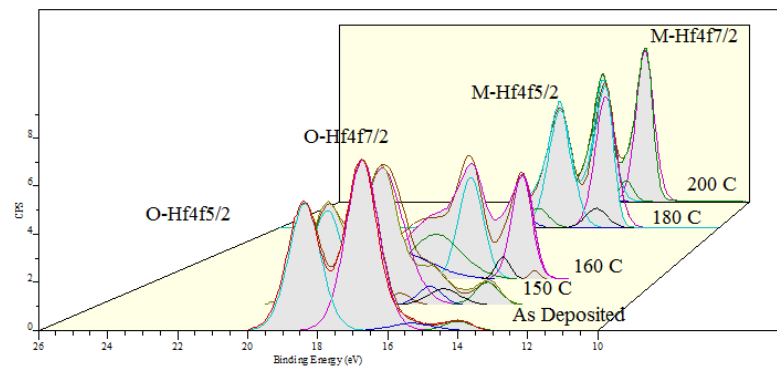
Oleg Malyshev



EDX
(determination of
film composition)



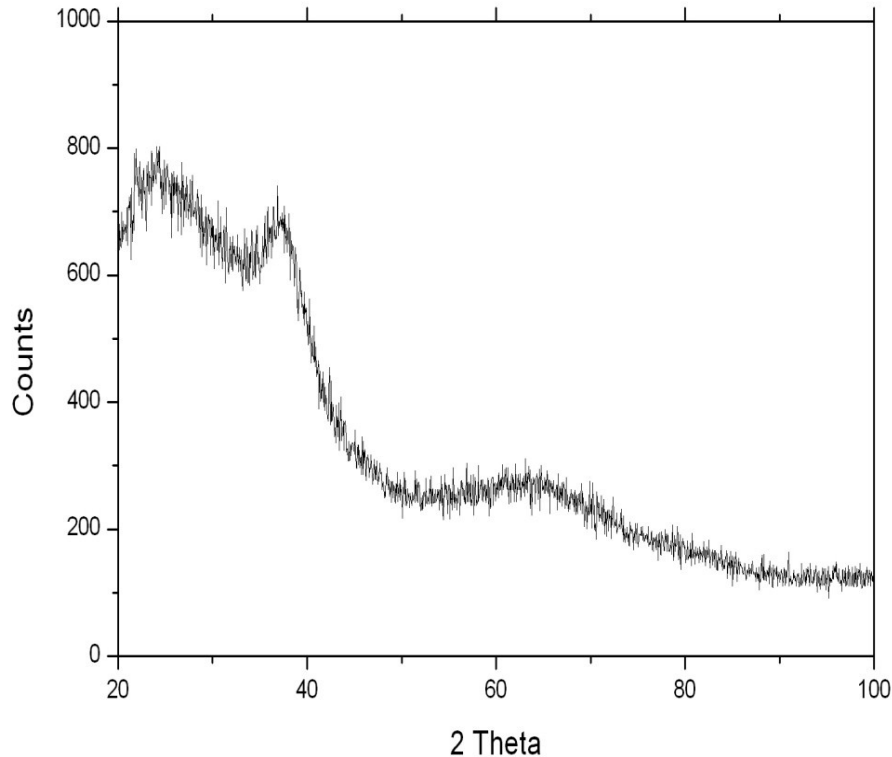
Region scan of XPS core levels of Ti, Zr, C, Hf and V of a Ti-Zr-V-Hf film (surface composition and chemical bounding)



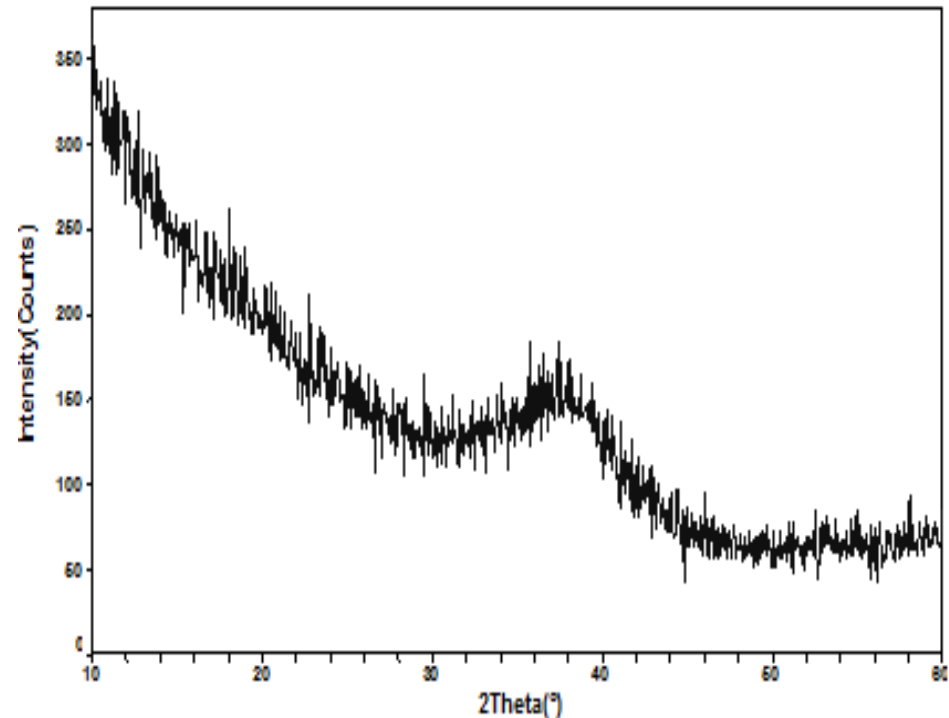


XRD of Film deposited from Ternary and Quaternary alloy wire as target.

TiZrV Film deposited on Si by cylindrical magnetron using Alloy wire



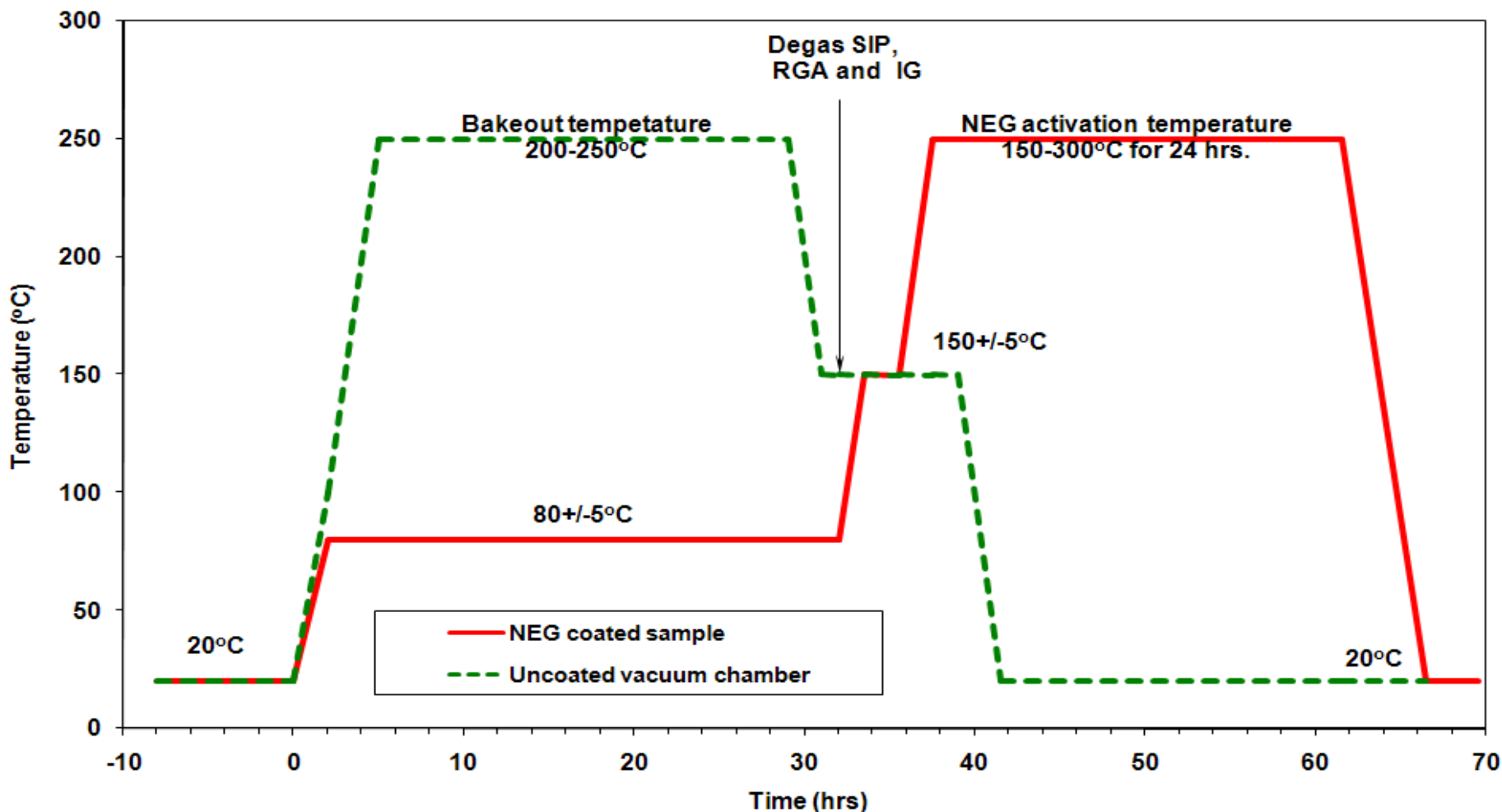
TiZrVHf film deposited on Si by cylindrical magnetron using Alloy wire



In Both cases there is only one broad peak near $2\theta = 36.8^\circ$
The film is nearly amorphous.



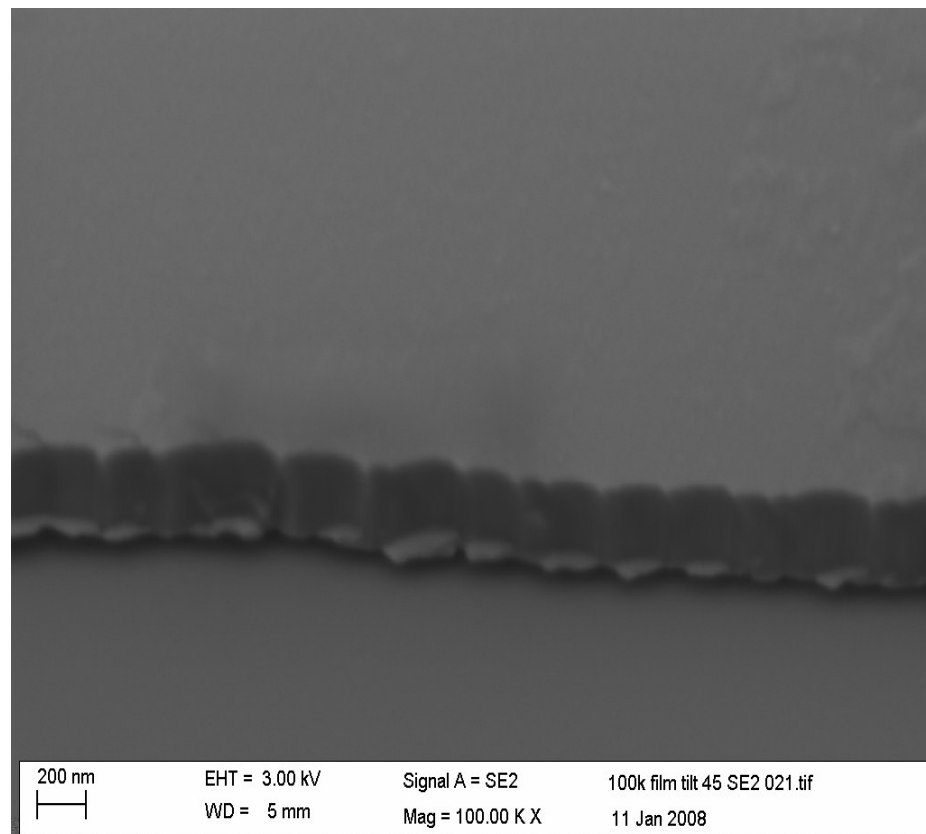
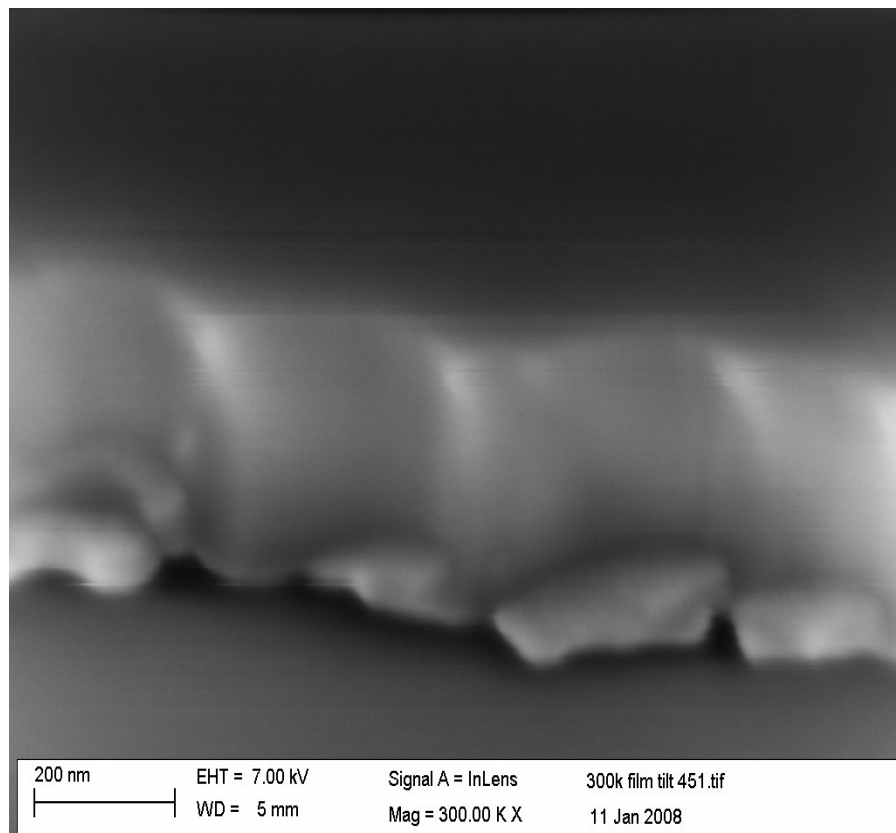
ASTeC activation procedure



O.B. Malyshev, K.J. Middleman, J.S. Colligon and R. Valizadeh. J. Vac. Sci. Technol. A 27 (2009), p. 321.



Quaternary NEG alloy film deposited on Si test sample from twisted Ti, V, Zr, and Hf wires.



Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.12 nm/s, $T = 120^{\circ}\text{C}$.
Very glassy structure.



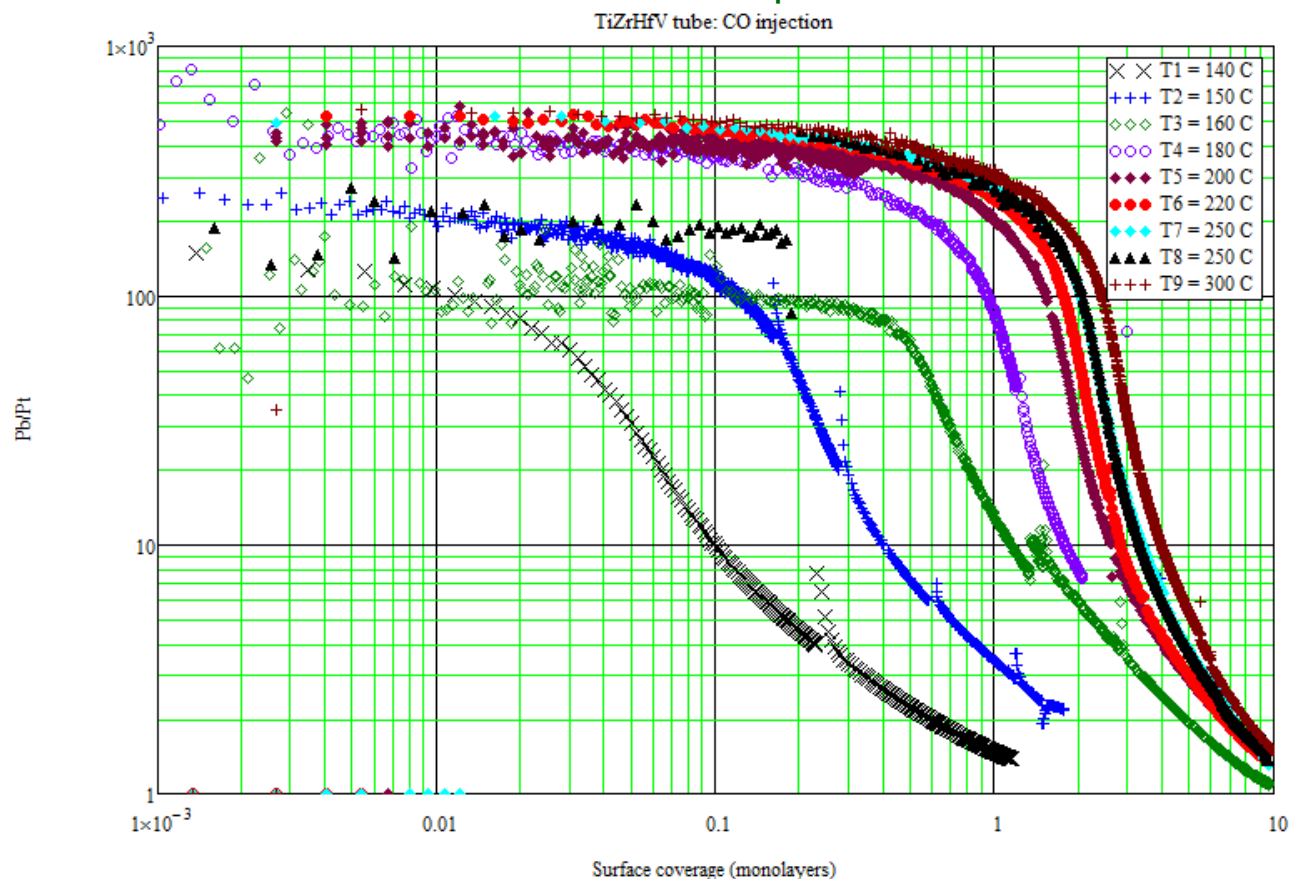
Pumping properties measurements

H₂, CO and CO₂ injection

pumping capacity taken at $P_{\text{bottom}} / P_{\text{top}} = 10$

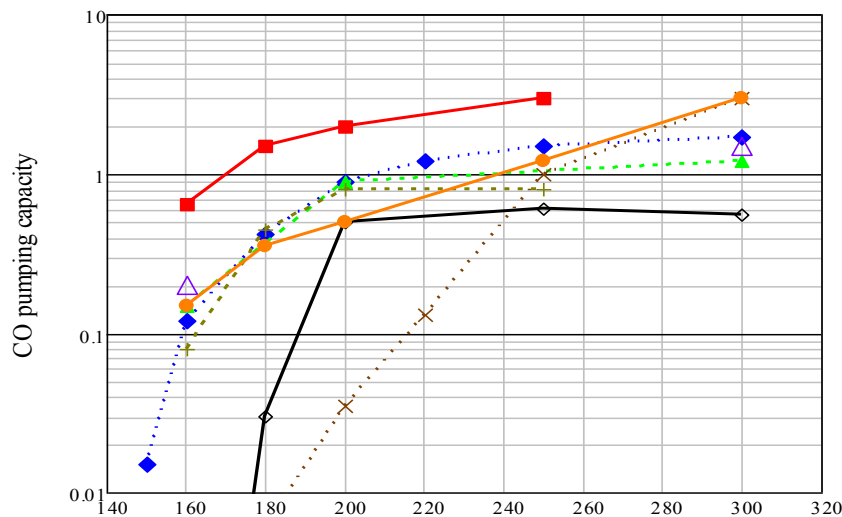
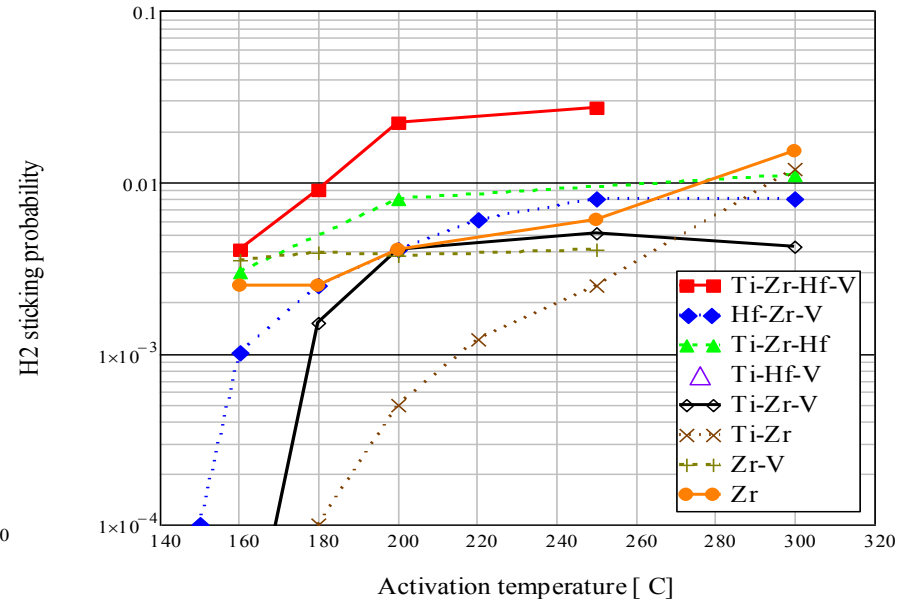
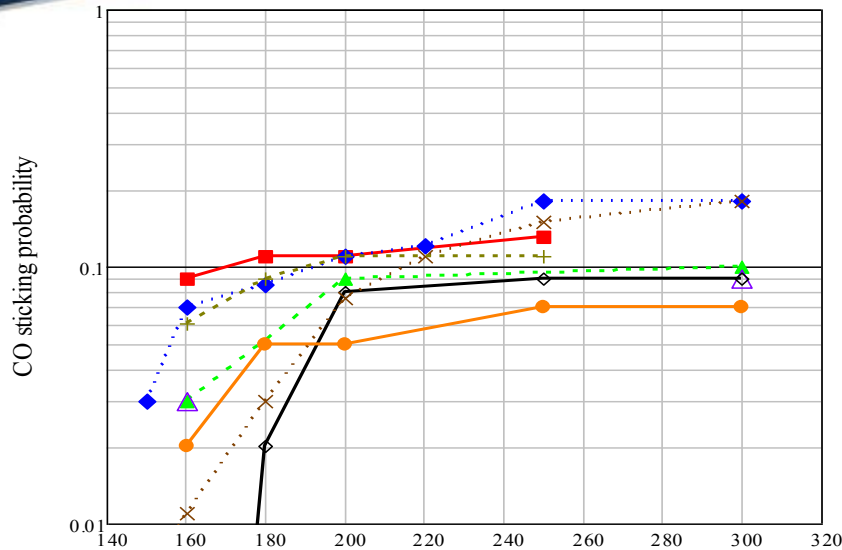
Initial sticking
probability can be
obtained from
pressure ratio

$P_{\text{bottom}} / P_{\text{top}}$





Pumping properties of some films



- **Ti-Zr-Hf-V** is the best
- **Hf-Zr-V, Ti-Zr-Hf, Ti-Hf-V** and **Zr** are comparable
- **Ti-Zr-V** is lower
- **Zr-V** (best binary alloy) has the lowest activation temperature



Pressure in the accelerator vacuum chamber

$$P \propto \frac{\eta}{\alpha}$$

where

- η - desorption yield
- α - sticking probability

- Improving the pumping properties is limited:

$$\alpha \leq 1.$$

$$0.005 < \alpha_{H_2} < 0.01$$

$$0.1 < \alpha_{CO} < 0.5$$

$$0.4 < \alpha_{CO_2} < 0.6$$

- Reducing the desorption yields η in orders of magnitude is a realistic task



Reducing the gas desorption from the NEG coatings

- Main gases in the NEG coated vacuum chamber are H_2 and CH_4
 - Only H_2 can diffuse through the NEG film under bombardment or heat
 - CH_4 is most likely created on the NEG surface from diffused H_2 and C (originally from sorbed CO and CO_2)
 - Therefore the H_2 diffusion must be suppressed
 - Where H_2 come from?



Reducing the gas desorption from the NEG coatings

Gas molecules are contained

on the NEG coating surface

after exposure to air

minimise exposure to air

inside the NEG coating

trapped during deposition

purity of discharge gas

background pressure

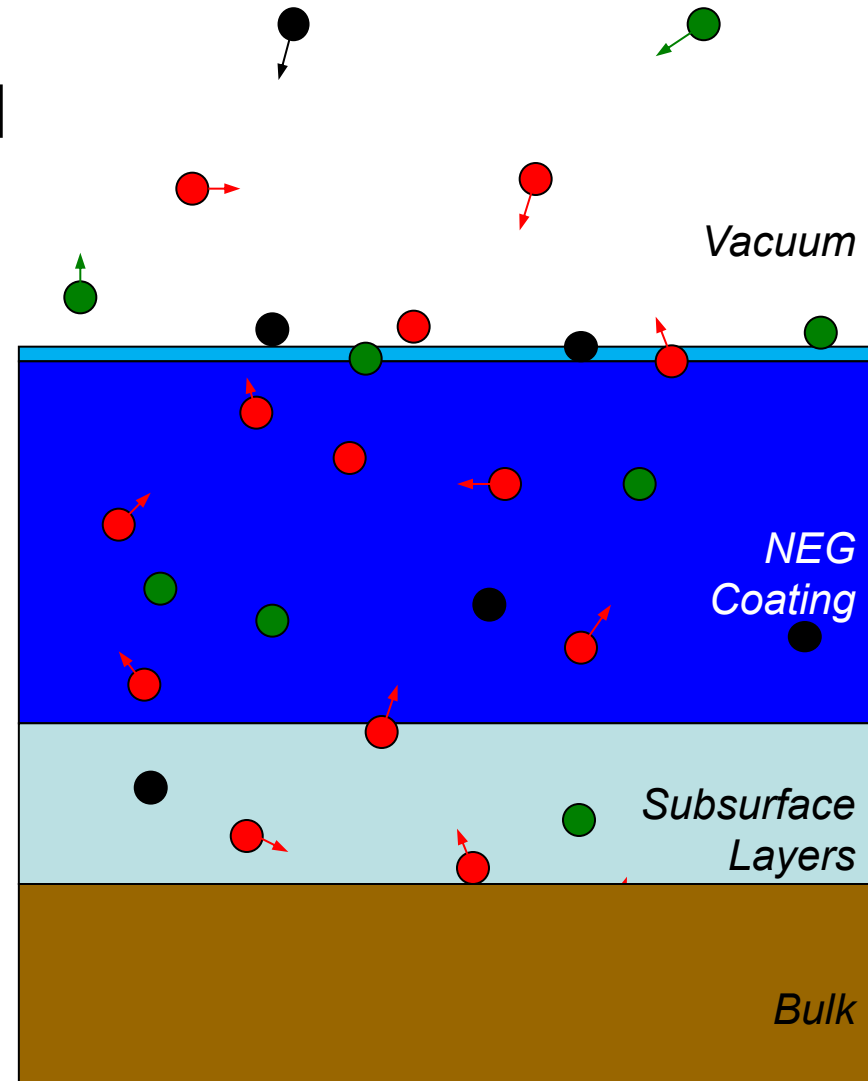
in subsurface substrate layer

substrate bakeout before NEG

deposition

in the substrate bulk

vacuum firing





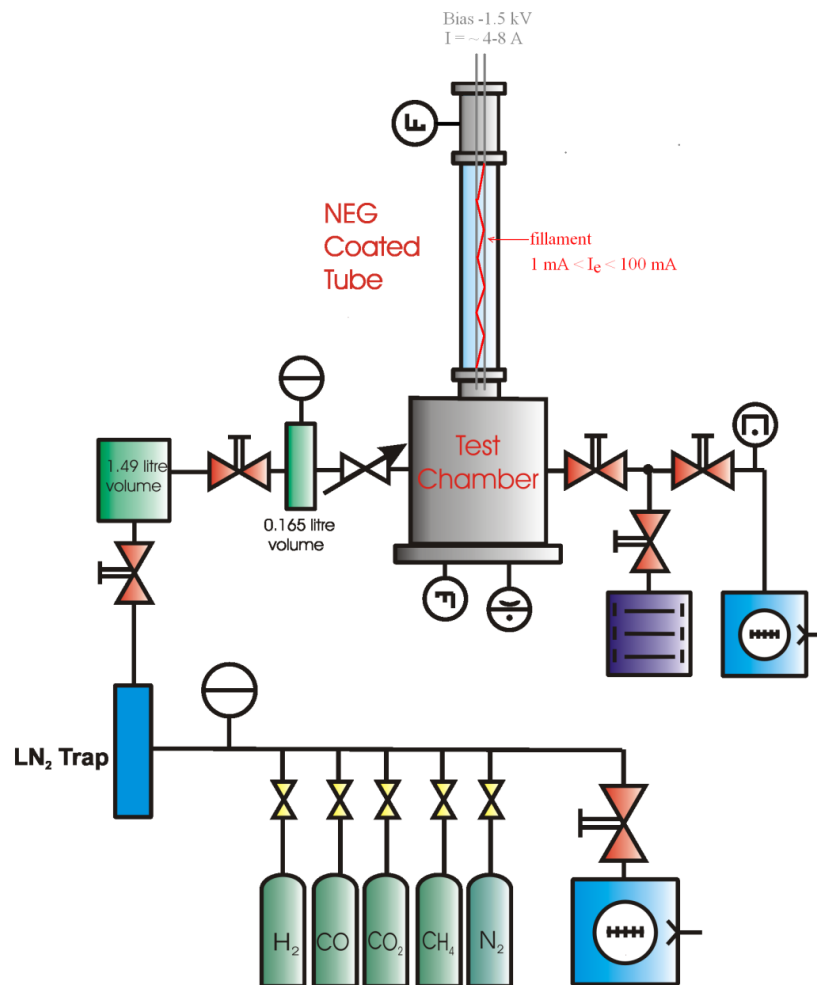
Electron stimulated desorption rig

ESD is studied as a function of

- Electron energy
- Dose
- Wall temperature (-5 to +70°C)
- Activation/bakeout temperature

Can be used for samples with:

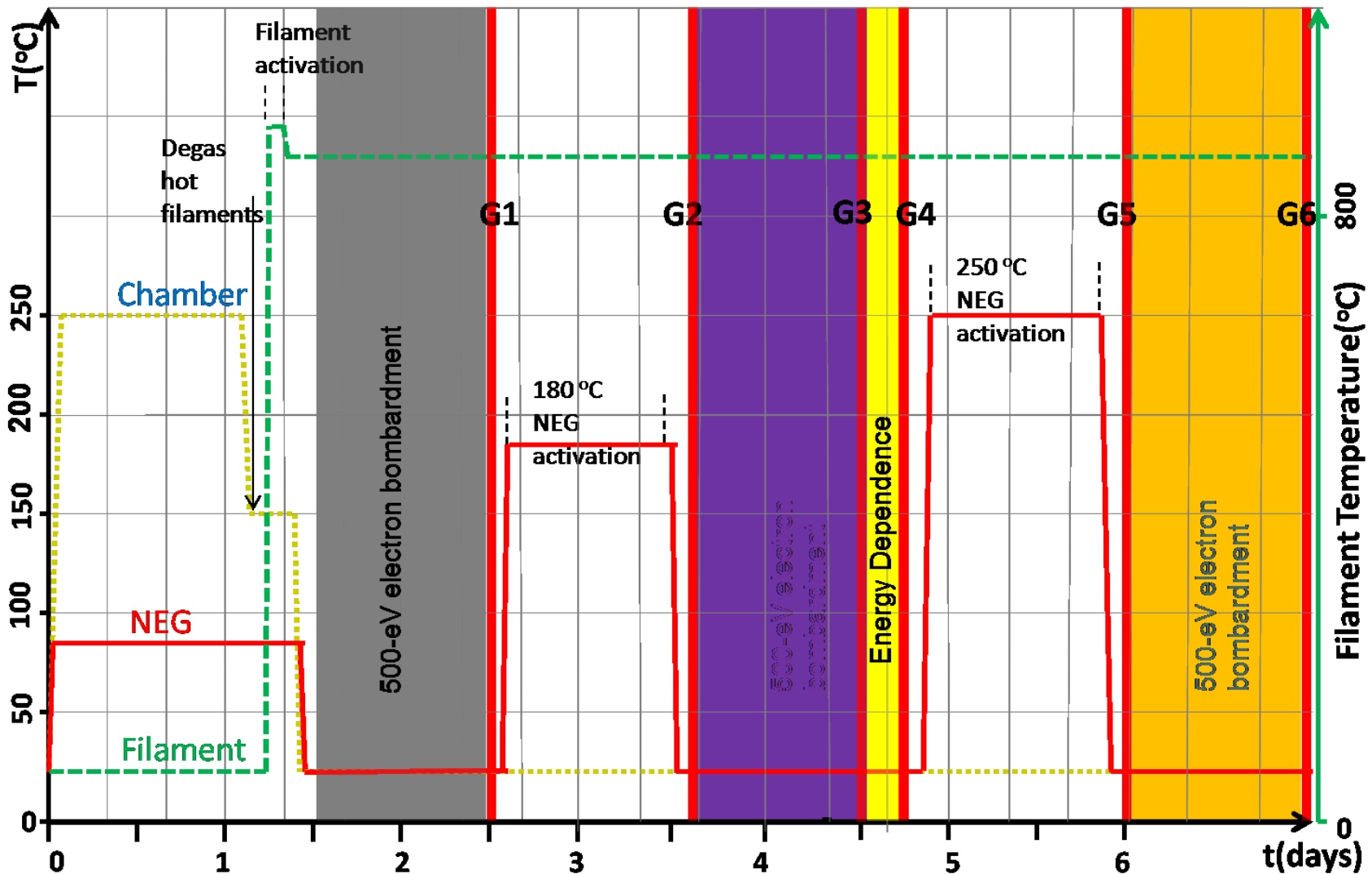
- Specially treated samples
 - Vacuum fired, polished, etc.
- Low desorption coating
- No coatings
- NEG coating
 - ESD measurements
 - Sticking probability measurements



O.B. Malyshev, A.P. Smith et al. J. Vac. Sci. Technol. A 28 (2010), p. 1215.



Experimental procedure for NEG coated samples

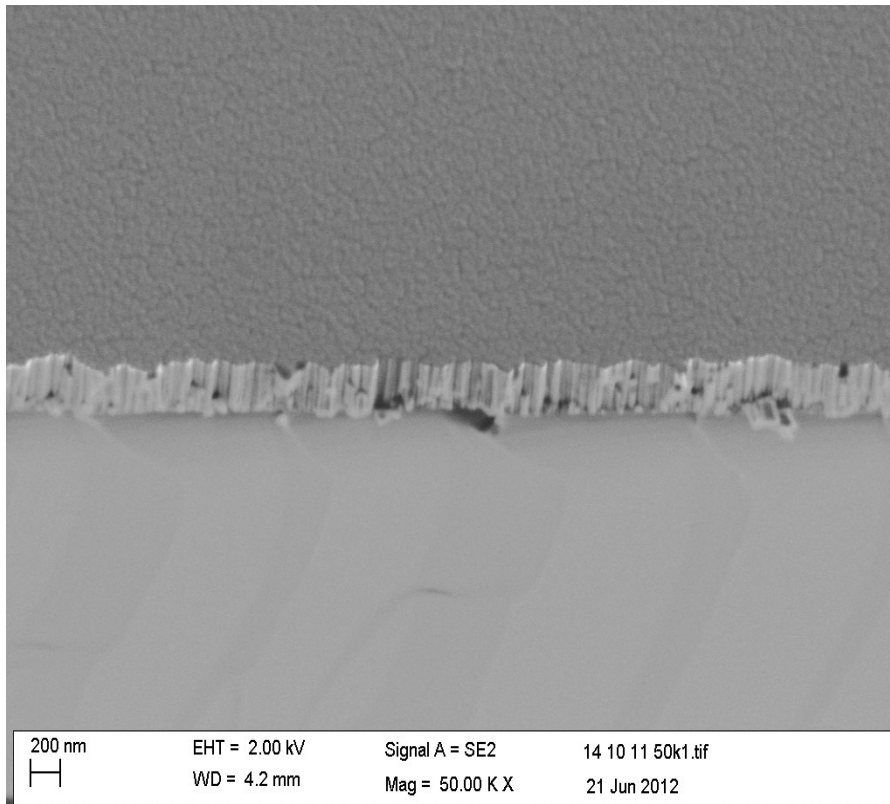




SEM images of films (film morphology)

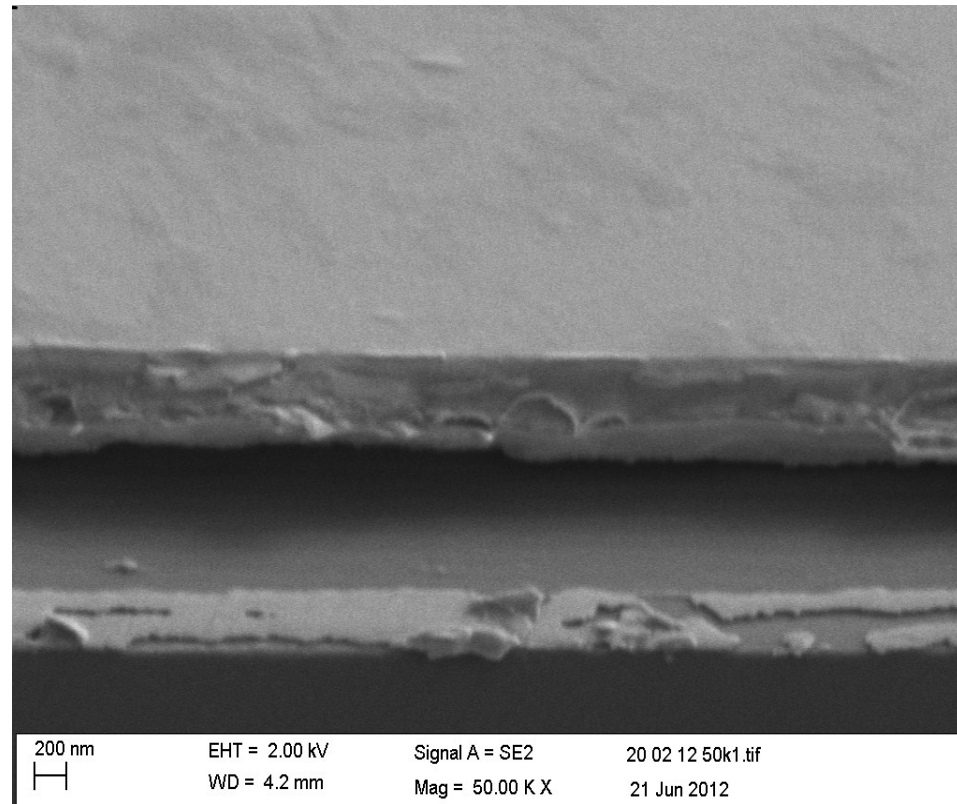
columnar (TiZrVHf)

Best for pumping



dense (TiZrVHf)

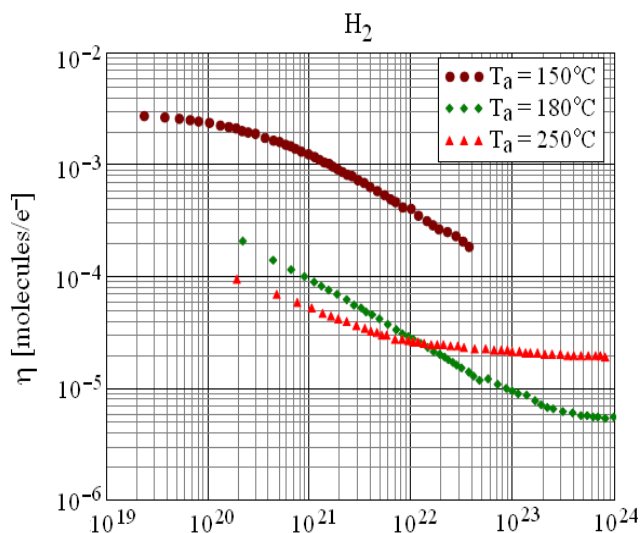
A first candidate for a barrier



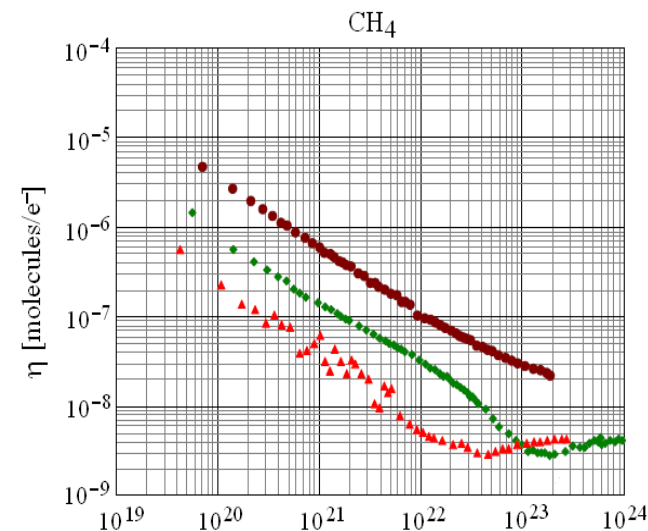
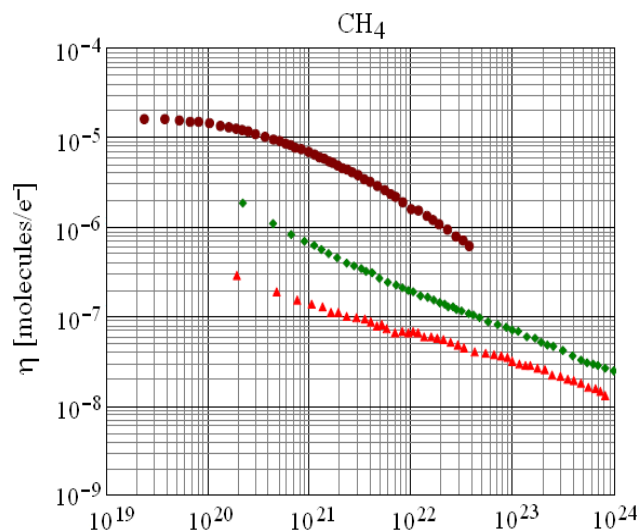
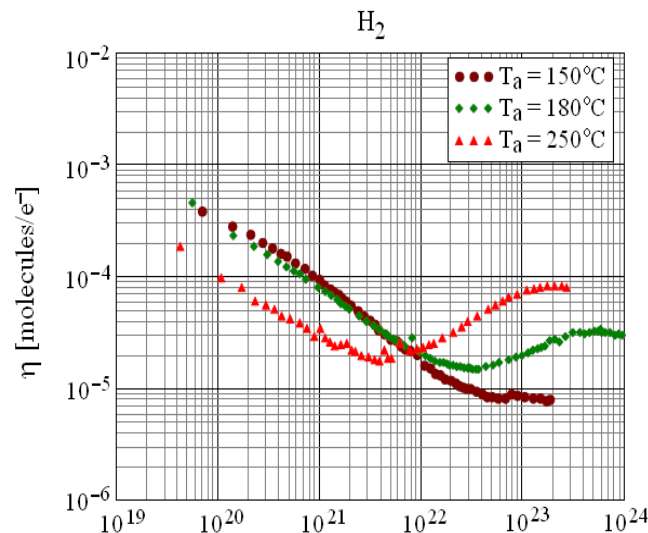


ESD yield from NEG coated samples

(a) Dense film



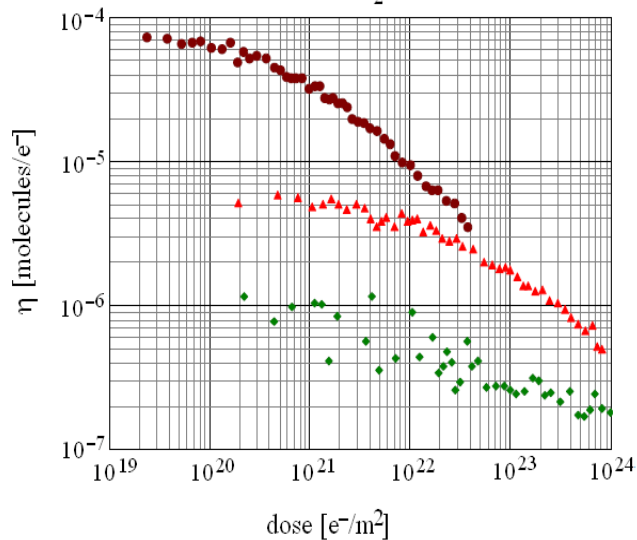
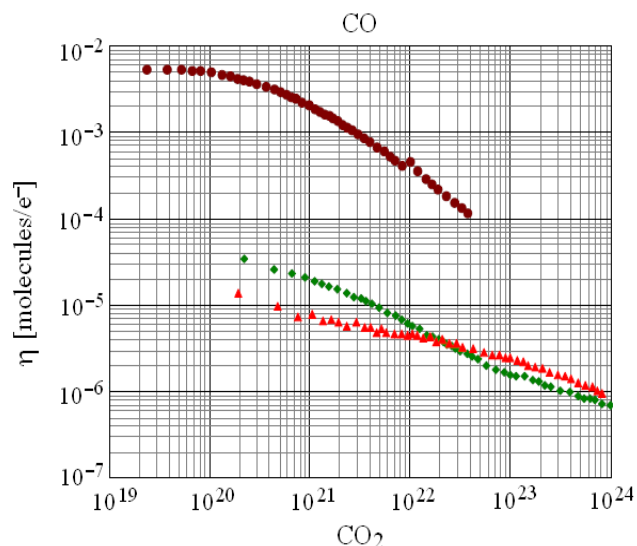
(b) Columnar film



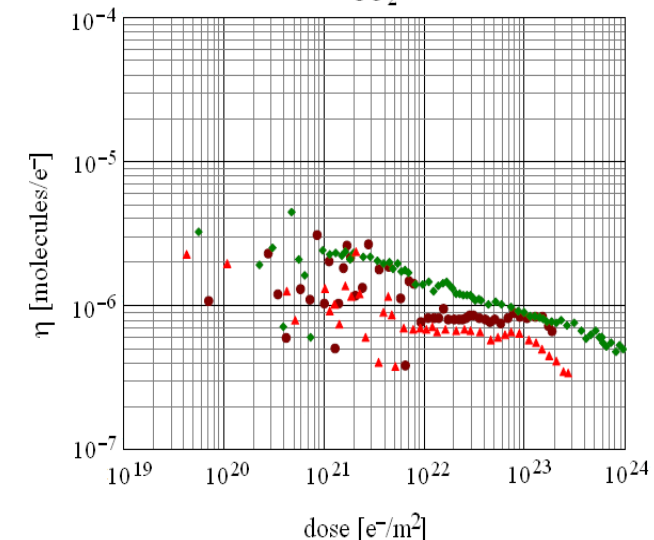
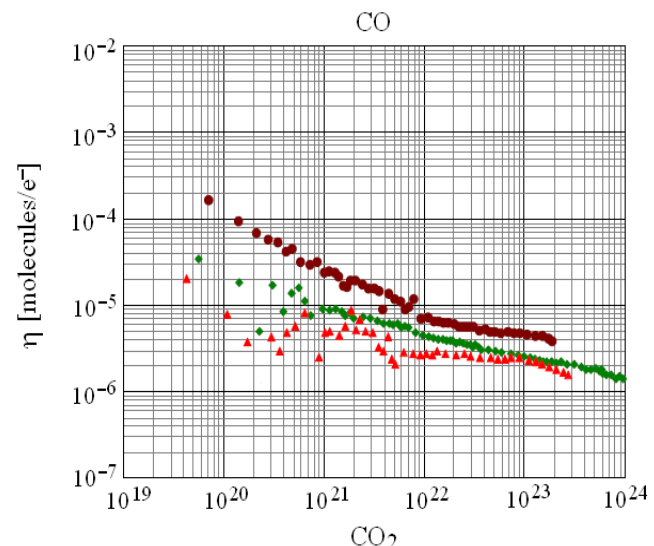


ESD yield from NEG coated samples

(a) Dense film



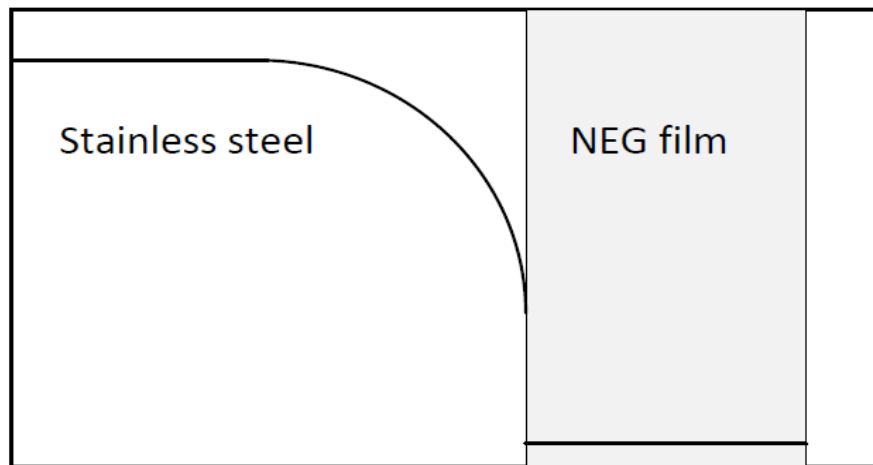
(b) Columnar film



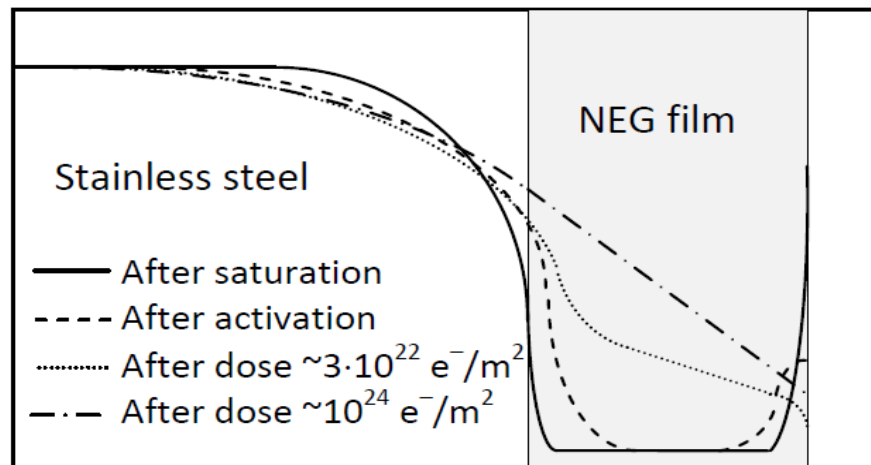


ESD yield from NEG coated samples

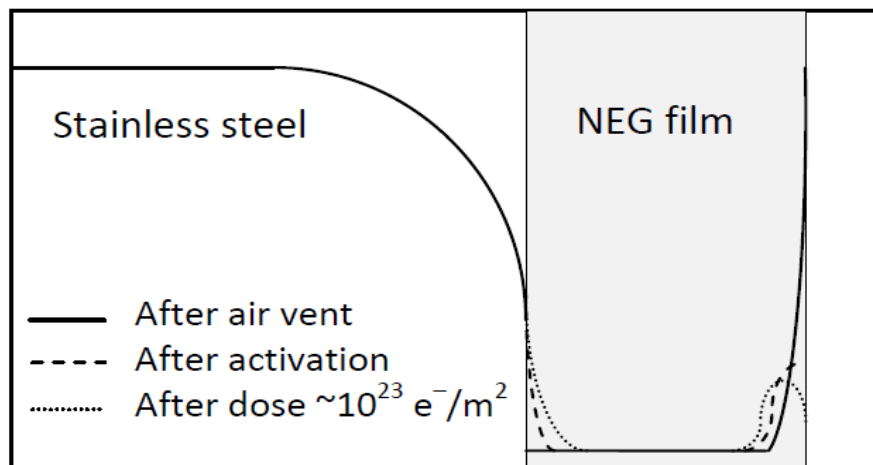
(a) after deposition before vent to air



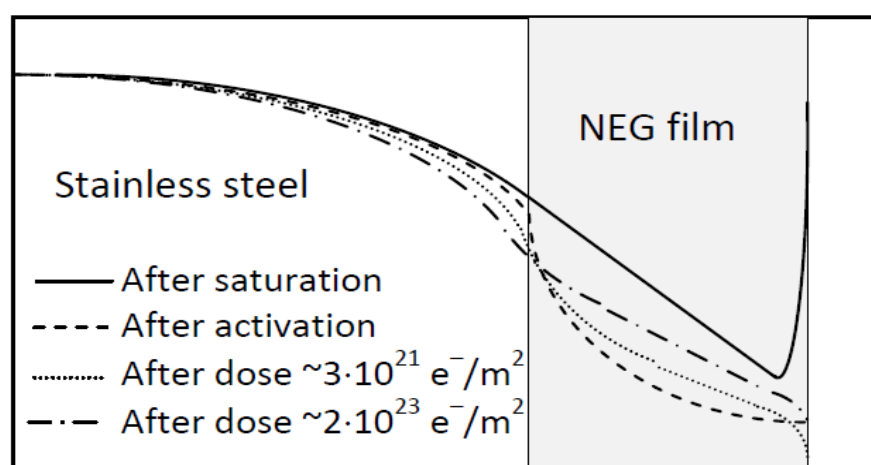
(c) activation at 180 °C



(b) activation at 150 °C



(d) activation at 250 °C





Conclusions

- ASTeC activation procedure minimises NEG poisoning from non-coated vacuum chamber components
- Role of element:
 - Zr-based – highest sticking probability and capacity, lowers activation temp.
 - Ti-based – lowest sticking probability and capacity, highest activation temp
- Role of grain size
 - Activation temperature reduces with a grain size due to increase the grain boundary density
- Quaternary alloy demonstrated the lowest activation temperature and best pumping properties;
 - Pure Zr film is good as well
- Alloy target is better than twisted wires
- The improvement and further development of NEG coatings requires
 - Intensive use surface analysis techniques
 - Evaluation under photon, electron and ion bombardment.