

Optimization of non-evaporable getter coating for accelerator beam pipe

Dr. Oleg B. Malyshev

Senior Vacuum Scientist

Dr. Reza Valizadeh

Senior Surface Scientist



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

Outlet

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

What are usual considerations for vacuum

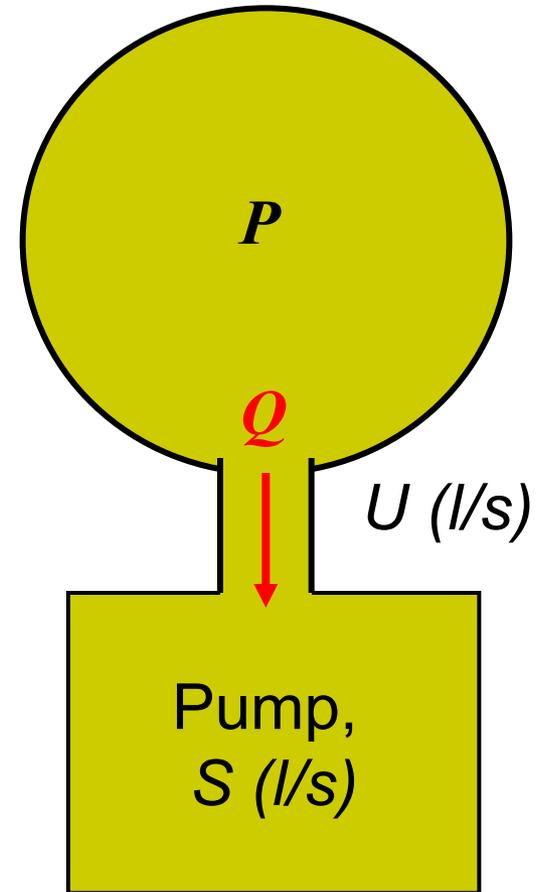
Required pressure P is defined by gas desorption Q in the vessel and effective pumping speed S_{eff}

In a simple case it is

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

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

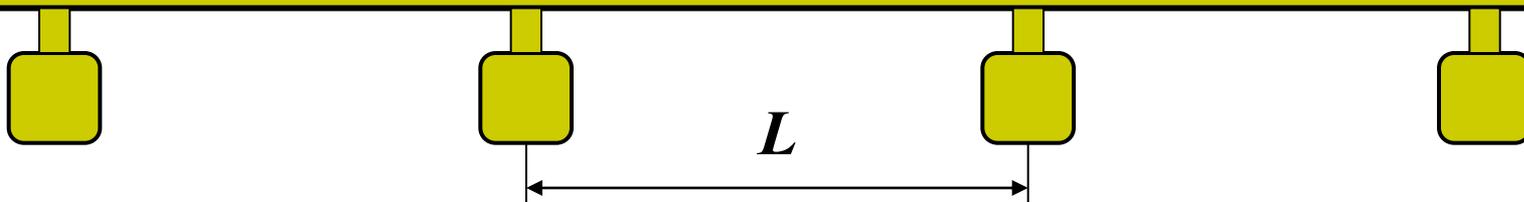
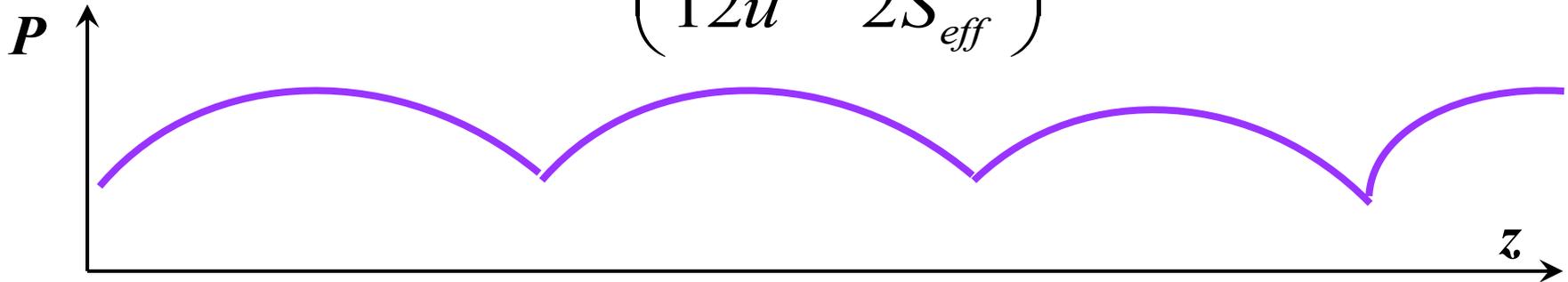
Thermal, photon, electron and ion stimulated desorption



Usual accelerator vacuum chamber

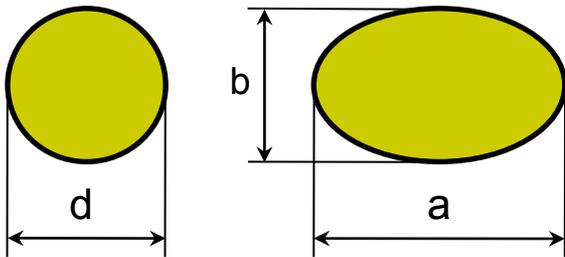
- Long tube with length $L \gg a$, where a - transversal dimension
- Average pressure depends on vacuum conductance $u(L, a)$ of the beam vacuum chamber

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

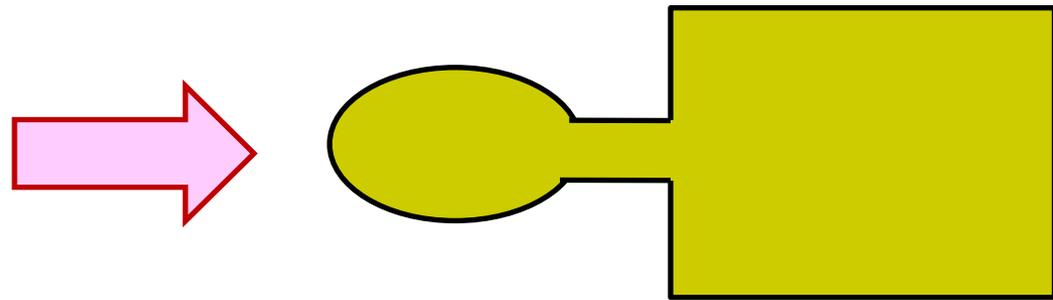


Vacuum chamber cross sections

Beam pipe
Circular or elliptical
 $4 \text{ mm} \leq d, a, b \leq 200 \text{ mm}$

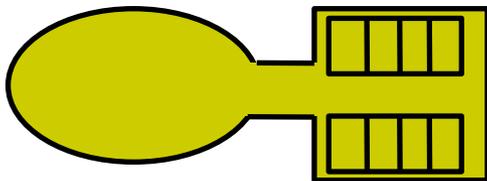


Vacuum chamber with an antechamber
for larger vacuum conductance, U

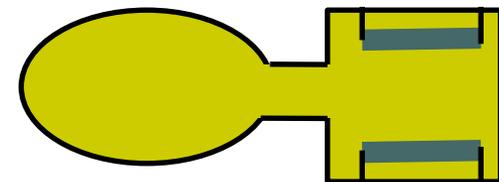
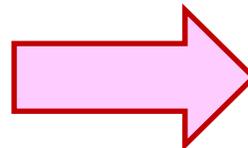


Distributed pumping

In dipole magnetic field



With NEG strips
(LEP in CERN)



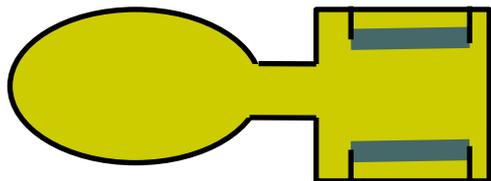
Two concepts of the ideal vacuum chamber

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

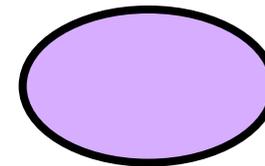


'New' (C. Benvenuti, CERN, ~1998):

- 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

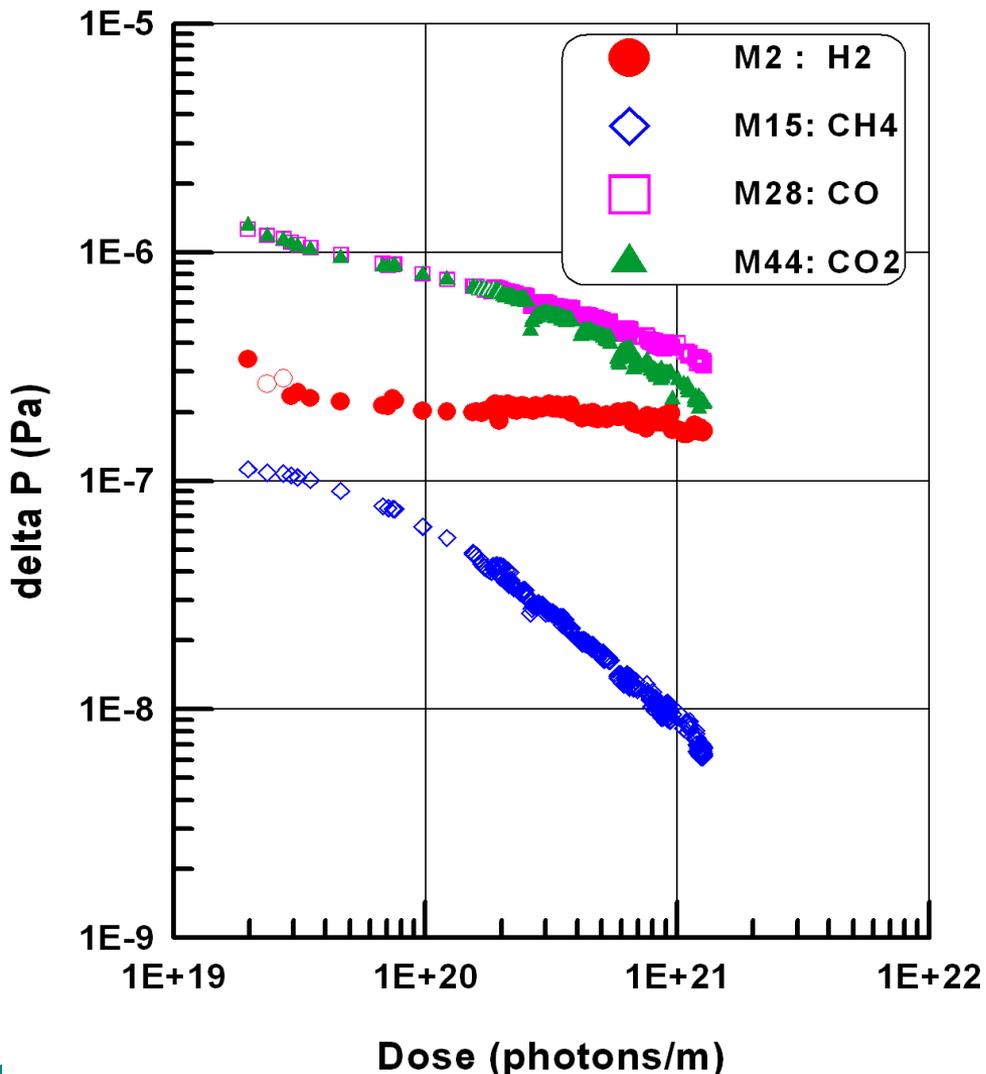
- NEG coated surface
- There should be no un-coated parts
- Activating (baking) *in-situ* at **150-180°C**
- Small pumps for C_xH_y and noble gases



Stainless steel vs. NEG coated vacuum chamber under SR

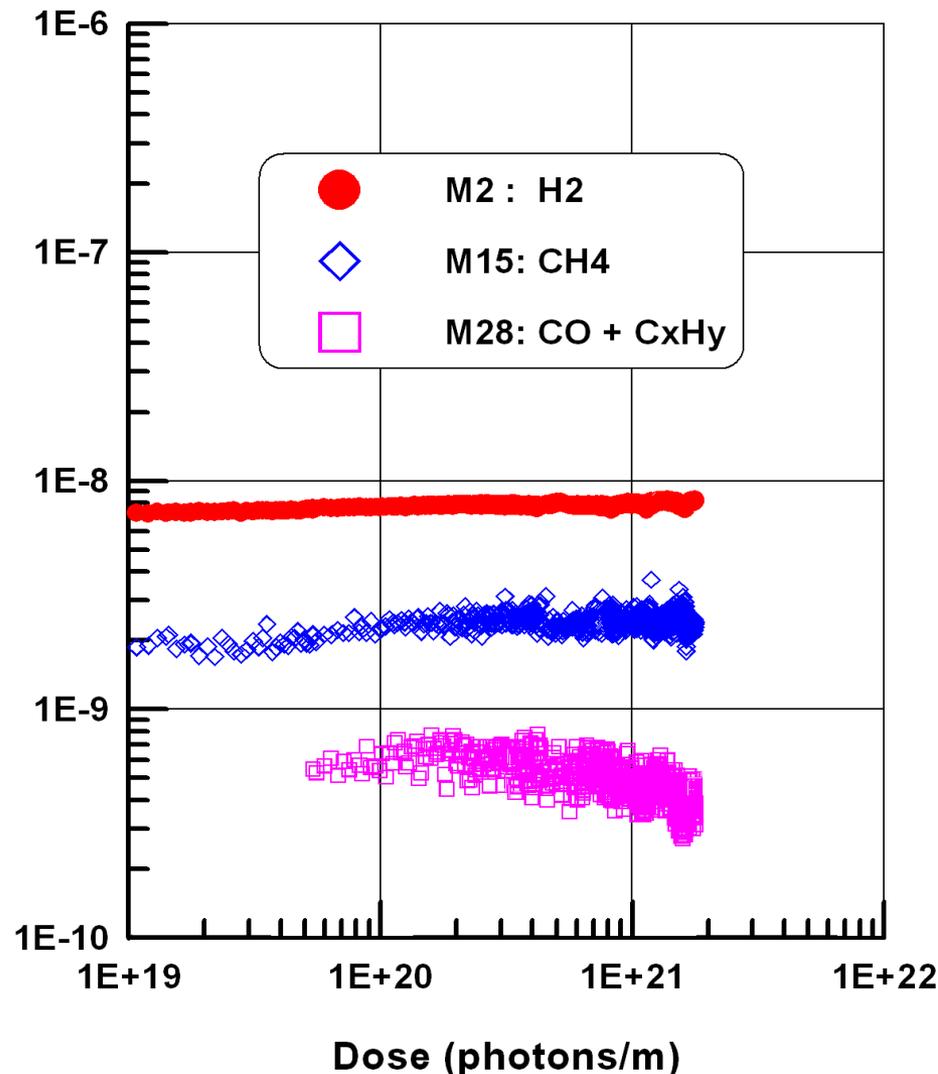
Baked at 300°C for 24 h

Stainless steel



Activated at 180°C for 24 h

TiZrV



NEG coating for accelerators

- First used in the ESRF (France);
- ELETTRA (Italy);
- Diamond LS (UK);
- Soleil (France) – first fully NEG coated;
- LHC (Switzerland) – longest NEG coated vacuum chamber;
- SIS-18 (Germany);
- and many others.
- **NEG film capacity for CO and CO₂ is ~1ML:**
 - If $P = 10^{-9}$ mbar then 1 ML can be sorbed just in $\sim 10^3 - 10^4$ s;
 - Lab measurements of different NEG coatings often don't repeat CERN's data on sticking probability and capacity;
 - However, NEG coated parts of accelerators work well.

NEG coating for accelerators (2)

- What is required:
 - Input data for accelerator design:
 - $\eta(D, E, T_a)$, $\alpha(M, T_a)$, pumping capacity;
 - Better understanding:
 - what and why;
 - practical 'do's and 'don't's';
 - Further development of this coating:
 - lower η , T_a , SEY;
 - higher $\alpha(M)$, pumping capacity;
 - optimising for an application.

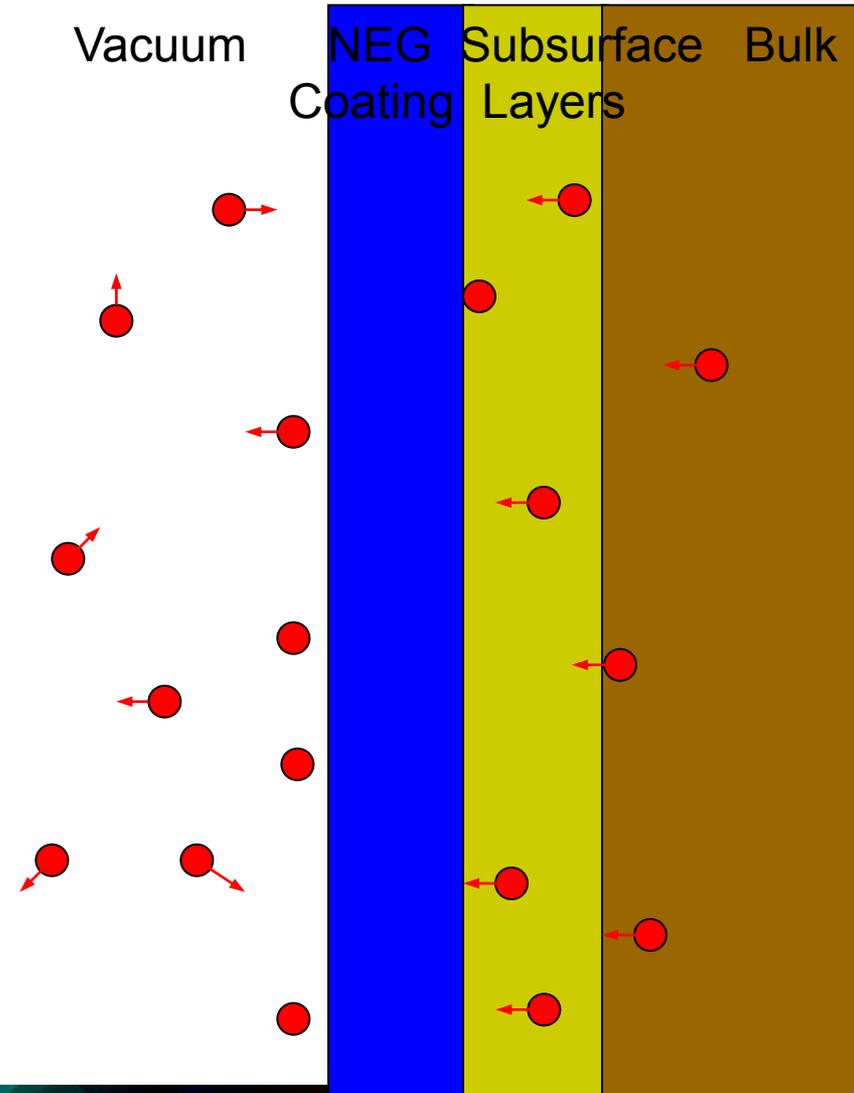
What NEG coating does

- **Reduces gas desorption:**
 - A pure metal film ~1- μm thick without contaminants.
 - A barrier for molecules from the bulk of vacuum chamber.

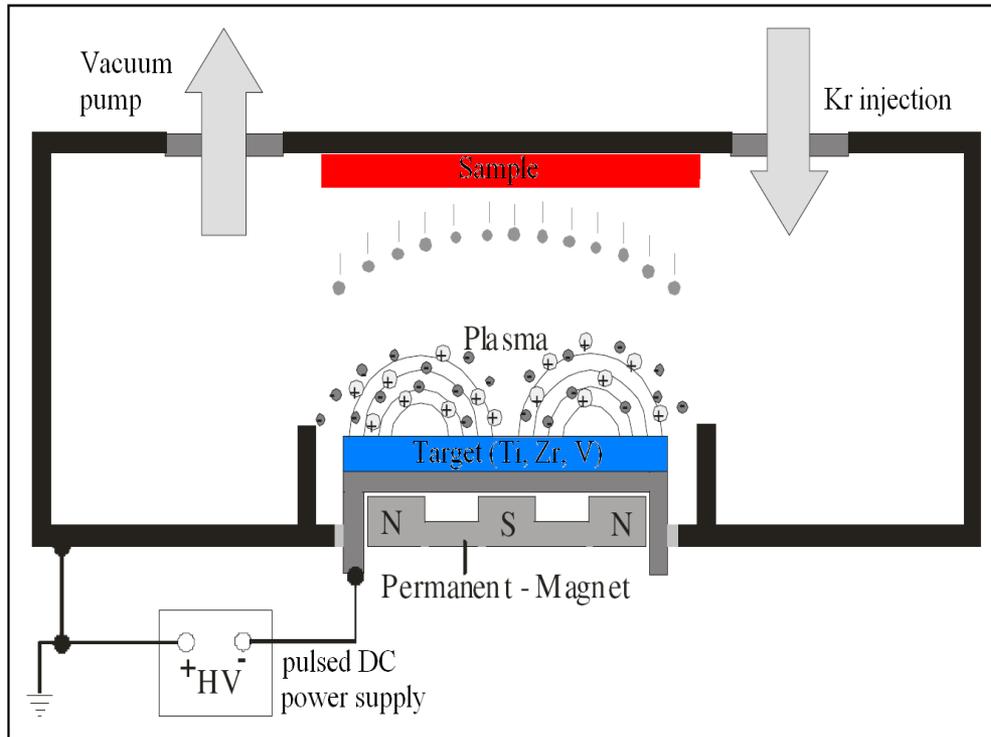
- **Increases distributed pumping speed, S:**
 - A sorbing surface on whole vacuum chamber surface

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

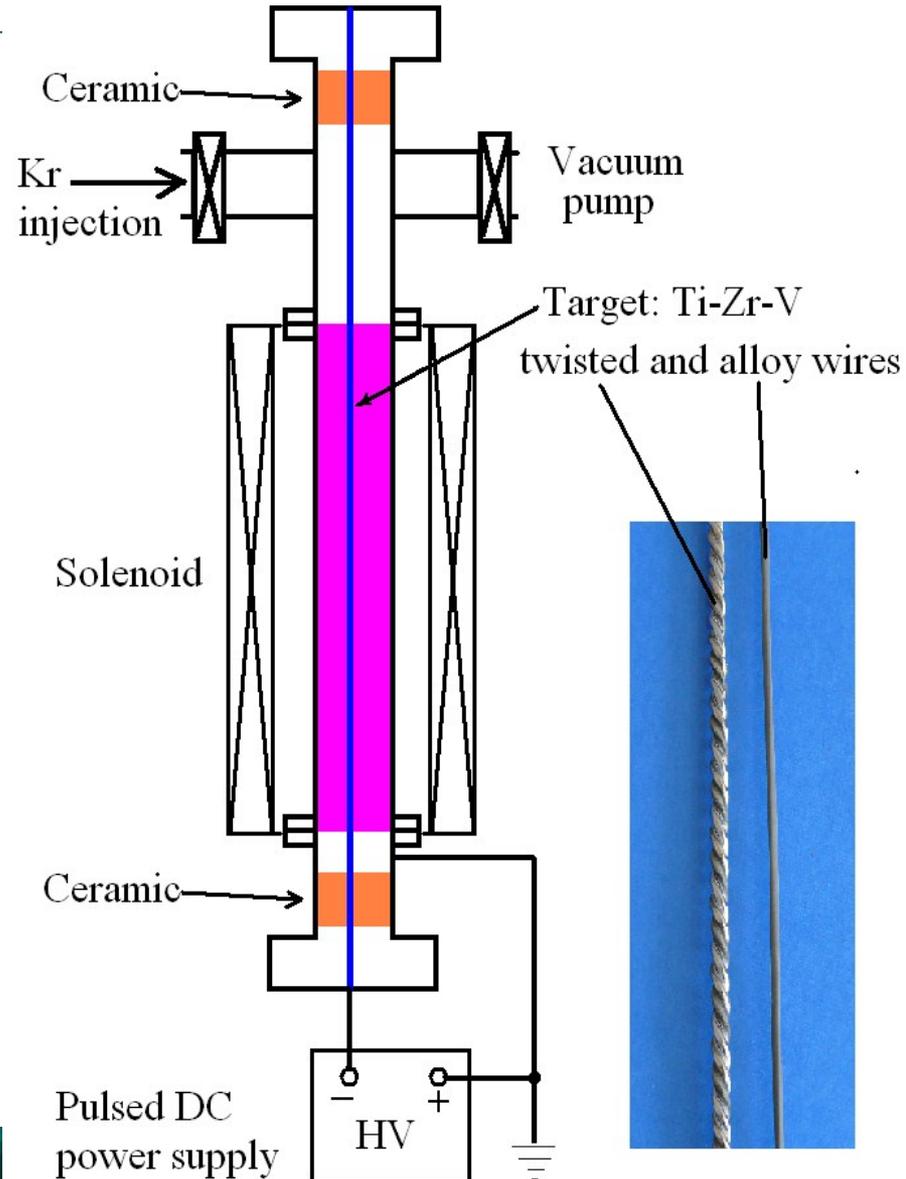
where α – sticking probability,
 A – surface area,
 v – mean molecular velocity



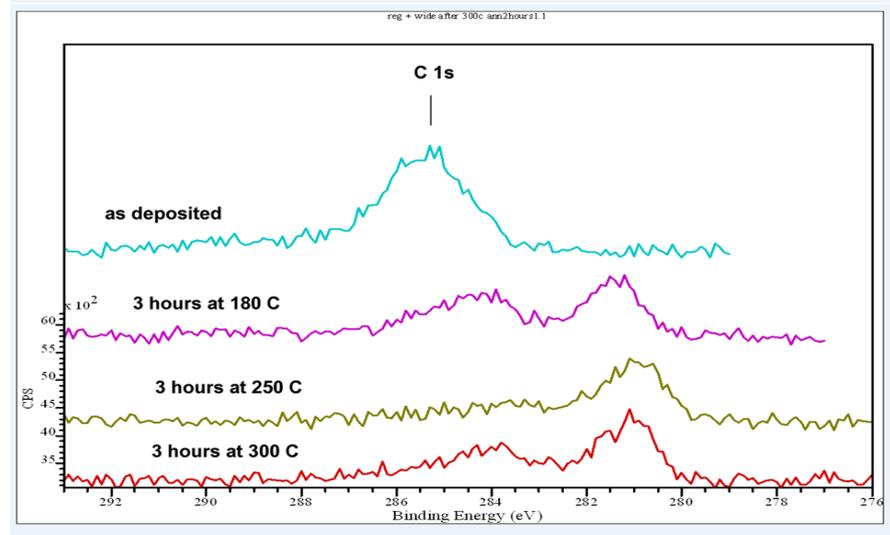
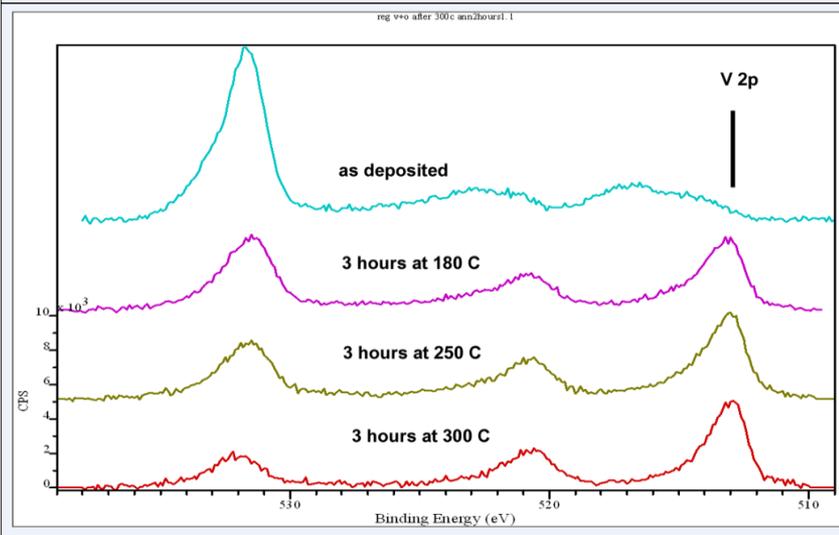
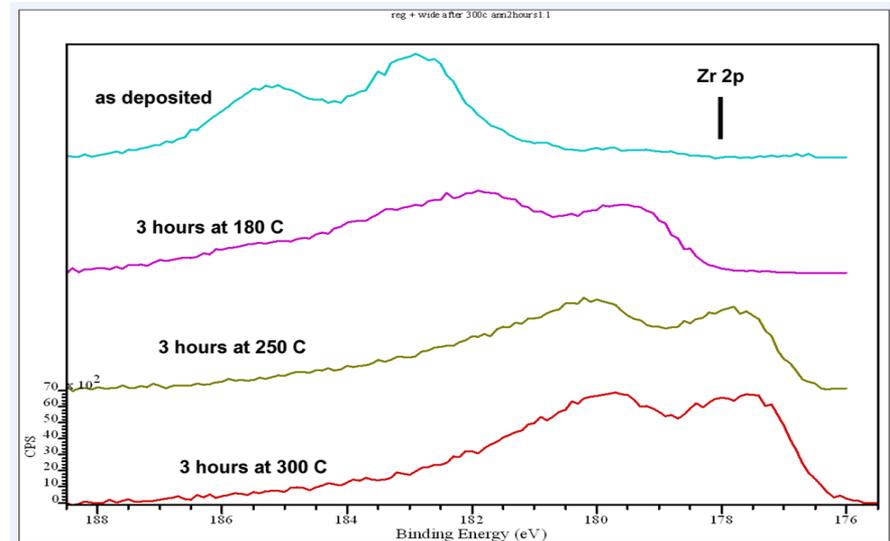
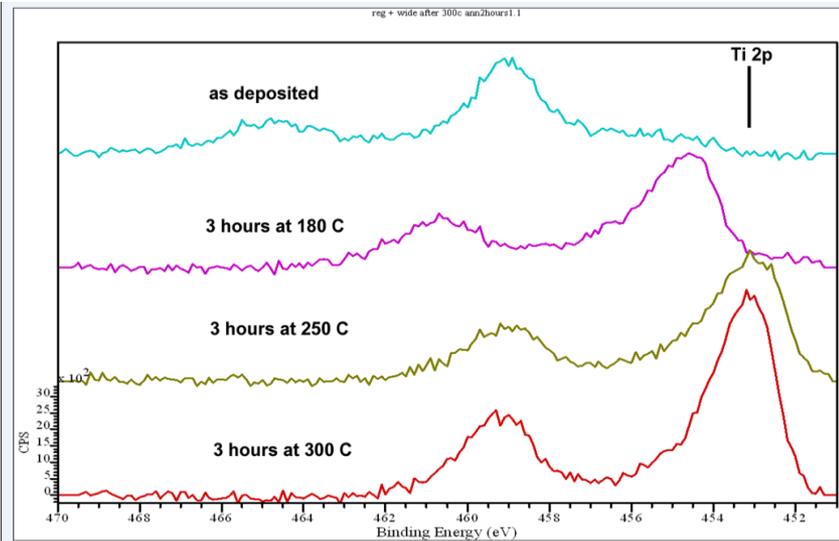
Planar magnetron deposition



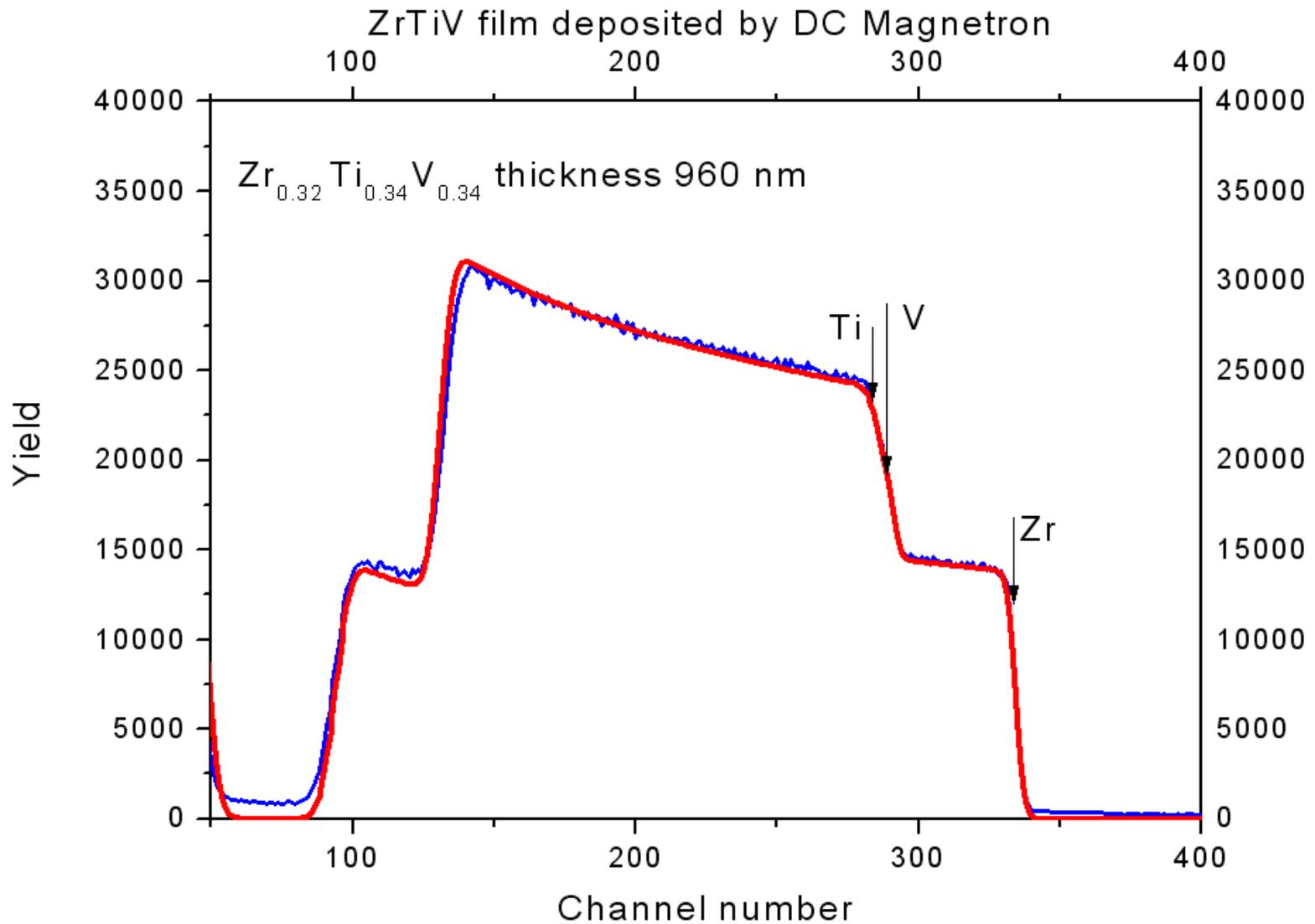
Cylindrical magnetron deposition



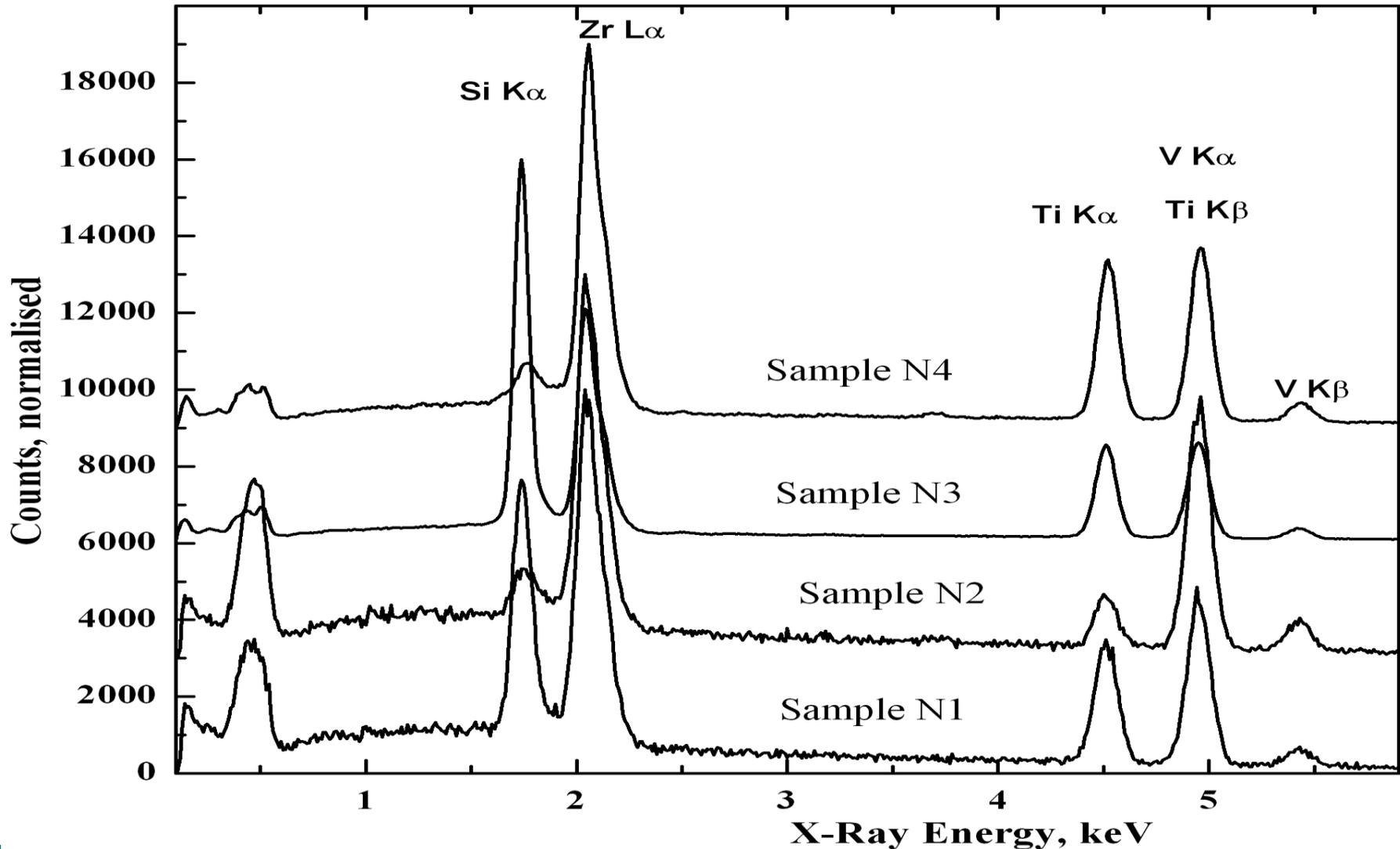
Region scan of XPS core levels of Ti, Zr, C and V of a Ti-Zr-V film (surface composition and chemical bounding)



RBS (film compositions in bulk)

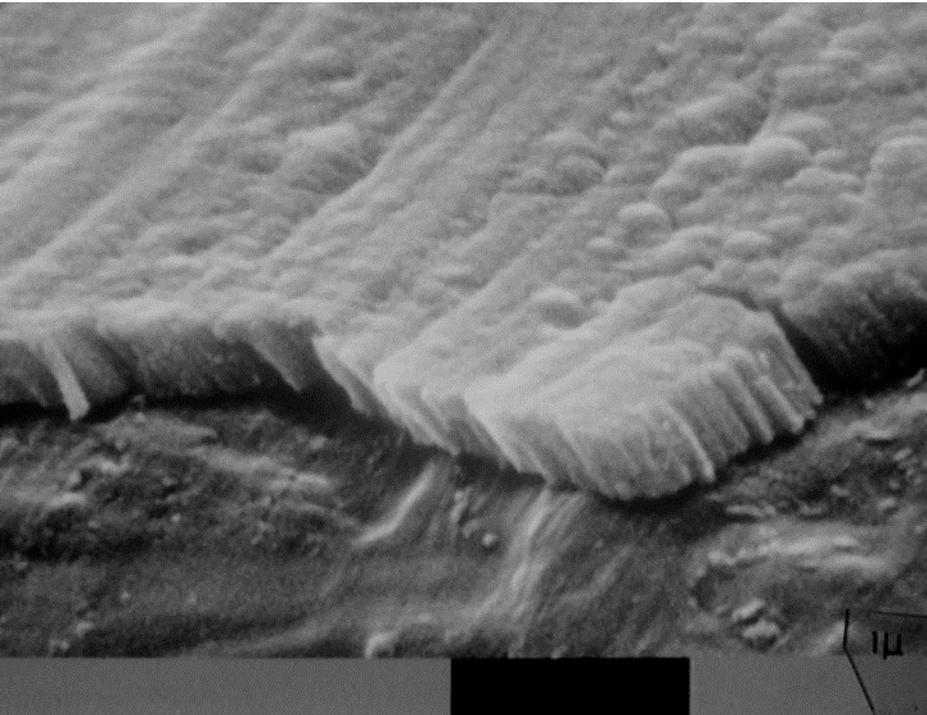


The EDX analysis for determination of film composition

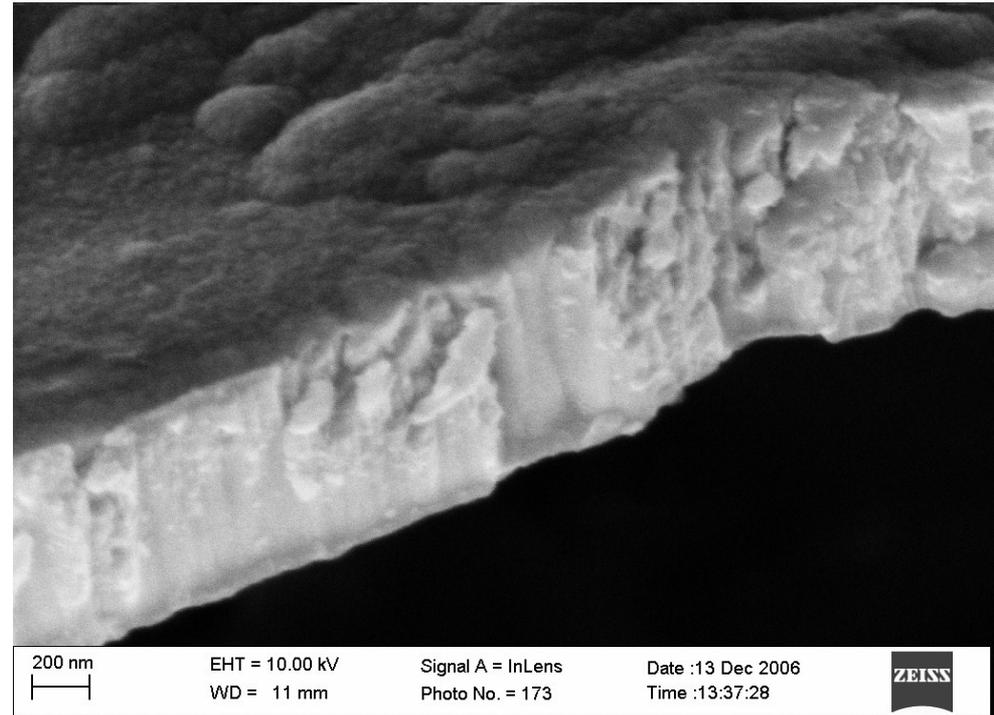


SEM images of films (film morphology)

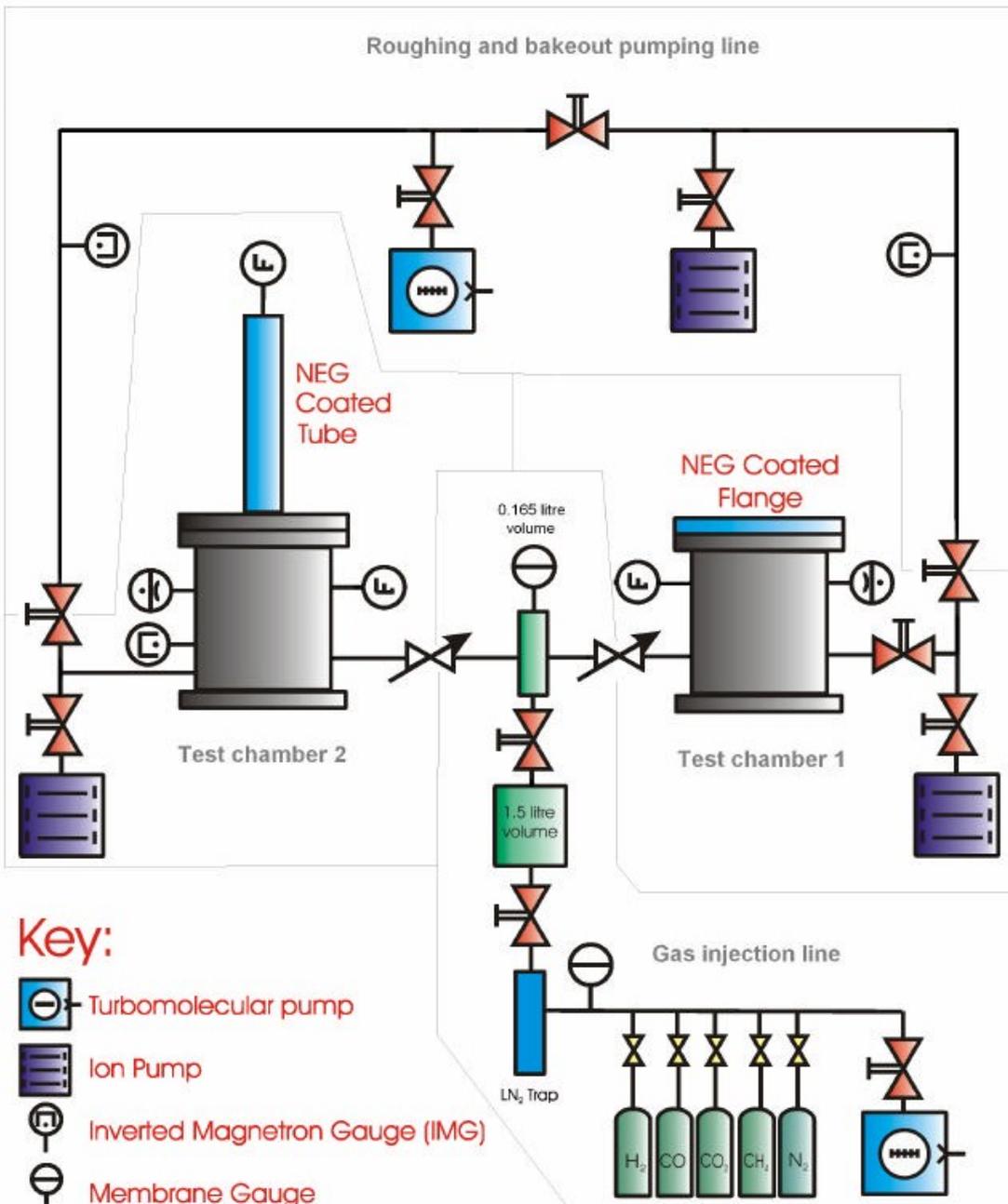
columnar



dense



O.B. Malyshev, R. Valizadeh, J.S. Colligon *et al.* J. Vac. Sci. Technol. A 27 (2009), p. 521.

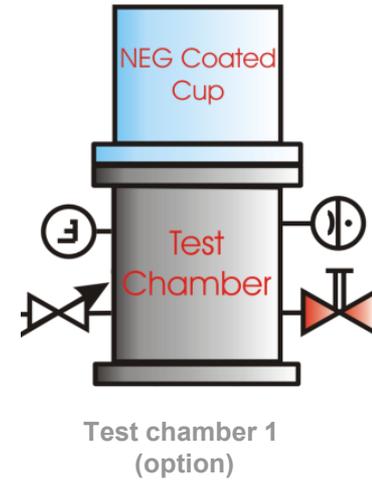


Key:

- Turbomolecular pump
- Ion Pump
- Inverted Magnetron Gauge (IMG)
- Membrane Gauge

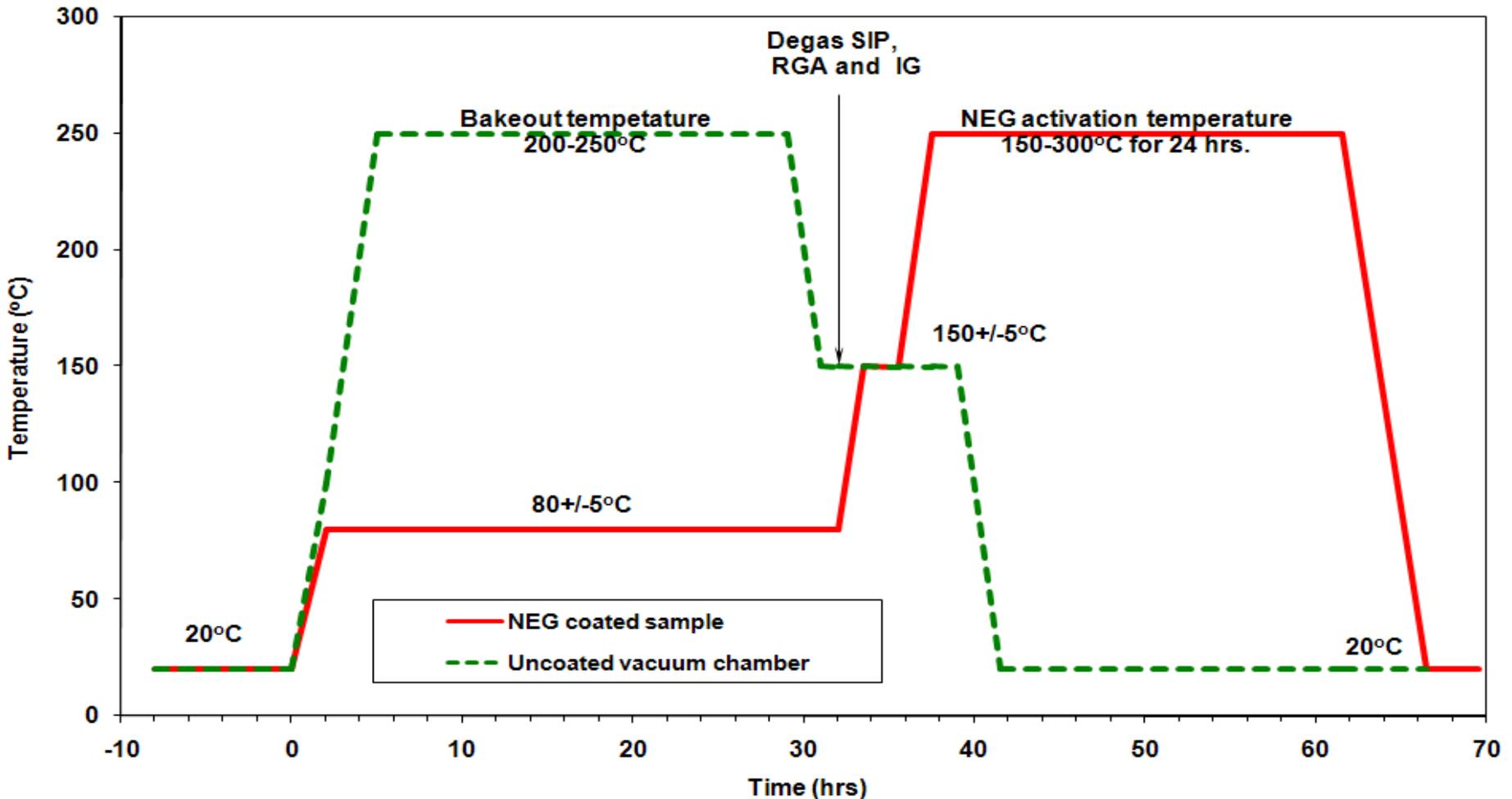
*O.B. Malyshev and K.J. Middleman.
Vacuum 83 (2009), p. 976.*

*O.B. Malyshev et al.
J. Vac. Sci. Technol. A 27 (2009), p. 321.*

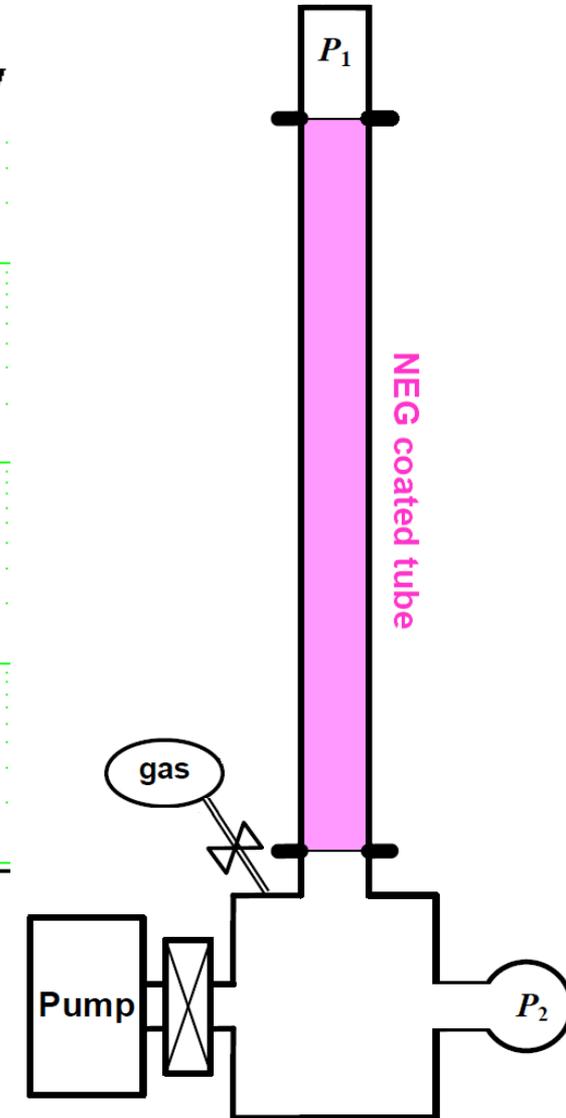
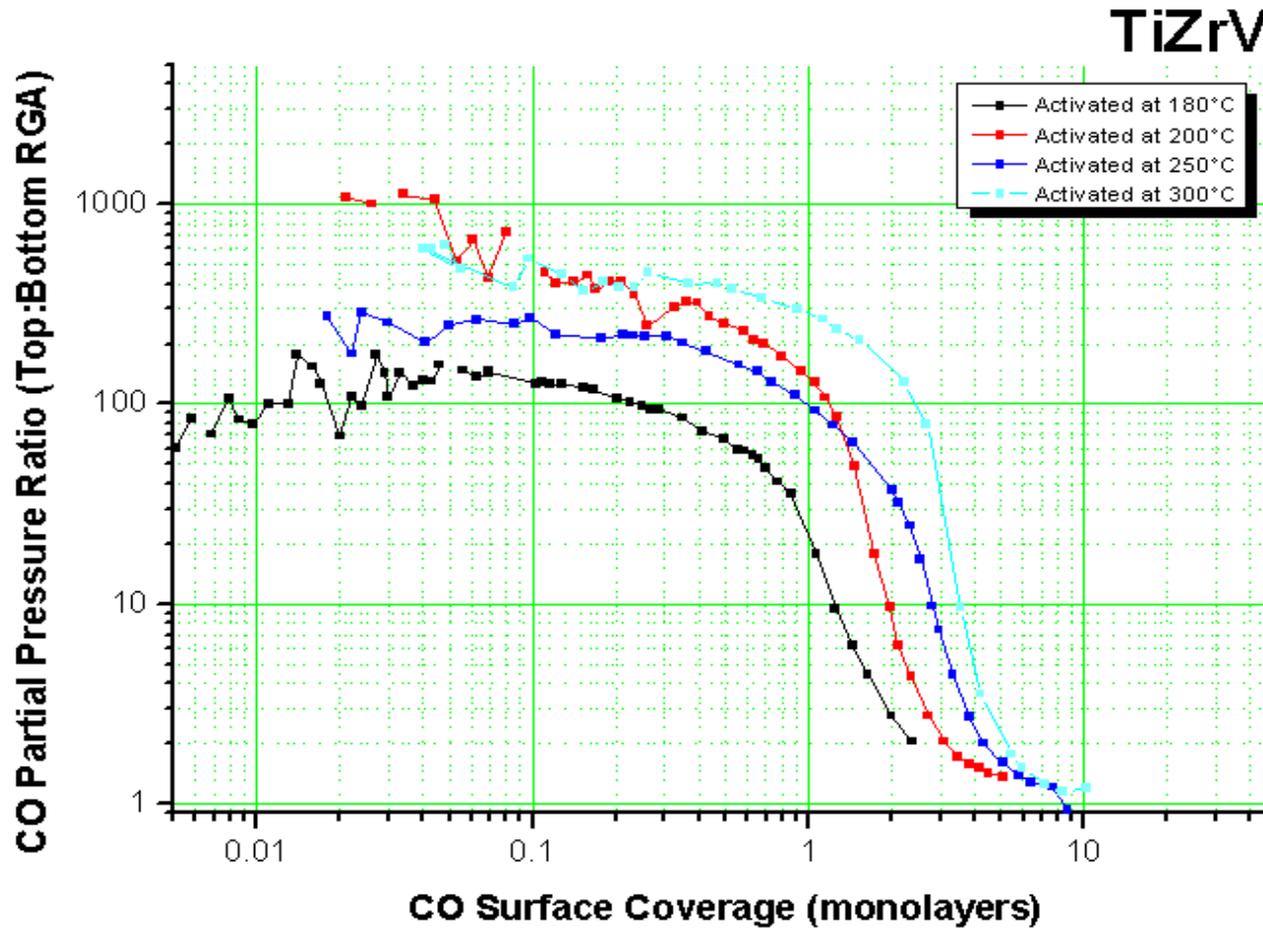


- Residual Gas Analyser (RGA)
- Extractor Gauge
- Fine Leak Valve

ASTeC activation procedure



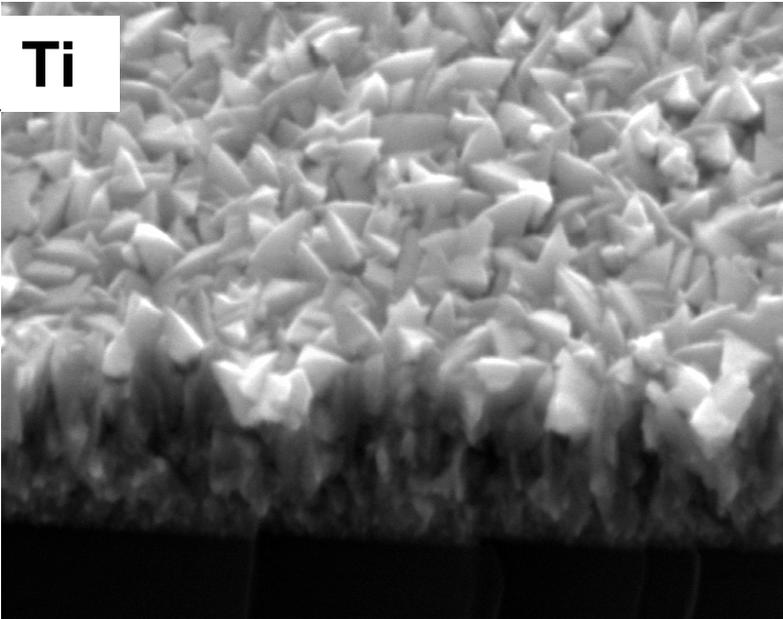
O.B. Malyshev, K.J. Middleman, J.S. Colligon and R. Valizadeh. *J. Vac. Sci. Technol. A* 27 (2009), p. 321.



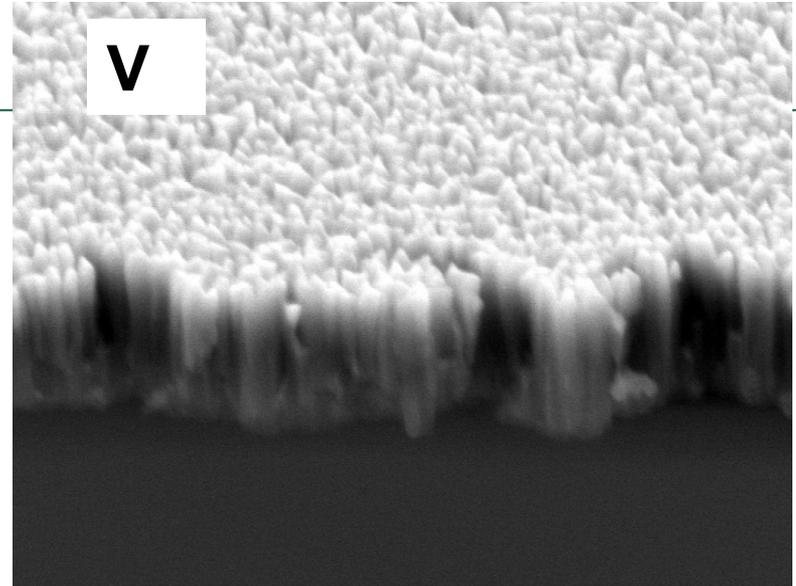
Pressure ratio P_1/P_2 measured during gas injection is used to estimate:
 initial sticking probability and sorption capacity

Ti, Zr, Hf and V film deposited on Si test sample from a single Ti wire

Ti



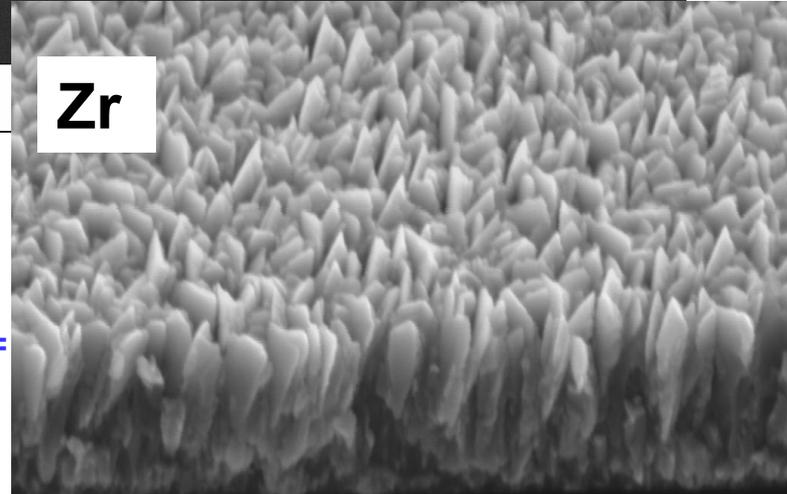
V



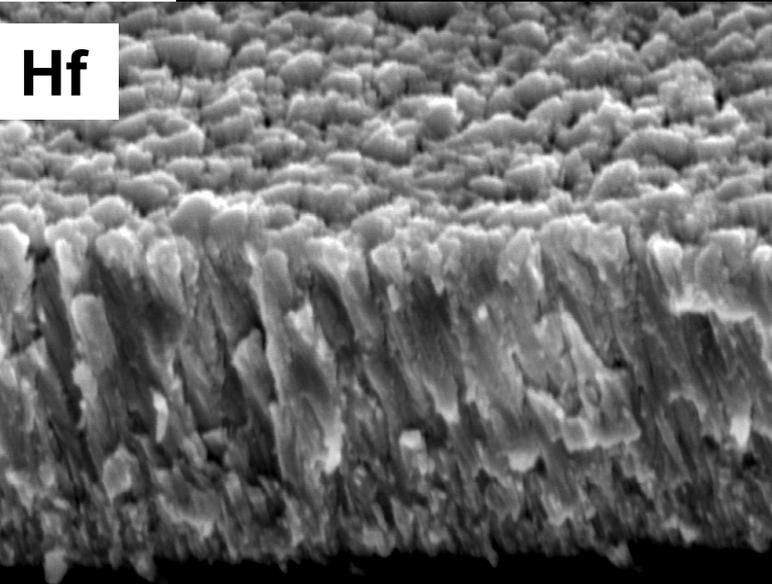
200 nm



Zr



Hf



Cylindrical
Magnetron:
Power = 60 W,
 $P_{Kr} = 10^{-2}$ mbar,
deposition rate =
0.14 nm/s,
 $T = 120^{\circ}C$.
Average grain
size 100 – 150
nm.

200 nm



EHT = 10.00 kV

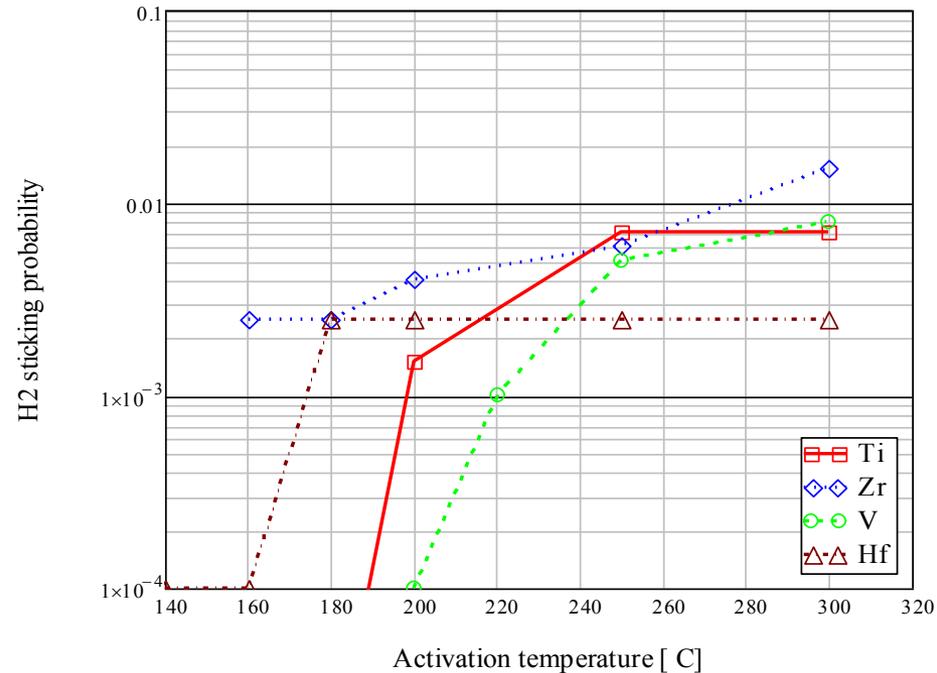
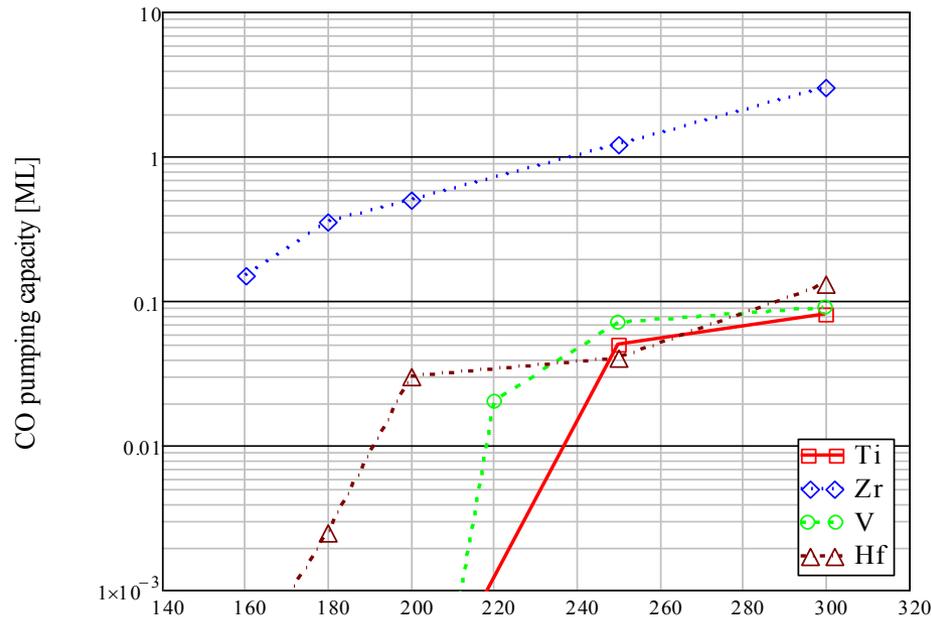
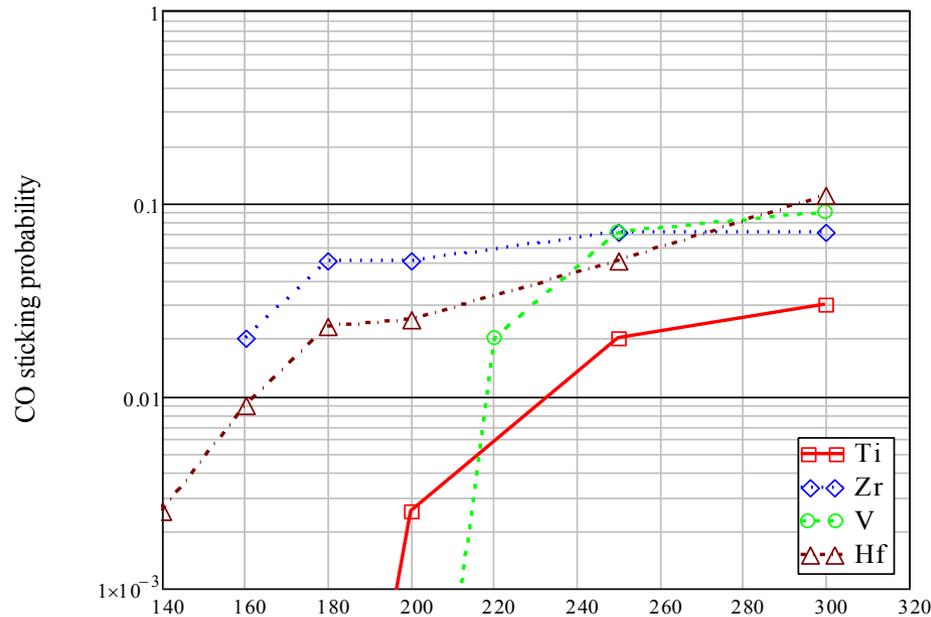
WD = 7 mm

Signal A = SE2

Mag = 100.00 K X

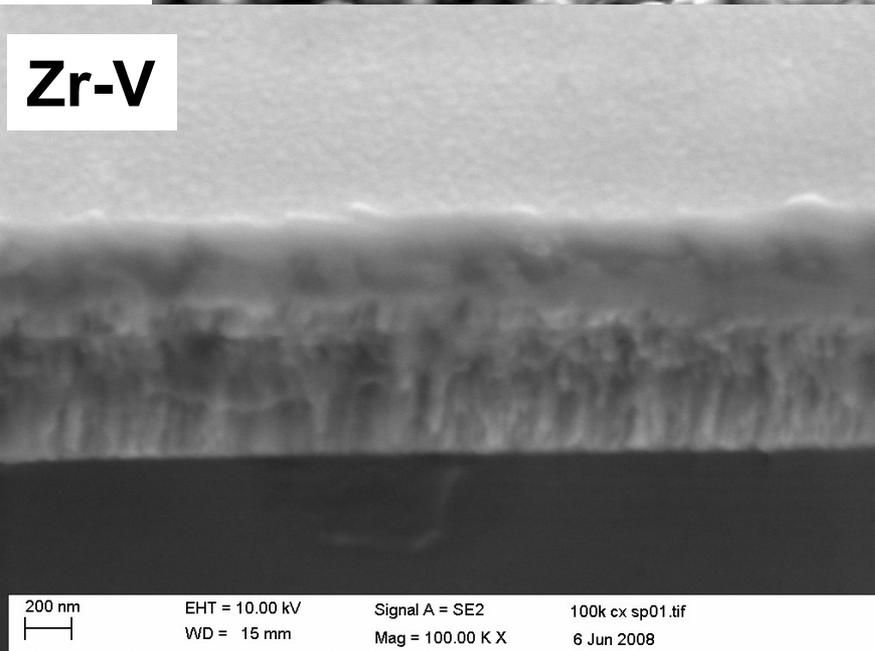
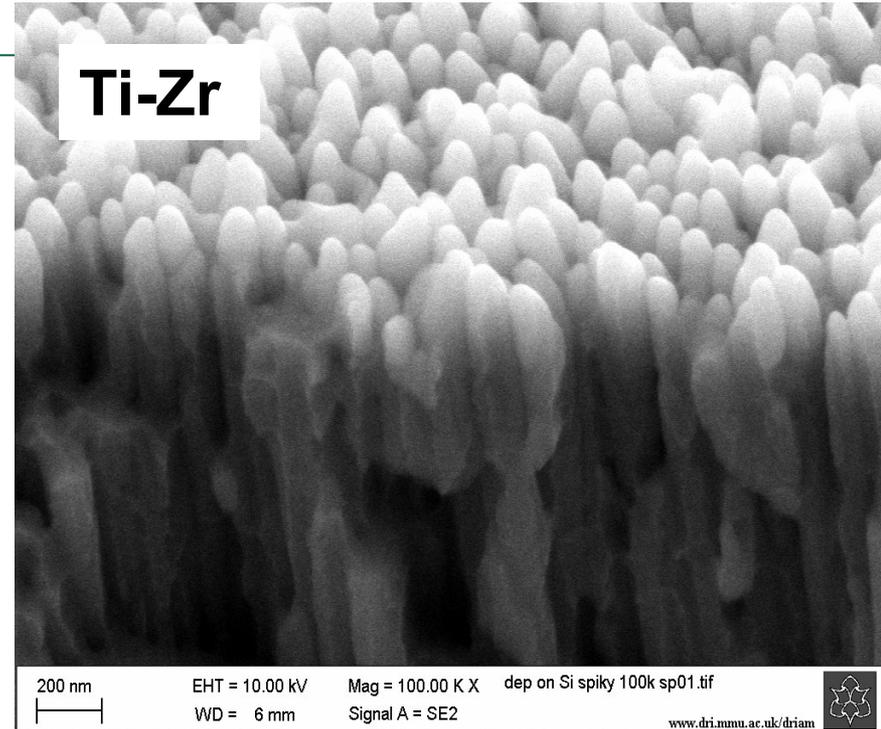
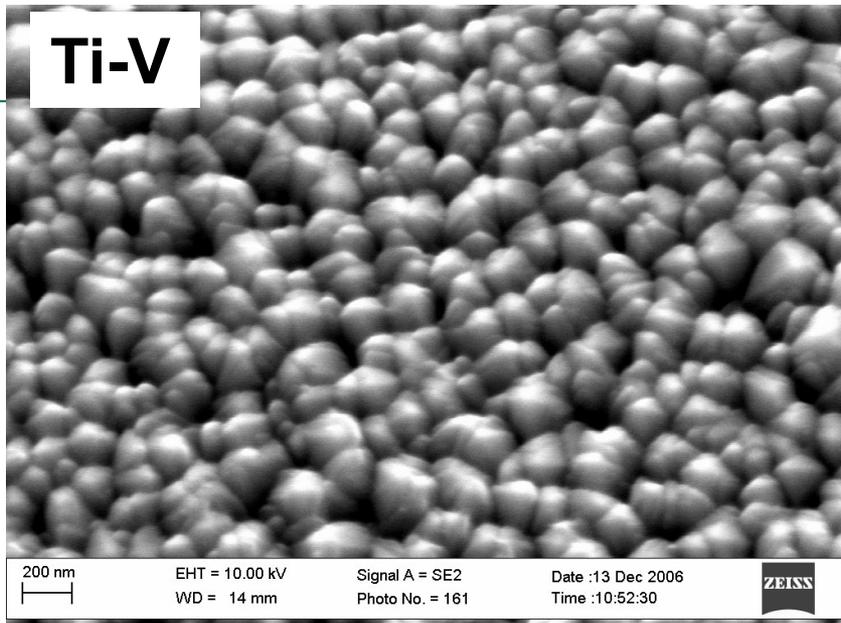
Si 100k.tif

18 May 2009

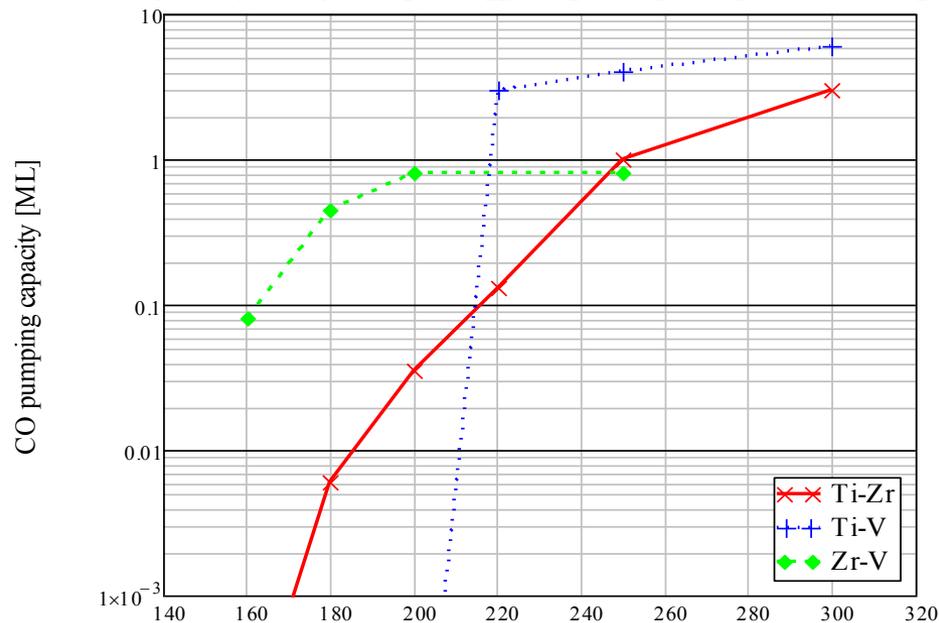
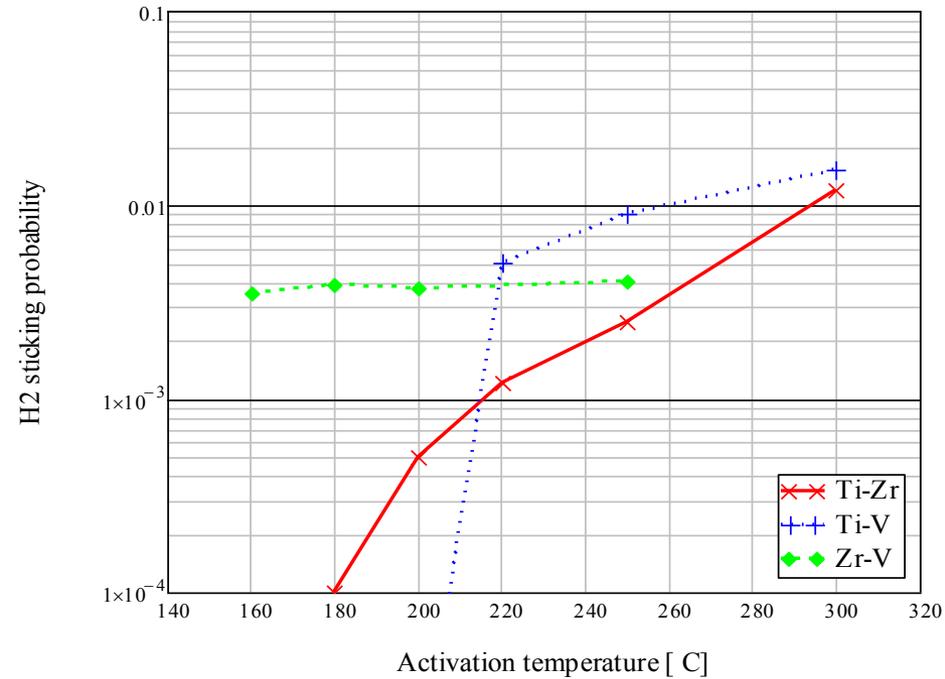
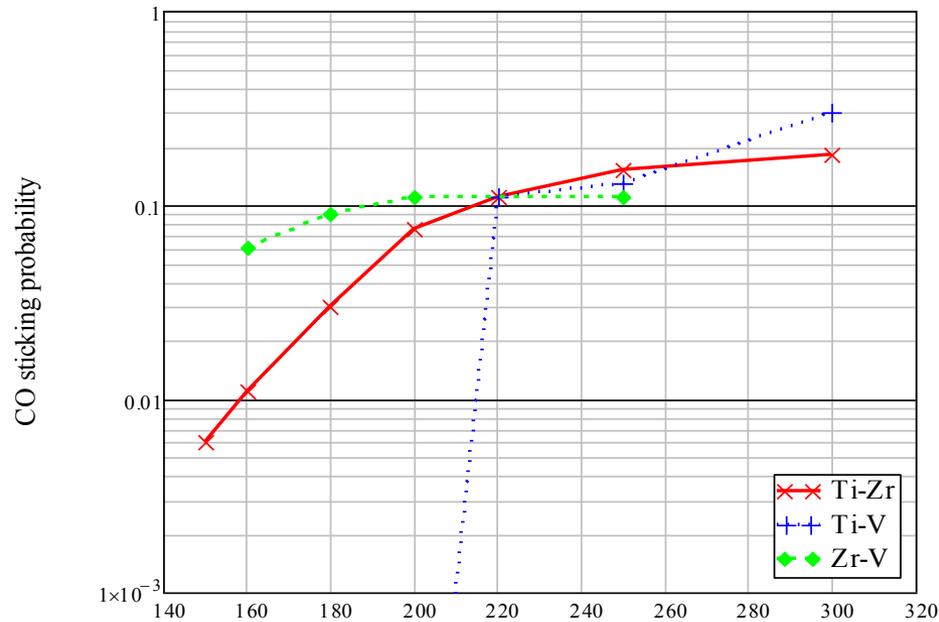


Zr is best:
 Lowest activation Temp. and
 highest capacity
Hf
Ti
V has highest activation
 temperature

Binary alloy films deposited on Si test sample from twisted wires.



Cylindrical Magnetron:
 Power = 60 W, $P_{Kr} = 10^{-2}$ mbar,
 deposition rate = 0.13 nm/s, $T = 120^{\circ}\text{C}$.
 Average grain size 50 – 100 nm.
 Hexagonal lattice structure.



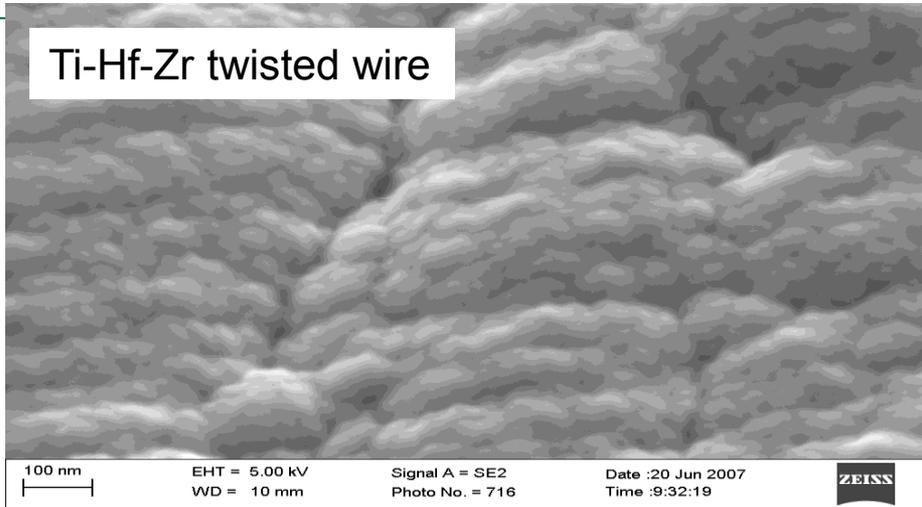
Zr-V is best

Ti-Zr activation temperature is lower than for Ti-V

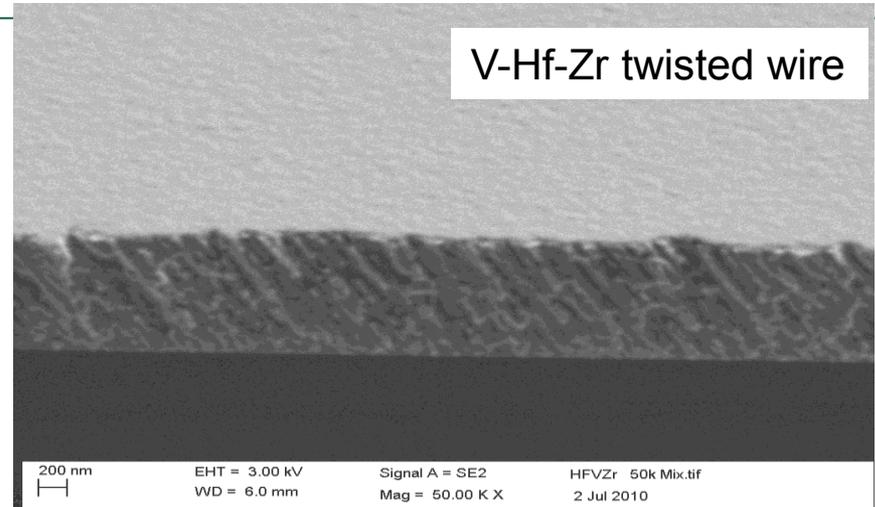
Zr-Hf was not studied

Ternary NEG film deposited on Si test sample from twisted Ti, V, Zr, and Hf wires and TiZrV alloy wire

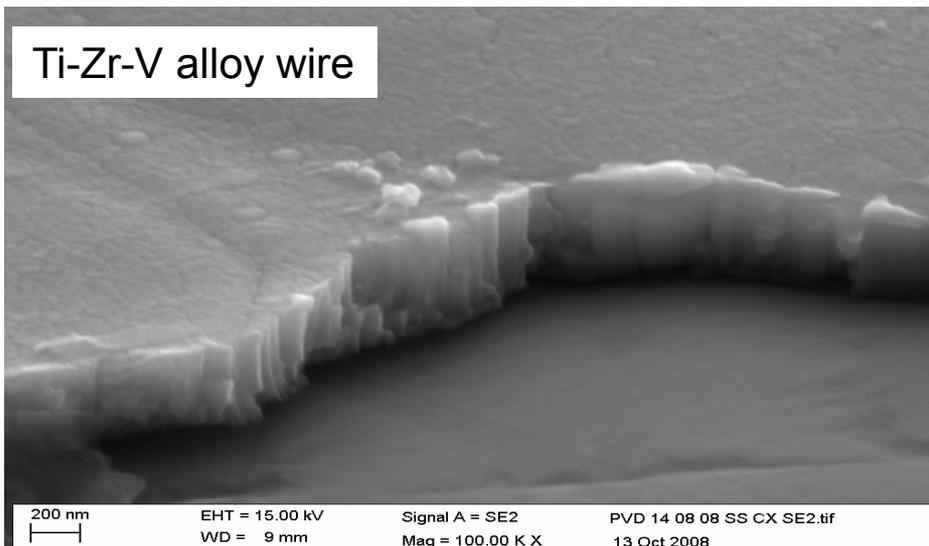
Ti-Hf-Zr twisted wire



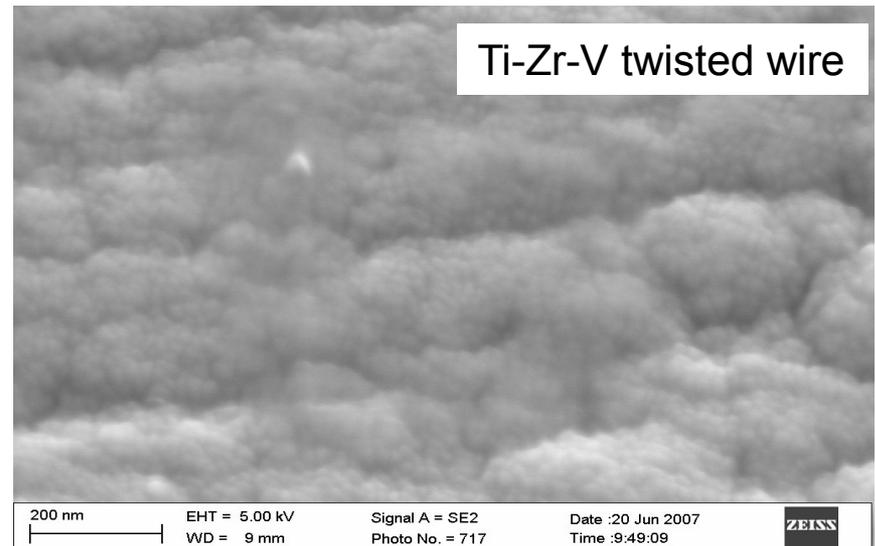
V-Hf-Zr twisted wire



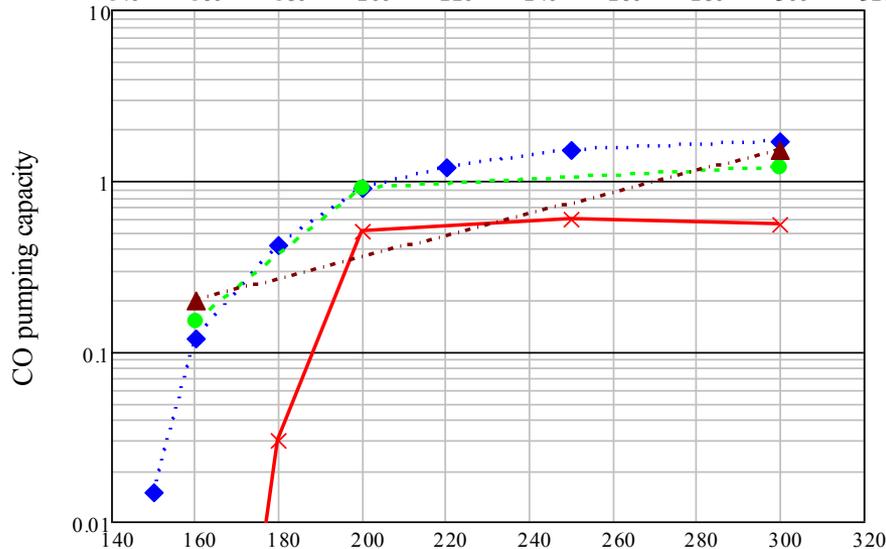
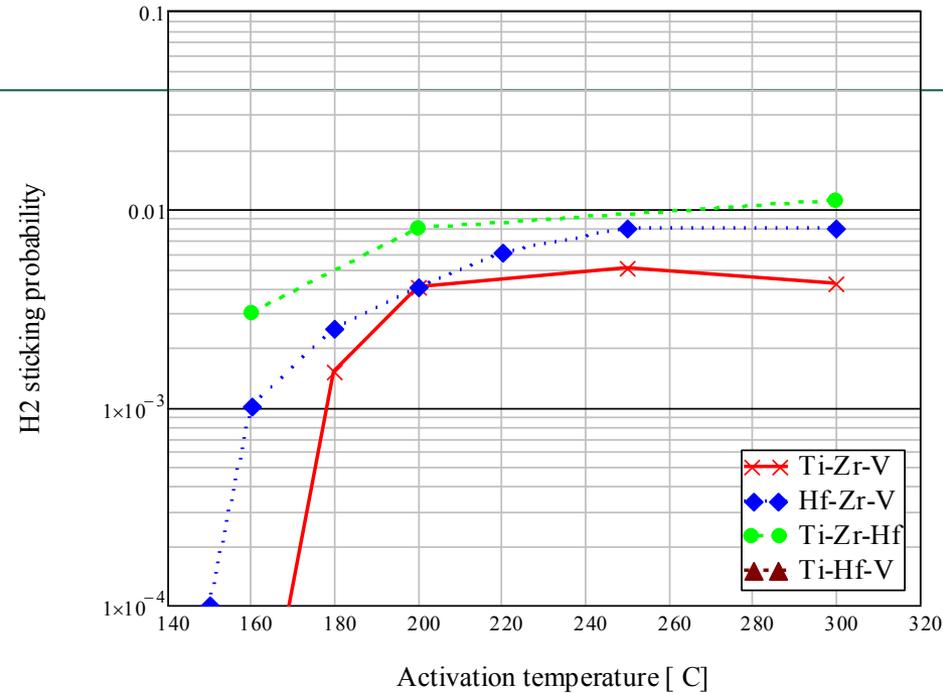
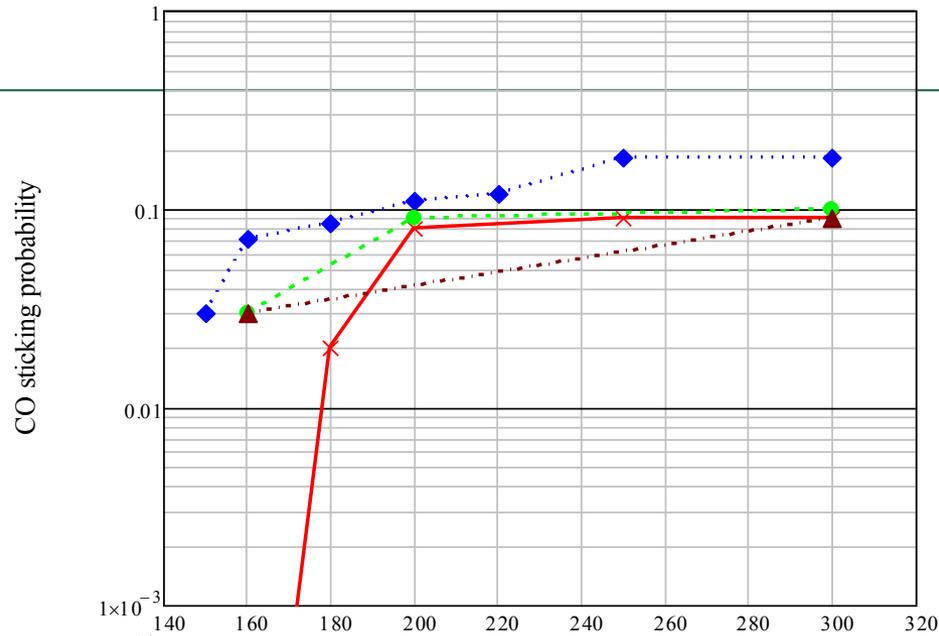
Ti-Zr-V alloy wire



Ti-Zr-V twisted wire



Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.12 nm/s, $T = 120^\circ\text{C}$.
 Average grain size 5 nm. Hexagonal lattice structure.

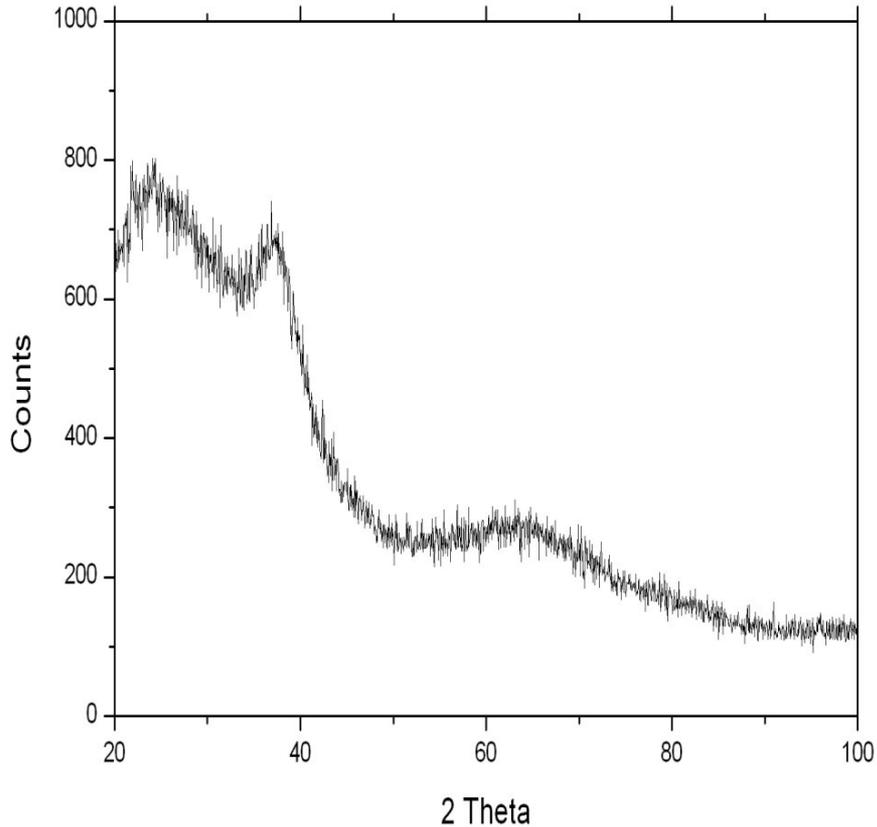


Hf-Zr-V, Ti-Zr-Hf and **Ti-Hf-V** are comparable

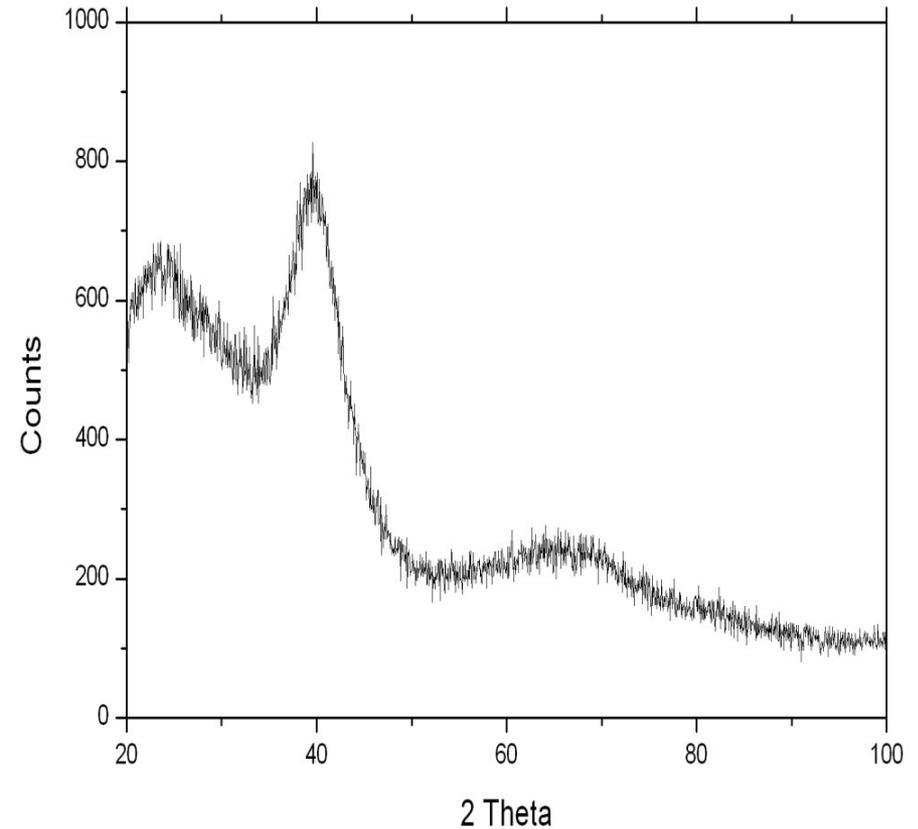
Ti-Zr-V has the highest activation temperature

XRD of Ti-Zr-V film: alloy wire vs. twisted wires as target.

TiZrV Film deposited on Si by cylindrical magnetron using Alloy wire

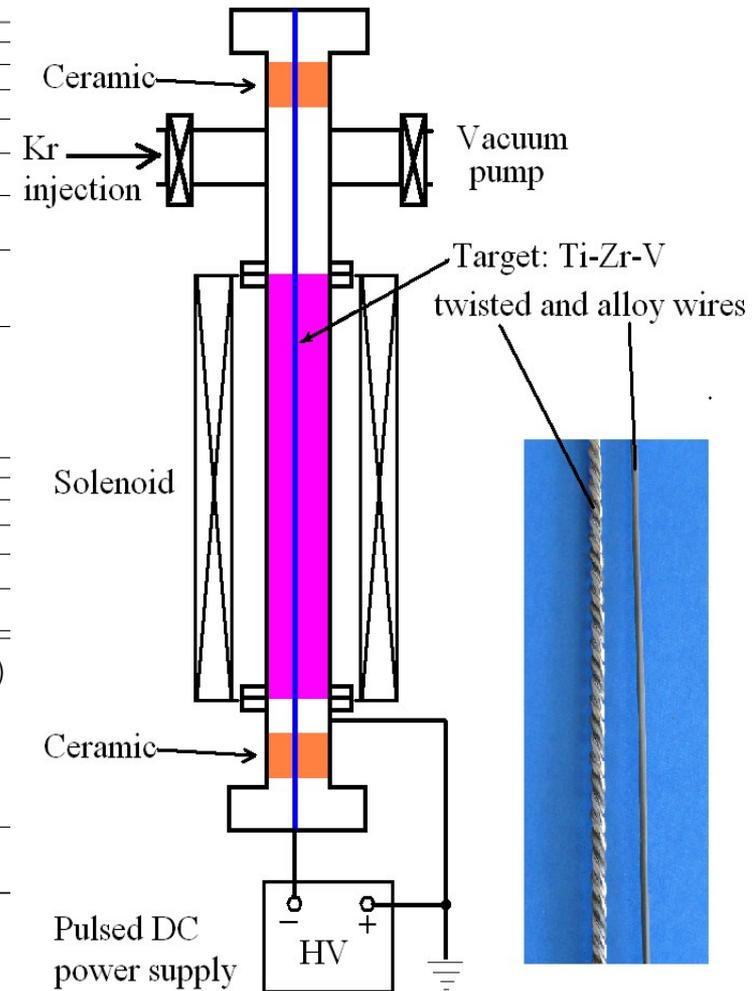
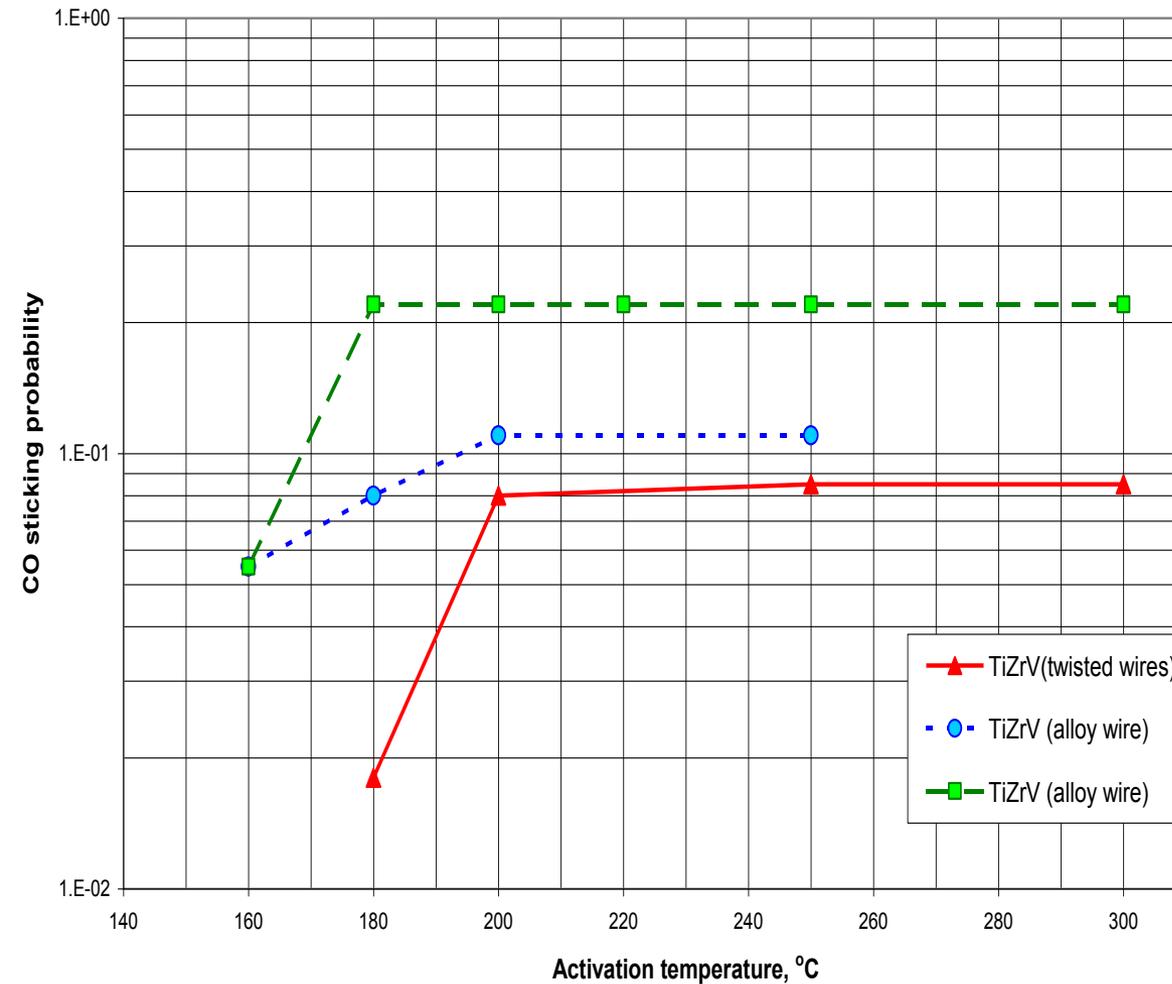


TiZrV Film deposited on Si by cylindrical magnetron using twisted wire



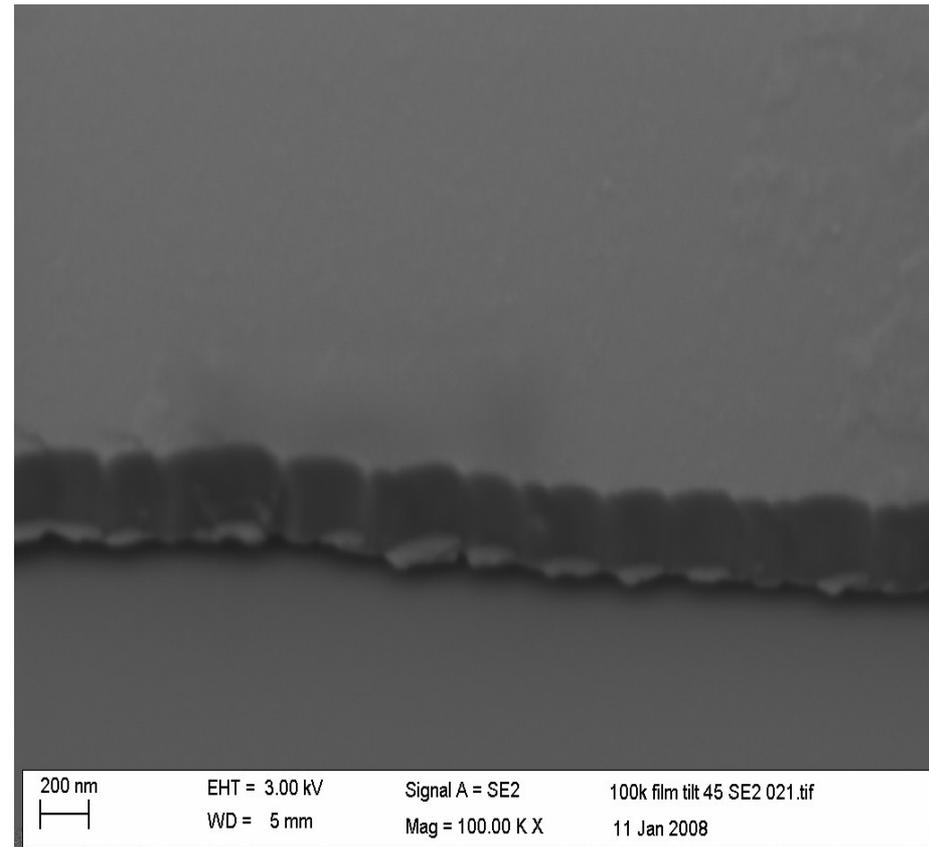
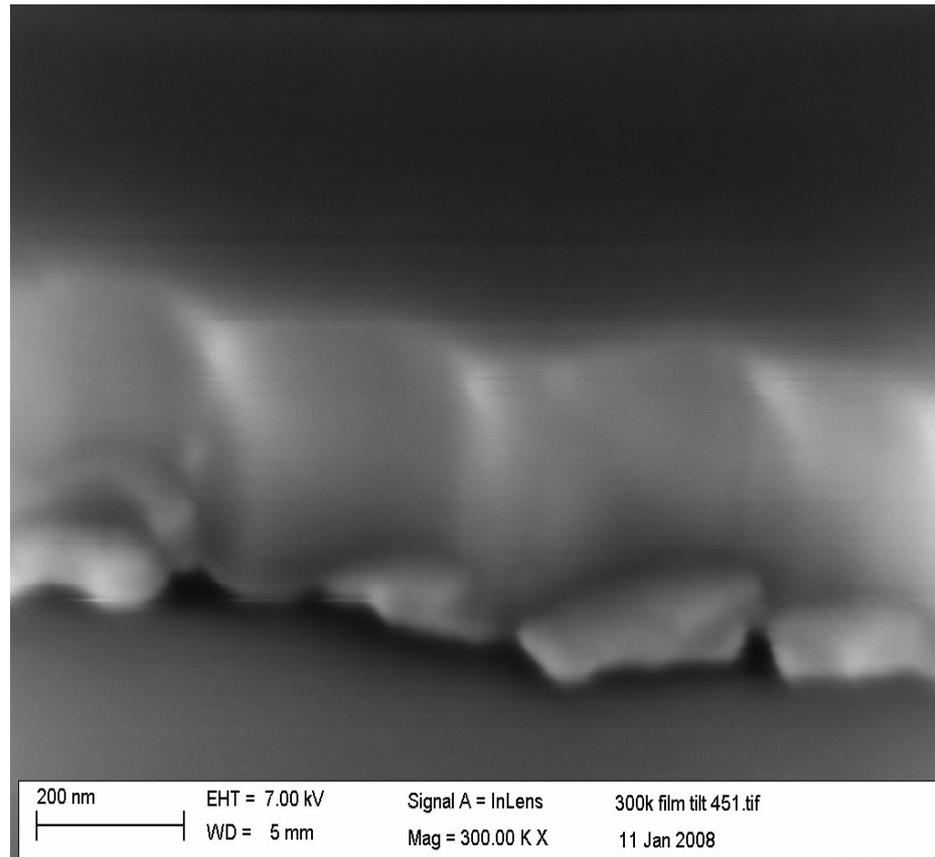
In Both cases there is only one broad peak near $2\theta = 40^\circ$
The film is nearly amorphous.

Twisted wires vs. alloy target: reducing T_a

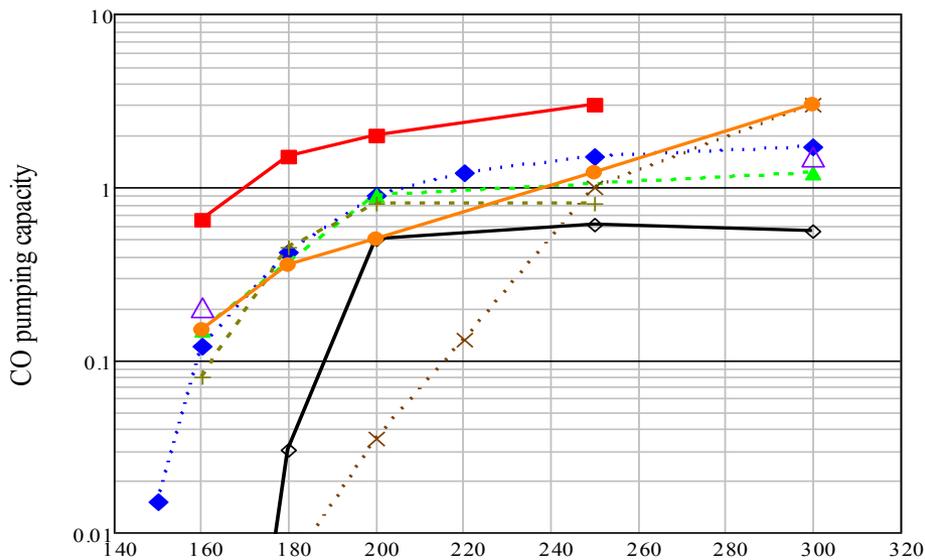
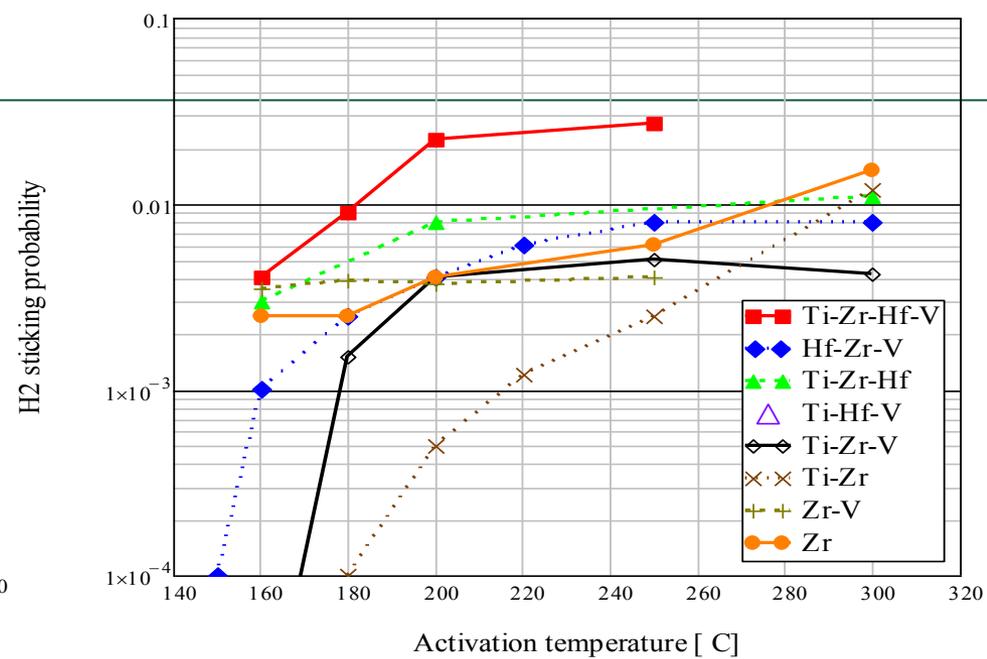
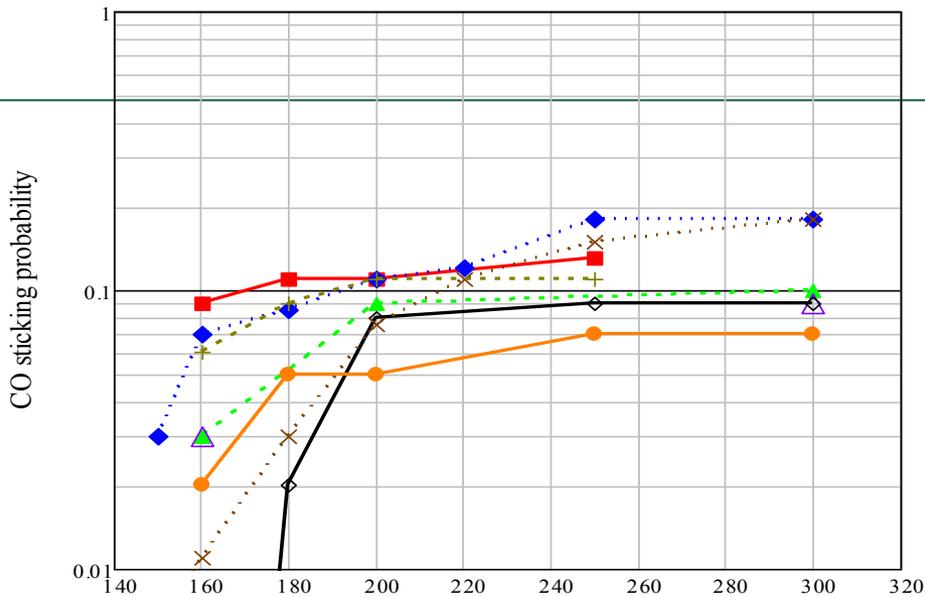


R. Valizadeh, O.B. Malyshev, J.S. Colligon, V. Vishnyakov. Accepted by J. Vac. Sci. Technol. Aug. 2010.

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.



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 pumping properties is limited:

$$\alpha \leq 1.$$

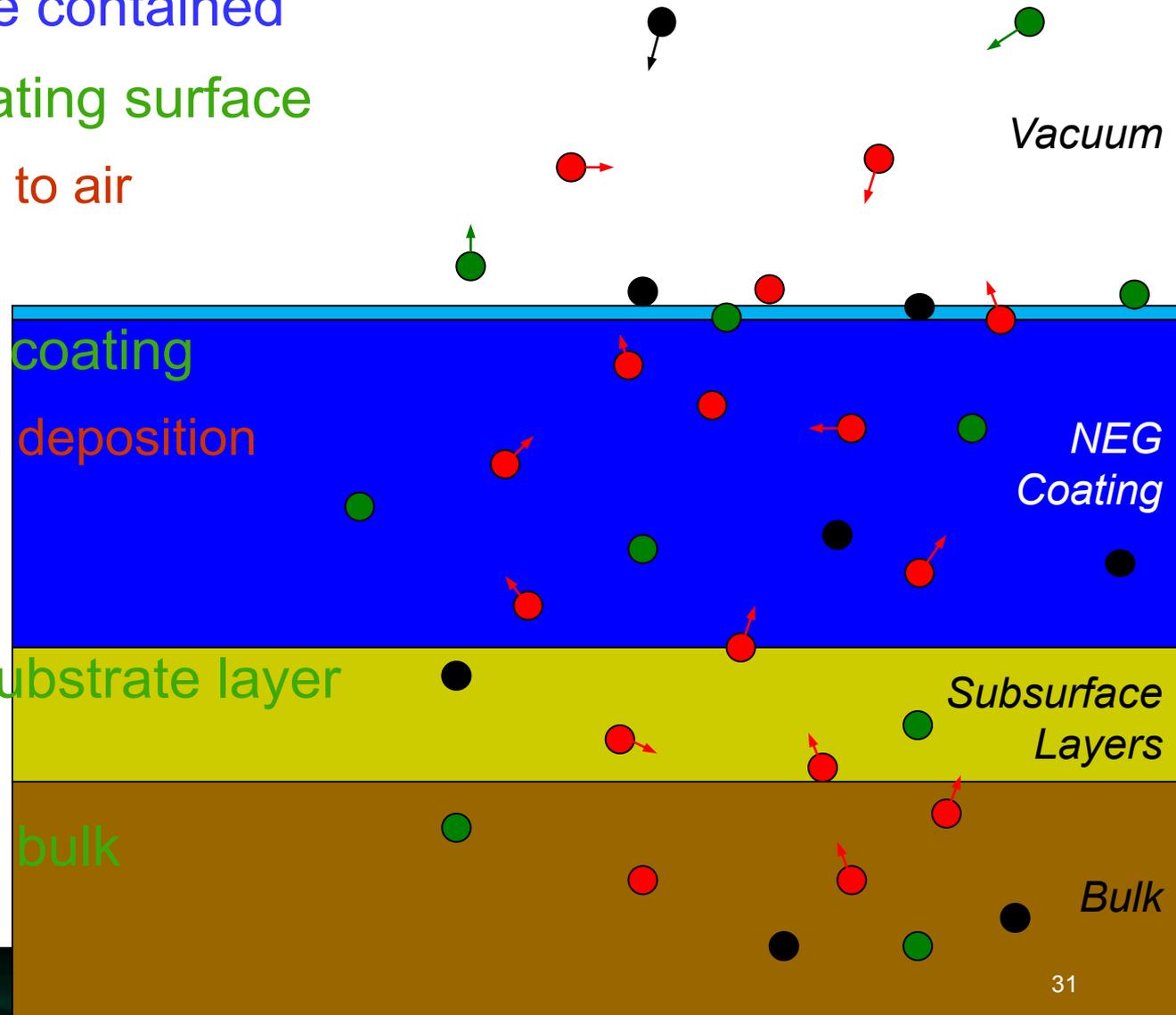
- $0.005 < \alpha_{H_2} < 0.01$
- $0.1 < \alpha_{CO} < 0.5$
- $0.4 < \alpha_{CO_2} < 0.6$
- Reducing the desorption yields η
• in orders of magnitude
is a realistic task

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?

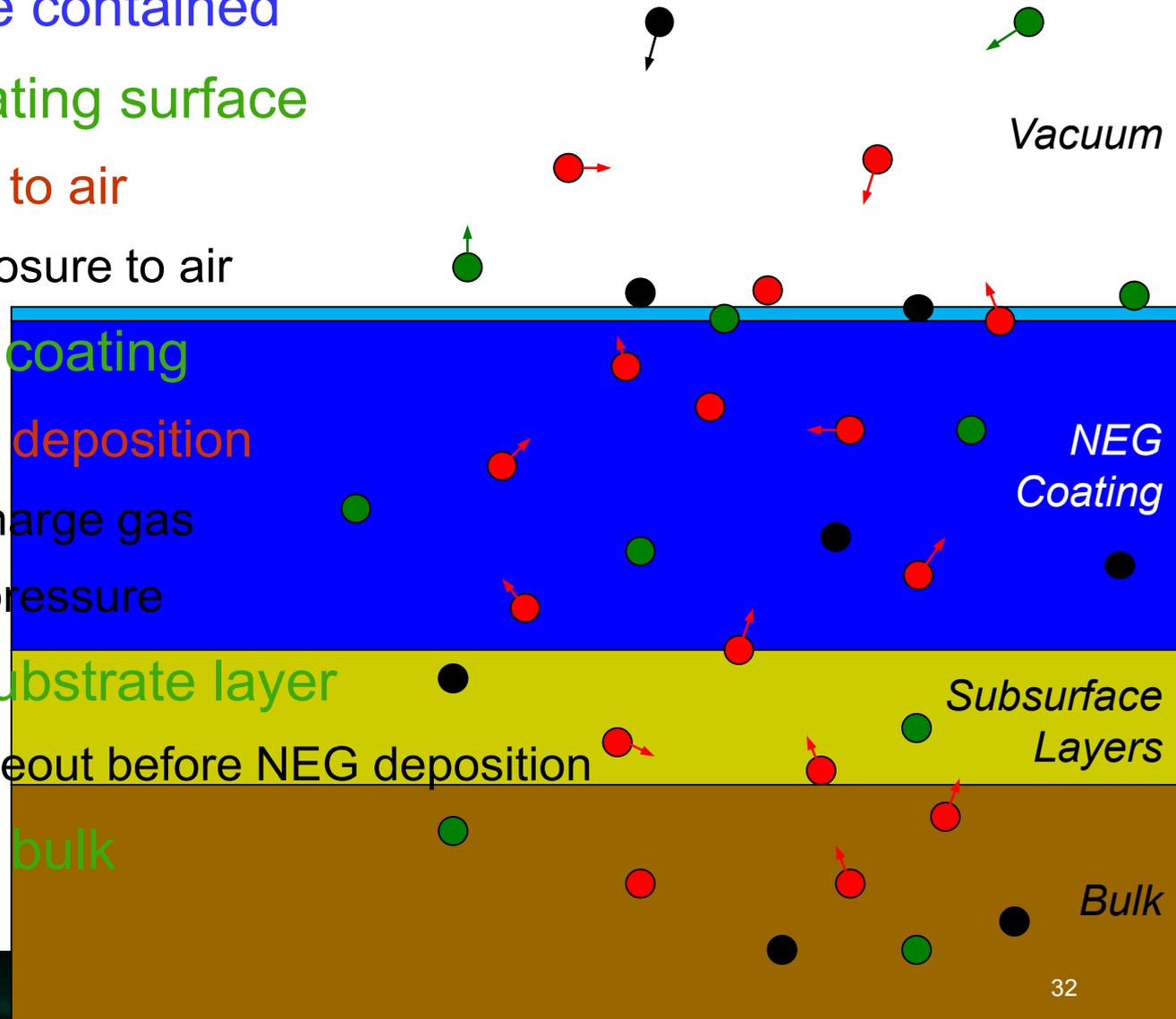
Reducing the gas desorption from the NEG coatings

- Gas molecules are contained
 - on the NEG coating surface
 - after exposure to air
 - inside the NEG coating
 - trapped during deposition
 - in subsurface substrate layer
 - in the substrate bulk



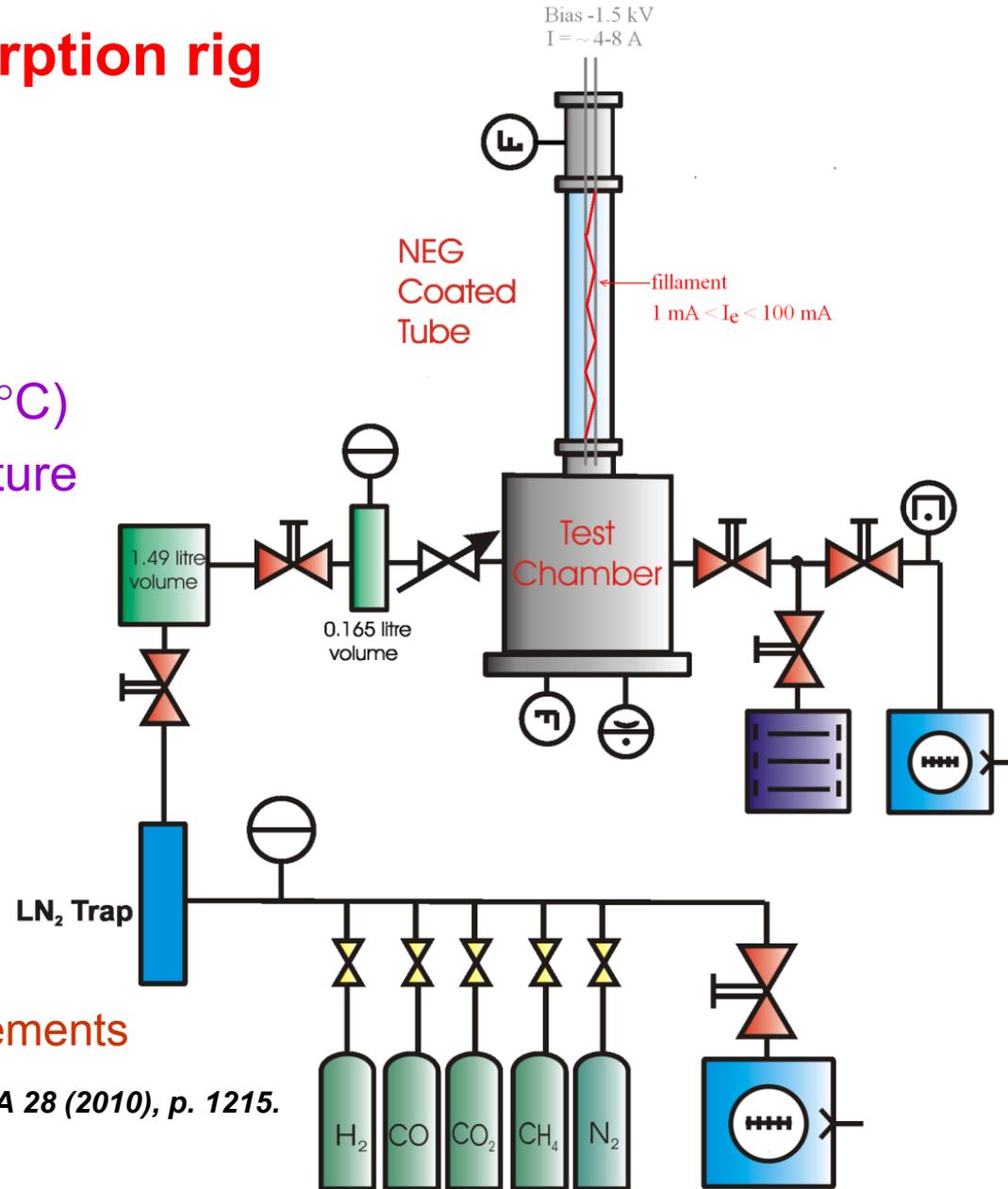
Reducing the gas desorption from the NEG coatings

- 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



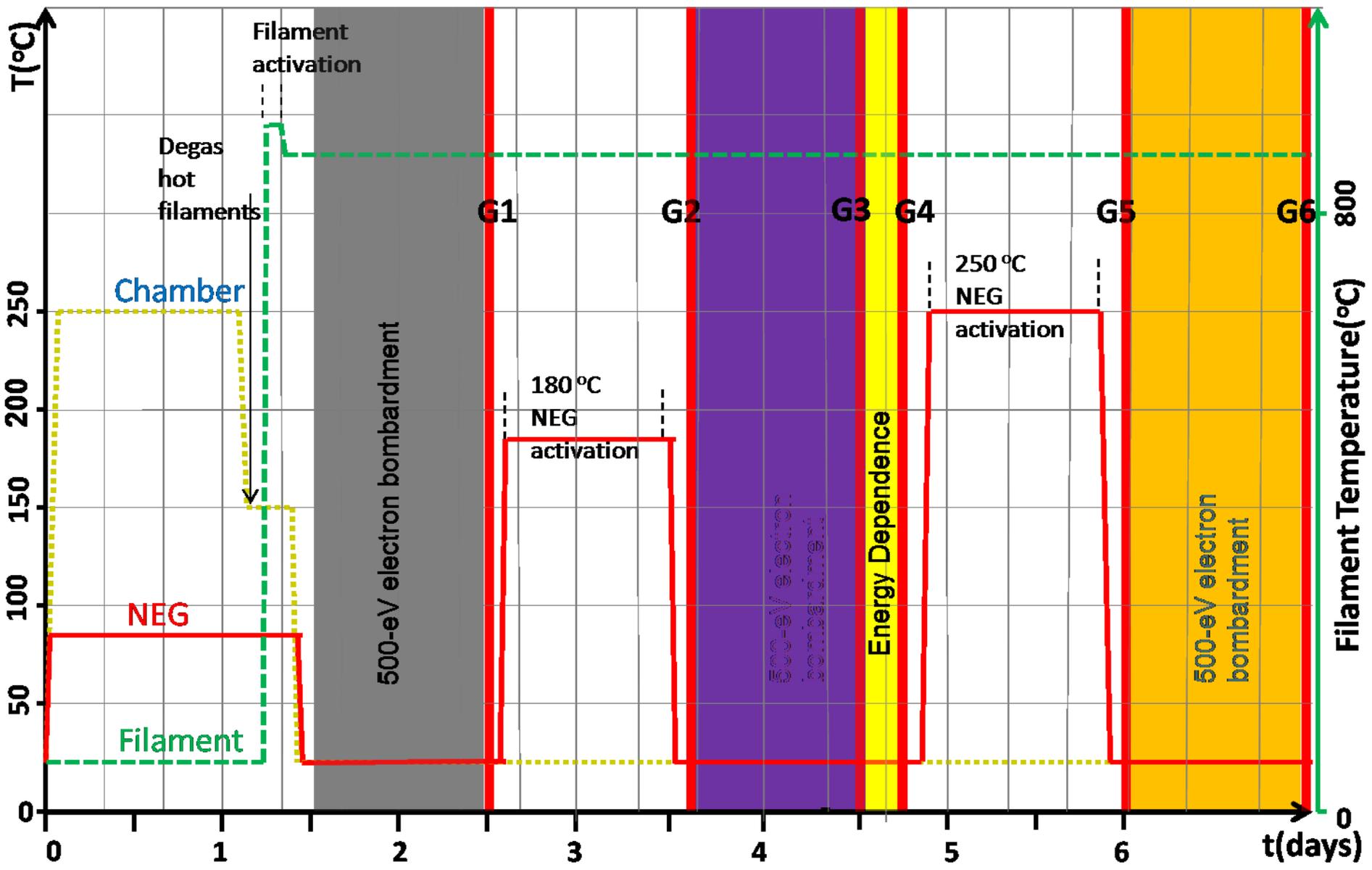
Electron Stimulated Desorption (ESD) studies programme

- ESD from different materials
 - Stainless steel
 - Al, Cu, other materials
 - Coating for low outgassing
 - NEG coated samples
- ESD as a function of
 - Activation/bakeout temperature
 - Electron energy
 - Electron dose
 - Coating density, morphology and structure
 - Wall temperature

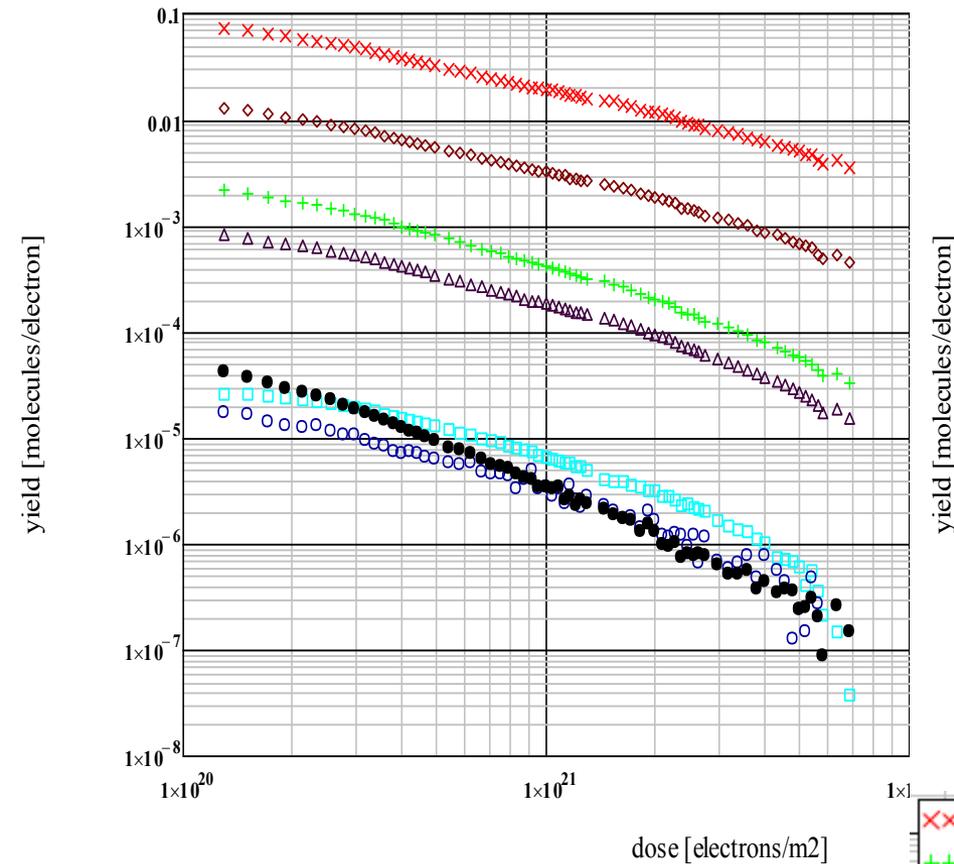
Motivation

- Collecting the data for design of accelerator vacuum systems and comparing different materials used for accelerator vacuum chamber under SR, electron and ion bombardment:
 - SR beamline is not available at DL, and expensive
 - Electrons are easily produced with a filament and bias
 - Ions can also be done but at higher cost
 - Heavy ions with high energies are most expensive
- Reducing the gas desorption from these materials by applying different processes and technologies:
 - Cleaning
 - Polishing
 - Coatings
 - etc.

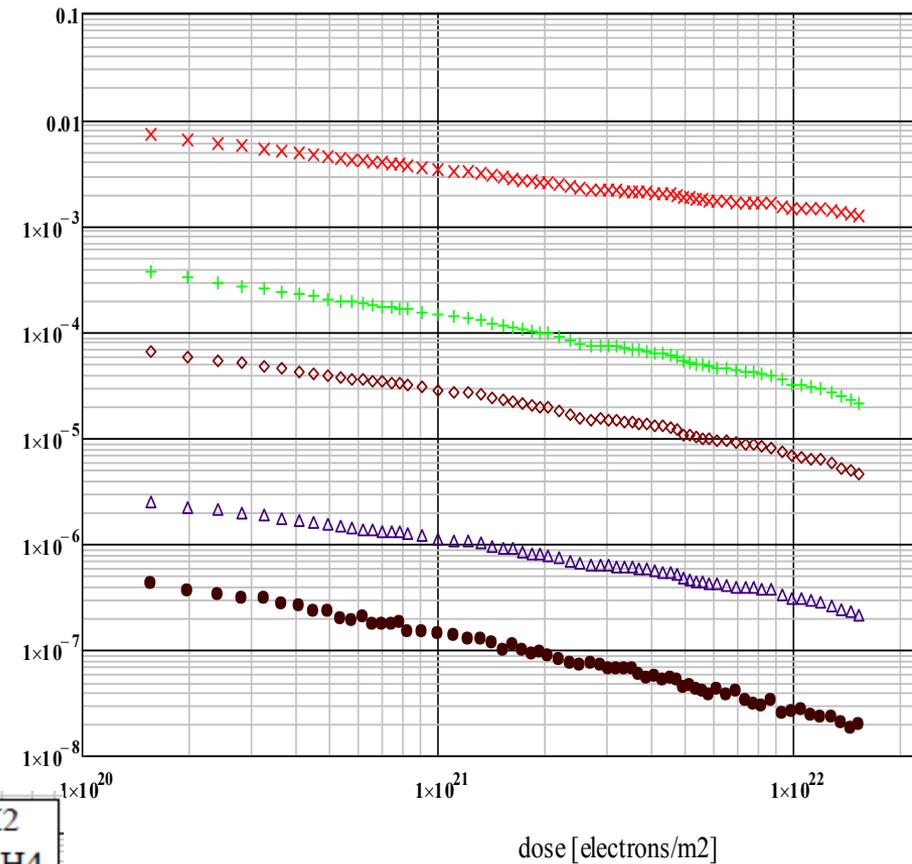
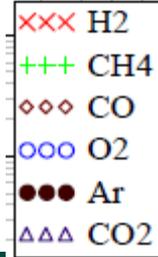
Experimental procedure for NEG coated samples



ESD: stainless steel vs non-activated NEG coated vacuum chamber

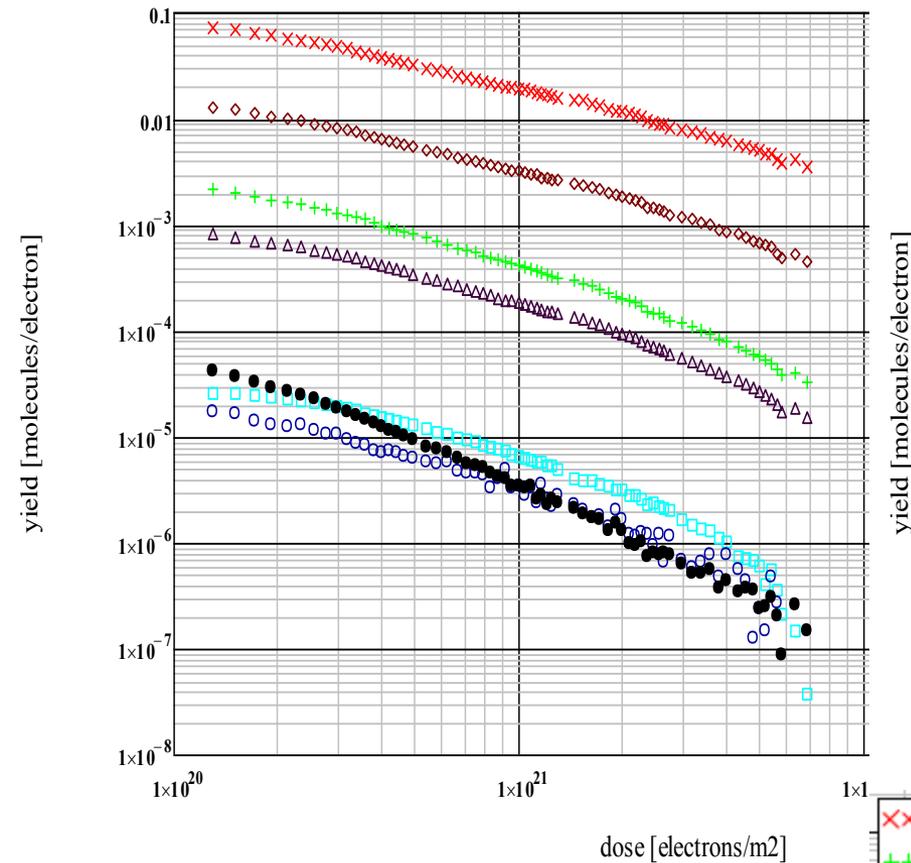


Baked to 250°C for 24 hrs
Pumped for 30 days

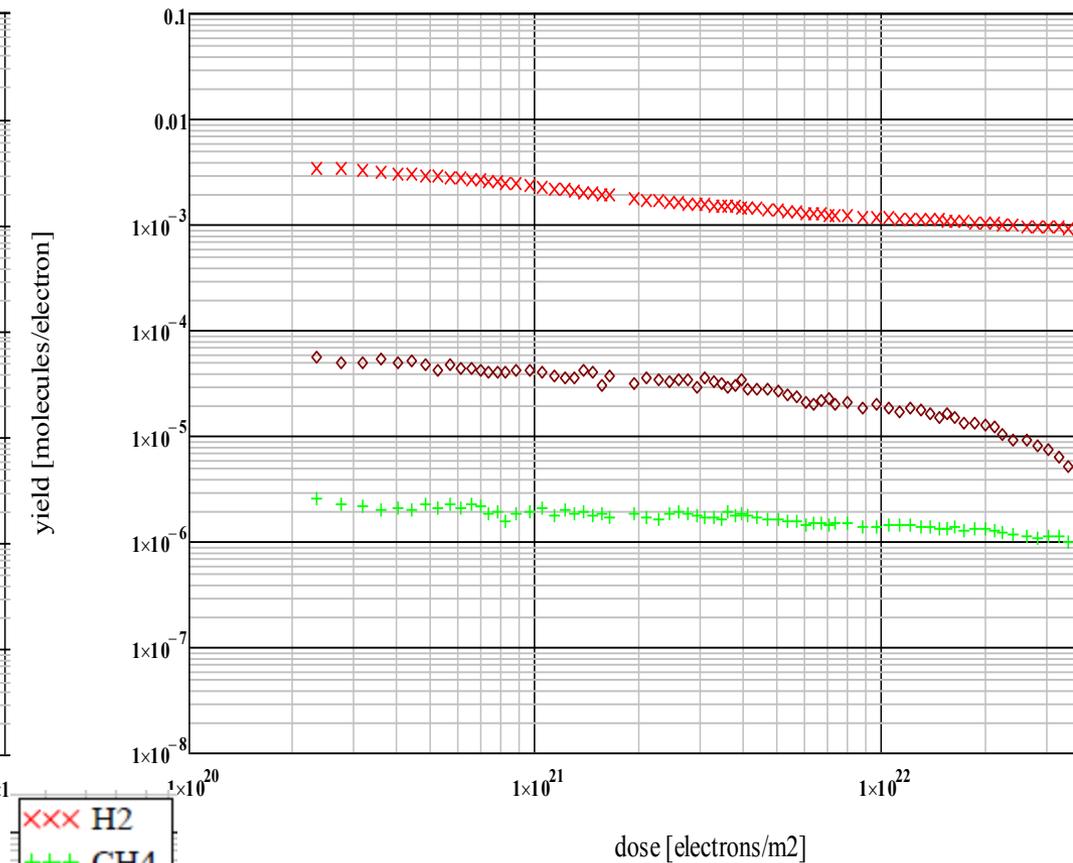


Baked to 80°C for 24 hrs
pumped for 1 day

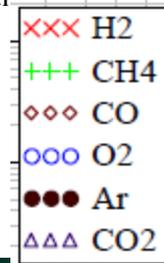
ESD: stainless steel vs activated NEG coated vacuum chamber



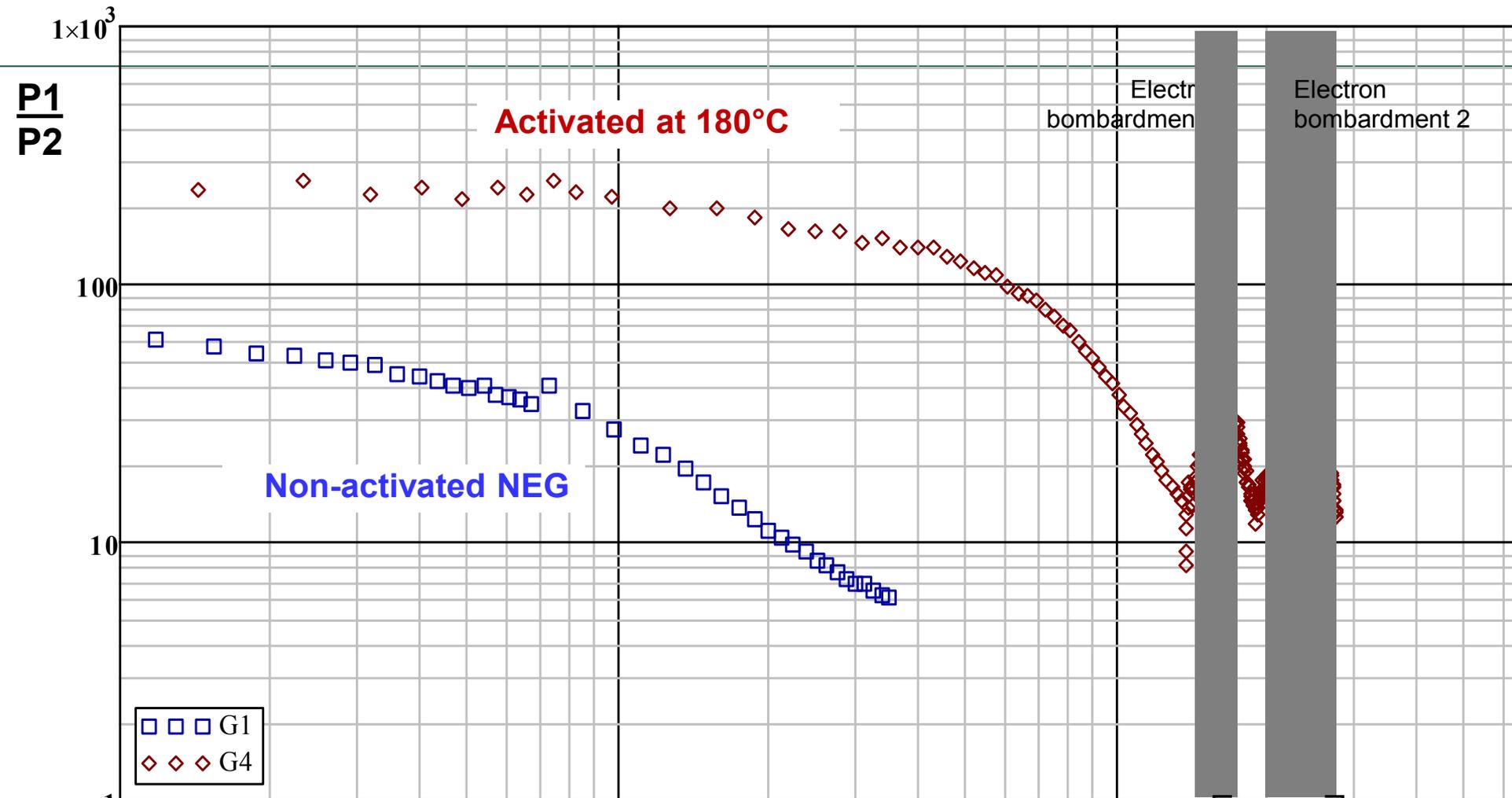
Baked to 250°C for 24 hrs



Activated to 180°C for 24 hrs

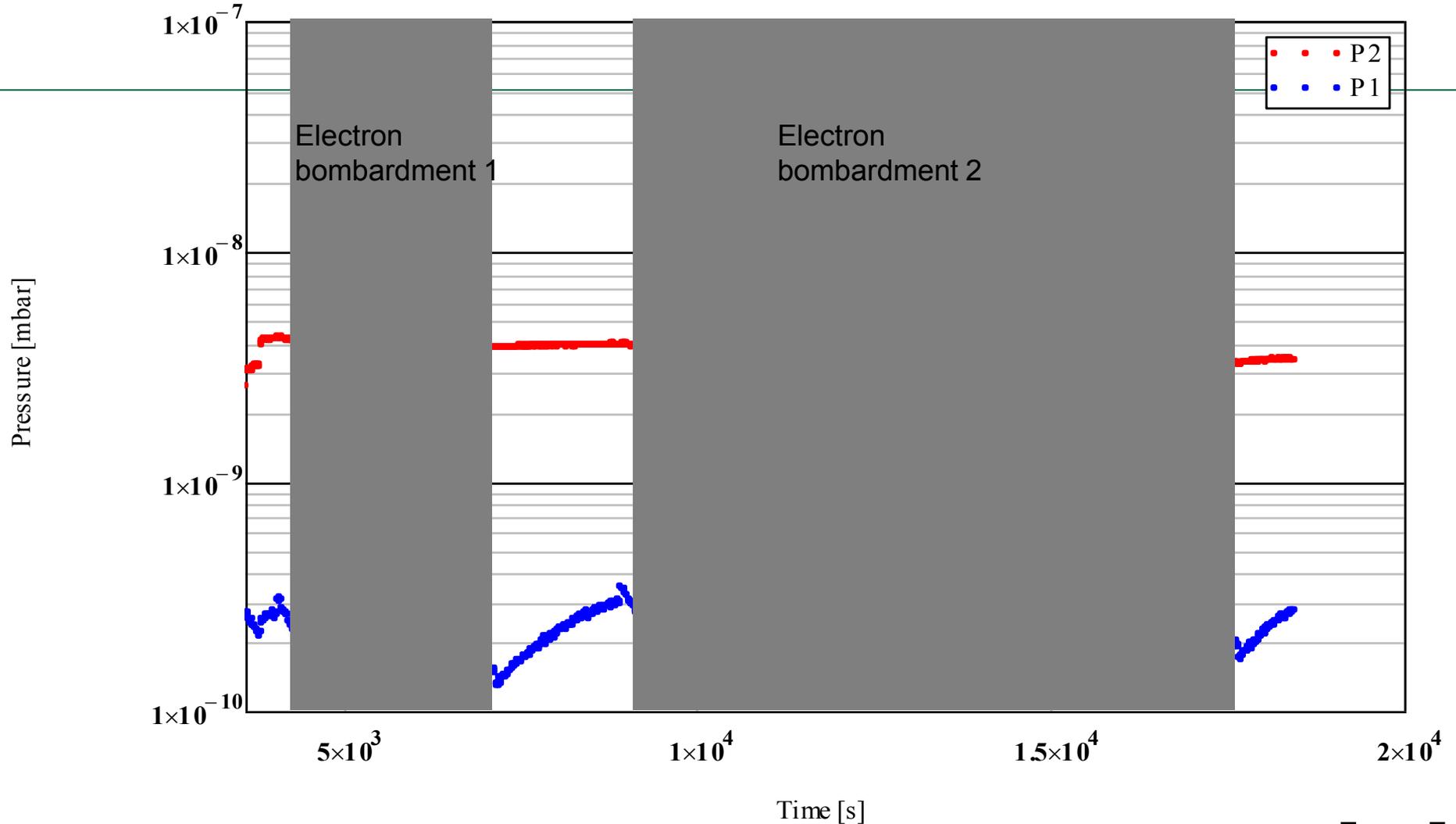


*O.B. Malyshev, A. Smith, R. Valizadeh, A. Hannah.
Accepted by J. Vac. Sci. Technol., Aug. 2010.*



The electron stimulated NEG activation efficiency estimated as $7.9 \times 10^{-4} < \sigma_1 < 2.4 \times 10^{-3} \text{ [CO/e}^-]$

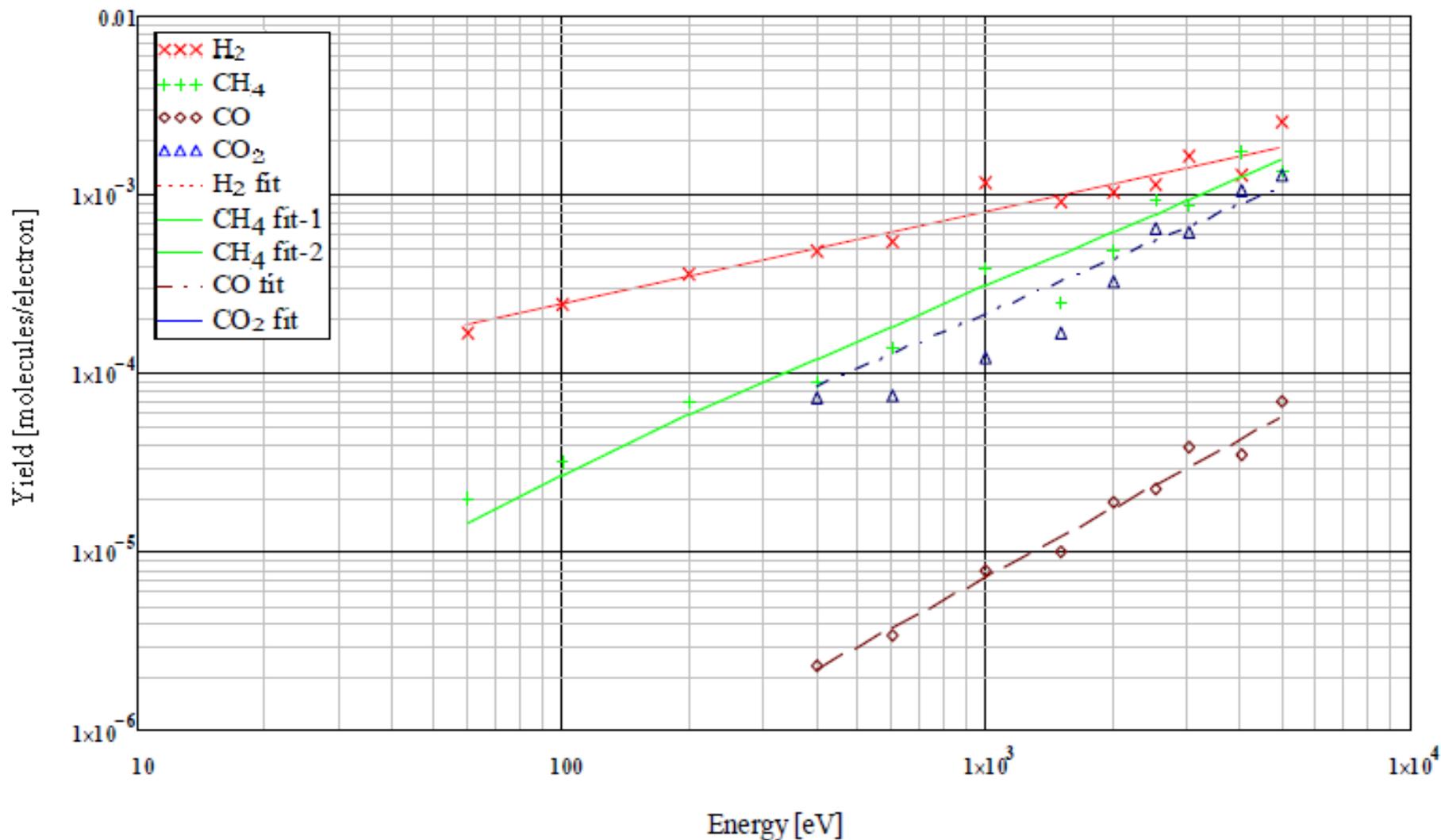
$$\sigma_1 \frac{CO}{e^-} = \frac{\Omega_{CO}}{D}$$



The electron stimulated NEG activation efficiency estimated as

$$\sigma_2 = \frac{Q_{CO}}{k_B T} \frac{q_e}{I} = 2.2 \times 10^{-3} \left[\frac{CO}{e^-} \right]$$

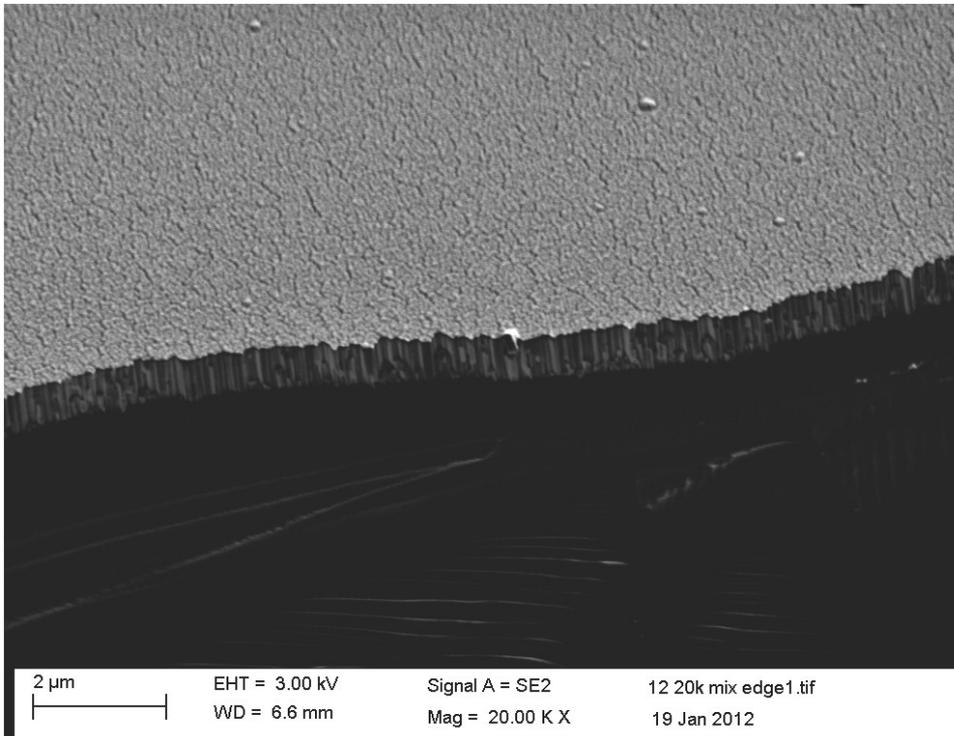
$\eta(E_e)$ for different gases for NEG coating



SEM images of films (film morphology)

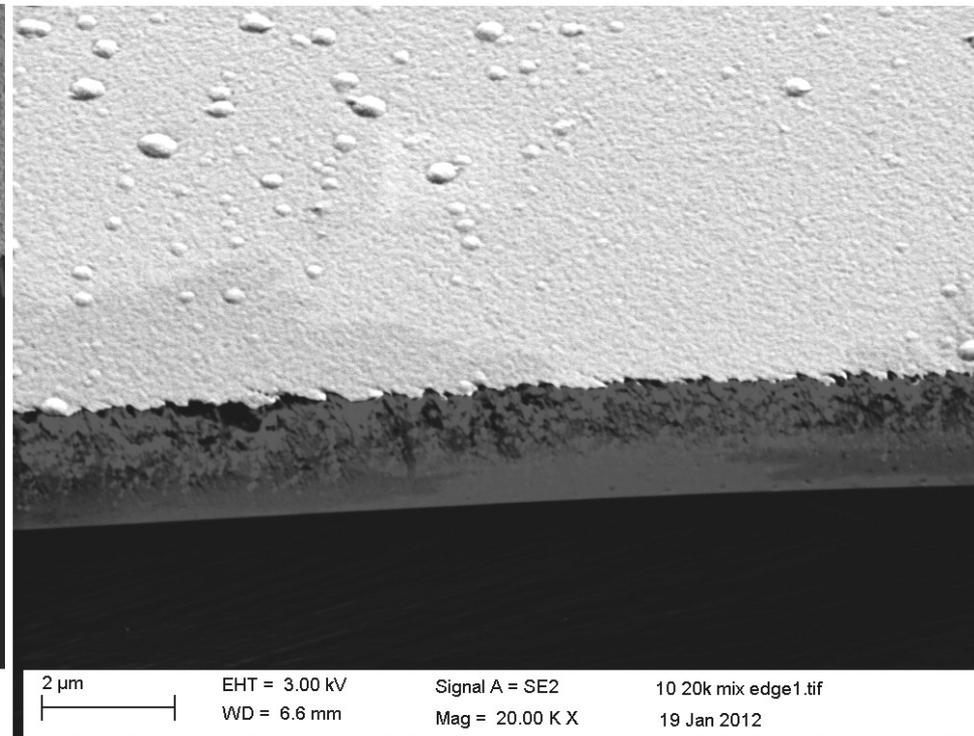
columnar

Best for pumping

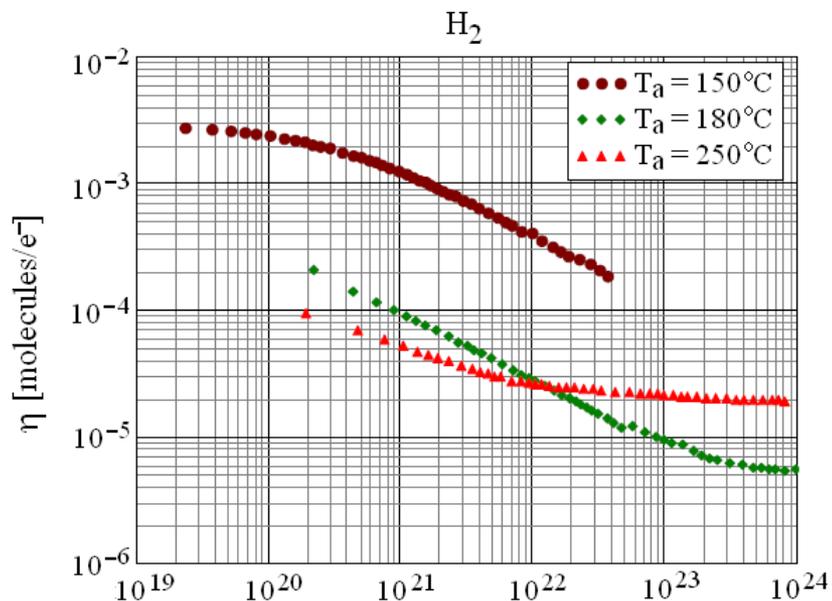
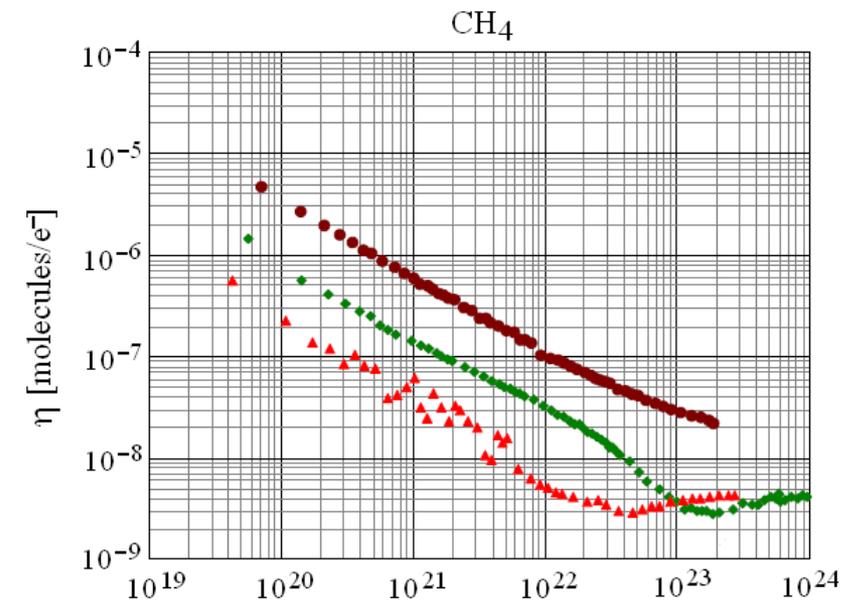
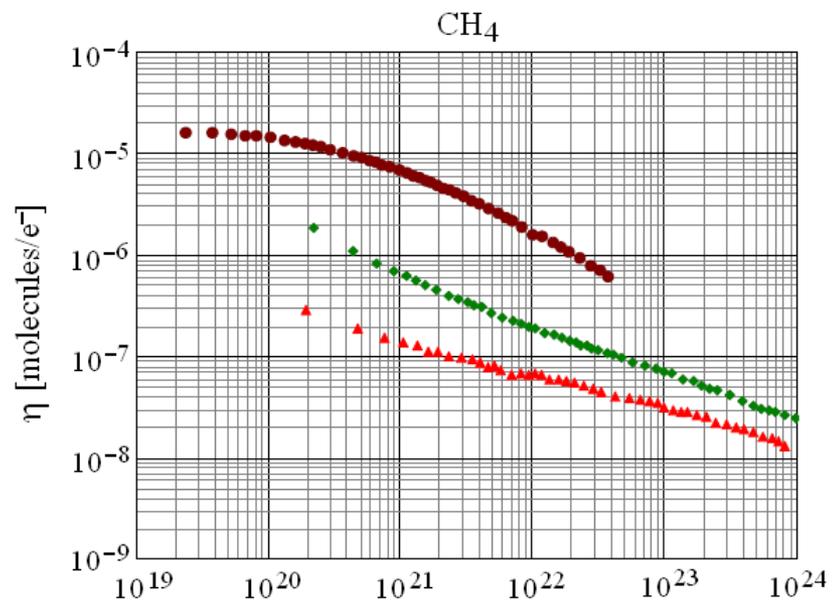
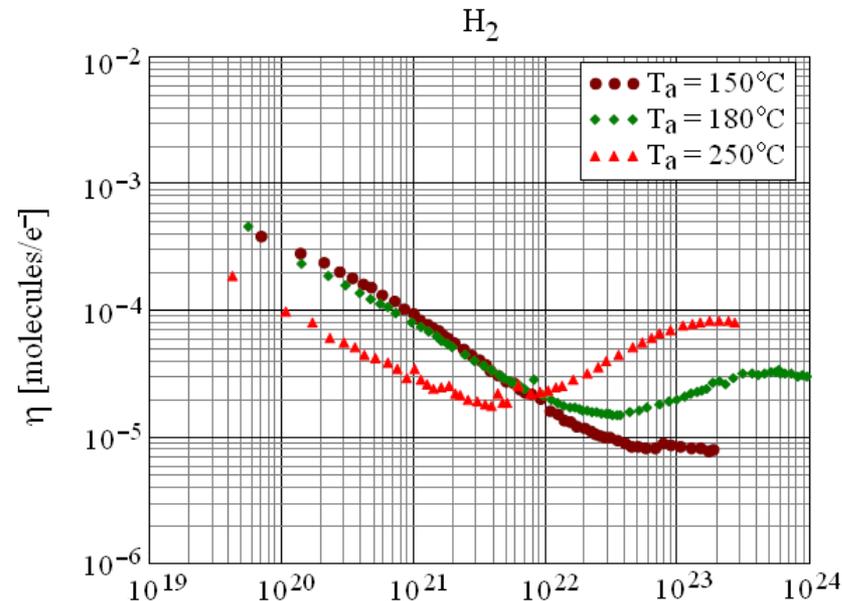


dense

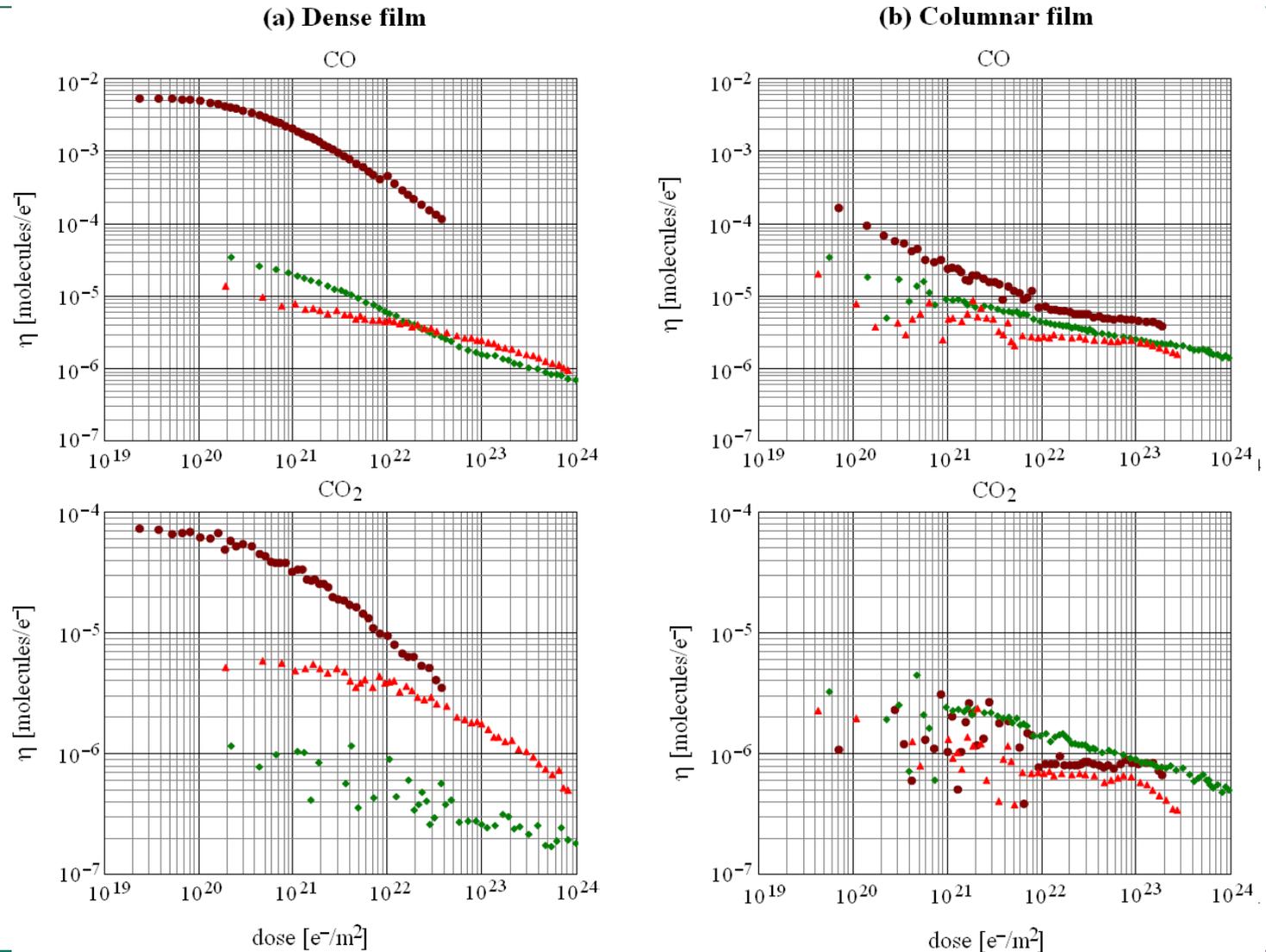
A first candidate for a barrier



O.B. Malyshev et al. Vacuum 86 (2012) 2035-2039

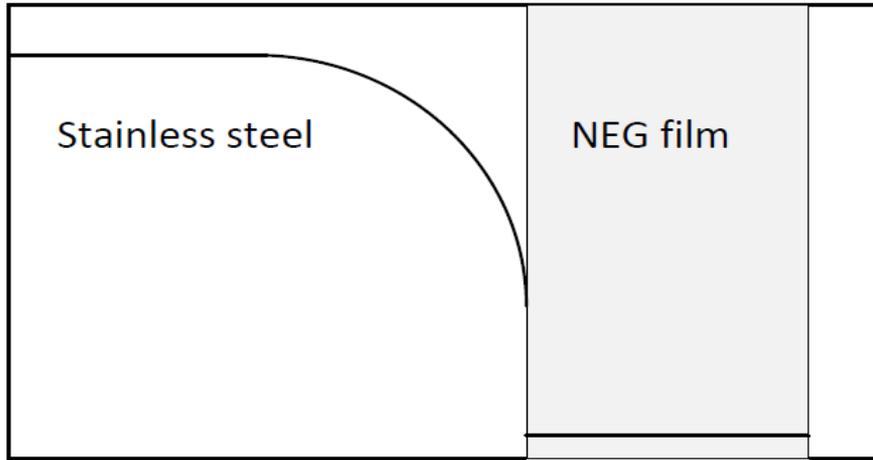
(a) Dense film

(b) Columnar film


ESD yield from NEG coated samples

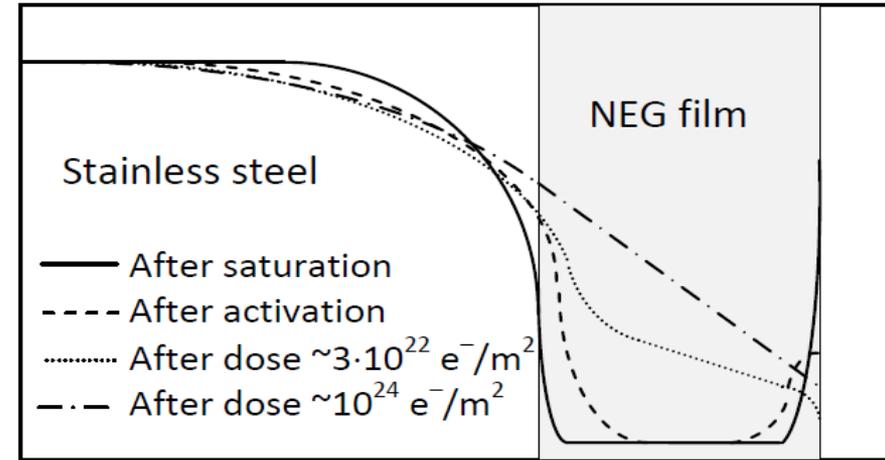


ESD yield from NEG coated samples

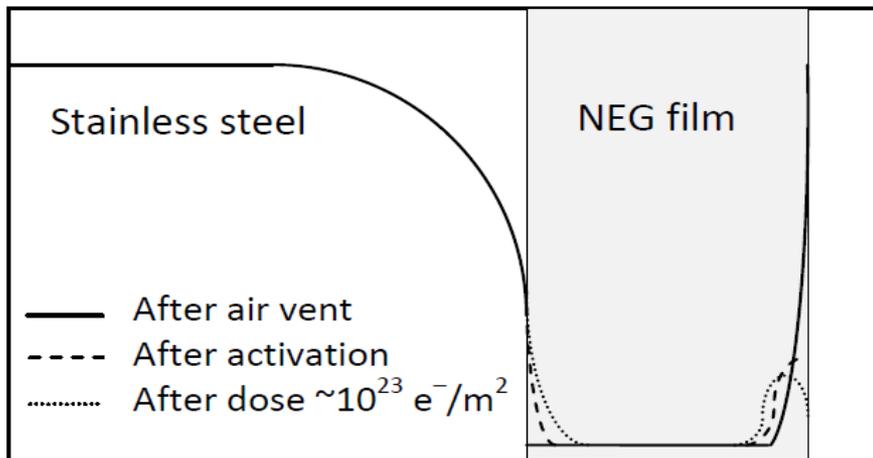
(a) after deposition before vent to air



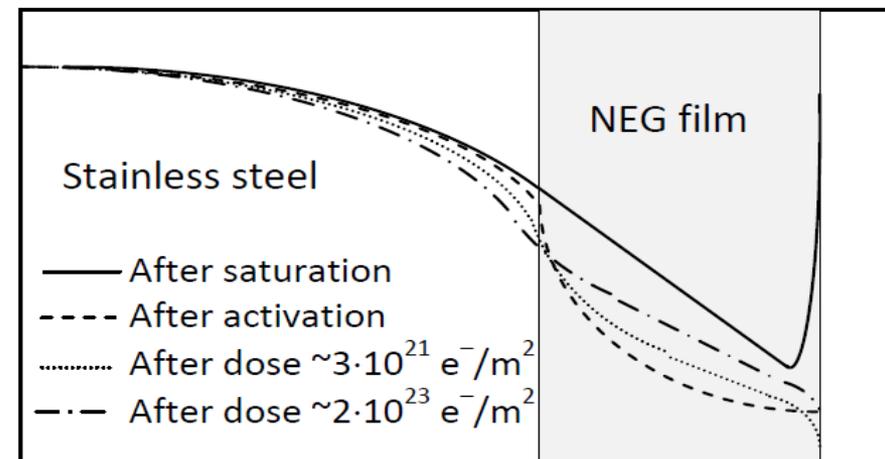
(c) activation at 180 °C



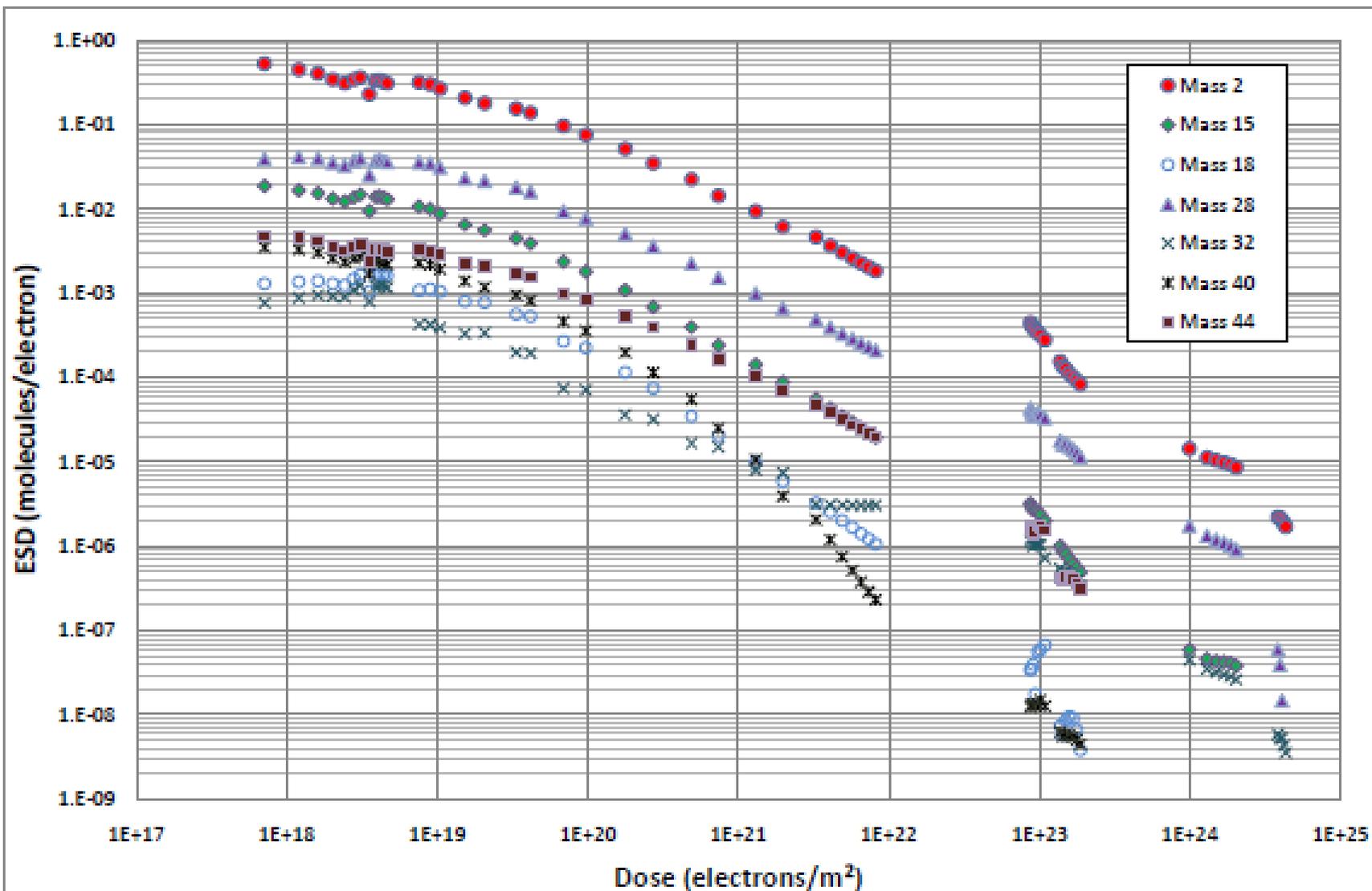
(b) activation at 150 °C



(d) activation at 250 °C

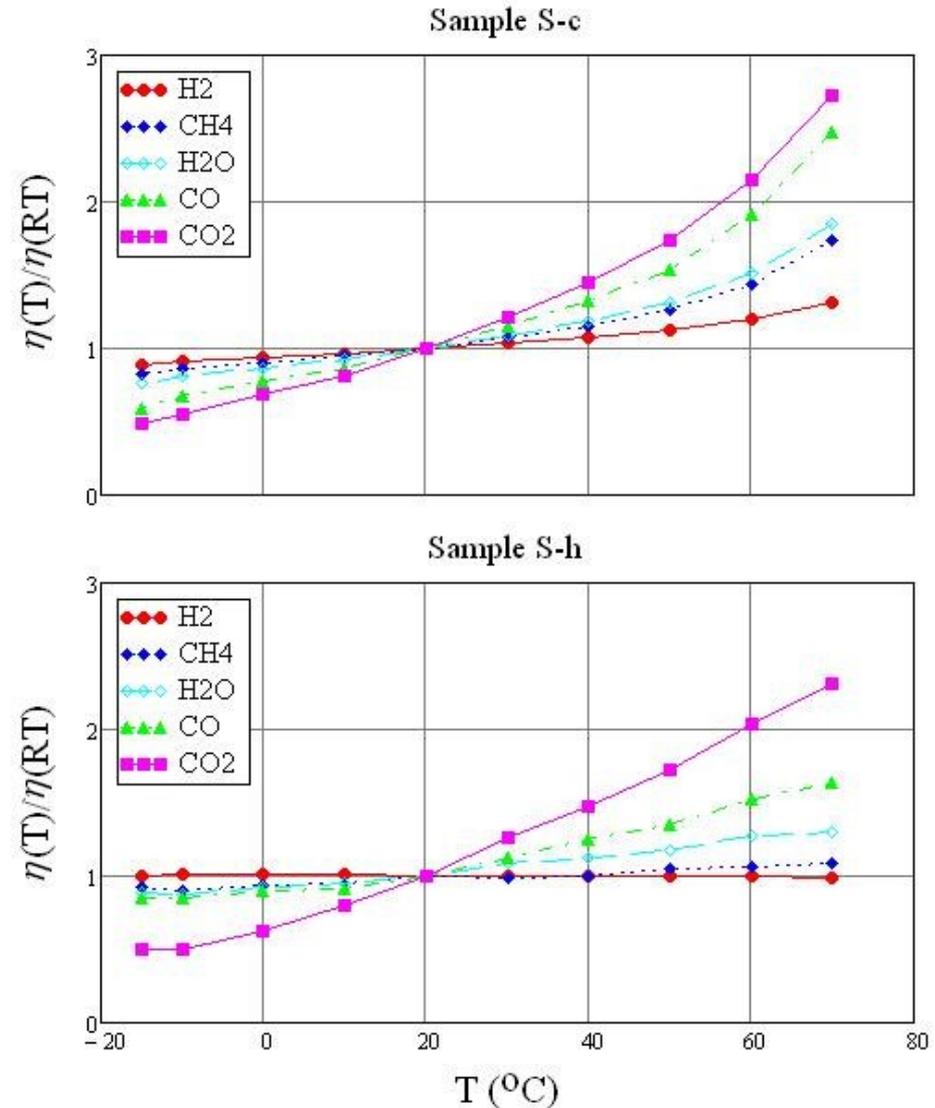


EDS as a function of dose for 316L stainless steel

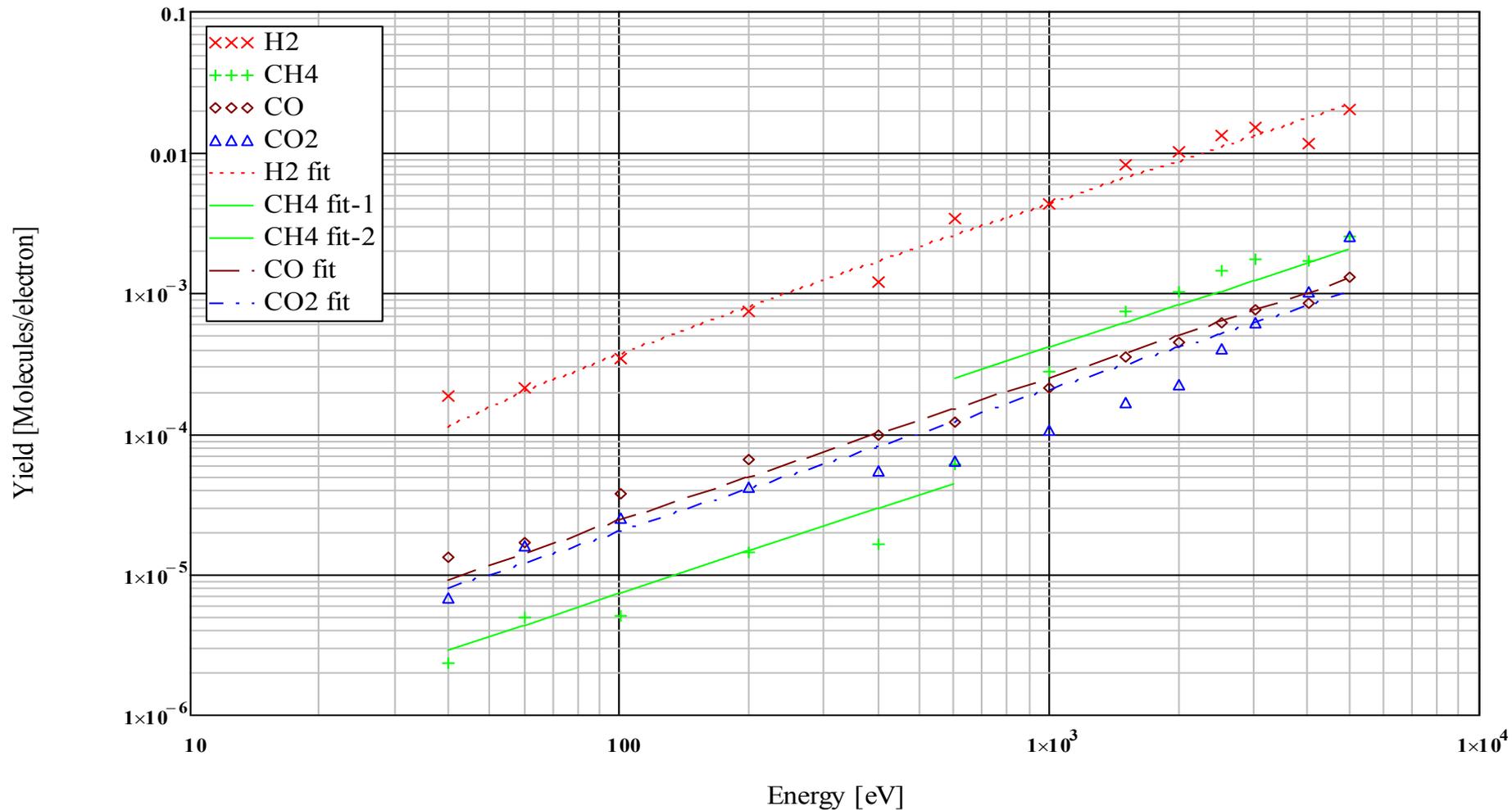


EDS as a function of temperature

- 3 samples were bombarded with electrons with $E = 500$ eV at
 - $T = -15$ °C
 - $T = +20$ °C
 - $T = +70$ °C
- after large dose (10^{23} - 10^{25} e⁻/m²) each sample temperature was varied between
 - -15 °C < $T = +70$ °C

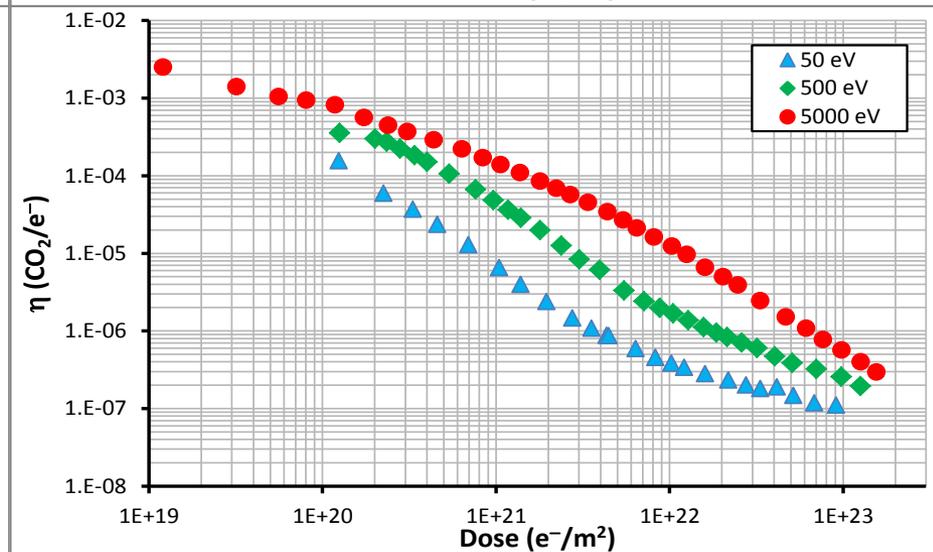
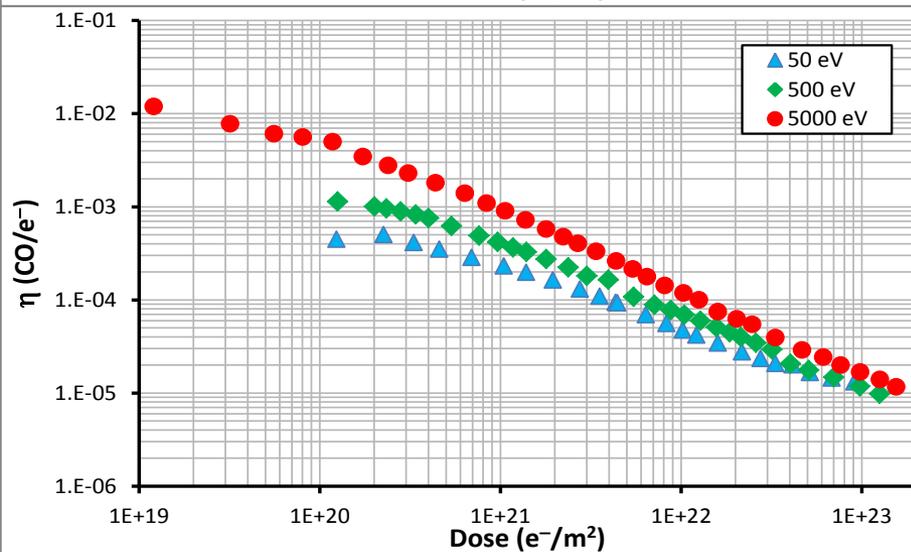
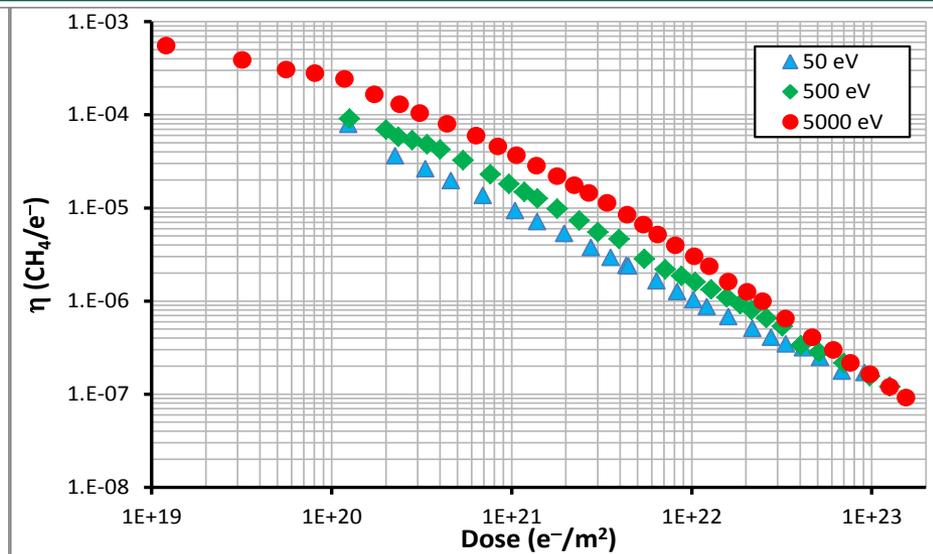
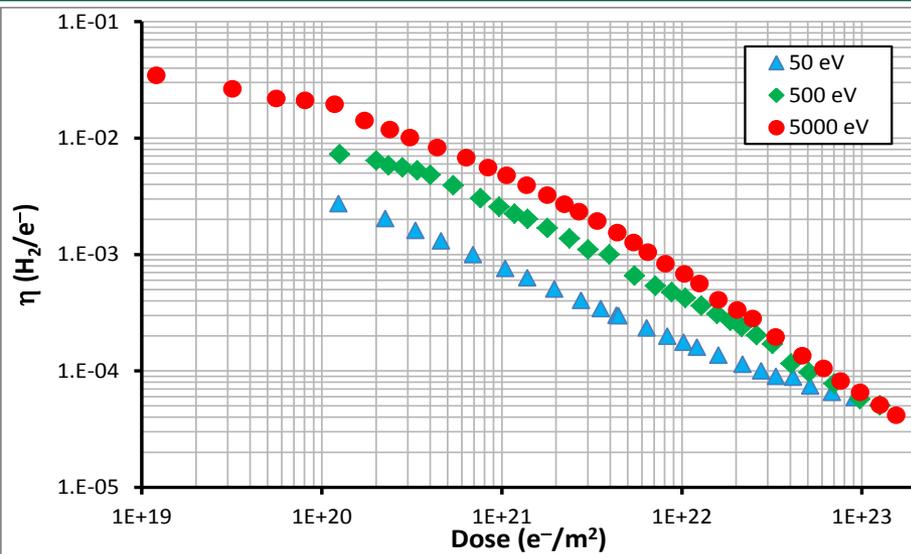


$\eta(E_{e^-})$ for different gases for 316LN

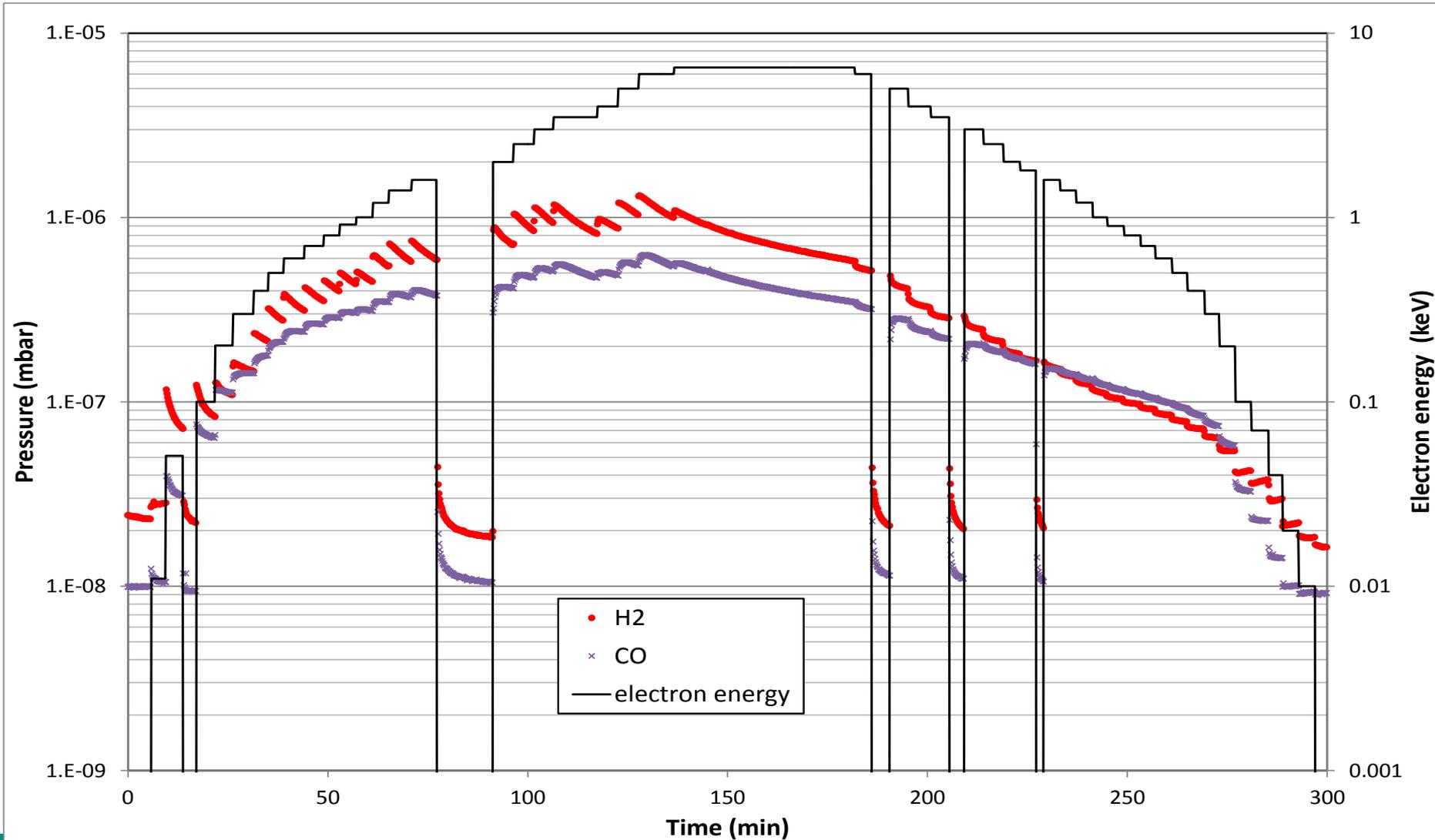


O.B. Malyshev, A.P. Smith et al. *J. Vac. Sci. Technol. A* 28 (2010), p. 1215.

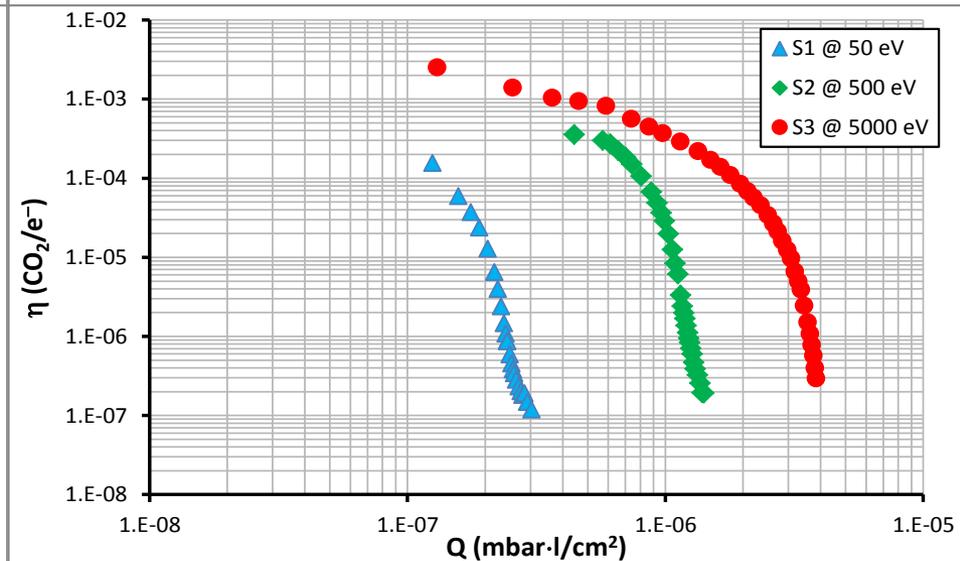
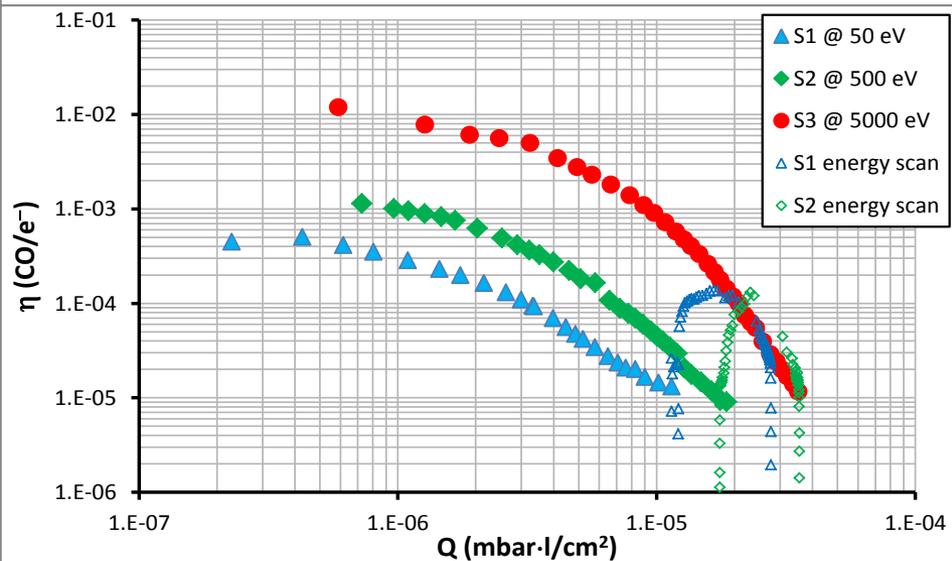
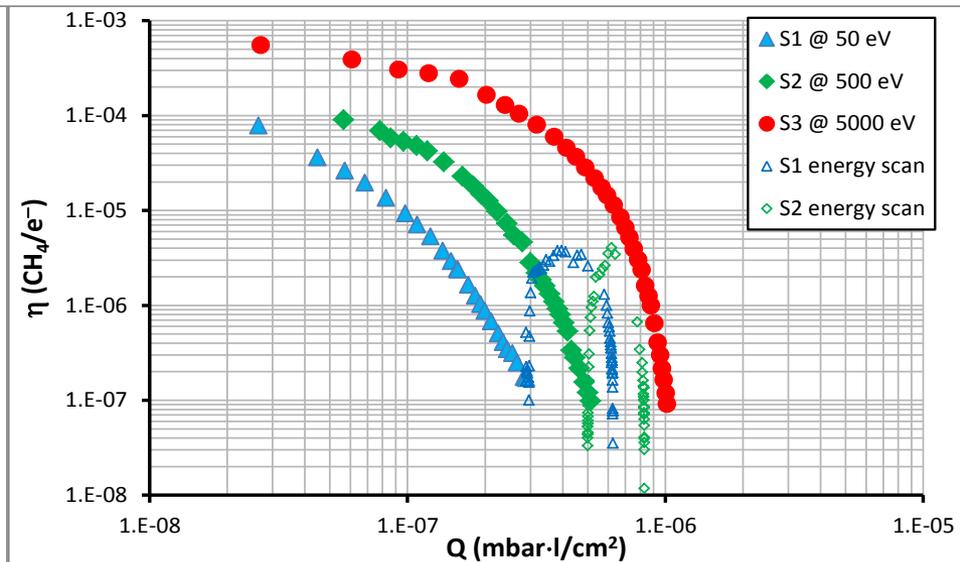
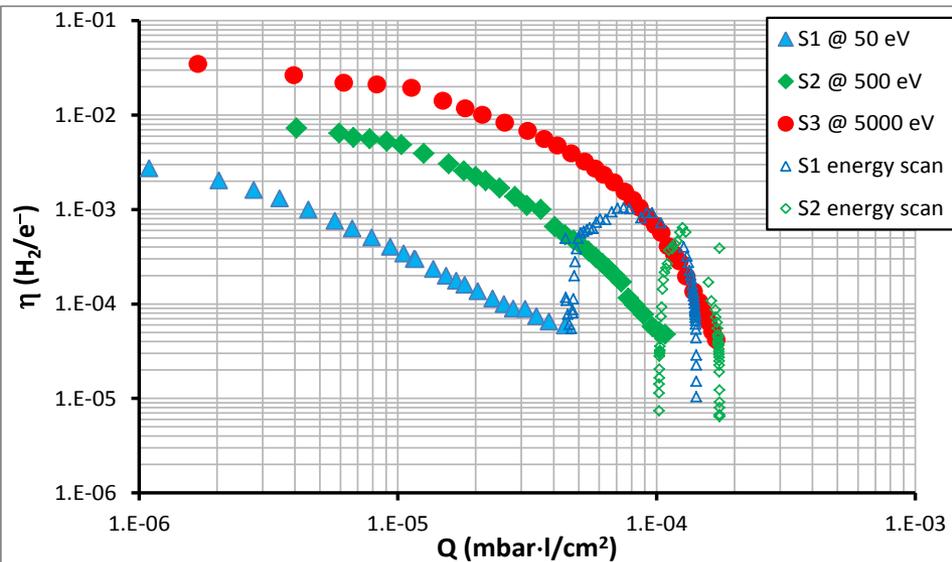
ESD as a function of dose and energy



Pressure as a function of time and energy



ESD as a function of dose and energy



EDS as a function of pumping time after bakeout

- 316 LN stainless steel
 - ESD yields from a sample pumped for 24 days is ~10 smaller than for from a sample pumped for 24 hours (1 day)
- Al
 - ESD yields from a sample pumped for 26 days is very similar to a sample pumped for 1 day
 - O.B. Malyshev et al. Vacuum 85 (2011) p. 1063.

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 die 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.

Conclusions (2):

- An ESD set-up for tubular sample
 - Uniform bombardment along the tube
 - From both pumping and non-pumping samples.
- The ESD yields as a function of electron dose :
 - 316L stainless steel sample after bakeout at 250°C
 - Ti-Zr-V coated before NEG activation and after activation at 180°C and 250 °C.
 - Desorption yields from SS are comparable with earlier results from literature;
 - The initial desorption yields from NEG coating are 20 times lower for H₂, 1000 times lower for CH₄ and 200 times lower for CO, the desorption yields for other gases below the installation sensitivity.
- The ESD yields as a function of electron energy:
 - were measured in the energy range between 40 eV and 6.5 keV.
 - ESD yield increases with energy
- The electron bombardment induced pumping of the CO saturated NEG film was observed for a first time
 - this effect is similar to photon induced pumping of the NEG film observed earlier.

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Acknowledgments

Co-authors (team):

ASTeC

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- Mr. A. Smith
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- Dr. S. Patel

MMU

- Prof. J.S. Colligon
- Dr. V. Vishnyakov

