Science & Technology Facilities Council

Progress in NEG Coatings for Particle Accelerators

O.B. Malyshev and R. Valizadeh,

ASTeC Vacuum Science Group,
STFC Daresbury Laboratory, UK

ASTeC

7th Low Emittance Rings Workshop

15-17 January 2018
CERN, Geneva, Switzerland

Outlook

- Introduction
- Pumping properties
- Desorption properties
- Bombardment induced activation and pumping
- Surface resistance
- SEY
- Summary

Science & Technology Facilities Council

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

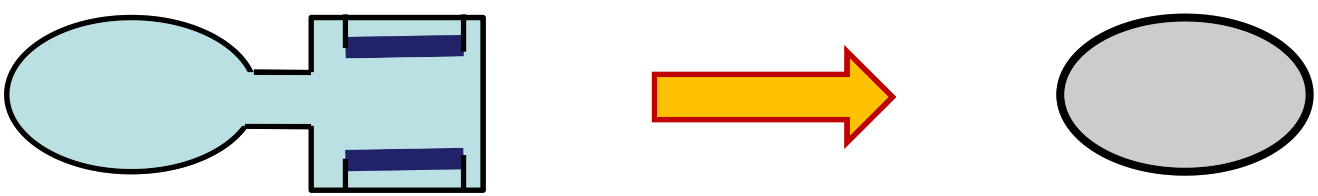
- Surface cleaning, conditioning, coatings
- Vacuum firing, *ex-situ* baling
- Baking *in-situ* to up to **300°C**
- Separate pumps

'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 NEG coating does

1) Reduces gas desorption:

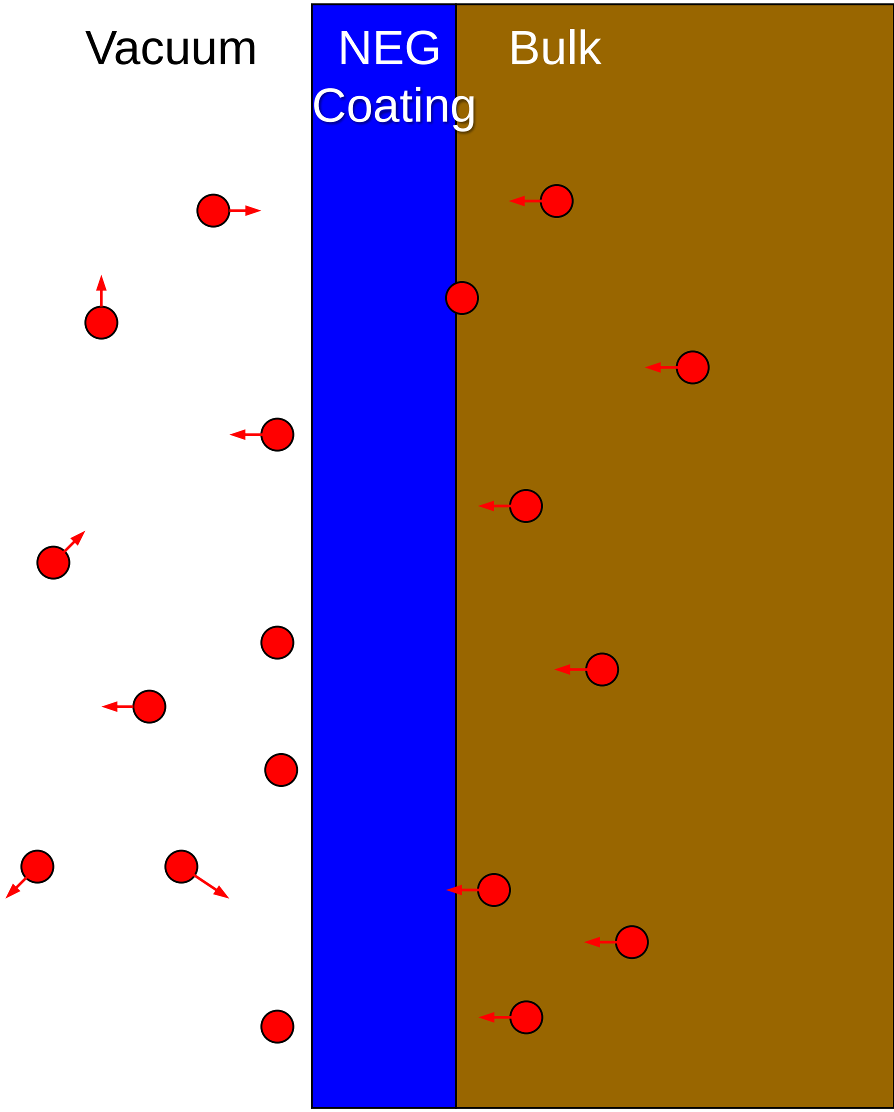
- **A pure metal (Ti, Zr, V, Hf, etc.) film ~1-μm thick without contaminants.**
- **A barrier for molecules from the bulk of vacuum chamber.**

2) Increases distributed pumping speed, *S*:

- **A sorbing surface on whole vacuum chamber surface**

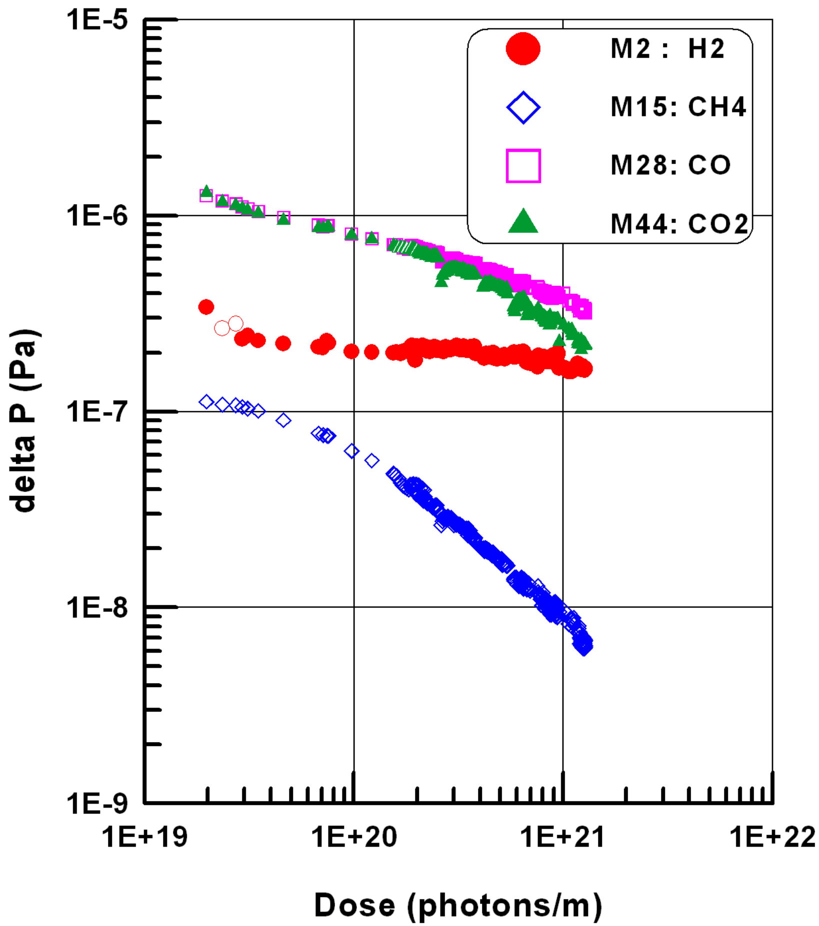
$$S = \alpha \cdot A \cdot v/4;$$

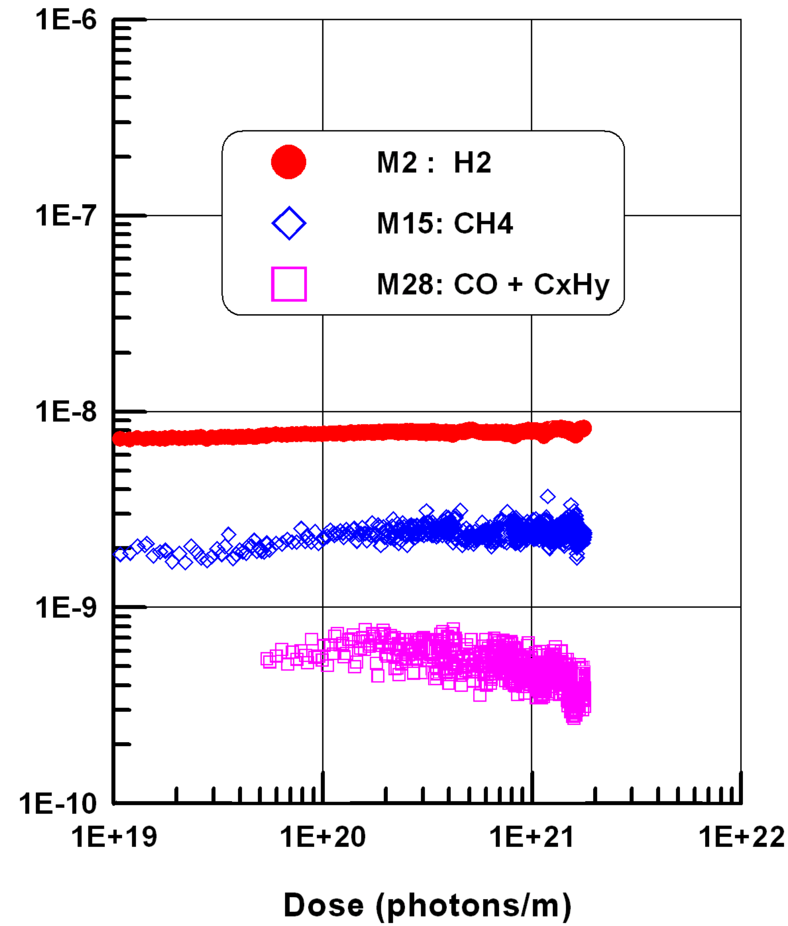
where α **– sticking probability,**
A – surface area,
v – mean molecular velocity



Comparison of PSD from 316LN and NEG

Samples coated with Ti-Zr-V at CERN (Switzerland)
Experiments on the SR beam line at BINP (Russia)





Stainless Steel (baked at 300°C for 24 hrs)

V.V. Anashin *et al*, Vacuum 75 (2004) p. 155.

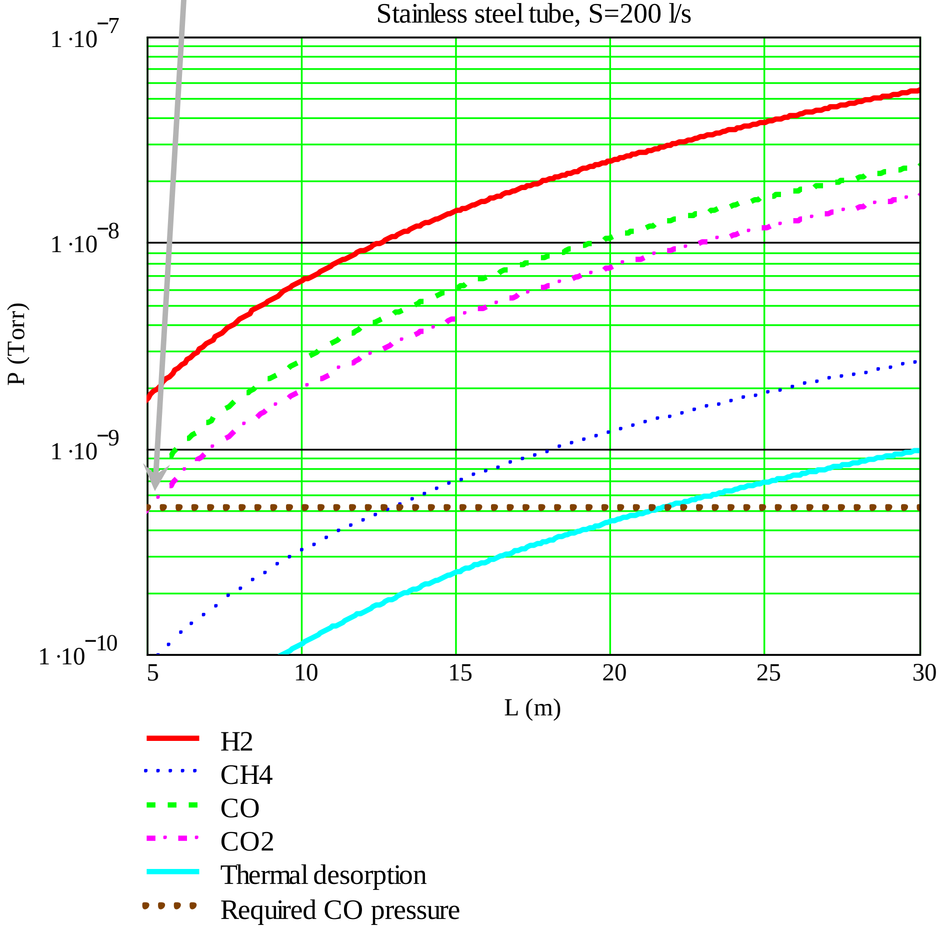
TiZrV coated vacuum chambers (activated at 190°C for 24 hrs)

Using these result for the ILC-DR design

Average pressure after 100 Ahr beam conditioning:

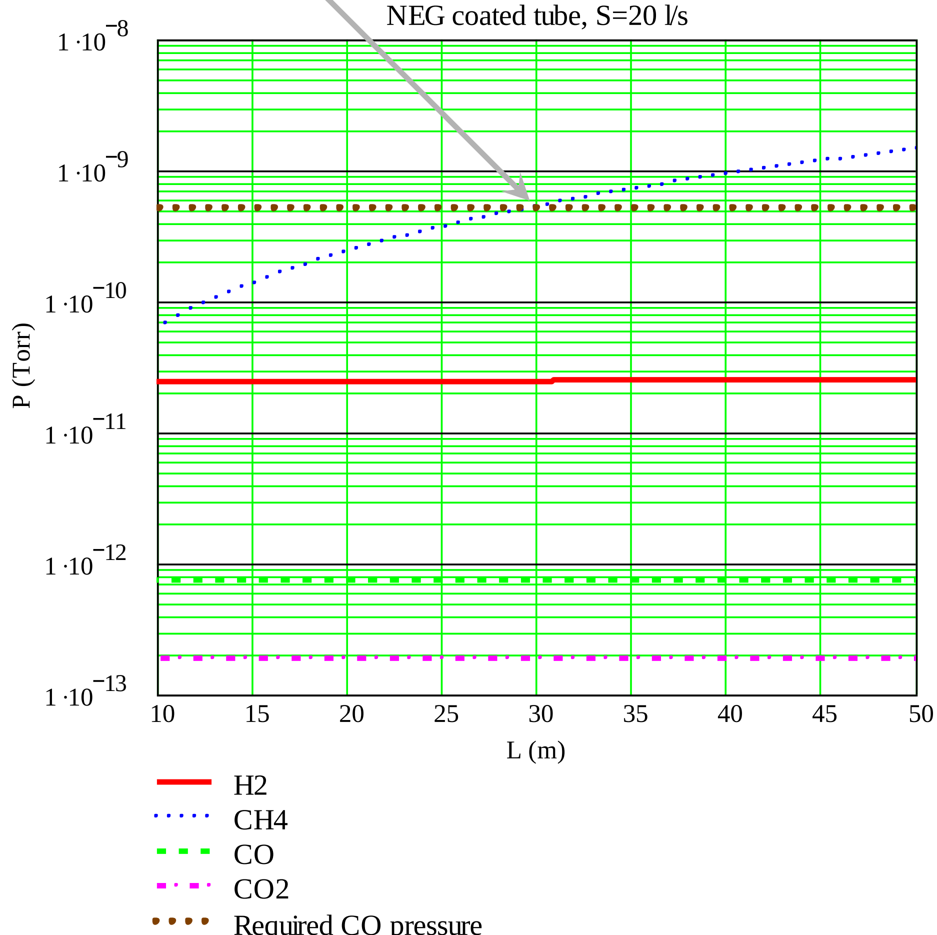
inside a stainless steel tube

S_{eff} = 200 l/s every 5 m



inside a NEG coated tube

S_{eff} = 20 l/s every 30 m



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); MAX-IV (Sweden); Solaris (Poland)
- and many others.

Meanwhile:

- NEG film capacity for CO and CO_2 is ~1 ML:
  - If P = 10^{-9} mbar then 1 ML can be sorbed just in ~10^3 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.**

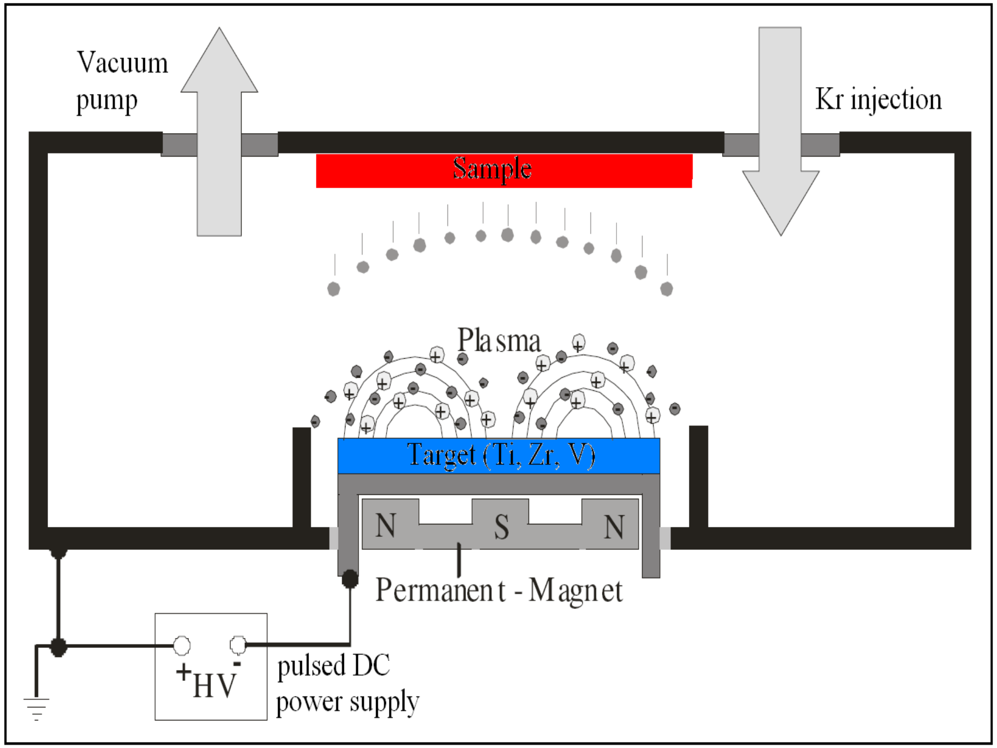
What else is required?

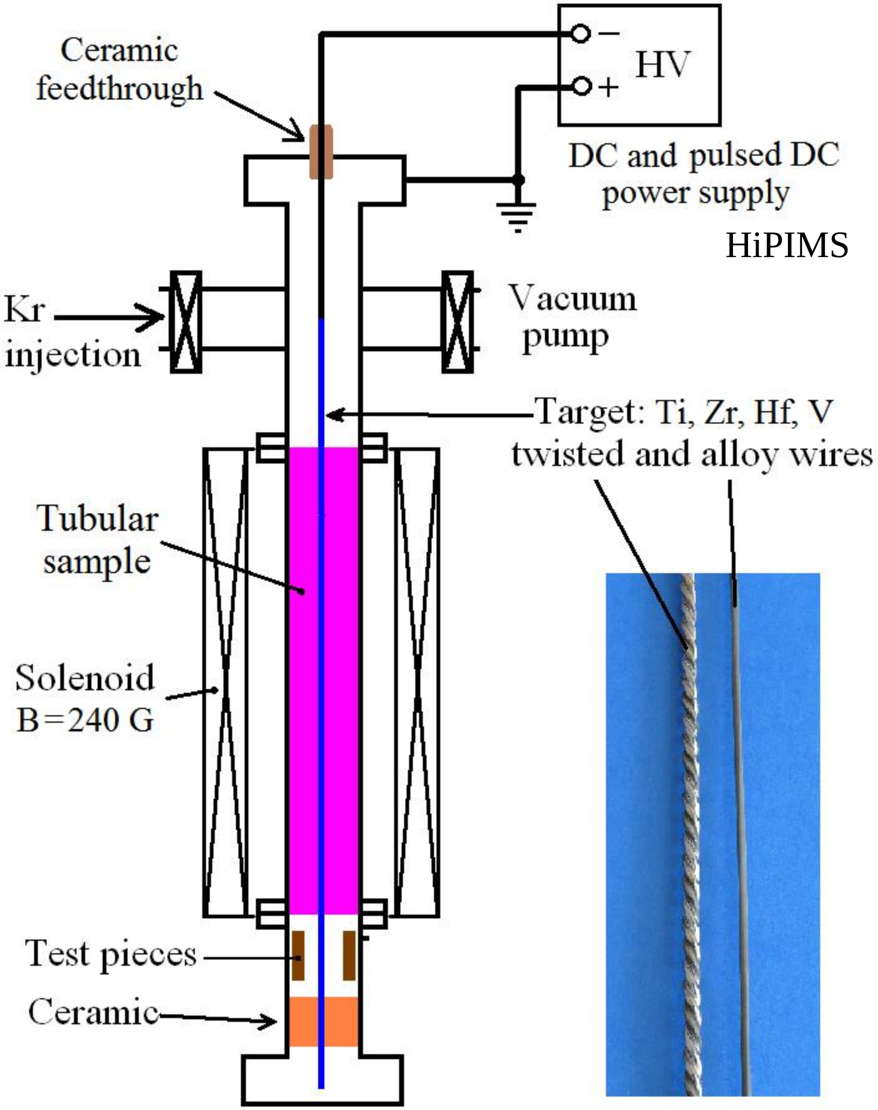
- Input data for accelerator design:
  - η(D,E,T_a), α(M,T_a), pumping capacity;
- Better understanding:
  - what and why;
  - practical ‘do’s and ‘don’t’s;
- Further development of this coating:
  - lower η, T_a, SEY;
  - higher α(M), pumping capacity;
  - optimising for an application.

Deposition method

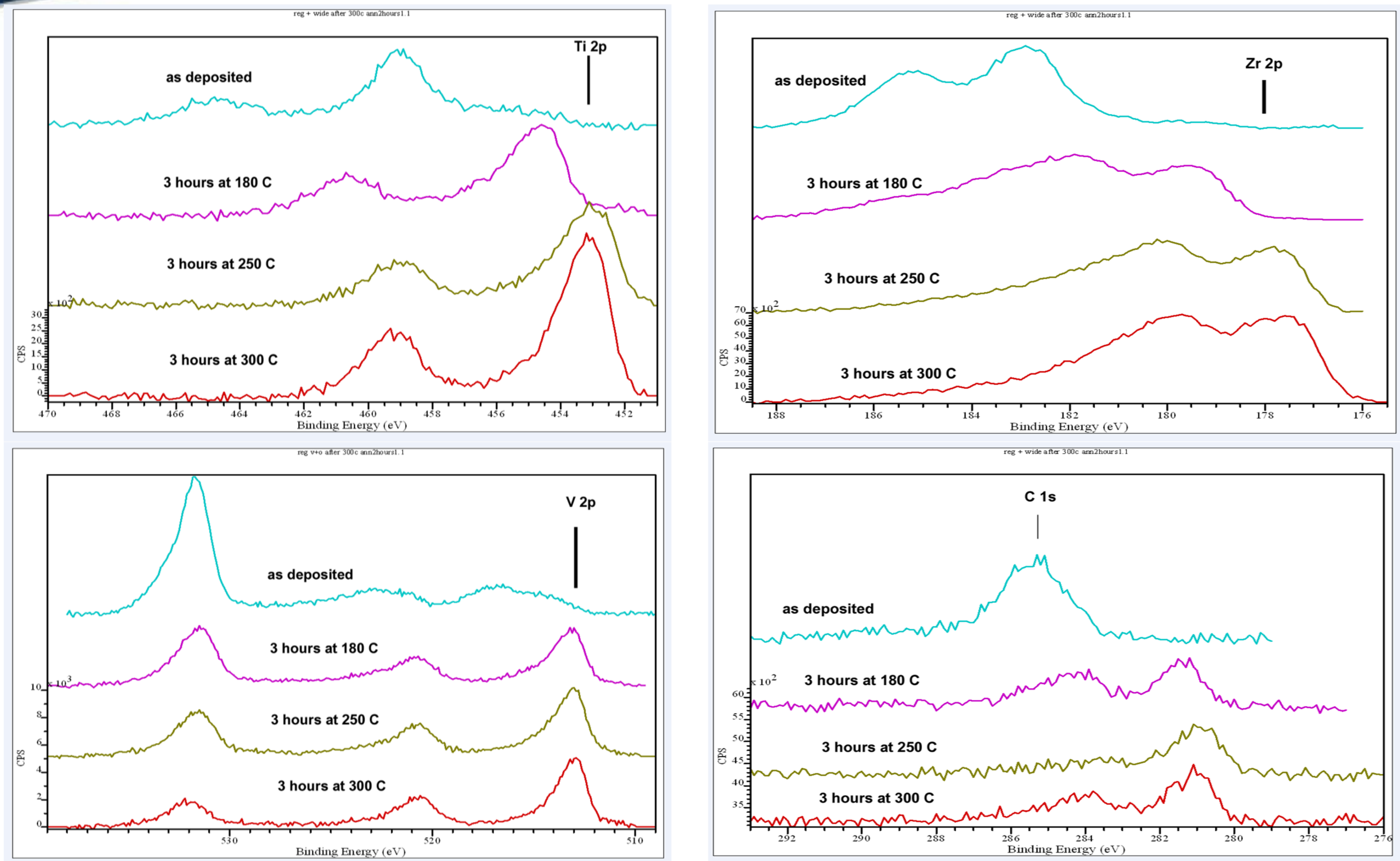
Cylindrical magnetron deposition for vacuum chambers

Commonly used planar magnetron deposition

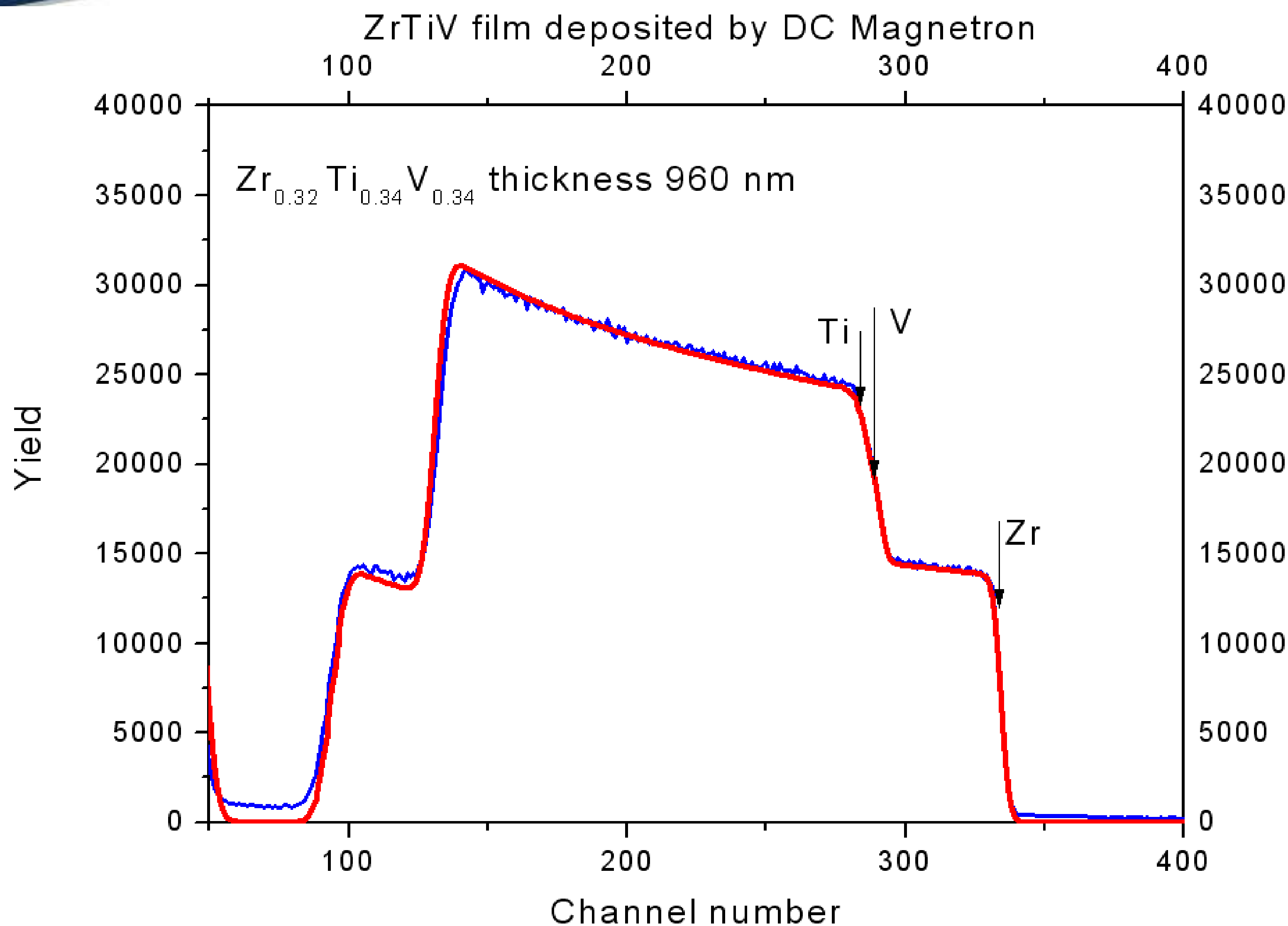




Region scan of XPS core levels of Ti, Zr, C and V of a Ti-Zr-V film (surface composition and chemical bounding)

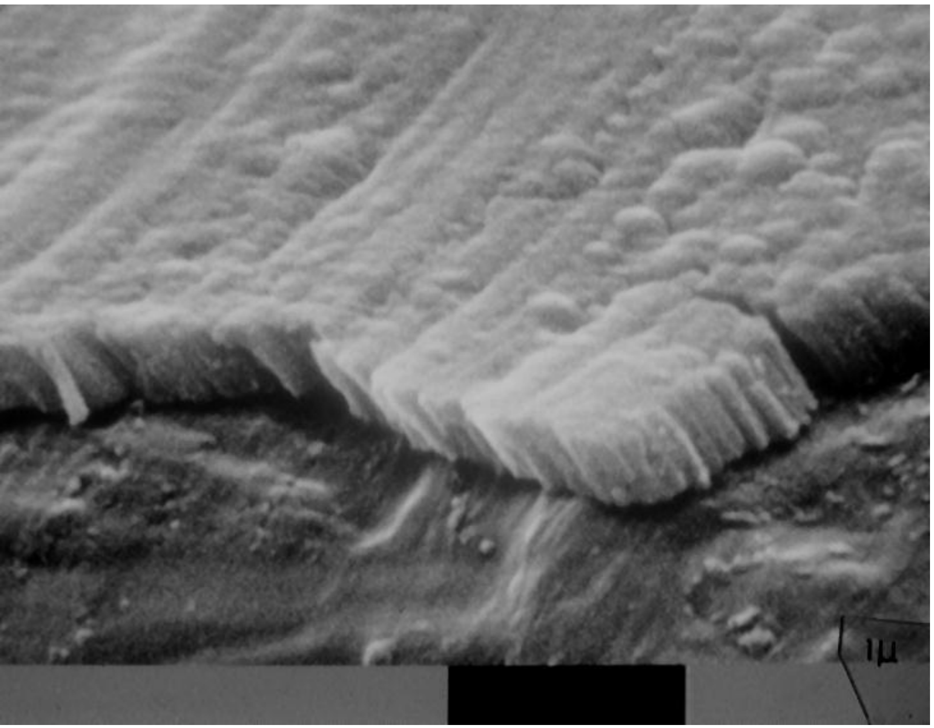


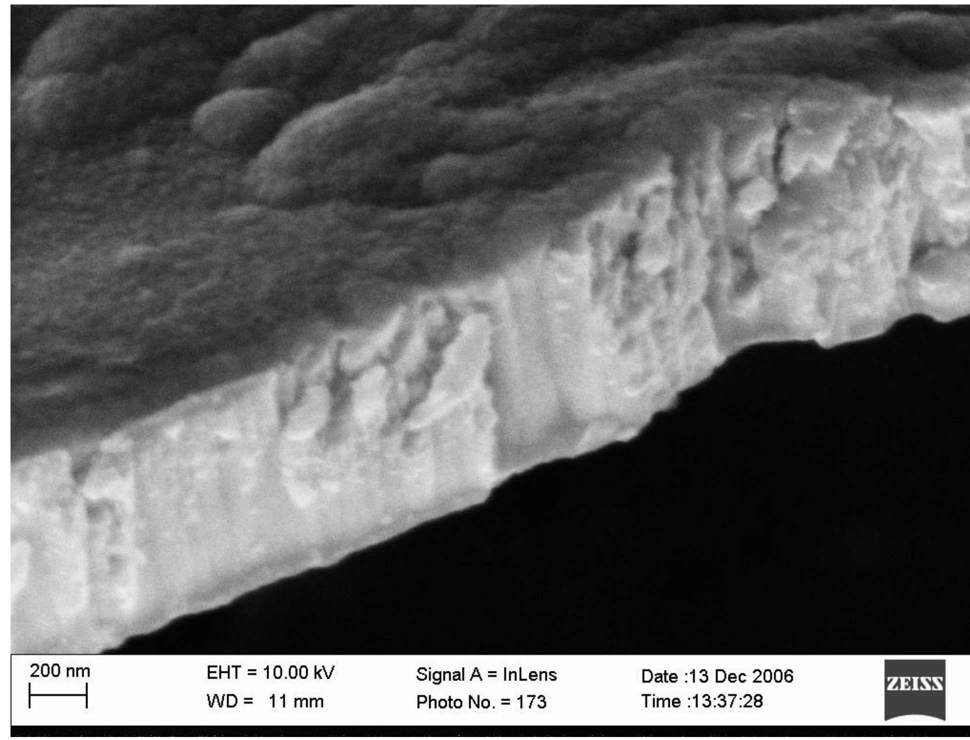
RBS (film compositions in bulk)



SEM images of films (film morphology)

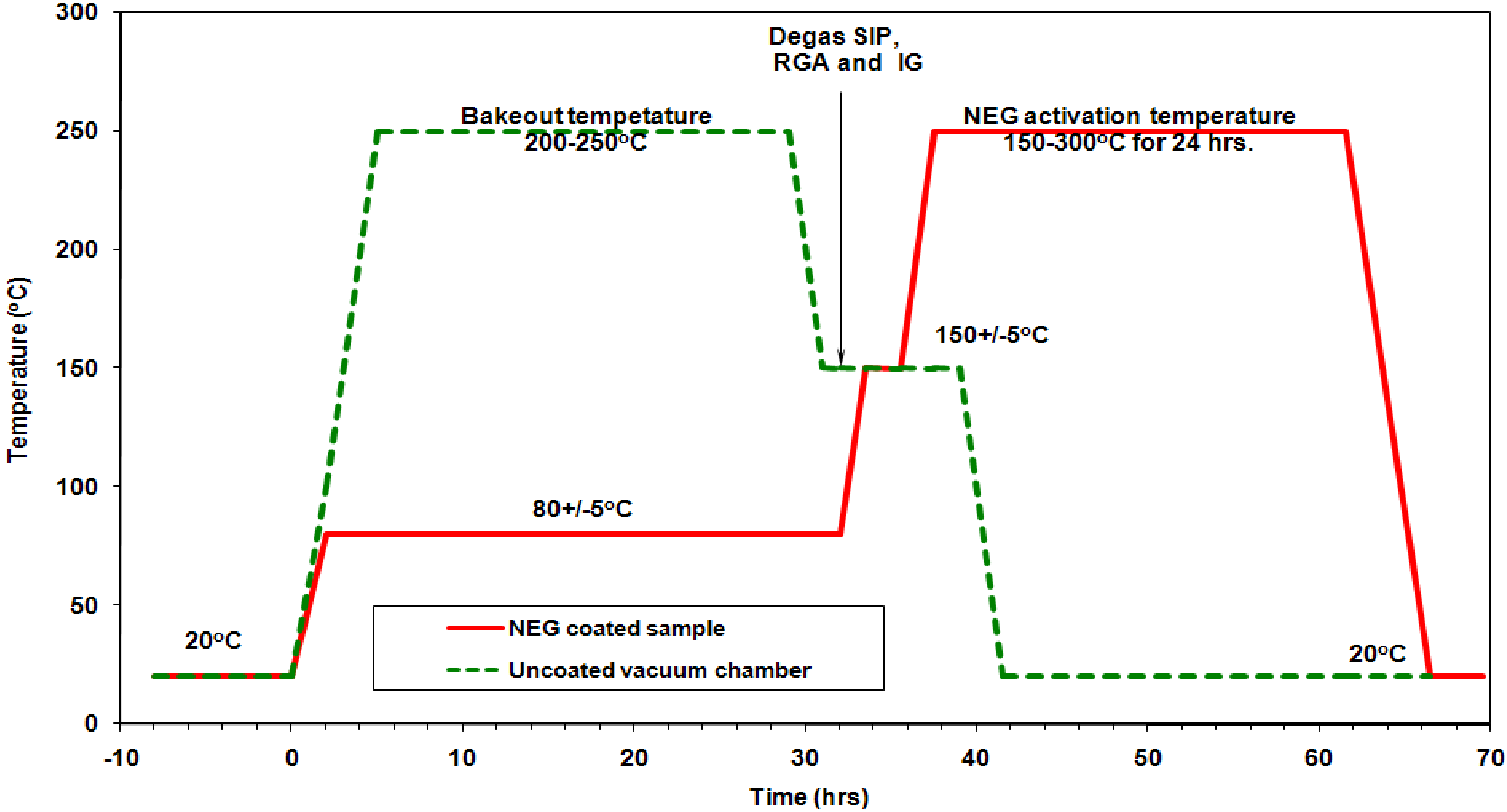
columnar | dense





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

ASTeC activation procedure

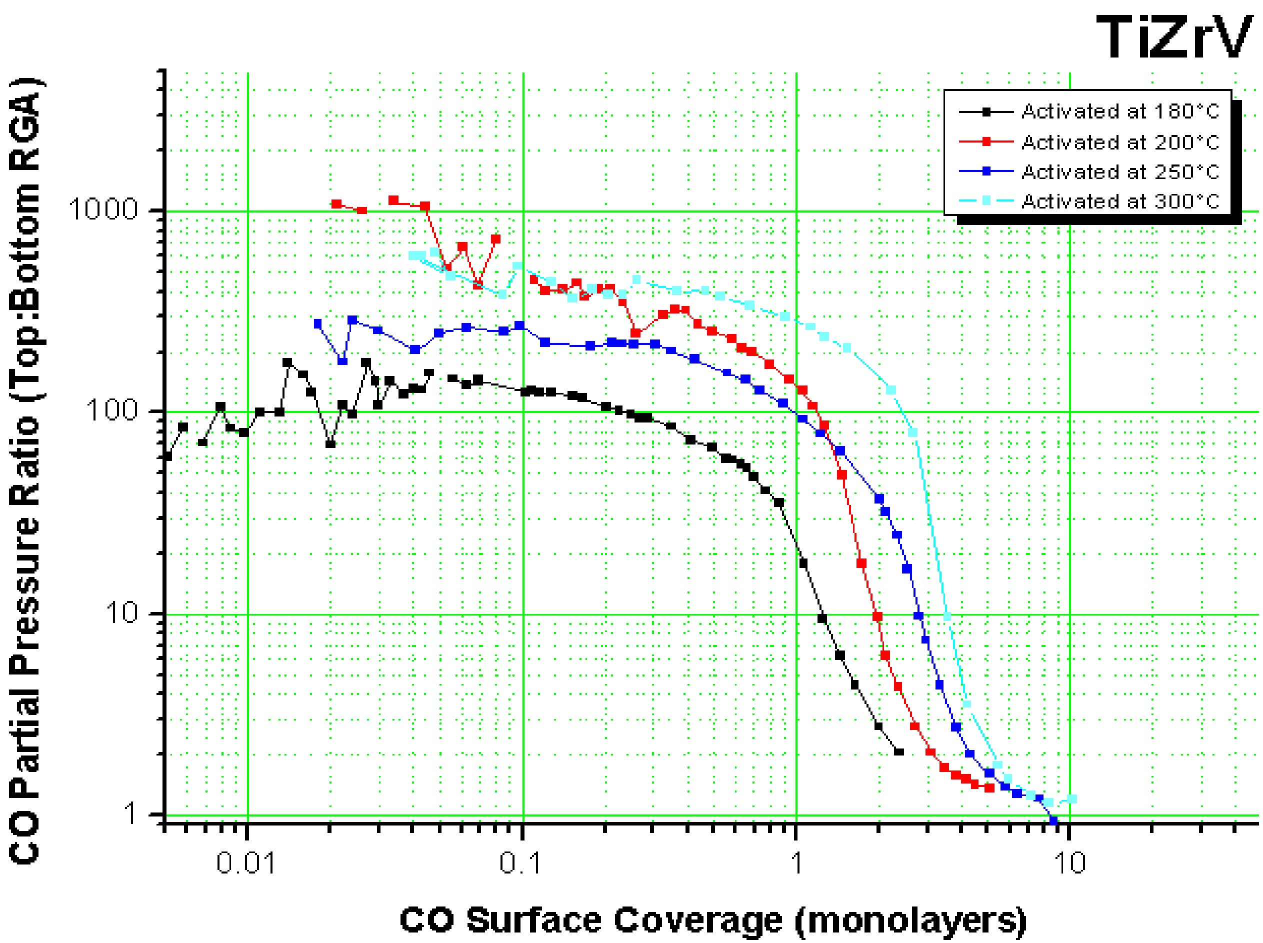


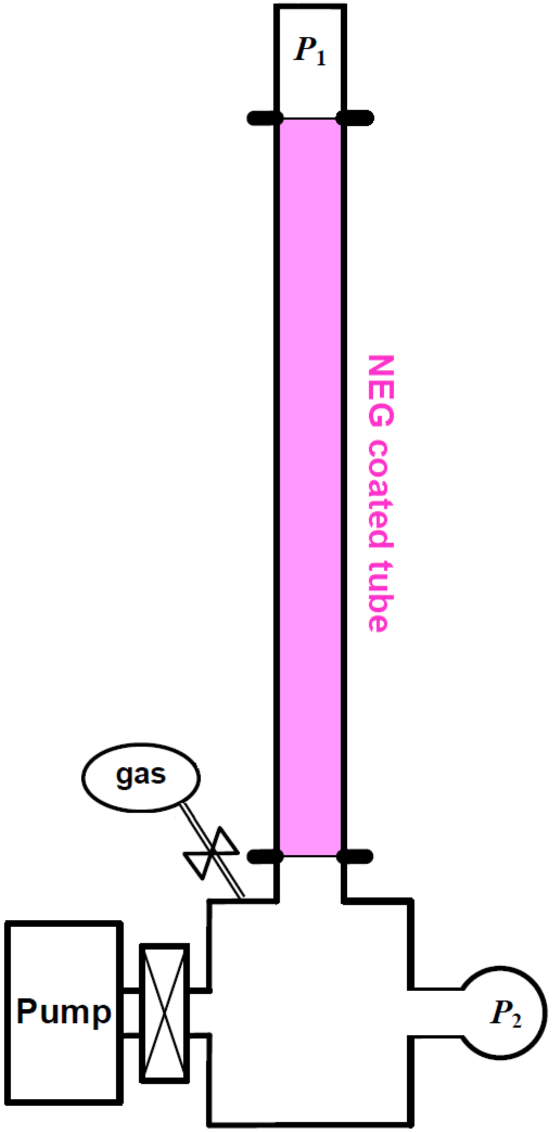
Advantages of ASTeC activation procedure:

- *better activation (less poisoning by das from uncoated parts),*
- *lower electricity cost,*
- *lower total thermal expansion.*

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

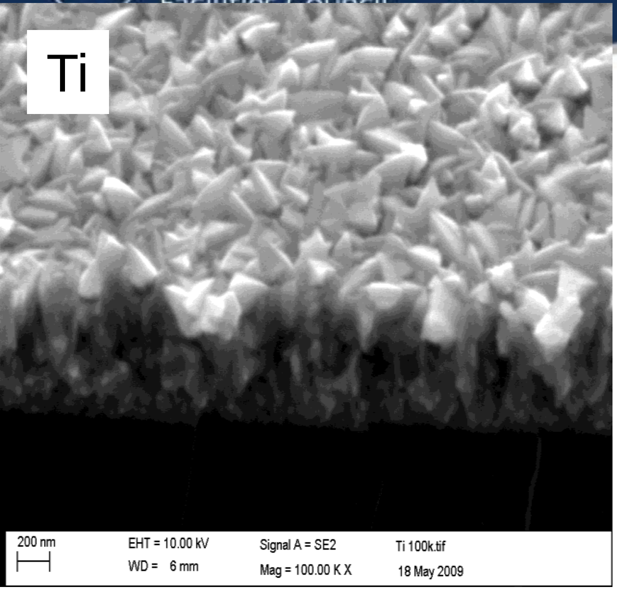
NEG pumping properties

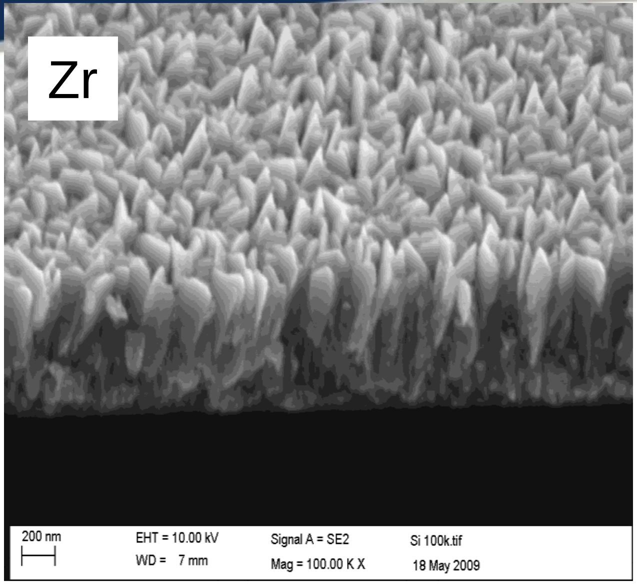


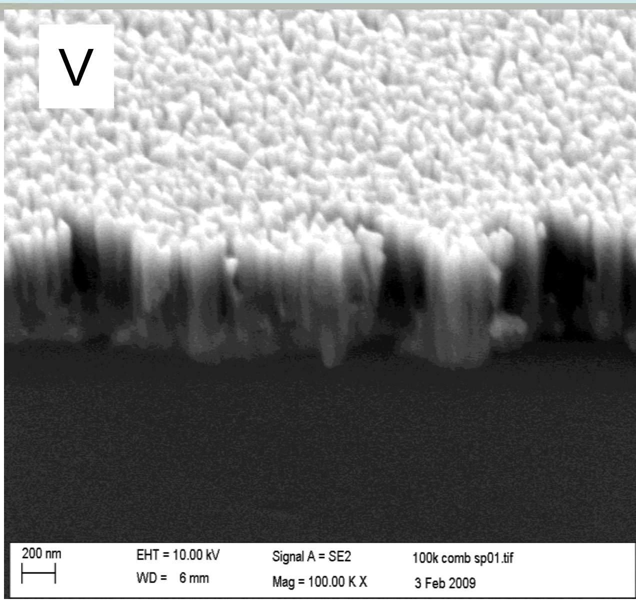


Pressure ratio P_1/P_2 measured during gas injection is used to estimate:
initial sticking probability and sorption capacity

Thin films deposited on Si sample from a single metal wire







Cylindrical Magnetron:

Power = 60 W, $P_{Kr} = 10^{-2}$ mbar,

Deposition rate = 0.14-0.16 nm/s,

T = 120°C.

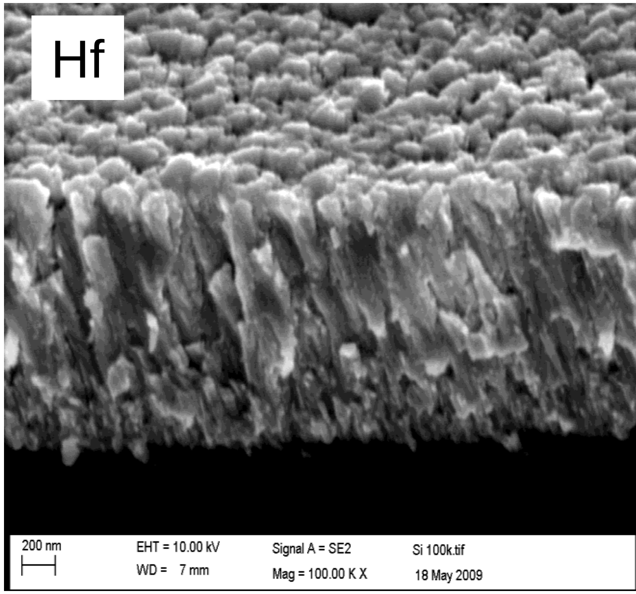
Average grain size: 100 – 150 nm.

Ti:

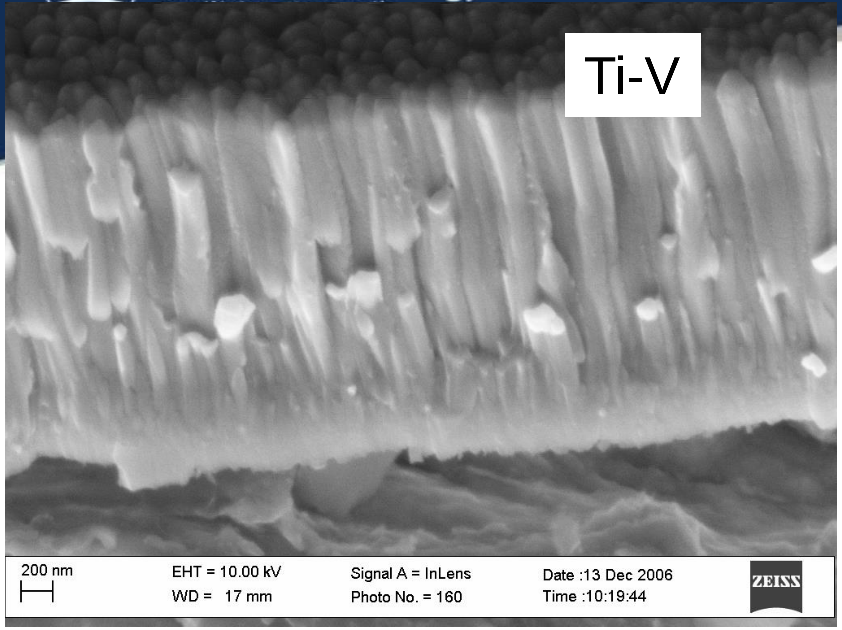
Zr: Hexagonal lattice structure

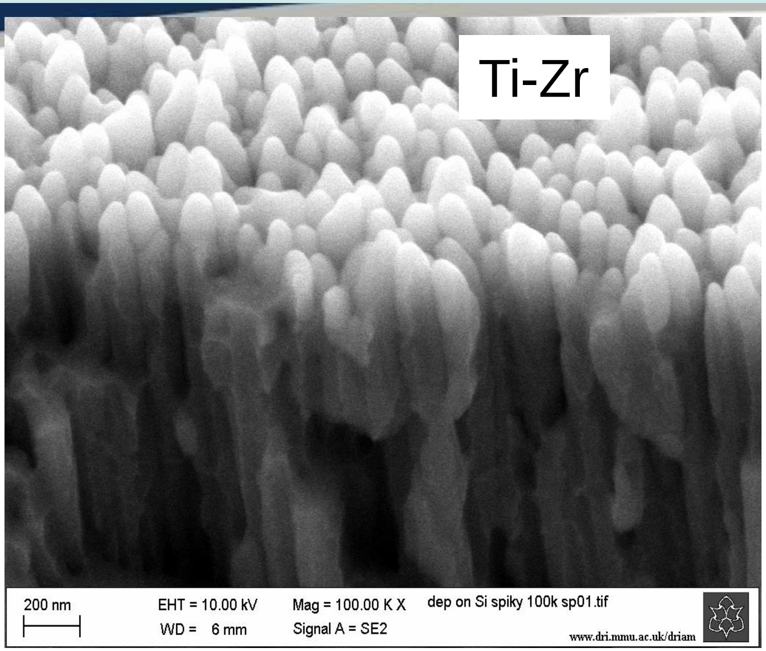
V: Rhombohedral lattice structure

Hf: Hexagonal lattice structure



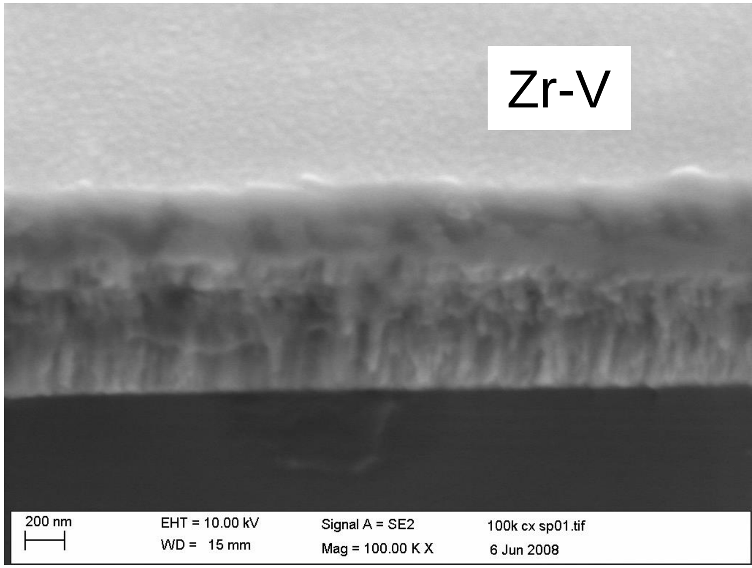
Thin film deposited on Si sample from two twisted wires





Cylindrical Magnetron:

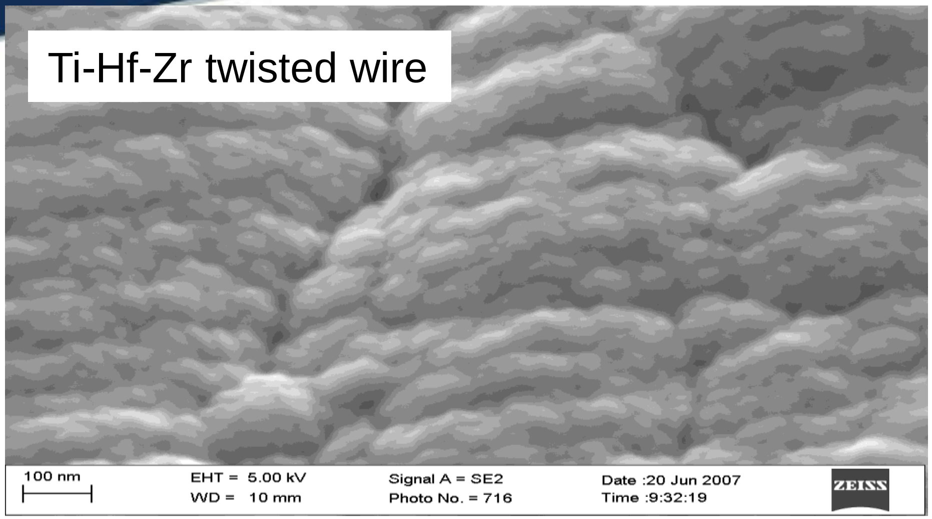
Power = 60 W, P_{Kr} = 10^{-2} mbar,
Deposition rate = 0.13-0.16 nm/s,
T = 120°C.

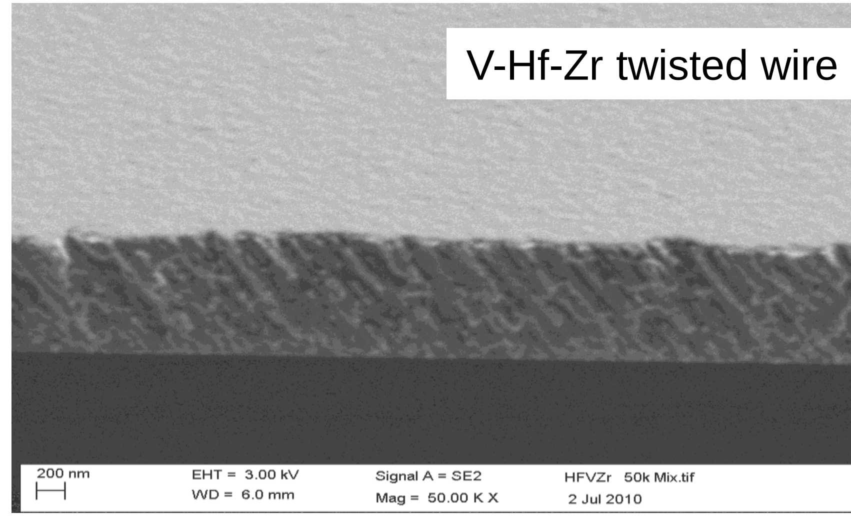


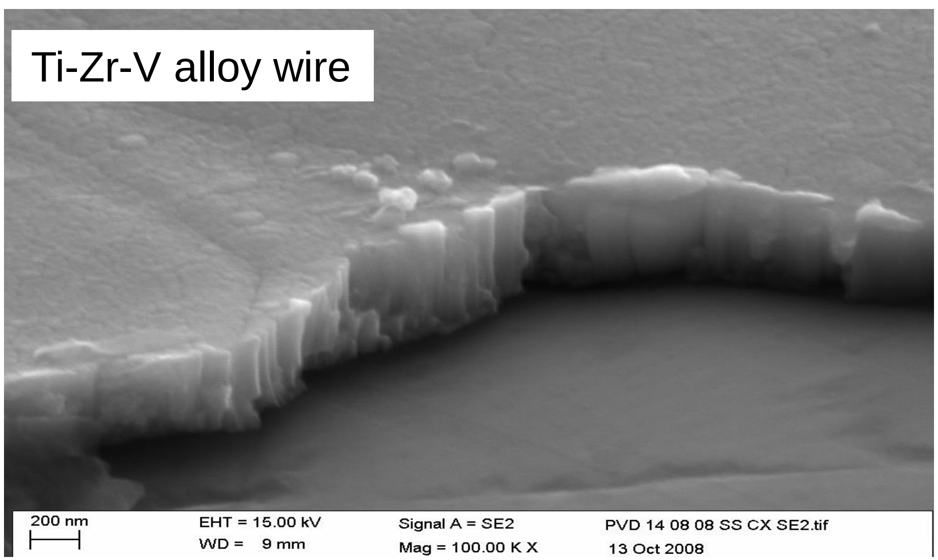
Average grain size:

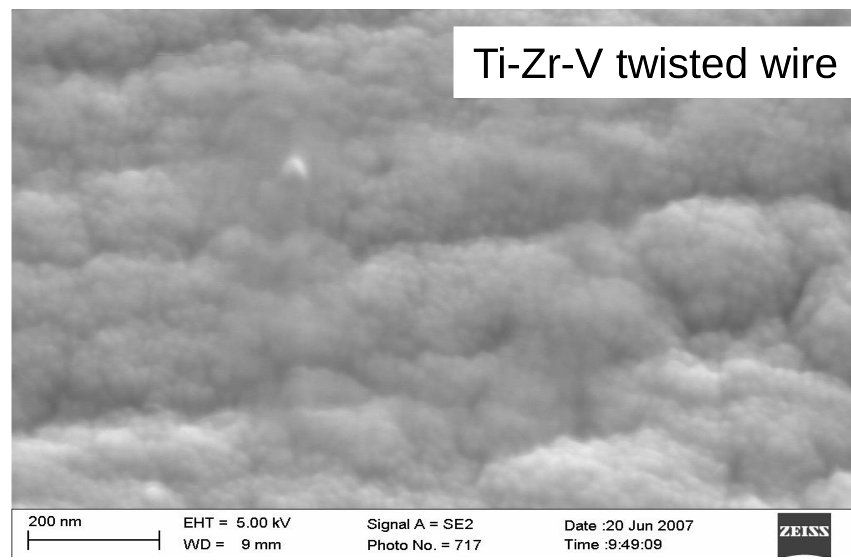
Ti-V: 50 – 100 nm, Hexagonal lattice structure
Ti-Zr: 50 – 100 nm, Hexagonal lattice structure
Zr-V: **10 – 20 nm,** Rhombohedral lattice structure

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





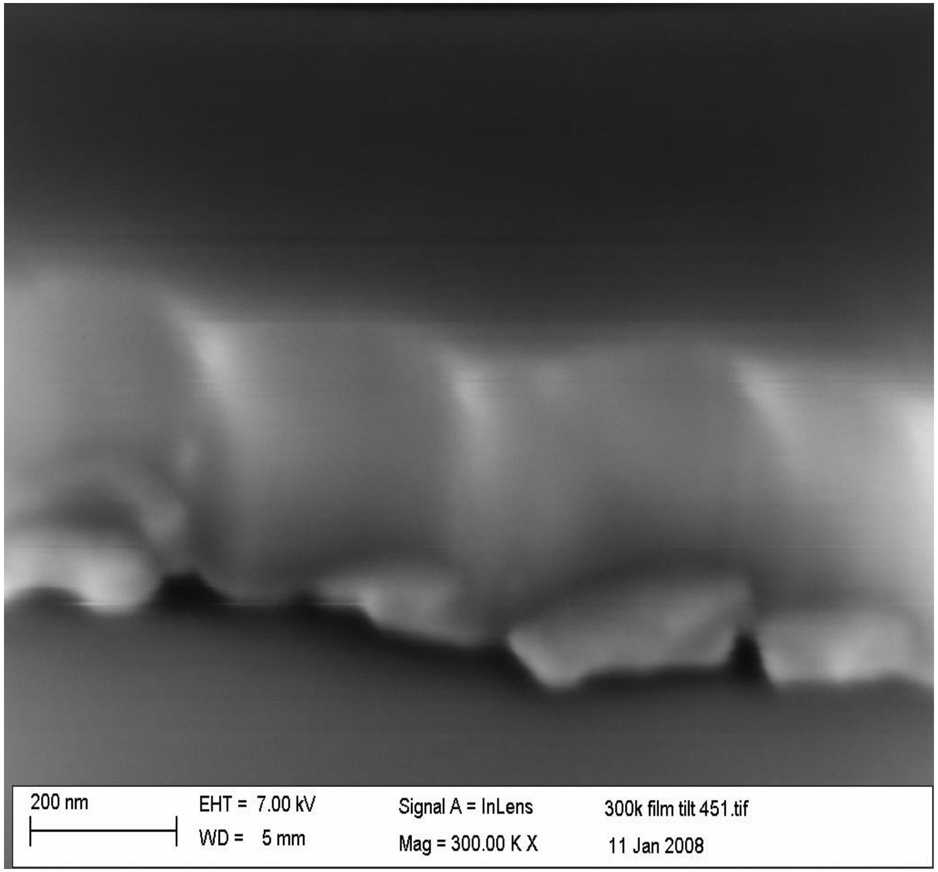


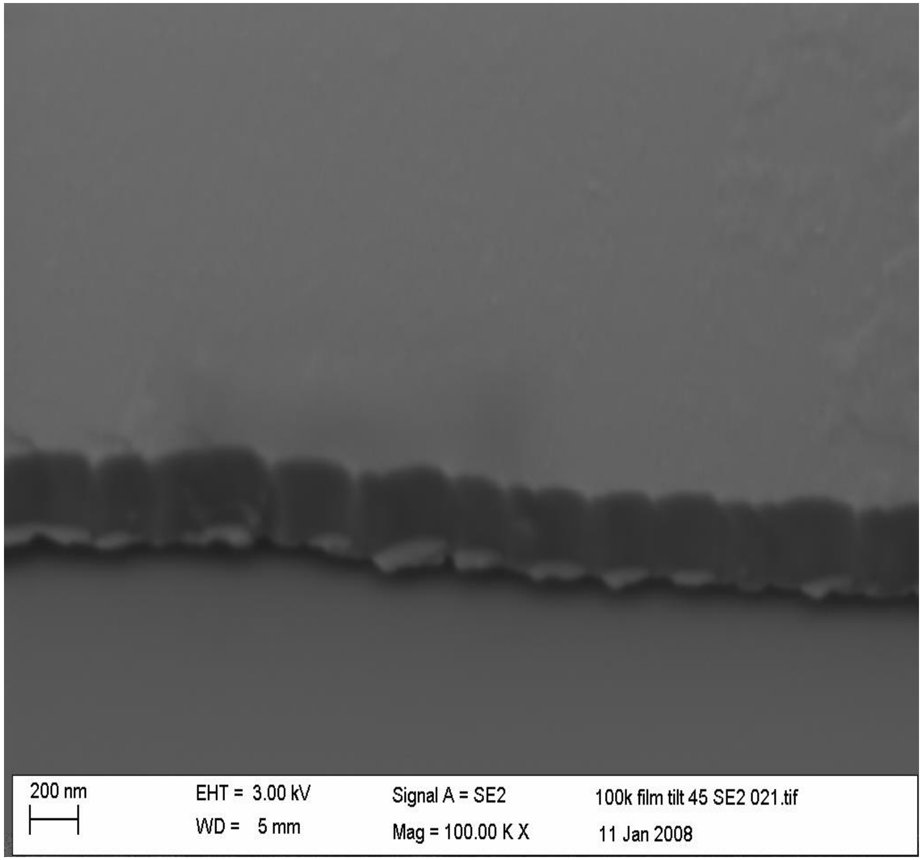


Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.12 nm/s, T = 120°C.

Average grain size 5 nm. Hexagonal lattice structure.

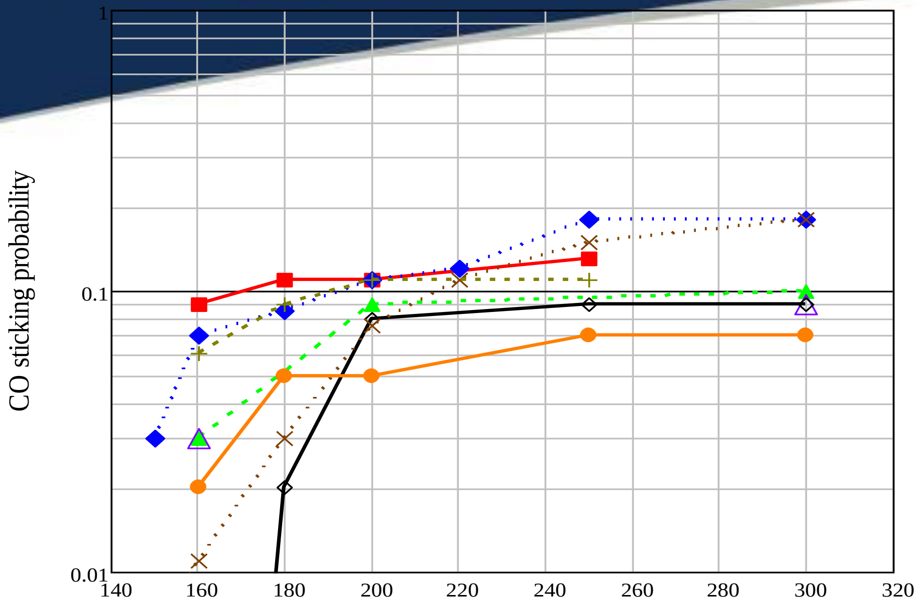
Quaternary NEG alloy film deposited on Si test sample from twisted Ti, V, Zr, and Hf wires

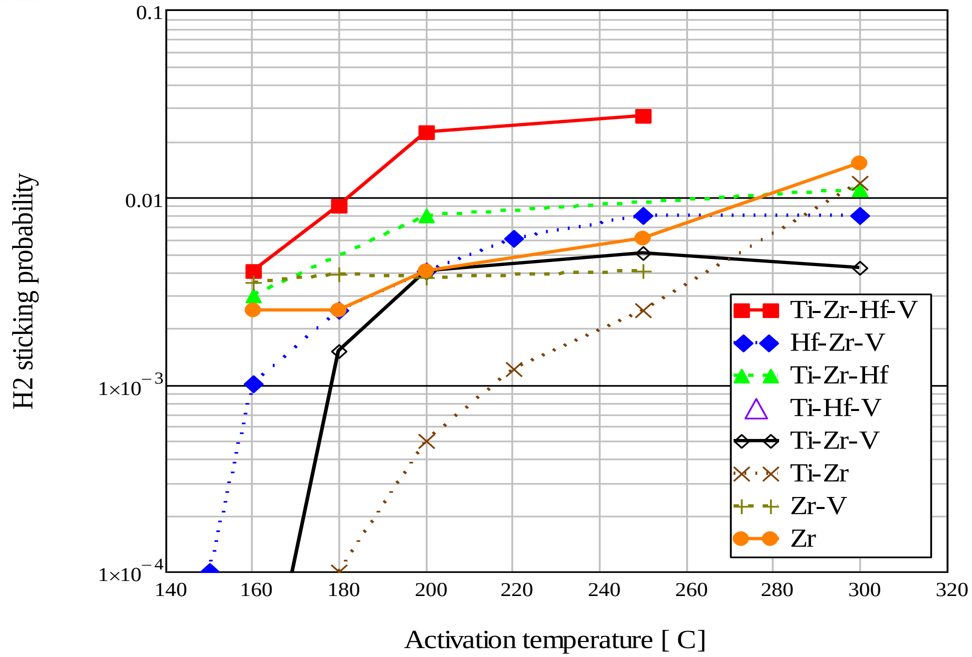


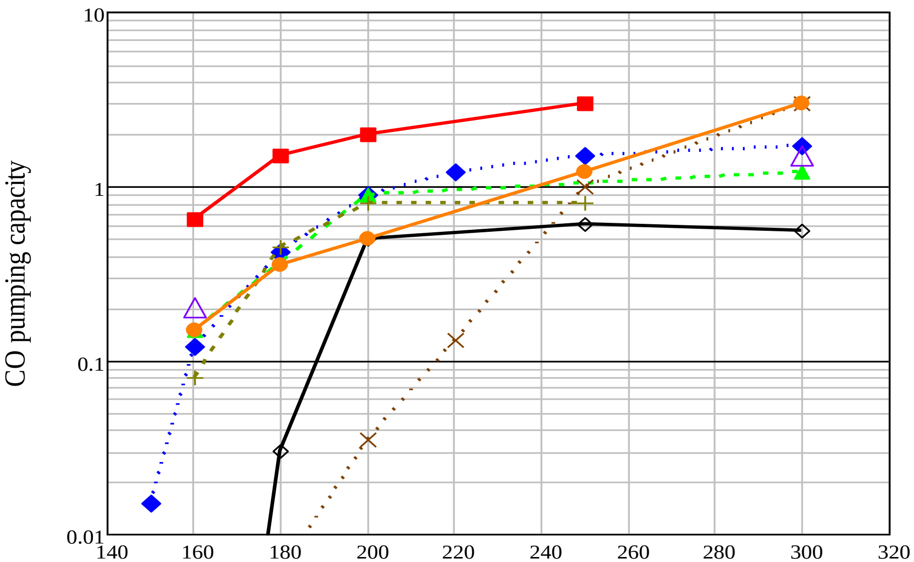


Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.12 nm/s, T = 120°C.
Very glassy structure.

Pumping properties of some NEG 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 (photon, electron or ion stimulated desorption)

α - sticking probability

- Improving pumping properties is limited:

  $\alpha \leq 1$

  - $0.005 < \alpha_{H2} < 0.02$
  - $0.1 < \alpha_{CO} < 0.5$
  - $0.4 < \alpha_{CO2} < 0.6$

- Reducing the desorption yields η <u>in orders of magnitude</u> was our aim

Reducing the gas desorption from the NEG coatings

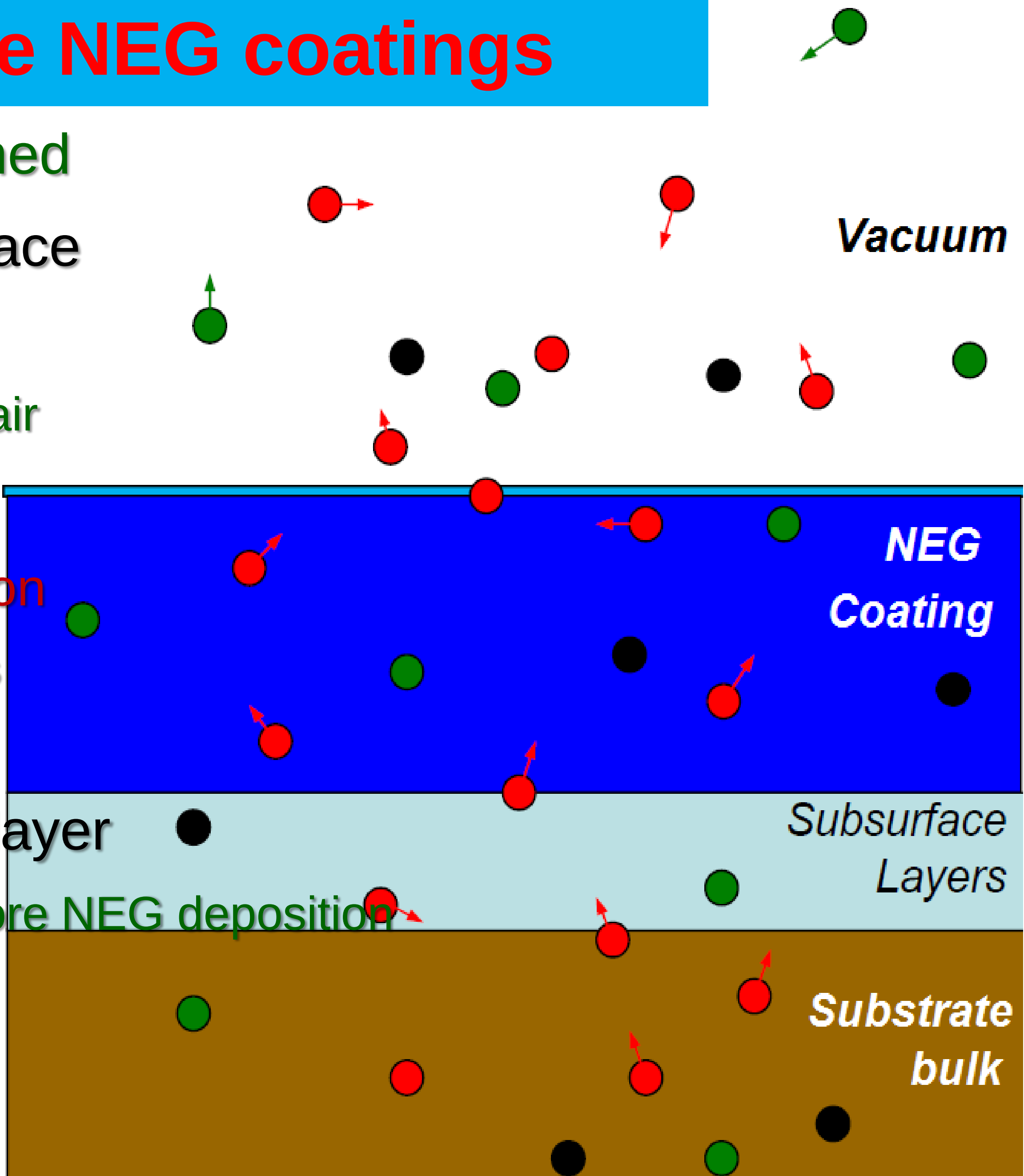
- Main gases in the NEG coated vacuum chamber are H_2 and CH_4
  - Only H_2 can diffuse through the NEG film under bombardment or heat
  - CH_4 is most likely created on the NEG surface from diffused H_2 and C (originally from sorbed CO and CO_2)
  - Therefore the H_2 diffusion must be suppressed

    - **Where H_2 come from?**

Reducing the gas desorption from the NEG coatings

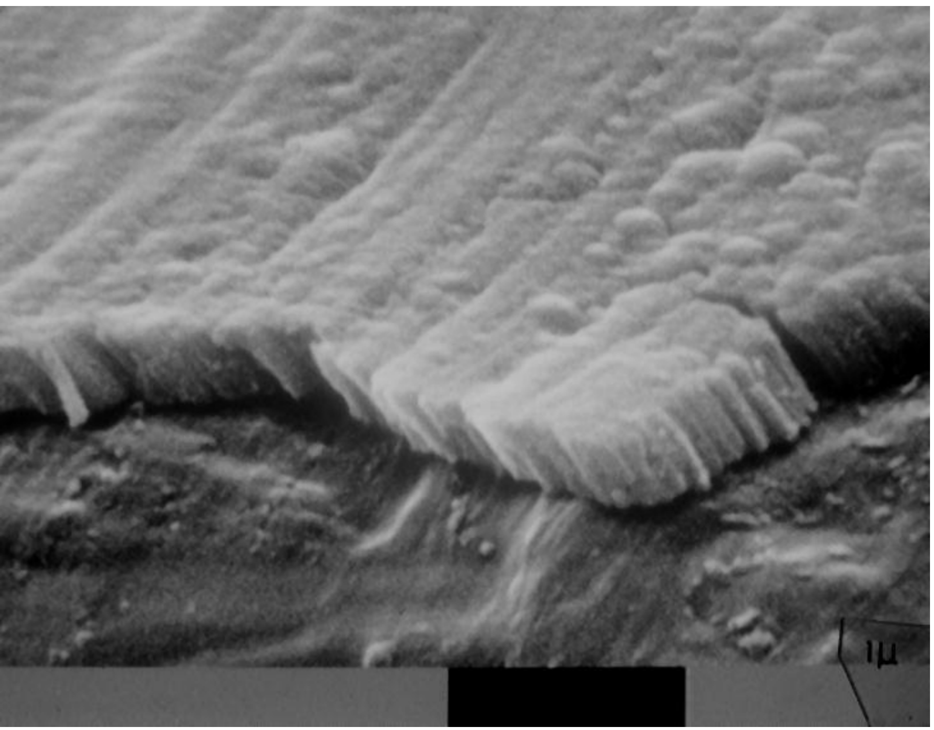
Gas molecules are contained

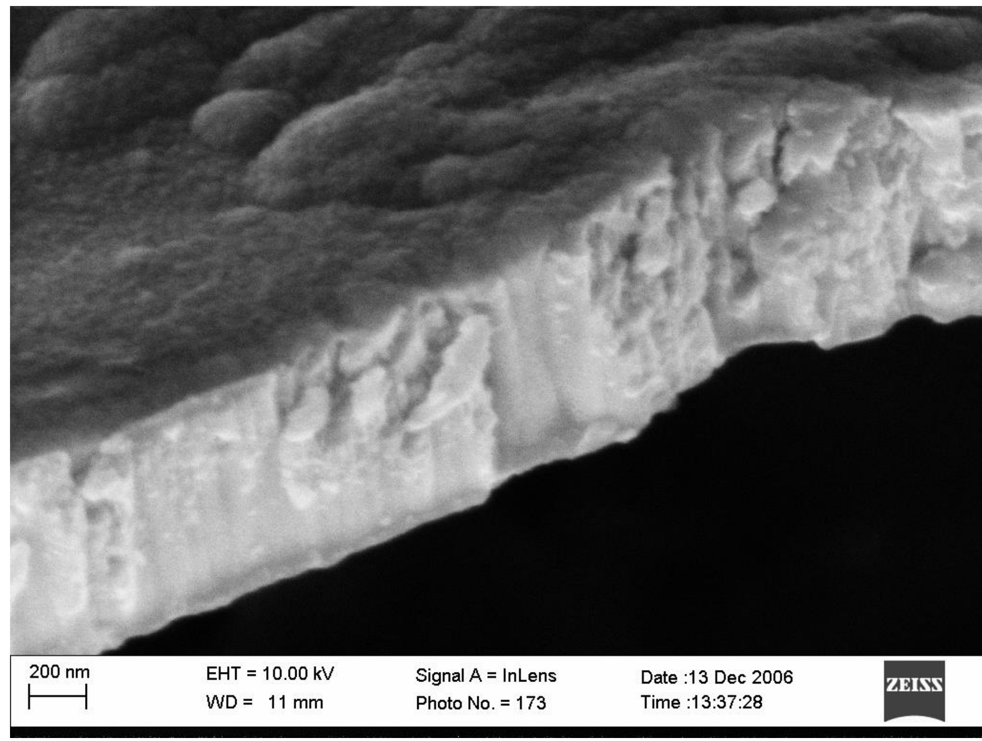
- on the NEG coating surface
  - after exposure to air
    - minimise exposure to air
- inside the NEG coating
  - trapped during deposition
    - purity of discharge gas
    - background pressure
- in subsurface substrate layer
    - substrate bakeout before NEG deposition
- in the substrate bulk
    - vacuum firing



SEM images of films (film morphology)

columnar	dense
Best for pumping	A first candidate for a barrier





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

Facilities Council

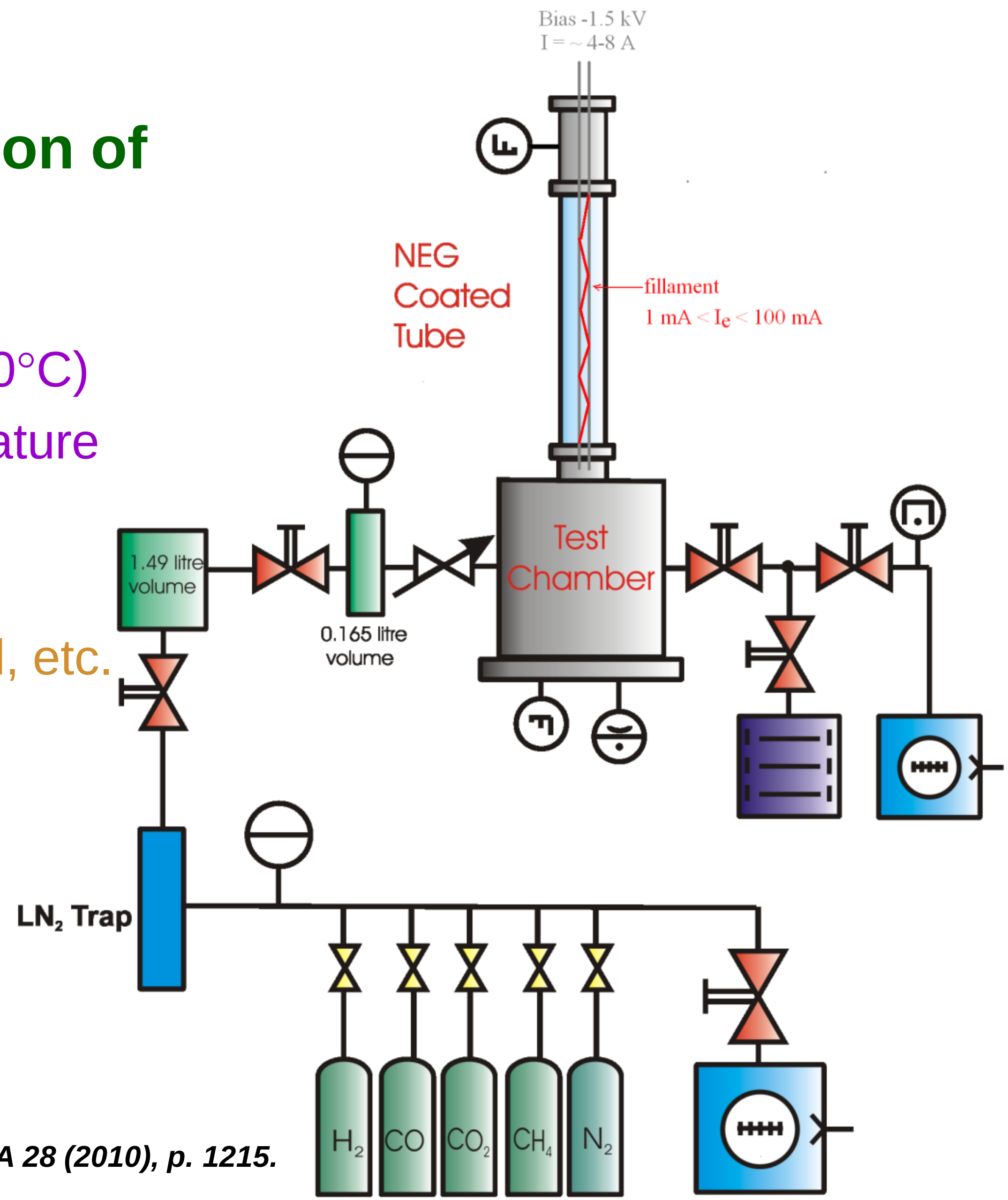
Electron stimulated desorption facility

ESD is studied as a function of

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

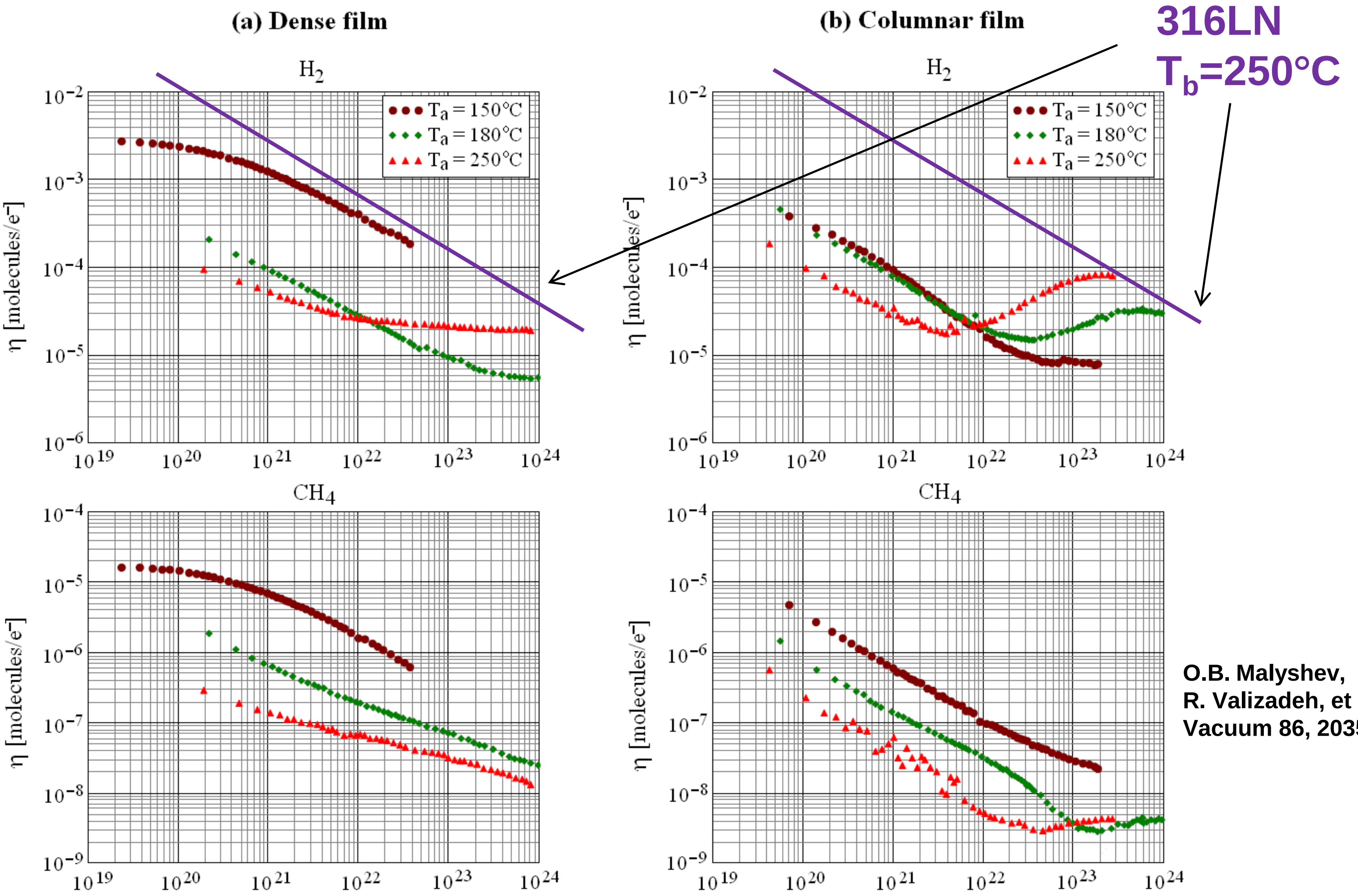
Can be used for samples with:

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



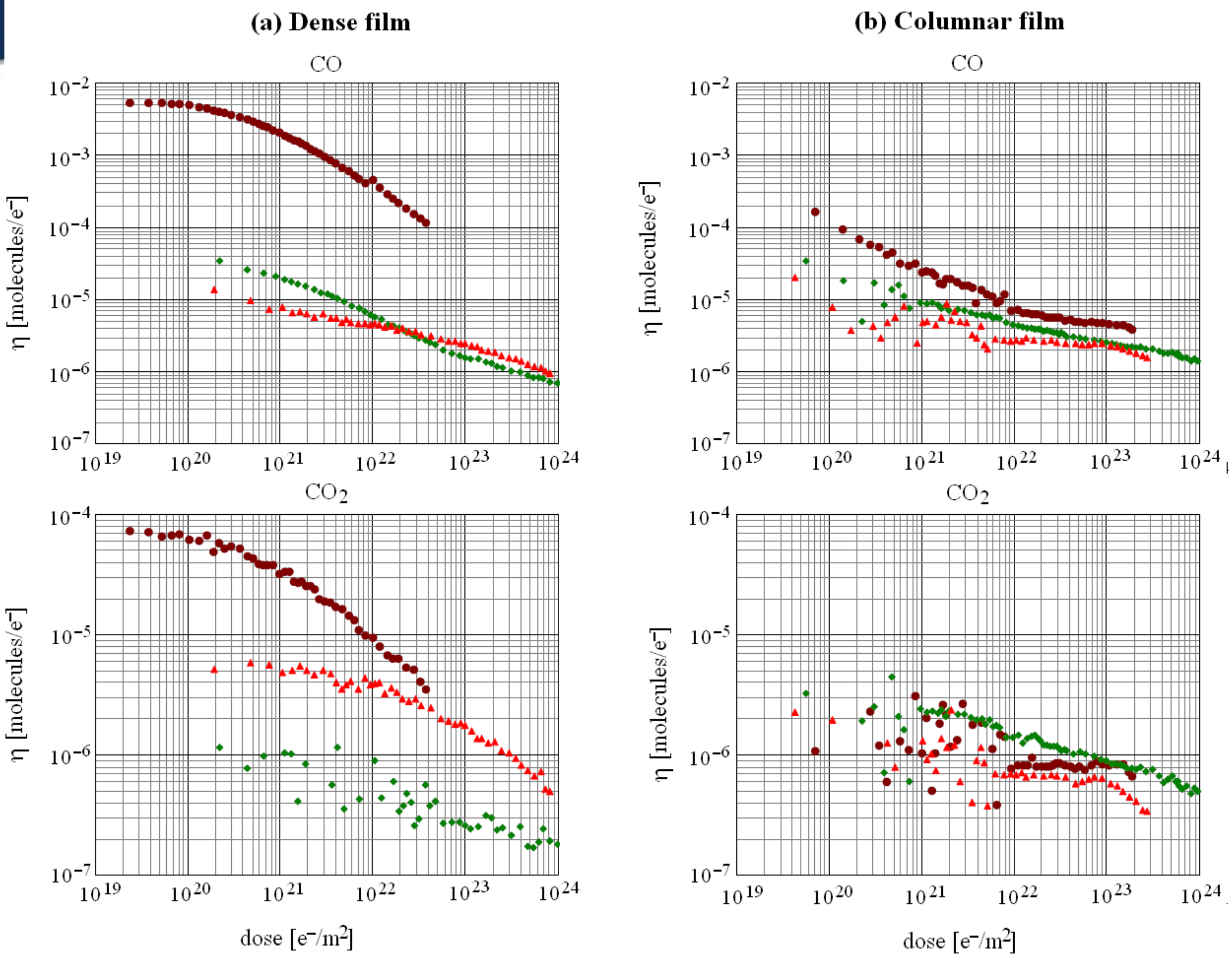
O.B. Malyshev, A.P. Smith et al. J. Vac. Sci. Technol. A 28 (2010), p. 1215.

ESD yield from NEG coated samples



O.B. Malyshev,
R. Valizadeh, et al.
Vacuum 86, 2035 (2012).

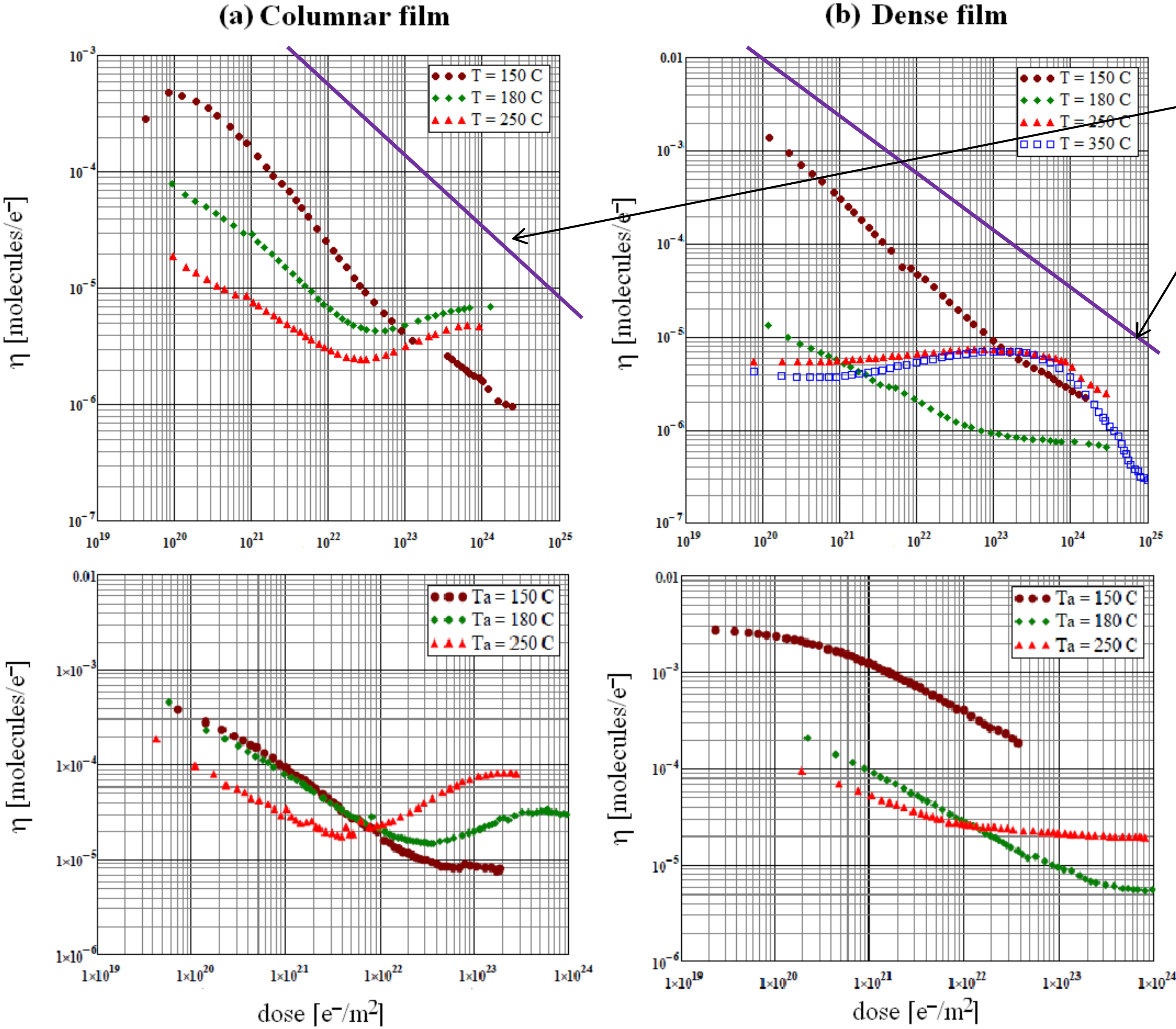
ESD yield from NEG coated samples



H_2 ESD from NEG coated vacuum fired 316LN

Vacuum fired

No vacuum firing



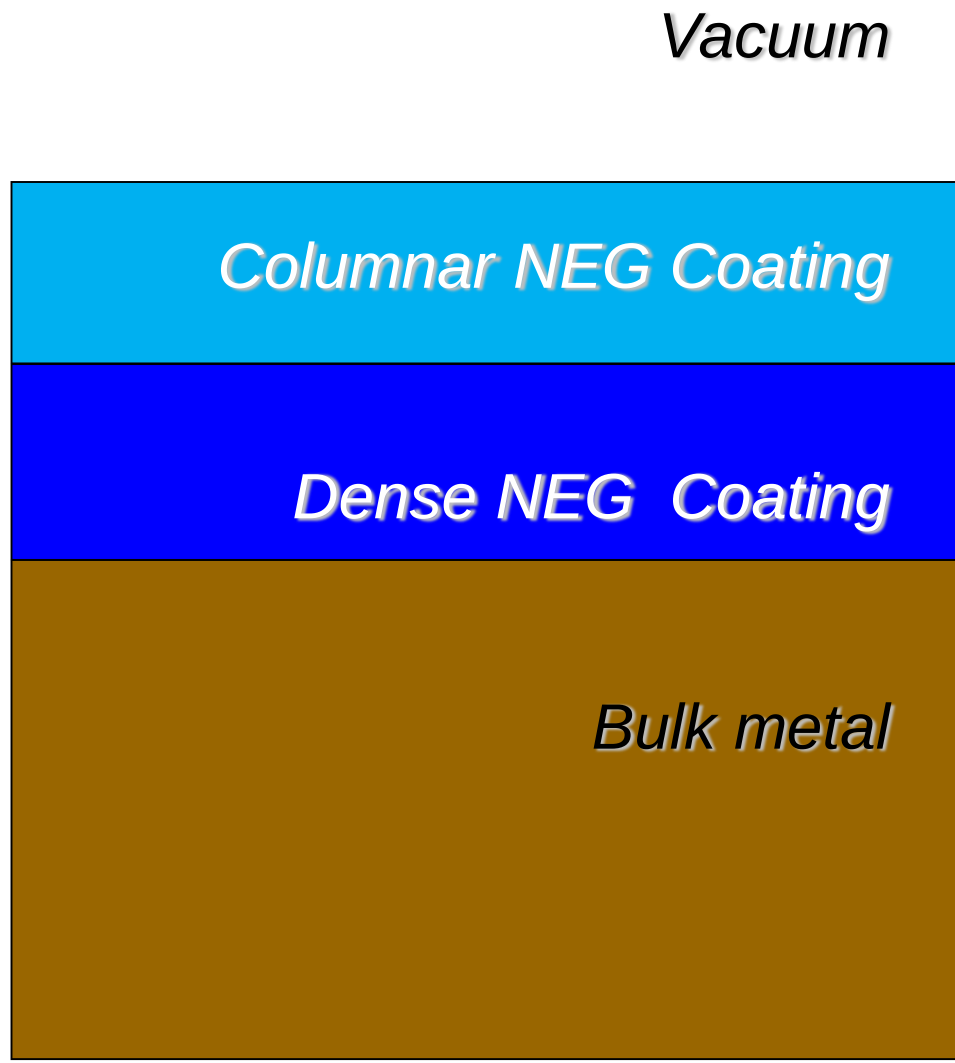
316LN
T_b=250°C

O.B. Malyshev, R. Valizadeh, et al. JVST A 32, 061601 (2014)

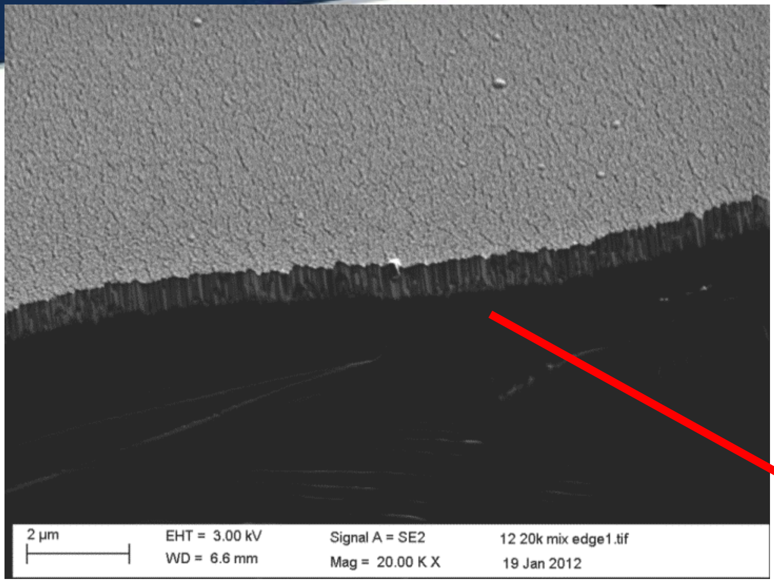
O.B. Malyshev, R. Valizadeh, et al. Vacuum 86, 2035 (2012).

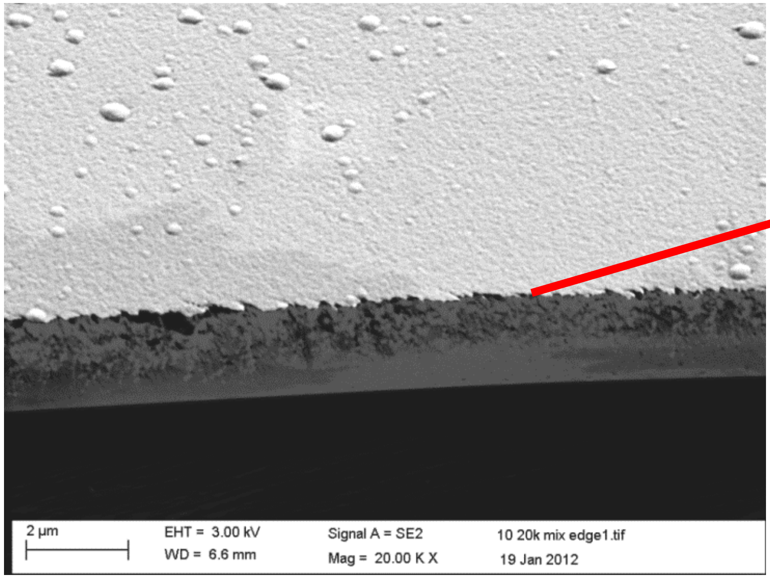
Dual layer

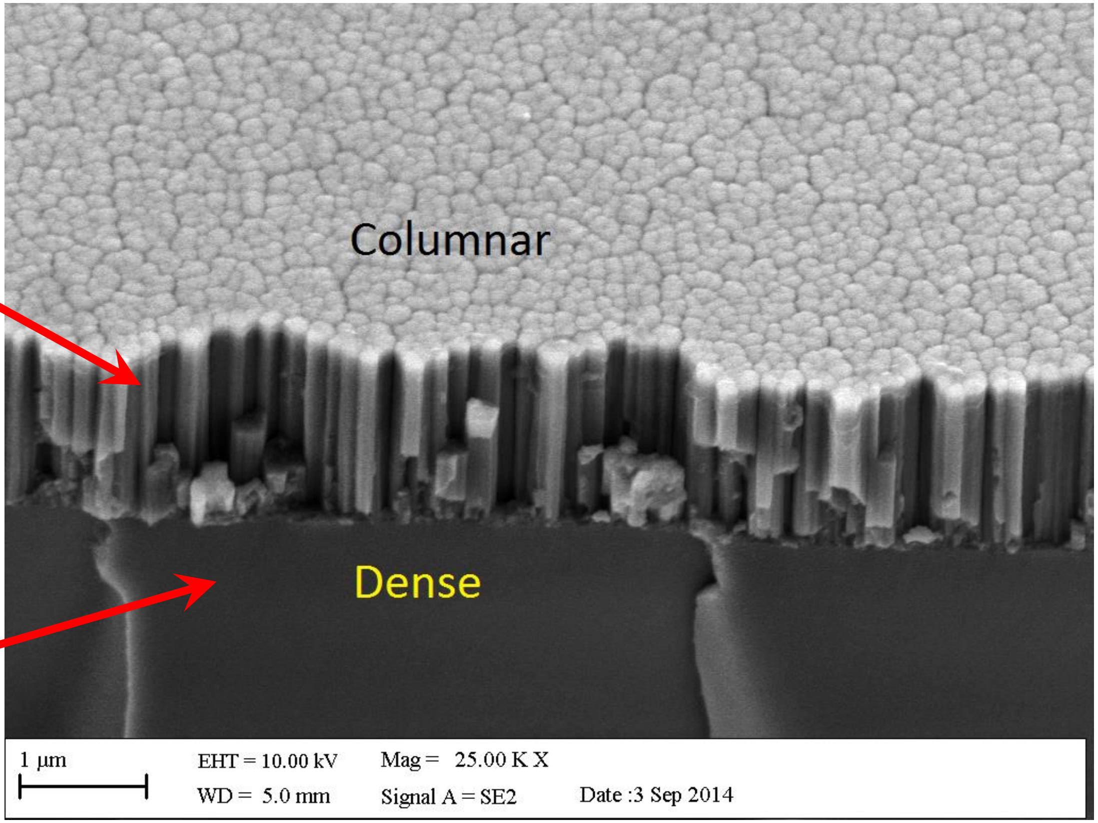
- Columnar layer:
  - Activated at lower temperature
  - Provides higher sticking probability and pumping capacity
- Dense layer:
  - Provides lower ESD
- Dual Layer:
  - Combines benefit of both
  - For more details: see A. Hannah’s poster EM286 on Thursday



Dual layer

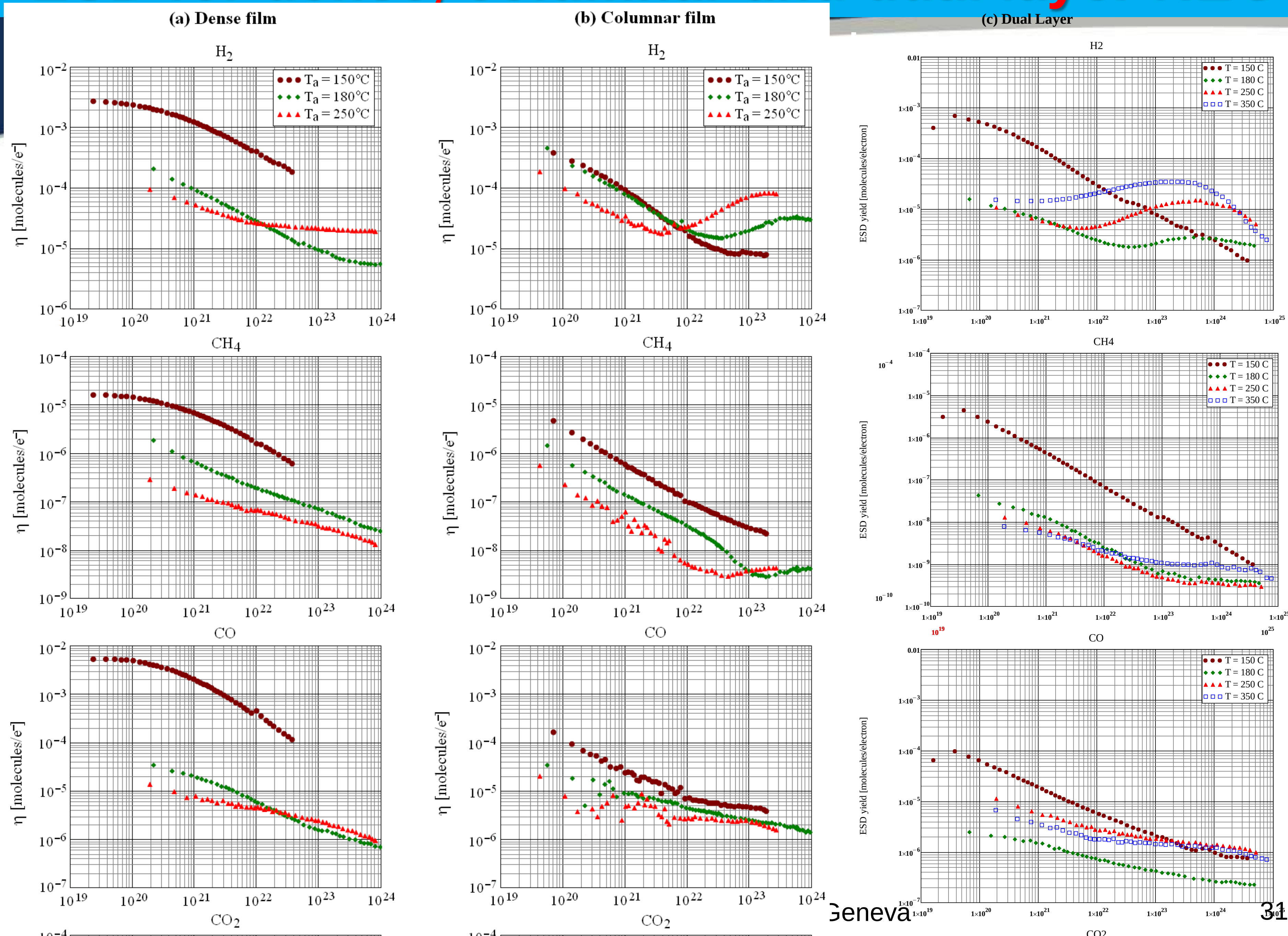






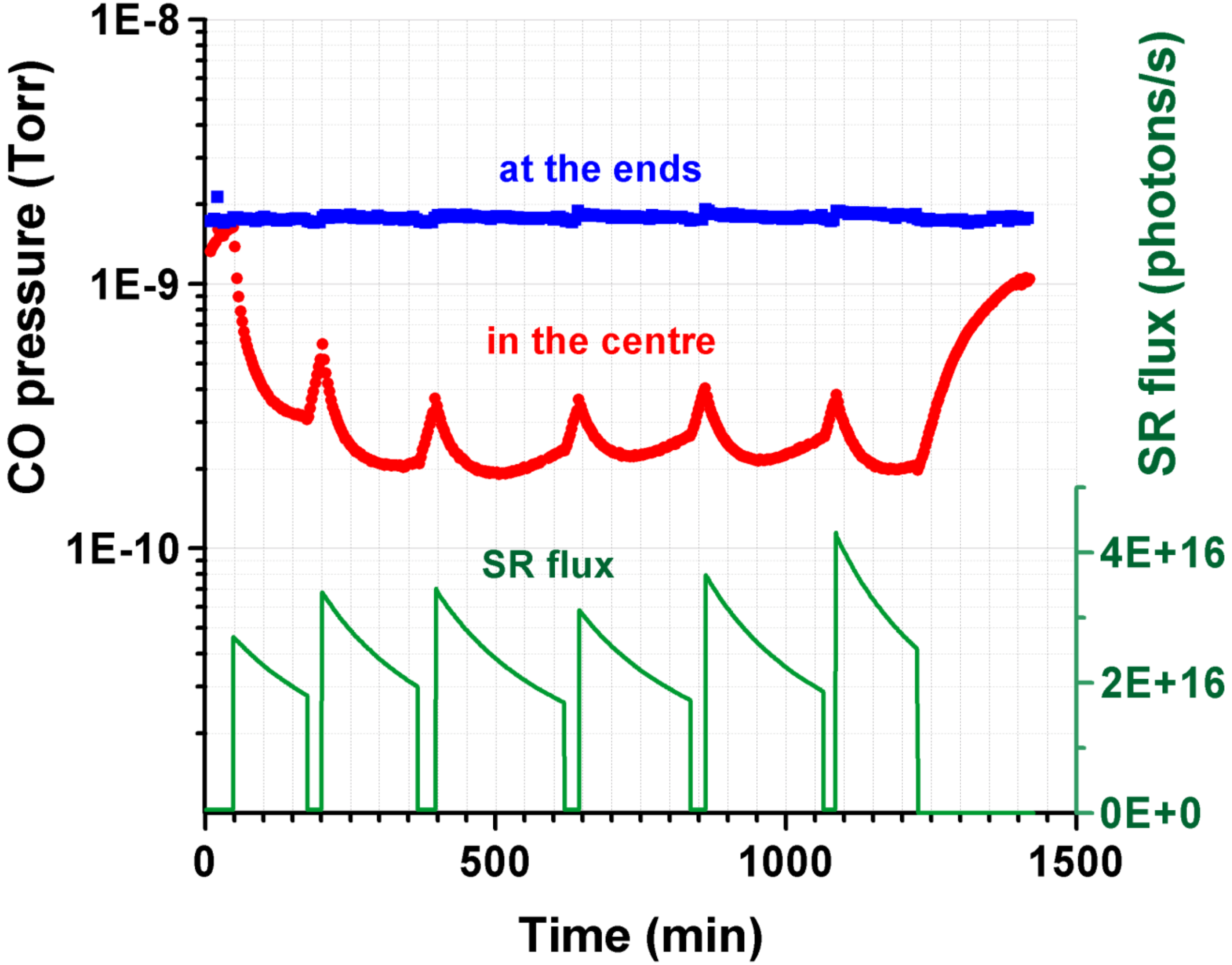
O.B. Malyshev, R. Valizadeh and A.N. Hannah. JVST A 34, 061302 (2016)

ESD for dense, columnar and dual layer NEG



NEG Coated Vacuum Chamber: SR Induced Pumping

NEG TiZrV coated surface saturated with CO (i.e. no pumping speed) exposed to SR

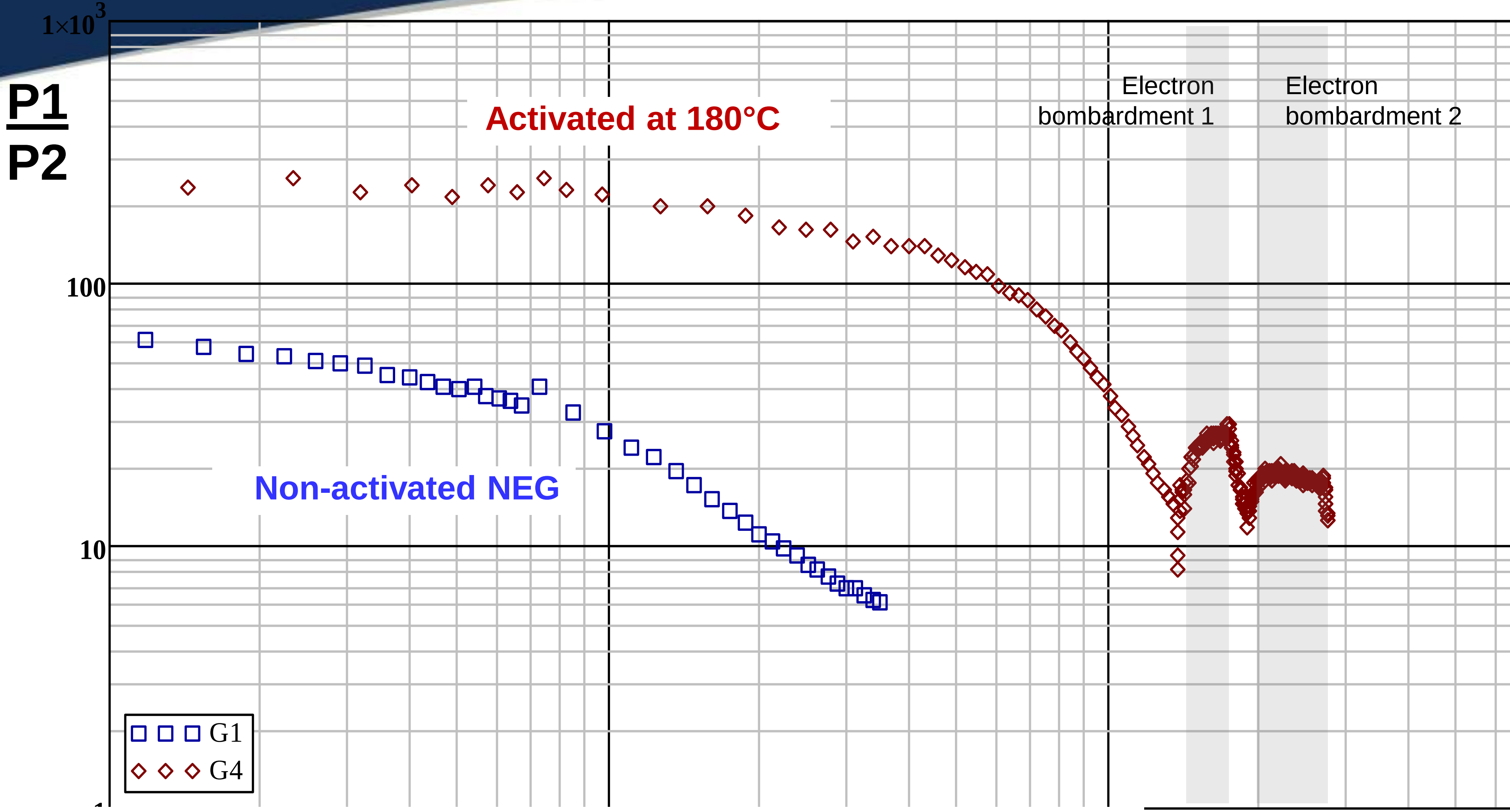


The photon stimulated NEG activation efficiency estimated as

$\sigma_\gamma = 2\times10^{-5}$ [CO/γ]

V.V. Anashin et al. Vacuum 75 (2004), p. 155.

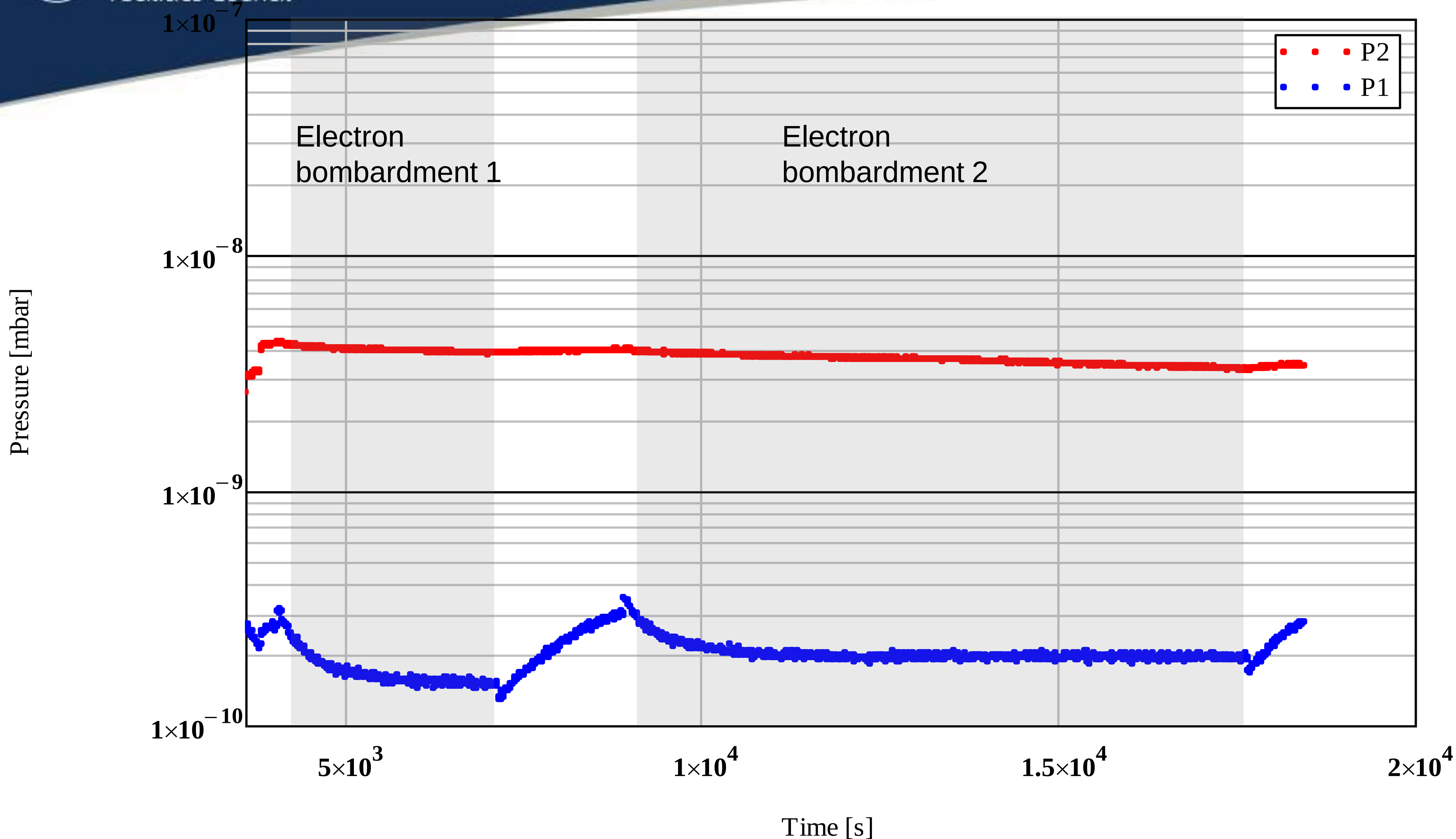
Electron stimulated NEG activation



The electron stimulated NEG activation efficiency estimated as **$7.9\times10^{-4} < \sigma_1 < 2.4\times10^{-3}$ [CO/e-]**

$$\sigma_1\left[\frac{CO}{e^-}\right]=\frac{\Omega_{CO}}{D}$$

Electron stimulated NEG activation

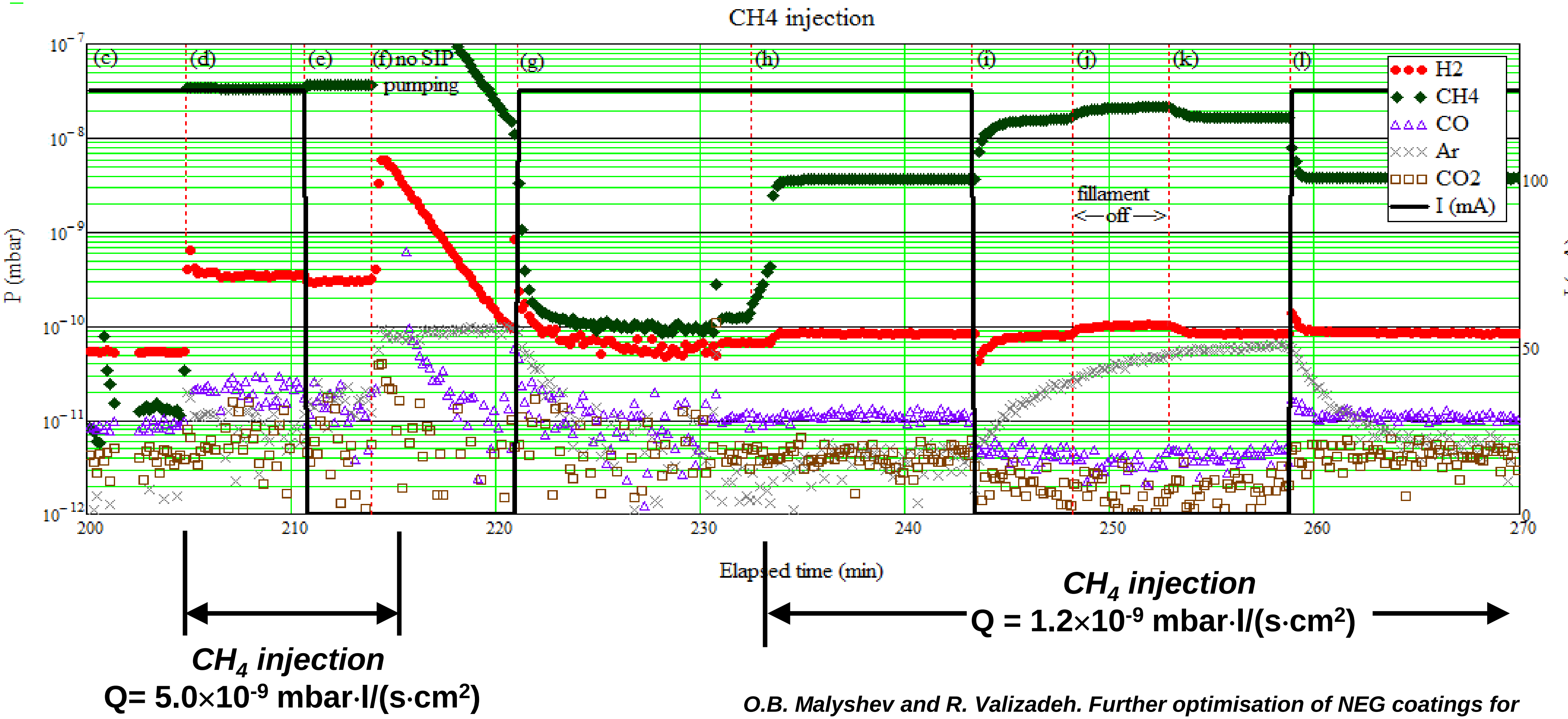


The electron stimulated NEG activation efficiency estimated as

$$\sigma_2 = \frac{Q_{CO}}{k_B T}\frac{q_e}{I} = 2.2\times10^{-3}\left[\frac{CO}{e^-}\right]$$

CH_4 problem

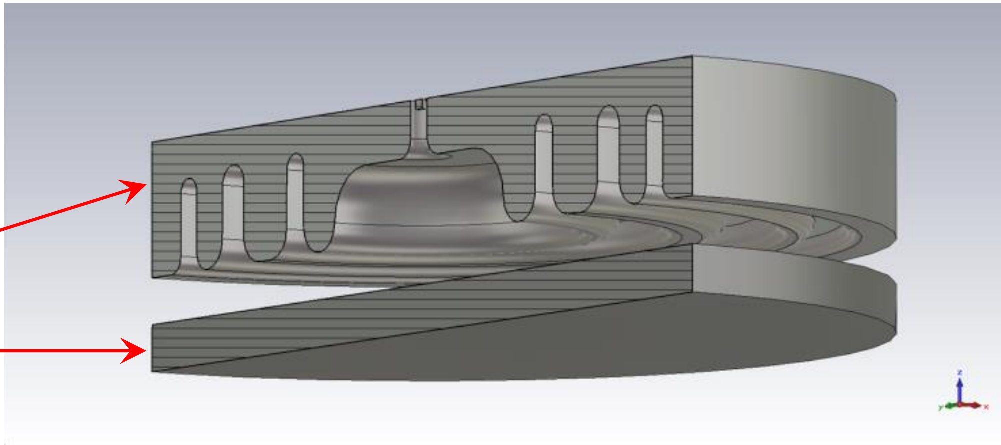
- NEG does not pump CH_4 and other hydrocarbons
- However, CH_4 can be pumped in a presence of SR or electron bombardment: $\chi = 2.3\times10^{-5}$ CH_4/e^-.



O.B. Malyshev and R. Valizadeh. Further optimisation of NEG coatings for accelerator beam chamber. Proc. IPAC-5 (2014) p. 2399.

Surface resistance: method

- The cavity geometry consists of two parts:
  - a body of the cavity
  - a planar sample,
  - separated by an air gap.
- Contactless
- RF chokes in order to keep the RF power within the cavity

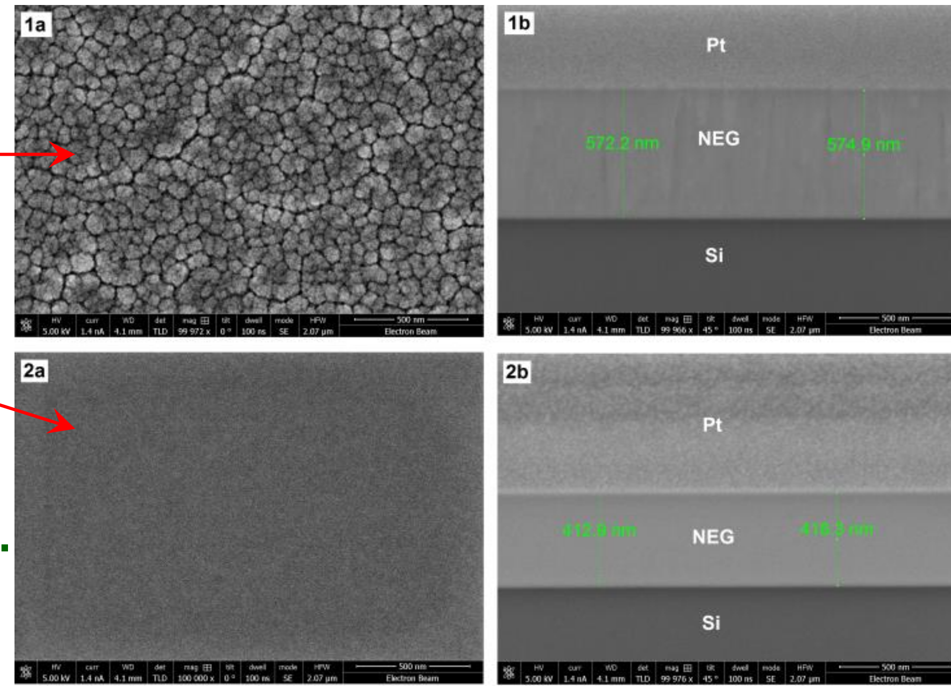


$$R_S^{sam} = \frac{G\,Q_0^{-1} - R_S^{cav}\,p_c}{p_s}$$

- Modelled with CST Microwave Studio.
- G = 235 Ω.
- The field ratios $p_c = 0.625$ and $p_s = 0.375$ for perfect electric conductor boundary conditions.

NEG coatings

- NEG films
  - columnar
  - dense
- Deposited on:
  - polycrystalline copper
  - silicon Si(100) substrates.
- The substrate size was 100 mm × 100 mm × 2 mm
- Sample thickness:
  - from 0.7 to 18 μm



O.B. Malyshev, L. Gurran, P. Goudket, K. Marinov, S. Wilde, R. Valizadeh and G. Burt.. Nucl. Instrum. Methods Phys. Res., A 844, 99-107 (2017)

Analytical model

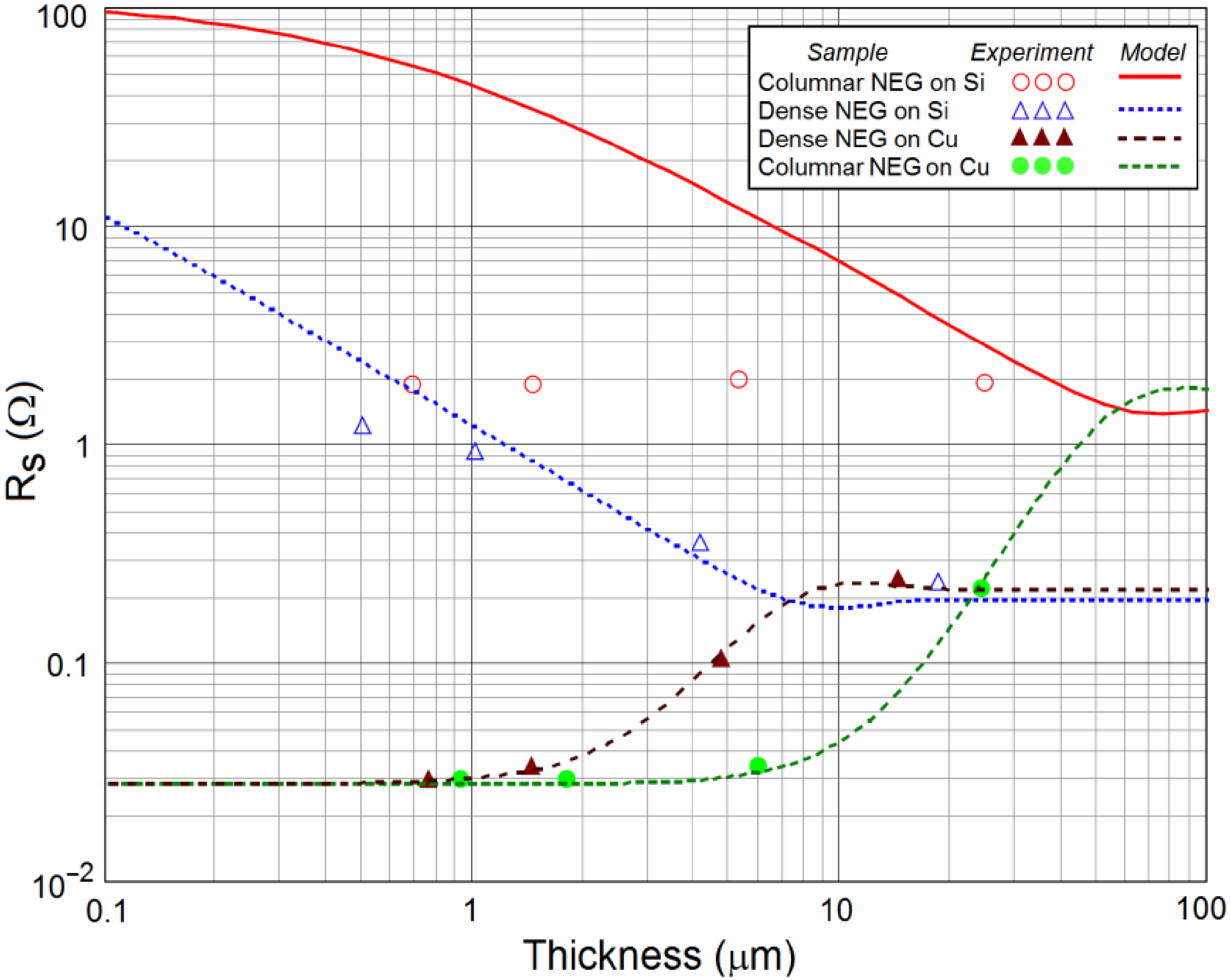
- The expressions for the surface impedance of a planar metallic film deposited on a substrate (dielectric or metallic) are derived by following the standard approach employed in calculating the transmission and reflection coefficients in layered media

$$R_S = R_1 \frac{1-\delta^2 \exp\left(-4\kappa_1 d_1\right) - 2\delta \sin\left(2\kappa_1 d_1\right)\exp\left(-2\kappa_1 d_1\right)}{1+\delta^2 \exp\left(-4\kappa_1 d_1\right) + 2\delta \cos\left(2\kappa_1 d_1\right)\exp\left(-2\kappa_1 d_1\right)} \quad \text{for NEG on metal substrate;}$$

$$R_S = R_1 \frac{1-\exp\left(-4\kappa_1 d_1\right) + 2\sin\left(2\kappa_1 d_1\right)\exp\left(-2\kappa_1 d_1\right)}{1+\exp\left(-4\kappa_1 d_1\right) - 2\cos\left(2\kappa_1 d_1\right)\exp\left(-2\kappa_1 d_1\right)} \quad \text{for NEG on Si substrate.}$$

O.B. Malyshev, L. Gurran, P. Goudket, K. Marinov, S. Wilde, R. Valizadeh and G. Burt.. Nucl. Instrum. Methods Phys. Res., A 844, 99-107 (2017)

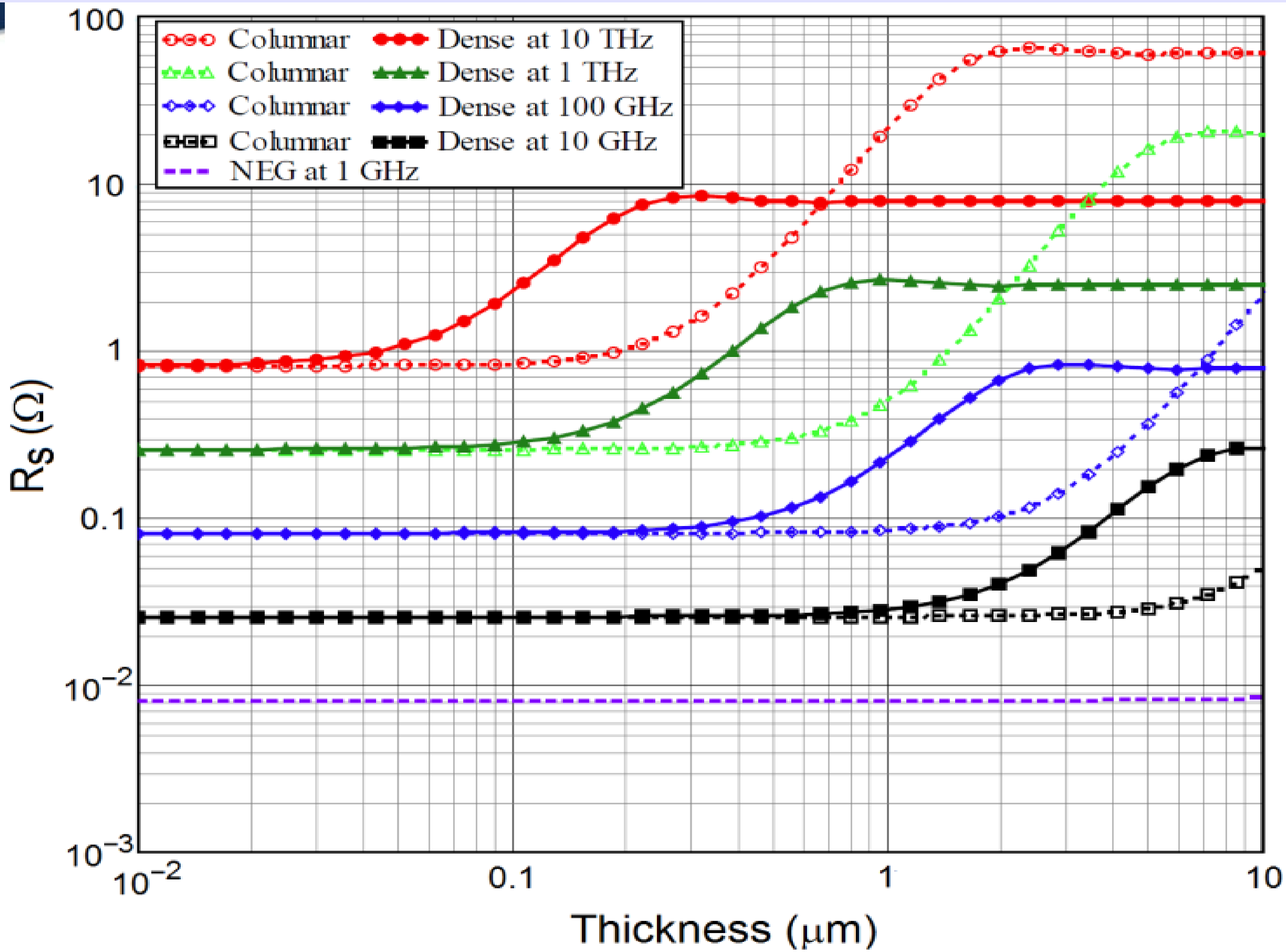
The surface resistance RS of dense and columnar NEG coatings on copper and silicon substrates as a function of film thickness



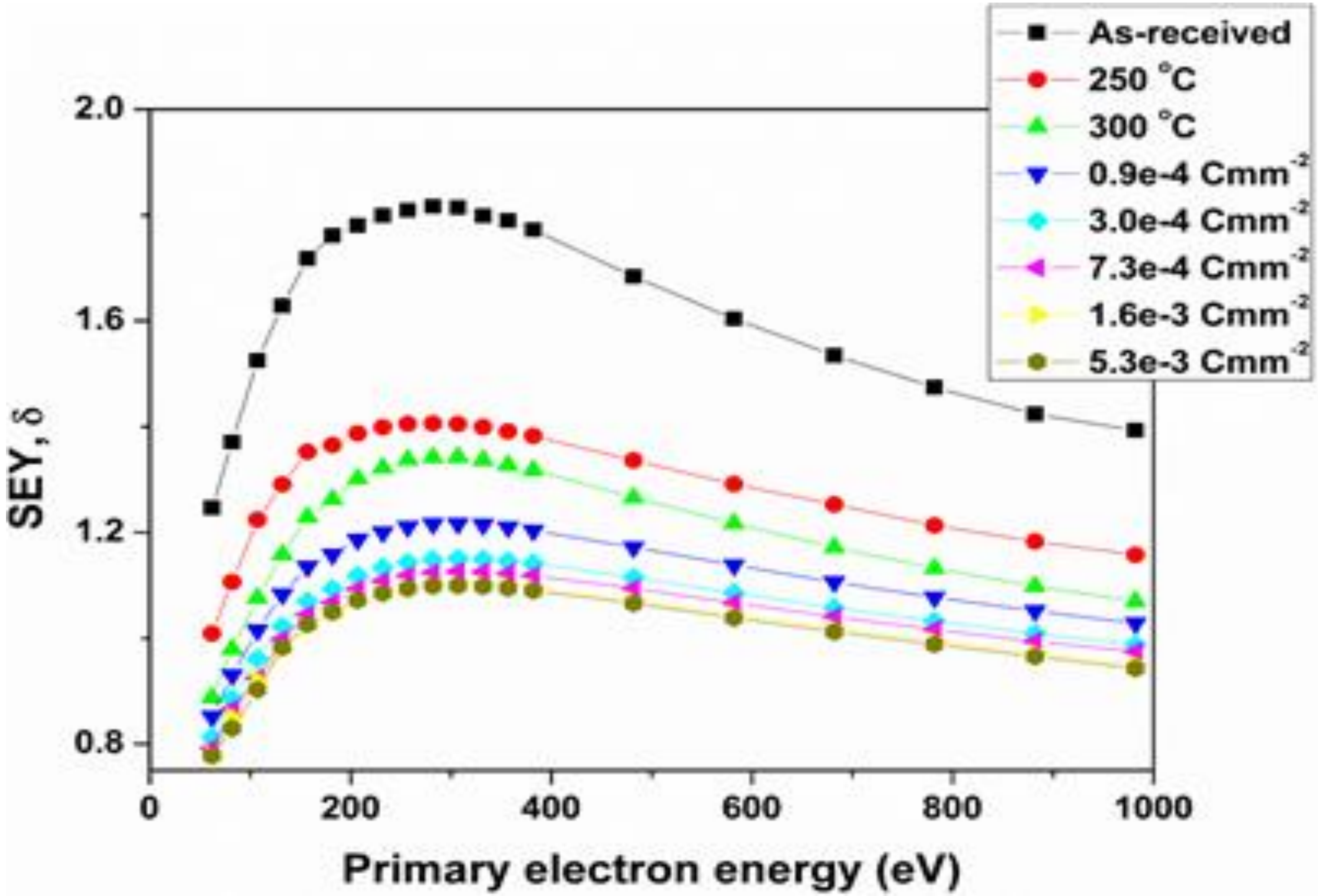
The bulk conductivity was obtained with the analytical model:

- $\sigma_d = 1.4\times10^4\ S/m$ for the columnar NEG coating
- $\sigma_d = 8\times10^5\ S/m$ for the dense NEG coating

The surface resistance R_S as a function of NEG film thickness on copper at various frequencies



SEY from columnar NEG



Science & Technology Facilities Council

Ongoing studies

- NEG coating of narrow tubes < 10 mm diameter
  - See presentations at IPAC’2018

- Higher electric conductivity NEG coatings
  - i.e. better than for Ti,Zr,Hf and V alloys
  - See presentations at IPAC’2018

Conclusions

- NEG coating is a technology that allows to meet **UHV/XHV** vacuum specification win long narrow vacuum chambers.
  - PSD and ESD After NEG activation at 180°C the initial η(316LN)/η(Ti-Zr-V) =
    - =20 for H_2, =1000 for CH_4 and =200 for CO.
    - Vacuum firing => an order of magnitude lower ESD
    - η(Ti-Zr-Hf-V) < η(Ti-Zr-V).
    - Best results is for the dense and dual layer NEG activated at 180 °C
  - Often the only vacuum solution
  - Lower cost of pumping system
- NEG film requires *activation* at 150-180 °C in stead of 250-300 °C usual bakeout:
  - Shorter bellows or less number of bellows
  - Wider choice of material for vacuum chamber and components
- SR (or electron bombardment) induced activation/pumping:
  - NEG can be (re-)activated by irradiation/bombardment
  - NEG can pump CH_4 molecules *during* irradiation/bombardment
- The bulk conductivity:
  - σd = 1.4×10^4 S/m for the columnar NEG coating
  - σd = 8×10^5 S/m for the dense NEG coating
- SEY < 1.1 can be obtained after activation or by conditioning

Acknowledgments

Co-authors (team):

ASTeC

- Dr. R. Valizadeh
- Mr. A.N. Hannah
- Mr. B.T. Hogan
- Mrs. R.M.A. Jones
- Mr. A.P. Smith
- Dr. K.J. Middleman
- Dr. K. Marinov
- Dr. P. Goudket
- Mr. L. Gurran
- Mr. S. Wilde
- Dr. S. Wang

MMU / Huddersfield Uni.

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

Lancaster University

- Dr. G. Burt

ASTeC / ISIS

- Dr. S. Patel