Progress in NEG Coatings for Particle Accelerators

O.B. Malyshev and R. Valizadeh,

ASTeC Vacuum Science Group,
STFC Daresbury Laboratory, UK

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Outlook

• Introduction
• Pumping properties
• Desorption properties
• Bombardment induced activation and pumping
• Surface resistance
• SEY
• Summary
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 CₓHᵧ and noble gases
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 $\alpha$ – 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)

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Using these result for the ILC-DR design

Average pressure after 100 Ahr beam conditioning:

inside a stainless steel tube
\( S_{\text{eff}} = 200 \text{ l/s every 5 m} \)

inside a NEG coated tube
\( S_{\text{eff}} = 20 \text{ l/s every 30 m} \)
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 $\sim$1 ML:
  • If $P = 10^{-9}$ mbar then 1 ML can be sorbed just in $\sim 10^3$ 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:
  • \( \eta(D, E, T_a) \), \( \alpha(M, T_a) \), pumping capacity;
• Better understanding:
  • what and why;
  • practical ‘do’s and ‘don’t’s;
• Further development of this coating:
  • lower \( \eta \), \( T_a \), SEY;
  • higher \( \alpha(M) \), pumping capacity;
  • optimising for an application.
Deposition method

Cylindrical magnetron deposition for vacuum chambers

Commonly used planar magnetron deposition

Diagram showing deposition method with labeled components:
- Vacuum pump
- Kr injection
- Ceramic feedthrough
- DC and pulsed DC power supply
- HiPIMS
- Target: Ti, Zr, Hf, V twisted and alloy wires
- Tubular sample
- Solenoid B=240 G
- Target (H, Zr, V)
- Permanent Magnet
- Pulsed DC power supply
- Test pieces
- Ceramic
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)

ZrTiV film deposited by DC Magnetron

\[ Zr_{0.32} Ti_{0.34} V_{0.34} \text{ thickness 960 nm} \]
SEM images of films (film morphology)

columnar
dense

Advantages of ASTeC activation procedure:

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

NEG pumping properties

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

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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,
  \( T = 120^\circ C \).

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,
\( T = 120^\circ \text{C} \).

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, $T = 120^\circ$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, $T = 120^\circ$C.

Very glassy structure.
Pumping properties of some NEG films

**CO sticking probability**

**H2 sticking probability**

**CO pumping capacity**

**Activation temperature [°C]**

**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

- \( \eta \) - desorption yield (photon, electron or ion stimulated desorption)
- \( \alpha \) - sticking probability

- Improving pumping properties is limited:
  \[ \alpha \leq 1 \]
  - \( 0.005 < \alpha_{H_2} < 0.02 \)
  - \( 0.1 < \alpha_{CO} < 0.5 \)
  - \( 0.4 < \alpha_{CO_2} < 0.6 \)

- Reducing the desorption yields \( \eta \) in orders of magnitude 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**
  - Best for pumping

- **dense**
  - A first candidate for a barrier

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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

ESD yield from NEG coated samples

(a) Dense film

(b) Columnar film

316LN

$T_b = 250^\circ C$

O.B. Malyshev, R. Valizadeh, et al.
Vacuum 86, 2035 (2012).
ESD yield from NEG coated samples

(a) Dense film

(b) Columnar film

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H$_2$ ESD from NEG coated vacuum fired 316LN

(a) Columnar film

(b) Dense film

316LN
$T_b = 250^\circ$C

Vacuum fired

η [molecules/e$^-$]

No vacuum firing

η [molecules/e$^-$]

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

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

(a) Dense film

H₂

CH₄

CO

CO₂

dose [electrons/m²]

ESD yield [molecules/electron]

T = 150 °C
T = 180 °C
T = 250 °C
T = 350 °C

(b) Columnar film

H₂

CH₄

CO

CO₂

dose [electrons/m²]

ESD yield [molecules/electron]

T = 150 °C
T = 180 °C
T = 250 °C
T = 350 °C

(c) Dual Layer

H₂

CH₄

CO

CO₂

dose [electrons/m²]

ESD yield [molecules/electron]

T = 150 °C
T = 180 °C
T = 250 °C
T = 350 °C
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

\[ \sigma_\gamma = 2 \times 10^{-5} \ [\text{CO}/\gamma] \]

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

\[ \sigma_2 = \frac{Q_{\text{CO}}}{k_B T} \frac{q_e}{I} = 2.2 \times 10^{-3} \left[ \frac{\text{CO}}{e^{-}} \right] \]
• NEG does not pump CH$_4$ and other hydrocarbons
• However, CH$_4$ can be pumped in a presence of SR or electron bombardment: $\chi = 2.3 \times 10^{-5}$ CH$_4$/e$^-$.

$\text{CH}_4$ injection

$Q = 5.0 \times 10^{-9}$ mbar l/(s cm$^2$)

$\text{CH}_4$ injection

$Q = 1.2 \times 10^{-9}$ mbar l/(s cm$^2$)

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.

Surface resistance: method

\[ R_{sam}^S = G Q_0^{-1} - R_{cav}^S \frac{p_c}{p_s} \]

- Modelled with CST Microwave Studio.
- \( G = 235 \, \Omega \).
- 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
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:

\[ R_s = R_i \frac{1 - \delta^2 \exp(-4\kappa_1 d_1) - 2\delta \sin(2\kappa_1 d_1) \exp(-2\kappa_1 d_1)}{1 + \delta^2 \exp(-4\kappa_1 d_1) + 2\delta \cos(2\kappa_1 d_1) \exp(-2\kappa_1 d_1)} \]

for NEG on metal substrate;

\[ R_s = R_i \frac{1 - \exp(-4\kappa_1 d_1) + 2\sin(2\kappa_1 d_1) \exp(-2\kappa_1 d_1)}{1 + \exp(-4\kappa_1 d_1) - 2\cos(2\kappa_1 d_1) \exp(-2\kappa_1 d_1)} \]

for NEG on Si substrate.
The surface resistance $R_S$ 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:
- $\sigma_d = 1.4 \times 10^4 \text{ S/m}$ for the columnar NEG coating
- $\sigma_d = 8 \times 10^5 \text{ S/m}$ 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

![Chart showing SEY as a function of primary electron energy for different conditions.]

- As-received
- 250 °C
- 300 °C
- 0.9e-4 Cmm^-2
- 3.0e-4 Cmm^-2
- 7.3e-4 Cmm^-2
- 1.6e-3 Cmm^-2
- 5.3e-3 Cmm^-2

Primary electron energy (eV)
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 $\eta(316LN)/\eta$(Ti-Zr-V) =
    • $\approx$20 for H$_2$, $\approx$1000 for CH$_4$ and $\approx$200 for CO.
    • Vacuum firing => an order of magnitude lower ESD
    • $\eta$(Ti-Zr-Hf-V) < $\eta$(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:
  • $\sigma d = 1.4 \times 10^4$ $S/m$ for the columnar NEG coating
  • $\sigma d = 8 \times 10^5$ $S/m$ for the dense NEG coating

• SEY < 1.1 can be obtained after activation or by conditioning
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Co-authors (team):

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