Optimization of non-evaporable getter coating for accelerator beam pipe

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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 $S_{\text{eff}}$.

In a simple case it is

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

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

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

\[
\langle P \rangle = qL \left( \frac{L}{12u} + \frac{1}{2S_{\text{eff}}} \right) k_B T
\]
Vacuum chamber cross sections

Beam pipe
Circular or elliptical
4 mm ≤ d, a, b ≤ 200 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 CₓHᵧ and noble gases
Stainless steel vs. NEG coated vacuum chamber under SR

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 CO$_2$ is $\sim$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 $\eta$, $T_a$, 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 \( \alpha \) – sticking probability,
  - \( A \) – surface area,
  - \( v \) – mean molecular velocity
Deposition method

Planar magnetron deposition

Cylindrical magnetron deposition

Vacuum pump

Kr injection

Sample

Plasma

Target (Ti, Zr, V)

Pulsed DC power supply

Permanent Magnet

Solenoid

Target: Ti-Zr-V twisted and alloy wires

Vacuum pump

Ceramic

Pulsed DC power supply

HV

WS-63, 14-19 September 2010, Ávila
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

$Zr_{0.32}Ti_{0.34}V_{0.34}$ thickness 960 nm

- Yield
- Channel number

WS-63, 14-19 September 2010, Ávila, Spain
The EDX analysis for determination of film composition
SEM images of films (film morphology)

columnar

dense

Set-up for NEG pumping evaluation


ASTeC activation procedure

Pressure ratio $P_1/P_2$ measured during gas injection is used to estimate:
initial sticking probability and sorption capacity.
Titanium film deposited on Si test sample from a single Ti wire

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.
Vanadium film deposited on Si test sample from a single V wire.

Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.16 nm/s, $T = 120^\circ$C. Average grain size 100 nm. Rhombohedral lattice structure.
Hf film deposited on Si test sample from a single Hf wire.

Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.16 nm/s, $T = 120^\circ$C. Average grain size 100 – 150 nm. Hexagonal lattice structure.
Zr film deposited on a Si test sample from a single Zr wire

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. Hexagonal lattice structure.
Single metal pumping properties

Zr is best:
Lowest activation Temp. and highest capacity

Hf

Ti

V has highest activation temperature
Ti-V film deposited on Si test sample from twisted Ti and V wires.

Cylindrical Magnetron:
Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.13 nm/s, $T = 120^\circ$C.
Average grain size 50 – 100 nm.
Hexagonal lattice structure.
Ti-Zr film deposited on Si test sample from twisted Ti and Zr wires

Cylindrical Magnetron: Power = 60 W, \( P_{Kr} = 10^{-2} \) mbar, deposition rate = 0.16 nm/s, \( T = 120^\circ C \).

Average grain size 50 – 100 nm. Hexagonal lattice structure.
Zr-V film deposited on Si test sample from twisted Zr an V wires.

Cylindrical Magnetron: Power = 60 W, $P_{Kr} = 10^{-2}$ mbar, deposition rate = 0.15 nm/s, T = 120°C. Average grain size 10 – 20 nm.
**Binary alloy pumping properties**

\[ egin{array}{c}
\text{Zr-V is best} \\
\text{Ti-Zr activation temperature is lower than for Ti-V} \\
\text{Zr-Hf was not studied}
\end{array} \]
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.
Ternary alloy pumping properties

**Graph 1:**
- CO sticking probability
- Activation temperature [°C]
- Ti-Zr-V, Hf-Zr-V, Ti-Zr-Hf, Ti-Hf-V are comparable
- Ti-Zr-V has the highest activation temperature

**Graph 2:**
- H2 sticking probability
- CO pumping capacity
- Ti-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.

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 Ta

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.
**Quaternary alloy pumping properties**

- **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
- \( \alpha \) - 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 \( \eta \)
  - in orders of magnitude is a realistic task
Average gas density in the ILC undulator: \(d=4\) mm
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
SEM images of films (film morphology)

**columnar**

Best for pumping

**dense**

A first candidate for a barrier

Electron stimulated desorption

**Modified NEG pumping properties evaluation rig:**

- To measure sticking probability $\alpha$
- To measure electron stimulated gas desorption as a function of
  - Electron energy
  - Dose
  - Wall temperature (20-100°C)
  - Activation/bakeout temperature
- Can be used for samples with:
  - NEG coating
  - Low desorption coating
  - No coatings
Electron Bombardment

Filament: Th/W, Th/Ir or Y/Ir

$\text{CH}_4$, CO, $\text{H}_2$, $\text{CO}_2$
**Electron Stimulated Desorption (ESD) studies programme**

- ESD as a function of
  - Activation/bakeout temperature
  - Electron energy
  - Electron dose
  - Coating density, morphology and structure
  - Deposition conditions
  - Substrate
Experimental procedure for NEG coated samples

- Filament activation
- Degas hot filaments
- Chamber
- 500-eV electron bombardment
- 180 °C NEG activation
- 250 °C NEG activation
- Energy Dependence
- 500-eV electron bombardment
- 500-eV electron bombardment
- Filament

EVC-11, 20-24 September 2010, Salamanca, Spain
ESD: stainless steel vs non-activated NEG coated vacuum chamber

Baked to 250°C for 24 hrs  Baked to 80°C for 24 hrs
Pumped for 30 days 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

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

\[ \sigma_2 = \frac{Q_{CO}}{k_B T} \frac{q_e}{I} = 2.2 \times 10^{-3} \left[ \frac{CO}{e^-} \right] \]
Normalised pressure $P_1$

Columnar vs. Dense

\[ \alpha_c(H_2) = 1.5 \alpha_d(H_2); \quad \alpha_c(CO) = 1.5 \alpha_d(CO) \]
ESD Yields
Columnar vs. Dense
$\eta(E_{e-})$ for different gases for NEG coating
η(E_{e-}) for different gases for 316LN

[Graph showing yield versus energy for different gases including H2, CH4, CO, CO2 with respective fits.]
η(E_{e-}) for different gases for aluminium alloy

![Graph showing yield vs energy for various gases including H2, CH4, H2O, CO, O2, Ar, CO2, H2 fit, CH4 fit 1, CH4 fit 2, H2O fit, CO fit, O2 fit, Ar fit, CO2 fit. The graph plots yield in molecules/electron on the y-axis against energy in eV on the x-axis.]
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 $\text{H}_2$, 1000 times lower for $\text{CH}_4$ 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 5 keV.
  - a linear dependence was measured for most of gases
  - except for $\text{H}_2$, for which the dependence is: $\eta(E) \propto E^{2/3}$

- 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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