Vacuum Science and Technology for Accelerator Vacuum Systems

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- Vacuum Instrumentation
- **Vacuum Pumps**
  - Vacuum Components/Hardware
  - Vacuum Systems Engineering
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SESSION 3.2B: Getters

- Getters pump gases by chemically bonding molecules upon impingement
- Two definitions of pumping capacities:
  - Activation capacity
  - Termination capacity
- Based on activation manner, there are two types of getters:
  - Titanium sublimation pumps (TiSPs)
  - Non-evaporable getters (NEGs)
- Both TiSPs and NEGs are widely employed in accelerator vacuum systems
NEG – The Basics

- Porous alloys with very large active metallic surface area, when activated.

- Bulk Getters - gases diffuse into the interior of the getter material upon heating.

- Gases are categorized into four families based on their interactions with NEGs:
  2. CO, CO₂, O₂, and N₂ - adsorbed irreversibly.
  3. H₂O, hydrocarbons - adsorbed in a combination of reversible and irreversible processes. Hydrocarbons are adsorbed very slowly.
  4. Noble gases - not adsorbed at all.
Commercial NEG

- NEG is available only from:
  
  SAES Getters S.p.A.
  Viale Italia, 77
  20020 Lainate (Milano) Italy

  SAES Getters U.S.A., Inc.
  1122 E. Cheyenne Mountain Blvd.
  Colorado Springs, CO 80906
Hydrogen

- Hydrogen does not form a stable chemical composition with a NEG alloy. It diffuses rapidly into the bulk of the getter and is stored as a solid solution.

- Sievert’s Law describes the relationship between $H_2$ concentration within its NEG and its equilibrium pressure.

$$\log P = A + 2 \log q - \frac{B}{T}$$

$q = H_2$ concentration in NEG, Torr - liters/gram

$p = H_2$ equilibrium pressure, Torr

$T =$ getter temperature, K

$A$, $B$ constants for different NEG alloys
**NEG Pumping Characteristics (2)**

**CO, CO₂, O₂, N₂, other O-, C-containing molecules**

- Active gases are chemisorbed irreversibly by NEGs.

- The chemical bonds of the gas molecules are broken on the surface of the NEG.

- The various gas atoms are chemisorbed forming oxides, nitrides, and carbides.

- High temperatures do not break these chemical bonds. High temperatures promote diffusion into the bulk of the NEG.
H₂O and Hydrocarbons

- Water vapor and hydrocarbons are “cracked” on the surface of the NEG.
- H₂ is chemisorbed reversibly.
- O₂ and C are chemisorbed irreversibly.
- However, hydrocarbons sorption efficiency below 500°C is extremely low.
Noble gases

- NEG do not sorb Ar, He, Kr, Xe.

- Ion pumps are required in combination with NEG for pumping rare gases.
NEG Pumping Characteristics (5)

- At low throughput, NEG pumping speeds are constant, independent of pressure.
- Pumping speeds do, however, vary with NEG temperature.
Activation Process for NEG

Ref. SAES Getters
Application Notes for NEGs

- NEG performance deteriorates due to successive exposures to air (oxygen and water) or N₂.

- Further improvement can be obtained if Argon is used as a protective gas, during long term storage.

- NEG pumps should never be exposed to air while at temperatures higher than 50°C.

- Degassing of NEGs during initial pump-down.

Ref. SAES Getters
SAES ST101® Non-evaporable Getters

- Metal alloy made up of 84% Zr, 16% Al.

- First Zirconium based getter alloy introduced and still widely used today after 30 years.

- The operating temperature range of ST101 is 0 to 450°C.

- ST101 chemisorbs CO, CO₂, H₂O, N₂, and O₂ at high rates.

- ST101 activates at temperatures from 550 to 900°C.

Ref. SAES Getters

ST 101 Alloy Activation Efficiency
SAES ST101® NEG – Pumping

Ref. SAES Getters
SAES ST101® NEG – Hydrogen Solubility

H₂ dissolved within St 101 alloy
ST101 Surface Composition vs. Temperature
SAES ST707® Non-evaporable Getter

- **Metal alloy made up of 70% Zr, 24.6% Va, and 5.4% Fe.**

- **The operating temperature range of ST707 is 20 to 100°C.**

- **ST707 chemisorbs CO, CO₂, H₂O, N₂, and O₂ at high rates.**

- **ST707 has much lower activation temperature.**

![Fig. 1. Activation conditions and gettering efficiency of St 707](image-url)
SAES ST707® NEG Pumping Performance

St 707 powder alloy: 100 mg
Geometric surface: 50 mm$^2$
Activation: 450°C for 10 min.
Sorption: At the indicated temperatures
**ST707 Surface Composition vs. Temperature**

![Graph showing Surface Composition vs. Temperature](image)

- **Surface Composition (Atomic %)**
- **Temperature (C)**
Other SAES NEG Alloys

- ST 172 - Zr, V, and Fe alloy.

- ST 175 - Ti and Mo powder mixture, sintered form.

- ST 185 - Ti-V alloy (obsolete !)
NEG Cartridge Pump Module – CapaciTorr®

- Complete compact pumping system, with matching controller for easy activation
- NEG materials: st172 blades/disks
- Pump sizes from 50 l/s to 2000 l/s, for H₂
- For large sizes, the NEG cartridges are replaceable
CapaciTorr® Pumping Performance

CapaciTorr D 2000 MK5 (nude)

Pumping Speed (l/s)

Sorption temperature: 25 °C
Activation: 450 °C x 45'
Sorption pressure: 3e-6 Torr

Sorbed Quantity (Torr.l)

H₂, N₂, CO

nude — with CF100 body

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NEG – Ion Pump Combination – NexTorr®

500 l/s VacIon Plus

NexTorr D500-5
Pumping Performance – NexTorr®

Sorption temperature: 25 °C
Activation: 1h @ 550°C
## Main Technical Parameters – NexTorr® D500-5

<table>
<thead>
<tr>
<th>Initial pumping speed (l/s)</th>
<th>Gas</th>
<th>NEG activated</th>
<th>NEG saturated</th>
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<tbody>
<tr>
<td>O₂</td>
<td>500</td>
<td>4</td>
<td></td>
</tr>
<tr>
<td>H₂</td>
<td>500</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>CO</td>
<td>340</td>
<td>5</td>
<td></td>
</tr>
<tr>
<td>N₂</td>
<td>200</td>
<td>4</td>
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</tr>
<tr>
<td>CH₄</td>
<td>13</td>
<td>5</td>
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<tr>
<td>Argon¹</td>
<td>6</td>
<td>6</td>
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</table>

<table>
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<tr>
<th>Sorption capacity (Torr·l)</th>
<th>Gas</th>
<th>Single run capacity</th>
<th>Total capacity</th>
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<tbody>
<tr>
<td>O₂</td>
<td>17</td>
<td>&gt;1500</td>
<td>N/A</td>
</tr>
<tr>
<td>H₂</td>
<td>670</td>
<td>N/A</td>
<td>&gt;360</td>
</tr>
<tr>
<td>CO</td>
<td>1.4</td>
<td>&gt;75</td>
<td></td>
</tr>
<tr>
<td>N₂</td>
<td>0.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CH₄</td>
<td>137</td>
<td>50,000 hours at 10⁻⁶ Torr</td>
<td></td>
</tr>
</tbody>
</table>

### NEG section
- Getter alloy type: St 172
- Alloy composition: ZrVFe
- Getter mass (g): 68 g
- Getter surface (cm²): 570

### ION section
- Voltage applied: DC +5kV
- Number of Penning cells: 4
- Standard bake-out temperature: 150°C
Other NEG forms – Build your own pumps
Distributed Pumping with NEG strips

APS Beampipe with NEG strips
LLNL NEG Pump in a PEP-II Vacuum Chamber
Combination Pumping . . .
Ion Pumps with TSP or NEG

- Combination pumping produces greater pumping speeds for all gases.
  - TSP and NEG provide high pumping speeds for getterable gases.
  - Ion Pumps provide pumping of argon and light hydrocarbons (usually Noble Diode pumps are chosen).

- Combination pumping can be attained by:
  - Commercial combination pumps
  - Custom built combination pumps
  - Use of multiple types of pumps

- NEGs are used on systems where high constant pump speeds are required or on systems requiring distributed pumping.

- TSPs are used on systems with sudden large gas bursts, localized gas sources and/or frequent venting takes place.
Commercial Combination Pumps . . .
Ion Pumps with TSP or NEG

Ion Pump with TSP filaments
Ion Pump with NEG cartridge
NEG Thin Film for Accelerators

- Developed at CERN, by Bevenuti, et al

Low Outgassing Rates

Discrete Pumping

Distributed Pumping

Integrated Pumping

NEG Coating

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Typical Sputtering Arrangement – A CLASSE Setup

- Cathode – Twisted wires
- Electric field (ion energy) 
  ~ 600 V
- Magnetic field :
  200 ~ 500 Gauss
- Sputtering gas : Ar or Kr 
  $P = 2 \sim 20$ mtorr

- DC or Magnetron Sputtering arrangement is commonly used.
- Coating surface cleanness is essential for good adhesion
- Sputtering gas purity extremely important
NEG Thin Film Characteristics

- *Most commonly deposited NEG thin films have elementary composition of Zr\textsubscript{x}V\textsubscript{y}Ti\textsubscript{z}, with x, y, z, close to unity.*

- *Stoichiometric balanced thin film tend to have lower activation temperature, probably due to smaller grain sizes.*

- *Pumping can be achieved at activation temperature as low as 150°C, though typical ~250°C. Thus an in-situ bakeout can activate the NEG coating.*

- *Typical NEG thin film thickness: 2~4 μm.*
NEG Coating Pumping Performance (1)

$T_{act}=350^\circ C$

![Graph showing CO and H₂ pumping performance with time in hours.]
NEG Coating Pumping Performance (2)

Pumping Speed vs. Gas-load
Activation Temperature Dependence (48-hr activation)
**NEG Film Total Capacity & Aging Effects**

- Total pumping capacity of a NEG thin film depends on the film’s solubility to oxygen, carbon, nitrogen, etc., and the film thickness

  Using solubility of 5%, 1-nm saturated surface oxide layer
  Estimated saturation/venting cycles for 1 \( \mu \text{m} \) NEG film > 50

- Gradual aging is a deterioration of the thin film performance due to accumulation of oxygen in the film

  - Reduction of pumping speed and capacity
  - Increase of activation temperature

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NEG Film Aging Effect
NEG Film Aging – More

![Graph showing hydrogen pumping speed versus number of heating/venting cycles at different temperatures.](image-url)
Successful Applications of NEG Coatings

• NEG coating is an idea solution for long narrow-gapped undulator vacuum chambers
• All LHC warm beampipes were NEG coated.
• ESRF has had a very successful experience with the NEG-coated undulator chambers.
• Other new 3rd generation SR light sources, such as SOLEIL and DIAMOND, also used the NEG coatings for the undulator chambers.
• A NEG Coating Workshop was held at DIAMOND site, on 23/24 September 2002.
CERN’s NEG Coating Facility

~ 8m
**CERN’s NEG Coating Facility – Details**

Solenoid

- L = 8m
- φ = 60cm

Manifold

Chambers

Extensions

3mm wires of Ti, Zr and V
CERN’s NEG Coating Production

More than 1300 chambers coated with TiZrV NEG for the LHC.
Standard chambers are 7 m long, 80 mm diameter.
ESRF’s NEG Coating Facility

A New NEG Coating Building @ESRF

Extruded Al-Chamber 5-m long, 11-mm Gap

Motorized Air-cool Solenoid (500 G @100Amp)
IntegraTorr® – SAES Getters’ NEG Coating

- **SAES Getters is licensed by CERN to provide commercial NEG coating services.**

- **All components to be coated by SAES are cleaned by CERN facility, to ensure good thin film adhesiveness.**

- **Known projects used this services: RHIC, CesrTA, etc.**

One of the SAES sputtering systems for NEG coating, capable to coat up to 6.5 meter long chambers with a 2m height coil.
Hydrogen Embrittlement of NEGs are well known

**Word of Caution**

- The original coating had excellent bonding, by visual inspection and/or via ‘tape testing’
- Believe the coating was damaged by excessive $H_2$ sorption. More investigation planned

*Powder substance were found on the orifice disk, as well as on the coated surface, after extensive pumping tests*
Powder Confirmed to Be NEG

Powder SEM Image  Powder EDX Spectrum
NEGs or TiSPs

- Both TiSPs and NEGs are great in dealing with hydrogen gas load, the main gas in an UHV system
- If space available, TiSPs are the first choice
  - Much less cost
  - More operational friendly
  - 'Un-limited' capacity
- Some practical questions regarding NEG
  - How to reduce hydrogen from NEG?
  - Should the NEG be thoroughly de-hydrogen before installation? Or is that possible?
  - What's sources of hydrogen in the commercial NEG modules/cartridges (in the NEG materials, or in the heating elements)?
  - What's the best way to passivate NEG for air exposure?