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Vacuum Science and Technology for Accelerator Vacuum Systems

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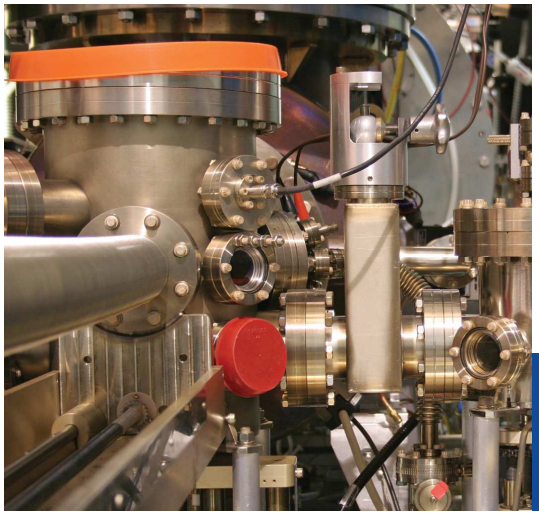

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- Vacuum Fundamentals
- Vacuum Instrumentation
- **Vacuum Pumps**
- Vacuum Components/Hardware
- Vacuum Systems Engineering
- Accelerator Vacuum Considerations, etc.

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

NEGs – 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:**
  - ✓ **1. Hydrogen and its isotopes - adsorbed reversibly.**
  - ✓ **2. CO, CO_2, O_2, and N_2 - adsorbed irreversibly.**
  - ✓ **3. H_2O, hydrocarbons - adsorbed in a combination of reversible and irreversible processes. Hydrocarbons are adsorbed very slowly.**
  - ✓ **4. Noble gases - not adsorbed at all.**

Commercial NEGs

- **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

NEG Pumping Characteristics (1)

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.*

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

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, CO_2, O_2, N_2, 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.

NEG Pumping Characteristics (3)

H_2O and Hydrocarbons

- Water vapor and hydrocarbons are "cracked" on the surface of the NEG.
- H_2 is chemisorbed reversibly.
- O_2 and C are chemisorbed irreversibly.
- However, hydrocarbons sorption efficiency below 500°C is extremely low.

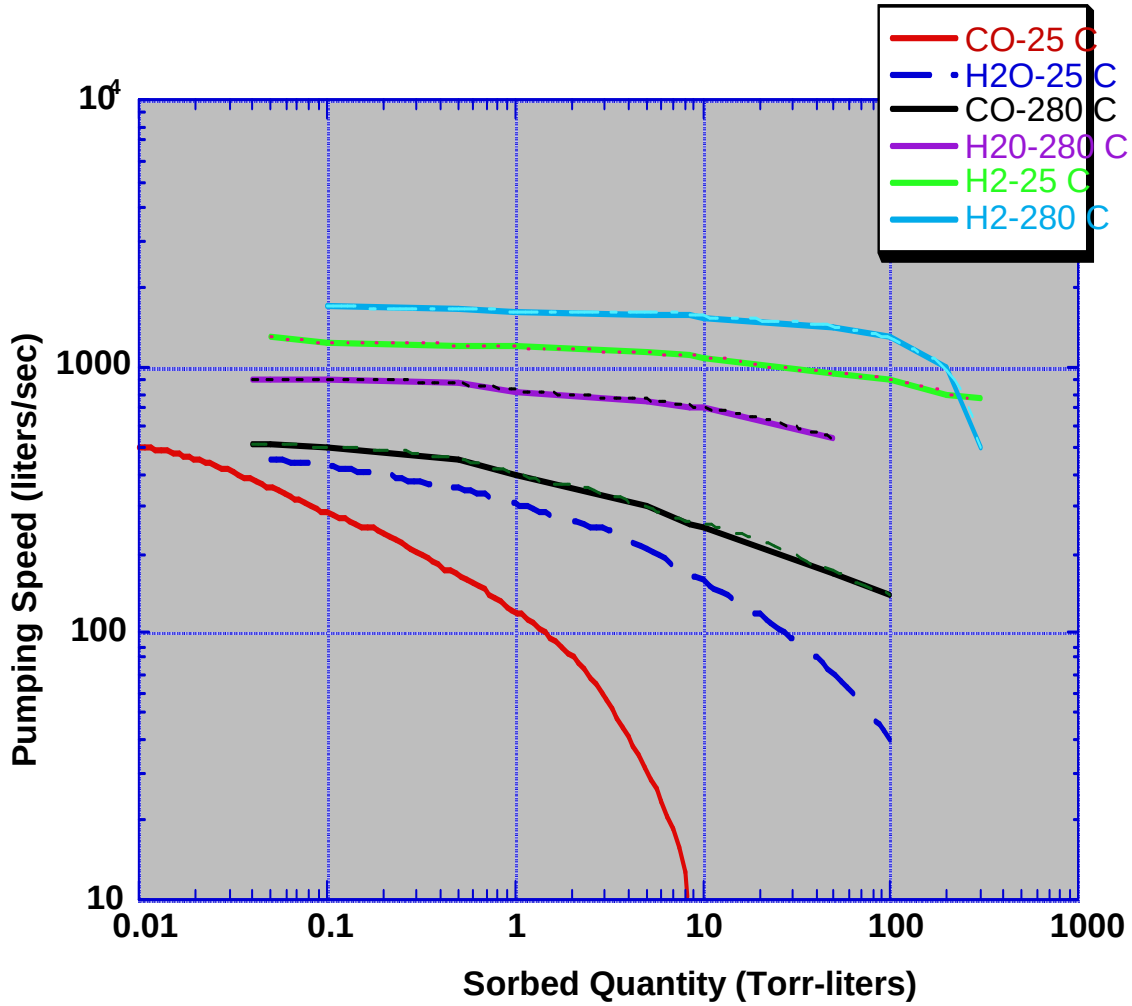
NEG Pumping Characteristics (4)

Noble gases

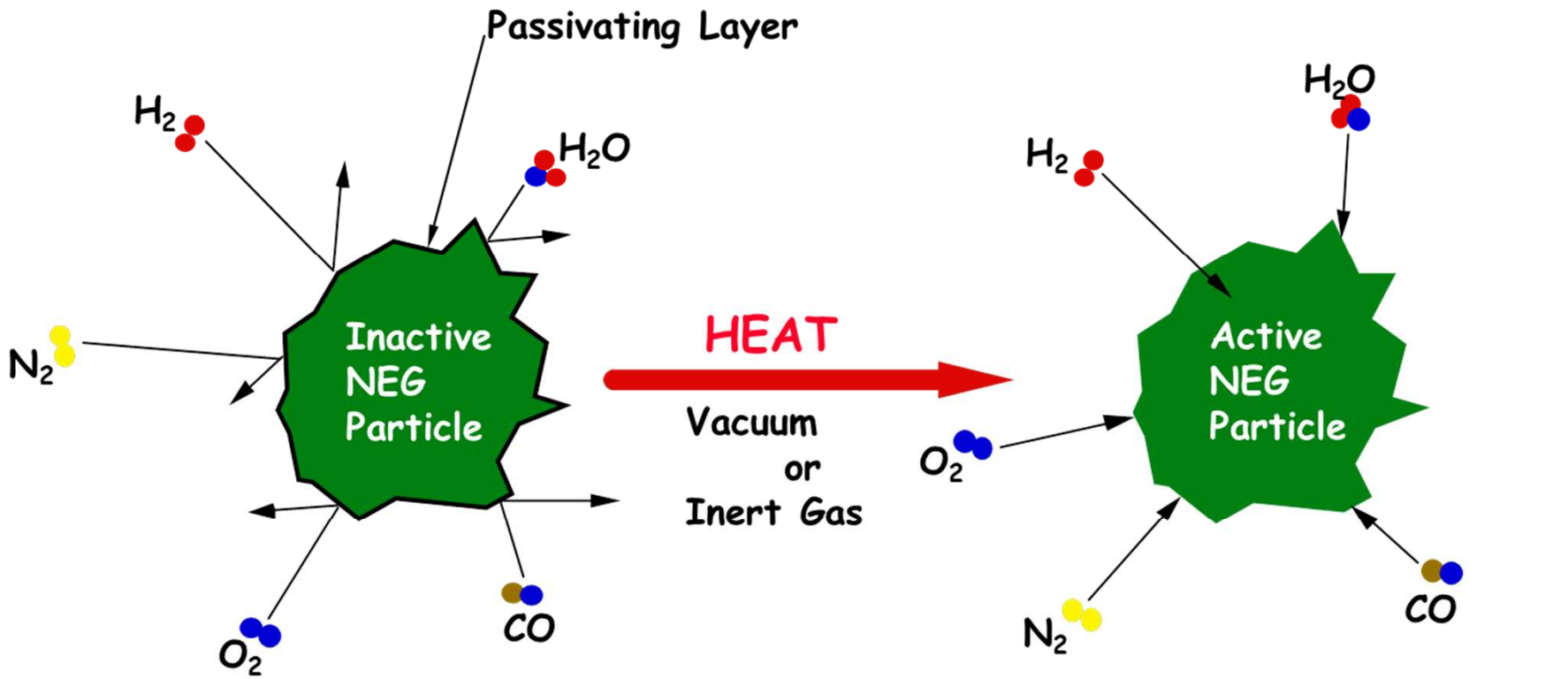
- NEGs do not sorb Ar, He, Kr, Xe.
- Ion pumps are required in combination with NEGs for pumping rare gases.

NEG Pumping Characteristics (5)

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



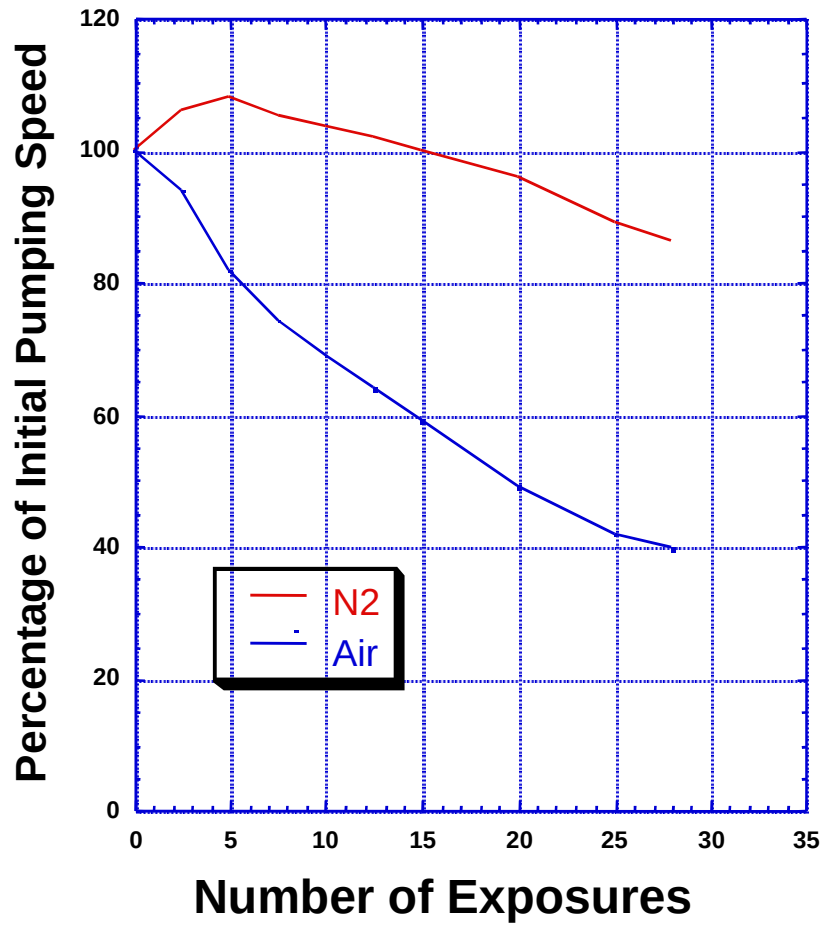
Activation Process for NEG



Ref. SAES Getters

Application Notes for NEGs

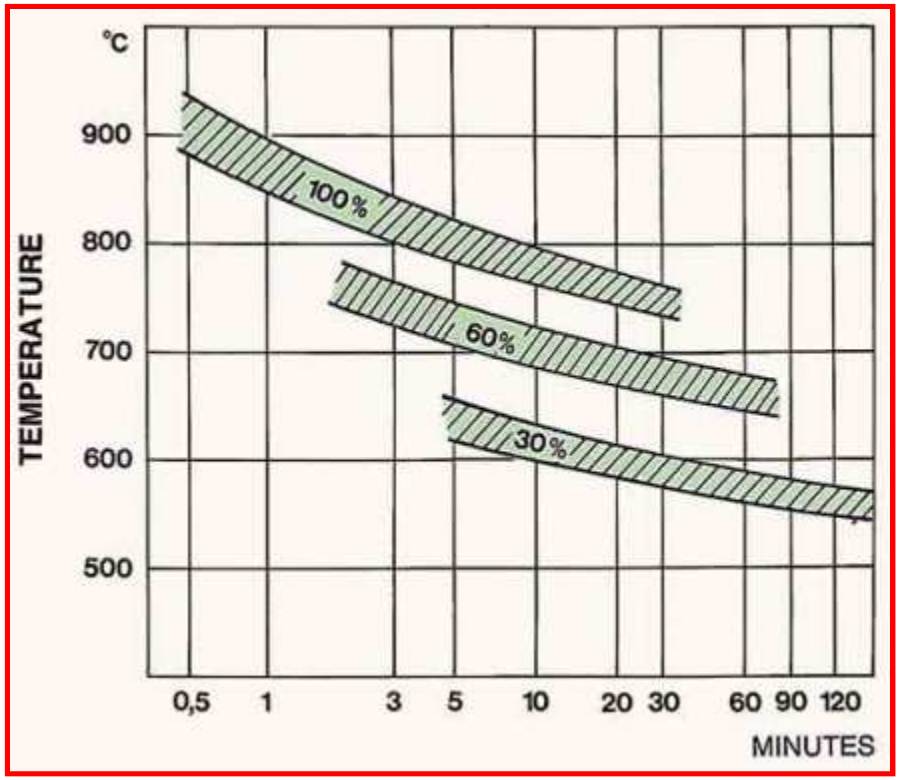
- *NEG performance deteriorates due to successive exposures to air (oxygen and water) or N_2.*
- *Further improvement can be obtained if Argon is used as a protective gas, during long term storage .*
- *NEG pumps should never be exposed to air while at temperatures higher than 50°C.*
- *Degassing of NEGs during initial pump-down.*



Ref. SAES Getters

SAES ST101® Non-evaporable Getters

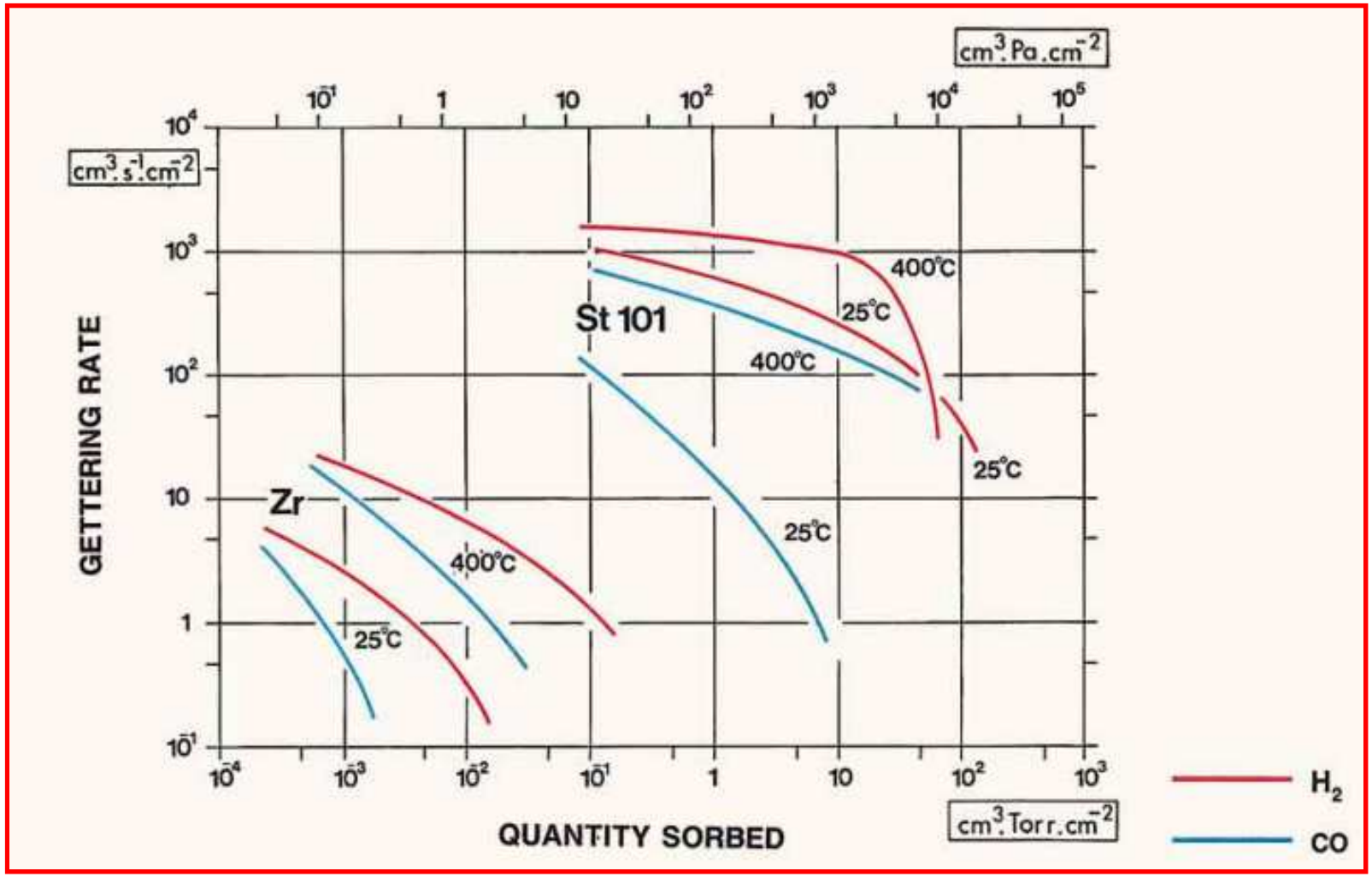
- ***Metal alloy made up of 84% Zr, 16% Al.***
- ***First Zirconium based getter alloy introduced and still widely used today after 30 years.***
- ***The operating temperature range of ST101 is 0 to 450° C.***
- ***ST101 chemisorbs CO, CO_2, H_2O, N_2, and O_2 at high rates.***
- ***ST101 activates at temperatures from 550 to 900° C.***



ST 101 Alloy Activation Efficiency

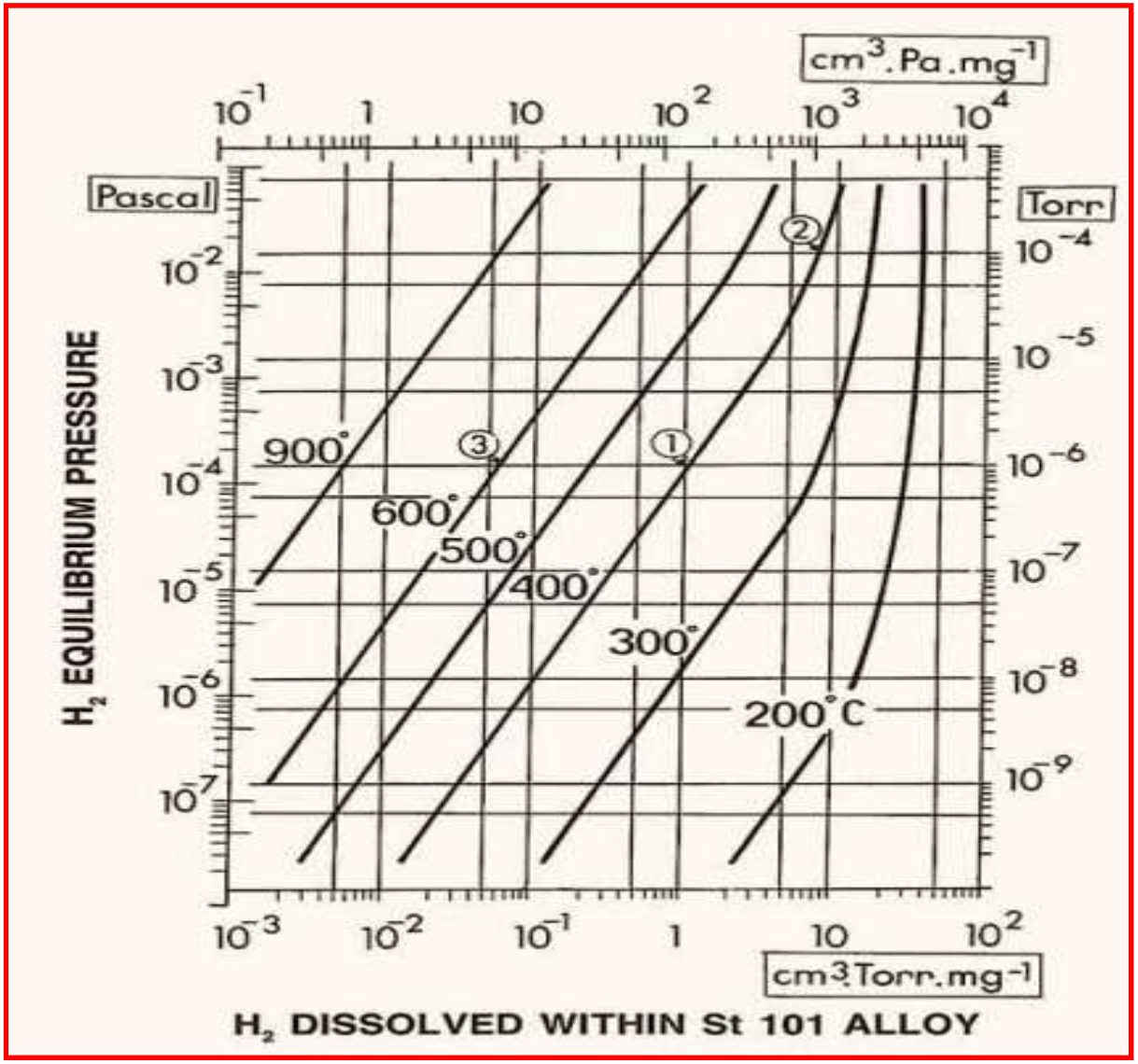
Ref. SAES Getters

SAES ST101® NEG – Pumping

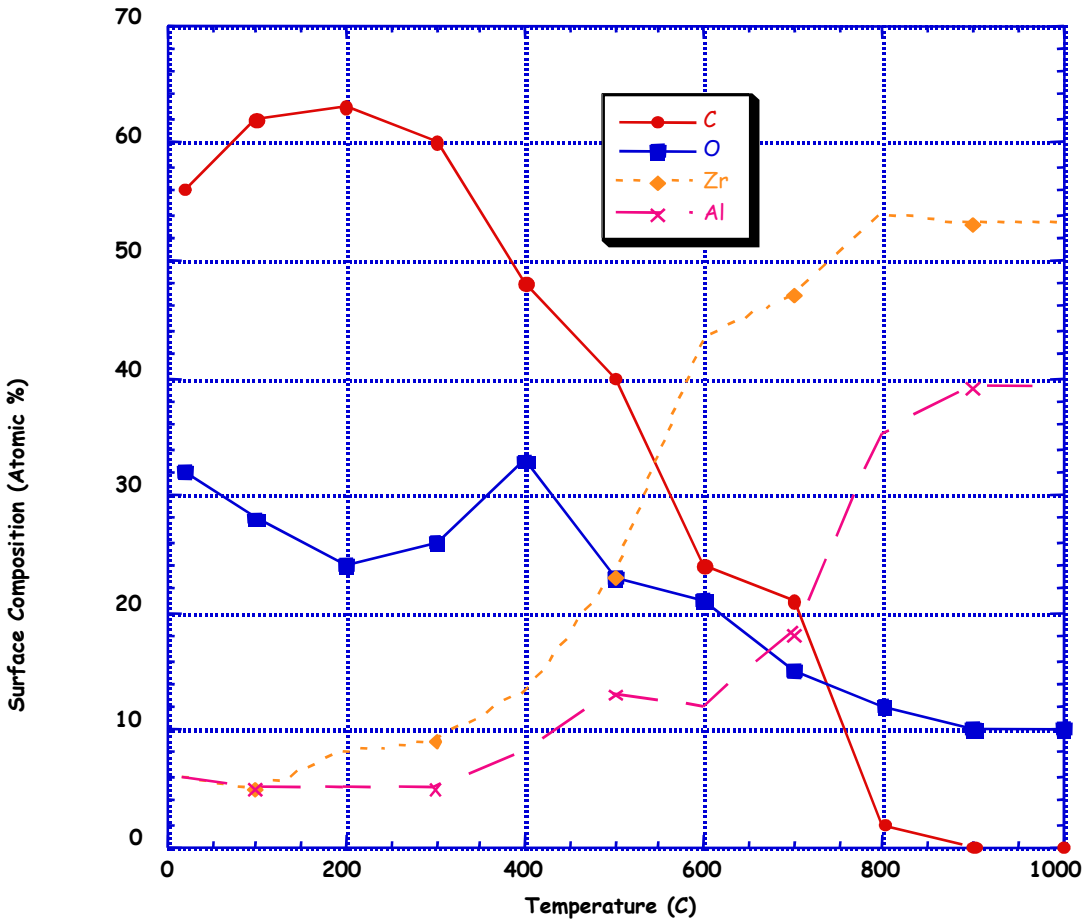


Ref. SAES Getters

SAES ST101® NEG – Hydrogen Solubility



ST101 Surface Composition vs. Temperature



Ref. SAES Getters

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, CO_2, H_2O, N_2, and O_2 at high rates.
- ST707 has much lower activation temperature.

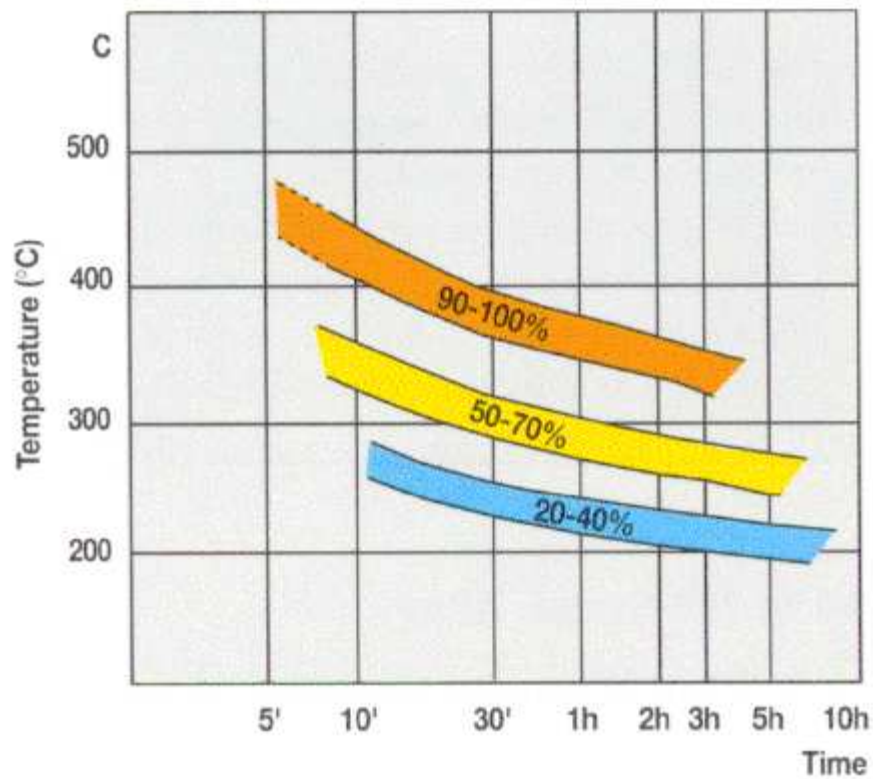
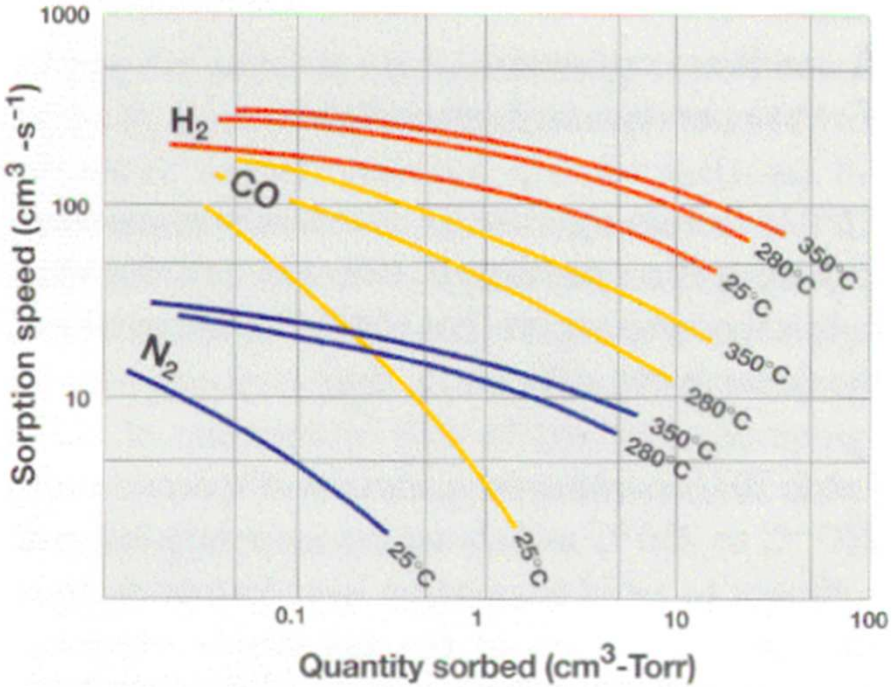


Fig. 1. Activation conditions and gettering efficiency of St 707

SAES ST707® NEG Pumping Performance

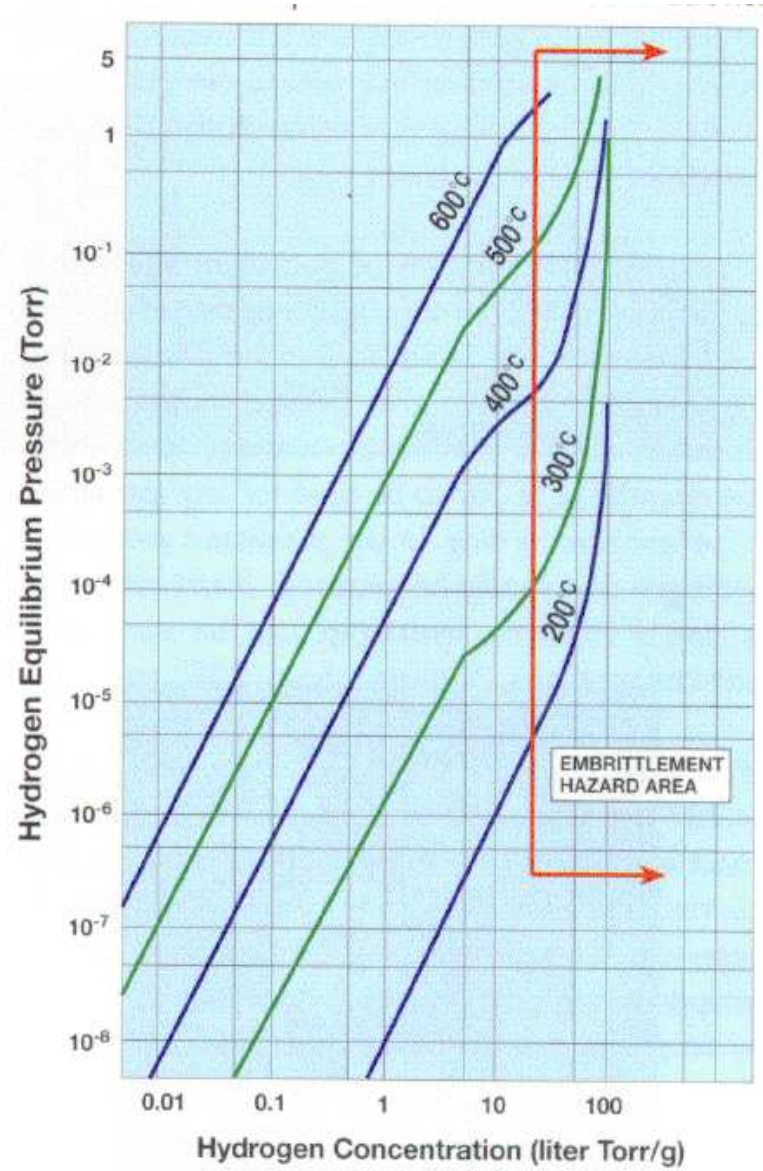


St 707 powder alloy: 100mg

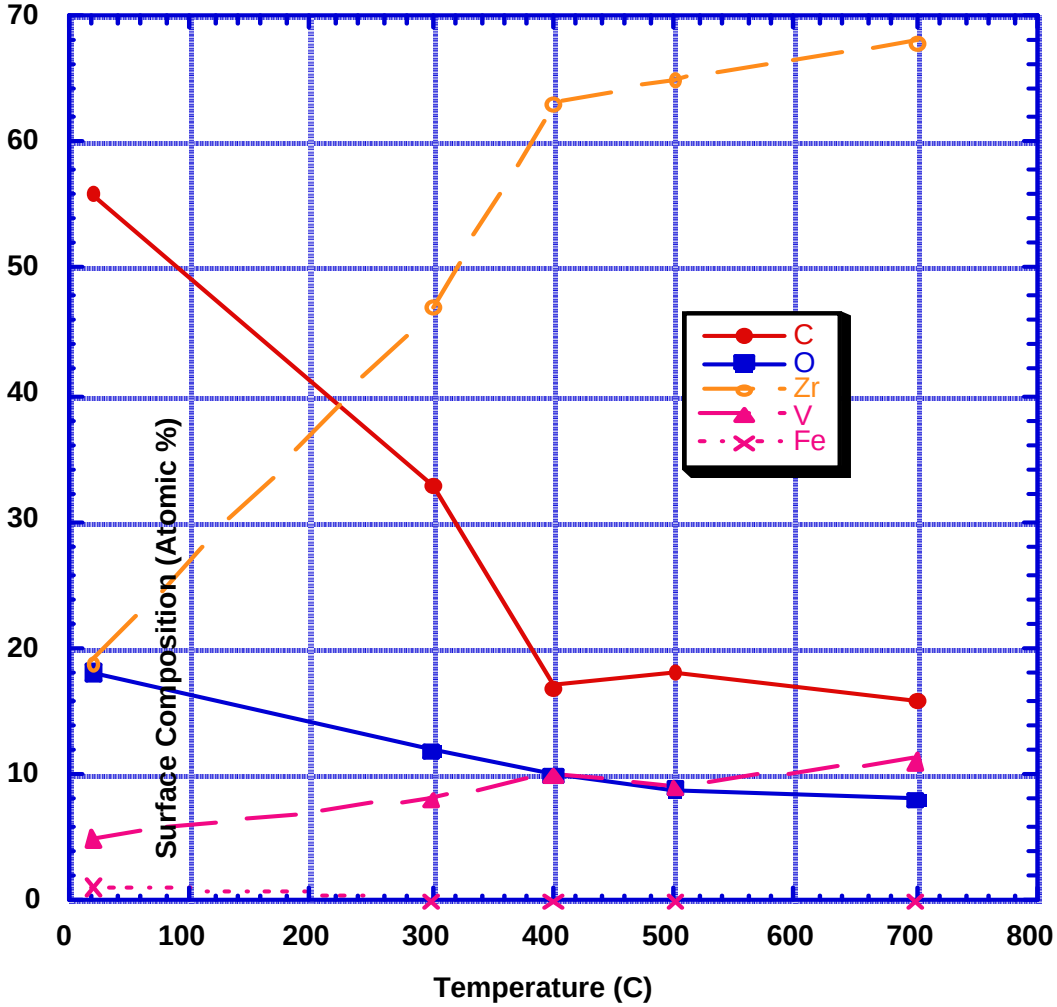
Geometric surface: 50 mm^2

Activation: 450°C for 10 min.

Sorption: At the indicated temperatures



ST707 Surface Composition vs. Temperature

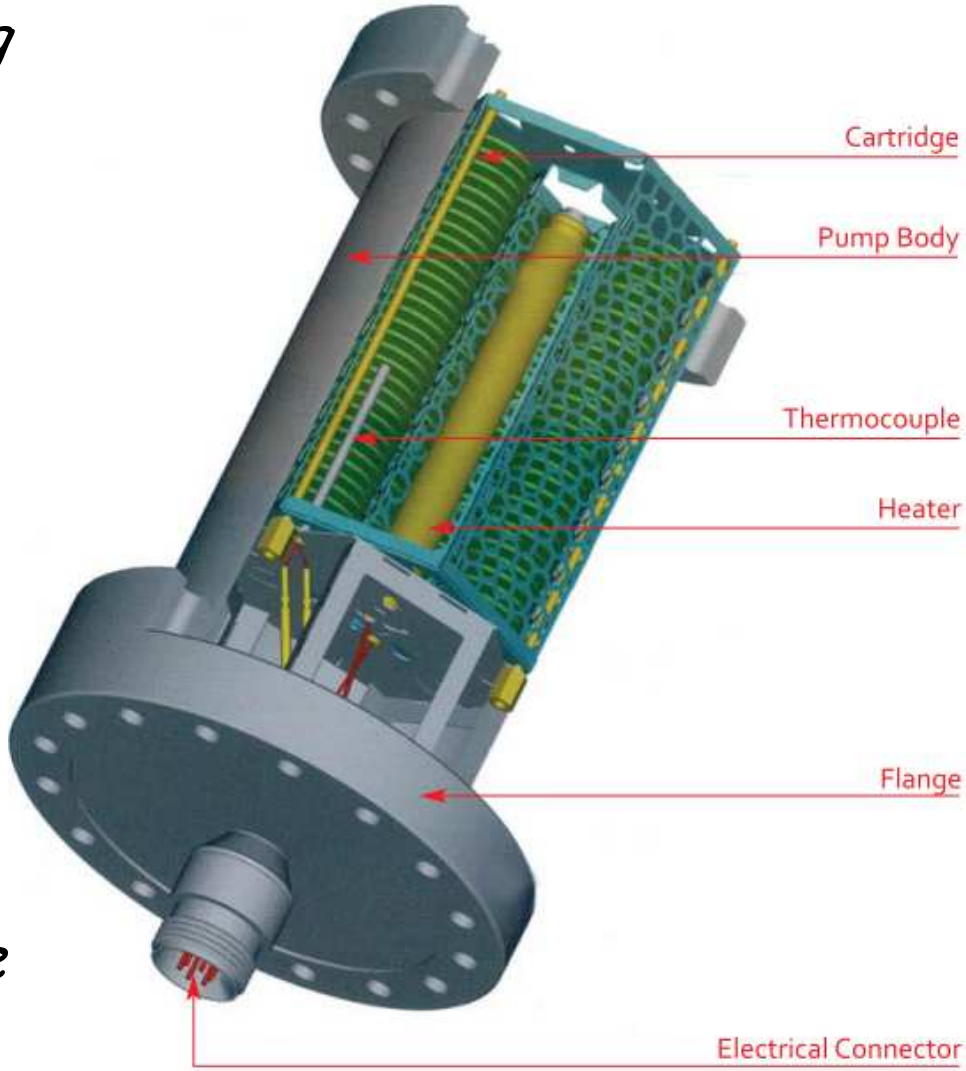


Other SAES NEG Alloys

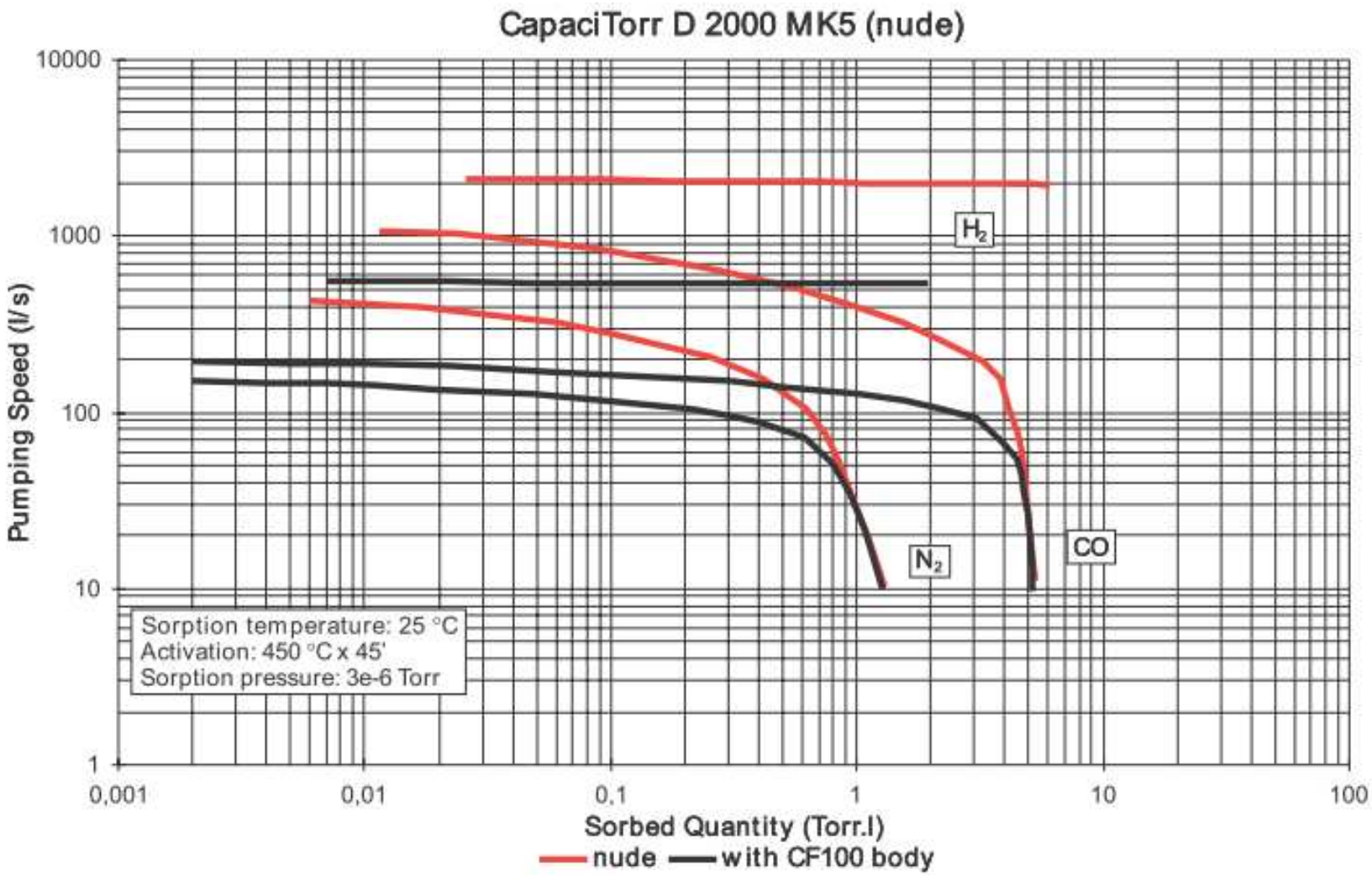
- *ST 172 – Zr, V, and Fe alloy.*
- *ST175 – Ti and Mo powder mixture, sintered form.*
- *ST185 – Ti-V alloy (obsolete !)*

NEG Cartridge Pump Module – CapaciTorr®

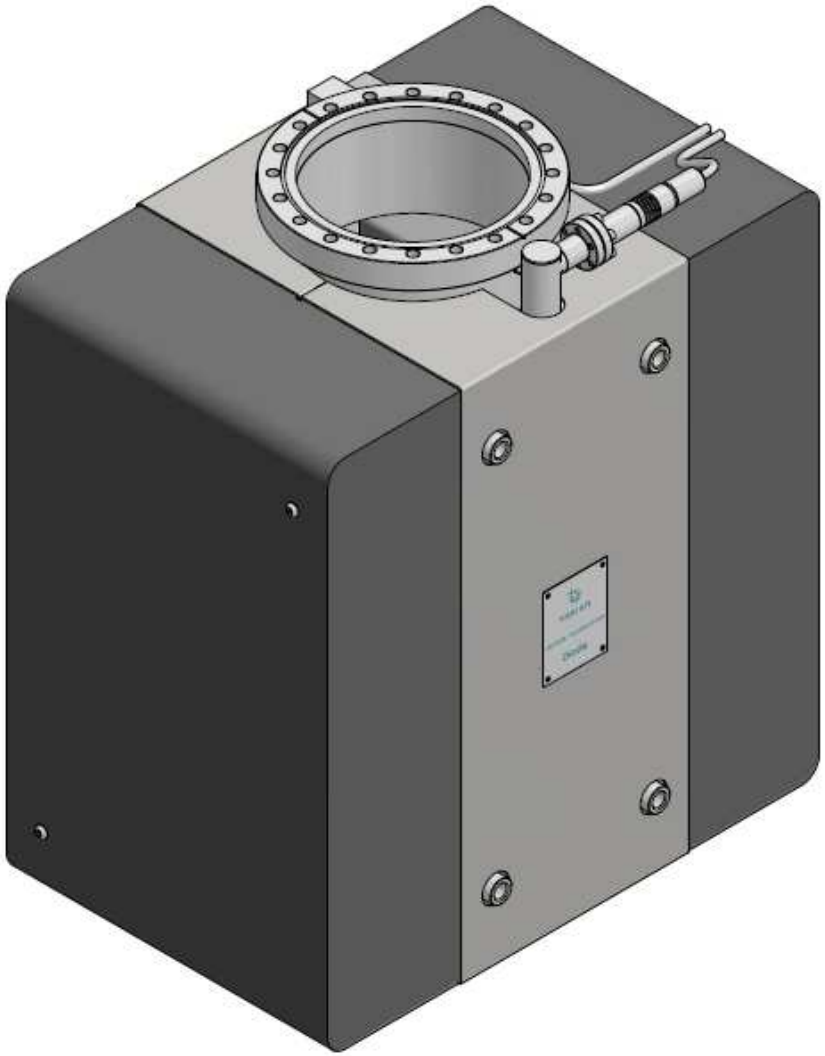
- *Complete compact pumping system, with matching controller for easy activation*
- *NEG materials: st172 blades/disks*
- *Pump sizes from 50 l/s to 2000 l/s, for H_2*
- *For large sizes, the NEG cartridges are replaceable*



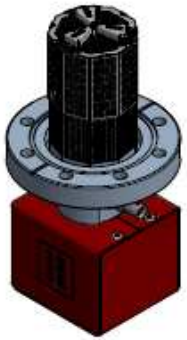
CapaciTorr® Pumping Performance



NEG – Ion Pump Combination – NexTorr®

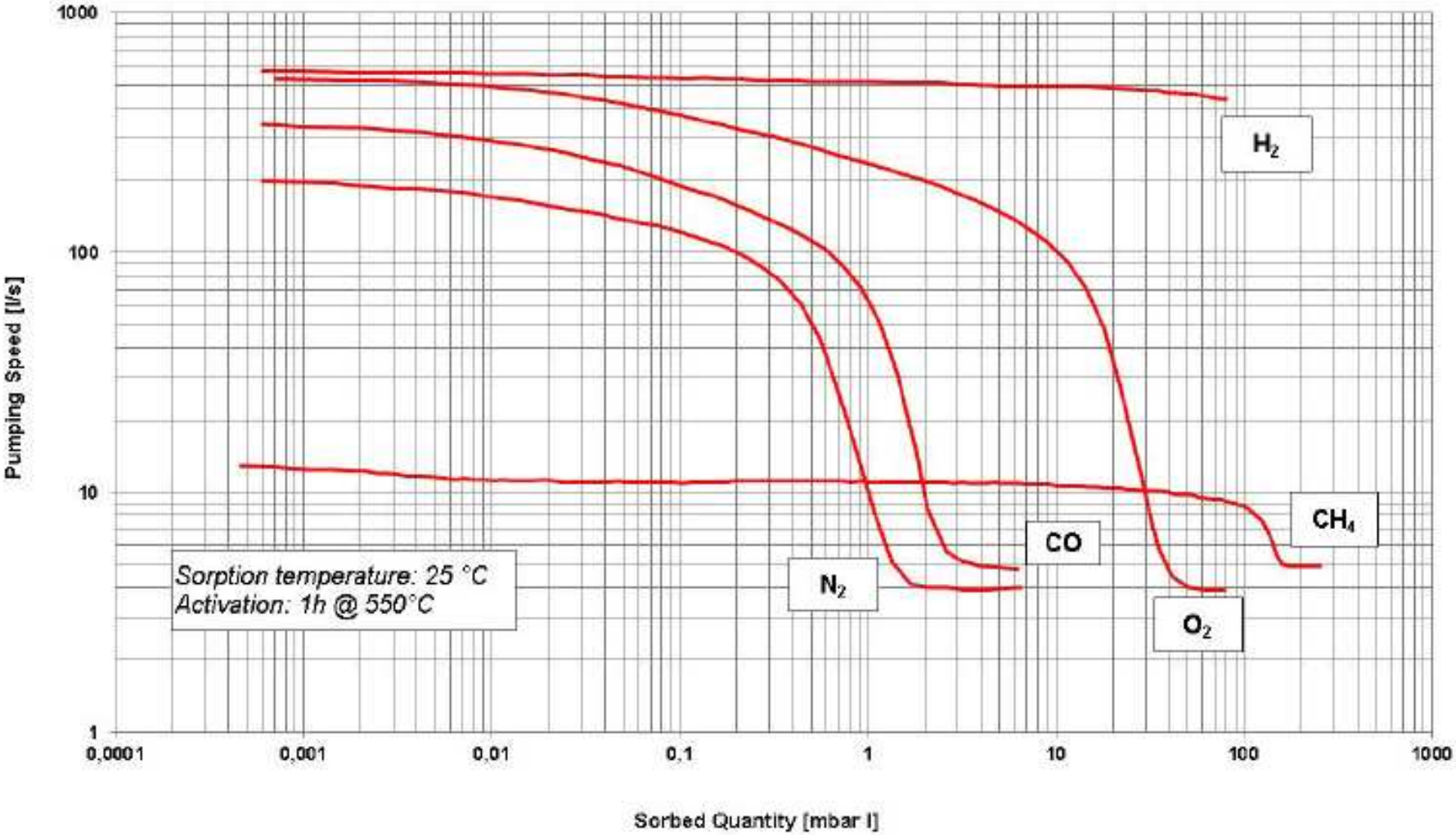


500 l/s VacIon Plus



NexTorr D500-5

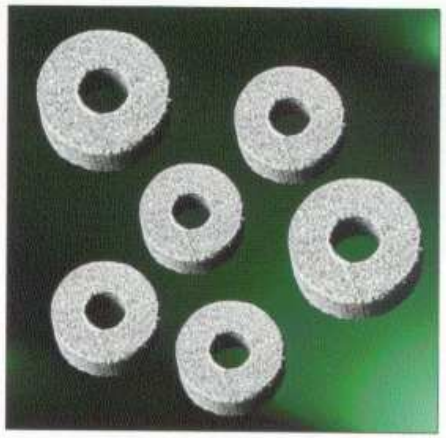
Pumping Performance – NexTorr®

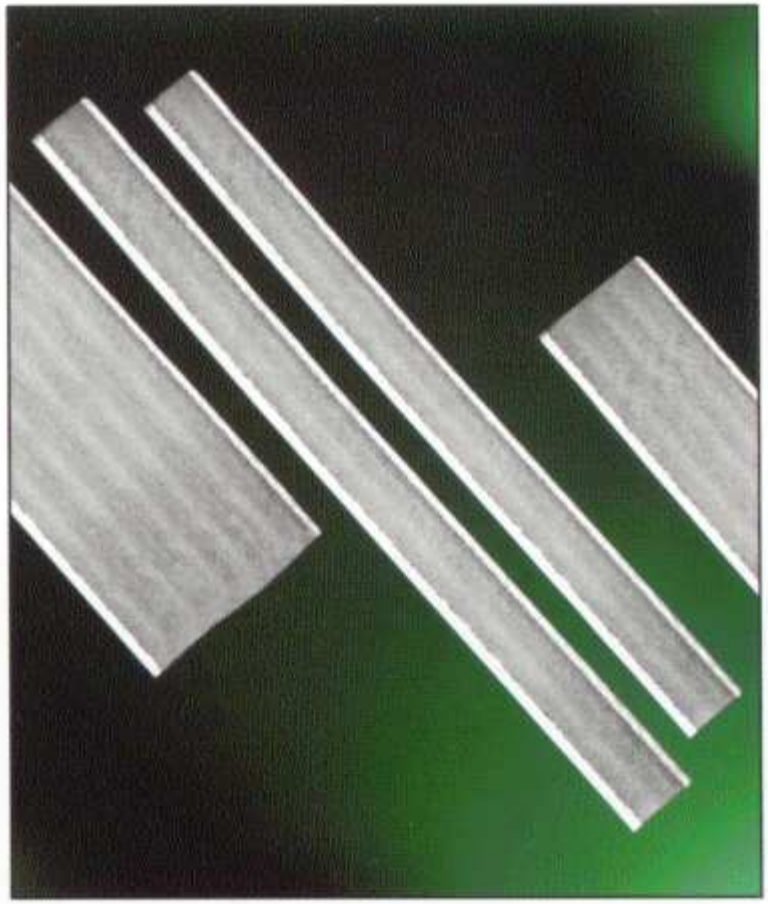


Main Technical Parameters – NexTorr® D500-5

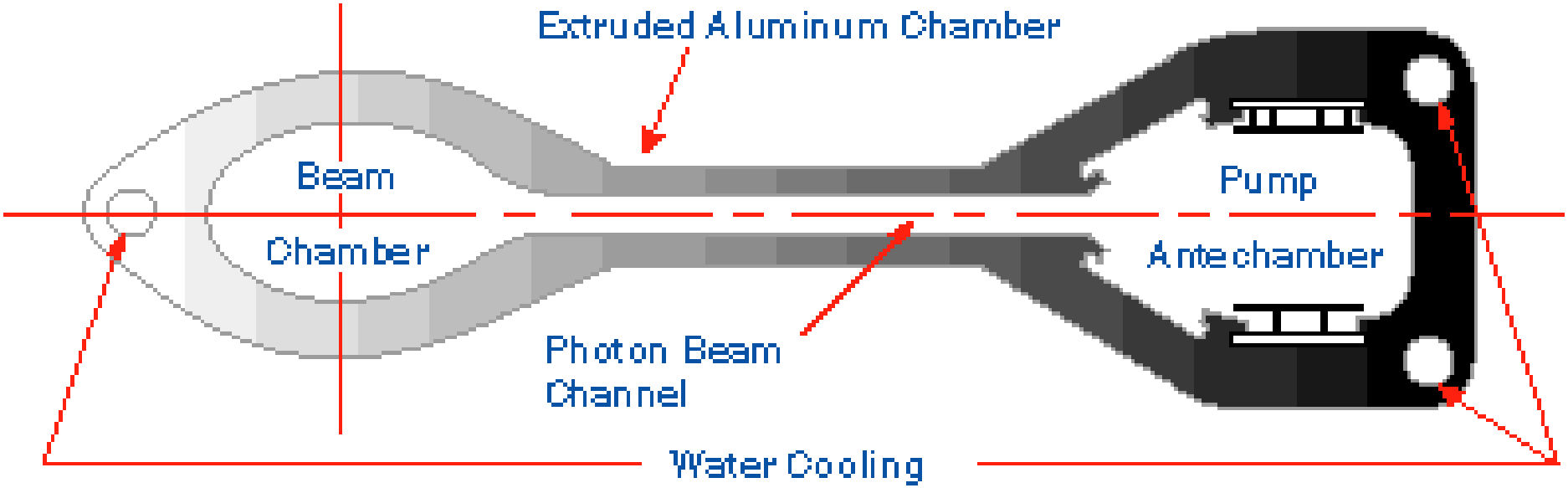
Initial pumping speed (l/s)	Gas	NEG activated	NEG saturated
	O_2	500	4
	H_2	500	6
	CO	340	5
	N_2	200	4
	CH_4	13	5
	Argon[1]	6	6
Sorption capacity (Torr·l)	Gas	Single run capacity [2]	Total capacity [3]
	O_2	17	>1500
	H_2	670	N/A [4]
	CO	1.4	>360
	N_2	0.8	>75
	CH_4	137	50,000 hours at 10^{-6} Torr
NEG section	Getter alloy type		St 172
	Alloy composition		ZrVFe
	Getter mass (g)		68 g
	Getter surface (cm^2)		570
ION section	Voltage applied		DC +5kV
	Number of Penning cells		4
	Standard bake-out temperature		150°C

Other NEGs forms – Build your own pumps



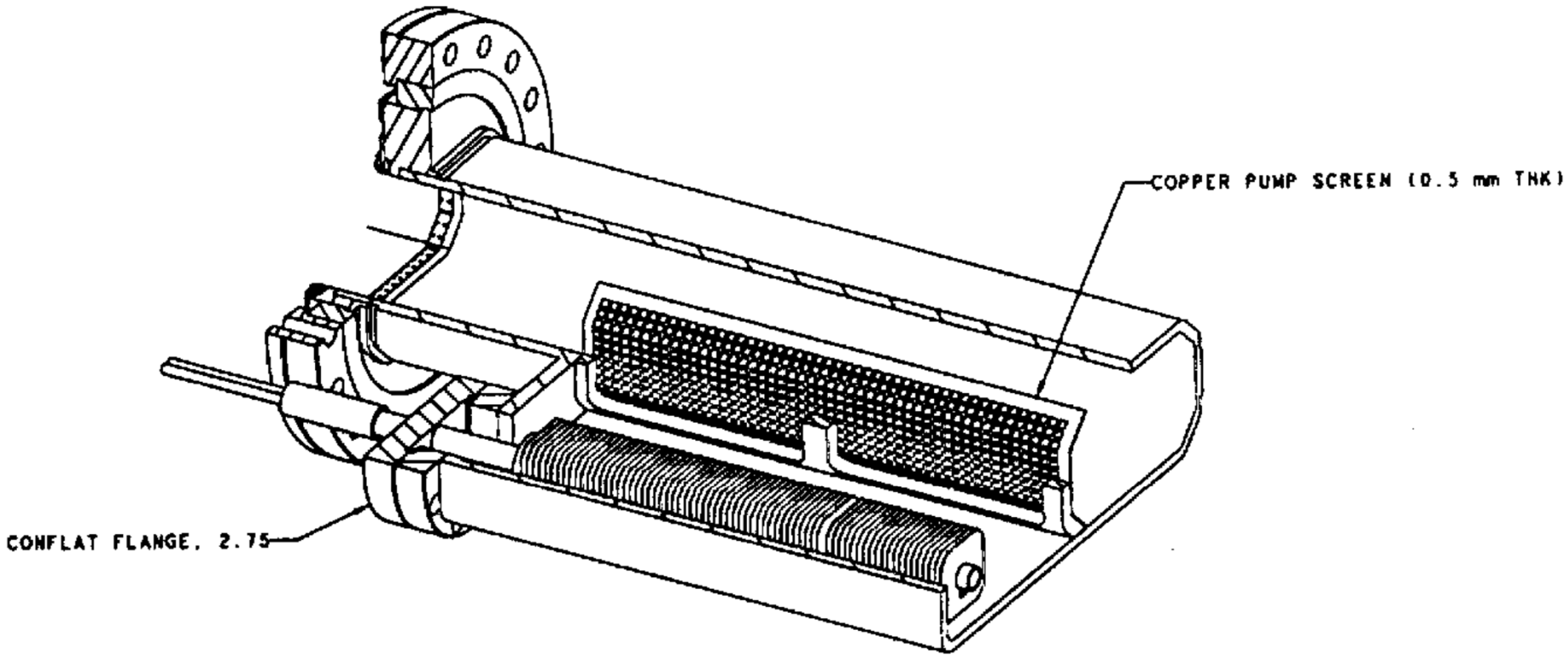


Distributed Pumping with NEG strips



APS Beampipe with NEG strips

LLNL NEG Pump in a PEP-II Vacuum Chamber



CUTAWAY ISOMETRIC
REFERENCE ONLY

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.

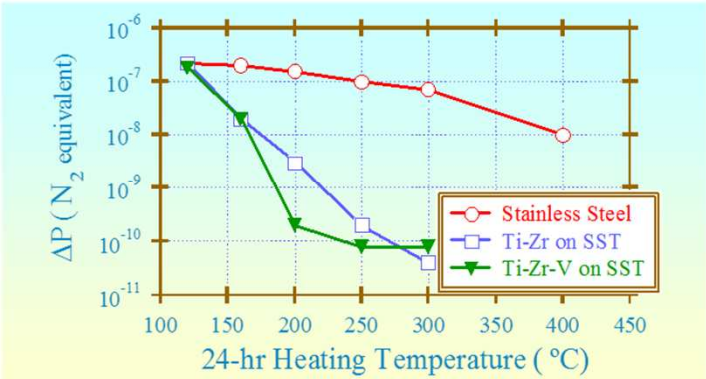
Commercial Combination Pumps . . .
Ion Pumps with TSP or NEG

Ion Pump with TSP filaments

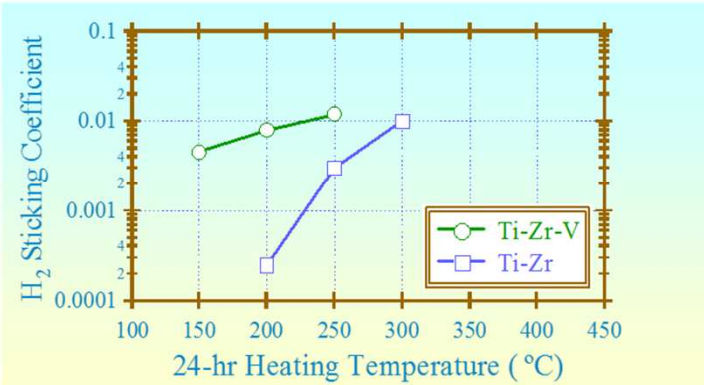
Ion Pump with NEG cartrdge

NEG Thin Film for Accelerators

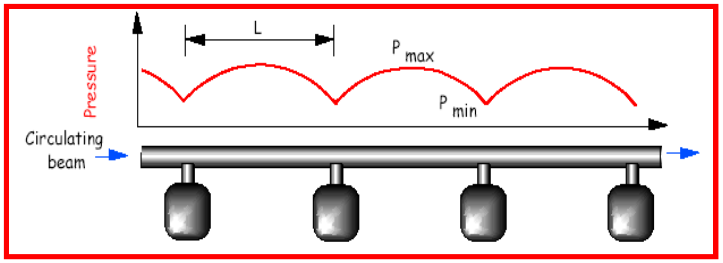
- Developed at CERN, by *Bevenuti, et al*



Low Outgassing Rates



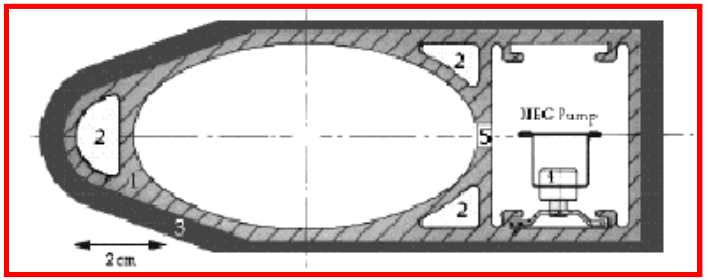
LOCAL pumping



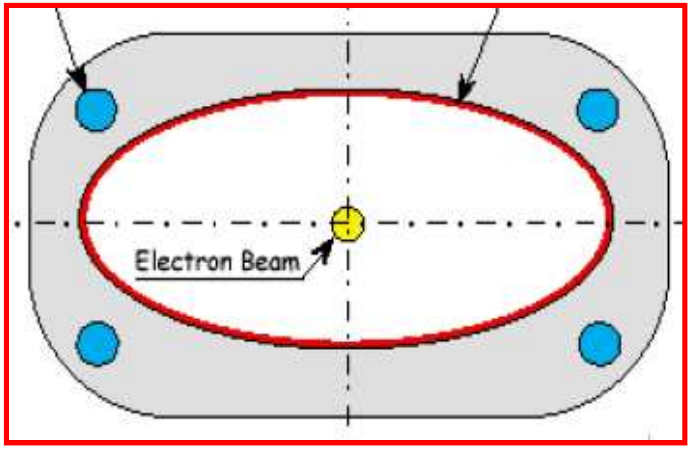
Discrete Pumping

Integrated Pumping

NEG Coating

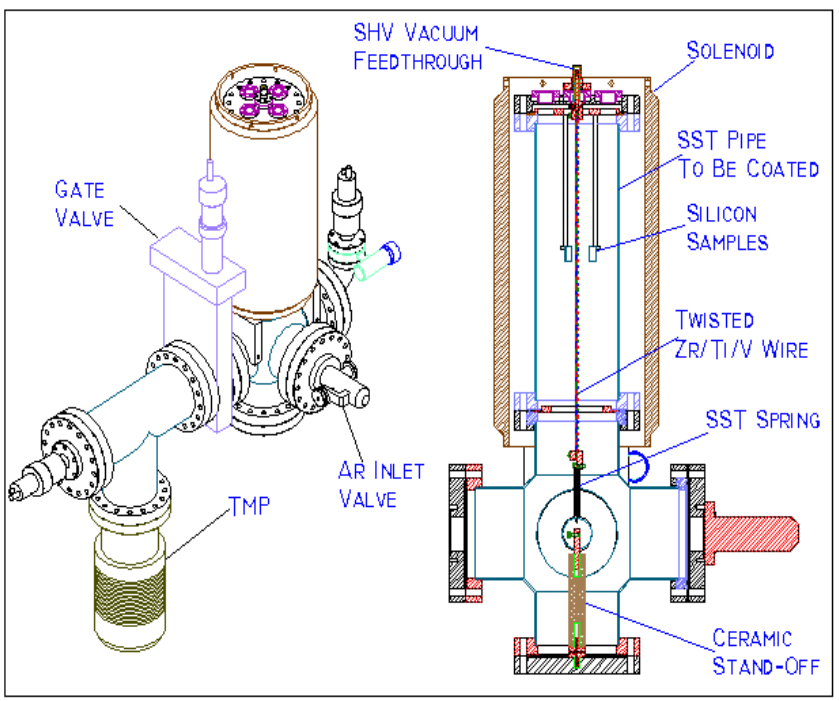


Distributed Pumping



Deposition of NEG Thin Films

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 ~ 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 $Zr_xV_yTi_z$, with x, y, z, close to unity.

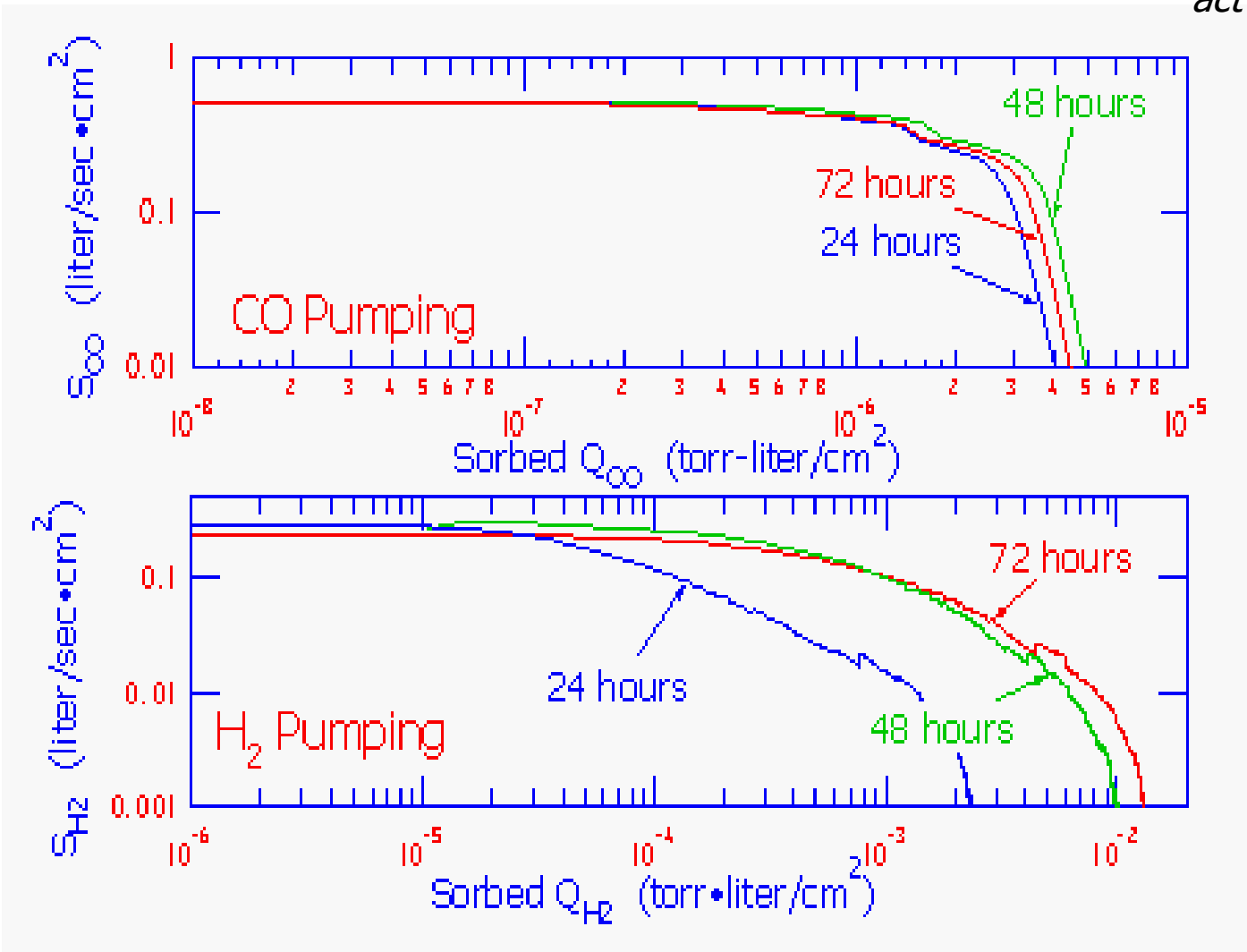
- Stoichiometric balanced thin film tend to have lower activation temperature, probably due to smaller grain sizes.

- 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)

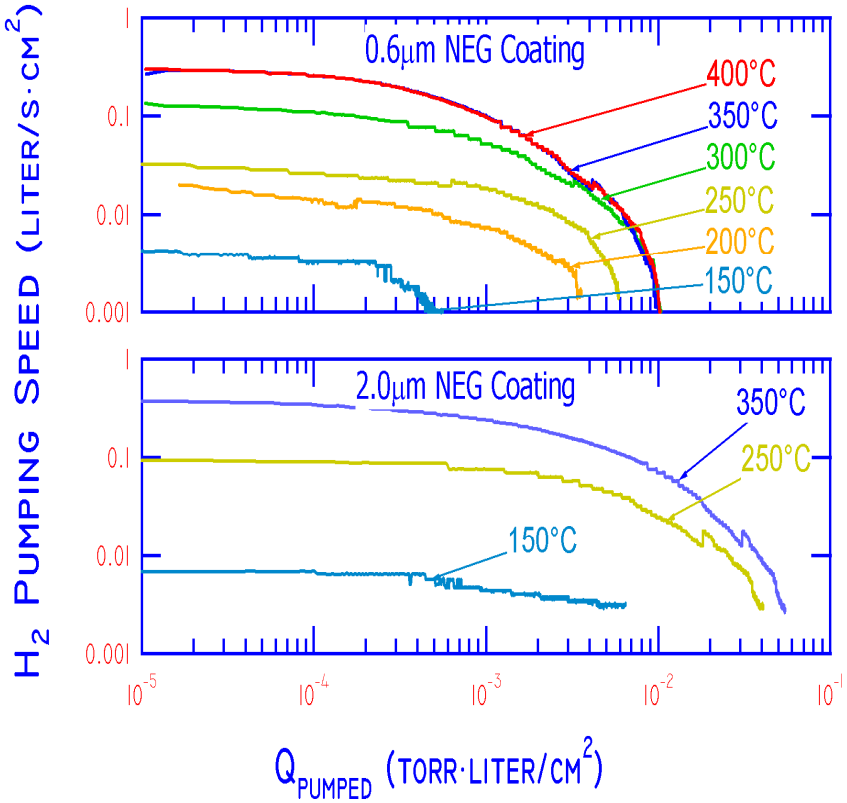
T_{act}=350°C

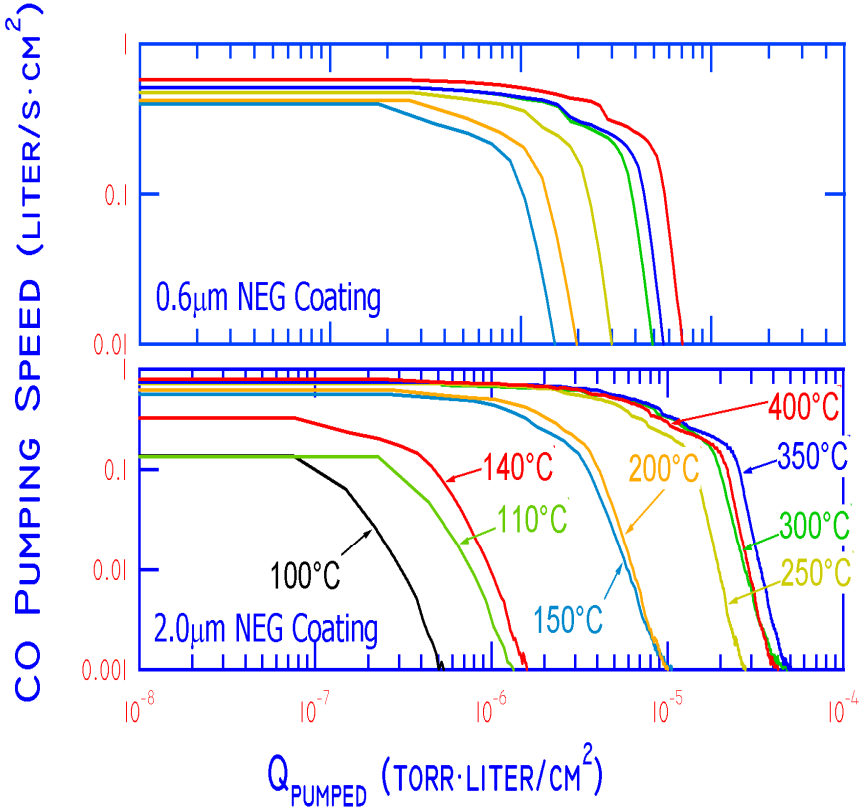


NEG Coating Pumping Performance (2)

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

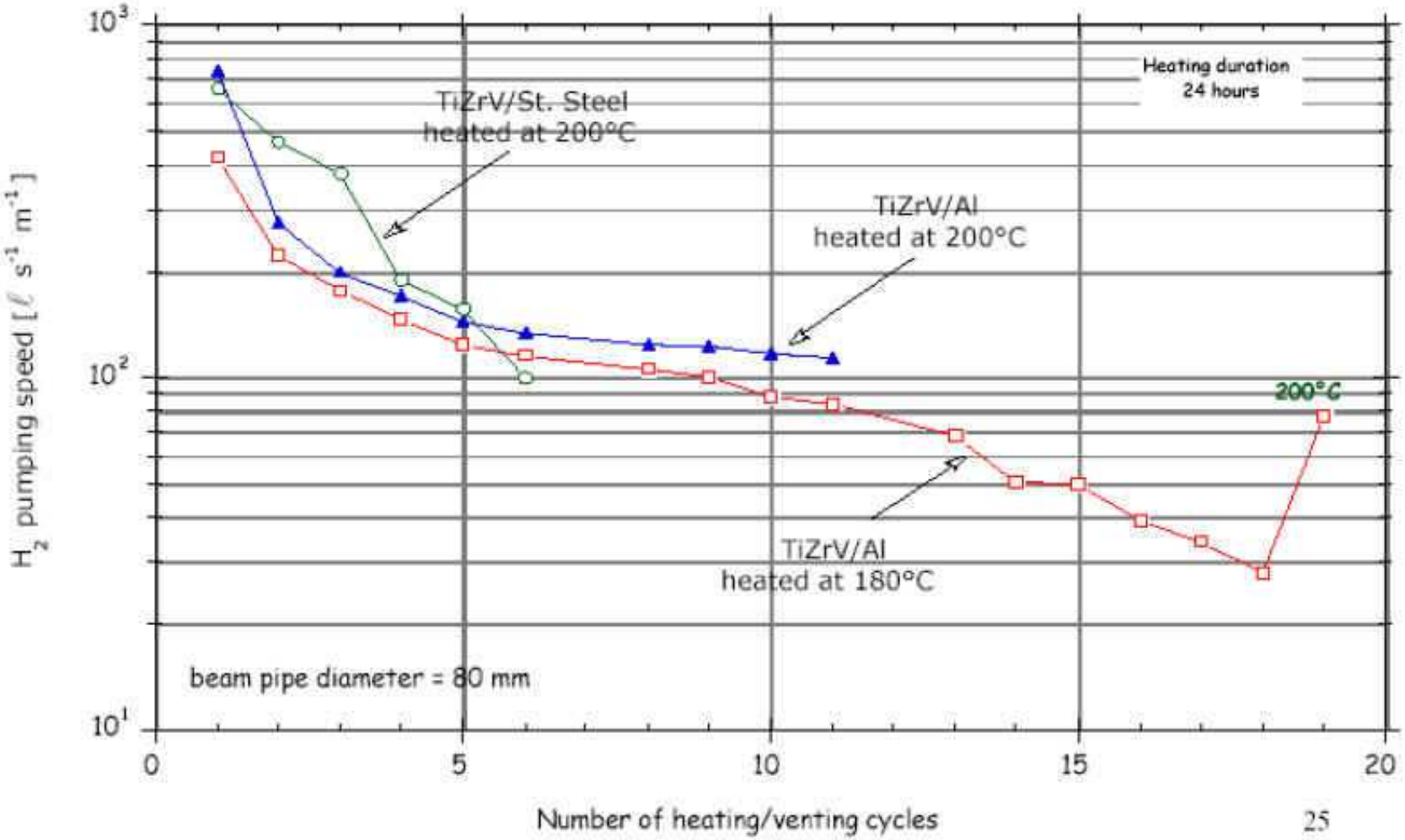
  Using solubility of 5%, 1-nm saturated surface oxide layer
  Estimated saturation/venting cycles for 1 μm NEG film > **50**

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

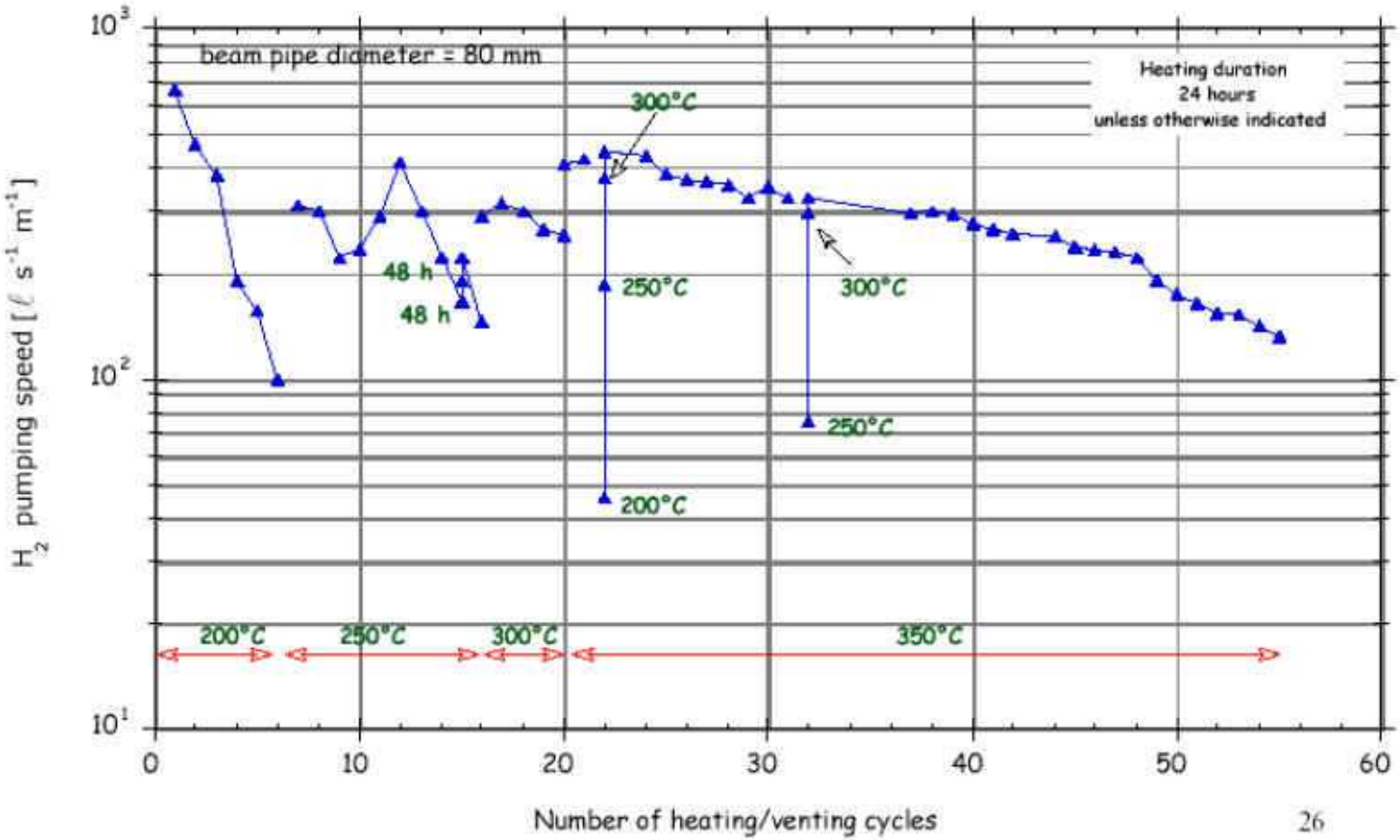
  → Reduction of pumping speed and capacity

  → Increase of activation temperature

NEG Film Aging Effect



NEG Film Aging – More

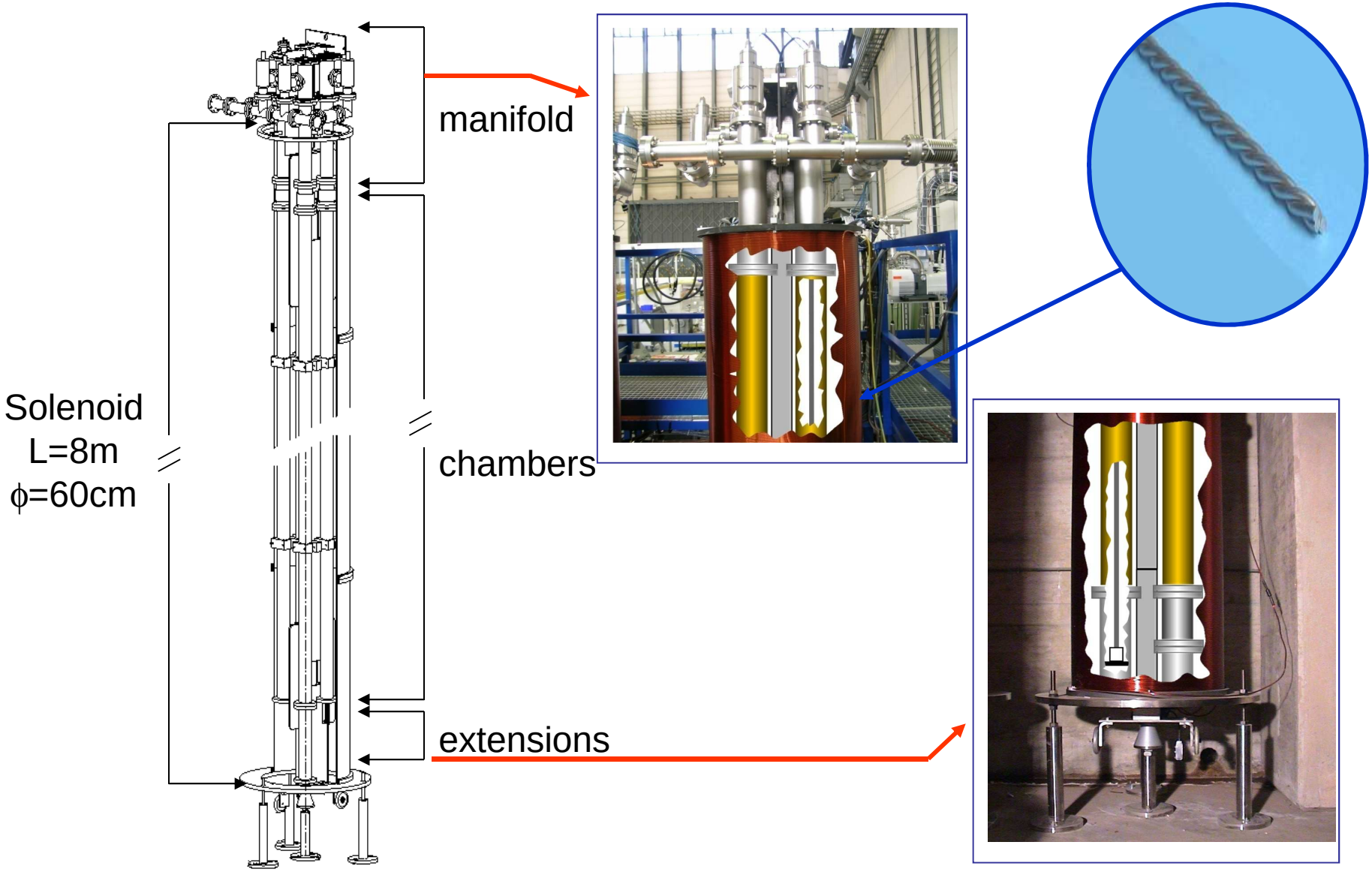


Successful Applications of NEG Coatings

- 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

CERN's NEG Coating Facility – Details



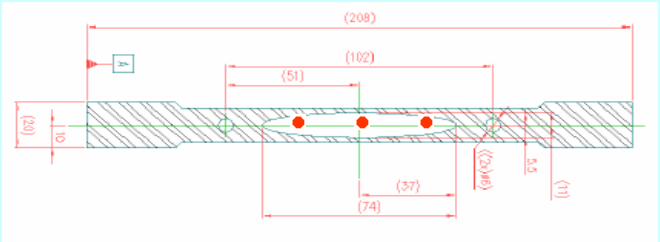
CERN's NEG Coating Production

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.*

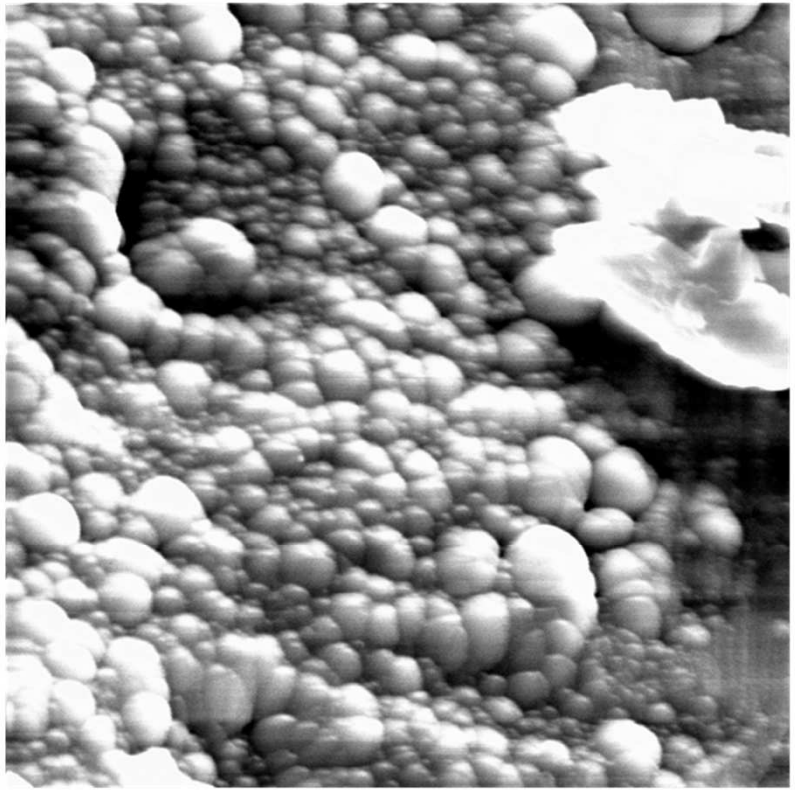
One of the SAES sputtering systems for NEG coating, capable to coat up to 6.5 meter long chambers with a 2m height coil.

Word of Caution

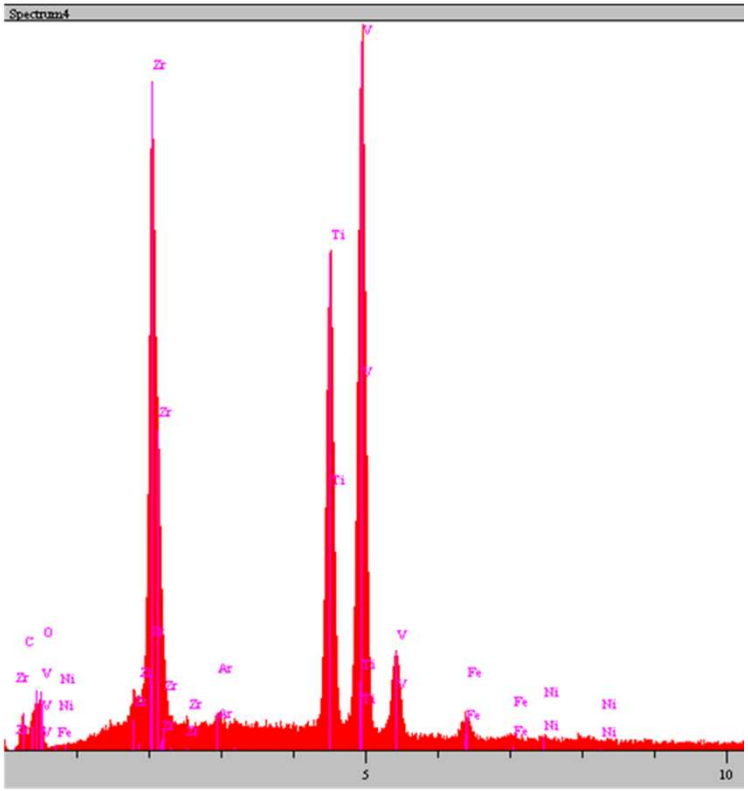
Powder substance were found on the orifice disk, as well as on the coated surface, after extensive pumping tests

- *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 Confirmed to Be NEG



Powder SEM Image



Powder EDX Spectrum

NEGs or TiSPs

- *Both TiSPs and NEGs are great in deal 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 NEGs*
  - → How to reduce hydrogen from NEGs ?
  - → Should the NEGs 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 NEGs for air exposure ?