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

## SESSION 4.3: Getters

- Getters pump gases by chemically bonding molecules to surfaces 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 used in accelerator vacuum systems

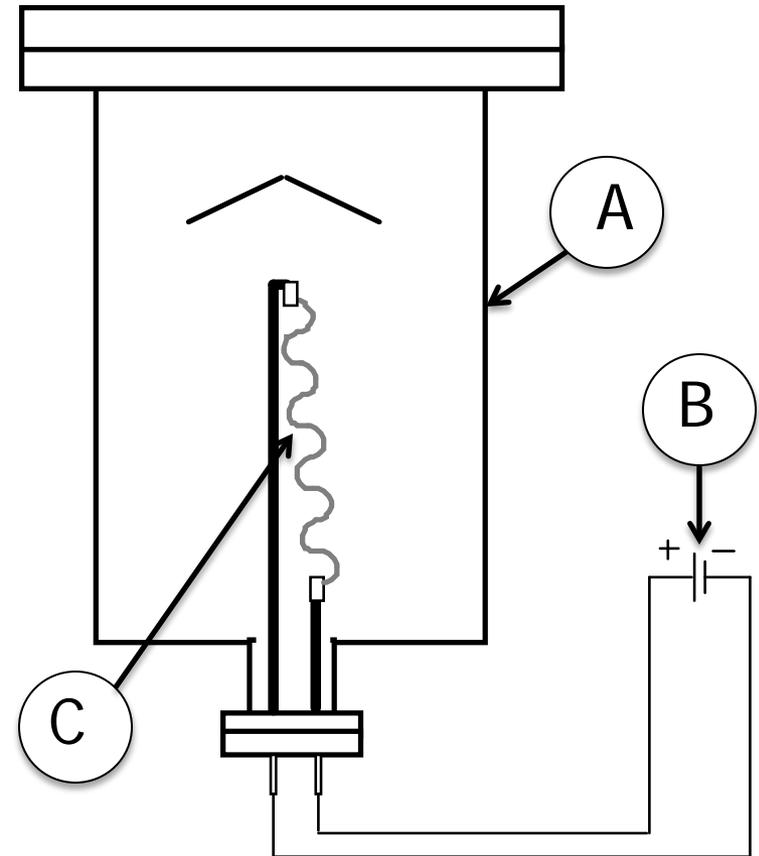


# Session 4.3A

## Titanium Sublimation Pumps



- *A TiSP simply consists of three basic elements:*
  - *A source from which titanium is sublimed (C)*
  - *A power supply to heat the source (B)*
  - *A surface onto which the titanium is sublimed, and is accessible to the arriving active gas. (A)*
- *Thus no manufacturer sells a TiSP 'pump', only the Ti sources (and a power supply)*



*Example of a TiSP System*

# Titanium Sources – Filamentary Types



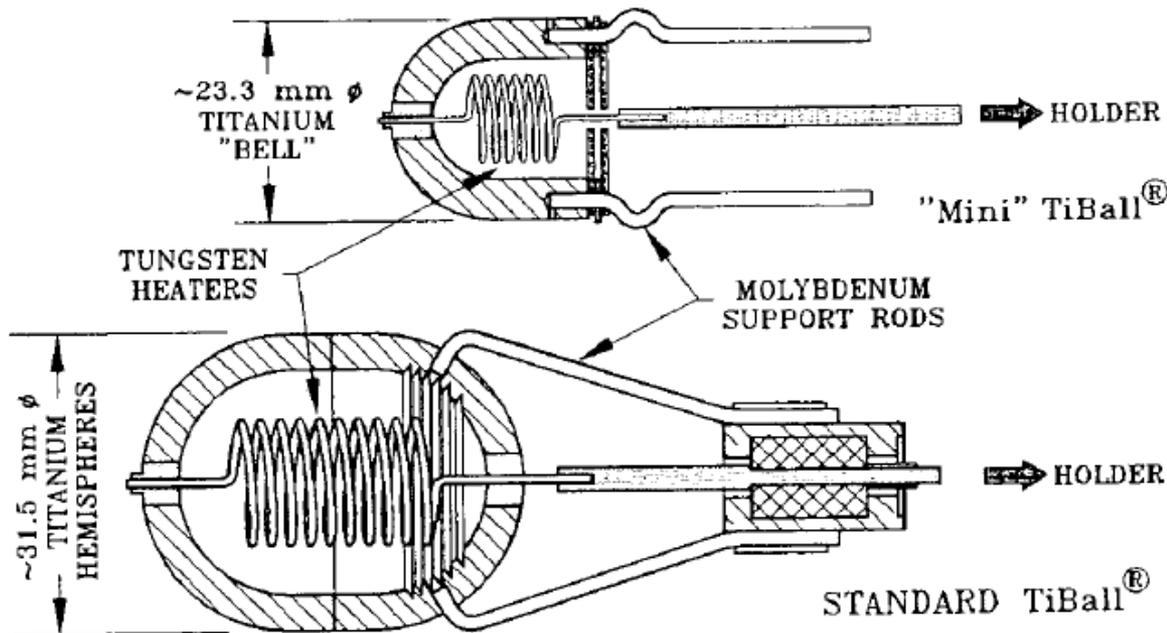
- ❑ *Filaments made of 85%Ti-15%Mo are most common sources of titanium used in TiSPs.*
- ❑ *The filament is resistively heated during sublimation process.*
- ❑ *In most cartridges, multiple filaments are loaded.*



# Titanium Sources – Radiated Heating



More suitable for very high throughput pumping application.



*Varian Vacuum*

Ref. "A Radiant Heated Titanium Sublimator," Proc. 5th Int. Vacuum Congress, 1971, JVST 9 (1), 552 (1972)

- Sources require operation at some level of standby power to maintain Titanium temperature above 900°C.
- Very inefficient heating, and require relatively high heating power.



# TiSPs – Pumping Speed



*A TiSP is simply a surface conductance limited pump. The pumping speed depends on unit surface conductance ( $C_i$ ) to the Ti-covered surfaces ( $A$ ) and gas sticking coefficient.*

$$S_i (m^3/s) = \alpha_i C_i A = 36.24 \sqrt{\frac{T}{M}} \cdot \alpha_i A (m^2)$$

*Or*

$$S_i (L/s) = 3.624 \sqrt{\frac{T}{M}} \cdot \alpha_i A (cm^2)$$

*T - Temperature of gases (Kevin)*

*M - Gas molar mass*

*A - Ti covered area (in  $m^2$  or  $cm^2$ )*

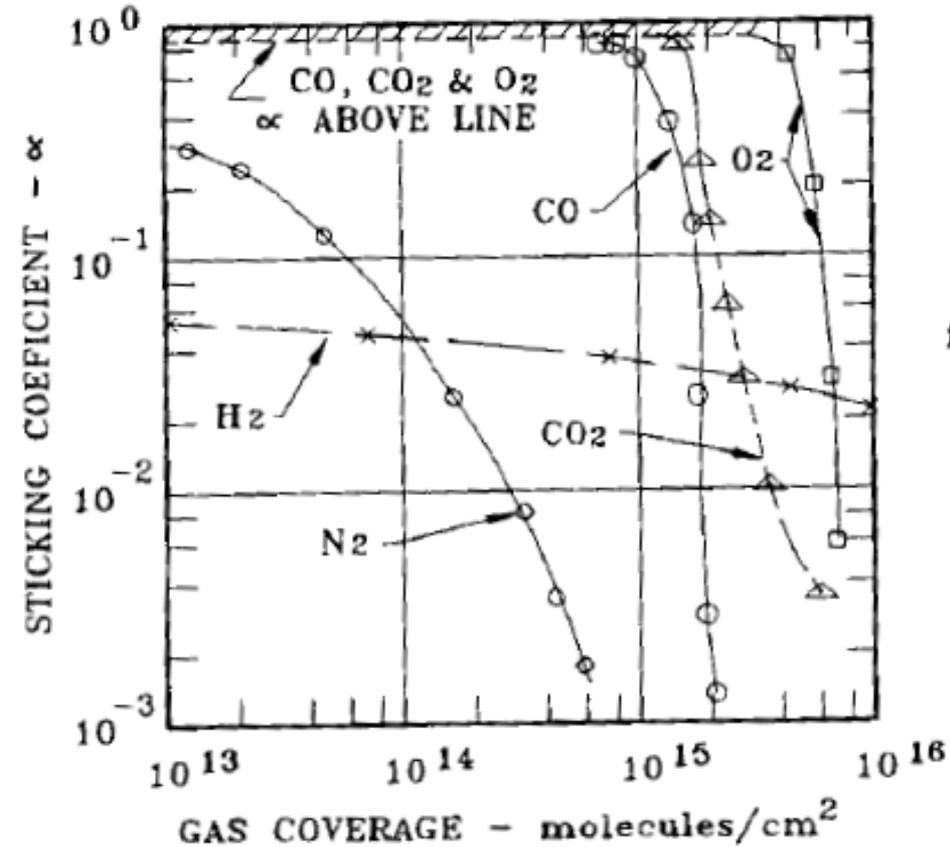
*$\alpha_i$  - sticking coefficient for "i" gas molecules*



# Sticking Coefficient



- ✓ Sticking coefficient is strongly gas reactivity dependent
- ✓ For most active gases, the sticking coefficient decrease with adsorbed quantity, with various behavior, due to surface deactivation.
- ✓ Ti film has very high capacity for hydrogen, indicating 'bulk' diffusion for dissociated H atoms.



Ti layer:  $\sim 10^{16}$  atoms/cm<sup>2</sup>





- *Thickness of Titanium film*
- *Ratio of pumping speed to Titanium sublimation rate*
- *Surface temperature at the time of sublimation*
- *Surface temperature at the time of gas sorption*
- *Film deposition process (batch or continuous)*
- *Gas species*
- *Gas desorption and synthesis at Titanium source*
- *Partial pressures of gases at time of sublimation*
- *Contamination of film by some gas*
- *Effects of film annealing*
- *Variations of surface and bulk diffusion processes*



# Pumped or/and Displaced Gases

Pumped Gas	Displaced Gas				
	CH <sub>4</sub>	N <sub>2</sub>	H <sub>2</sub>	CO	O <sub>2</sub>
CH <sub>4</sub>		no	no	no	no
N <sub>2</sub>	yes		no	no	no
H <sub>2</sub>	yes	yes		no	no
CO	yes	yes	yes		no
O <sub>2</sub>	yes	yes	yes	yes	
$\alpha_m$	$< 10^{-3}$	0.3	0.05	0.85	0.95

This is controversial and probably only true for CH<sub>4</sub> and H<sub>2</sub>.

# Typical Engineering Values for TiSP



Test Gas	Max. Sticking Coefficient- $\alpha_m$		Max. Speed <sup>a</sup> (liters/sec-cm <sup>2</sup> )		Max. Capacity of Film- $\times 10^{15}$ (molecules/cm <sup>2</sup> ) <sup>b</sup>	
	300 K	77 K	300 K	77 K	300 K	77 K
H <sub>2</sub>	0.06	0.4	2.6	17	8-230 <sup>c</sup>	7-70
D <sub>2</sub>	0.1	0.2	3.1	6.2	6-11	-
H <sub>2</sub> O	0.5	-	7.3	14.6	30	-
CO	0.7	0.95	8.2	11	5-23	50-160
N <sub>2</sub>	0.3	0.7	3.5	8.2	0.3-12	3-60
O <sub>2</sub>	0.8	1.0	8.7	11	24	-
CO <sub>2</sub>	0.5	-	4.7	9.3	4-12	-

a) Speed calculated at RT

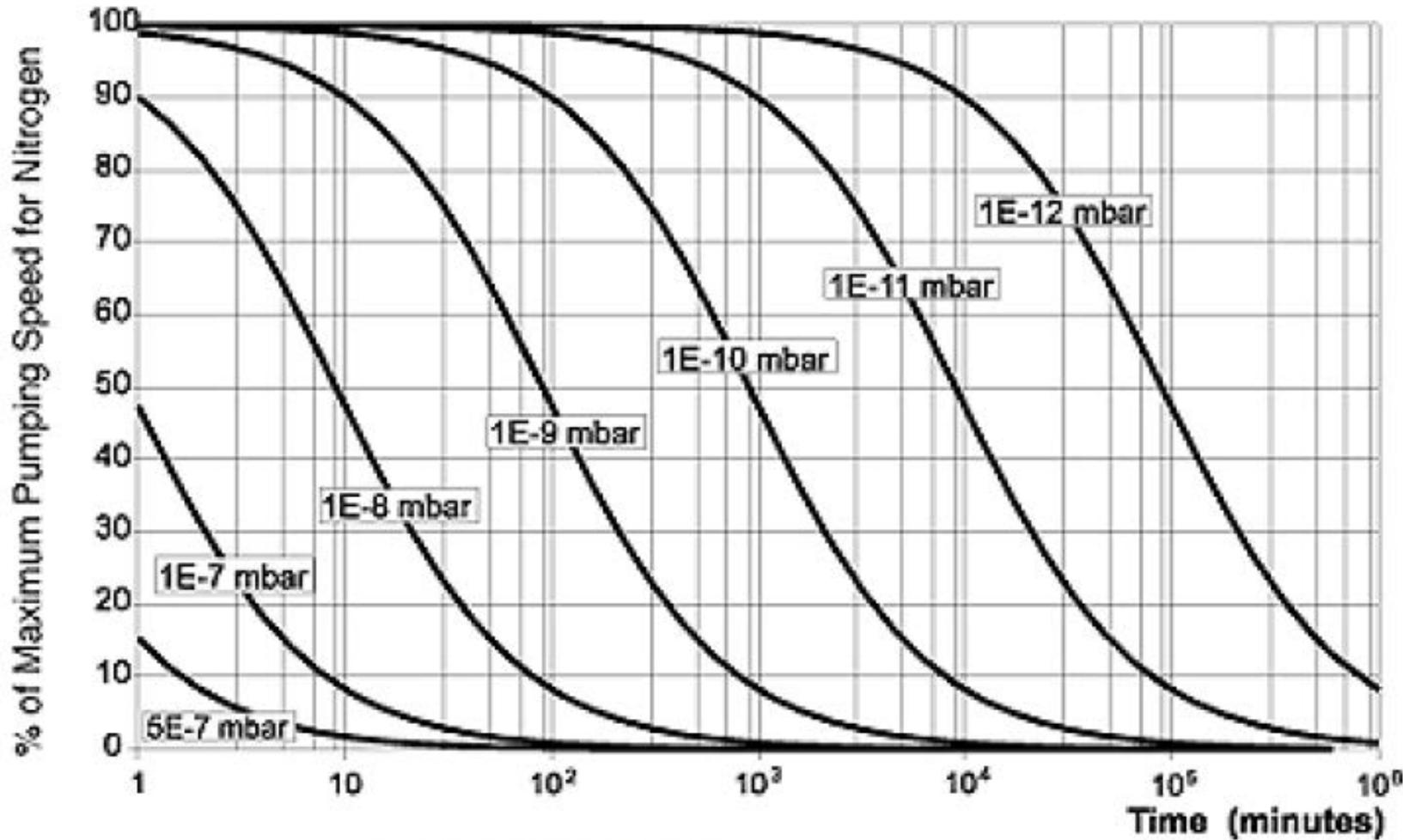
b) Wide variations due to film roughness

c) Wide variations due to bulk diffusion into film

(Ref. "Sorption of Nitrogen by Titanium Films," Harra and Hayward, Proc. Int. Symp. On Residual Gases in Electron Tubes, 1967)



# Pumping Capacity – Just an Example



Ref. Varian Vacuum





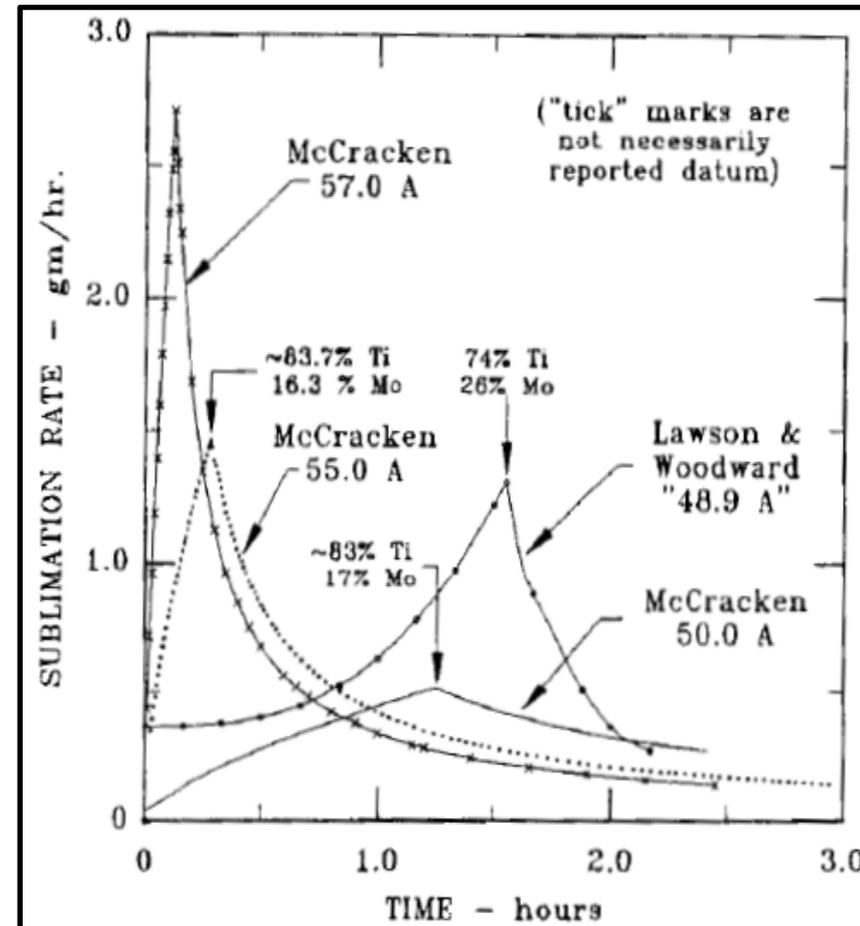
- ❑ For some high gas load, high throughput applications, Ti may be continuously sublimated. In the continuous sublimation mode, proper cooling must be considered.
- ❑ In most applications, Ti is periodically sublimated as the Ti layer is saturated. This is referred to as "batch sublimation". In a batch sublimation mode, the timing of the sublimation is usually based on independent pressure measurement.
- ❑ In batch-mode sublimation, one may choose various control modes: constant current, constant voltage or constant power.



# Sublimation Mode – Constant Current



- ❑ *Constant current operation of Titanium filaments produces increases in sublimation rates early in the filament life.*
- ❑ *This is probably due to the progressively leaner mixture of titanium in the filaments.*
- ❑ *Filaments develop rougher surface textures as the mixture changes.*
- ❑ *Rougher texture*
  - = greater surface area*
  - = higher emissivity*
  - = lower operating temperature*
  - = lower sublimation rates.*



# Sublimation Mode – Constant Voltage



- ❑ *Constant voltage operation is rarely done.*
- ❑ *Constant voltage operation in conjunction with RT cycling produces more predictable sublimation rates*

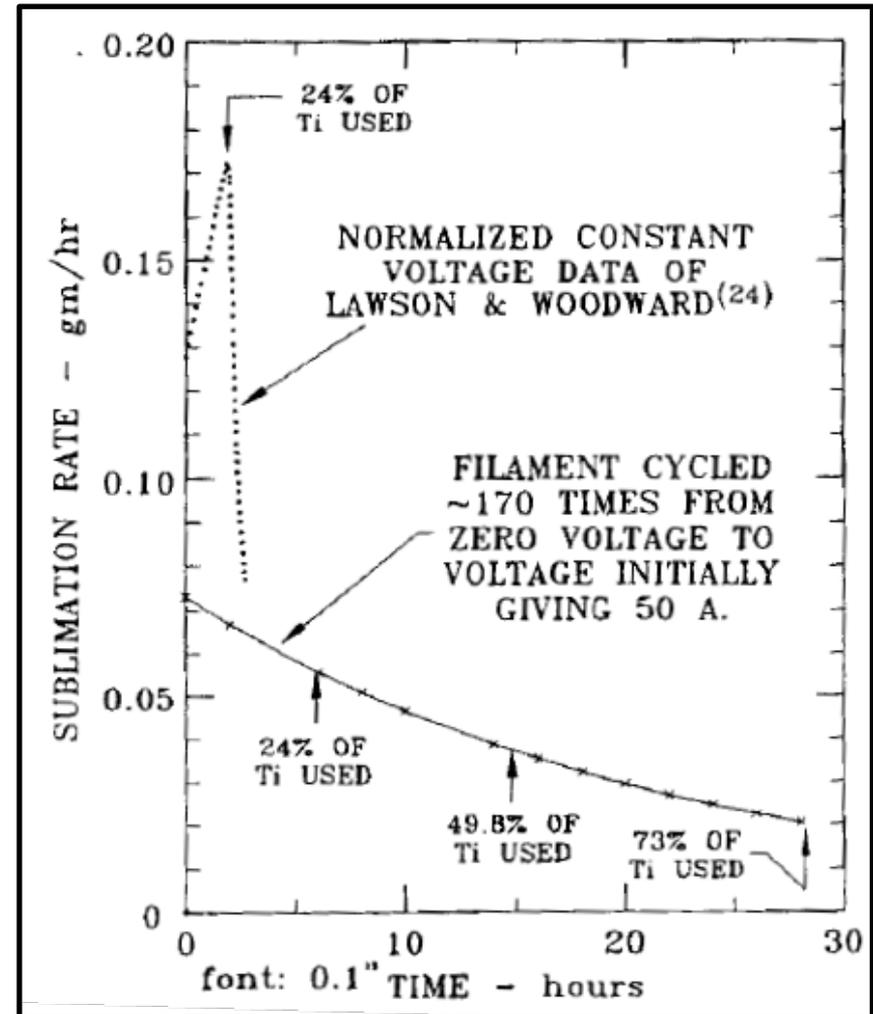
$$R(t) = R_0 e^{-at}$$

where  $R_0$  = initial sublimation rate

$a$  = constant

$t$  = cumulative sublimation time

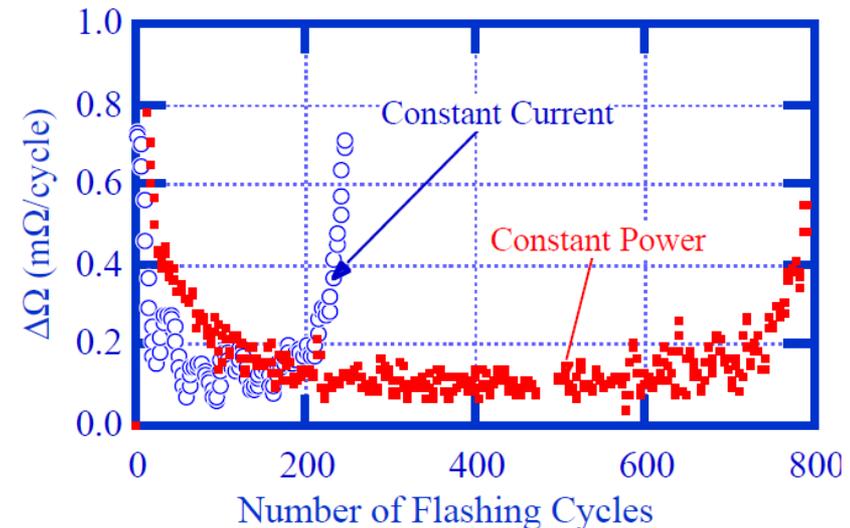
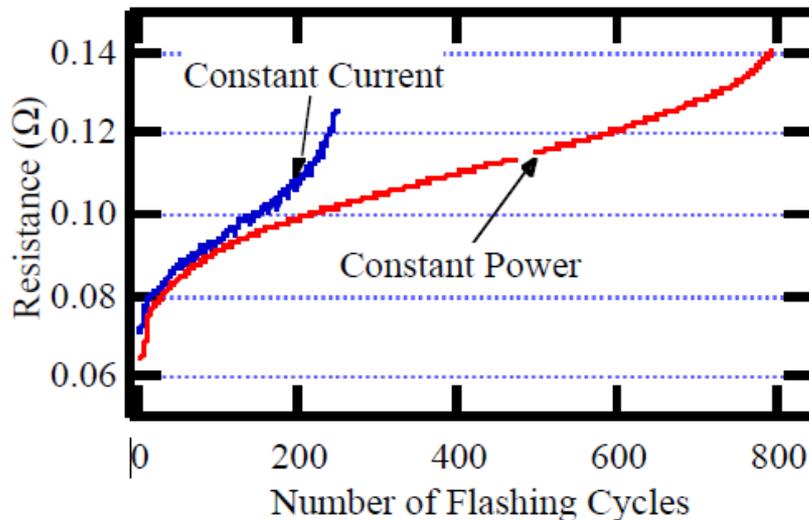
- ❑ *Titanium sublimation rates are dependent on Ti and Mo proportions and the number of temperature cycles through the crystallographic transformation temperature.*



# Sublimation Mode – Constant Power



- ❑ *At CESR, we choose a constant power approach for Ti sublimation (with a LabView® PID controller).*
- ❑ *Using resistance change as a measure of sublimation rate, the constant power mode provide very long term stability of the sublimation rate.*
- ❑ *Constant power mode also ensures longer lifetime of Ti filament.*





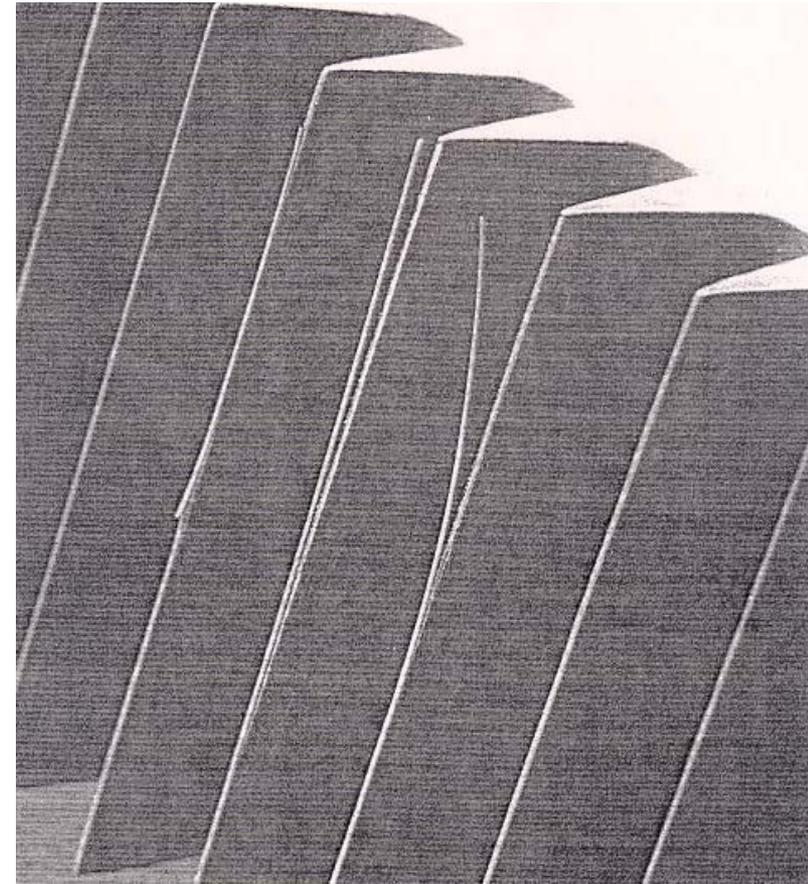
- ❑ *Gas throughput must be estimated for use of TiSPs, so that the sublimation period may be reasonable for the accelerator operations. Measures should be taken in design to maximize Ti covered surface area.*
- ❑ *Baffles must be in place to block all line-of-sight between the Ti filaments and the particle beam space.*
- ❑ *For very long term operations, Ti thin film peeling may be an issue. Orientation and placement of Ti filaments play a role in minimize particle generations to the beam space.*
- ❑ *Ti filaments may become EMI antennae when not properly shielded against short bunched particle beam. Sometime RF filtering is necessary.*
- ❑ *Adequate protections (mechanical and corrosive) are important for the electric feedthroughs on the Ti cartridges.*



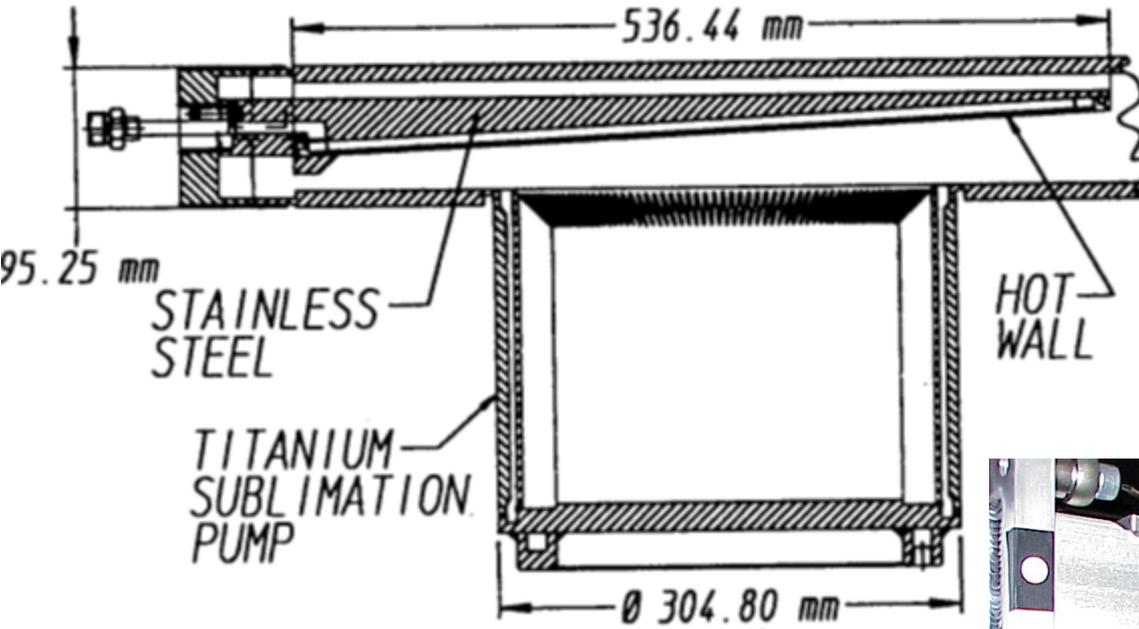
# Peeling of Titanium Films



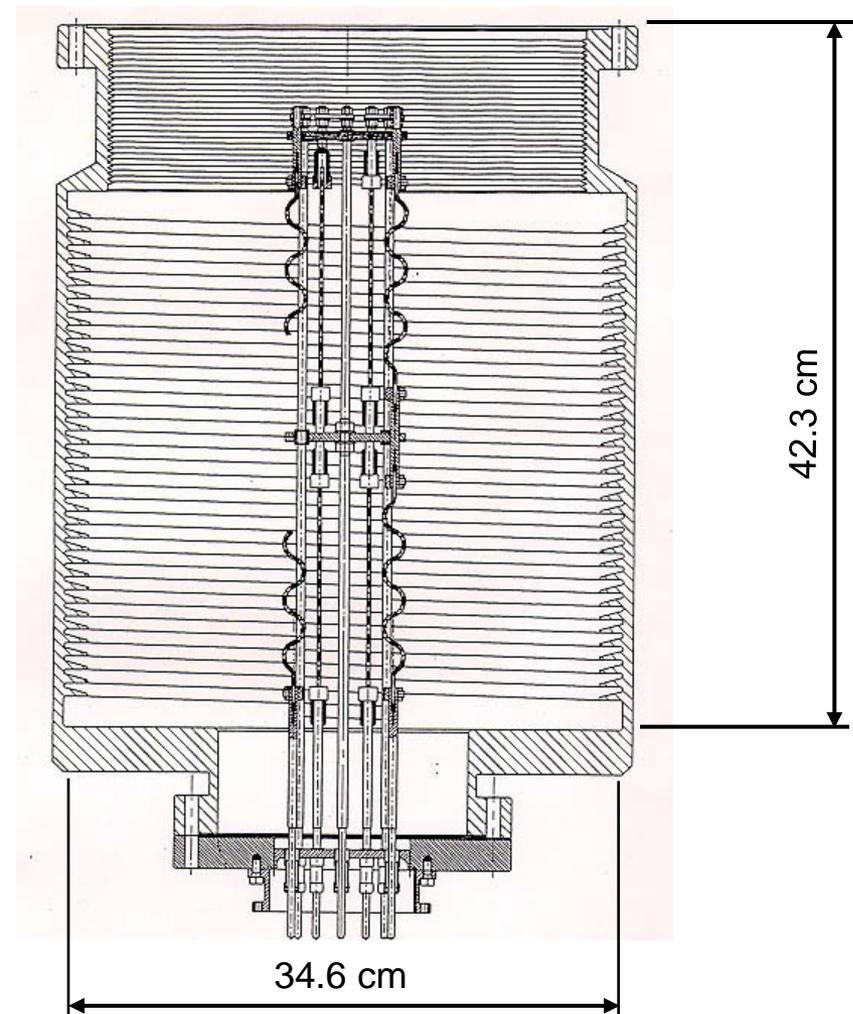
- *As Titanium builds-up on a pumping surface, it will begin to peel.*
- *A typical thickness where peeling begins is 0.05 mm.*
- *Peeling produces dust particles and increases surface temperatures during sublimation.*
- *Because of peeling, pumping surfaces may require periodic cleaning (glass bead blasting and/or chemical cleaning).*
- *If peeling is a problem, a TSP was probably a bad choice or you are misusing the pumps.*



# PEP-II LER Arc TSP and Photon Stop



- Used at photon stops
- Specially ordered cartridge with more filaments
- Grooved interior surfaces to increase pumping speed and capacity

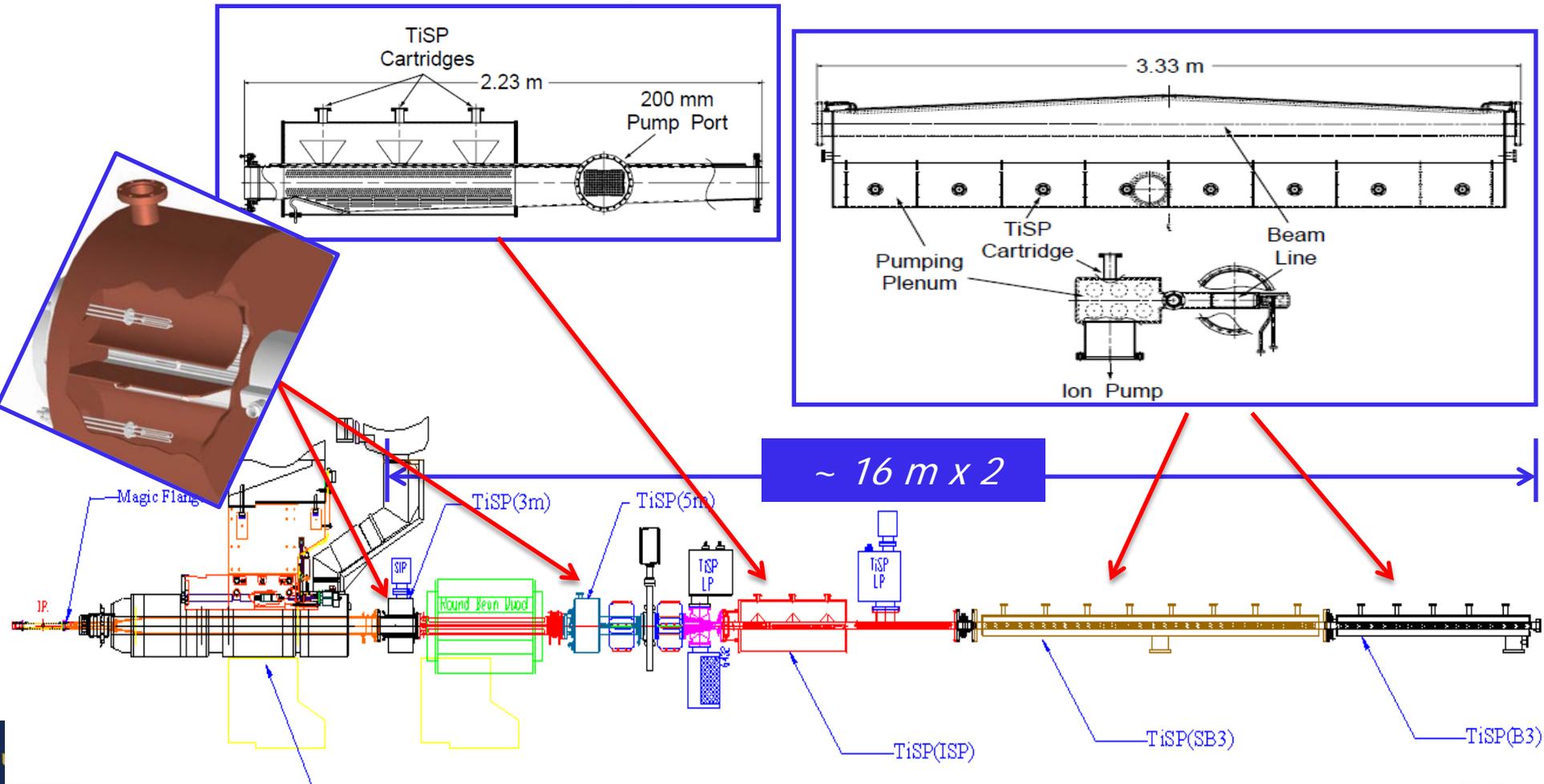


Courtesy: C. Vaccarezza, INFN

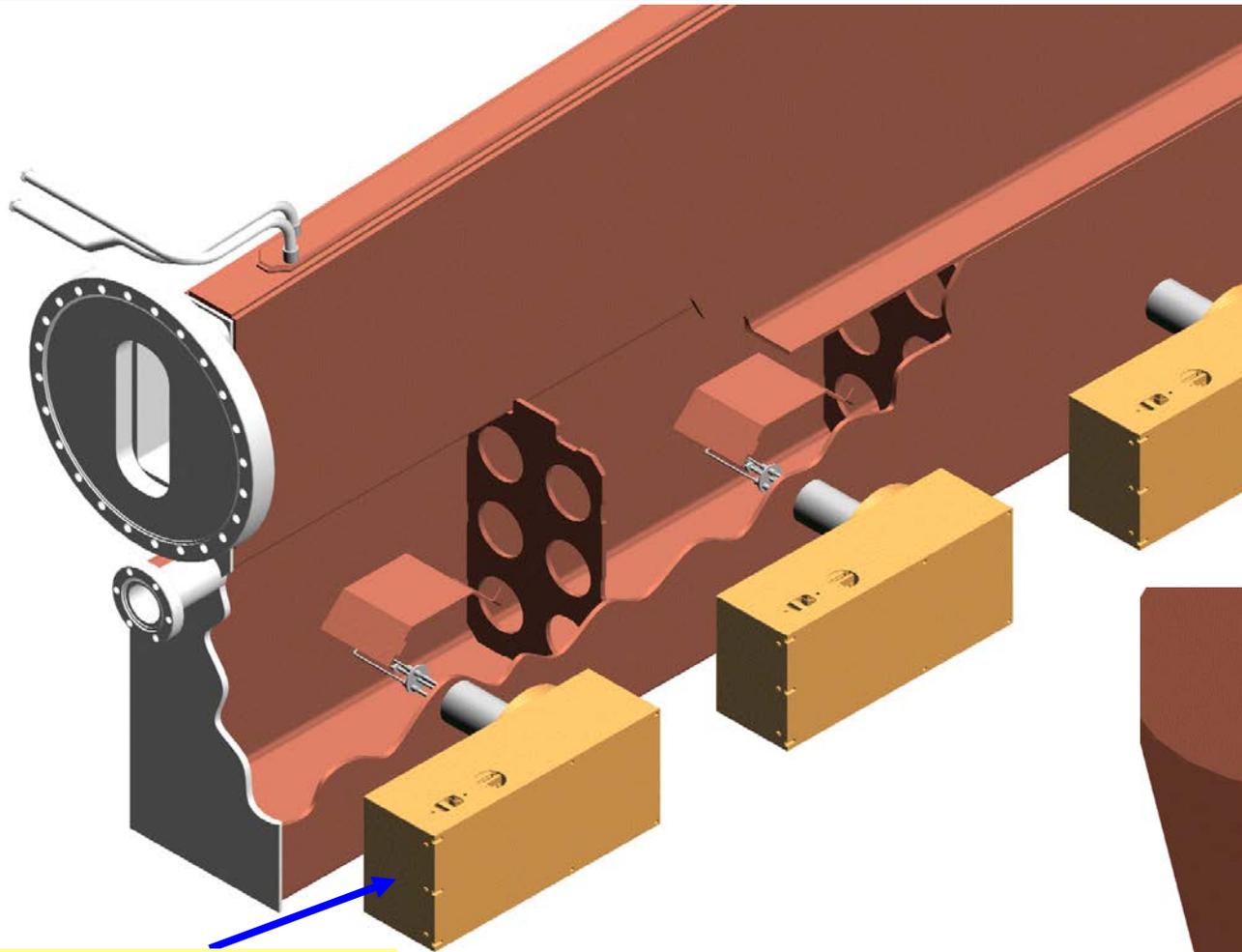
# TiSPs in CESR Interaction Region



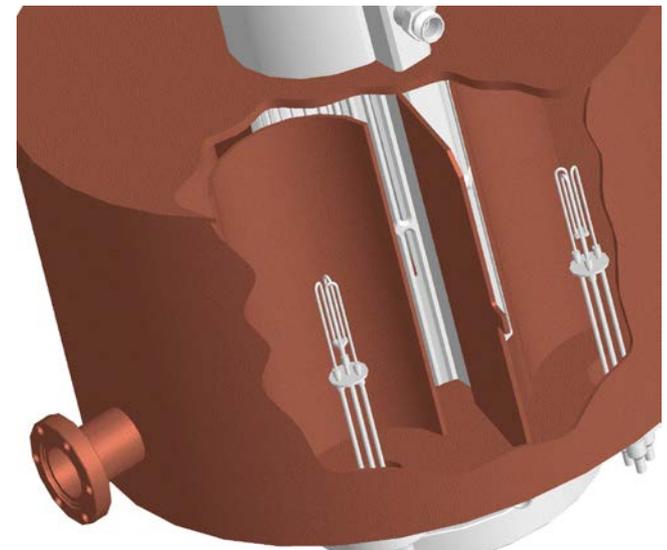
- During CESR/CLEO III era, distributed TiSPs were implemented as the main pumping system.
- 2X26 TiSP cartridges populated ~32 m. A RF-filter, multiplexing TiSP control system was also deployed.



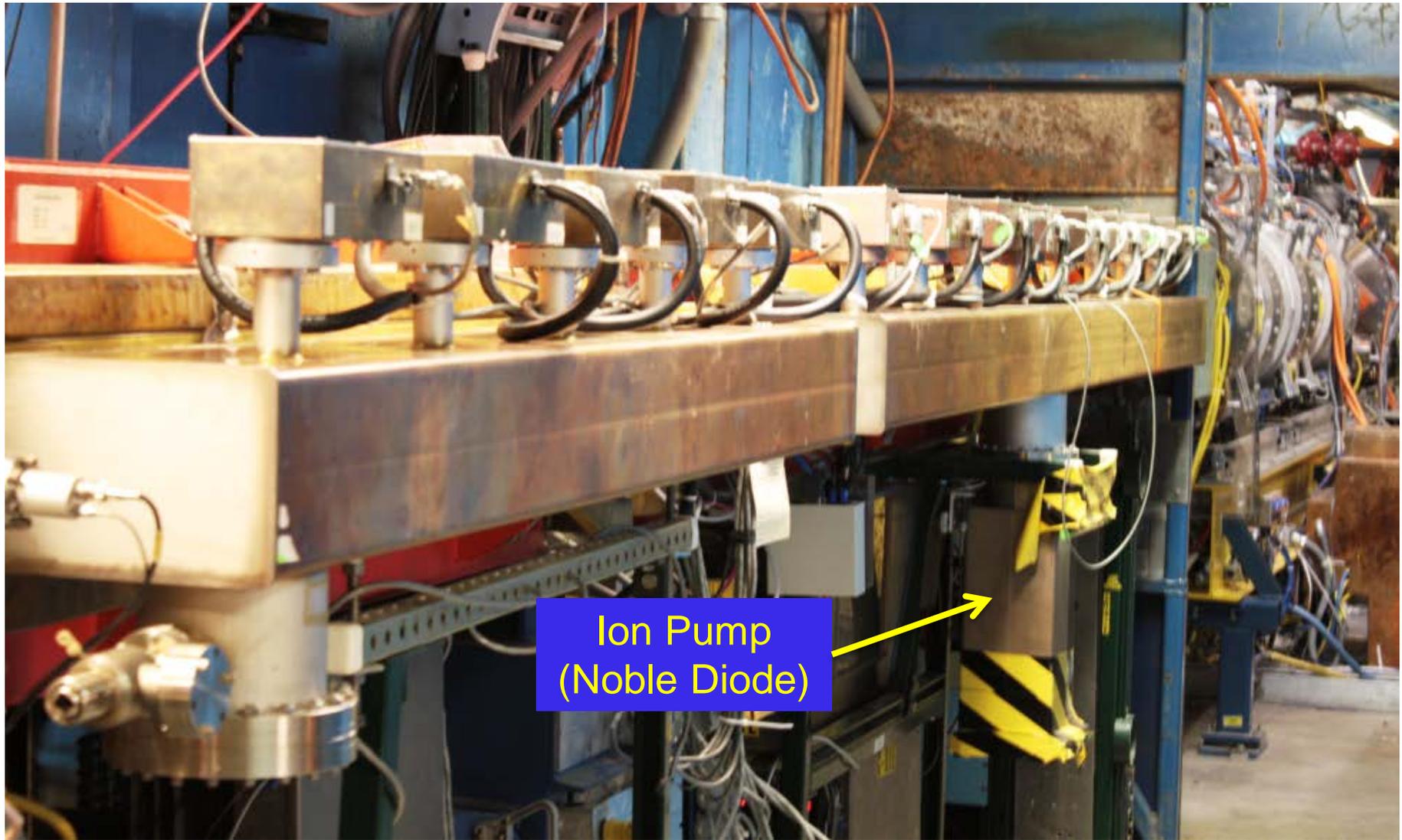
# TiSP Chambers in CESR – A Close Look



Relay & EMI  
Filter Boxes



# *TiSPs in CESR Interaction Region*



# TiSPs in CESR IR – Performance History

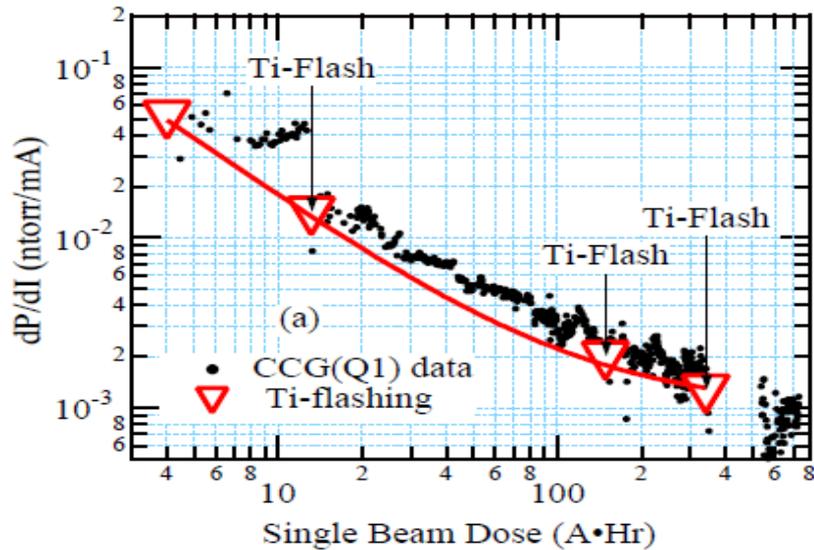
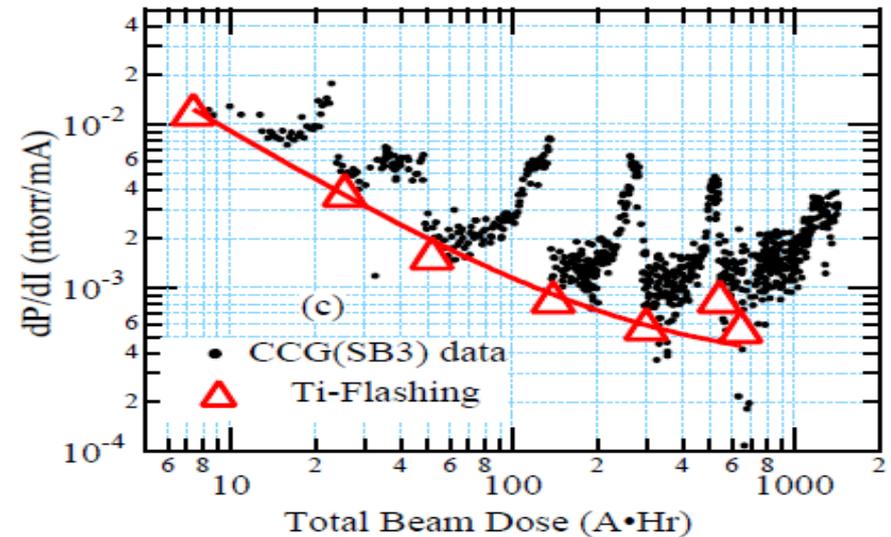
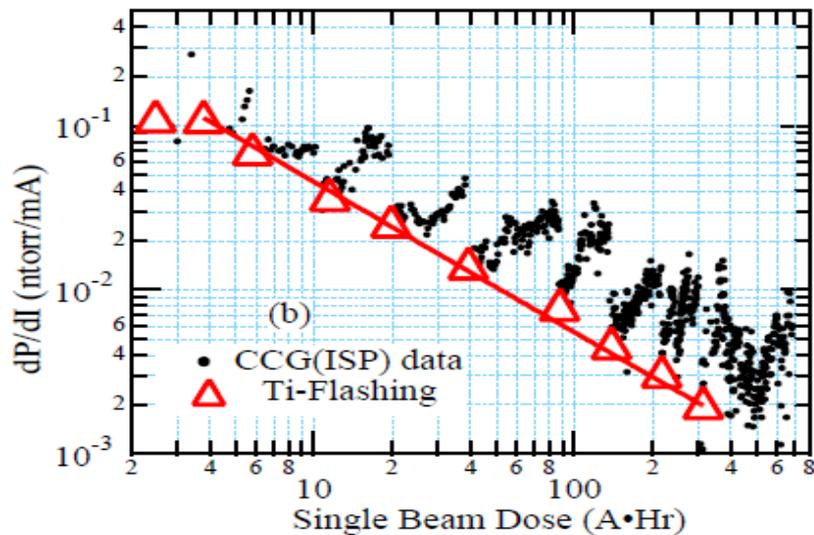


TABLE I. Synchrotron Radiation Flux and Gas Load in the CESR IR for 300 mA  $e^\pm$  Stored Beams.

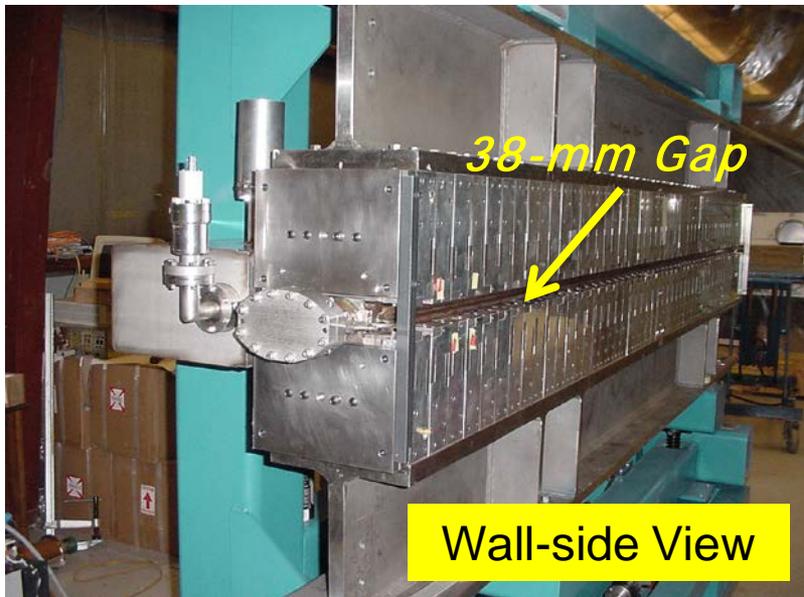
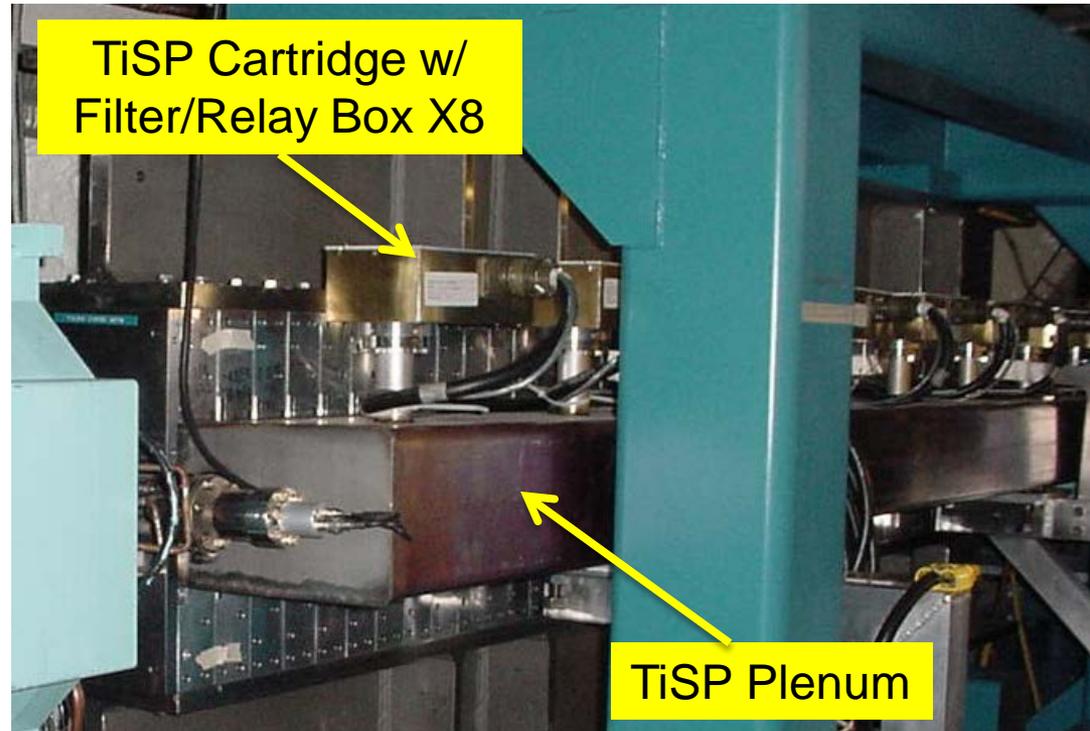
Location	Flux Density (Photon/s/m)	Total Flux (Photons/s)	CO GasLoad [Torr-l/s]
Q1-Pump	$1.3 \cdot 10^{18}$	$2.5 \cdot 10^{17}$	$7.9 \cdot 10^{-8}$
ISP absorb.	$1.1 \cdot 10^{19}$	$1.9 \cdot 10^{18}$	$2.0 \cdot 10^{-7}$
Soft-Bend	$3.3 \cdot 10^{18}$	$3.5 \cdot 10^{18}$	$6.7 \cdot 10^{-7}$
Hard-Bend	$6.9 \cdot 10^{18}$	$2.3 \cdot 10^{19}$	$1.3 \cdot 10^{-6}$



# TiSPs in a Wiggler Vacuum Chamber



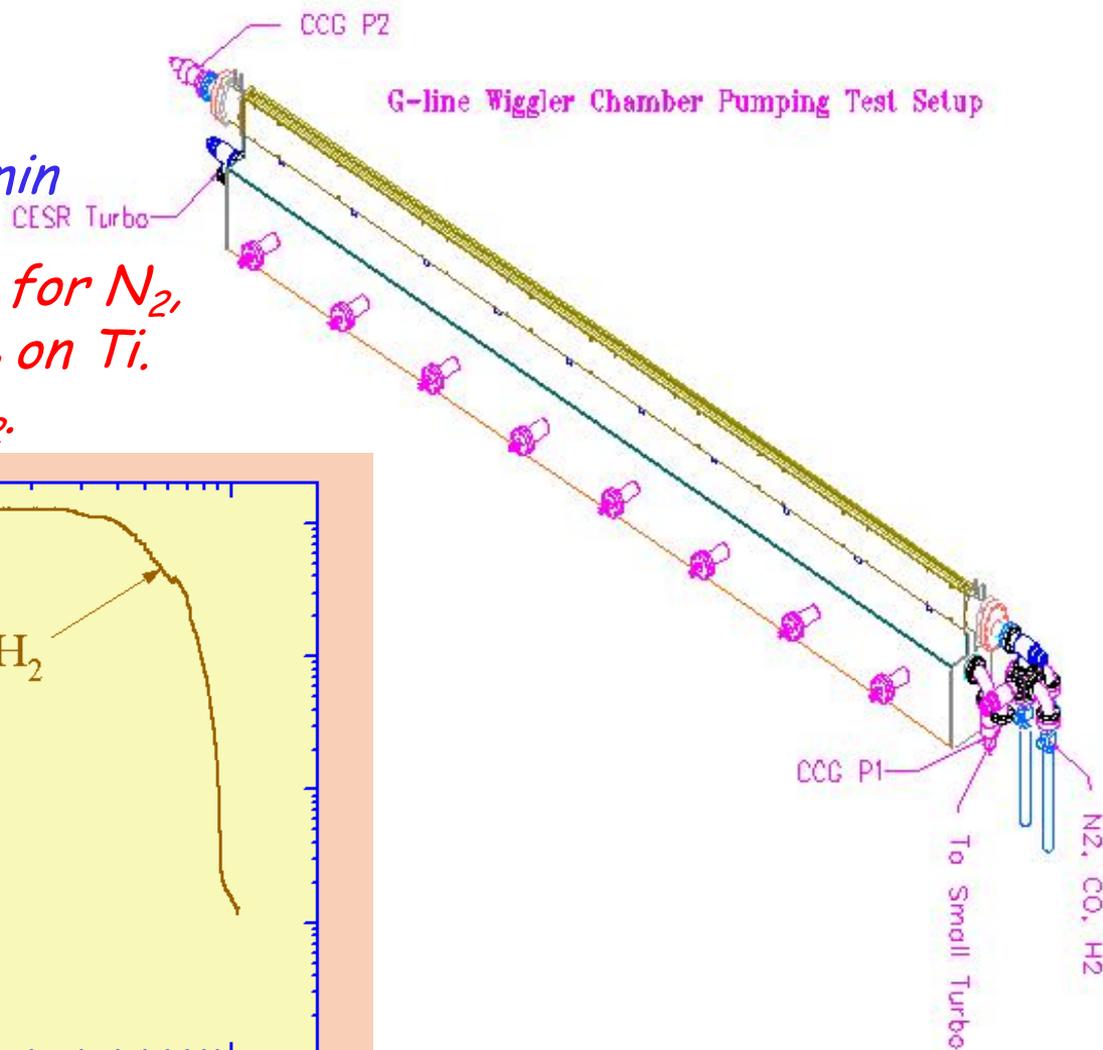
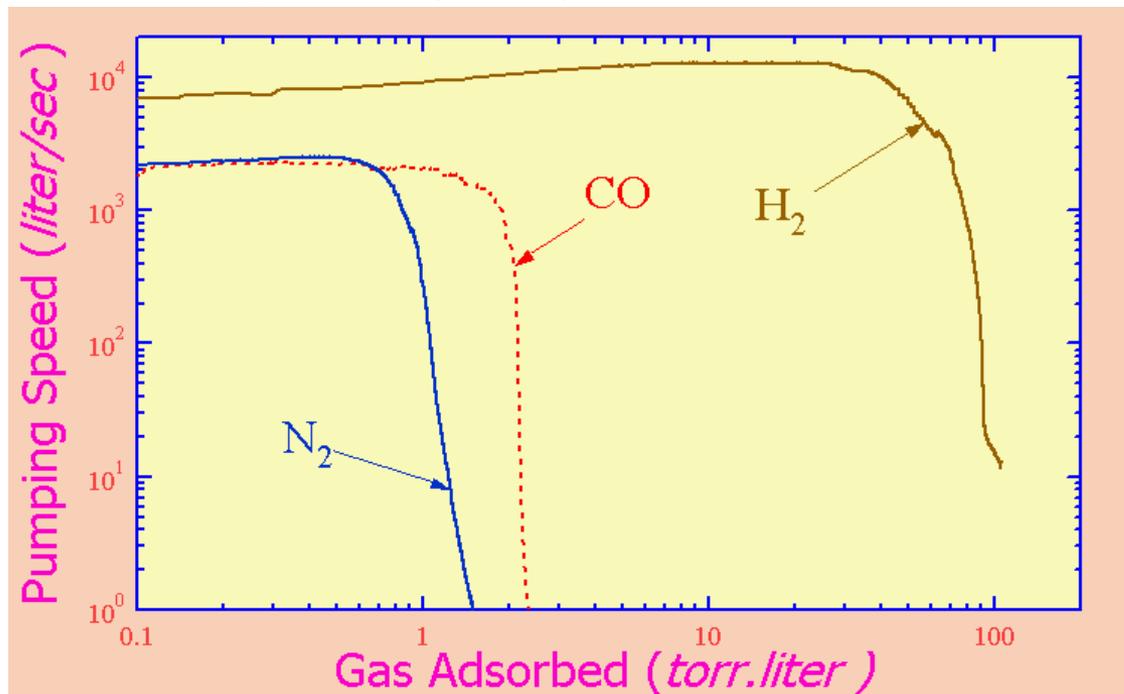
TiSP was incorporated in narrow gapped wiggler chamber for the CHESS G-line



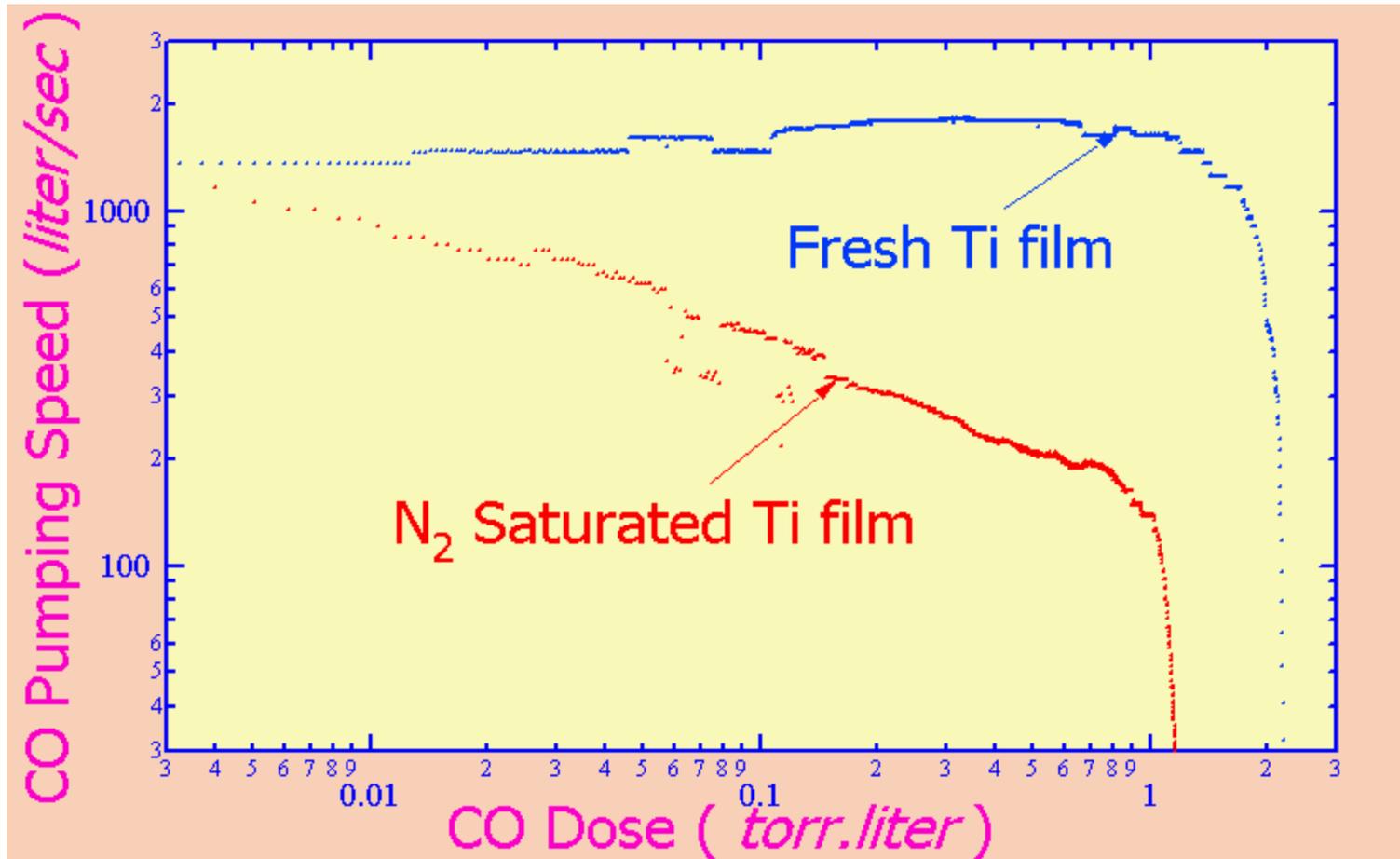
# TiSPs in a Wiggler VC – Pumping Speed



- ✓ Calibrated leaks used to measure pumping speed
- ✓ TiSPs sublimation: 200W-2min
- ✓ Twice as capacity for CO as for N<sub>2</sub>, indicating dissociation of N<sub>2</sub> on Ti.
- ✓ Much higher capacity for H<sub>2</sub>.



# TiSPs Pumping – Another Look



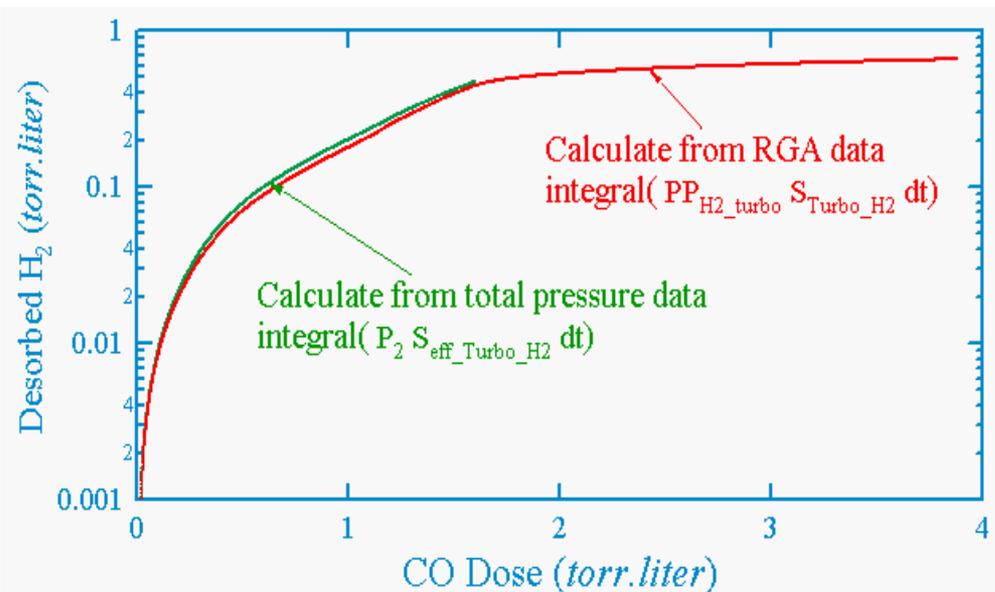
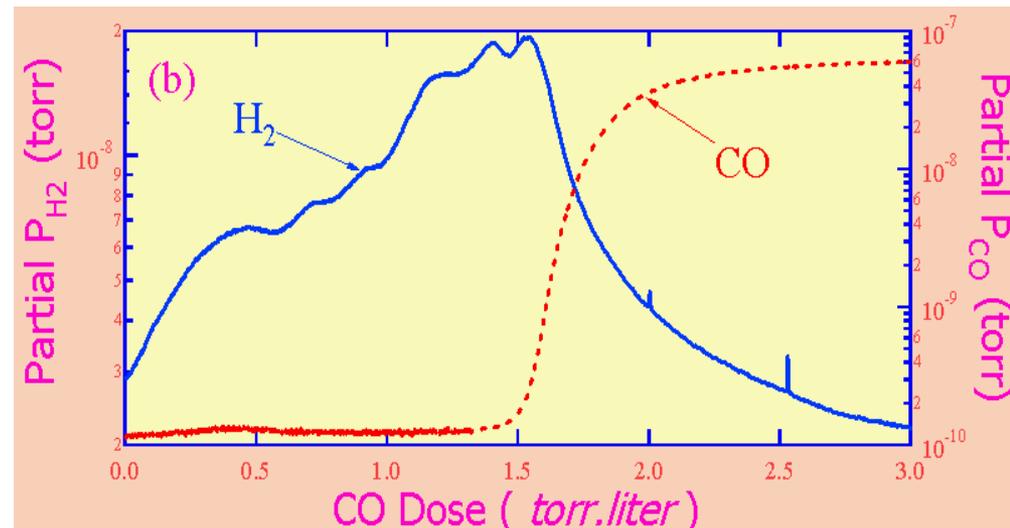
*More reactive CO re-arrange adsorbed N atoms on Ti surfaces. Note the (1/2)-capacity for CO*



# CO Adsorption on Hydrogen-saturated Ti Film



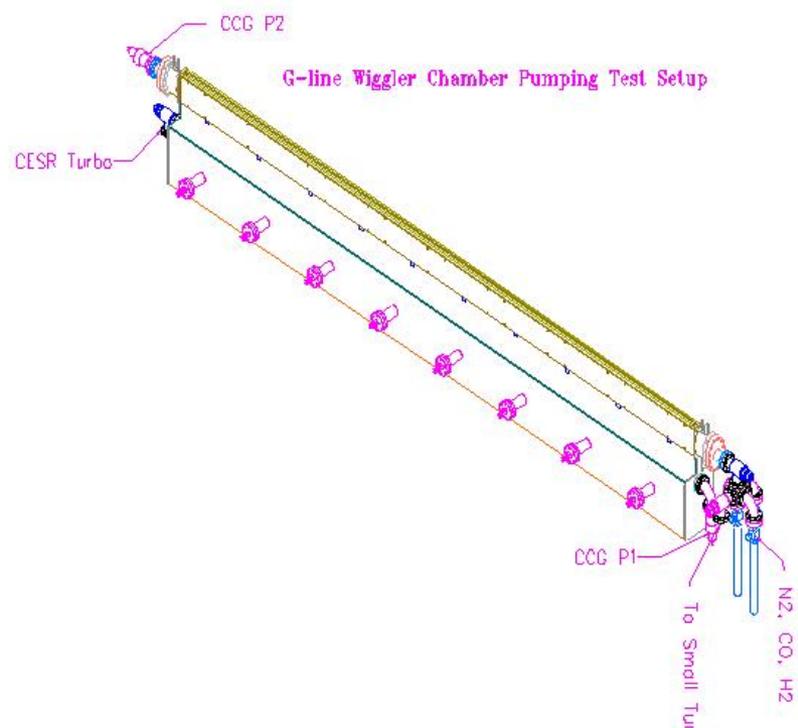
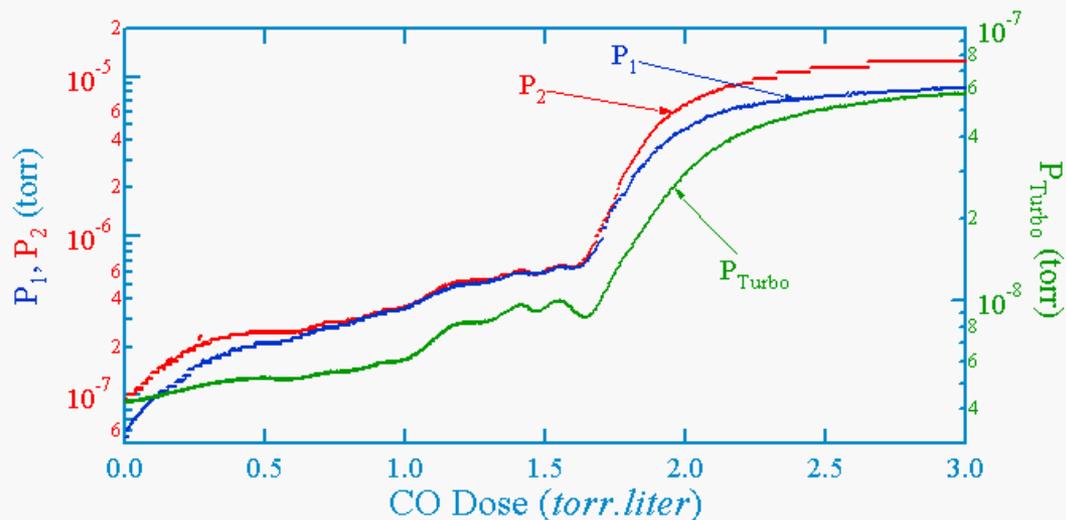
- After saturating Ti surface with  $\sim 100$  torr-liter  $H_2$ , introduce CO.
- RGA data clearing indicating further adsorption of CO, and desorption of  $H_2$  simultaneously.



- Careful quantitative analysis showed CO promoted H recombination desorption.
- 1.5 torr-liter of CO replaced  $\sim 0.7$  torr-liter of  $H_2$ !

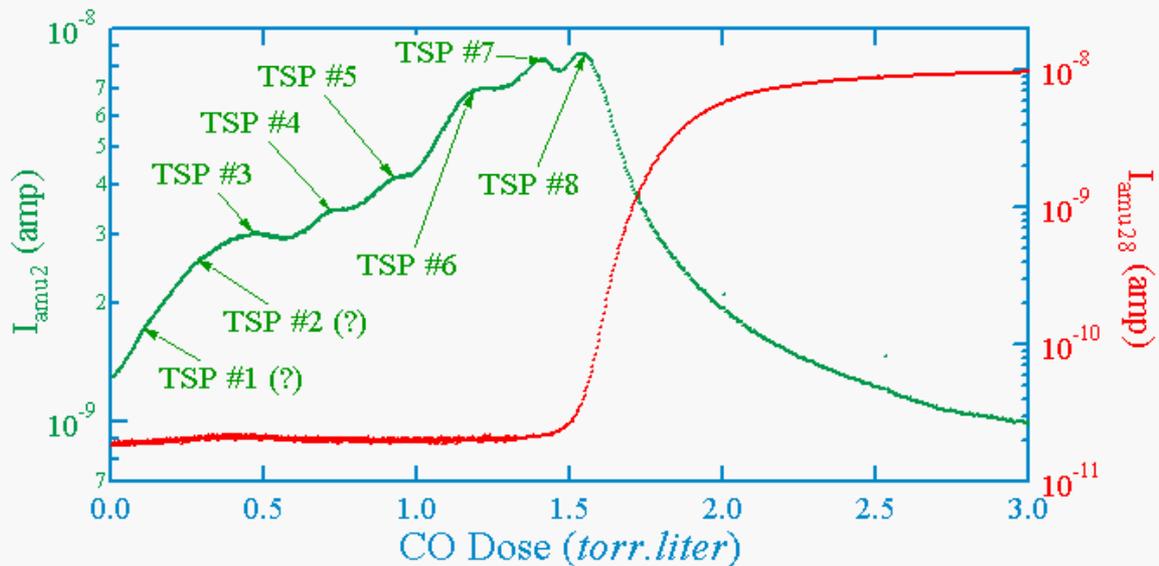


# Important Partial Pressure Info



Total pressure gauges data.  
Hard to tell the displacement  
of  $H_2$  by CO.

RGA data clearly  
showing CO  
replacing  $H_2$ .





# Session 4.3B

## Non-Evaporable Getters (NEGs)





- Porous alloys with very large active metallic surface area, when activated.
- Bulk Getters - gases diffuse into the interior of the getter material upon heating (activation).
- Gases are categorized into four families based on their interactions with NEGs:
  - ✓ 1. **Hydrogen and its isotopes** - adsorbed reversibly.
  - ✓ 2. **CO, CO<sub>2</sub>, O<sub>2</sub>, and N<sub>2</sub>** - adsorbed irreversibly.
  - ✓ 3. **H<sub>2</sub>O, hydrocarbons** - adsorbed in a combination of reversible and irreversible processes. Hydrocarbons are adsorbed very slowly.
  - ✓ 4. **Noble gases** - not adsorbed at all.





- NEGs are primarily 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

- NEGs are also available only from:

Gamma Vacuum, Edwards Vacuum Inc.

2915 133rd Street West

Shakopee, MN 55379 • USA





## Hydrogen

- ❑ *Hydrogen does not form a stable chemical compounds with a NEG alloy. It dissociatively chemisorbs on active NEG surfaces and atomic hydrogen diffuses rapidly into the bulk of the getter and is stored as a solid solution. Thus at ambient temperature, NEGs are very effective pumps for hydrogen.*
- ❑ *Sieverts' Law describes the relationship between  $H_2$  concentration within its NEG and its equilibrium pressure. Usually, high  $H_2$  pressure, ranging  $10^{-7}$  to  $10^{-4}$  torr, is expected during NEG activation.*

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

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

$p$  =  $H_2$  equilibrium pressure, Torr

$T$  = getter temperature, K

$A, B$  constants for different NEG alloys





*CO, CO<sub>2</sub>, O<sub>2</sub>, N<sub>2</sub>, other O-, C-containing molecules*

- ❑ *Active gases are chemisorbed irreversibly by NEG.*
- ❑ *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.*
- ❑ *At activation temperatures, these chemical bonds do not 'break'. Instead, the elevated activation temperature promotes diffusion into the bulk of the NEG, to 'regenerate' active surface 'sites'.*





## *H<sub>2</sub>O and Hydrocarbons*

- *Water vapor and hydrocarbons are "cracked" on the surface of the NEG.*
- *H<sub>2</sub> is chemisorbed reversibly.*
- *O<sub>2</sub> and C are chemisorbed irreversibly.*
- *However, hydrocarbons sorption efficiency at ambient temperature is extremely low. However, HC pumping may be enhanced with 'hot' NEGs.*



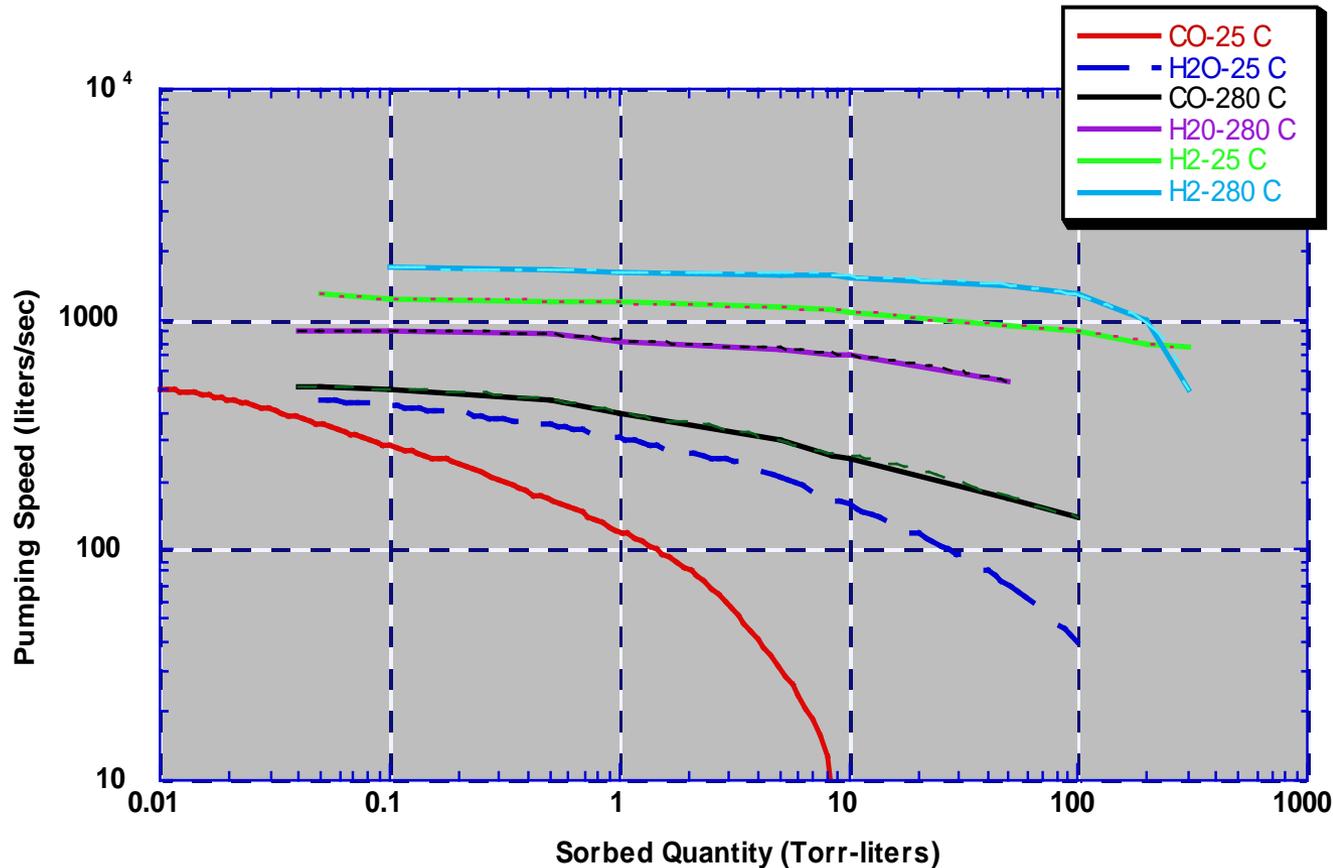


## *Noble gases*

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



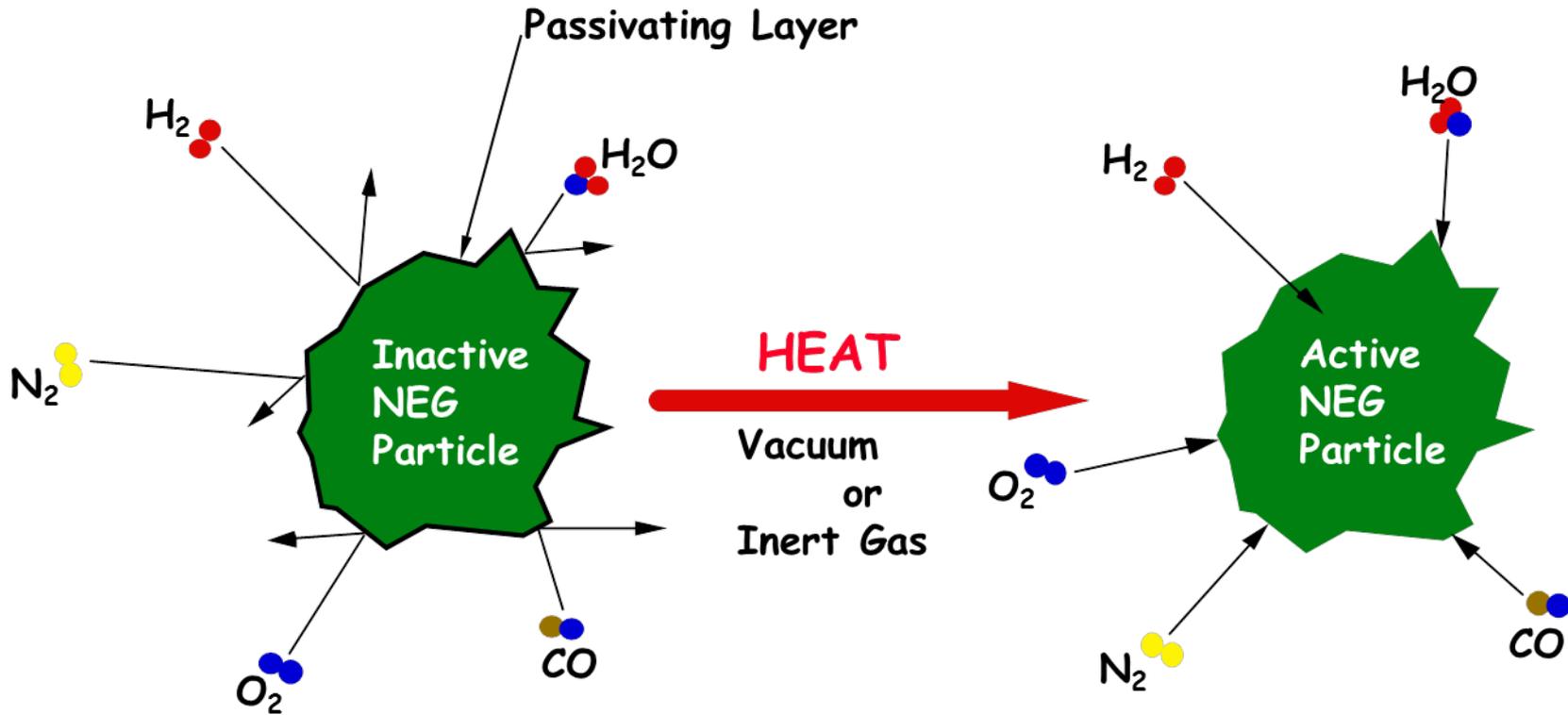
# NEG Pumping Characteristics (5)



- *At low throughput, NEG pumping speeds are constant, independent of pressure.*
- *Pumping speeds usually increase at higher sorption temperature.*



# Activation Process for NEG



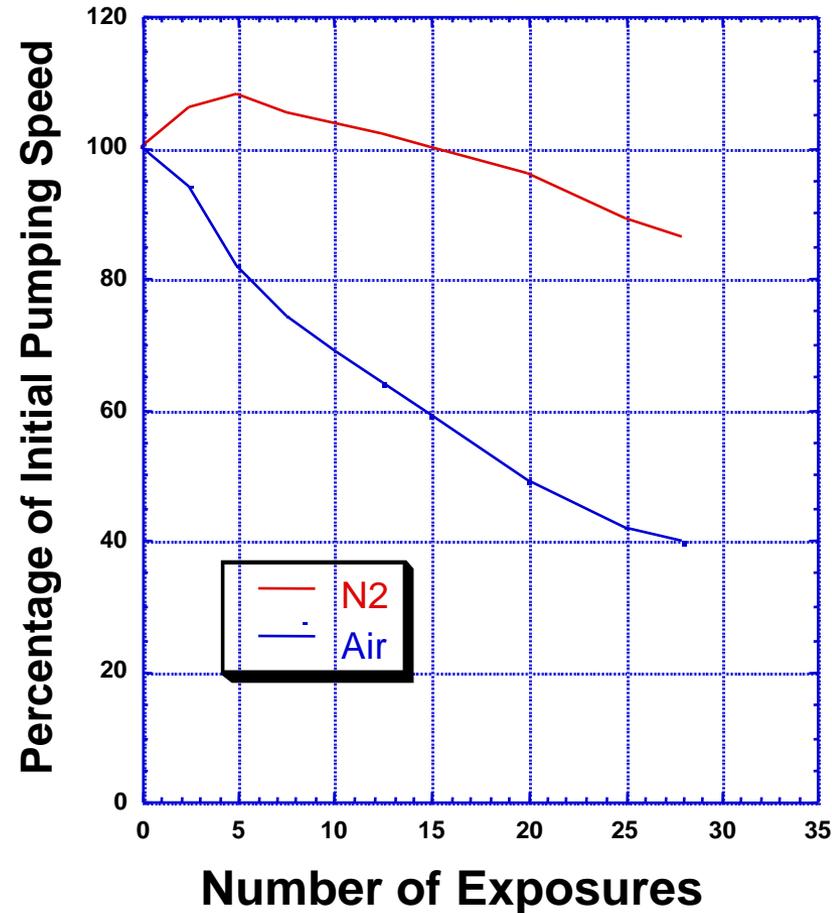
Ref. SAES Getters



# Application Notes for NEGs



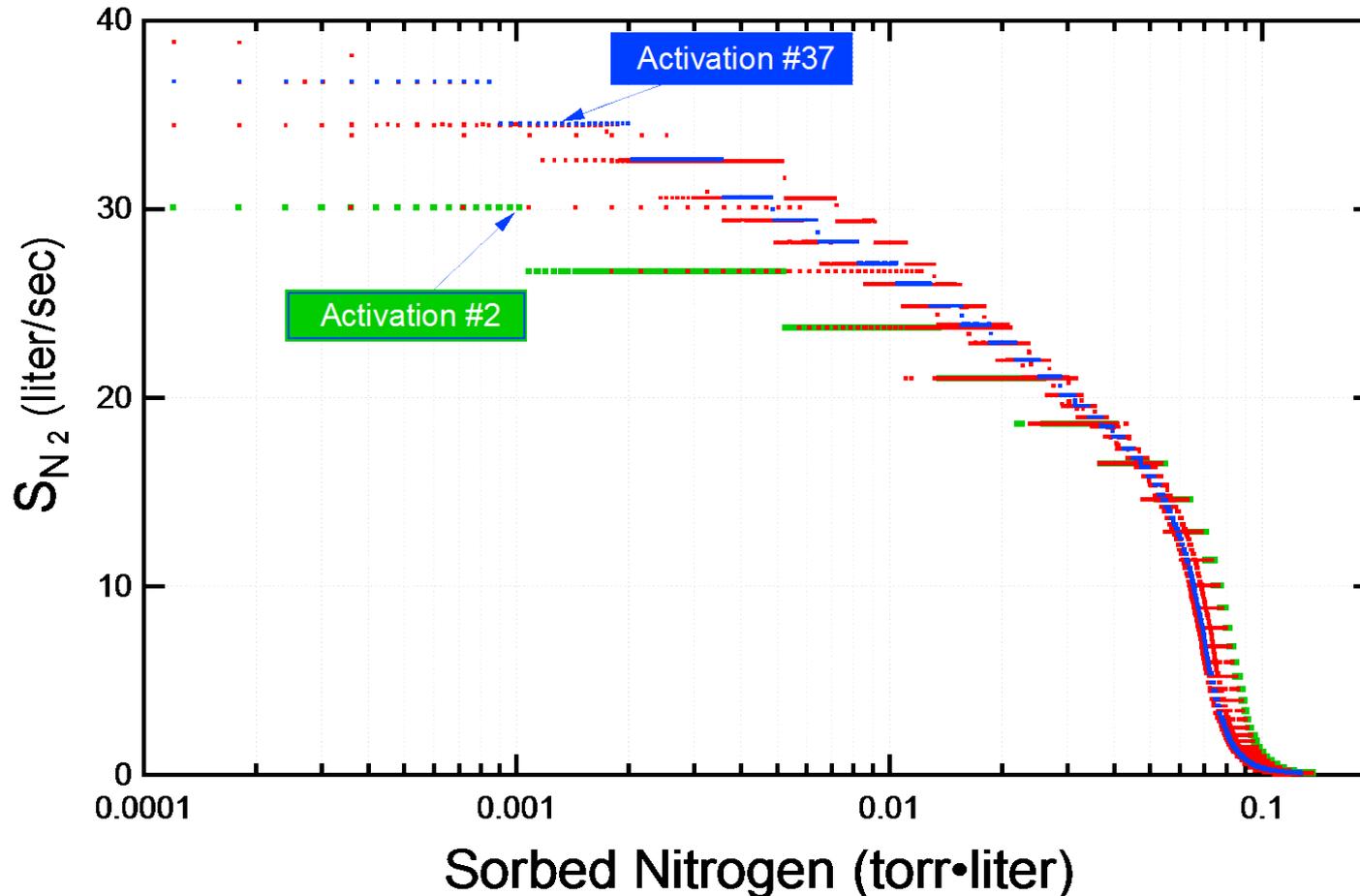
- ❖ *NEG performance deteriorates due to successive exposures to air (oxygen and water) or N<sub>2</sub>.*
- ❖ *Further improvement can be obtained if Argon is used as a protective gas, during long term storage.*
- ❖ *NEG pumps should never be exposed to air while at temperatures higher than 50°C.*
- ❖ *Degassing (or conditioning) of NEG's after initial pump-down.*
- ❖ *Whenever practical, activate all NEG's in a system at the same time.*



Ref. SAES Getters



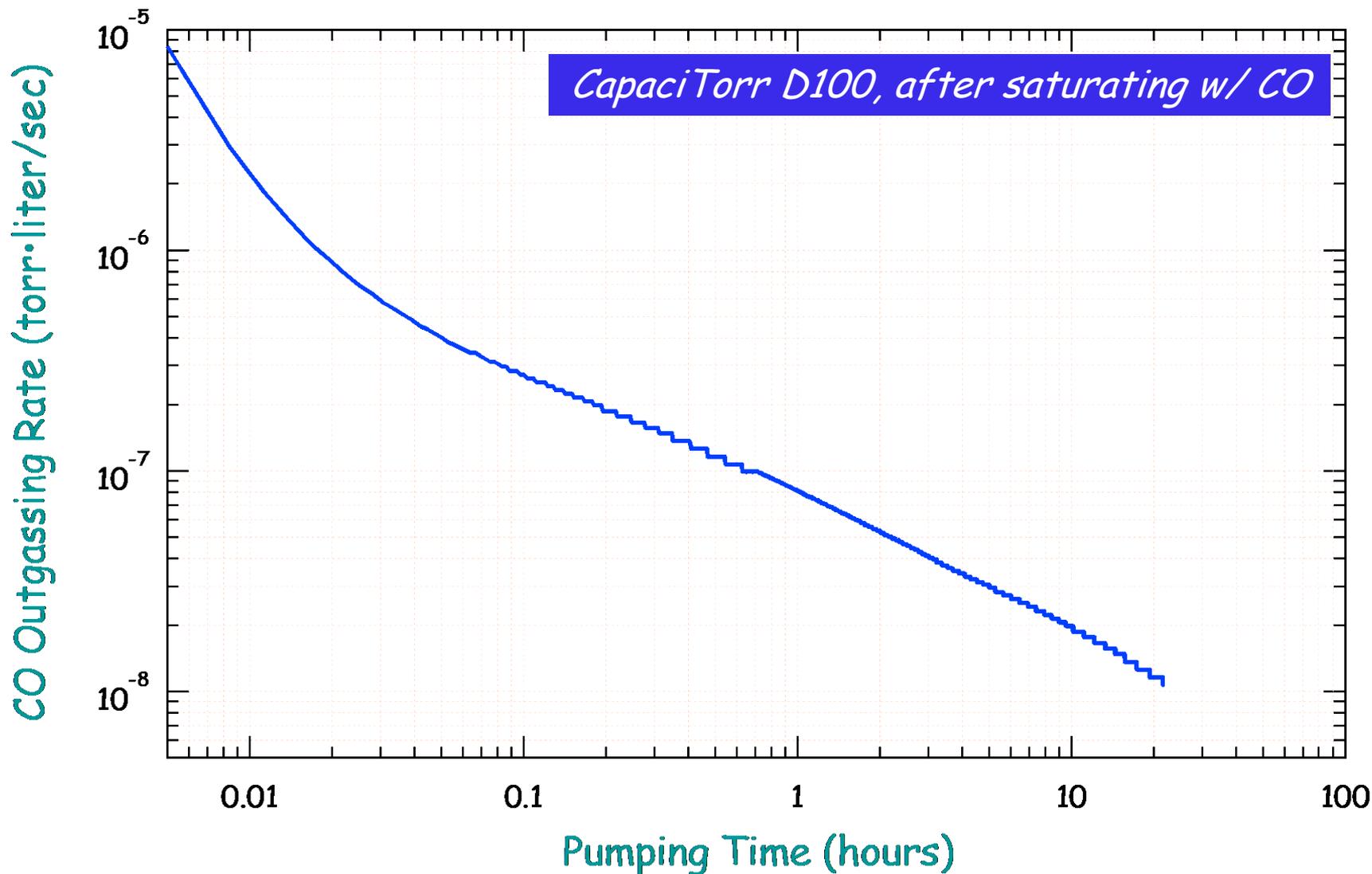
# Pumping with Repeated Activations



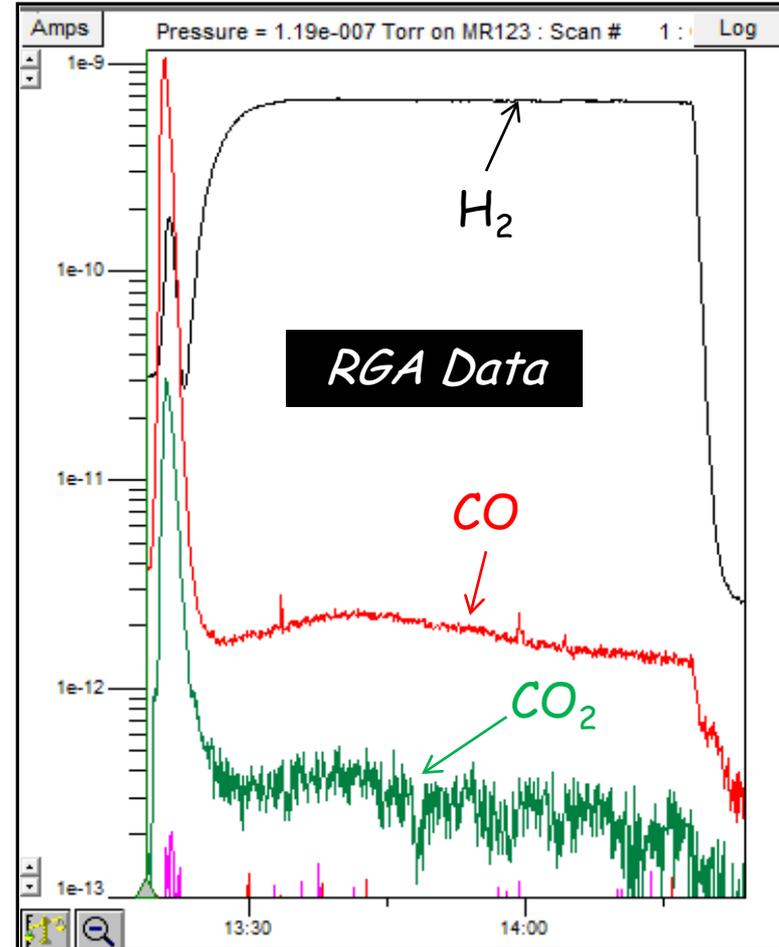
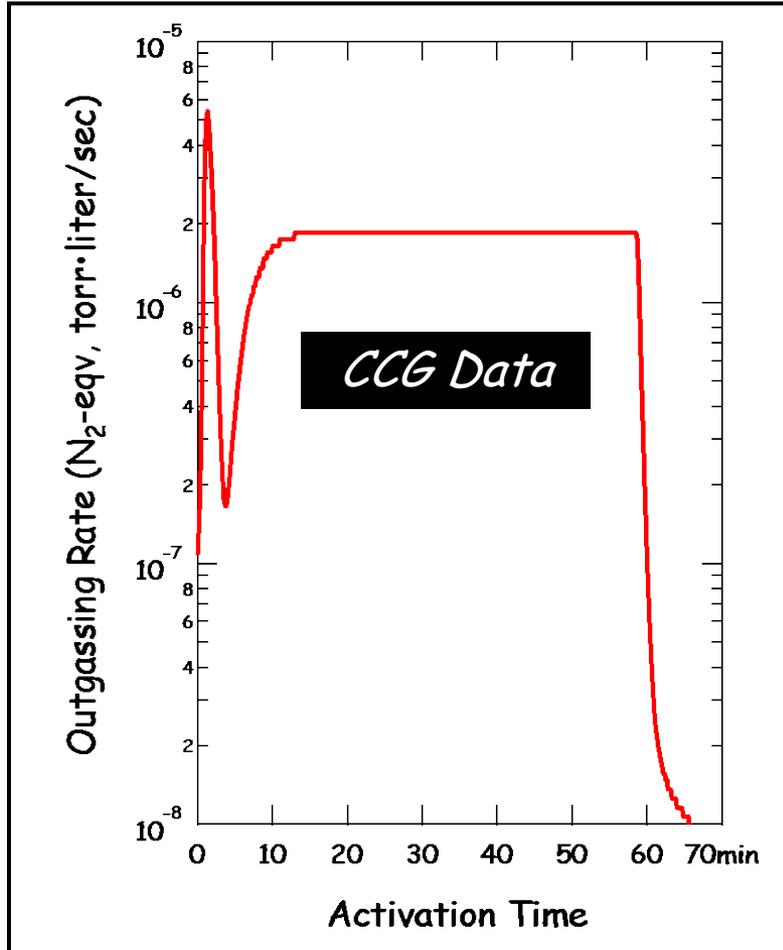
*Measured  $N_2$  pumping on a CapaciTorr D100 pump with repeated activation/saturation cycles w/o venting*



# Outgassing of a fully saturated NEG



# Take a look at NEG activation



*This activation of a SAES' CapaciTorr D100 pump shows typical behavior. (The pump was previously saturated with CO gas, after a 24-hr pumping.)*

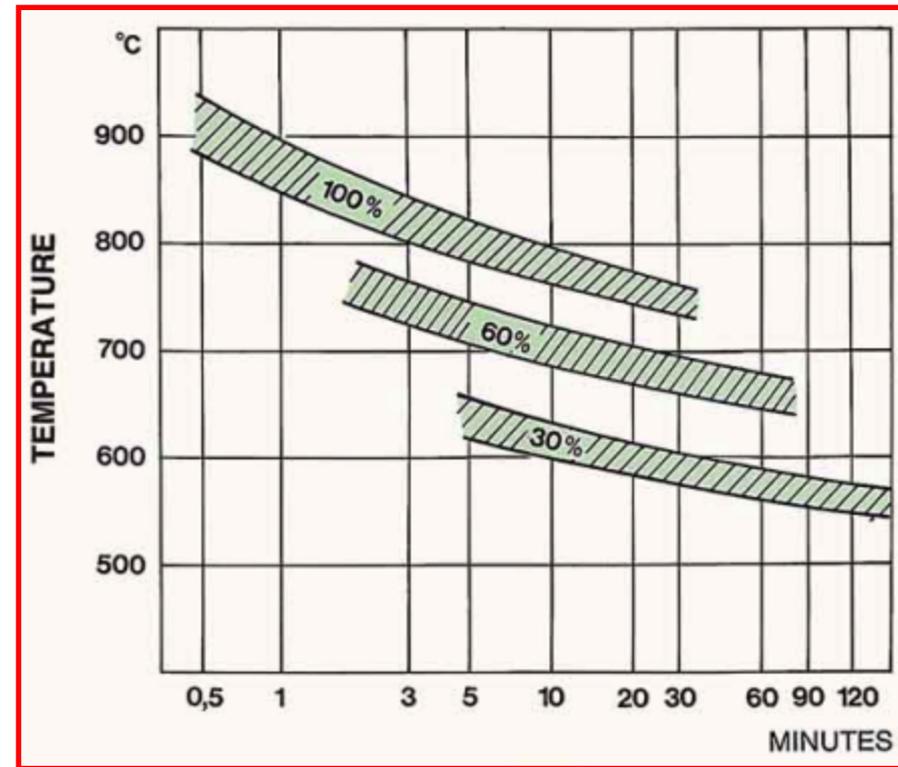


# Examples of SAES Getter's Available NEG Alloys

# SAES ST101<sup>®</sup> 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<sub>2</sub>, H<sub>2</sub>O, N<sub>2</sub>, and O<sub>2</sub> at high rates.
- ❑ ST101 activates at temperatures from 550 to 900°C.
- ❑ ST101 alloy has been replaced by new alloys with lower activation T.

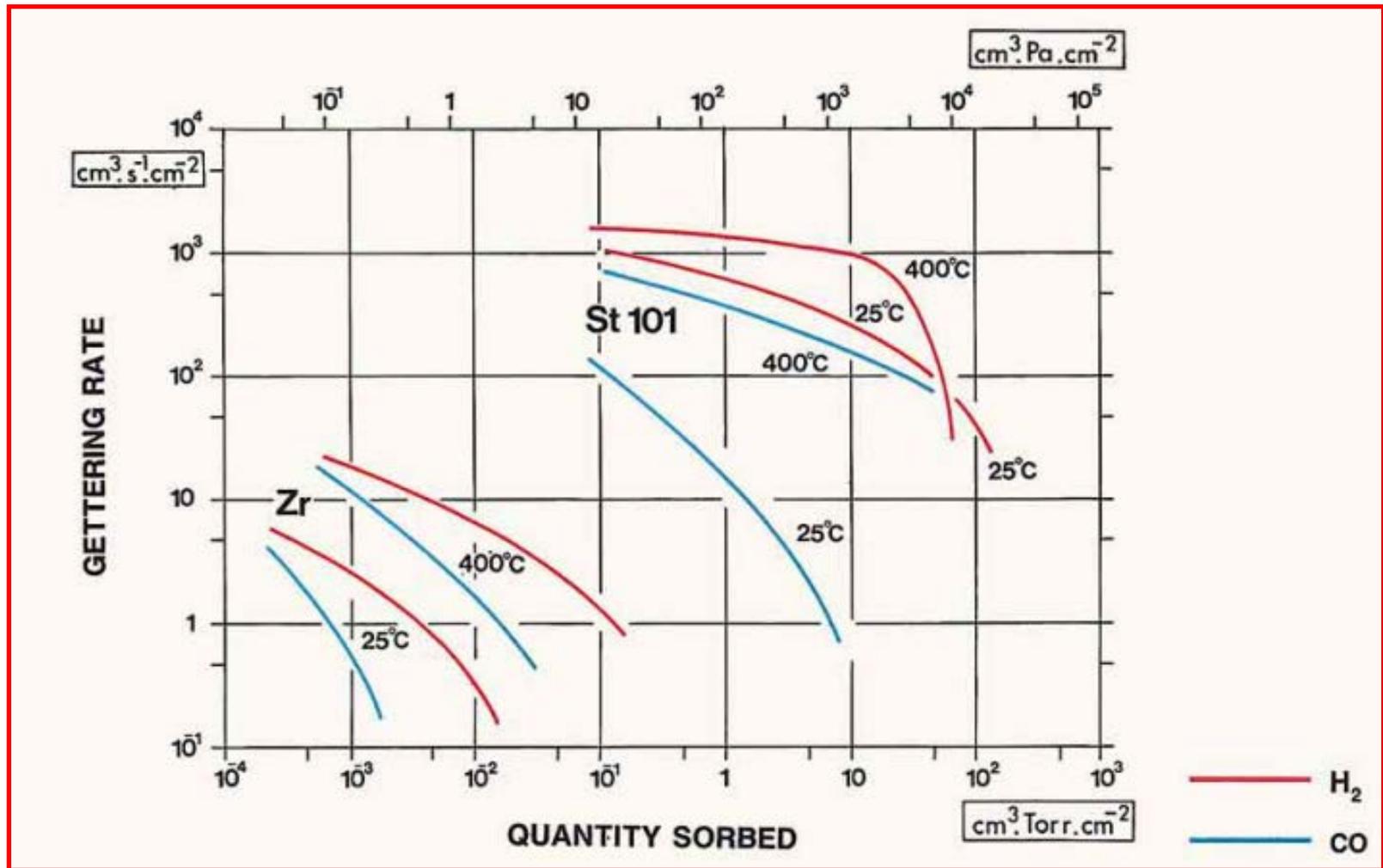


ST 101 Alloy Activation Efficiency

Ref. SAES Getters



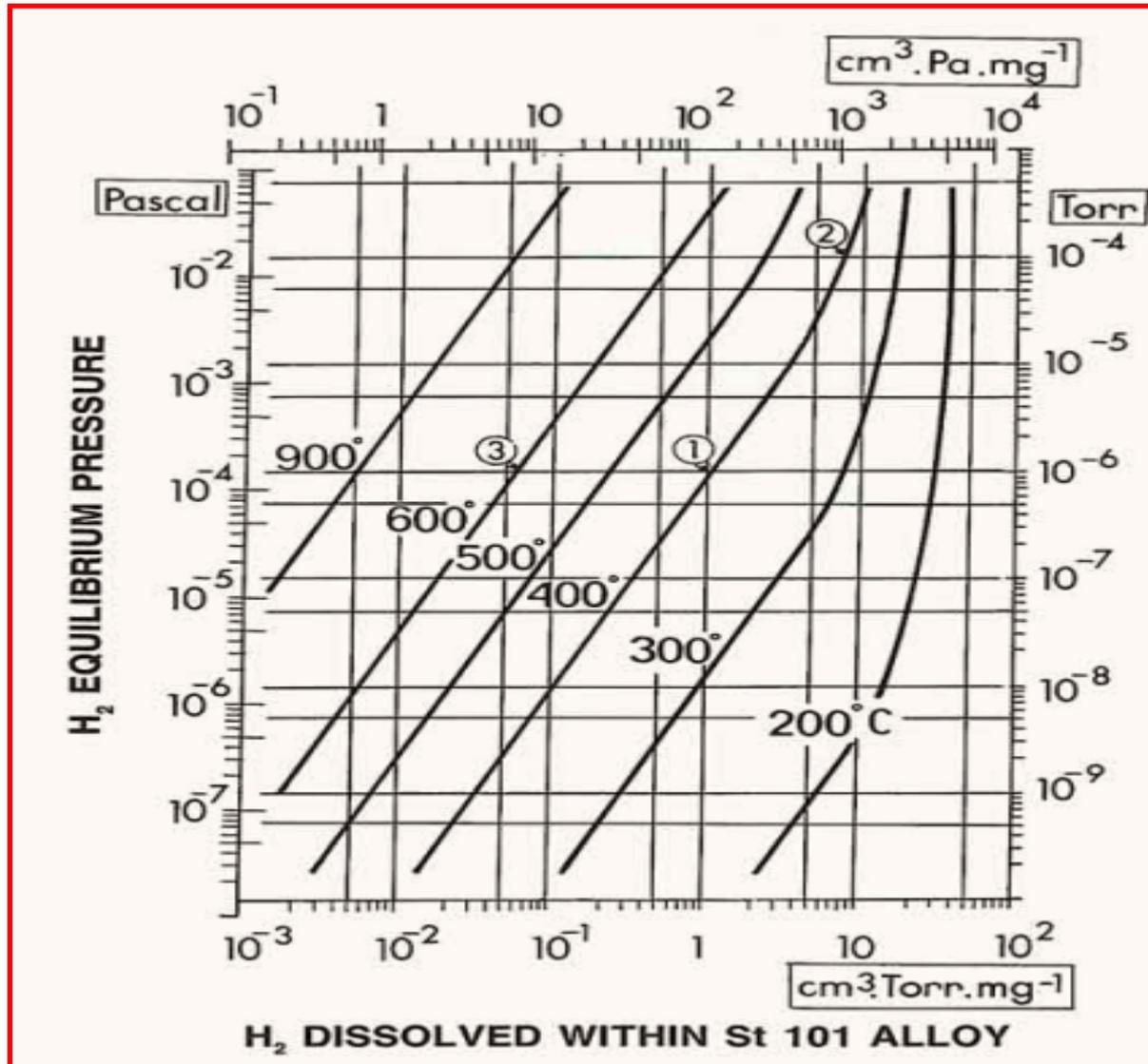
# SAES ST101<sup>®</sup> NEG – Pumping



Ref. SAES Getters



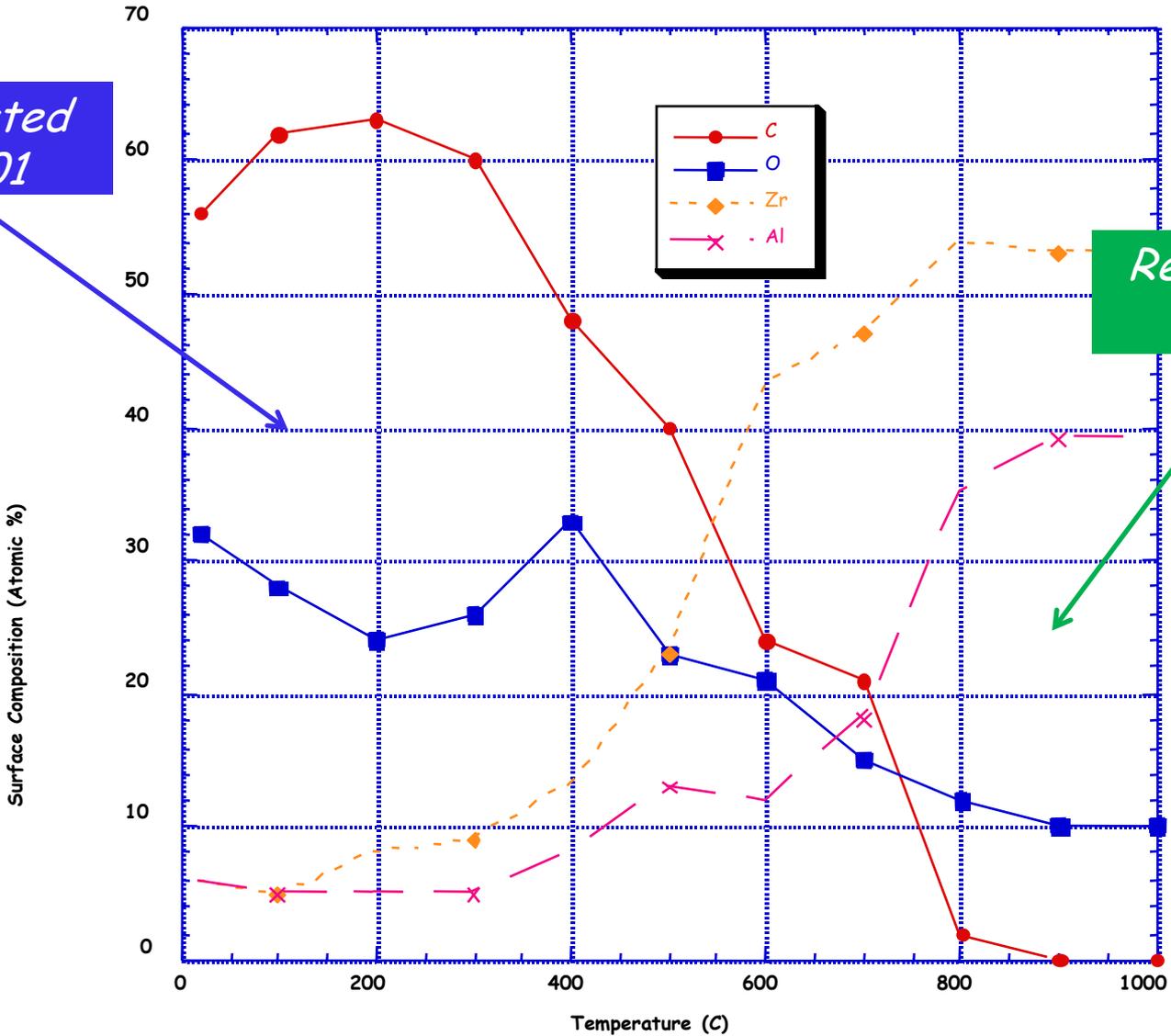
# SAES ST101<sup>®</sup> NEG – Hydrogen Solubility



# ST101 Surface Composition vs. Temperature

Saturated ST101

Regenerated ST101



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<sub>2</sub>, H<sub>2</sub>O, N<sub>2</sub>, and O<sub>2</sub> at high rates.
- ❖ ST707 has much lower activation temperature.

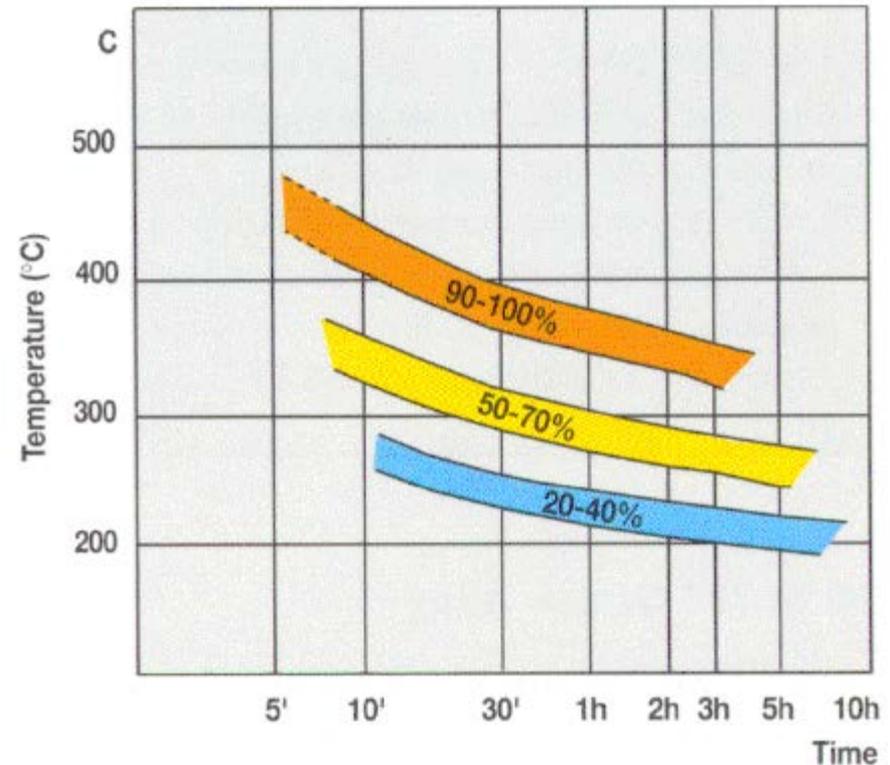
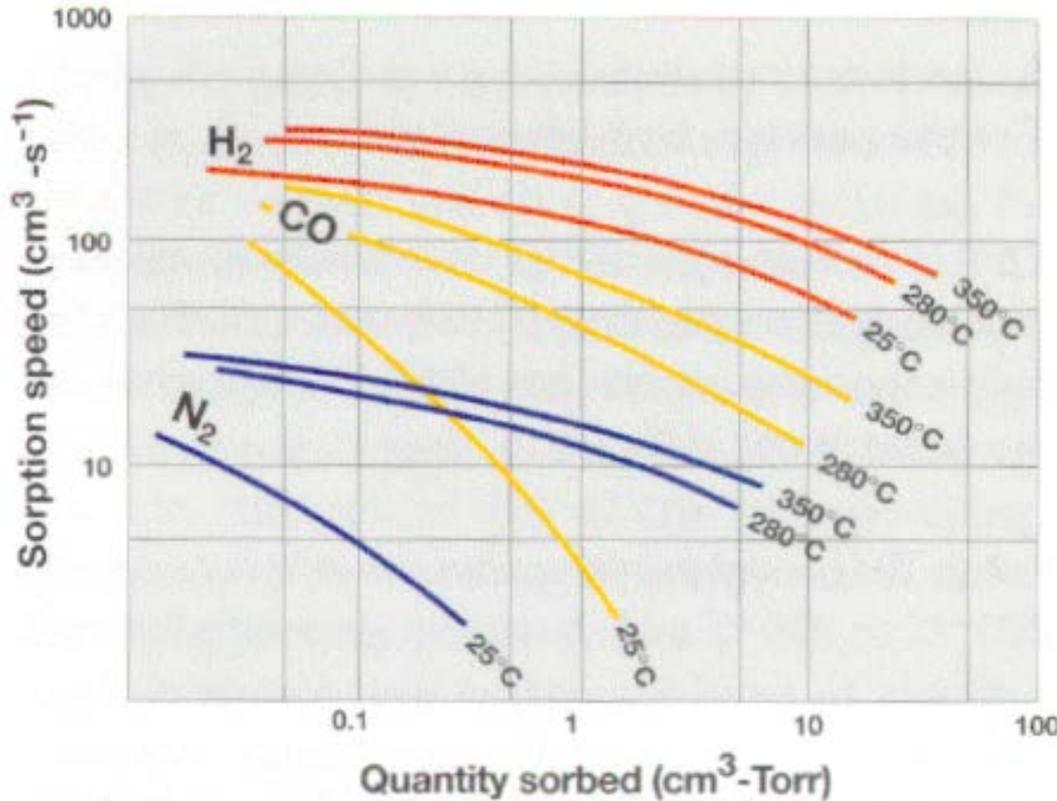


Fig. 1. Activation conditions and gettering efficiency of St 707



# SAES ST707® NEG Pumping Performance



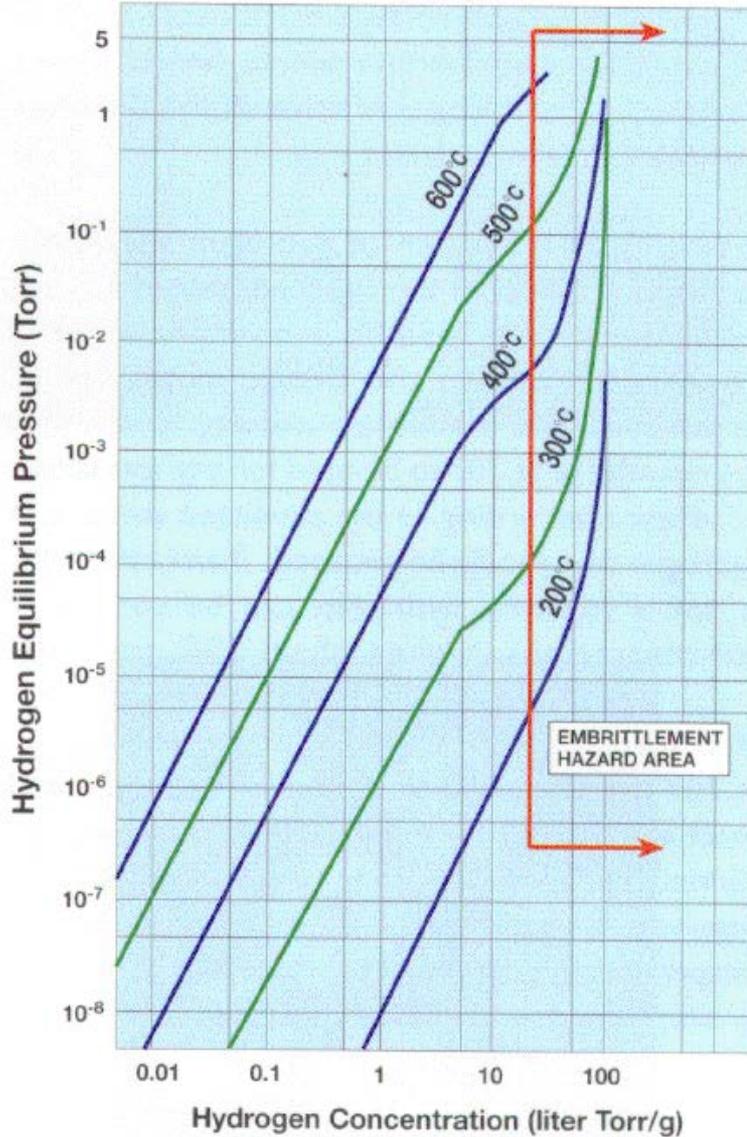
St 707 powder alloy: 100mg

Geometric surface: 50 mm<sup>2</sup>

Activation: 450°C for 10 min.

Sorption: At the indicated temperatures





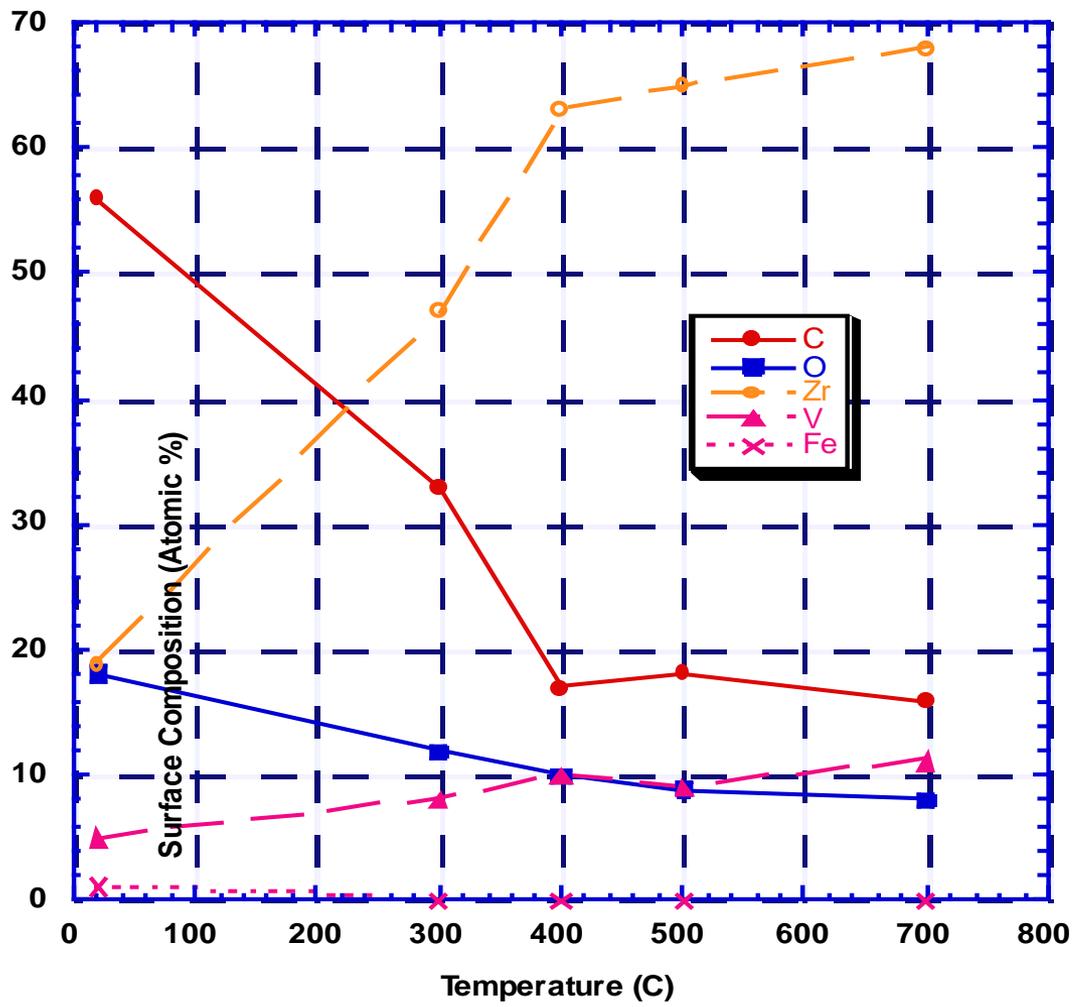
## Sievert's Law for ST707

$$\text{Log } P = 4.8 + 2 \log Q - \frac{6116}{T}$$

- $P$  = H<sub>2</sub> equilibrium pressure (torr)
- $Q$  = H<sub>2</sub> concentration (torr-l/g)
- $T$  = Temperature in °K



# ST707 Surface Composition vs. Temperature



# Other SAES NEG Alloys

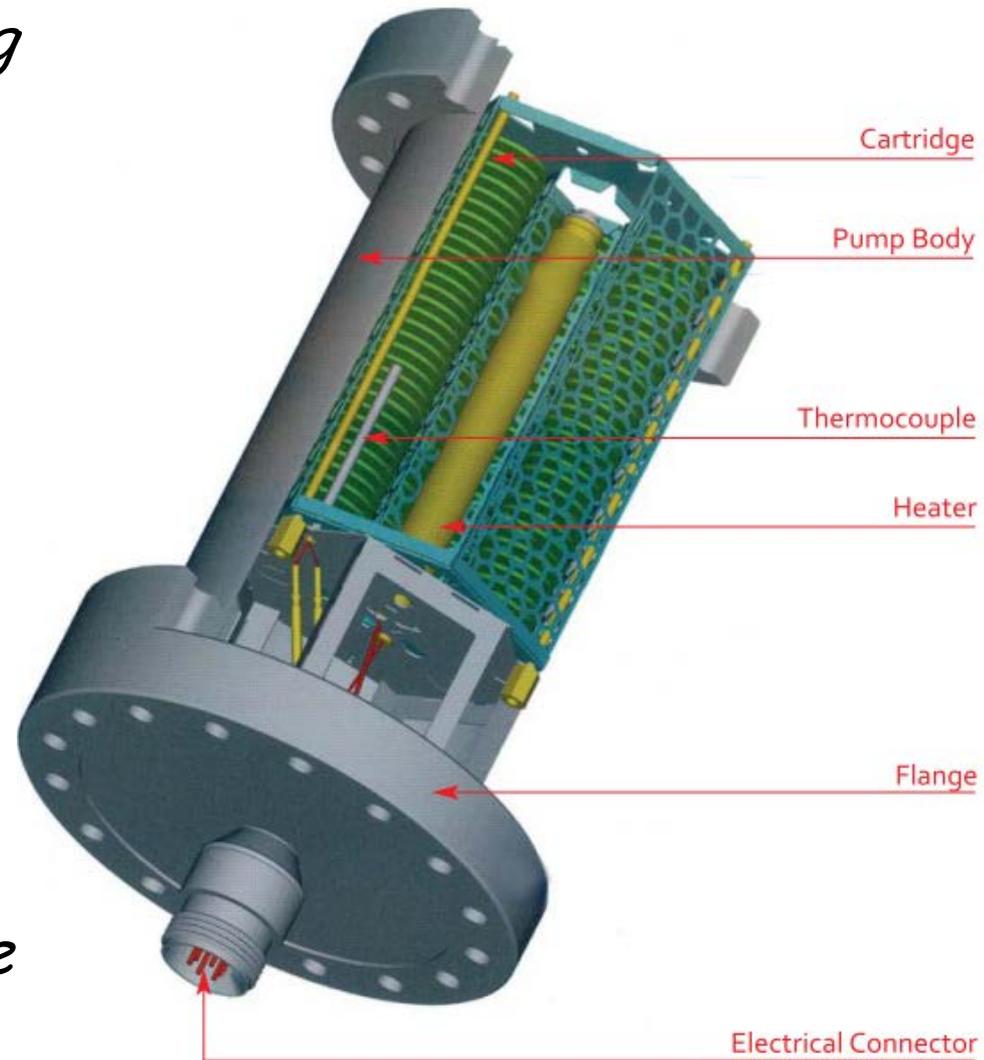


- ❑ *ST 172 - ST707 + Zr.*  
One of most used alloys, used in SAES' CapaciTorr and NexTorr pump series.
- ❑ *ST175 - Ti and Mo powder mixture, sintered form.*
- ❑ *ST185 - Ti-V alloy (obsolete !)*
- ❑ *ZAO - a new Zr-based alloys, lower gas emissions, higher capacity*

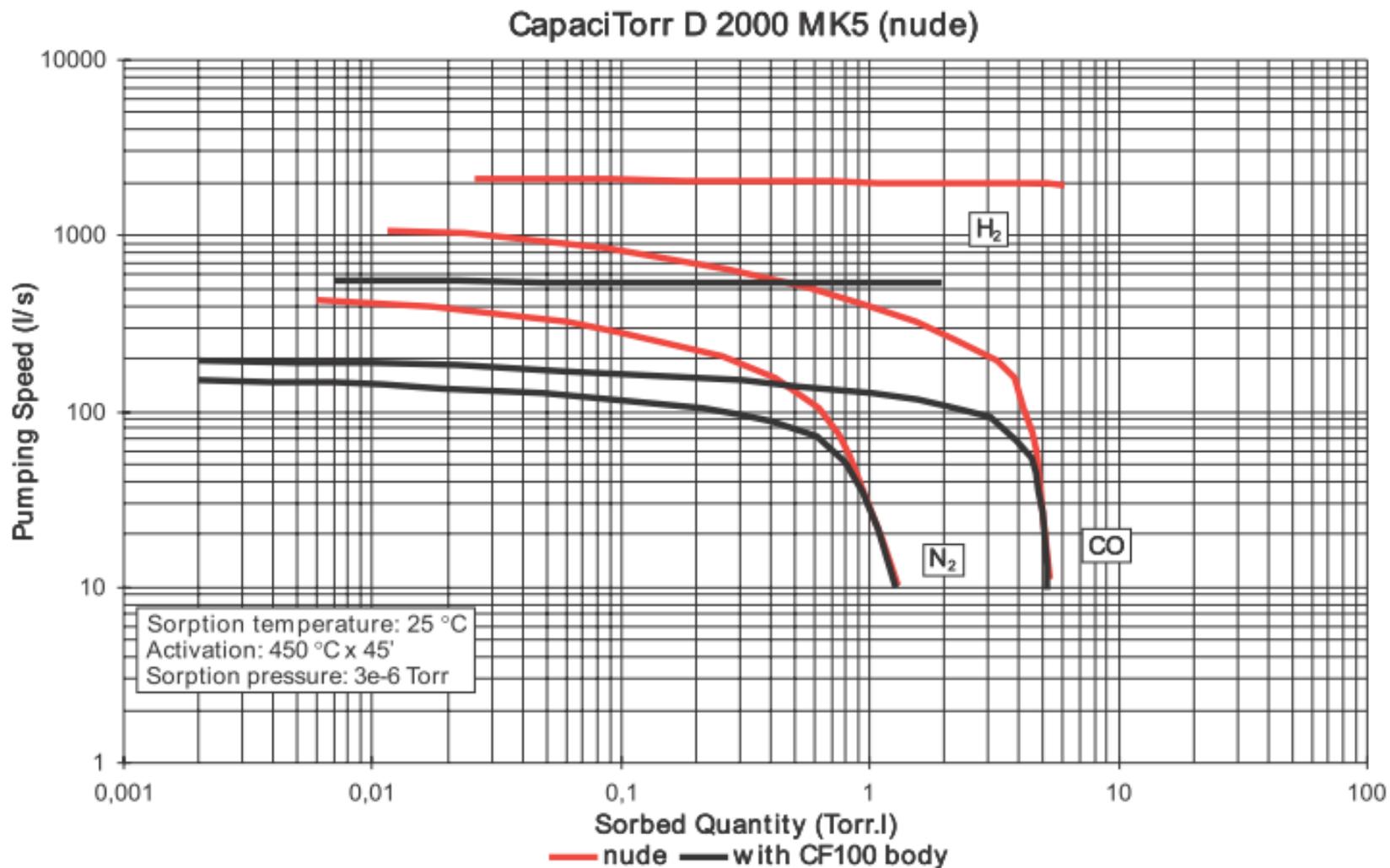


# Examples of SAES Getter's Available NEG Pumps

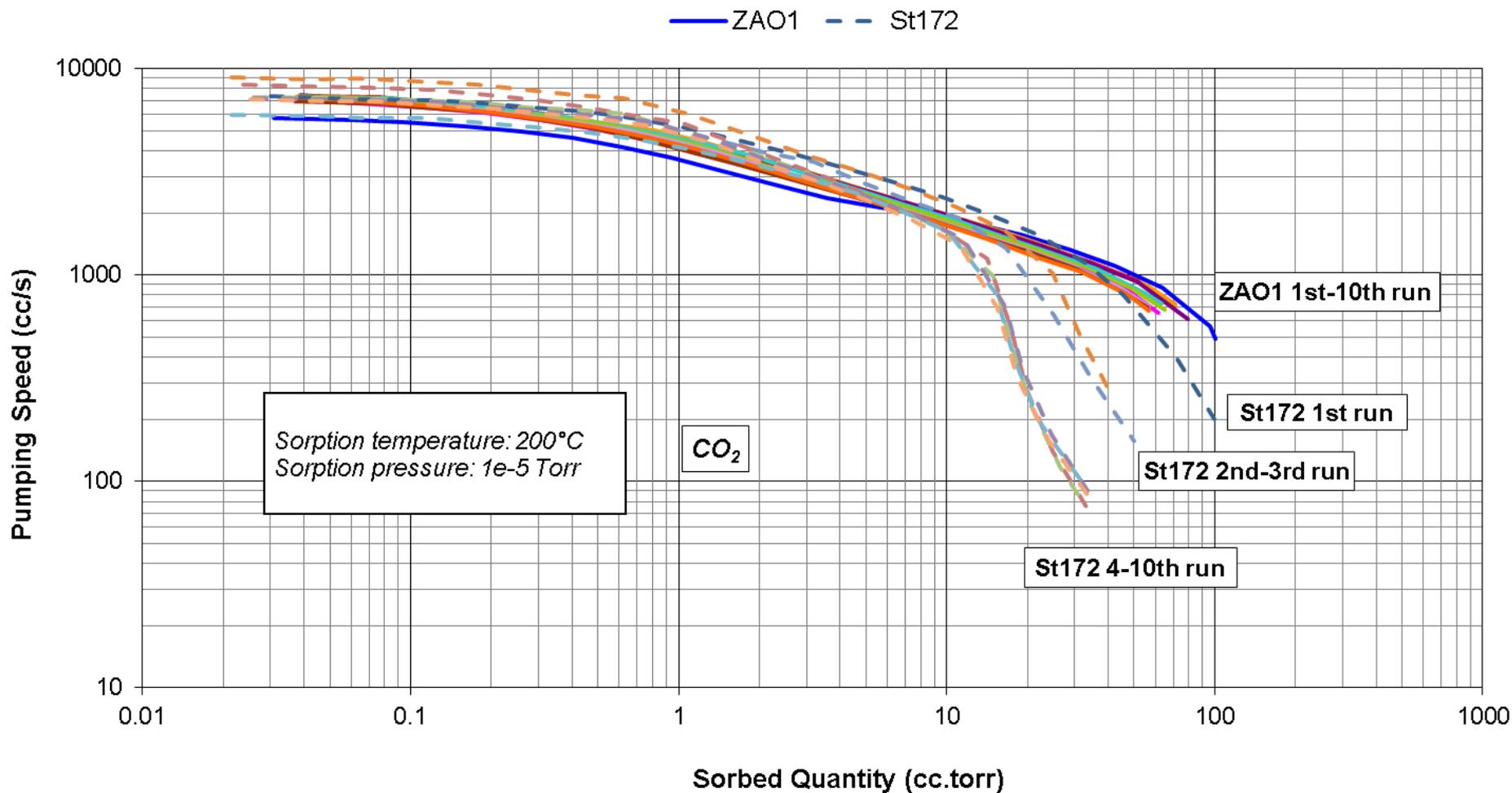
- ❑ *Complete compact pumping system, with matching controller for easy activation*
- ❑ *NEG materials: st172 sintered blades/disks*
- ❑ *Pump sizes from 50 l/s to 2000 l/s, for H<sub>2</sub>*
- ❑ *For large sizes, the NEG cartridges are replaceable*



# CapaciTorr® Pumping Performance



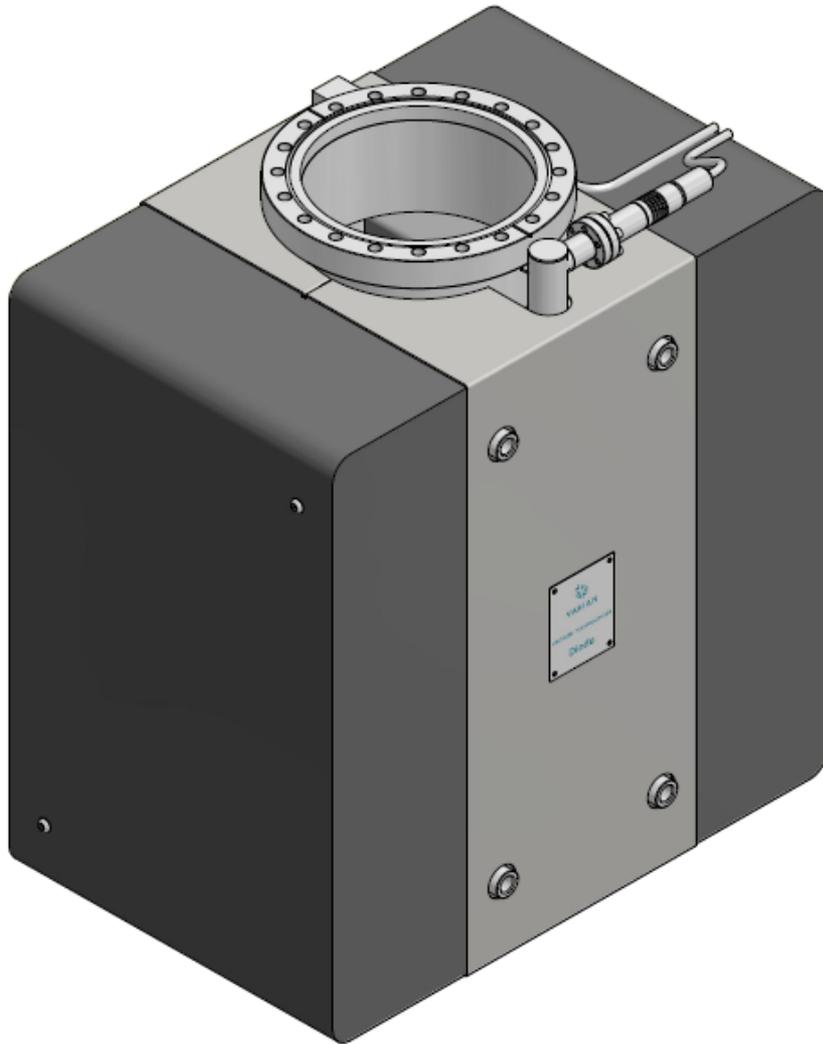
# CapaciTorr® st172 vs ZAO HV



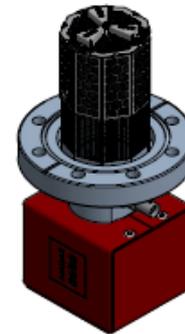
F.Siviero AVS 61<sup>st</sup>, November 2014 Baltimore



# NEG – Ion Pump Combination – NexTorr®



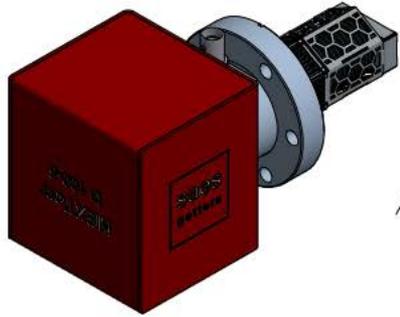
*500 l/s Vaclon Plus*



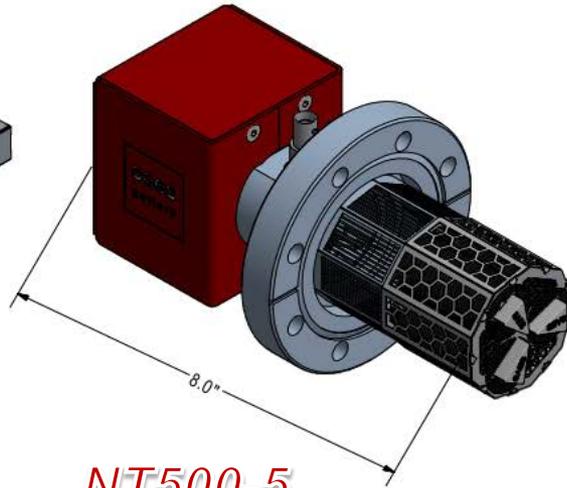
*NexTorr D500-5*



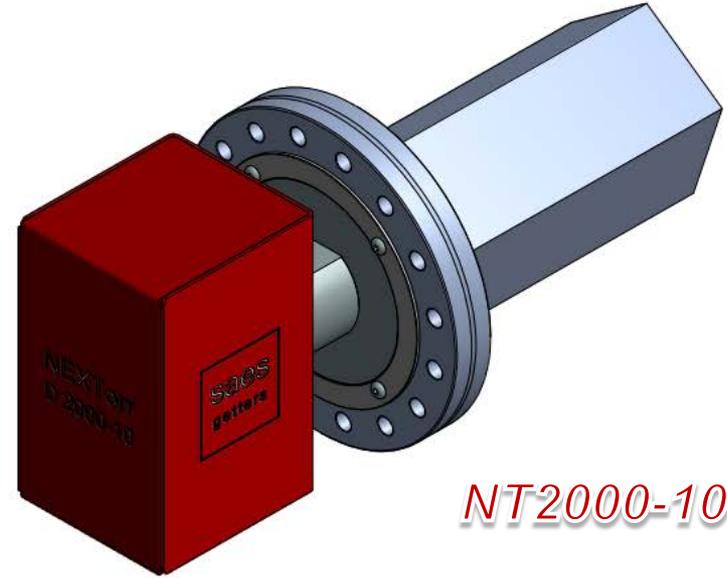
# Available NexTorr<sup>®</sup> Pumps from SAES



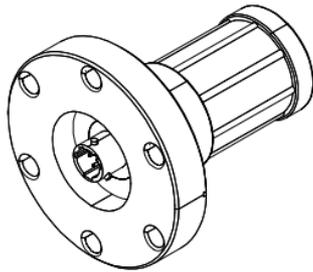
*NT100-5*



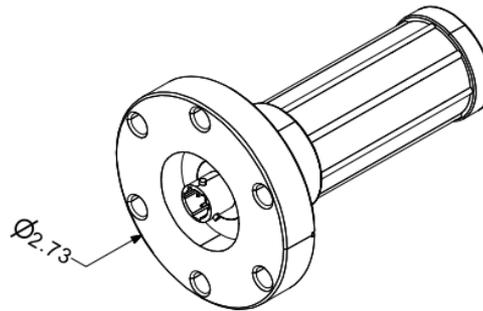
*NT500-5*



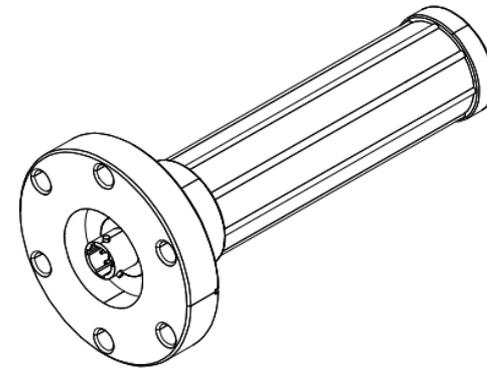
*NT2000-10*



*Gamma N100*



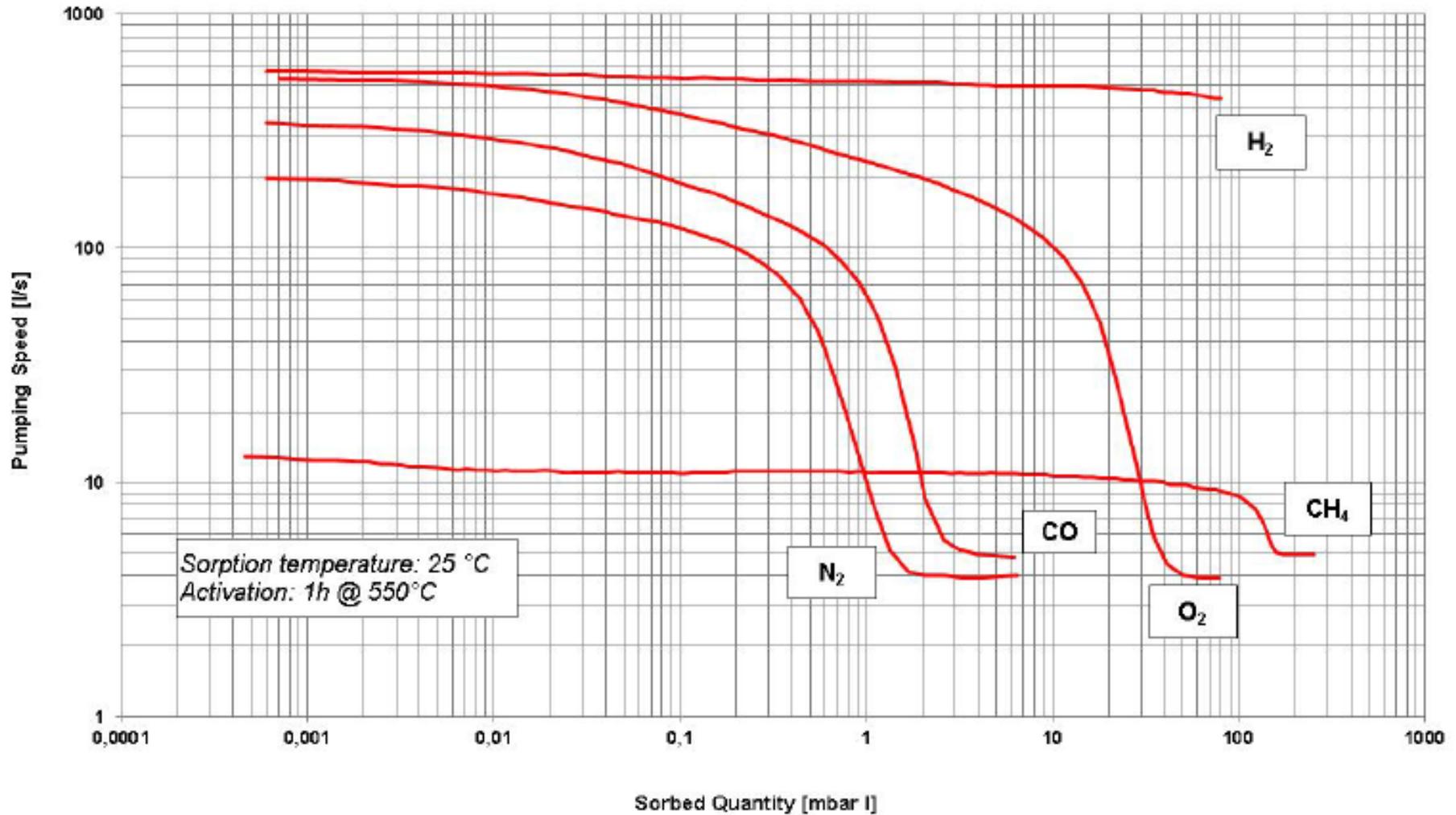
*Gamma N200*



*Gamma N400*



# Pumping Performance – NexTorr®



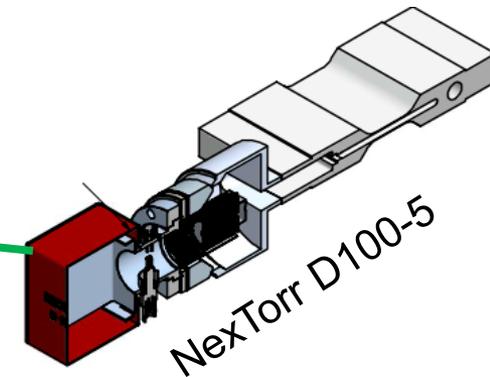
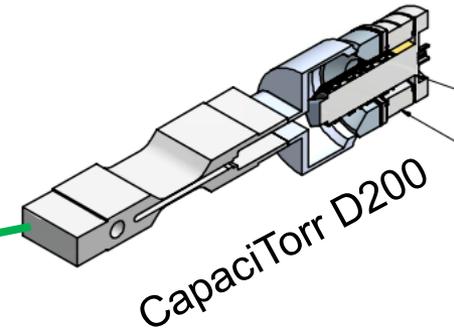
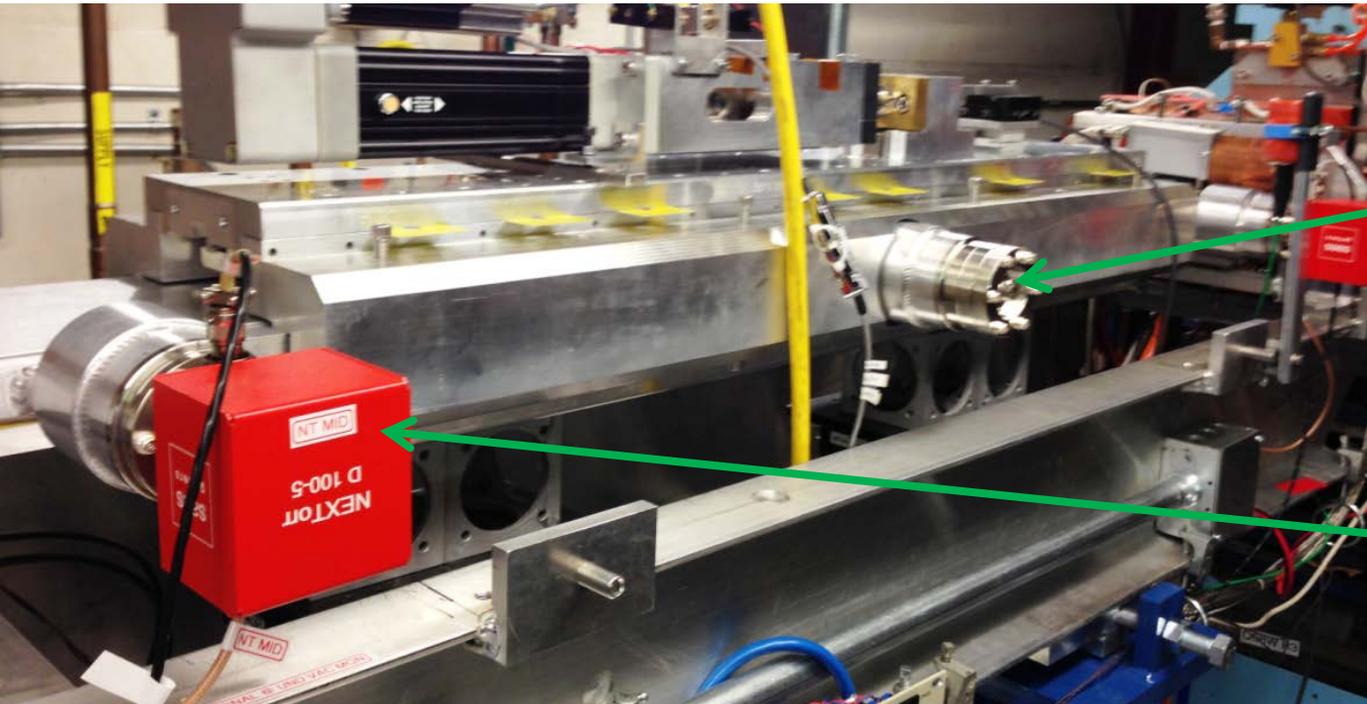
# Main Technical Parameters – NexTorr® D500-5



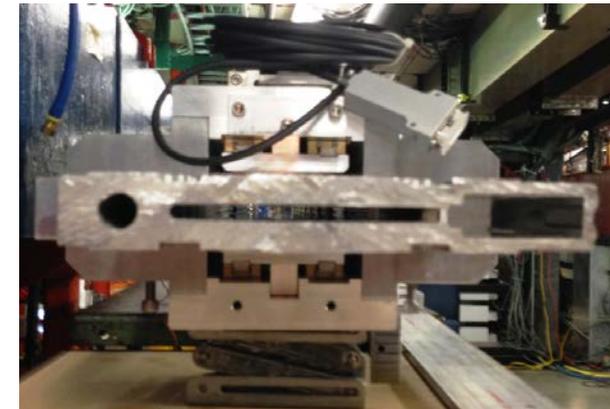
Initial pumping speed (l/s)	Gas	NEG activated	NEG saturated
	O <sub>2</sub>	500	4
	H <sub>2</sub>	500	6
	CO	340	5
	N <sub>2</sub>	200	4
	CH <sub>4</sub>	13	5
	Argon <sup>1</sup>	6	6
Sorption capacity (Torr·l)	Gas	Single run capacity <sup>2</sup>	Total capacity <sup>3</sup>
	O <sub>2</sub>	17	>1500
	H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



# CapaciTorr and NexTorr Application Examples



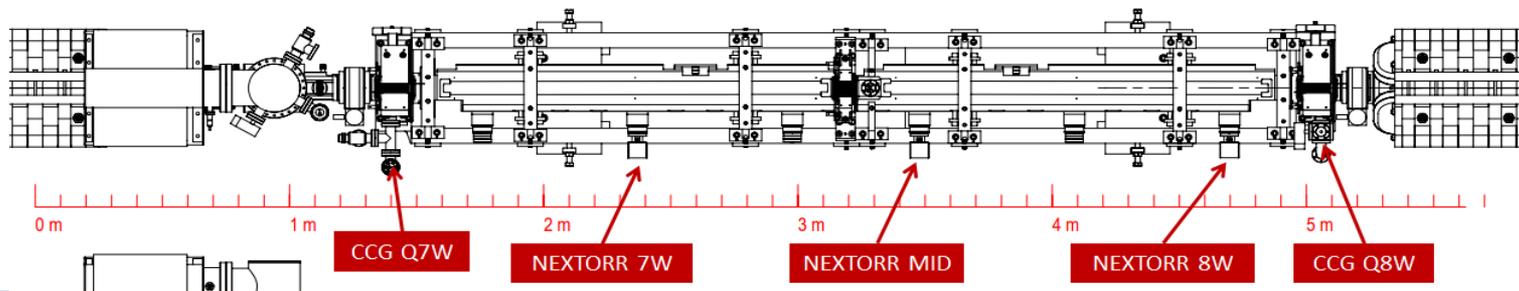
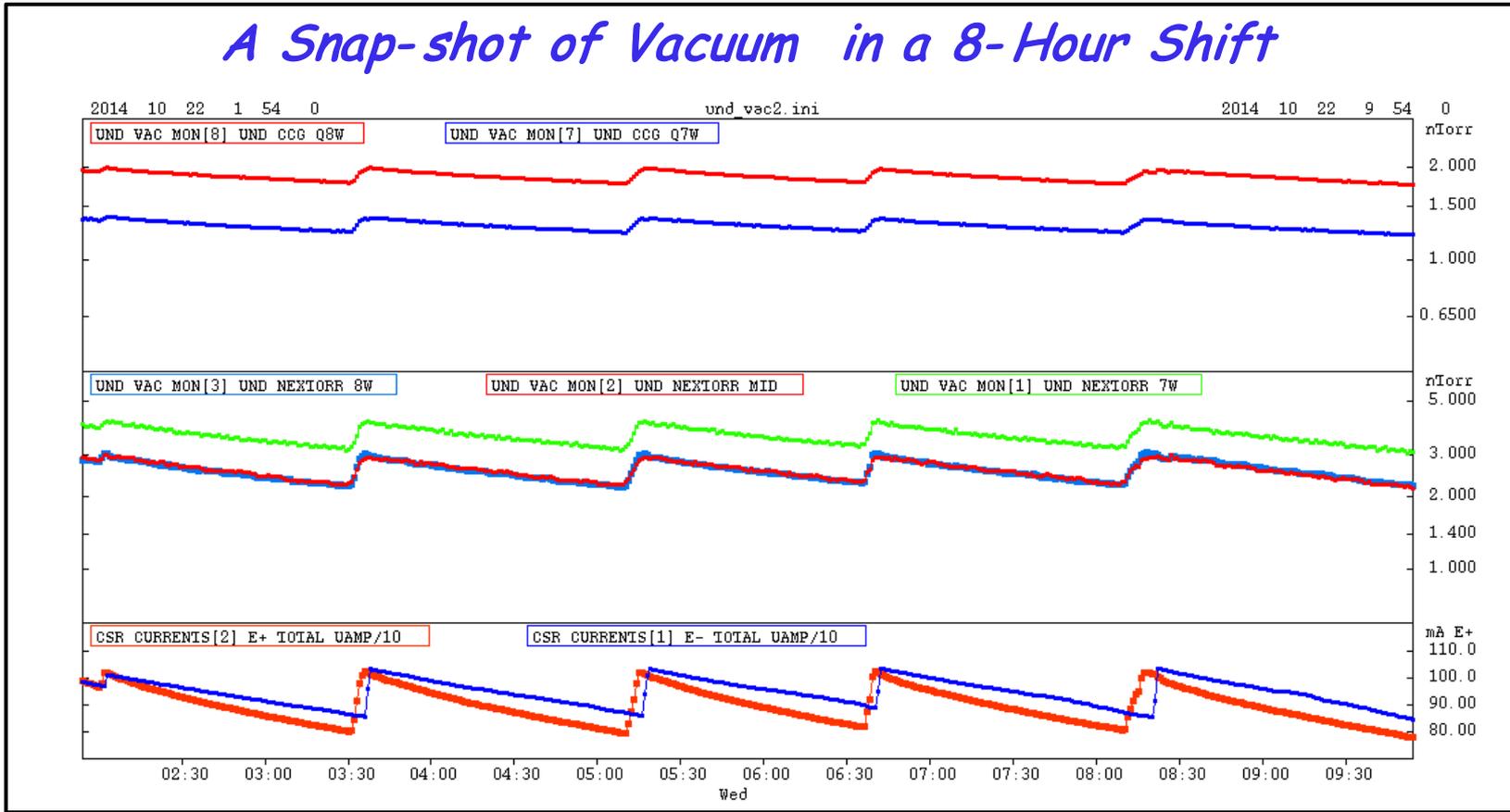
- ❑ At a recent upgrade project for CESR/CHESS, a 3.5-m long undulator vacuum chamber was designed and installed to accommodate a pair of Cornell Compact Undulator (CCU)
- ❑ Three NexTorr D100-5 and three CapaciTorr D200 pumps were used for vacuum pumping, taking full advantage of light weight of the NEG's.

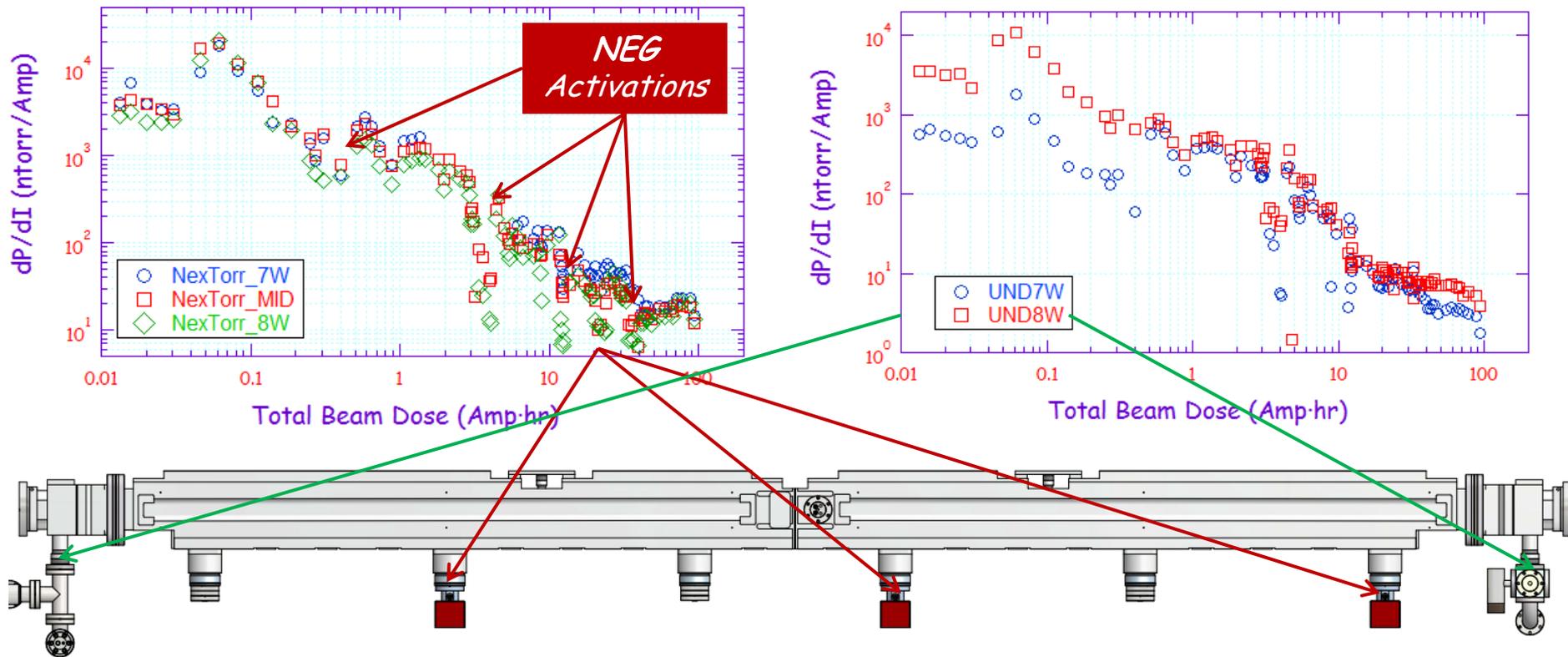


# CapaciTorr and NexTorr Performance at CCU – 1



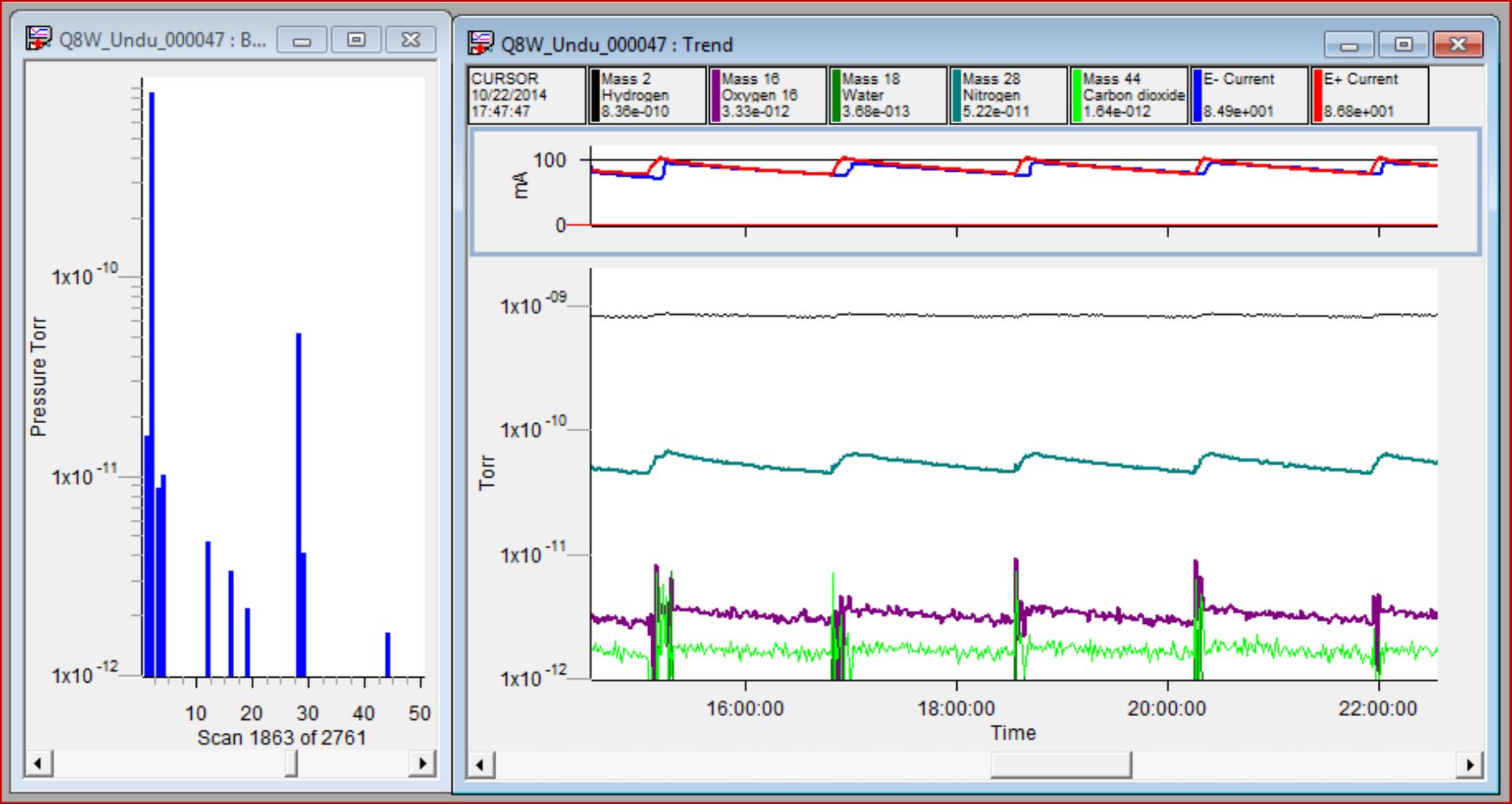
## A Snap-shot of Vacuum in a 8-Hour Shift





- *The chamber vacuum conditioning are progressing as expected.*
- *The NEGs were re-activated four times during regularly scheduled tunnel access, to keep optimum pumping.*
- *$dP/dI$  values indicating  $\eta_{PID}$  approaches  $10^{-6}$  mol/ph @~100 Amp-hr beam dose.*

# CapaciTorr and NexTorr Performance at CCU – 3

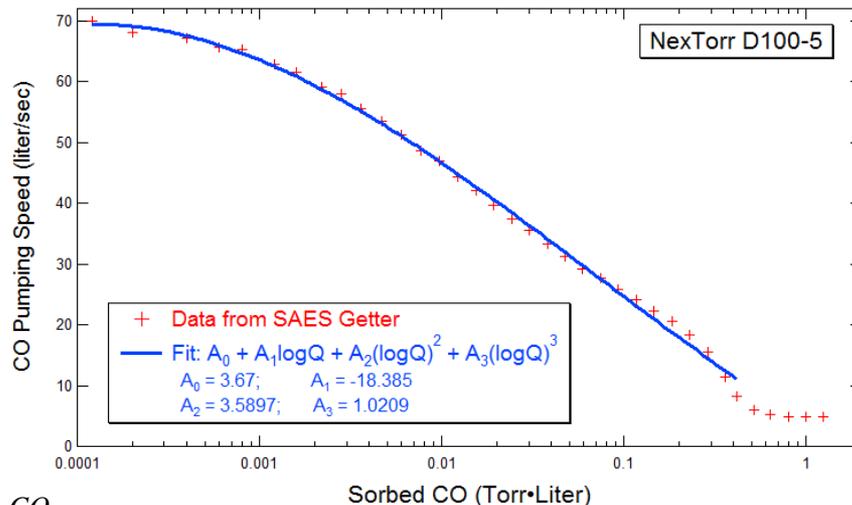


*RGA data showed hydrogen being the dominant gas, with trace of other gases (probably produced by RGA filament)*



# Estimate NEG Pumping 'on-the-Fly' – 1

- ❑ In many systems, full vacuum instrumentation is not always possible (lack of space!), so one has to have ways in evaluating NEG's saturation status.
- ❑ For NexTorr pumps, SIP current may be used for evaluating sorbed quantity of a NEG, though gas composition info is needed.



- ❑ Estimated sorbed CO-equivalent load  $Q_t^{CO}$ :

$$Q_t^{CO} = \sum_i^t \alpha_i^{CO} P_i^{NT_m} \times S_{i-1}^{NT_m} \times \Delta t$$

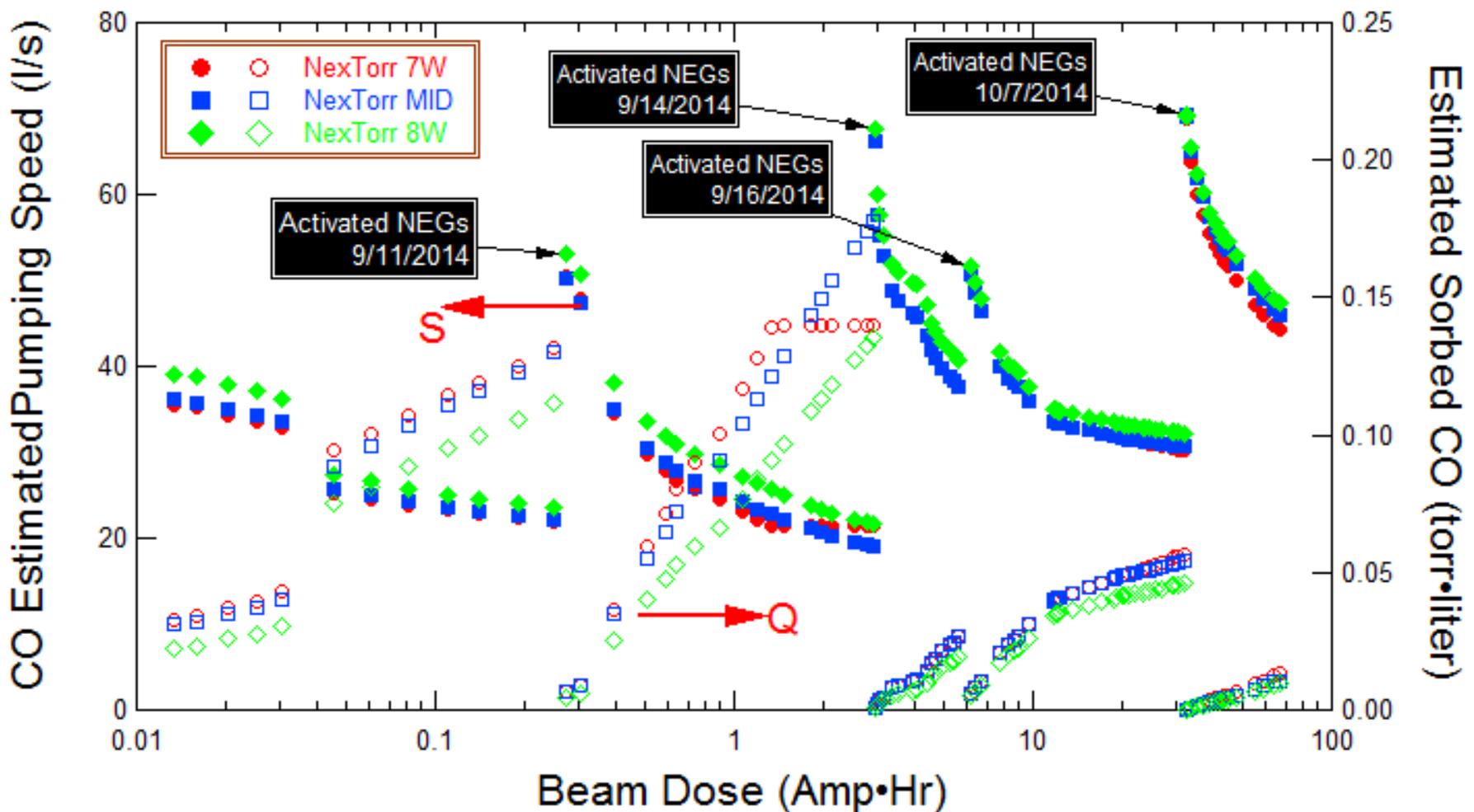
where:  $P_i^{NT_m}$  is recorded pressure at  $t_i$  from NexTorr  $m$  ( $m=Q7W, MID$  and  $Q8W$ );

$\alpha_i^{CO}$  is estimated CO-equivalent percentage from RGA data;

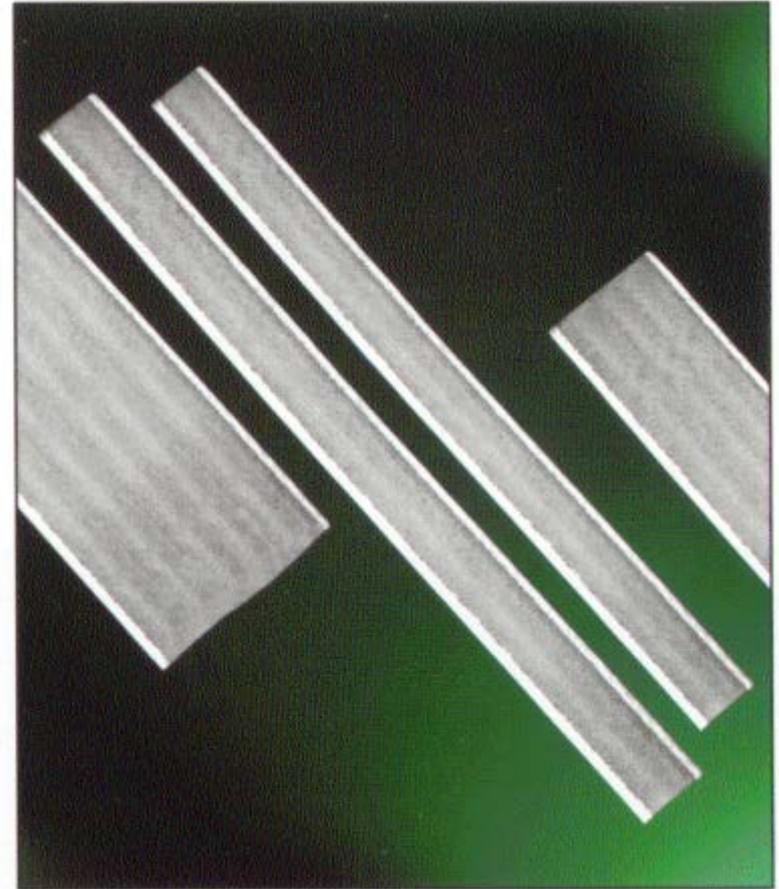
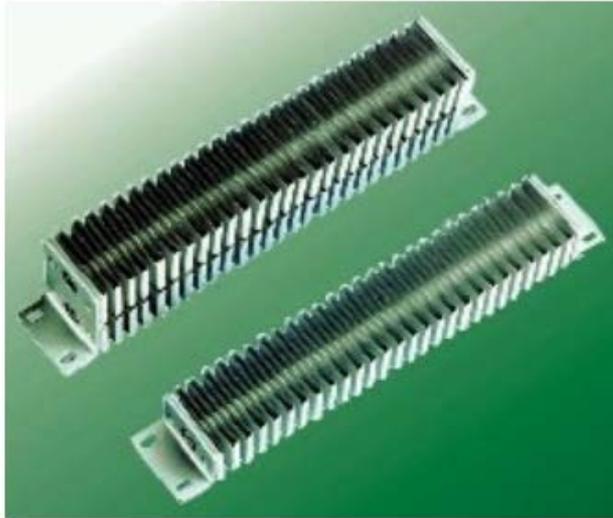
$S_{i-1}^{NT_m}$  is calculated CO pumping speed using fitted formula above;

$\Delta t = 60$  seconds for data from 1-minute logit files

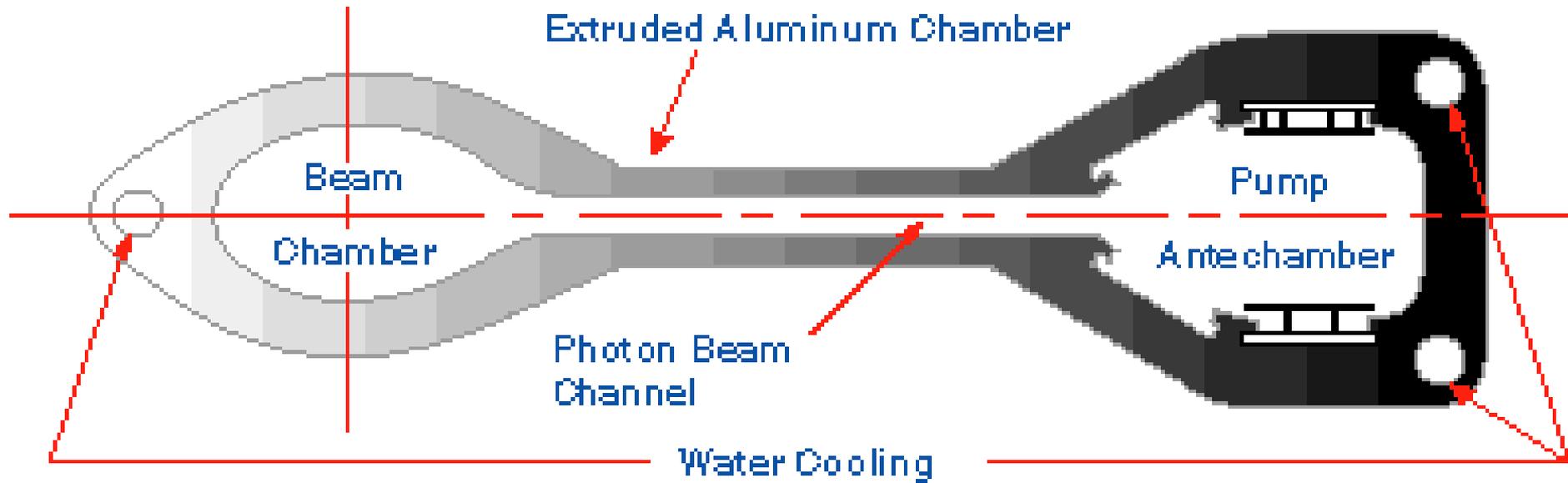
# Estimate NEG Pumping 'on-the-Fly' – 2



# Other NEG forms – Build your own pumps



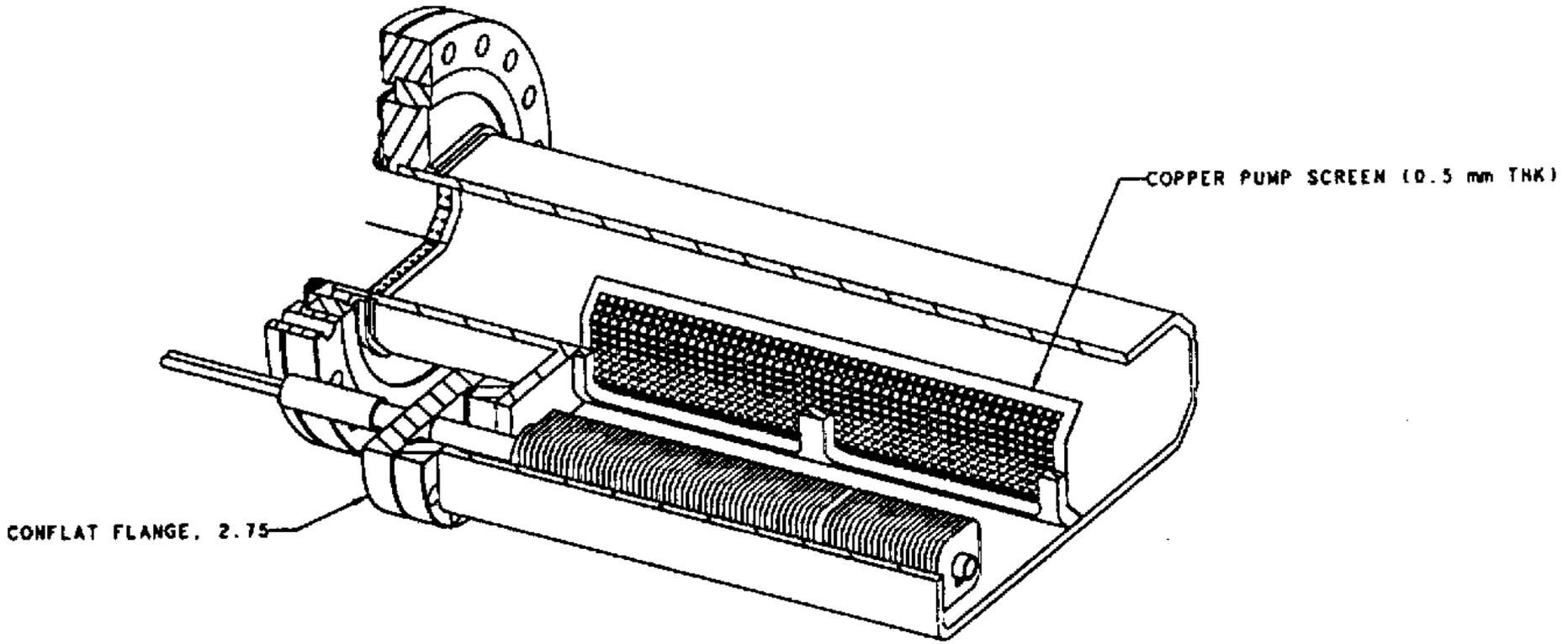
# Distributed Pumping with NEG strips



APS Beampipe with NEG strips



# LLNL NEG Pump in a PEP-II Vacuum Chamber



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**



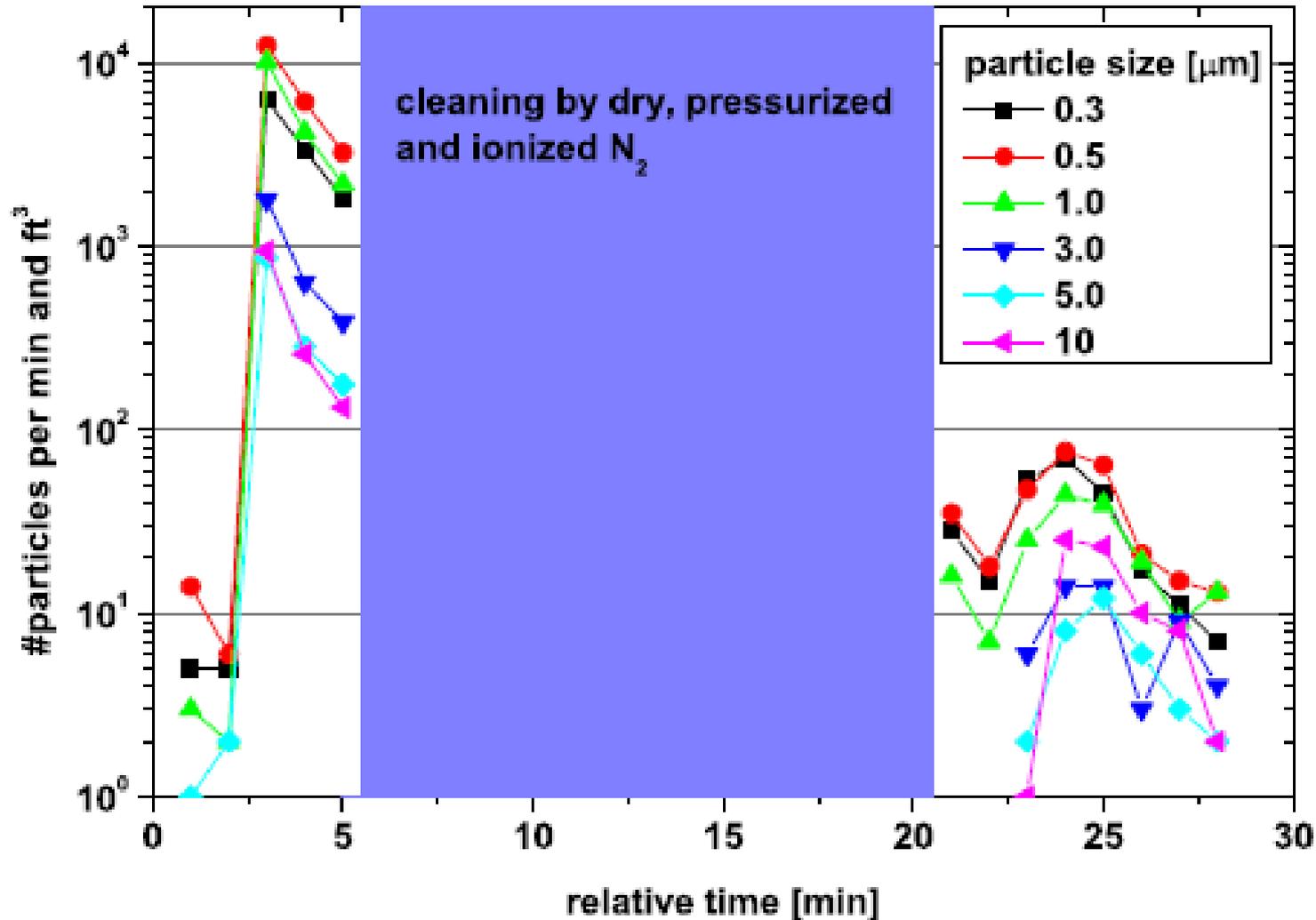
**Ion Pump with NEG cartridge**



- ❑ *Most NEG pumping elements are formed through powder metallurgy, either through cold-press or sintering processes. Thus the NEGs are prone to particulate creation, if not treated.*
- ❑ *Particulates creation from NEGs can be a major concern for many UHV systems, such as superconductivity RF cavities, HV DC photon-cathode guns, etc.*
- ❑ *Strips with cold-press NEG materials are mechanically less stable than sintered disks/blades. So careful placement consideration should be taken in using these NEG elements.*
- ❑ *NEG pumps using sintered materials (such as NexTorr, CapaciTorr) can be cleaned to achieve clean-room compatibility. The proven cleaning methods include N<sub>2</sub>-blowing and solvent rinsing (in a Clean-Room condition).*
- ❑ *However, cleaned sintered NEG elements may still generate particulates during initial activations. This is currently under further investigations.*



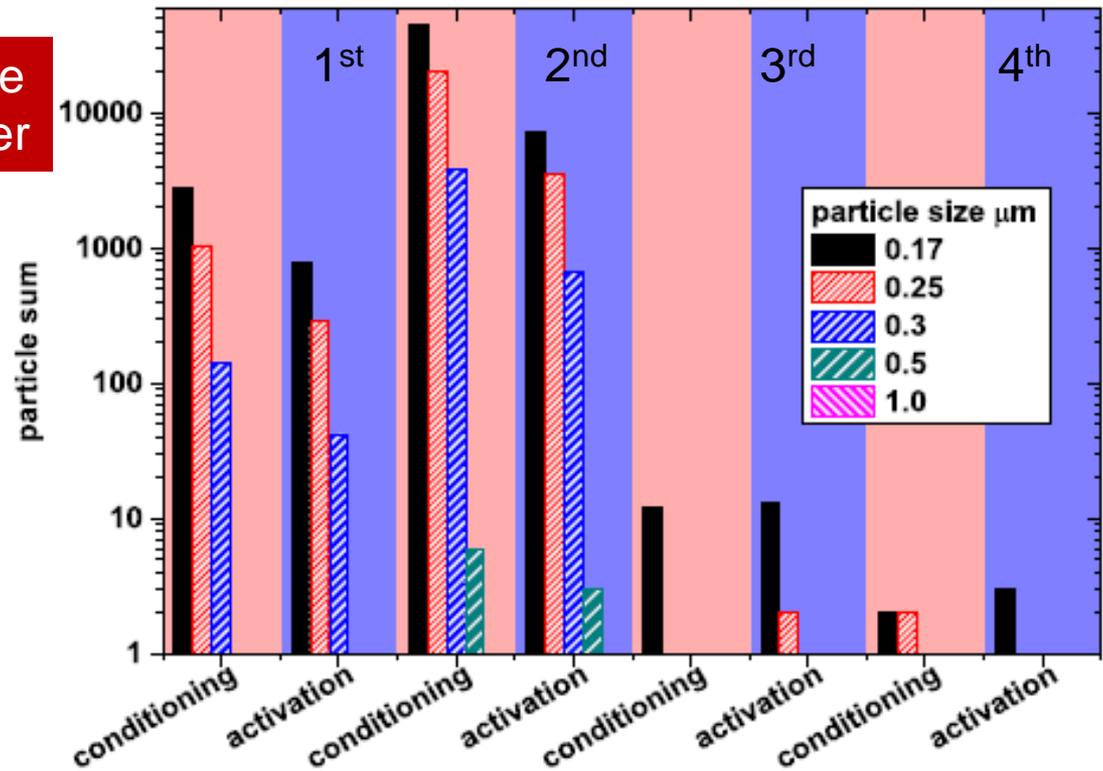
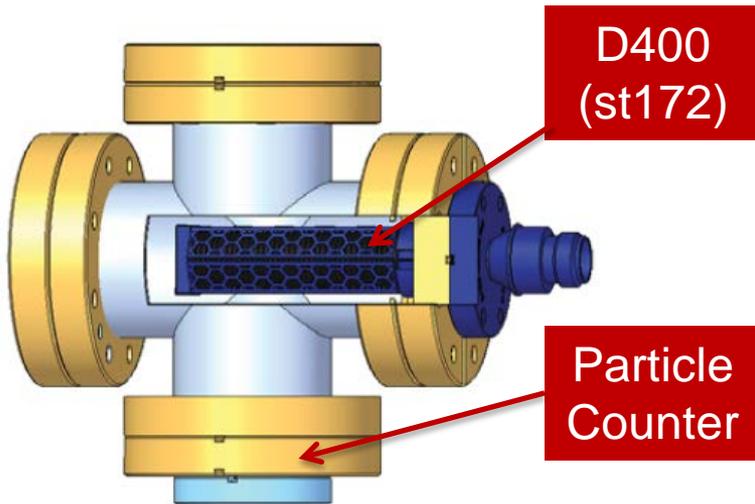
# Particles: St172® cartridges



From: S. Lederer, L. Lilje, P. Manini, F. Siviero, E. Maccallini (DESY & SAES)



# Particles: St172® cartridges



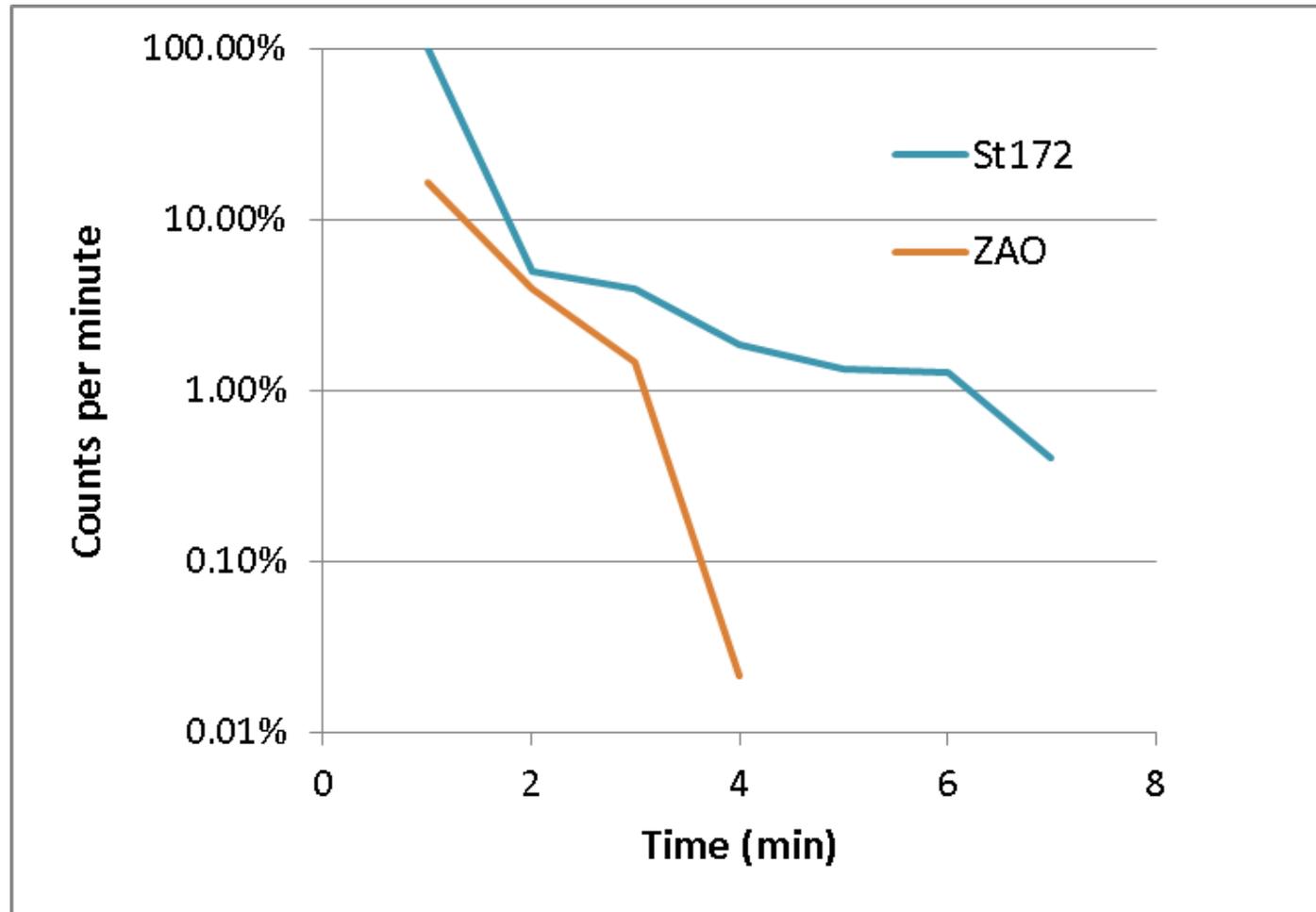
From: S. Lederer, L. Lilje, P. Manini, F. Siviero, E. Maccallini (DESY & SAES)



# Particles: St172 vs ZAO HV1



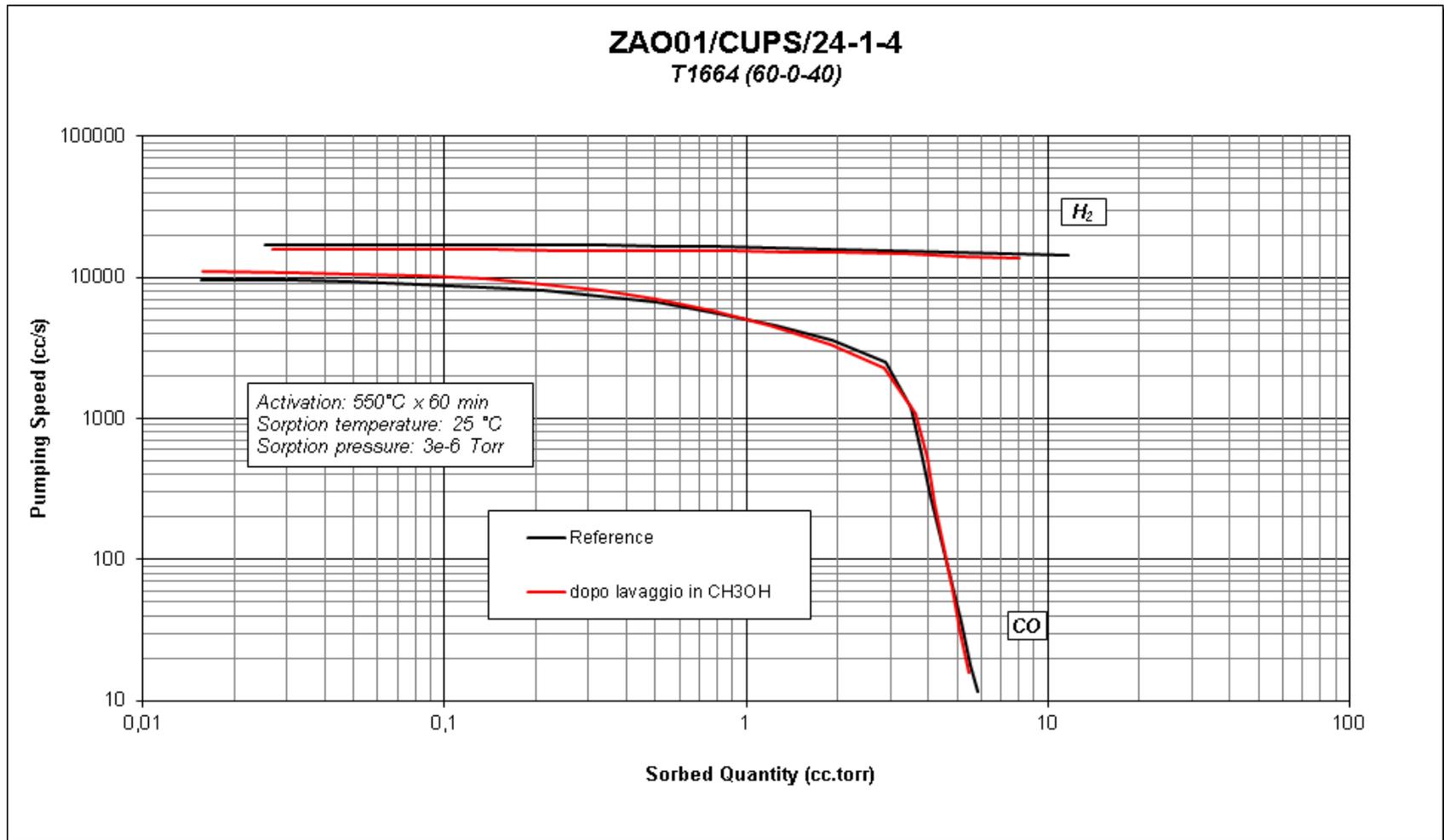
Laser particle counter under N<sub>2</sub> flushing



*Measurements at INFN Milan*



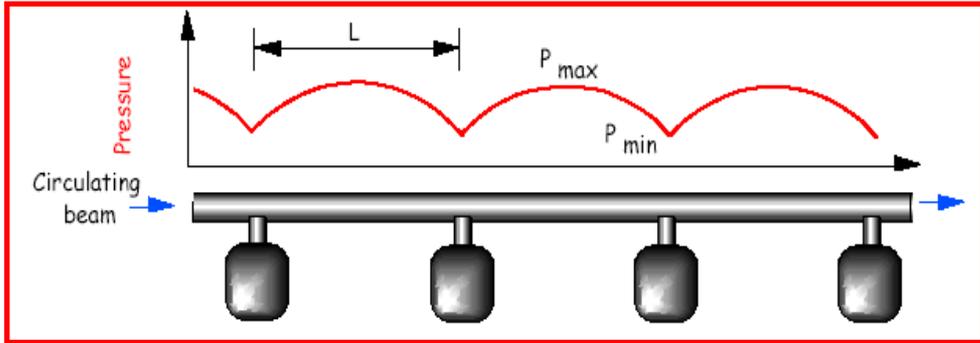
# Solvent Cleaning of Sintered NEG



A test by SAES Getters showed no effect on NEG pumping after methanol dipping

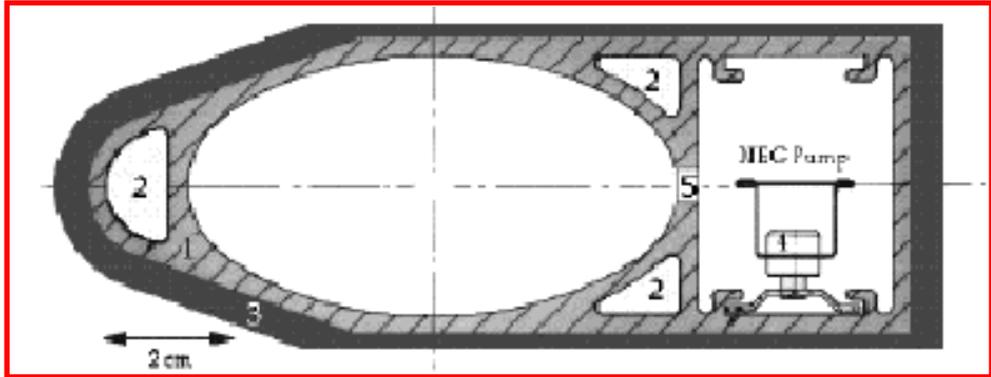


# NEG Thin Film – Integrated Pumping

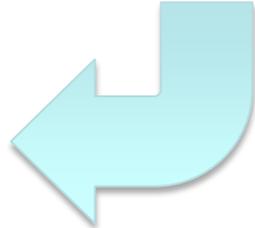
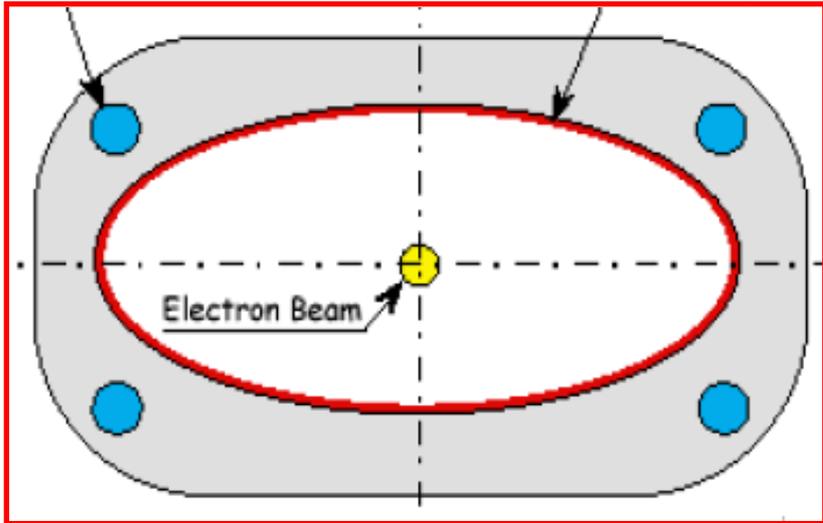


*Distributed Pumping*

*Discrete Pumping*



NEG Coating



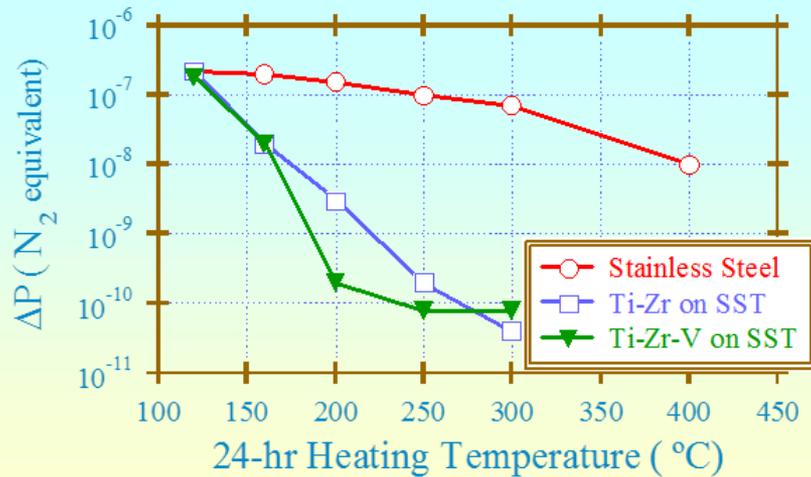
*Integrated Pumping*



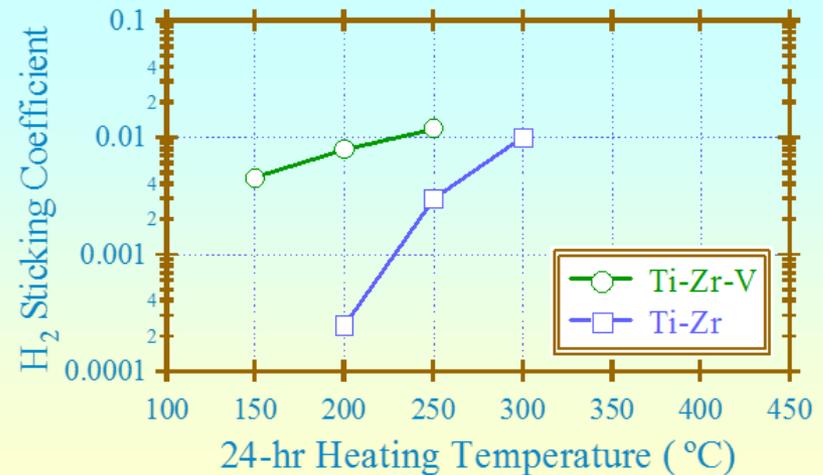
# NEG Thin Film – Benefits



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



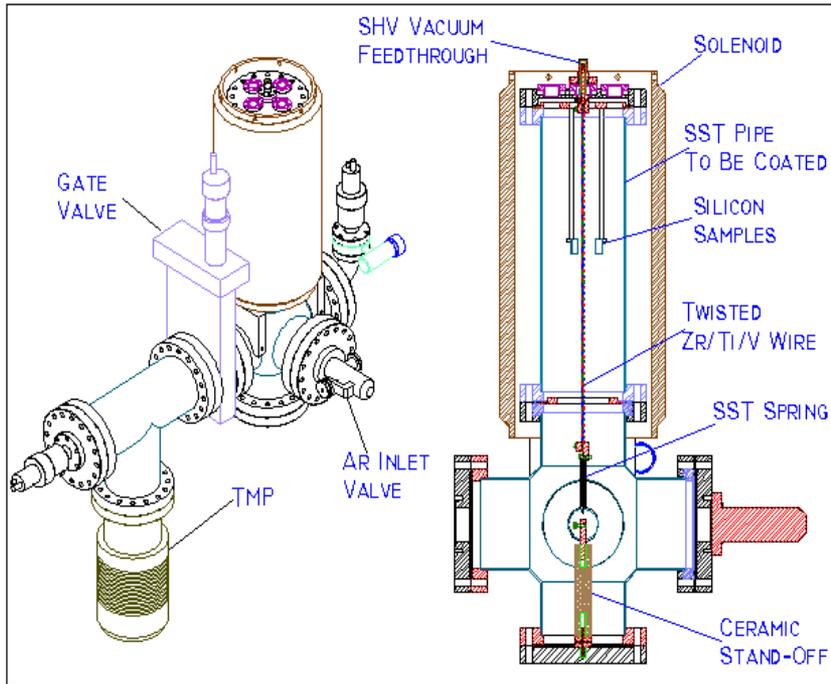
*Low Outgassing Rates*



*LOCAL pumping*



## 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



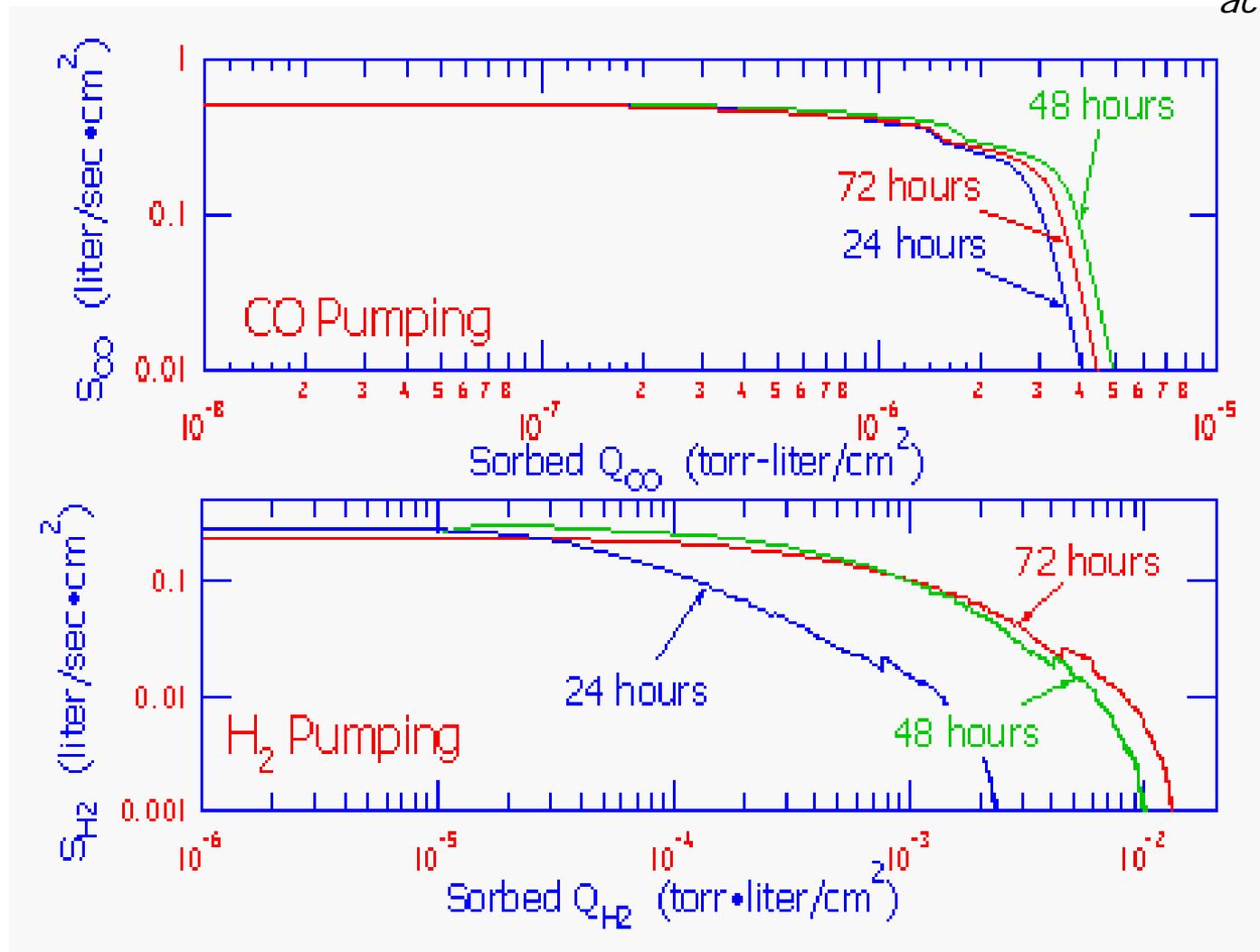
- ❑ *Most commonly deposited NEG thin films have elementary composition of  $Zr_xV_yTi_z$ , with  $x, y, z$ , close to unity.*
- ❑ *Stoichiometric balanced thin film tend to have lower activation temperature, probably due to smaller grain sizes.*
- ❑ *Pumping can be achieved at activation temperature as low as  $150^\circ\text{C}$ , though typical  $\sim 250^\circ\text{C}$ . Thus an in-situ bakeout can activate the NEG coating.*
- ❑ *Typical NEG thin film thickness:  $2\sim 4\ \mu\text{m}$ .*



# NEG Coating Pumping Performance (1)



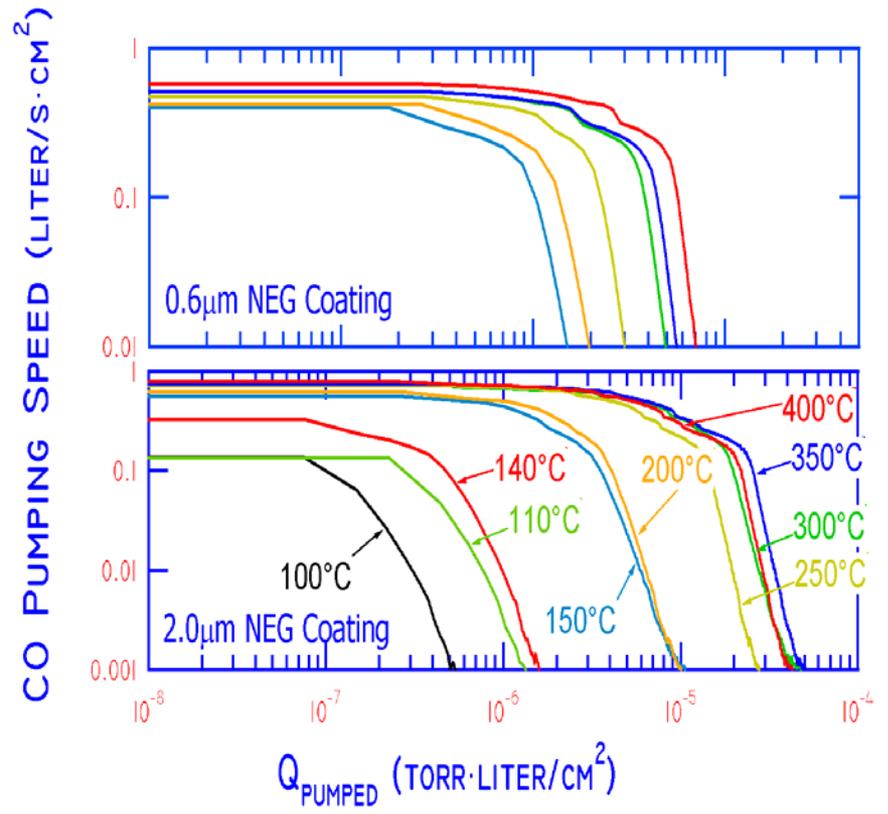
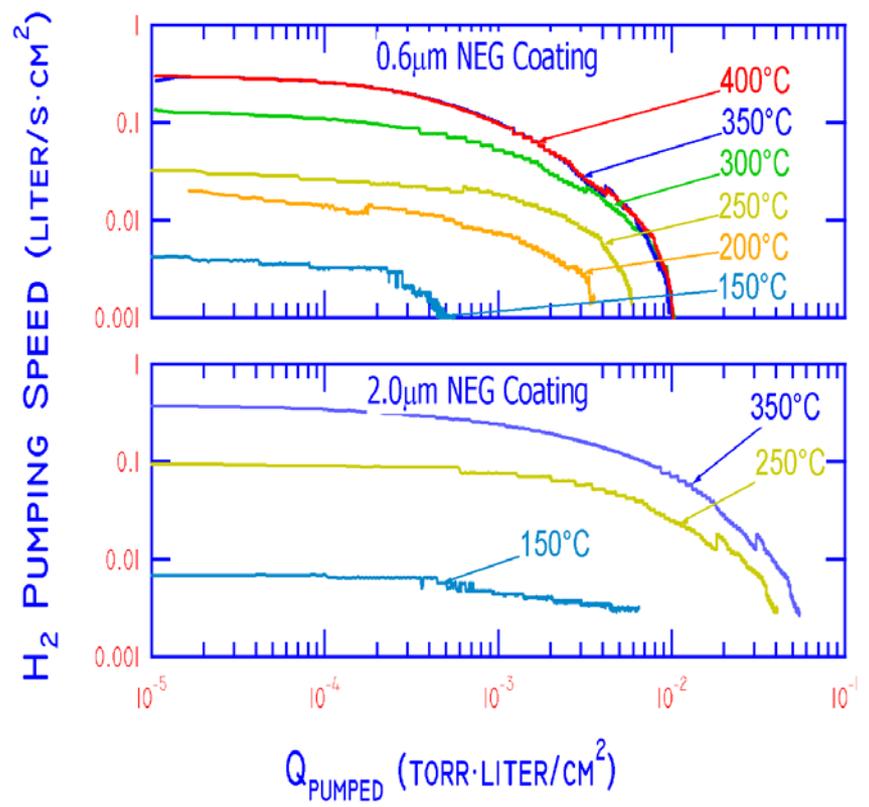
$T_{act} = 350^{\circ}C$





## Pumping Speed vs. Gas-load

Activation Temperature Dependence (48-hr activation)





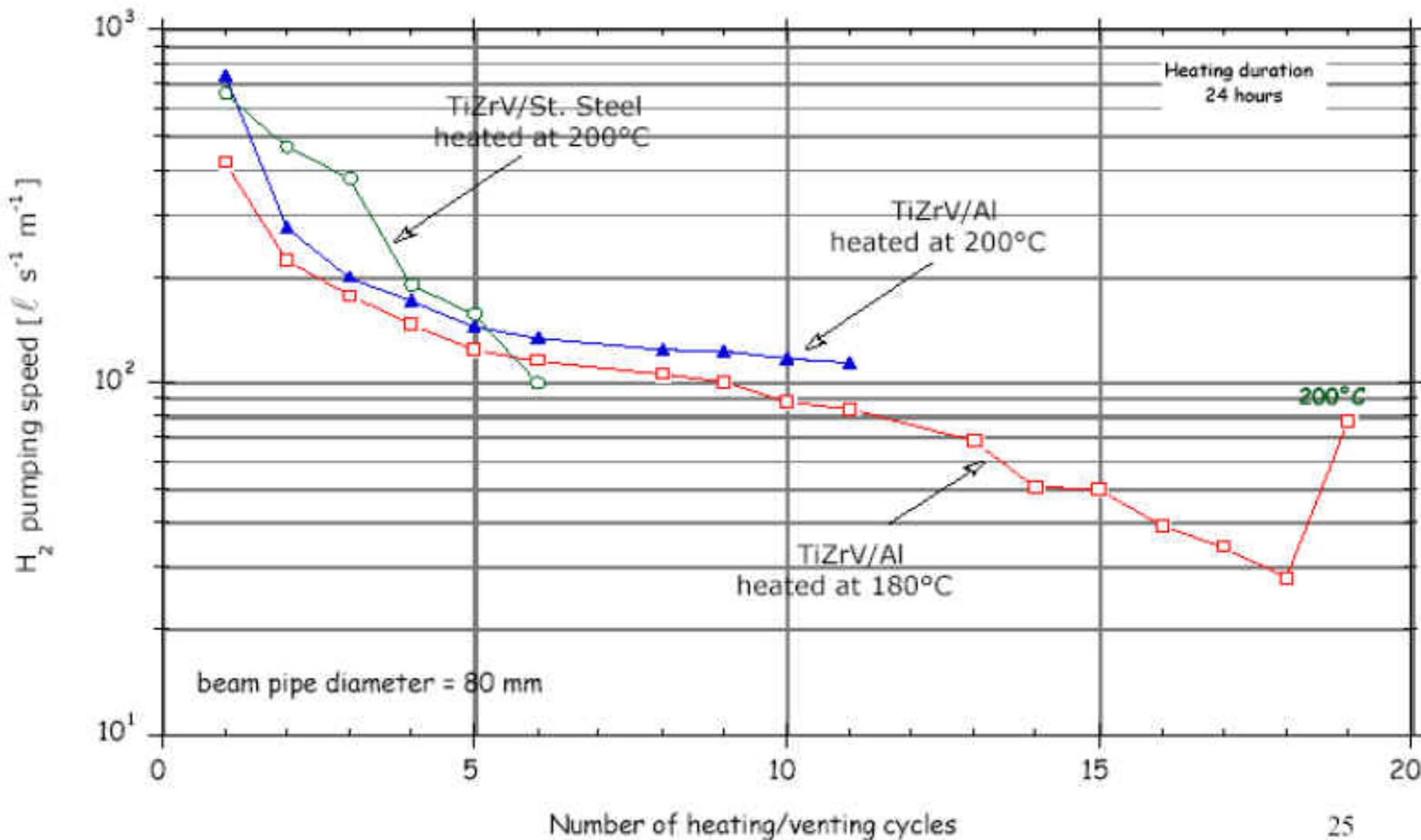
- Total pumping capacity of a NEG thin film depends on the film's solubility to oxygen, carbon, nitrogen, etc., and the film thickness

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

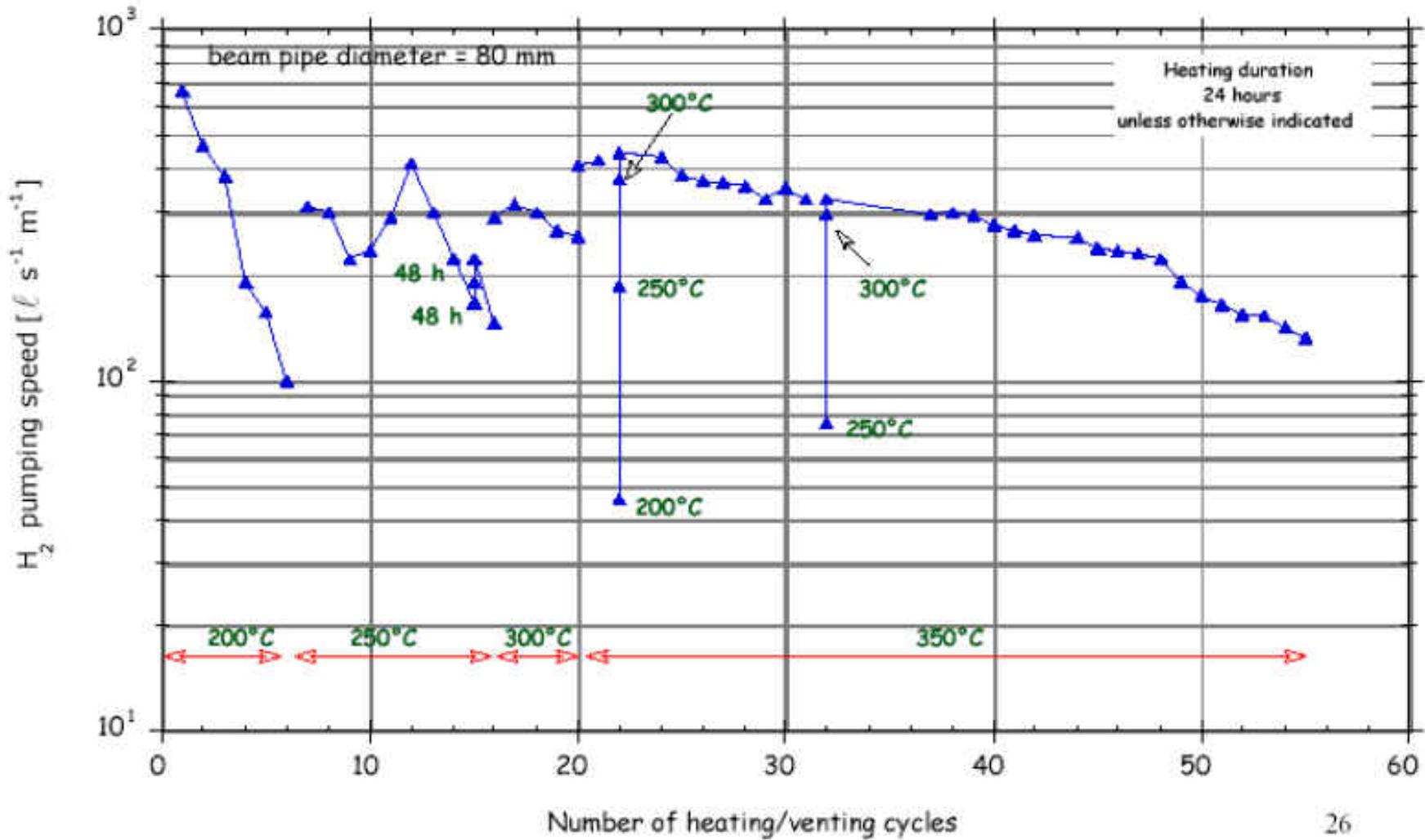
- Gradual aging is a deterioration of the thin film performance due to accumulation of oxygen in the film
  - Reduction of pumping speed and capacity
  - Increase of activation temperature



# NEG Film Aging Effect



# NEG Film Aging – More





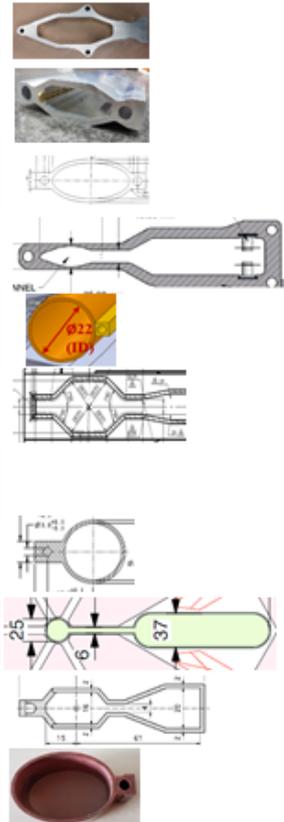
- NEG coating is an idea solution for long narrow-gapped undulator vacuum chambers
- All LHC warm beampipes were NEG coated.
- *ESRF* has had a very successful experience with the NEG-coated undulator chambers.
- RHIC coated ~600m of warm beampipes to suppress e-Cloud and associated abnormal pressure rises, which resulted in significant increase in heavy ion beam performances.
- Other new 3<sup>rd</sup> generation SR light sources, such as *SOLEIL* and *DIAMOND*, also used the NEG coatings for the undulator chambers.
- A *NEG Coating Workshop (45th IUVSTA Workshop)* was held at *Catania Italy*, in April 2006.



# Comparison of SR facilities in attendance @ 80<sup>th</sup> IUVSTA



Machine	Country	Status	Lattice	Energy (GeV)	Current (mA)	Circum (m)	Chamber ID (mm)	NEG coat
SOLEIL	France	Active (2006)	DBA	2.75	400	354	25 x 70	56%
PLS-II	S. Korea	Active (2012)	DBA	3	400	281	N/A	0%
TPS	Taiwan	Active (2016)	DBA	3	500	518	38 x 88	0%
NSLS-II	USA	Commissioning	DBA	3	500	792	25 x 76	0%
MAX-IV	Sweden	Commissioning	7BA	3	500	528	22	100%
EBS	France	In Design (2020)	7BA	6	200	844	20	0%
HEPS	China	In Design (2024)	7BA	6	200	1296	22	>80%
APS-U	USA	In Design (2025)	7BA	6	200	1104	22	20%
KEK-LS	Japan	Proposed	8BA	3	500	570	25	>50%
Spring-8 U	Japan	Proposed	5BA	6	100	1436	16 x 30	0%
Diamond-II	UK	Proposed	6BA	3	300	562	18 x 27	0%
ALS-U	USA	Proposed	DLSR	2	500	197	N/A	>20%



*Courtesy of Jason Carter of APS*

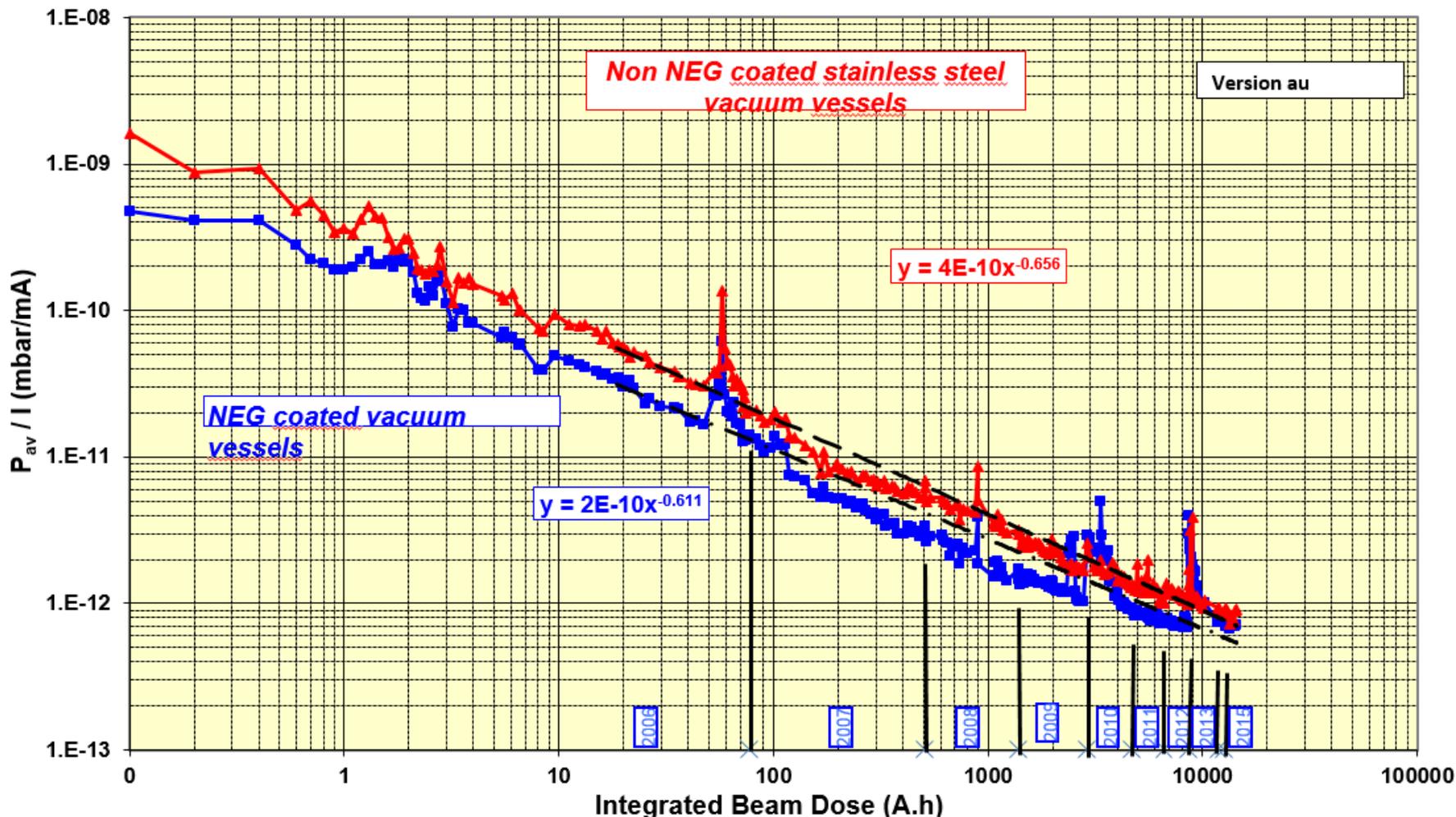


# 10 year experience of operation with NEG coating at the SOLEIL synchrotron light source



Christian HERBEAUX, N. BECHU, Vacuum Group

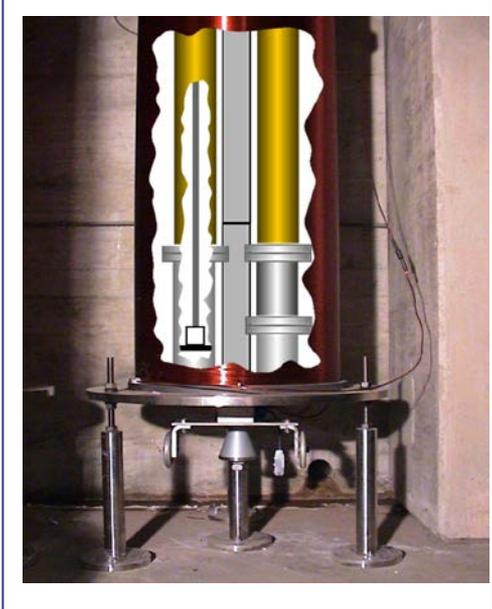
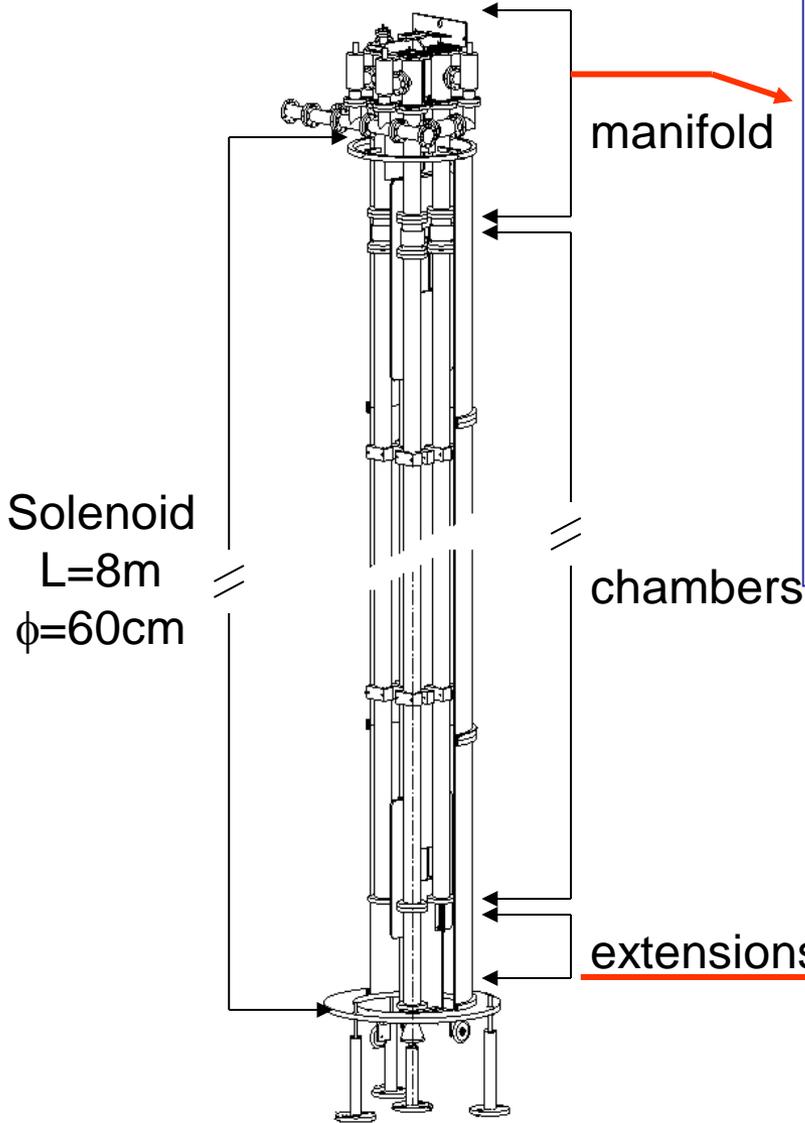
Average pressure rise in Cell C07 normalised to current Vs. the beam dose



# CERN's NEG Coating Facility



# CERN's NEG Coating Facility – Details



# CERN's NEG Coating Production



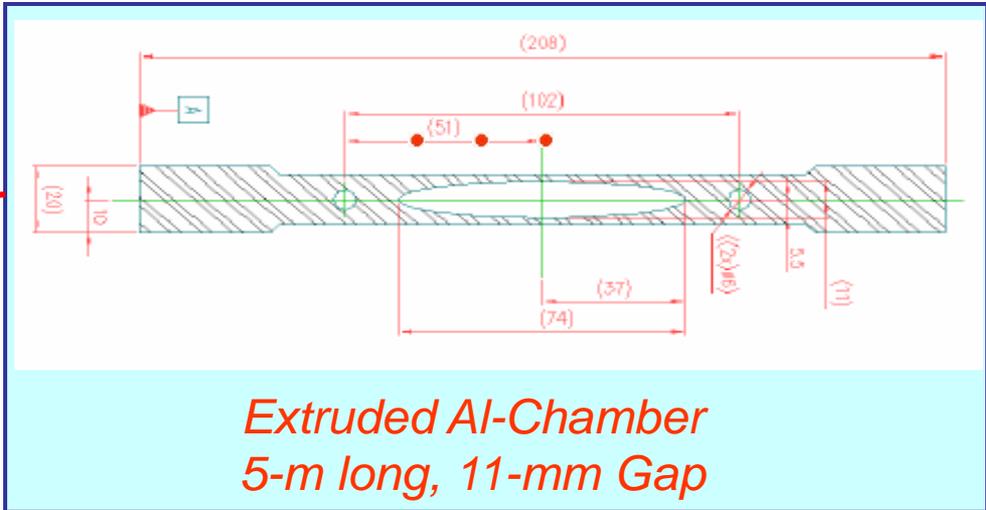
More than 1300 chambers coated with TiZrV NEG for the LHC.  
Standard chambers are 7 m long, 80 mm diameter.



# ESRF's NEG Coating Facility



A New NEG Coating Building @ESRF



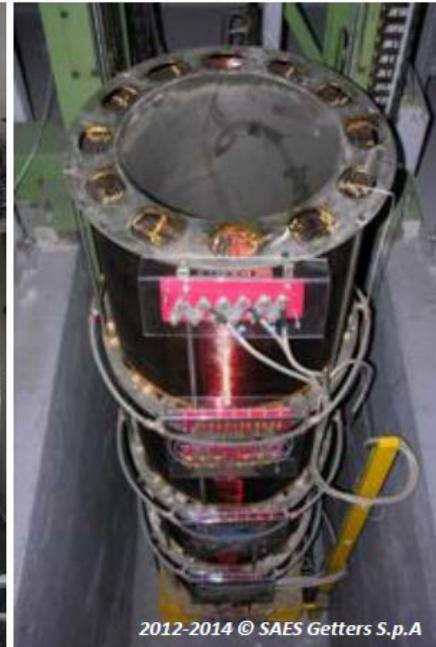
Motorized Air-cool Solenoid (500 G @100Amp)



# IntegraTorr® – SAES Getters' NEG Coating



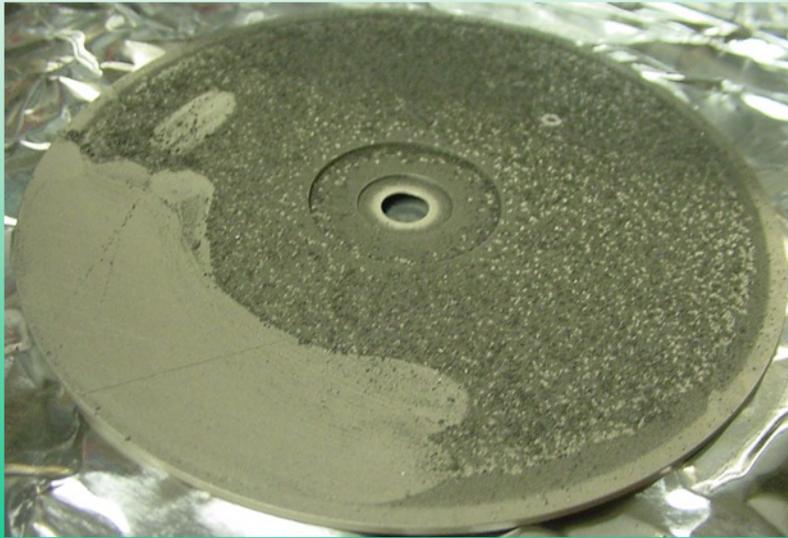
- ❑ *SAES Getters is licensed by CERN to provide commercial NEG coating services.*
- ❑ *All components to be coated by SAES are cleaned by CERN facility, to ensure good thin film adhesiveness.*
- ❑ *Known projects used this services: RHIC, CesrTA, etc.*



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

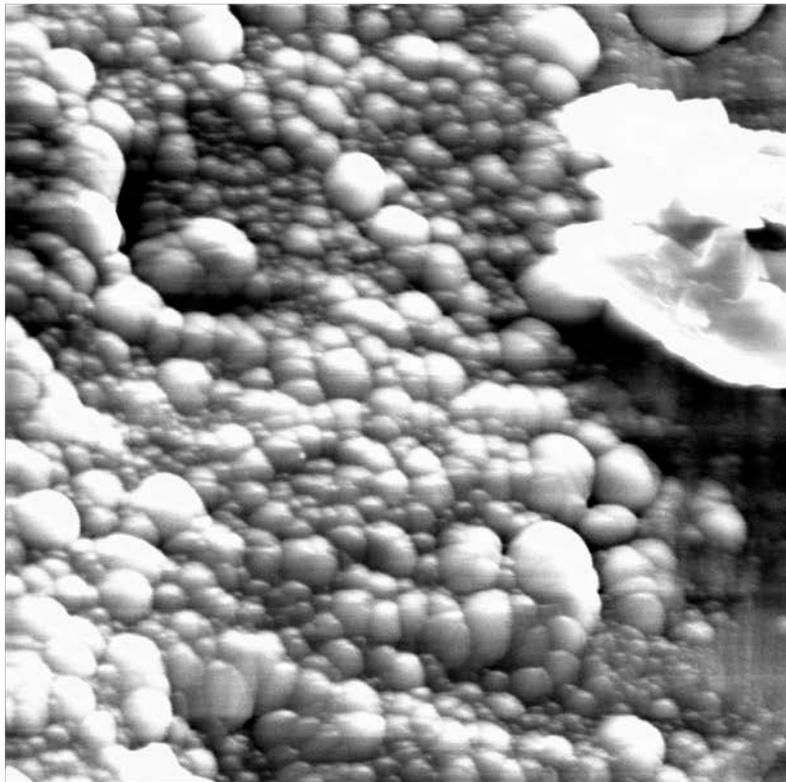
## *Word of Caution*

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

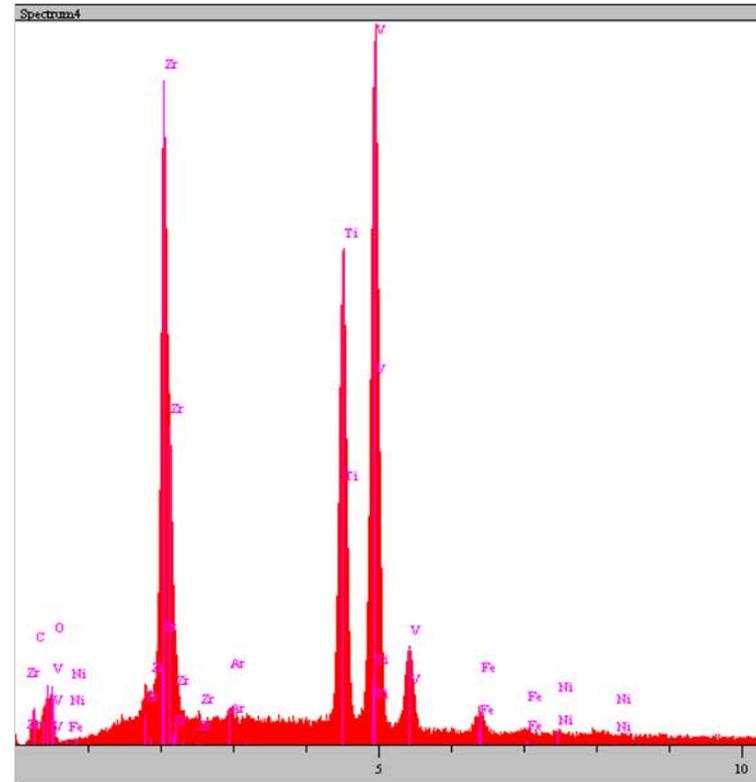


- The original coating had excellent bonding, by visual inspection and/or via 'tape testing'*
- Believe the coating was damaged by excessive H<sub>2</sub> sorption. More investigation planned*

# *Powder Confirmed to Be NEG*



*Powder SEM Image*



*Powder EDX Spectrum*



- *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 lower cost*
  - *More operational friendly*
  - *'Un-limited' capacity*
- *However, space is always tight in accelerators, NEGs are more favorable*
  - *They are more expensive, but similar to the ion pumps*
  - *NEGs are usually user-ready, little design involved*
  - *Capacity vs. dynamic gas-load needs to be evaluated.*
- *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 module/cartridge*  
*(in the NEG materials, or in the heating elements) ?*
  - *What's the best way to passivate NEGs for air exposure ?*

