The US Particle Accelerator School
Titanium Sublimation Pumps

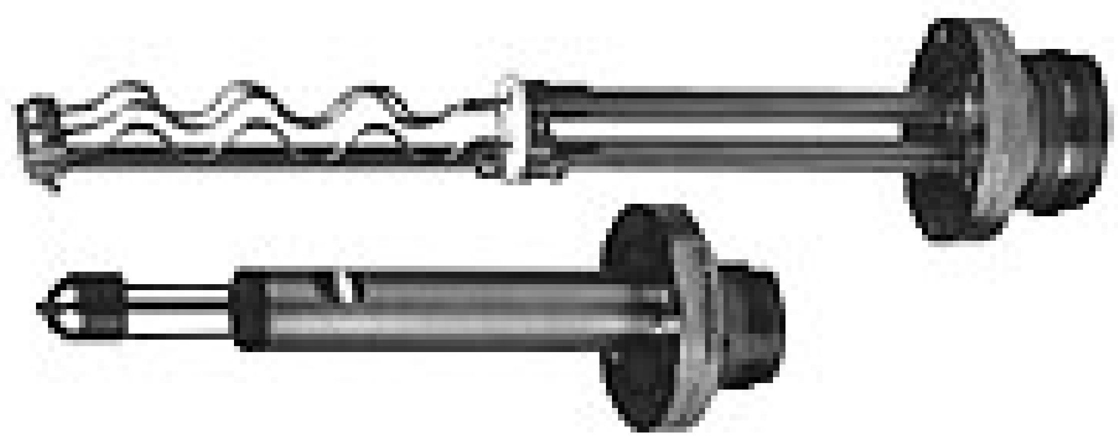
Lou Bertolini

Lawrence Livermore National Laboratory

June 10-14, 2002

Titanium Sublimation Pumping (TSP)

- Gases are pumped by "gettering." "Getterable" gases are pumped at high speeds by chemisorption.
- TSP is very practical and a cost effective mechanism.
- Manufacturers don't sell TSPs; they sell Titanium sources.



Titanium Sublimation Pumping

- **"Sticking Coefficients"** of gases are important in understanding TSPs.

- **Sticking coefficient** (α), is the probability that a specific gas molecule, when striking a surface will permanently stick.

$$\upsilon = 3.5x10^{22} \frac{P}{\sqrt{MT}}$$ Impingement rate

Titanium Sublimation Pumping

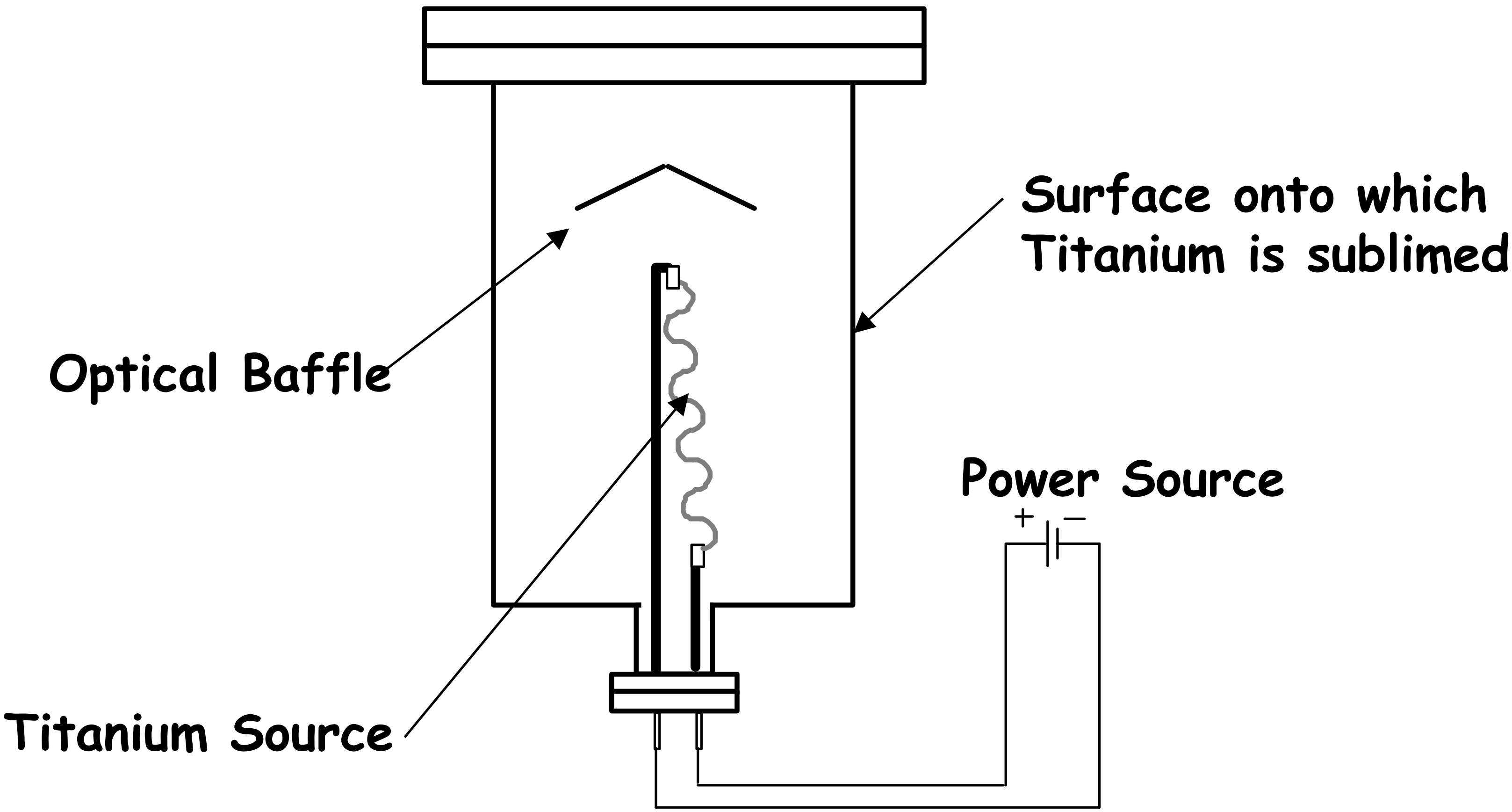
- TSPs are "surface" pumps that are surface conductance limited.

- There is a relationship between the number of active metal energy sites per unit area and the sticking coefficient.

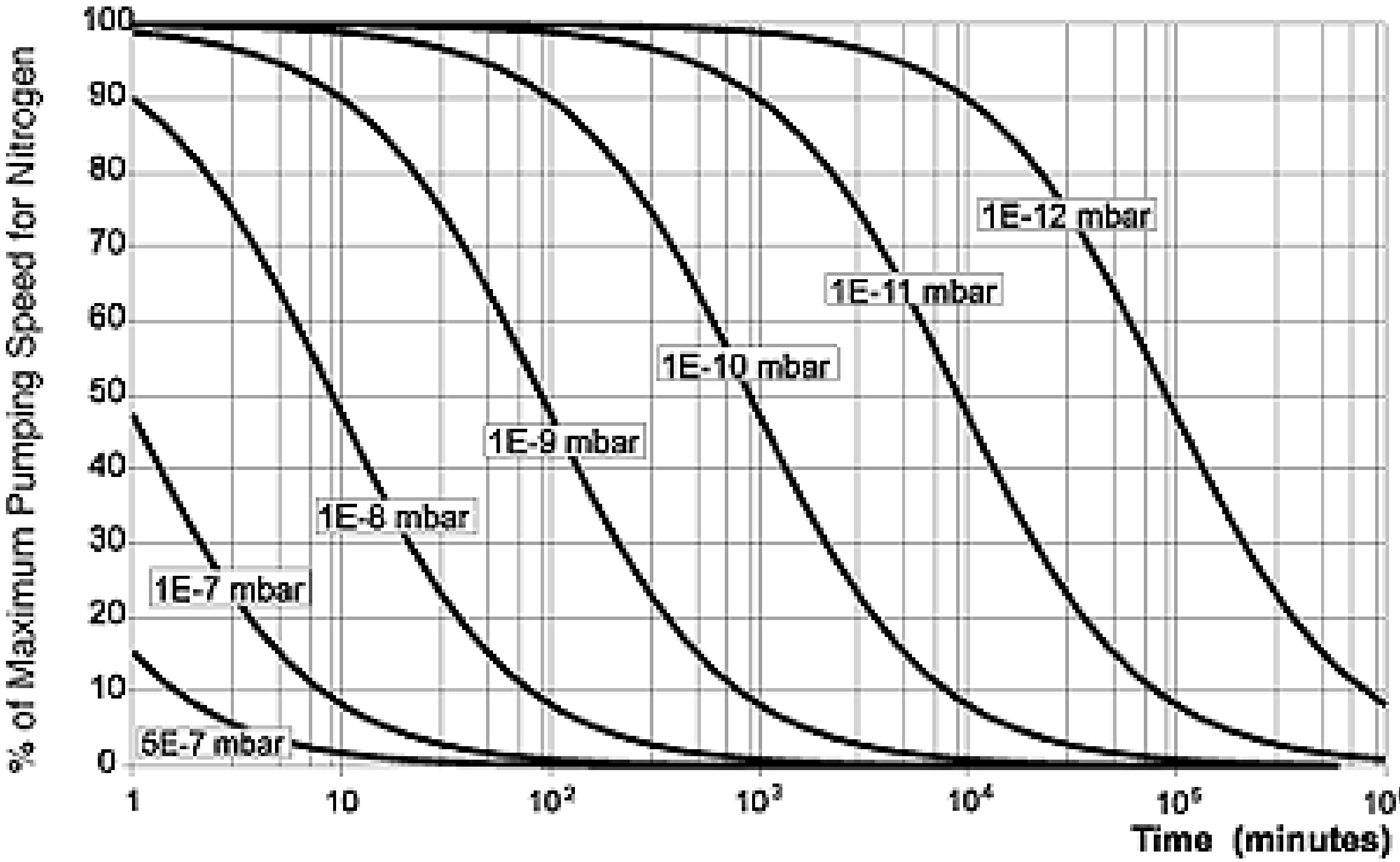
- The sticking coefficient will decrease as the active metal energy sites decrease.

Maintaining a balance between the incident flux of gas and the available chemisorption sites is key to Titanium Sublimation Pumping.

Features of a Titanium Sublimation Pump



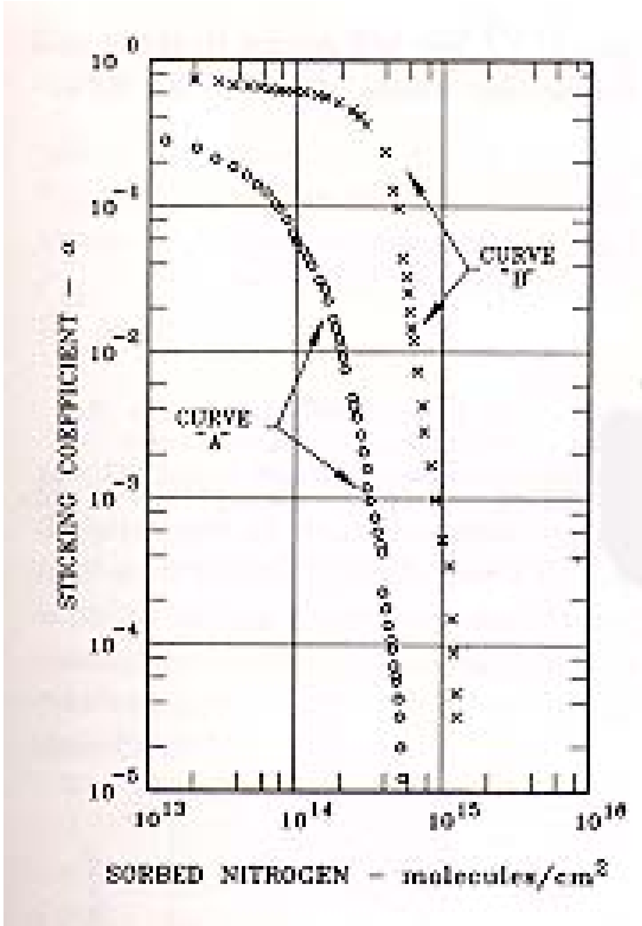
TSP vs. time (at various pressures)



Ref. Varian Vacuum

Sublimation Processes

1. Batch sublimation - periodic sublimation of titanium onto the pumping surface. The titanium film is then allowed to saturate with gas over time.



Curve A – batch layer of 8.0×10^{14} Ti atoms/cm^2 deposited on a previously exposed base of 1.7×10^{17} atoms/cm^2

Curve B – batch layer of 8.3×10^{14} Ti atoms/cm^2 deposited on a previously exposed base of 1.0×10^{18} atoms/cm^2

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

Sublimation Processes

2. Continuous Sublimation - titanium sublimation onto a pumping surface at a constant and continuous rate. Pumping sites are replenished at a rate equal to the rate at which they are occupied.

$$S_s = \alpha_i C_i A_c$$

where S_s = surface pump speed

α_i = sticking coefficient for gas species "i"

C_i = aperture conductance per cm^2 for gas species "i"

A_c = total pumping surface area

Factors that Influence Sticking Coefficient

- 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

Some have proposed that there is a "pecking order" of gases pumping by Titanium chemisorption

Pumped Gas	Displaced Gas				
	CH_4	N_2	H_2	CO	O_2
CH_4		no	no	no	no
N_2	yes		no	no	no
H_2	yes	yes		no	no
CO	yes	yes	yes		no
O_2	yes	yes	yes	yes	
α_m	$<10^{-3}$	0.3	0.05	0.85	0.95

This is controversial and probably only true for CH_4 and H_2.

Typical Engineering Values for TSP

Test Gas	Max. Sticking Coefficient-α_m		Max. Speed[a] (liters/sec-cm^2)		Max. Capacity of Film- x10^{15} (molecules/cm2) [b]	
	300 K	77 K	300 K	77 K	300 K	77 K
H_2	0.06	0.4	2.6	17	8-230[c]	7-70
D_2	0.1	0.2	3.1	6.2	6-11	-
H_2O	0.5	-	7.3	14.6	30	-
CO	0.7	0.95	8.2	11	5-23	50-160
N_2	0.3	0.7	3.5	8.2	0.3-12	3-60
O_2	0.8	1.0	8.7	11	24	-
CO_2	0.5	-	4.7	9.3	4-12	-

a) Speed calculated at RT
b) Wide vaiations 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)

Titanium Sublimation Sources

There are three types of sources for sublimating Titanium on pumping surfaces:

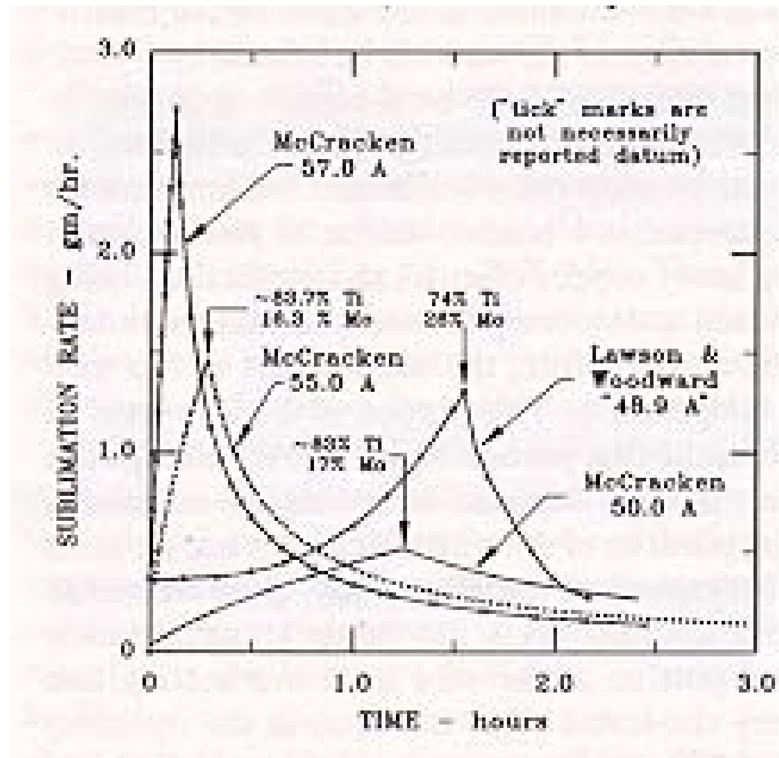
1. Filamentary sources
2. Radiantly heated sources
3. Electron-gun sources

Titanium Filamentary Sources

- Titanium has poor mechanical strength at sublimation temperatures.
- Titanium filaments must be coupled with other materials for mechanical strength.
  - One method is to wrap the Titanium filaments on a structural member (Ta or W)
  - The most prevalent solution is to alloy Titanium with Molybdenum (85% Ti; 15% Mo by weight)

Constant current operation of TSPs

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



Ref."Properties of Titanium-Molybodenum Alloy Wire as a Source of Titanium for Sublimation Pumps," Lawson and Woodward, Vacuum 17, 205 (1967)

"Titanium Filaments for Sublimation Pumps" McCracken and Pashley, JVST, 3, 96 (1966)

Constant Voltage Operation of TSP Filaments

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

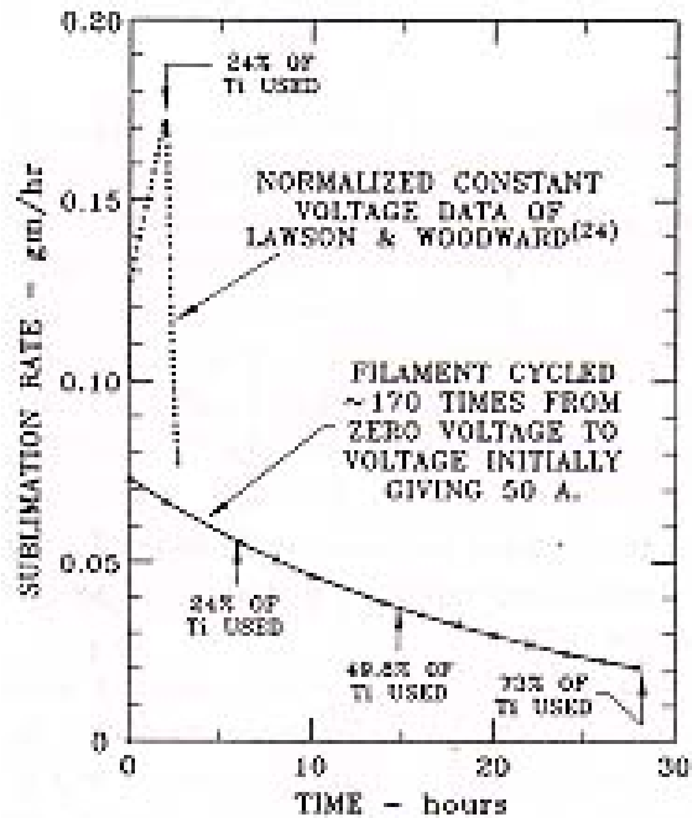
$$R(t) = R_o e^{-at}$$

where R_o = initial sublimation rate

a = constant

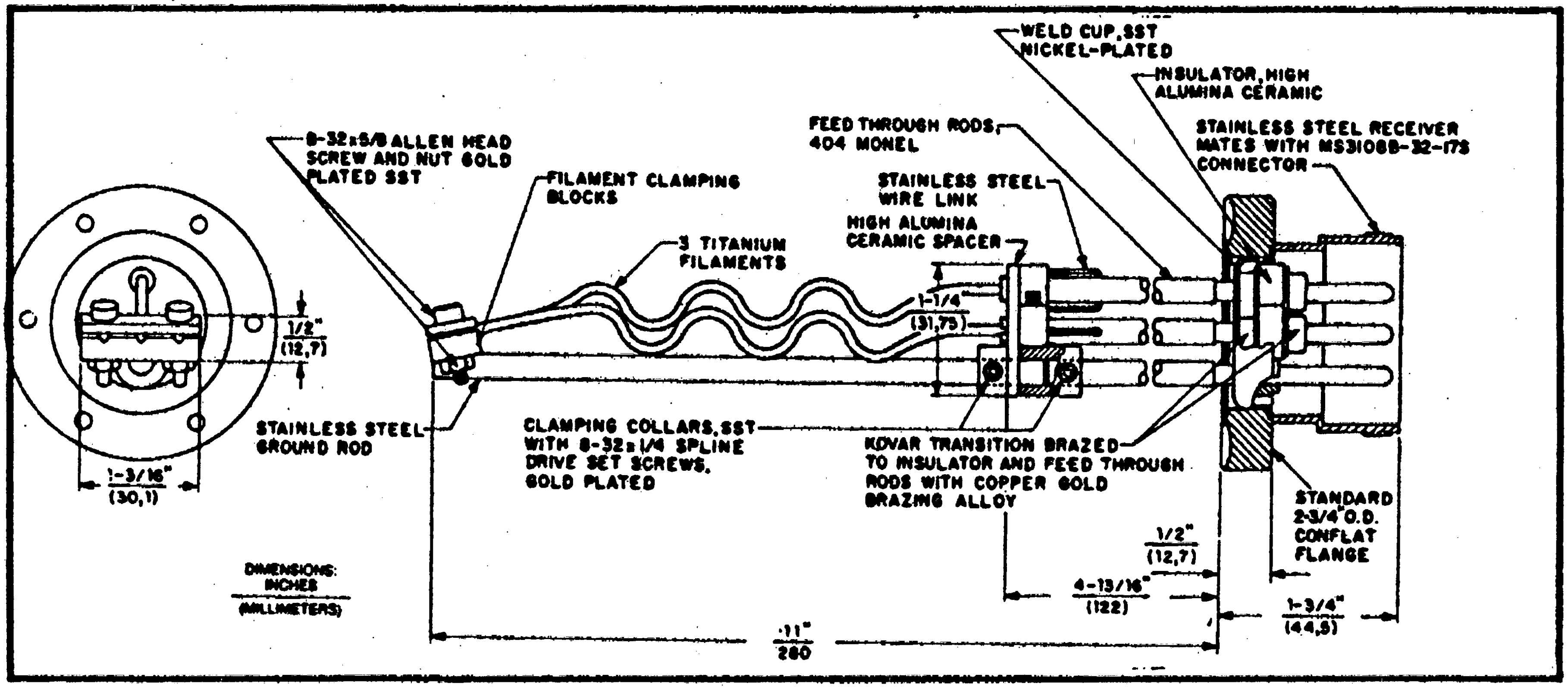
t = cumulative sublimation time

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



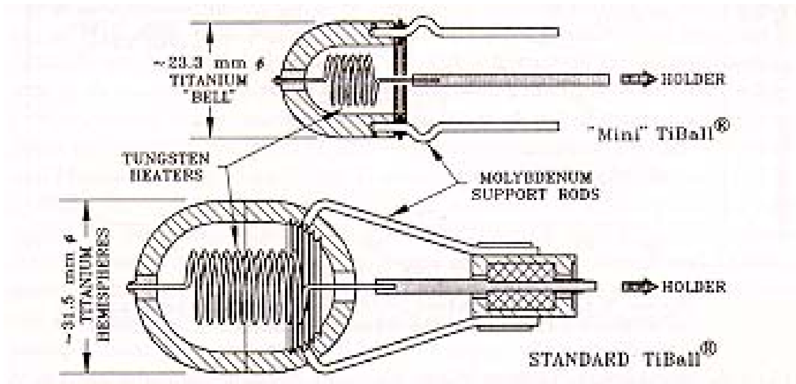
Ref. "Properties of Titanium-Molybodenum Alloy Wire as a Source of Titanium for Sublimation Pumps," Lawson and Woodward, Vacuum 17, 205 (1967)

Titanium Sublimation Cartridge

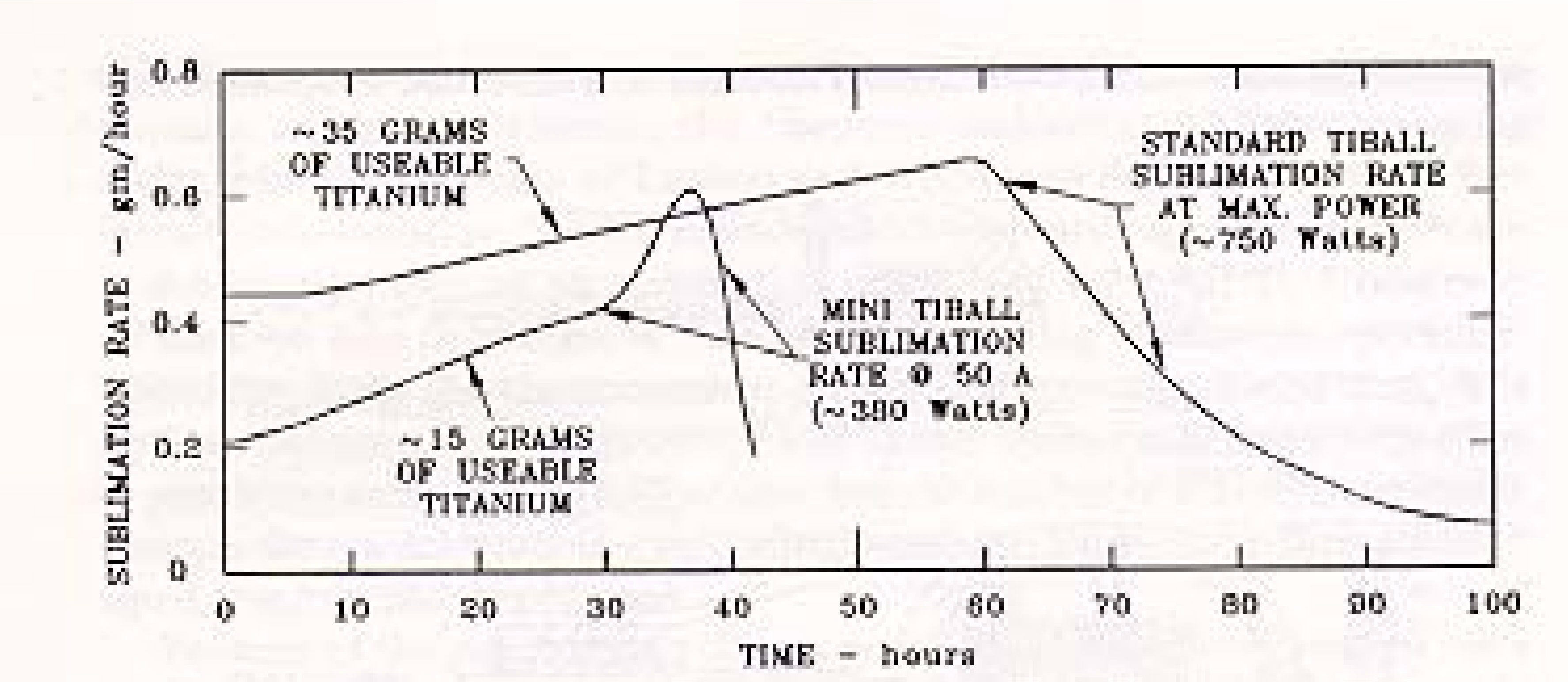


Radiantly Heated Titanium Sources

- Titanium heating occurs primarily through radiation from a secondary Tungsten (W) filament.
- The Titanium sphere surrounds the filament and provides the return current path.
- Titanium source assembly is mounted as a removable flange with an electrical feedthrough.
- Functional life ends when a "hole" opens up in the Titanium sphere.



Sublimation rates for Varian TiBall and "Mini" TiBall Sources



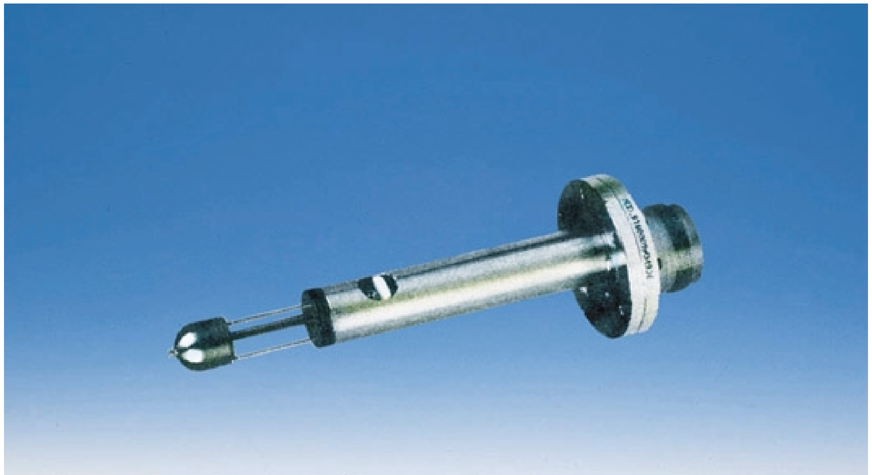
Ref. "A Radiant Heated Titanium Sublimator," Proc. 5th Int. Vacuum Congress, 1971, JVST 9 (1), 552 (1972)

Disadvantage of Radiantly Heated Sources

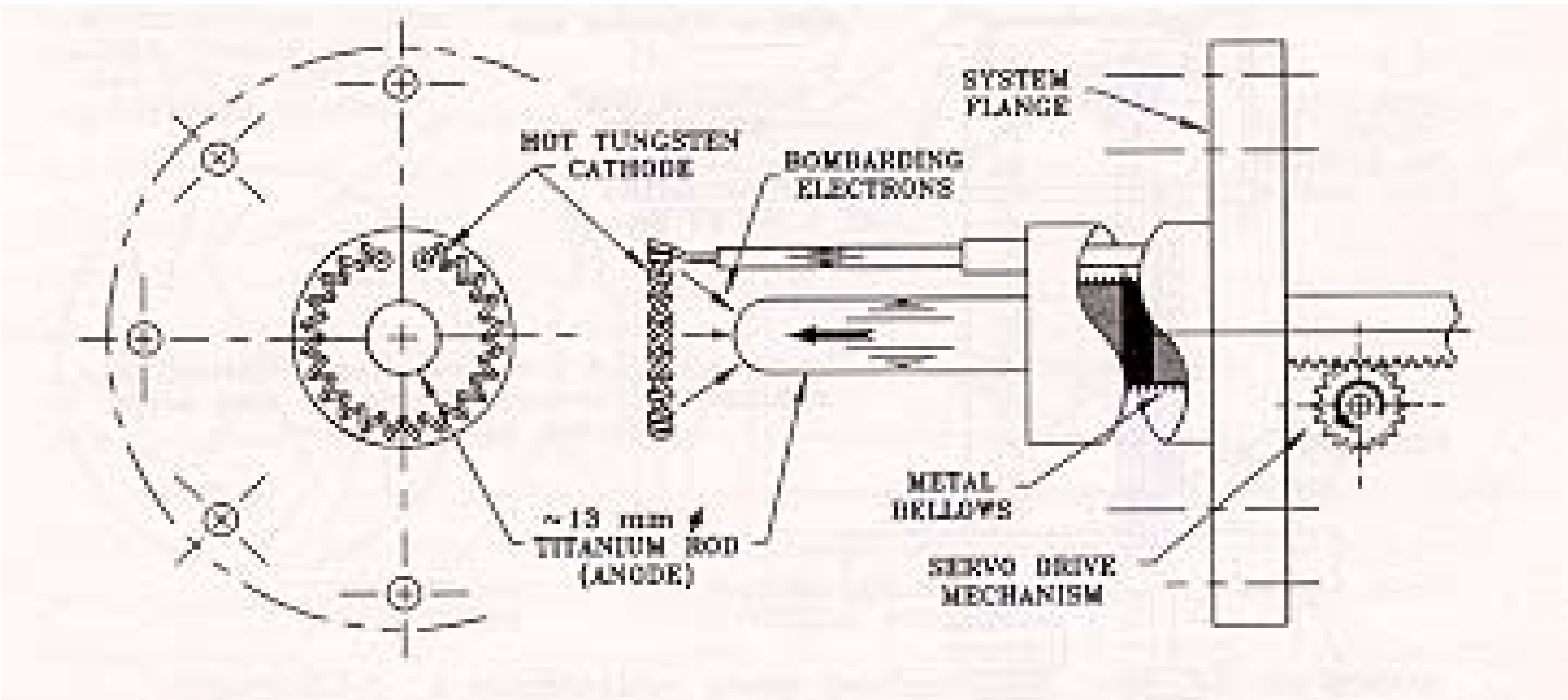
- Sources require operation at some level of standby power to maintain Titanium temperature above 900°C.
- Temperature cycling through the crystalographic phase transformational temperature of 880°C eventually results in distortion.
- Two things can happen (both bad):
  1. Distortion leads to electrical shorting of W filament.
  2. Distortion leads to increased emissivity, lower temperatures, and reduced sublimation rates.

Summary of Radiantly Heated Titanium Sources

- Standby power (100-200 W) required to prolong source life.
- Heat from standby mode can increase gas load.
- In storage rings (operating at 10^{-10} Torr), Titanium quantity is less important than reliability.



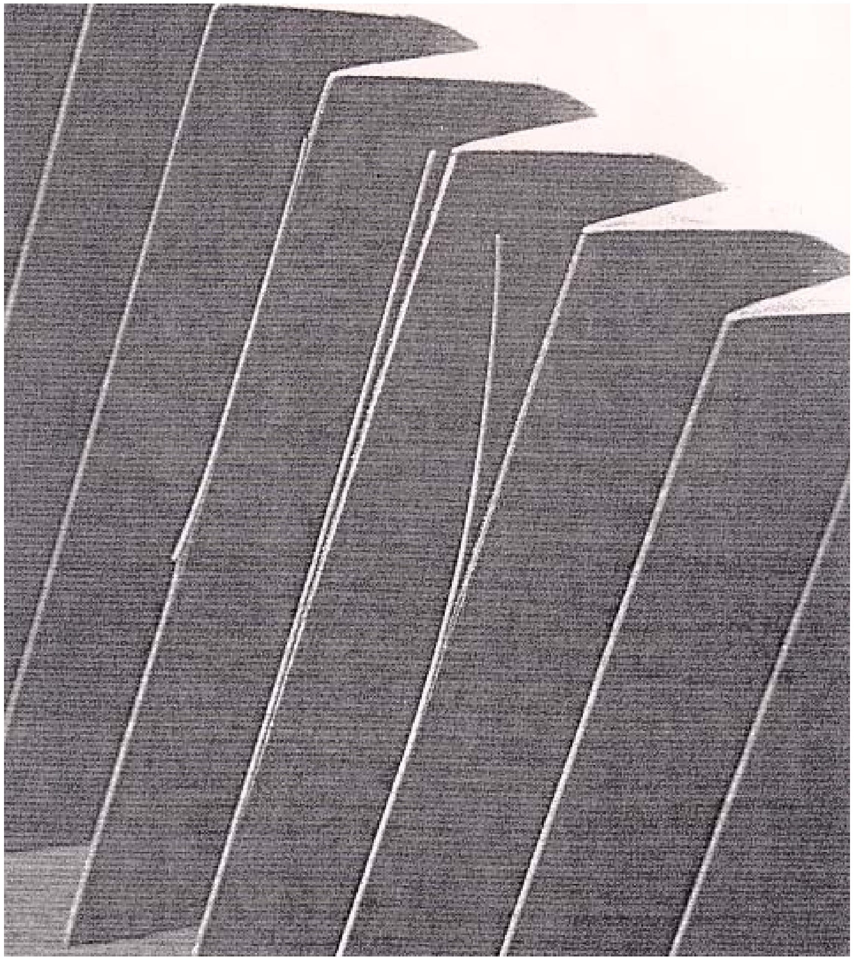
Electron-gun Titanium Sources



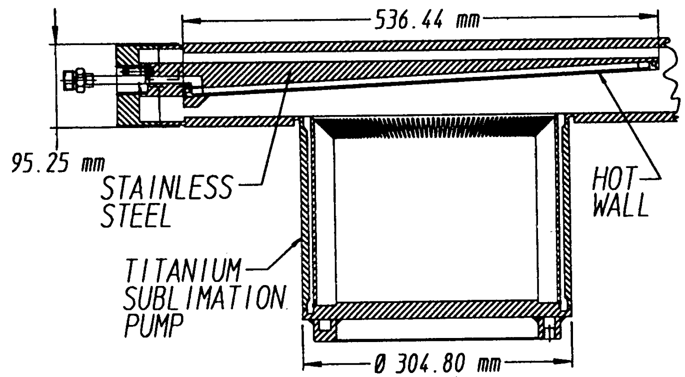
Ref. Perkin-Elmer Corporation

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



Pumping Speed as a Function of Gas for an Extended Surface 6" TSP PUMP

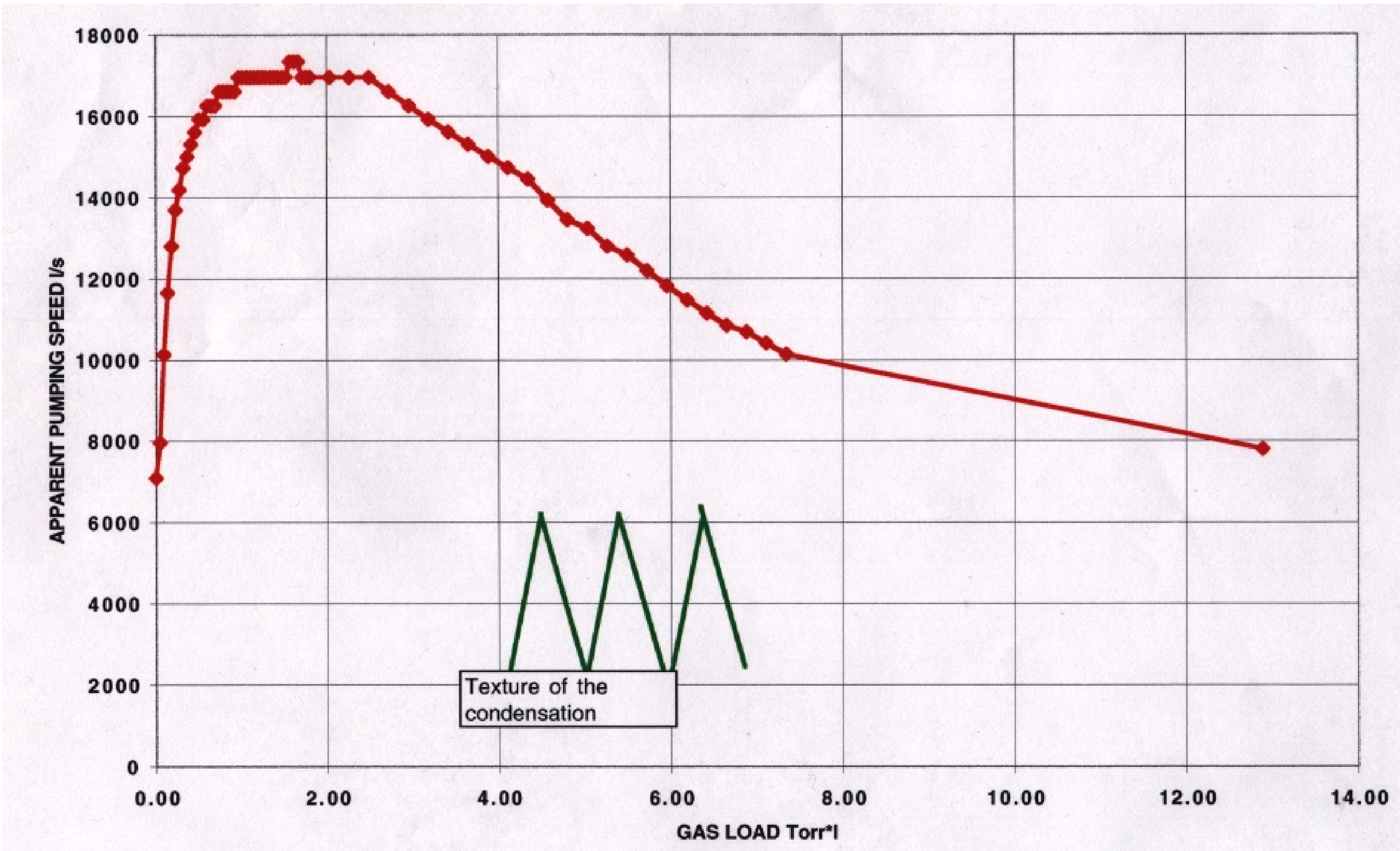
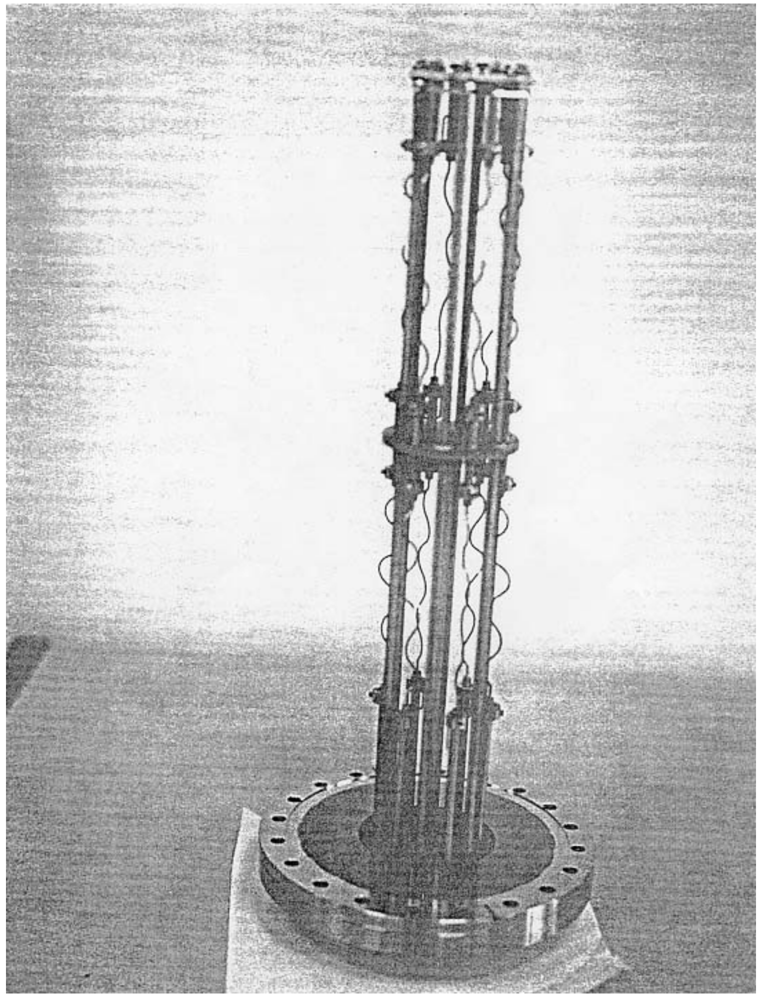
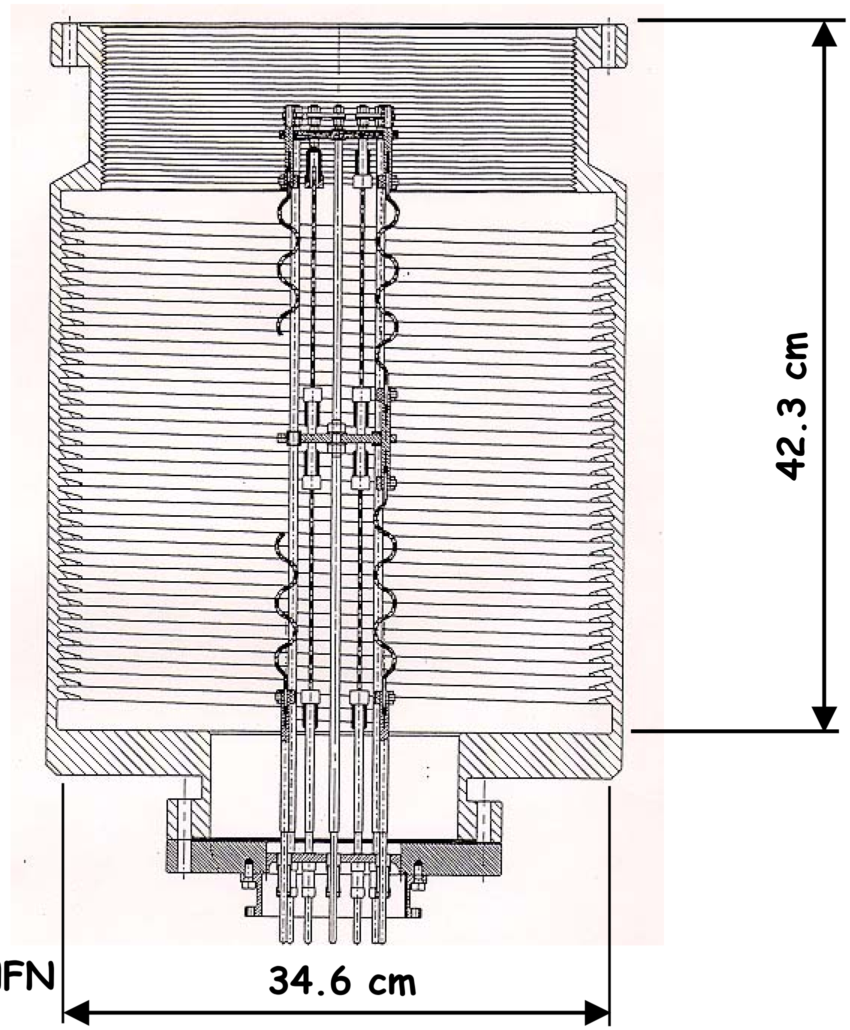


Photo of custom Varian TSP designed for ALS and DAFNE



DAFNE Collider TSP



Courtesy: C. Vaccarezza, INFN