

# The Pumping of Helium and Hydrogen by Sputter-Ion Pumps Part II: Hydrogen Pumping

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**R H I C   P R O J E C T**

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# THE PUMPING OF HELIUM AND HYDROGEN BY SPUTTER-ION PUMPS\*

## PART II: HYDROGEN PUMPING

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### ABSTRACT

The pumping of helium by various forms of sputter-ion pumps (i.e., SIPs) is given in Part I.<sup>1</sup> The pumping of hydrogen in diode and triode SIPs is herein discussed. The type of cathode material used in these pumps is shown to have a significant impact on the effectiveness with which hydrogen is pumped. Examples of this include data for pumps with aluminum, titanium and titanium-alloy cathodes. Diode pumps with aluminum cathodes are shown to be no more effective in the pumping of hydrogen than in the pumping of helium. The use of titanium anodes and titanium *shielding* of a pump body is also shown to measurably impact on the speed of a pump at very low pressures. This stems from the fact that hydrogen is  $\times 10^6$  more soluble in titanium than in stainless steel. Hydrogen becomes resident in the anodes because of fast neutral burial. Ions and fast neutrals of hydrogen are also buried in the walls of pump bodies. Outgassing of this hydrogen from the anodes and pump bodies results in a gradual increase in pump base pressure and consequential decrease in hydrogen pump speed at very low base pressures.

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## INTRODUCTION

There are three types of SIP configurations: i) conventional or *standard* diode pumps; ii) *noble* diode pumps; and, iii) *triode* pumps. The variety of presently available commercial SIPs and their pumping mechanisms are discussed in detail elsewhere.<sup>2</sup> In brief, the configuration of single-cell triode and diode SIPs is shown in Fig. 1. The cathodes are customarily made of some chemically active material. Gas is ionized in the hollow, Penning discharge *cells* by swirling clouds of electrons. These electrons are trapped within the cells by orthogonal electric and magnetic fields. Gas ions bombard and sputter the chemically active cathode material. The lighter gases, such as He and H<sub>2</sub>, do not effectively sputter the cathodes. Gases chemically active with the cathode materials are pumped by chemisorption and the inert gases by physisorption.

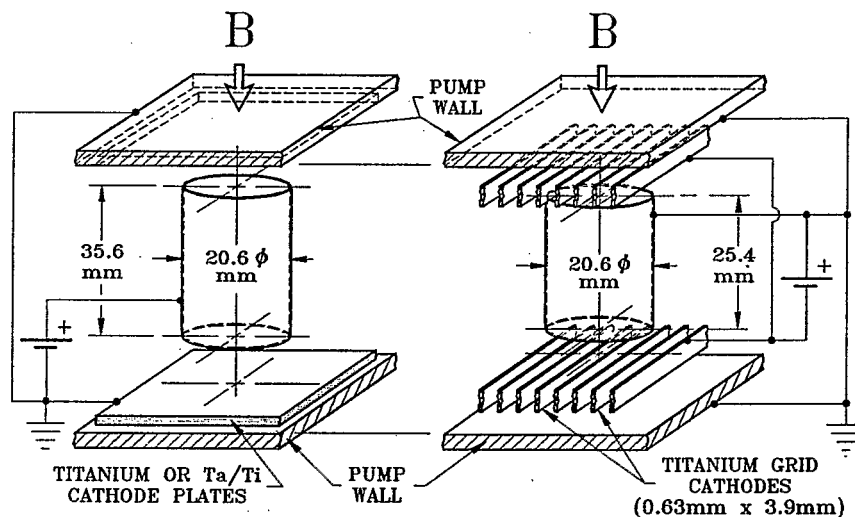


Figure 1. Configurations of single-cell diode and triode sputter-ion pumps.

If an energetic gas ion strikes a metal surface, there is a probability that it will *steal* an electron from the surface, and rebound as an energetic neutral atom. These energetic neutrals are reflected back from the cathodes and buried as neutrals in exposed pump surfaces. If a gas is not chemically active with the cathode material, it can only be pumped by burial as a high energy neutral or gas ion. Therefore, physisorption mechanisms include the burial of cathode-reflected high energy neutrals and ion implantation in the cathodes.

The only difference between standard and noble diodes is in the selection of cathode material. In the case of the noble diode, sometimes called DI® pumps, one cathode comprises Ti and the second Ta. The Ta atoms in the one cathode serve as a high-inertia crystal lattice for the reflection and burial of neutrals in other surfaces. In the conventional diode, both cathodes comprise Ti. The diode anode is operated at a positive voltage with respect to ground and the cathodes at ground potential.

The triode anode - the word *triode* is a misnomer - is operated at ground potential, and the cathodes at a negative potential with respect to ground. The cathodes of triode pumps are somewhat transparent to high energy neutrals created by charge exchange processes at these surfaces. Some of the gas ions created in the discharge cell pick up an electron on impingement with the cathodes, and pass on through the transparent grids as high energy neutrals. These neutrals are implanted in the walls of the pump, immune to subsequent sputter-desorption.

## UNIQUE HYDROGEN PUMPING CHARACTERISTICS

In order to understand the pumping of hydrogen by SIPs, we must first understand how the heavier chemically active gases are pumped. When these gases are ionized and bombard the cathodes they very effectively sputter these surfaces. For example, the sputter-yield of Ar ions on Ti - the ratio of the number of atoms sputtered off the cathodes to the number of bombarding ions - is greater than unity at  $\geq 1$  keV.<sup>3</sup> Therefore, chemically active cathode atoms are liberally spewed onto the surfaces of the anodes and juxtaposed cathodes. Later in the process, these atoms chemically combine with neutral gas molecules. The preponderance of gas is pumped by chemisorption on the pump anodes. Jepsen noted that the efficiency,  $\eta$ , with which  $N_2$  is pumped is 0.25 - 0.5.<sup>4</sup> The *efficiency* is merely the ratio of the number of gas atoms removed from the system to the number of ions bombarding the cathodes.

Hydrogen pumping by SIPs primarily stems from diffusion into the cathodes.<sup>5</sup> Molecular hydrogen ions bombard the cathodes, dissociate at the cathode surfaces, or at some modest implantation depth therein, and diffuse on into the bulk. Surface oxides on the cathodes inhibit this process. The sputter-yield for 7.5 keV  $H_2$  ions on Ti is only  $\sim 0.011$ .<sup>6</sup> A moderate oxide (or nitride) surface coverage on the cathodes inhibits pumping by  $H_2$  dissociation on the cathode surfaces. This effect is illustrated in Fig. 2 wherein the  $H_2$  speed of a pump with  $\sim 190$  cm<sup>2</sup> of cathode surface area is given as a function of the amount of pumped gas.<sup>7</sup> At point "A" in this figure, the speed of the pump is  $\sim 35\%$  of the steady-state value (*i.e.*, point "C"). This low initial speed is due to surface oxides.

As the cathodes are sputter-cleaned by  $H_2$  ions, the speed gradually increases to a value of  $\sim 160\%$  of the steady-state value (*i.e.*, point "B"). At this point  $\eta$  is found to be  $\sim 3$ . This startling result indicates that hydrogen *molecules* are being spontaneously dissociated at the cathode surfaces, and H atoms are diffusing into the bulk. This effect was first reported by Rutherford and Jepsen.<sup>8</sup> They noted the spontaneous pumping of  $H_2$  by an SIP for almost 60 h after turning off the high voltage supply. However, because of the low sputter-yield of  $H_2$  ions, even slight traces of a contaminant gas such as CO, CO<sub>2</sub>, N<sub>2</sub>, *etc.*, will result in an  $\eta \leq 1$ . The decrease in  $H_2$  speed noted after point "B" of Fig. 2 stems from complex  $H_2$  implantation effects described elsewhere.<sup>9</sup>

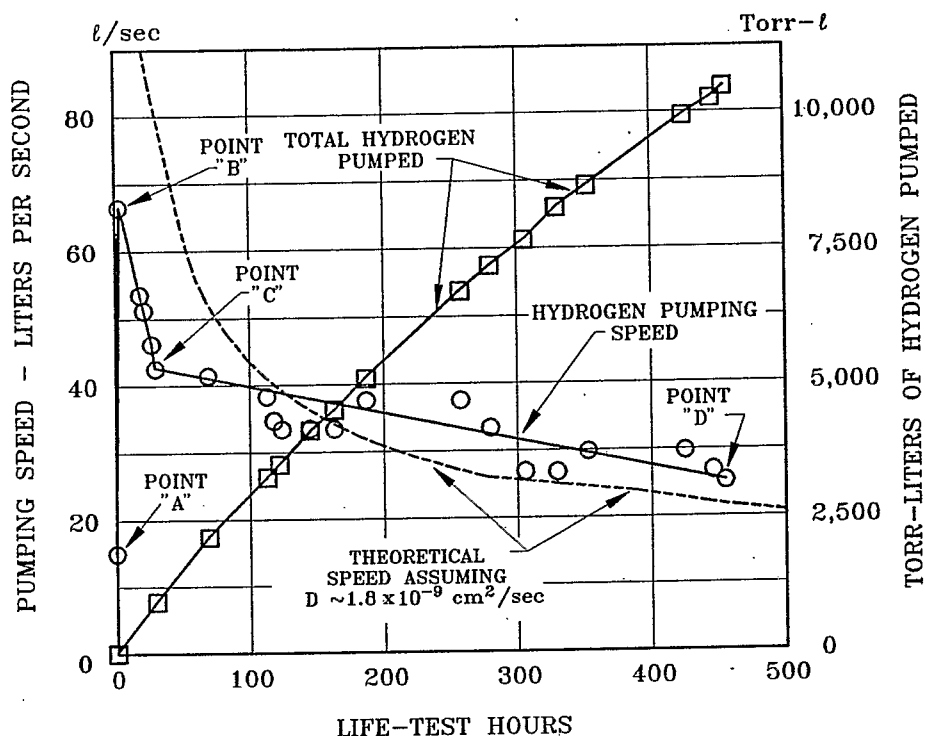


Figure 2. Speed and quantity of hydrogen pumped with Ti-6Al-4V cathode material.

The above findings make evident the complexity of making meaningful  $H_2$  speed measurements. Also, it is very difficult to interpret published  $H_2$  speed data of SIPs in the absence of knowledge of partial pressures of other gases at the time of the speed measurements. For obvious reasons, even slight traces of argon in the  $H_2$  gas will lead to inordinately high  $H_2$  speed results. For example, Rutherford noted that the  $H_2$  speed of a pump could be increased by  $\times 10$  by first sputter-cleaning the cathodes with argon.<sup>10</sup> Conversely, slight traces of  $CO$ ,  $CO_2$  or  $N_2$  will result in decreases in measured  $H_2$  speeds over that of pure hydrogen. This in part may explain Singleton's finding that speeds of SIPs do not follow the I/P ratio of the discharge at very low partial pressures of  $H_2$ .<sup>11</sup>

## CATHODE MATERIALS

Gurewitsch and Westendorp first proposed the use of Ti SIP cathodes. They also proposed C, Mg, SS (*i.e.*, stainless steel) and Al cathodes in single-cell SIPs in 1953.<sup>12</sup> Most quantitative work to follow involved pumps featuring Ti cathodes. Many papers indicated possible alternate cathode materials, but few gave quantitative data related to these materials. Rutherford and Jepsen noted work with Zr cathodes.<sup>8</sup> In 1966 James and Tom proposed combining one Ta and one Ti cathode in an SIP pump element for noble gas pumping.<sup>13</sup> This cathode pairing and other heavy metals paired with Ti was extensively studied.<sup>14</sup> In 1961 Jepsen worked with a sintered cathode matrix material, comprising Ti particles 10-25 microns in size, for the purpose of

enhancing  $H_2$  pumping in SIPs.<sup>15</sup> In 1969 Singleton studied  $H_2$  pumping in single-cell diode pumps with Ti and Al cathodes.<sup>16</sup> He later reported on tests of single-cell diode pumps with Ta, Ti, Zr, Mo, SS and Al cathodes, and reported that "... all gave virtually the same pumping speed ..." In 1971 Tom reported on the use of Mg/Ti cathode pairs in an SIP.<sup>17</sup> However, there were some technical problems associated with the use of Mg, the solutions of which were fiscally unattractive.

Little quantitative work was done thereafter with alternate cathode materials for  $H_2$  pumping until Hill reported on the use of Ti-alloy cathodes.<sup>18</sup> Hill was assisting Viissers with hydrogen concentration monitors used for detecting water leaks into sodium-cooled reactor heat exchangers.<sup>19</sup> Instruments comprising single-cell SIPs were coupled through Ni diaphragms to the Na loops. In the event of a water leak into the Na loop the water would dissociate and some of the resultant nascent hydrogen in the Na would diffuse through the Ni diaphragm and into the SIP. It was hoped that by monitoring the changes in the SIP current one could thereby deduce the concentration of hydrogen in the Na loop and be alerted of a water leak problem.

The problem with this scheme was that the pumping speed of SIPs for  $H_2$  varied so significantly over time that the pump current was of marginal benefit in deducing the actual hydrogen concentration in the Na loop. This problem was solved,<sup>20</sup> and work started in comparing the  $H_2$  pumping characteristics of various forms of titanium alloys.<sup>21</sup> Three materials were tested: 1) pure titanium; 2) Ti-6Al-4V alloy; and, 3) Ti-10V-11Cr-3Al alloy. These three materials are unique in their respective diffusivities. The pure Ti, prior to  $H_2$  implantation, has an hcp crystal lattice; the Ti-6Al-4V material is referred to as an " $\alpha$ - $\beta$  alloy", the  $\alpha$ -phase comprising an hcp crystal lattice and the  $\beta$ -phase comprising a bcc crystal lattice; the Ti-10V-11Cr-3Al alloy, referred to as  $\beta$ -stabilized, has a bcc crystal lattice. Findings of  $H_2$  life tests of these materials are summarized in Table I.

Table I. Hydrogen speed and capacity for a water-cooled sputter-ion pump with cathodes of different titanium alloys.

CATHODE MATERIAL	NOMINAL <sup>1</sup> SPEED-l/s	$H_2$ PUMPED @ END OF LIFE <sup>2</sup> Torr-l/cm <sup>2</sup>	INITIAL <sup>3</sup> DIFFUSIVITIES cm <sup>2</sup> /s	CAUSE OF END-OF-LIFE
TITANIUM	20	15.8	$5.0 \times 10^{-12}$	Diffusion Limited
Ti-6Al-4V	30	55.3	$1.8 \times 10^{-9}$	Diffusion Limited
Ti-10V-11Cr-3Al	50	84.2	$5.0 \times 10^{-7}$	$H_2$ Embrittlement

Notes: 1) Speed measured at  $\sim 10^{-5}$  Torr.

2) Life data taken at a pressure of  $1-2 \times 10^{-4}$  Torr, and with water-cooled cathodes.

3) Diffusivities decreased markedly with  $H_2$  pumping.

The cathodes of the pumps were water-cooled, and speed measurements were conducted at a pressure of  $\sim 10^{-4}$  Torr. End of life

in pumps with pure Ti cathodes and cathodes of the Ti-6Al-4Va material stemmed from diffusion limitation. End of life of pumps featuring the Ti-10V-11Cr-3Al material stemmed from catastrophic shattering of the cathodes because of H<sub>2</sub> embrittlement.<sup>22</sup>

## PUMPS WITH ALUMINUM CATHODES

The present work with Al cathodes started about three years ago. Dr. Derek Lowenstein, the Brookhaven AGS Department Head, found an article published in an obscure journal which championed the benefits of the use of Al cathodes in SIPs. Because of argon pumping instabilities in LINAC SIPs, we had just incurred great expense for Ta to convert 30% of these pumps to DI® diodes. We were asked: "Why not use Al cathodes in these pumps?" We had reservations about H<sub>2</sub> pumping with Al cathodes.

About this same time Liu and his colleagues reported on the stable pumping of gases, including H<sub>2</sub>, with a triode with Al cathodes.<sup>23</sup> Liu's results are shown in Fig. 3. The H<sub>2</sub> speed data were reported as steady-state and taken under *saturated* conditions. The H<sub>2</sub> results were very puzzling as the relative solubilities for H<sub>2</sub> in the metals Al:SS:Ti are ~1:10<sup>6</sup>:10<sup>12</sup>.<sup>24</sup> From this we speculated that the steady-state pumping of H<sub>2</sub> in the triode pump resulted from the burial of neutral hydrogen in the SS walls of the triode pump.

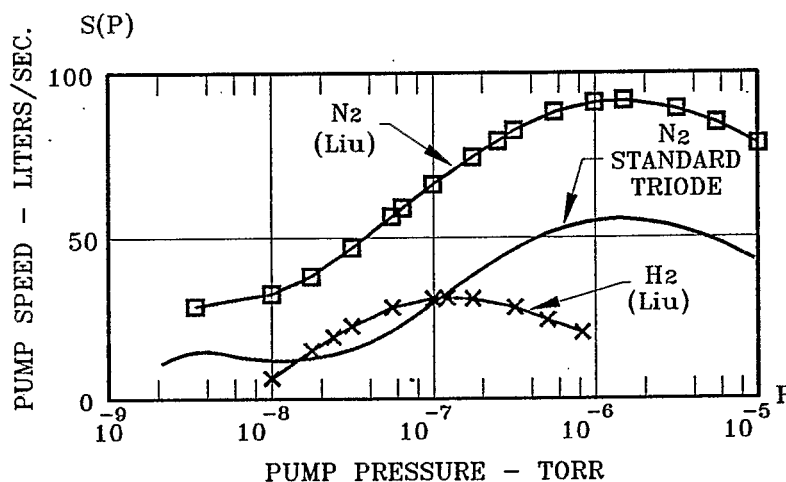


Figure 3. Nitrogen speed for a conventional triode sputter-ion pump and nitrogen and hydrogen speed for a triode pump with grided aluminum cathodes (Liu).<sup>(19)</sup>

To test this hypothesis, we built and tested a diode SIP with Al cathodes. The pump comprised two elements having a total of 126, 20.3mm  $\phi$  by 25.4 mm long anodes and a total projected cathode surface area of 1176 cm<sup>2</sup>. At the time we were unable to measure the amount and rate of H<sub>2</sub> being input to the system. Therefore, we had to use speed measurement data, using a CERN dome and calibrated Bayard-Alpert gauge, to determine the amount of H<sub>2</sub> pumped in time. This diode pump proved extremely

unstable when pumping  $H_2$ , the speed was erratic and end of life was manifest by pressure runaway. However, after a thorough analysis of the data we concluded that we had pumped 2-4 Torr-l of  $H_2$  at end of life. This corresponds to an  $H_2$  capacity of  $1.7-3.4 \times 10^{-3}$  Torr-l/cm<sup>2</sup>, and is comparable to findings of the more refined He capacity measurements made with this pump at a later date (i.e., ~3.4 Torr-l He prior to pressure runaway).<sup>1</sup> As the result of these measurements we concluded that hydrogen and He have comparable solubilities and diffusivities in Al.

We verified Liu's results for the steady-state pumping of  $N_2$  with Al cathodes. Steady-state  $N_2$  speed with Al cathodes at a pressure of  $\sim 10^{-7}$  Torr, and after pumping ~0.17 Torr-l  $N_2$ , was 140 l/s vs. 110 l/s with Ti cathodes. Also, we noted a steady-state CO pumping speed, with the same Al cathodes, of ~110 l/sec at  $\sim 3.5 \times 10^{-6}$  Torr after pumping ~10 Torr-l of CO.

## ALUMINUM CATHODES AND TITANIUM ANODES

Liu's results, in conjunction with the above diode pump results with Al cathodes, suggested that substantive quantities of  $H_2$  neutrals were being implanted in the walls of the triode pump. These neutrals on subsequent outgassing would result in reduced pump speeds at very low pressures.

Andrews and others, using autoradiography techniques, established that significant amounts of inert gas neutrals are buried in the anodes of pumps.<sup>25-28</sup> This suggested to us that the same low pressure outgassing problem would hold true for  $H_2$  in diode pumps with SS anodes. That is, H and  $H_2$  neutrals are implanted in the anodes and thereafter outgas at low pressures, thus reducing the speed at these low pressures to values disparate with known I/P values.

We modified the diode SIP described above to include new Al cathodes and Ti anodes. We speculated that if H and  $H_2$  neutrals were implanted in the anodes during  $H_2$  speed measurements, even with Al cathodes, we should observe some low, steady-state pumping speed after pumping 2-4 Torr-l of  $H_2$ . This low steady-state pumping speed could only be attributable to H and  $H_2$  implantation and retention in the anodes. The refined apparatus used in making these experiments is described elsewhere.<sup>1</sup>

As shown in Fig. 4, this proved to be one of those rare instances when one has a *hunch* about a complex mechanism, and it proves to be the case. An initial  $H_2$  speed of ~120 l/s was observed. The speed gradually decayed to a value of ~6.6 l/s. Thereafter it remained steady-state at this value even after ~15.28 Torr-l of  $H_2$  had been pumped. Speed measurements were conducted at pressures ranging from  $10^{-8}$  to  $5 \times 10^{-6}$  Torr. At  $H_2$  pressures  $> 5 \times 10^{-6}$  Torr the pressure tended to gradually increase, indicating possible thermal runaway due to desorption of  $H_2$  from the cathodes (i.e., ~14 mW/cm<sup>2</sup>). At pressures  $\leq 5 \times 10^{-6}$  Torr the pump was totally stable. We continued these speed measurements for several hundred hours and then concluded the test. From these results we concluded that 5-6% of the primary H and  $H_2$  ions were reflected off the cathodes as neutrals and were buried in the anodes of the pump.

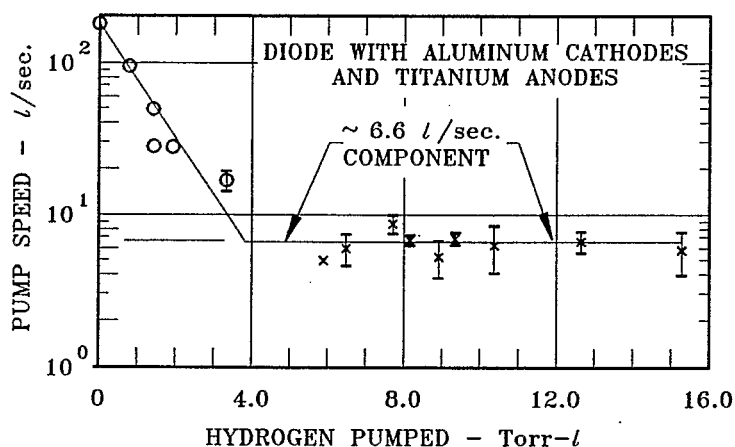


Figure 4. Hydrogen pumping speed of a diode sputter-ion pump with aluminum cathodes and titanium anodes.

## HYDROGEN BASE PRESSURE & SPEED MEASUREMENTS

As reported, when pumping He with a large diode SIP  $\geq 1.5\%$  of the He is buried in the walls of the pump.<sup>1</sup> This result and the above tests with Ti anodes suggested that the  $H_2$  pumping speed of a conventional SIP might be enhanced at low pressures if this pump was constructed with Ti cathodes *and* Ti anodes, and some effort was made to shield the SS pump body from stray  $H_2$  ions and neutrals emanating from the Penning discharge. Also, the base pressures of pumps with these features should be lower after high pressure  $H_2$  capacity tests or subsequent to a high temperature bakeout with the pump energized.

To test this hypothesis a direct comparison was made of the speed and base pressures of: 1) a conventional 270 l/s diode SIP with Ti cathodes and SS anodes (*i.e.*, the *standard pump* hereafter); 2) an identical SIP featuring Ti-alloy anodes, and without the benefit of pump body shielding. (Speed and base pressure results of these tests were only marginally better than that of the conventional diode. It was after these tests that we *stumbled onto* the fact that about 1.5% of the pumped gas was being buried in the walls of the pump body.); and, 3) the same diode SIP but with Ti anodes and body shielding (*i.e.*, the *shielded pump* hereafter).

The body shielding comprised  $\sim 1$  mm thick Ti sheet. This sheet was used to line the *pockets* of the SIP. The body was shielded from line-of-sight ions originating in the Penning cells and first-bounce neutrals from reflected the cathodes. This was done by leaning sheets of Ti on a diagonal to the foot of the element pockets. This left the top and diagonal portions of the sides of the pump plenum unshielded (*i.e.*,  $\sim 20\%$  of the pump body).

Comparisons of the standard and shielded pump were made with the same pump body and magnetic circuitry, but the pump elements were new in each instance. Dimensions of the pump elements and a description of the measurement apparatus have been

previously described.<sup>1</sup>

Experiments were first conducted on the standard pump with SS anodes. In each case we baked the pump at 250°C for ~48 hours into a turbopump. Thereafter, at a temperature of ~250°C, the SIP was started, the turbopump valved out, and the pump baked on itself at 250°C for an additional ~24 hours. The H<sub>2</sub> base pressure of the system was limited after bakeout by the H<sub>2</sub> speed of the pump and outgassing of the apparatus. All pressure data were taken using the calibrated QRGAs and are given in absolute H<sub>2</sub> pressure. Frequent electron multiplier gain measurements were taken, and the gain compensated for in the interpretation of the data.

The series of H<sub>2</sub> measurements included: i) system base pressure and pump speed measurements at various times subsequent to the initial system bakeout; ii) speed measurements at ~10<sup>-9</sup> Torr; iii) speed measurements at higher pressures in the course of pumping fixed quantities of gas; iv) system base pressure and speed measurements subsequent to pumping specific quantities of H<sub>2</sub>. The elapsed times and quantities of gas pumped in each case were not identical as we took leave from the apparatus from time-to-time. In all but one instance the pumps were operated at 7.0 kV.

Equations for calculating the speed of the pump did not include a term which compensated for the base pressure of the pump. The practice of including such a term when calculating (and reporting) speed data is utterly nonsensical as it obscures the real, useable speed of a pump at low pressures. Results of the tests are given in Table II.

When measuring speeds at the system base pressures, H<sub>2</sub> outgassing of the upper region of the CERN dome served as the source of gas. The outgassing of the upper dome region was determined by measuring the H<sub>2</sub> pressure difference across the dome aperture. In the comments section of Table II., the total outgassing from the dome is given. This number was arrived at by assuming the total dome outgassing was proportional to the total dome surface area.

The H<sub>2</sub> base pressure of the standard pump 144 hours after bakeout was  $6.4 \times 10^{-10}$  Torr. Using the dome outgassing data, we calculate that the speed of the pump was ~40 l/s at this pressure. With all pumps, speed data were taken over extended periods. In the case of the standard pump, the first high pressure speed test was conducted at  $\sim 2.6 \times 10^{-7}$  Torr. Prior to measuring the speed, ~6.6 Torr-l of H<sub>2</sub> was pumped. Thereafter, speed data were taken until ~11.9 Torr-l of H<sub>2</sub> had been pumped. The gas source was then turned off and the gas introduction system evacuated. The SIP pumped on the system for 43 hours, at which time the next base pressure measurements were taken. Thereafter, successively greater amounts of H<sub>2</sub> were pumped, etc. In instances when only a few speed datum were taken, the average (i.e., AVG.) of the data are reported. When greater than eight speed datum were taken, the standard deviations of the data are given (i.e.,  $\sigma$  of Table II). With a value of  $\sigma < 5\%$  we reported negligible (i.e., NEG) data spread.

Table II. Hydrogen base pressures and speeds at various pressures vs. the quantity of H<sub>2</sub> pumped for standard and shielded diode sputter-ion pumps.

TYPE PUMP	HYDROGEN BASE PRESS. Torr	H <sub>2</sub> PRESS. OF SPEED TEST -Torr	H <sub>2</sub> SPEED		$\int Q dt$ H <sub>2</sub> Torr-L	HOURS AFTER TEST	COMMENTS
			L/sec	$\sigma$			
Standard 270 L/sec Diode	$6.4 \times 10^{-10}$	-	ND	-	~0	144	After 48 hr., 250°C Bakeout.
	-	$2.6 \times 10^{-7}$	223	35	6.6-11.9	-	
	$1.6 \times 10^{-9}$	-	25	-	11.92	43	Dome Q ~ $4.0 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	-	$5.0 \times 10^{-7}$	207	NEG	57-74	-	
	$1.1 \times 10^{-9}$	-	23.6	-	74.2	47	Dome Q ~ $2.6 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	-	$6.0 \times 10^{-6}$	128	14	81-117	-	
	$1.1 \times 10^{-9}$	-	23.6	-	117.6	42	Dome Q ~ $2.6 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	$8.3 \times 10^{-10}$	-	31.3	-	159.5	48	Dome Q ~ $2.6 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
Standard 270 L/sec Diode w/ Ti Anodes & ~80% Internal Body Ti Shields	$1.8 \times 10^{-9}$	-	12.8	-	159.5	~49	Dome Q ~ $2.3 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	$2.5 \times 10^{-10}$	-	-	-	~0	24	After 24 hr., 250°C Bakeout.
	$2.0 \times 10^{-10}$	-	41.3	AVG.	~0	47	
	-	$2.9 \times 10^{-9}$	136	4.8	0.004	-	
	-	$1.6 \times 10^{-7}$	185	6.7	~0.23	-	
	$3.6 \times 10^{-10}$	-	47.2	-	0.241	14	Dome Q ~ $1.7 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	-	$2.6 \times 10^{-9}$	116.5	8.9	~0.241	-	
	-	$1.4 \times 10^{-6}$	235	10	6.3-8.6	-	
	$5.3 \times 10^{-10}$	-	28	4.5	8.65	16	
	$4.5 \times 10^{-10}$	-	46.7	-	8.65	39	Dome Q ~ $2.1 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	-	$2.6 \times 10^{-9}$	120	12	~8.65	-	
	-	$3.0 \times 10^{-6}$	240	12	55-61	-	
	$5.4 \times 10^{-10}$	-	-	-	61.91	28	
	$4.0 \times 10^{-10}$	-	50.0	-	61.91	72	Dome Q ~ $2.0 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	$4.2 \times 10^{-10}$	-	59.5	-	61.91	96	Dome Q ~ $2.5 \times 10^{-8}$ Torr-L/sec H <sub>2</sub> .
	-	$2.6 \times 10^{-9}$	168	16.3	~61.91	-	
	$4.3 \times 10^{-10}$	-	-	-	~61.91	20	After 2nd 24 hr., 250°C Bakeout.

We conclude that there is a gradual deterioration in base pressure of the standard pump as a consequence of pumping successively greater amounts of H<sub>2</sub>. There are two subtle and competing effects in this regard. On the one hand, the base pressure of the pump will deteriorate as a consequence of the burial of ions and neutrals in the pump anodes and pump walls. This has the effect of increasing the H<sub>2</sub> outgassing from these members, and thus decreasing the measured speed of the pump at low pressures. On the other hand, the pumping of significant amounts of H<sub>2</sub> eventually sputter-cleans the surfaces of the cathodes so that the H<sub>2</sub> pumping efficiency, and therefore speed, increases markedly in time. Therefore, the base pressure subsequent to the pumping of substantive quantities of H<sub>2</sub> may or may not be lower than prior to this pumping depending on the two competing effects. This explains why the base pressure of the pump in the middle of the series of measurements appeared to decrease as a consequence of H<sub>2</sub> pumping (i.e., an increase in pump H<sub>2</sub> speed), whereas at the conclusion of the experiment, sufficient gas had been buried in the SS members of the pump to overcome the benefits of increased H<sub>2</sub> speeds.

Results of the shielded pump test series indicated the following: 1) The base pressure of the shielded pump, after bakeout, was  $\times 2.7$  lower than that of the standard pump. 2) Subsequent to the pumping of substantive quantities of H<sub>2</sub> with the standard and shielded pumps, H<sub>2</sub> speeds in the low  $10^{-9}$  Torr range were  $\sim \times 10$  greater with the shielded pump. 3) After pumping  $\sim 0.24$  Torr-l of H<sub>2</sub> the speed of the shielded pump was 116 l/sec. This increased to 168 l/sec at the same pressure

(i.e.,  $2.6 \times 10^{-9}$  Torr) after pumping  $\sim 62$  Torr-l of  $H_2$ . However, the base pressure of the pump deteriorated by  $\sim 16\%$ . 4) Subsequent to pumping comparable quantities of gas, the base pressure of the shielded pump was in all cases  $\sim 3$  lower in pressure than that of the unshielded pump, and substantial speeds were observed at the lower pressures.

## CONCLUSIONS

From these tests we conclude the following: 1) The  $H_2$  capacity of a diode SIP with Al cathodes is  $\sim 1.7-3.4 \times 10^{-3}$  Torr-l/cm<sup>2</sup>. 2) This  $H_2$  capacity is comparable to the same pump's capacity for He (i.e.,  $2.89 \times 10^{-3}$  Torr-l/cm<sup>2</sup>). 3) Approximately 5.5% of the hydrogen ions impinging on the cathodes of a diode SIP are reflected and buried as neutrals in the anodes. 4) Substantive quantities of  $H_2$ , in the form of reflected neutrals, will be pumped in the anodes of an SIP if the anodes are made of Ti or a Ti alloy. 5) The use of Ti anodes is necessary but not sufficient to eliminate low pressure  $H_2$  outgassing effects. 6) Substantive quantities of ions and neutrals of gases are buried in the walls of all SIPs. 7) Shielding the walls of an SIP with Ti and constructing the anodes of this same material will result in a significant increase in pumping speeds for  $H_2$  at low pressures and thus improve on the base operating pressures.

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