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PUMPING MECHANISMS IN SPUTTER-ION PUMPS  
LOW PRESSURE OPERATION\*Kimo M. Welch  
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It is shown that significant H<sub>2</sub> pumping occurs in the walls of triode pumps. Also, H<sub>2</sub> is pumped in the anode cells of sputter-ion pumps. This pumping occurs in a manner similar to that by which the inert gases are pumped. That is, H<sub>2</sub> is pumped in the walls of the anode cells by high energy neutral burial. Hydrogen in the pump walls and anodes limits the base pressure of the pump.

BACKGROUND

Sputter-ion pumps (SIPs) are capture pumps. That is, they store the gases they pump. A detail treatment of the subject of capture pumps, including SIPs, is given elsewhere.<sup>(1)</sup> Sputter-ion pumps make use of a Penning discharge to ionize gases. Gas is ionized in hollow, Penning discharge "cells" by a swirling cloud of high energy electrons 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. Chemically active gases (i.e., with the cathode materials) are pumped by chemisorption and the inert gases by physisorption.

The primary physisorption mechanism is the burial of high energy neutral gas molecules. 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 molecule or atom. These energetic neutrals are reflected back from the cathodes and buried as neutrals in all pump surfaces.<sup>(2)</sup> Therefore, 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. Hydrogen is poorly chemisorbed by Al.<sup>(3)</sup> Therefore, if H<sub>2</sub> is pumped by an SIP with Al cathodes, it must be pumped by physisorption processes.

The configuration of single-cell triode and diode SIPs is shown in Fig. 1. The cathodes are customarily made of some chemically active material. The original triode SIPs had solid titanium "auxiliary" cathode plates separating the stainless steel walls of the pump from the cellular or grid-like cathodes.<sup>(4)</sup>

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The pumping of hydrogen by any sputter-ion pump is critically dependent on both the diffusivity and solubility of nascent hydrogen in the cathodes and other elements.<sup>(5)</sup> The solubility of H<sub>1</sub> in Al is approximately one part in 10<sup>12</sup> that of its solubility in Ti. The solubility of H<sub>1</sub> in 300 series stainless steel is approximately one part in 10<sup>6</sup> that of its solubility in Ti.<sup>(6)</sup> Therefore, from an H<sub>2</sub> pumping standpoint, stn. stl. is a better H<sub>2</sub>-pumping material than Al. Singleton and others were puzzled by the fall-off in hydrogen pumping speed of both triode and diode SIPs at low pressures. That is, speeds seem to fall off at a rate greater than the decrease in cell sensitivity.<sup>(7,8,9)</sup> Also, he noted that diode pumps with Al cathodes saturated very quickly.<sup>(7)</sup> However, Liu, et al., showed that a triode pump with Al cathodes would pump both nitrogen and hydrogen.<sup>(10)</sup>

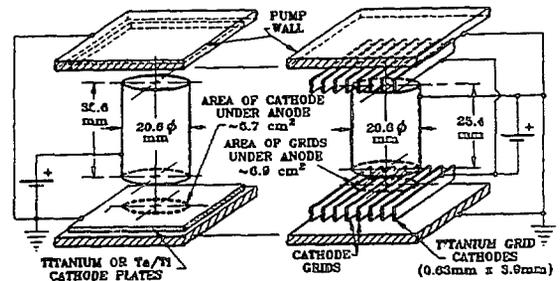


Figure 1. Single cell diode and triode pumps.

HYPOTHESES

Because of the very low solubility of H<sub>2</sub> in Al, diode pumps with Al cathodes make very poor H<sub>2</sub> pumps. Triode pumps with Al cathodes will pump hydrogen as a consequence of burial of energetic neutrals in the walls of the pump. Hydrogen can also be pumped in diodes with Al cathodes if some provision exists for the burial and accommodation of high energy neutrals in the anodes. The reason for the poor hydrogen pumping performance of all SIPs at very low pressures - regardless of the cathode material - is that reflected neutral H<sub>2</sub>, buried in elements other than the cathodes, becomes a source of outgassing at very low pressures. However the equilibrium pressure of TiH<sub>2</sub> is ~10<sup>-12</sup> Torr.

MASTER

## COROLLARIES

If a thin sheet of Ti was used to separate the gridded cathodes of a triode pump from the walls of the pump, hydrogen would not be buried in the walls and outgas at lower pressures (i.e., put the element back). If the anodes of both triode and diode SIPs were made of Ti, or some other material with high sorptive capacity for H<sub>1</sub>, the speed of these pumps would be improved at low pressures as a consequence of reduction in H<sub>2</sub> outgassing.

### TEST OF HYPOTHESES

"Saturated" pumping speed results of Liu, et al., for N<sub>2</sub> and H<sub>2</sub>, in a triode SIP with Al cathodes, are given in Fig. 2.<sup>(10)</sup> The data for Ti cathodes, were added by the writer. Note that the N<sub>2</sub> speed with Al cathodes is greater than that observed with Ti cathodes. However, the H<sub>2</sub> speed is much less than that of N<sub>2</sub>. We would have expected the steady-state H<sub>2</sub> speed to be  $\sim \times 1.2$  that of the N<sub>2</sub> speed (rather than comparable to that of He?).

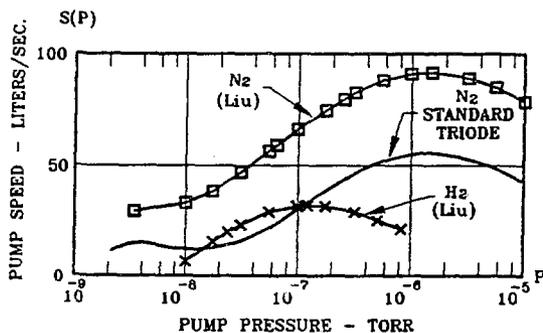


Figure 2. Nitrogen speed for a conventional triode sputter-ion pump and nitrogen and hydrogen speed for a triode pump with gridded aluminum cathodes (Liu)<sup>(10)</sup>

Assume that both H<sub>2</sub> and N<sub>2</sub> are pumped by chemisorption. If a triode pump with Al cathodes will pump H<sub>2</sub> and N<sub>2</sub> by chemisorption, then a diode pump with Al cathodes should also pump H<sub>2</sub> and N<sub>2</sub> by chemisorption. Tests were conducted with a diode pump with Al cathodes. Measurement was made for the gases N<sub>2</sub>, CO, air and H<sub>2</sub>. Results for the first three gases are shown in Fig. 3. The test vehicle was a new "100 L/sec" pump. Pure Al cathodes were substituted for the Ti cathodes. Speed measurements were taken with a CERN Dome, and the system baked to 250° C between tests. All results were steady-state.<sup>(11)</sup> In that the N<sub>2</sub> speed for the triode with Al cathodes (Liu) and diode with Al cathodes (Welch) is in fact greater than the N<sub>2</sub> speed of either pump with Ti cathodes, we may conclude that N<sub>2</sub> is in fact primarily pumped by chemisorption. This is deduced from the fact that Ar physisorption speeds of a diode pump, with Ti

cathodes, are only 2-3% that of its speed for N<sub>2</sub>.

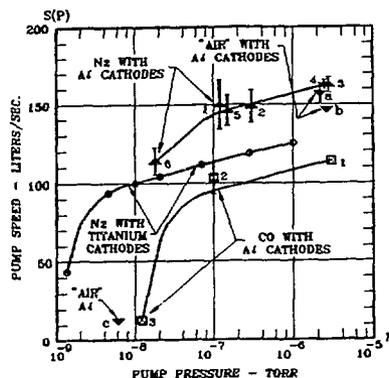


Figure 3. Pumping speed of the same diode sputter-ion pump for N<sub>2</sub>, "air" and CO; case 1): the N<sub>2</sub> speed for a diode with Ti cathodes; case 2): the diode speed for N<sub>2</sub>, "air" and CO with Al cathodes. The numbers beside respective datum represent the pressure sequence in which the measurements were conducted.

Hydrogen speed tests were conducted with the above diode with Al cathodes. After pumping  $\sim 0.06$  Torr-L of H<sub>2</sub>, the pump became very unstable and evidenced pressure "runaway" problems. The pump evidenced all of the symptoms of a conventional diode pump (i.e., not a "noble diode" pump) when pumping an inert gas. These symptoms included cyclical instabilities,<sup>(12)</sup> and pressure "run-away" (i.e., see Fig. 4). At no pressure setting  $\geq 10^{-7}$  Torr (corrected) could stable operation be achieved. The pump was very unstable after pumping  $\approx 0.19$  Torr-L H<sub>2</sub>.

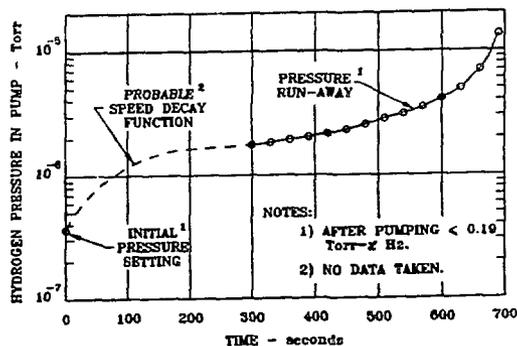


Figure 4. Decay in speed and pressure "run-away" of a diode sputter-ion pump with stainless steel anodes and aluminum cathodes after pumping  $< 0.19$  Torr-L H<sub>2</sub>.

We then replaced the anode material in the diode SIP with tubes of Ti alloy. We pumped  $\sim 6.5$  Torr-L H<sub>2</sub> at which time the speed of the pump equilibrated at  $\sim 5$  L/sec. From this we concluded that the equivalent of this amount of H<sub>2</sub> was being pumped as high energy neutrals in the Ti alloy anodes. Once the H<sub>2</sub> was sorbed in the anodes, it remained in solution. Results of the H<sub>2</sub> diode speed tests

with Al cathodes and the two different anode materials are summarized in Table 1.

Table 1.0. The hydrogen pumping speed of a diode sputter-ion pump with Al cathodes, and with both stainless steel and Ti-alloy anodes.

DATA SEQUENCE	TYPE GAS	QUANTITY PUMPED Torr-ℓ	PUMP SPEED ℓ/sec	PRESSURE TORR*	MATERIAL		NOTES
					ANODE	CATH.	
1	H <sub>2</sub>	~0.05	24	$2.2 \times 10^{-6}$	sta.stl.	Al	b
2	H <sub>2</sub>	<0.10	15	$4.0 \times 10^{-7}$	sta.stl.	Al	
3	H <sub>2</sub>	<0.10	34	$2.5 \times 10^{-6}$	sta.stl.	Al	c
4	H <sub>2</sub>	<0.10	20	$7.0 \times 10^{-6}$	sta.stl.	Al	d
1	H <sub>2</sub>	~0	181	$3.0 \times 10^{-6}$	Ti	Al	e
2	H <sub>2</sub>	0.78	98	$6.0 \times 10^{-6}$	Ti	Al	f
3	H <sub>2</sub>	1.42	50	$8.7 \times 10^{-6}$	Ti	Al	f
4	H <sub>2</sub>	1.43	28.3	$4.1 \times 10^{-6}$	Ti	Al	f
5	H <sub>2</sub>	1.98	29.3	$5.1 \times 10^{-6}$	Ti	Al	f
6	H <sub>2</sub>	3.33	$16.9 \pm 1.7$	$9.1 \times 10^{-6}$	Ti	Al	f
7	H <sub>2</sub>	3.69	5.0	$1.0 \times 10^{-7}$	Ti	Al	g,f
8	H <sub>2</sub>	6.55	$4.9 \pm 1.1$	$1.8 \times 10^{-6}$	Ti	Al	f
9	H <sub>2</sub>	6.49	$6.0 \pm 1.4$	$8.4 \times 10^{-6}$	Ti	Al	f
10	H <sub>2</sub>	6.42	0	$9.0 \times 10^{-6}$	Ti	Al	h,f

NOTES:

- a) All pressure readings corrected for H<sub>2</sub>.
- b) Unstable and problems of pressure run-away\*. Reduced pressure.
- c) Increased pressure to  $\sim 2.4 \times 10^{-7}$  Torr and observed sinusoidal pressure instabilities with  $\Delta P$  of  $\sim 1.8 \times 10^{-7}$  Torr and T  $\sim 2.5$  sec.
- d) Increased pressure to  $\sim 3.6 \times 10^{-7}$  Torr and observed pressure "run-away" shown in Fig. 3.0. Would not operate at pressure  $> 10^{-7}$  Torr thereafter.
- e) Ti-6Al-4V anodes with fresh Al cathodes installed.
- f) Though decay in speed noted, no evidence of instabilities was noted.
- g) See Fig. 4.0 for speed decay function.
- h) Base pressure of pump two days after tests.

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CONCLUSIONS

We believe that it has been conclusively shown that:

- 1) there is very little if any chemisorption of H<sub>2</sub> by Al;
- 2) there is significant chemisorption of CO, N<sub>2</sub> and other air gases by Al;
- 3) a significant amount of pumping of neutral H<sub>2</sub> in triode pumps occurs as high energy neutral burial of H<sub>2</sub> in the walls of the pump body;
- 4) a significant amount high energy H<sub>2</sub> (H<sub>1</sub>?) is buried in the walls of the diode SIP anode.

It is probable that this also occurs in the anodes of triode SIPs.

ACKNOWLEDGEMENTS

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There was initially a very high H<sub>2</sub> speed in the diode SIP with Al cathodes and Ti alloy anodes. A gradual decay in speed ensued. This probably stemmed from saturation of what I call the "implant laminate" of the Al cathodes.<sup>(13)</sup> Eventually, because of the negligible diffusion of H<sub>1</sub> into the Al, the net flux of gas becomes zero at the cathodes, and the remaining H<sub>2</sub> pumping speed component is that of high energy neutral burial in the anodes. The observed decay in H<sub>2</sub> speed, with accumulated gas load, is shown in Fig. 5.

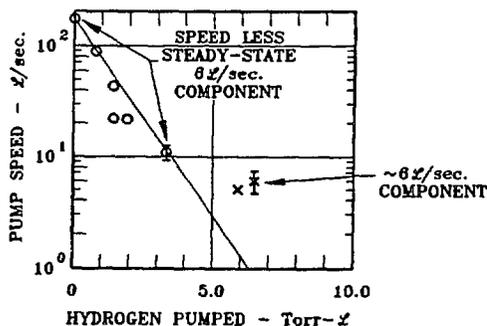


Figure 5. Hydrogen pumping speed as a function of the amount of hydrogen pumped.