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Informal Report

Some Important Developments in Capture Pumping Technology in the Last Forty Years

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R H I C P R O J E C T

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ABSTRACT

Highlights of the history of capture pumping technology are given. The subjects covered include some of the important chronological developments in sputter-ion, electrostatic-ion, sublimation, nonevaporable getter and cryogenic pumping in the last forty years. All of these forms of pumps proved to be essential tools for the attainment of ultra-high vacuum.

INTRODUCTION

It is a considerable challenge to review this subject in so brief a presentation. One approach might be to make historical commentary on the technologies that lent impetus to the development of particular forms of capture pumps. For example, microwave tubes prompted the development of sputter-ion pumps (*i.e.*, SIPs). The semiconductor industry *primed* the market for the development of today's small closed-loop gaseous helium cryopumps (*i.e.*, CLCs), whereas the larger cryopumps were developed much earlier for space simulation applications. Prior to the refinement of CLCs, Titanium sublimation pumps (*i.e.*, TSPs) were used on a commercial basis in the semiconductor industry for clean, *dry* pumping and in space simulation applications. They are presently used extensively in surface physics apparatus and in particle accelerators and colliders. Nonevaporable getters (*i.e.*, NEGs) followed decades of getter development work with applications in receiving, TV picture and transmitting tubes. They also are now widely used in particle accelerators, storage rings and colliders. All forms of the above capture pumps have been used one way or another in fusion-related work. All have been applied to attaining UHV.

How can one discuss the history of capture pumps and not give prominence to the great technologists who pioneered their development and use through the years? *Users* must be included, as certain large-scale applications of capture pumps required considerable courage and vision on the part of users. For example, Neal's successful use of SIPs to pump the two-mile Stanford Linear Accelerator was a courageous venture.[1] The decision of Benvinuti and Reinhard to use NEGs to pump the CERN LEP machine was equally courageous and successful.[2,3] Staff at IBM first championed the presently universal use of CLCs in PVD systems and ion implanters.

Lastly, we tend to forget the many companies who assumed the financial risks associated with developing and marketing these devices, and the universities and government sponsored institutions that supported research in their development. In this regard we must surely give prominence to the SAES-Getter Company, in Malano, Italy, for their development of certain NEGs. We must remember the companies of Aurthur D. Little (CTI), Cryomech, Air Products, Inc. and Leybold who pioneered the development of CLC refrigerators. N.V. Philips, General Electric, Varian and Perkin-Elmer must be recognized for their contributions to the development and refinement of SIPs. We must mention the support of the DoE, and NASA and their counterparts throughout the world, the *hands that ofttimes feed us* in our research.

This author made an effort to accomplish some of the above objectives in a recent publication. It includes some 574 references dealing with the above subjects.[4] If anything was learned in this effort it was that new pumping technologies are not *discovered*. They do not suddenly appear as historical *events*. Rather, products and devices are nurtured and grow to maturity over a period of decades. This maturation process includes many seasons of discoveries, ofttimes a process of *fumbling along*, and it includes an ample portion of serendipity. I will first briefly discuss pumping mechanisms, and then merely highlight brief interludes of the maturation processes related to some forms of capture pumps.

PUMPING MECHANISMS

Capture pumps retain gases by physisorption and chemisorption. Physisorption is a *catch-all* term which applies to a variety of physical phenomena. In the case of cryopumps, gases are physisorbed due to van der Walls' forces which exist between gas molecules or the gas and cold pumping surfaces. At a given temperature the gas might be weakly bound to the surface so that the surface population of gas is comparatively sparse, and is in equilibrium with the pressure of that gas. If the gas flux onto a cooled surface is greater than the equilibrium pressure established by the adsorption isotherm of that gas, the ensuing pumping is called *cryosorption*. *Cryocondensation* is the pumping of gas to the extent that many atomic layers of gas condensate are accumulated on the cooled surfaces. When the

efflux of gas due to the vapor pressure of an *ice* of that gas on a cooled surface is much less than the impinging flux of the same gas, *cryocondensation* pumping ensues.

The implantation of energetic gas ions or neutrals within pumping elements is another form of physisorption. This is the process whereby SIPs and ESPs (*i.e.*, Electrostatic ion pumps) capture and retain inert gases.

Chemisorption is the pumping of gas through the formation of compounds of that gas with chemically active materials proffered by the pump. Chemisorption may or may not be reversible in capture pumps. For example, NEGs chemisorb gases including H_2, CO, N_2, CO_2, etc. When pumping all gases but H_2, the gas molecules are dissociated on the surface of the NEG and thereon form stable compounds with the chemically active NEG materials. These gases stay on the surface as nitrides, oxides, or carbides until the NEG is heated. When the NEG is heated the gas atoms diffuse into and remain permanently resident in the bulk of the NEG, leaving behind renewed, chemically active surfaces. However, H_2 is chemisorbed by dissociation on the NEG surface and the subsequent room temperature diffusion of the nascent hydrogen into the bulk material. In this case, when later heating the NEG the H atoms diffuse back to the surface, recombine to form H_2, and are released into the vacuum system in a manner somewhat predictable by Sievert's Law.

CHRONOLOGY OF ELECTROSTATIC-ION PUMPS

Magnetic fields are not used in ESPs to confine electrons in the electrical discharge within the pump. Some ESPs merely ionize the gas with accelerated electrons emitted from heated filaments. The gas ions are then accelerated through a potential and are either chemisorbed by the chemically active grid materials sputtered on the walls, or they are physisorbed by implantation into the walls of the pump. These forms of ESPs were called *Electro-Ion*[5,6] and *Orb-Ion*[7,8] pumps.

The original ESP was developed by Herb, and it was first reported on in 1953.[9-12] It was called the *Evapor-Ion* pump. It differed from the above simplified ESPs in that a wire of Ti was continuously fed into the pump. This wire was heated by bombarding electrons emitted from a heated filament. The molten Ti evaporated onto pump surfaces and thereon chemisorbed the chemically active gases. The inert gases were pumped by

physisorption as in the case of the Electro-Ion and Orb-Ion pumps. The evaporation of Ti wire in Herb's pump is often credited as the harbinger of TSPs.

ESPs were manufactured and sold by companies including Consolidated Vacuum Corporation (Herb's Evapor-Ion), Granville-Philips (Electro-Ion), Leybold, and NRC-Varian (Orb-Ion). These pumps had three shortcomings: 1) most required water cooling; 2) the power required was high and invariant with pressure; and, 3) most were maintenance intensive. SIPs essentially replaced ESPs in the market by the early 1970s. A comprehensive bibliography of ESPs and SIPs is given elsewhere.[13]

CHRONOLOGY OF SPUTTER-ION PUMPS

Sputter-ion pumps have been more extensively investigated than the combination of all other forms of capture pumps. Scientific observations over a period of almost a century led to the development of SIPs. For example, Guentherschulze reported that sputtering was observed to occur in electrical discharges as early as 1858.[14] Some of the observations leading to the development of SIPs are highlighted in Table I. In 1949 Penning first looked with favor on the prospects made possible by the pumping effects of cross-field discharges.[20]

Only two decades after Penning's findings, SIPs reached a state of product maturity. Some of the important developments of the period are given in Table II. This table does an injustice of omission to the great technologists who, in the years that followed Penning's disclosures, studied the discharge characteristics of cross-field cells. These must include mention of the theoretical and experimental work of Con and Daglish,[42,43] Dow,[44] Helmer and Jepsen,[45,46] Knauer and Lutz,[47,48] Lange,[49] Redhead,[50] Rutherford,[51] and Rudnitskii,[52] to mention only a few. Lastly, the advent of ferrite magnets, which according to Casimir were discovered by accident in a Philips laboratory, resulted in significant weight and cost reductions in SIPs.[53]

NONEVAPORABLE GETTERS

The contributions of the staff and associates of the SAES-Company have been most significant to the development and characterization of the most popular forms of NEGs. The names of

Della Porta, Boffito, Ferrario, Gasparini, Giorgi, Martelli, Rosai, Sciuccati and Storey appear time and again in the literature in this regard. Some of this work is reviewed elsewhere.[54,55]

Of singular importance was the first publication of Della Porta, et al., in 1961, wherein results were first reported on the 84% Zr, 16% Al NEG alloy which later became known as the St-101® NEG.[56] Perhaps the next most important development, was the publication of Kindl and Rabusin in 1967, also of SAES, reporting on a method of bonding the NEG material to thin metal strips of either Ni-plated Fe or constantan.[57] Some of the developments and applications of coated NEG strips which followed this work are highlighted in Table III.

TITANIUM SUBLIMATION PUMPS

Early studies of the gettering properties of materials set the stage for the development of TSPs as well as NEGs. The names of Dushman, Wagner, Fast, Della Porta, and Ehrke and Slack appear time and again to those researching the literature on the chemisorption properties of various materials. Subsequent to Herb's use of evaporated Ti in ESPs,[9] and Gurewitsch and Westendorp's use of Ti in SIPs,[21] interest grew in the properties of Ti as a getter material. In 1955 Stout and Gibbons conducted the first comprehensive tests of the gettering properties of heated Ti filaments at temperatures $\leq$1200°C.[70] In these experiments they were the first to demonstrate that CH_4 was synthesized at the hot Ti filament. This was later confirmed by experiments of Gupta and Leck.[71] Clausing followed in 1961 with the first large-scale application of sublimation pumps using Ti.[72] He conceived of various clever schemes for supporting Ti filaments when they are heated to temperatures sufficiently high to produce substantial sublimation.

There are three forms of TPSs: 1) *filamentary*; 2) *radiantly heated*; and, 3) *E-gun* sources. All merely serve as a means for subliming Ti onto surfaces where it thereafter chemisorbs gas. Filamentary sources are resistively heated to sublimation temperatures. Radiantly heated sources, first reported on by Herb,[73] and later refined by Harra and Snouse,[74] heat a hollow Ti structure from within with a Mo or W filament. E-gun sources heat the Ti by electron bombardment.[75,76]

In 1961 Kuz'min patented a Ti/Mo alloy which he later

reported on in 1963.[77] It comprised a 15% Mo and 85% Ti alloy filament which had good hot-strength at temperatures where appreciable Ti could be sublimed. This filamentary source was later characterized by Lawson and Woodward.[78]

Very significant work in the characterization of TSP pumping in UHV and EHV applications has been done by Halama,[79] Ishimaru,[80] and Johnson,[81] to name a few.

The likelihood of an impinging gas being pumped on a sublimed Ti surface is called the *sticking probability*. Clausing first sought to quantify this probability for various gases and for films deposited under a variety of conditions and surface temperatures.[72] Further work was done in this area by technologists including Grigorov,[82] Sweetman,[83] Elsworth, et al.,[84] and Harra.[85] A review paper was published by Harra on the subject of gas sticking probabilities on Ti films.[86] A review of the above and additional TSP work is given elsewhere.[87]

CRYOPUMPING

Cryopumps are the simplest forms of capture pumps. The equilibrium vapor pressure of cryocondensed gases can be predicted from the Clausius-Clapeyron equation, having its origin in the second and third laws of thermodynamics.[88] All reading this paper have referenced numerous times the *classic* work product of Honig and Hook which reviewed and compiled the vapor pressures of various substances, including the gases.[89] This work was subsequently updated for the gases He,[90] and H_2.[91] All of this work is most relevant to cryocondensation pumping.

The relationship between the equilibrium pressure of a gas over a cooled surface (i.e., P) *vs.* the surface population of that gas at a known fixed temperature (i.e., σ) is called an *adsorption isotherm*. Theories are numerous for predicting adsorption isotherms of various gas-substrate systems. These include Henry's Law, the parabolic law, the BET method (taken from Brunauer, Emmett and Teller),[92] and the more recent work of Dubinin and Radushkevich (the DR method).[93] However, there is no universal theory which accurately predicts adsorption isotherms for all gas-substrate systems. That is, the BET method might work very well for certain sieve materials and gases, whereas the DR method might more accurately portray the behavior of gases on metal or glass substrates. For example, Hobson, in what I consider to be another *classic work*, found that the

adsorption isotherms of Ar, Kr and Xe on a porous silver bed very closely followed the DR method over pressure ranges >10 orders in magnitude.[94] Yet, this method did not accurately portray the behavior of He on porous silver.[95] Earlier comprehensive studies of adsorption isotherms are discussed by Dushman,[96] where in a later work Robens qualitatively classified some 13 distinct σ-P curves for given gas-substrate systems.[97]

Cryopumping technology reduces to simply devising the following: 1) methods of producing cold surfaces through the use of liquid cryogens or mechanical refrigerators; 2) practical methods for creating extended cold surface areas (*e.g.*, sieve materials) to facilitate cryosorption as well as cryocondensation pumping; and, 3) pump configurations, with staged chevrons for shielding cooled surfaces from room temperature radiation and the preferential pumping of gases on the different stages.

If one must choose, the development by Gifford and McMahon of the two-stage gaseous helium refrigerator was the singularly most important work in the area of cryopumping.[98,99] These machines were originally developed to produce liquid helium, the last stage being the Joule-Thompson expansion of He. They became known as *Collins Helium Cryostats*.[100] In a few decades, these machines were used to cool pumping arrays in today's gaseous helium cryopumps.

Theoretical treatment and descriptions of much of the hardware related to accomplishing the above three objectives have been published by Heafer,[90] and the present author.[101] Excelent abreviated cryopumping review papers are given by Bently,[102] Dawson and Haygood,[103] and Hands.[104]

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TABLE I. A CHRONOLOGY OF SOME OF THE OBSERVATIONS LEADING TO THE DISCOVERY OF SPUTTER-ION PUMPS	DATE	REF.
Philips experimented with cross-field vacuum discharge devices.	1896	15
Soddy reported on the ability of cross-field discharge devices to consume gas.	1908	16
Gaede experimented with cross-field vacuum gauges with heated cathodes. He was "troubled" by gauge pumping effects.	1934	17
Penning noted that cathode disintegration (i.e., sputtering) produced a reduction in pressure.	1935	18
Penning experimented with cross-field, single-cell vacuum devices for the purpose of sputtering thin films.	1936	19
Penning reported on the use of single-cell, cross-field devices without heated cathodes, as practical gauges and pumps.	1949	20

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TABLE II. A CHRONOLOGY OF SOME MAJOR MILESTONES IN THE DEVELOPMENT AND USE OF SPUTTER-ION PUMPS	DATE	REF.
Gurewitsch and Westendorp applied for US patent on a single-cell, cross-field SIP with either Ti, C, Mg, Al or SS cathodes.	1953	21
Gurewitsch and Westendorp reported on hydrogen pumping with titanium cathodes in a single-cell SIP.	1954	22
Gale reported on the practical application of a single-cell SIP.	1955	23
Reikhrudel, et al., reported on the first multi-cell SIP with coaxially aligned anode cells and placed in a magnet solenoid.	1956	24
Hall, Helmer and Jepsen applied for a US patent on the first multi-cell diode SIP with a planer anode array.	1957	25
Hall reported on the first multi-cell diode SIP.	1958	26
Hall reported on the SIP disclosed in the 1957 patent application and on larger pumps in the offing.	1958	27
Vanderslice applied for a US patent on a single-cell triode pump.	1958	28
Lloyd and Huffman filed for a US patent on larger pumps with "pockets" to accommodate a number of pumping elements.	1958	29
Brubaker applied for a patent on the first multi-cell triode SIP.	1959	30
Brubaker reported on performance of diode vs. triode SIPs and presented the first in-depth treatment of Ar instabilities in SIPs.	1959	31
Jepsen reported on quantitative performance of SIPs including their speeds and efficiencies for various gases.	1959	32
Jepsen applied for a US patent on diode SIPs with slotted cathodes; such pumps stably pumped Ar due to air leaks.	1960	33
Malter and Mandoli reported on Ar gas instabilities in SIPs.	1960	34
Jepsen, et al., reported SIP instabilities resulting from pumping Ar and reported on the remedy of diodes with slotted cathodes.	1960	35
Hamilton discovered that "triode" pump elements need not be operated at three distinct voltages. The anodes and pump body could be operated at ground potential and the cathodes negative.	1962	36
Neal reported on the use of diode SIPs to pump the two-mile Stanford Linear Accelerator.	1965	37
James and Tom applied for patent on the DI® diode SIP with either Ta and Ti or Ta and Cu cathode pairs. This pump, as in the case of triode SIPs, effectively pumped noble gases.	1965	38
Anashin, et al., reported on the use of distributed SIPs in an electron-positron storage ring in Novosibirsk, Russia.	1968	39
Jepsen established that the pumping of noble gases in DI® and slotted-cathode diode and triode pumps was due to the burial of energetic neutrals in pump elements. Ions, on impacting the cathodes, picked up an electron and were reflected as neutrals.	1969	40
James and Tom published noble and active gas pumping results for DI® diode SIPs with Ta and Ti cathode pairs.	1969	41

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TABLE III. CHRONOLOGY OF SOME DEVELOPMENTS & USES OF St-101® & St-707® NONEVAPORABLE GETTERS	DATE	REF.
Della Porta, et al., reported on the development of an 84% Zr, 16% Al NEG which later was defined as St-101®.	1961	56
Kindl and Rabusin reported on a method of bonding NEGs onto thin metal substrates while retaining system ductility and NEG activity.	1967	57
Della Porta and Ferrario report on the development of a "Magnetless" St-101® NEG pump with integral Bayard-Alpert gauge.	1968	58
Benvenuti and Decroux, at CERN, reported on tests with an St-101® NEG for application as a distributed pump for particle accelerators.	1977	59
Boffito, et al., reported on the development of St-707®, a NEG made of 70% Zr, 24.6% V and 5.4% Fe, and regenerating at 400°C.	1981	60
Knize, Cecchi and Dylla report on the compatibility of NEGs for use in a tokamak environment	1981	61
Lampert, et al., used ESCA to study the optimum activation temp. of St-171®, a graphite-Zr NEG.	1981	62
Benvenuti/Reinhard reported on plans to use St-101® NEGs to pump the 27 km CERN LEP e^+e^- collider, and Grobner on its success.	1983 1990	63/3 64
Hseuh, et al., studied St-707® for application in a 600 m heavy ion beam transport line.	1982/85	65/66
Audi, et al., reported on the development and characteristics of a SIP/NEG combination pump.	1987	67
Ichimura, et al., used SIMs and ESCA to study the optimum activation temperatures of NEGs including St-101® and St-707®.	1987	68
Halama and Guo demonstrated that the irreversible deterioration in the speed of an St-707® NEG stemmed primarily from pumping carbon bearing gases such as CO and CO_2 rather than from N_2 and O_2.	1990	69

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