

Submitted to: The Vacuum Design of Advanced and Compact
Synchrotron Light Sources Conference
Brookhaven National Laboratory, Upton, NY
May 16-17, 1988

CONF-580552-8

IN-SITU REACTIVE GLOW DISCHARGE CLEANING OF NSLS DISTRIBUTED ION PUMPS*

BNL-41639

E.D. Johnson and T.S. Chou

DE88 016574

Brookhaven National Laboratory, NSLS, Upton New York 11973

ABSTRACT

Based on our experience with the in-situ cleaning of optical systems by reactive r.f. glow discharges [1,2] and the conditioning and preparation of distributed ion pump (DIP) elements [3], we have sought to develop strategies for recovering from severe vacuum accidents by restoring DIP elements of storage rings such as those at the NSLS in-situ. In this paper we will describe a series of experiments conducted in a test apparatus to condition a so called 'egg-crate' DIP in-situ, (this older type element being common in older storage rings). A new untreated element which was unable to pump below 5×10^{-8} Torr in its initial condition was treated in oxygen and subsequent argon r.f. discharges utilizing the pump element as the discharge electrode producing a nitrogen pumping speed of 168 l/s at 2×10^{-8} Torr. A light bake at 75°C increased this to nearly 500 l/s at 5×10^{-8} Torr. After exposure to atmosphere the speed was reduced to nil at these pressures but subsequently recovered, without bakeout, by glow discharge cleaning.

INTRODUCTION

Distributed Ion Pumps (DIP) are a familiar and important part of the pumping complement of nearly every synchrotron facility in use today. The idea is to take advantage of the dipole magnet field to construct a sputter ion pump (SIP) which is distributed over the length of the dipole chamber. One problem which pervades the use of these pumps is that while the conventional SIP operates in magnetic fields of roughly 0.1 Tesla, the DIP is required to pump well in fields which may be over an order of magnitude greater. The result is that the physics which describes the operation and performance of the DIP is relatively poorly understood.

Historically these pumps have been developed using empirical formulae and testing the candidate designs for a particular set of machine parameters [5-10]. In a recent study by Chou [3], significantly improved pumps were developed and characterized for the operating envelope of the NSLS storage rings. The range of parameters includes magnetic fields from 0.4 to 1.2 Tesla corresponding to electron beam energies of 750 MeV to 2.5 GeV in the x-ray ring, and anode voltages up to 10 KeV. The apparatus

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*This work was performed under the auspices of the U.S. Dept. of Energy, under contract no. DE-AC02-76CH00016.

constructed by Chou for this work was in essence a full scale model of the dipole section of the NSLS rings with the additional instrumentation necessary to study the vacuum characteristics of either a particular pump, or the impact of various treatment procedures performed on it.

This apparatus provided us with a ready-made opportunity to study another familiar feature on the accelerator landscape; the up-to-air incident. More specifically we wished to investigate the possibility of utilizing an in-situ radio-frequency (RF) glow discharge as an alternative strategy for recovering from an accidental venting. The conventional method of recovering from a severe venting usually involves a bakeout followed by some period of beam induced conditioning which can add up to a considerable amount of lost beam time. For example a complete bakeout of the NSLS VUV ring would take a minimum of one week and the X-ray ring at least one month [4].

Recent results with RF glow discharge cleaning of optical systems [1,2] had shown improvement not only in the performance of the optics, but also in their associated vacuum systems. In addition it is also commonly known that the surface condition of new pumps and chambers has a significant impact on their ultimate performance, so we also considered the possibility of developing procedures for commissioning new pumps when we undertook these experiments.

It is important to be mindful of the fact that any in-situ cleaning method almost certainly has an impact not only on the pump, but on the chamber in which it is contained. Although our thinking is directed to the performance of the pump, many others have investigated the potential of in-situ cleaning of chambers by glow discharge techniques [11-17]. An in-situ argon glow discharge cleaning of the Japanese SOR-RING in 1978 [13] was so successful in enhancing the performance of their facility that this capability was designed into the Photon Factory ring [14]. A very careful study was undertaken for the European LEP machine to quantify the impact of glow discharge cleaning on vacuum system performance and electron stimulated desorption yields [15]. Although all of these previous studies were designed to evaluate chamber cleaning, they may have relevance to conditioning the pumping system as well.

At this point it is worthwhile to compare the 'philosophies' of these cleaning methods. One school of thought is to regard the discharge cleaning as a mechanical process: that is, contaminants which might either photodesorb or sputter off pump and chamber surfaces into the path of the beam are to be removed by the sputtering action of ions produced in the glow discharge. The sputtered material will then work its way out of the system with the discharge gas to a pumping port, either directly, or by a process of redeposition and subsequent resputtering repeated many times. Another approach is to 'chemically' clean the system with a reactant

produced in the plasma. For example an oxygen plasma might be used to produce atomic oxygen to react with adsorbed carbon monoxide forming carbon dioxide which will be pumped away. There is some overlap between these two concepts [16], but they provide a useful framework for this discussion.

In situations where sputter cleaning of the chamber has been chosen, a DC discharge is invariably employed. The DIP anode usually serves as the positive electrode in a noble gas discharge which will direct energetic positive ions to the surface of the chamber (and the cathode of the pump). This last point is of vital importance, since the ions must be energetic enough to sputter material from the pump and chamber surfaces, but not implant gas in the pump cathode which might later 'burp' out during operation. This is a very tricky business, where the proper discharge conditions must be experimentally determined, including the total discharge time. Further, local variations in the geometry of the DIP and its chamber will render this set of conditions a 'best average' choice which will leave some areas contaminated, and others with significant amounts of implanted gas.

In previous studies where the chemical cleaning approach was adopted, the problem of implantation was somewhat reduced by keeping the DC discharge potential high enough to produce excited species (the reactant) yet low enough to avoid implantation [17]. The success of such a technique will depend on the relative rates of these processes. One can improve the situation by using a time varying potential where the electric field gradient can be large enough to provide efficient electronic excitation, but impart negligible energy to the ions. RF discharges, such as that just described, are well known in the semiconductor industry where they are employed in a wide range of processes too numerous to mention here. The features which are of interest for the present work are the relatively high electron temperature (10,000°K or more) and low ion kinetic energies (less than 1 eV) associated with low power RF discharges [18,19]. Both of these attributes are exploited in our strategy for ion pump (and vacuum system) cleaning.

EXPERIMENTAL

To perform the cleaning, we first must decide what we wish to remove; in our case we presume that carbon contamination is our main problem. If we view the process as a principally chemical one, we must prepare a suitable reactant and deliver it to the contaminated surfaces; we choose atomic oxygen which we prepare in an RF glow discharge essentially line-of-sight to the contaminated surface. The ultimate product of this reaction will be carbon dioxide which we will pump away. This is a rather simplified representation, but with the exception of a few addition wrinkles, is pretty close to what we

wish to simulate for the purposes of this study. To that end we have utilized a vacuum system described in detail elsewhere [3], and equipment for driving the discharge which has also been previously described [2]. We therefore provide here only a brief summary of the salient features of the apparatus, shown schematically in figures 1 and 2.

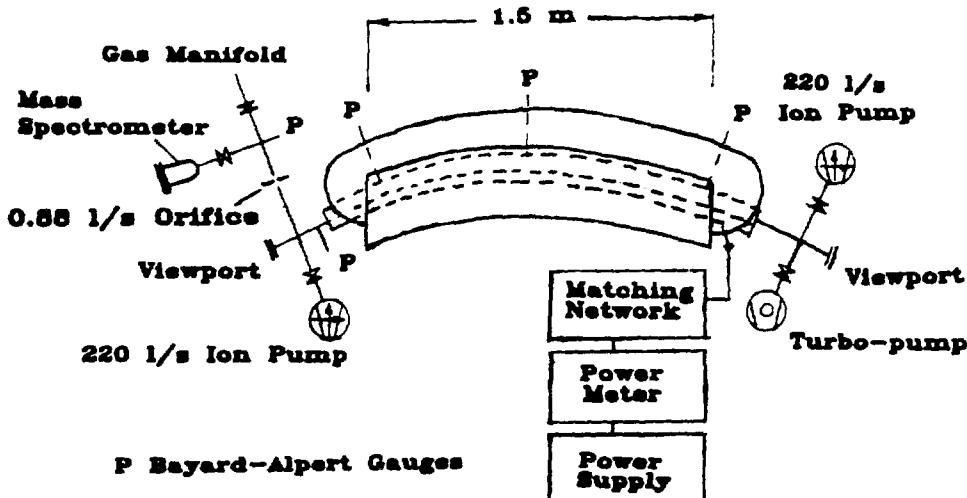


Figure 1 Distributed ion pump test stand [after ref. 3].

The test stand consists of a full scale NSLS dipole magnet with a specially fabricated arc chamber which was fitted with three magnetically shielded ion gauges, rather than the usual beam ports. One 220 l/s conventional ion pump was connected via a 20 cm cross to each end of the chamber, each pump being fitted with a 15 cm gate valve so it could be isolated from the system during the pumping speed measurements. A turbomolecular pumping station was attached to one end station for roughing out the system. Located at the opposite end station additional instrumentation provided a calibrated gas load for pumping speed measurements, which consisted of a 7 cm cross fitted with an ion gauge and a mass spectrometer, which were connected to a gas manifold through a leak valve. This instrumentation cross was in turn connected to the system by a 0.88 l/s conductance orifice. An ion gauge located in the endstation allowed the gas flowrate to be determined by measuring the pressure drop across the orifice.

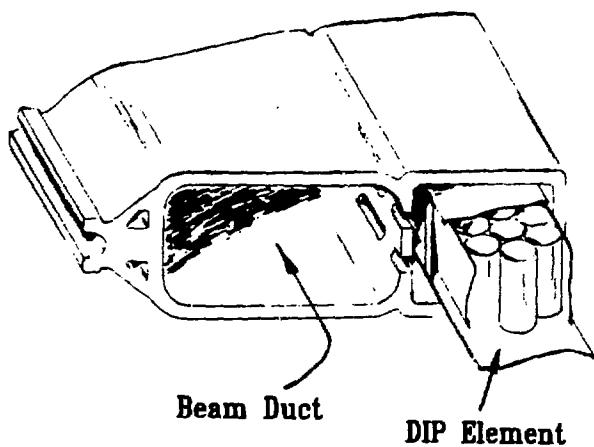


Figure 2 Cross section of NSLS Chamber and DIP.

Since the orifice is the smallest conductance aperture in the vacuum system, the speed at any point can be approximated as the ratio of the pressure drop from the high pressure side of the orifice to the local pressure, multiplied by the orifice conductance [3].

$$S(x) = \frac{C_i (P_i - P(x))}{P(x)}$$

Where $S(x)$ is the speed calculated for point x , C_i is the orifice conductance, P_i is the pressure before the orifice, and $P(x)$ is the local pressure.

The overall speed would be the integral of the local pumping speed over the length of the pump. In practice this is simply the average of the speeds determined at each of the gauges along the test stand scaled by the length of the pump to provide a speed with units of (1/sm). For measurement of the nitrogen pumping speeds described here, the magnetic field is fixed at 1.2 T and the DIP stainless steel anode biased to a positive voltage (usually +5.2 kV) with respect to the grounded titanium cathode. The pump used in this study is of the old NSLS "egg crate" design, which has been described in detail elsewhere [20].

For the discharge cleaning, an RF power supply operating at 13.56 MHz was connected via a power meter and impedance matching network to the DIP anode. The working gas, which was either oxygen or argon, entered the system from the same manifold as was used for the speed tests. As we have previously described in detail [2], the pressure during the discharge is varied from 100 to 500 mTorr to

obtain a distribution of collision trajectories and frequencies of the excited species in the plasma. Oxygen is utilized to produce atomic oxygen for the purpose of reacting with surface carbon. As will be described below, an argon discharge may be run after the oxygen treatment with the aim of chemically reducing any surface oxides which may have been formed, particularly on the titanium pump cathode.

RESULTS AND DISCUSSION

As the initial step of our study, an egg crate style pump which had not been vacuum fired or previously conditioned in any way was installed in the test chamber under a continuous dry nitrogen purge. Since the apparatus had just been used for commissioning a plate pump and was operating at 10^{-10} Torr, we felt that the major impact of any treatments we performed would be related to the new pump. As installed, the pump surfaces were dull and discolored. The system was evacuated to the low 10^{-8} Torr range with the auxiliary pumps before any voltage was applied to the DIP.

Once under vacuum we proceeded to perform the usual high voltage conditioning of the DIP which involves applying a DC bias to the pump anode in the absence of a magnetic field. The voltage is increased until a pressure burst is observed, held at that value until the pressure recovers, and subsequently increased. Although the operating voltage is 5.5 kV, we conditioned this pump up to 10 kV. Presumably this treatment results in the minimization of field concentrating protrusions, such as whiskers or sharp edges on the pump surfaces, by their ablation. This particular pump took an unusual amount of conditioning, which we attribute to its lack of prior surface preparation. It took several attempts to get the pump up to full field and voltage, an operation which was aided by the auxiliary pumps. Eventually the pump outgassing reached a steady state which allowed us to run a pumping speed test at a field of 1.2 T and voltage of 5.5 kV obtaining an equilibrium pressure of 8×10^{-7} Torr.

After this initial conditioning we ran an RF oxygen discharge between 100 and 500 mTorr with a forward power of 50 to 100 W for a total of 8 hours. The pressure was varied in 100 mTorr increments throughout the process with the power level determined by the pressure-dependant load. We observed the blue glow characteristic of oxygen discharges under these conditions extending out into the beam duct, but the discharge in the region of the pump was a pale green which has been attributed to the reduction of titanium oxide on the pump cathode [21]. As the discharge treatment, proceeded the green glow was lost in favor of blue in both the beam duct and pump chamber. After oxygen discharge treatment the titanium surfaces had

a shiny appearance, and in the absence of magnetic field, no pressure bursts were observed when the anode was biased up to 10 kV.

Although the pressure and pumping speed had improved considerably the equilibrium pressure of 2×10^{-8} Torr was still too high to be regarded as useful in application. Our visual observations of the oxygen discharge treatment lead us to believe that an oxide may have been formed on the cathode surfaces. Since electron and photon stimulated desorption of oxygen from the surfaces in this vacuum system are known to be fairly efficient processes [21,22], we decided to attempt to reduce the oxide with a noble gas discharge which would produce electrons as a 'reactant'. With these ideas in mind, we ran an argon discharge for two hours at 600 mTorr with 100 W of RF power.

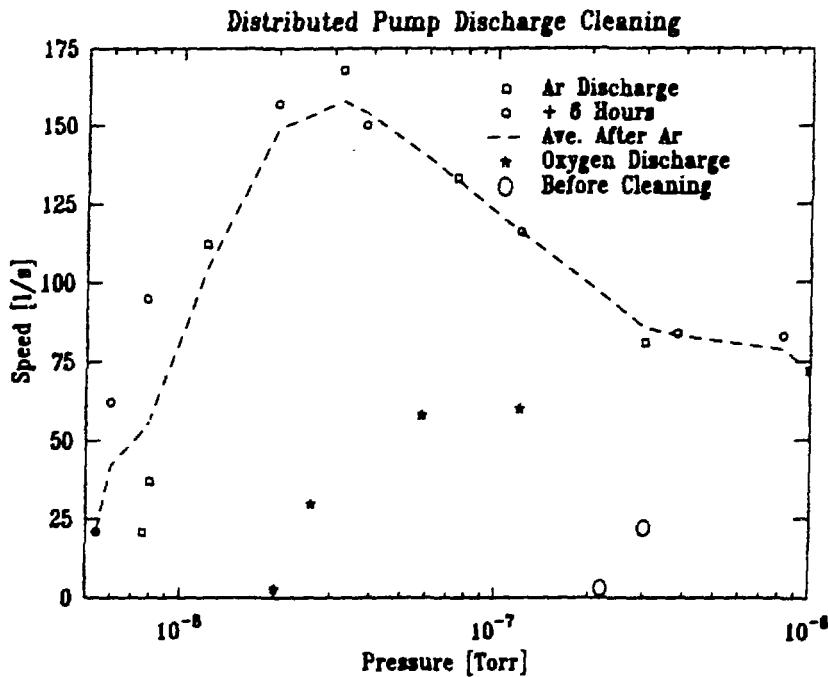


Figure 3 Pumping speed curves for the 'egg-crate' pump prior to treatment and after separate oxygen and argon RF glow discharge treatments.

After this short treatment the system pumped down almost immediately to the mid 10^{-8} Torr range. Subsequent tests showed that the equilibrium pressure was reduced to 6×10^{-8} Torr, and that the pumping speed had increased to over 150 l/s in the low 10^{-8} Torr range as shown in figure 3. Tests performed 5 days later showed that the pumping speed was essentially unchanged, and while these conditions represented a significant improvement over the original condition of the pump, they were still somewhat below the performance we would

like to obtain from a DIP. We therefore decided to bake the system to improve the base pressure.

We baked the system to 75°C for 24 hours which is relatively mild compared to the usual 100°C or more [4]. Even with this modest bake, the apparent pumping speed improved dramatically, as shown in figure 4. During the course of these tests the pump was operated for several hours at 5×10^{-6} Torr which degraded both the speed and base pressure. It is possible that the reduced pumping speed is caused by a loss in wall pumping speed as a result of saturation, or more likely by saturation of the cathode surface. Further study would be required to understand this process, since our data does not allow us to directly determine the cause of the diminished pump performance.

Discharge Cleaned and Baked to 75°C

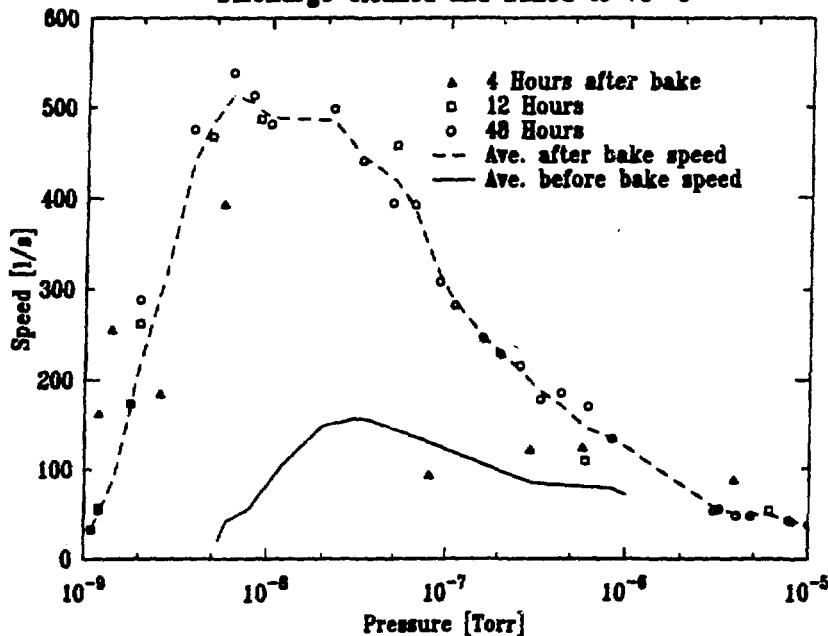


Figure 4 Pumping speed curves for the 'egg-crate' pump before and after a 75°C bakeout.

Having thus gained some experience with the behavior of this pump, we turned to simulating an up-to-air accident and attempting to recover from it by glow discharge conditioning. The results of this experiment are shown in figure 5. With the DIP and conventional ion pumps operating, the system was vented with room air to a pressure of 25 Torr for 20 minutes, then pumped back down with the turbomolecular pump to 10^{-6} Torr and all pumps restarted. In 24 hours the system had pumped itself to 3×10^{-8} Torr. After some high voltage commissioning, the pumping speeds shown in figure 5 were obtained, which were much lower than just prior to venting the system. We then ran an oxygen and argon discharges for 20 and 3 hours respectively, as described above. The performance had nearly recovered to its pre-accident

level. We did not however achieve the low pressure performance which was previously obtained by the addition of a light bake.

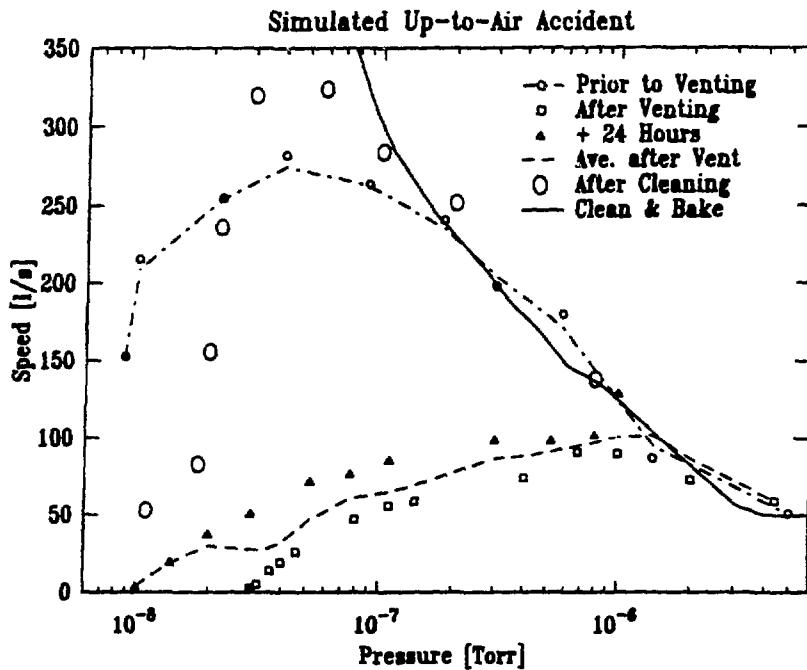


Figure 5 Simulated up-to-air accident pumping speed curves prior to venting, just after venting, and after RF glow discharge cleaning.

DISCUSSION AND CONCLUSIONS

We have shown that RF glow discharge conditioning as described in this work can improve DIP performance but we have not as yet developed the method to provide the level of performance which we have obtained either by bakeout, or with pumps of a superior design to the 'egg-crate' style utilized in this study. As a frame of reference for this statement we offer figure 6 where the pumping speed curves for the pump used in this study are compared with curves for the new NSLS plate pump [3]. These data were all taken in the same apparatus and the speeds calculated in an identical manner.

We also offer here some suggestions for future study. For example, from the behavior of our vacuum system and our experience in surface science we feel certain that the condition of the pump and chamber surfaces has a profound effect on the level of vacuum we achieve, but we are only able to infer its significance from the results of this work. A surface study with suitably designed test pump could be a big step in this direction. An additional

improvement would be to devise a way in which the behavior of the pump can be clearly distinguished from that of the vacuum chamber. Such a study might help us to understand the large drop in pumping speed we observed for the baked pump.

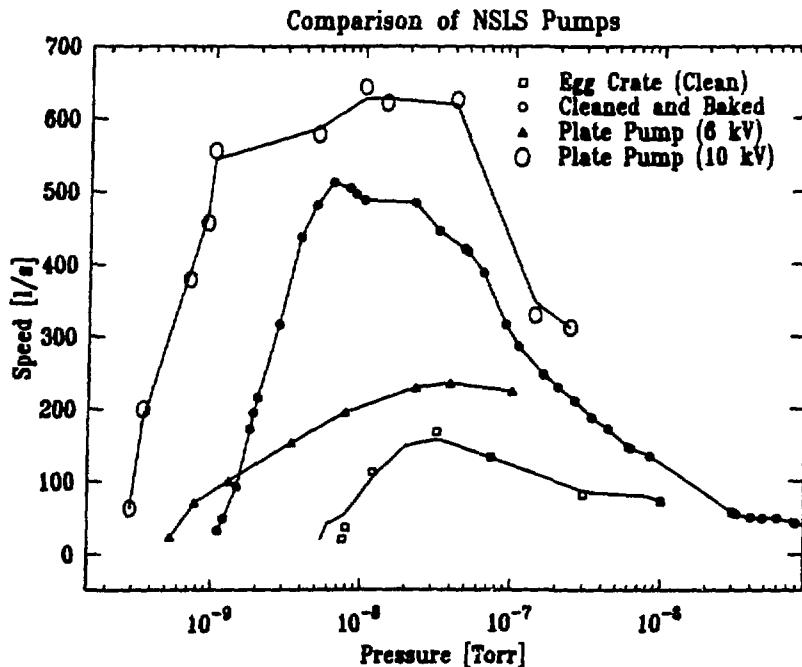


Figure 6 Comparison of pumping speed for the 'egg-crate' pump RF glow discharge cleaned both before and after 75°C bakeout, and the new NSLS plate pumps operating at 6 kV (nominally the same as in this study and in normal operation) and at 10 kV (optimum conditions).

Helium or neon might be considered for reducing surface oxides as an alternative to argon since, in machines such as the NSLS rings, ion trapping problems become more severe as the mean mass of the residual gases increases. Helium has in fact been proposed for DC discharge cleaning the LEP vacuum system [15], although the effect of residual helium on leak detection could be an important factor limiting its use. Other improvements are certainly possible.

In practice the type of accelerator to be cleaned and its operating envelope will dictate the choice of cleaning method and materials to be utilized. What we offer here should in that light be regarded as a step toward developing an alternative strategy for dealing with the NSLS vacuum system and its peculiarities. We hope that this work will serve as a useful reference frame to others endeavoring to deal with the eccentricities of their own machines.

REFERENCES

- 1 E.D. Johnson, S.L. Hulbert, R.F. Garrett, G.P. Williams, M.L. Knotek, *Rev. Sci. Inst.* 58, 1042 (1987)
- 2 E.D. Johnson, R.F. Garrett, *Nucl. Instr. and Meth.* A266, 381 (1988)
- 3 T.S. Chou, *Design Studies of Distributed Ion Pumps*, *J. Vac. Sci. Technol.* A5, 3446 (1987)
- 4 H. Halama *private communication*
- 5 M.D. Malev, E.M. Trachtenberg, *Vacuum* 23, 403 (1973)
- 6 H. Hartwig, T.S. Kouptsidis, *J. Vac. Sci. Technol.* 11, 1154 (1974).
- 7 W. Schuurman, *Physica* 36, 136 (1967)
- 8 R.J. Reid, B.A. Trickett, *Proc. 7th Int. Vacuum Congress*, Vienna, 89 (1977)
- 9 T.S. Chou, D. McCafferty, *J. Vac. Sci. Technol.* 18, 1148 (1981)
- 10 J.M. Laurent, O. Grobner, *IEEE Trans. Nucl. Sci.* 26, 3997 (1979)
- 11 A.G. Mathewson, J. Kouptsidis, L. Hipp, *Proc. 7th Int. Vacuum Congress*, Vienna, 235 (1977)
- 12 D. Edwards Jr, *J. Vac. Sci. Technol.* 15, 1586 (1978)
- 13 H. Kitamura, *Nucl. Instr. and Meth.* 177, 107 (1980)
- 14 M. Kobayashi, G. Horikoshi, H. Mizuno, *Nucl. Instr. and Meth.* 177, 111 (1980)
- 15 H. Stori, *Vacuum* 33, 171 (1983)
- 16 H.F. Dylla, *Proc. of 34th Natl. Symp. of the Am. Vac. Soc.*, Anaheim CA (1987) and these proceedings.
- 17 T.S. Chou, H.C. Hseuh, *Proc. of 10th Int. Symp. on Discharges and Electrical Insulation in Vacuum* (1982): BNL publication 31822
- 18 J.R. Hollahan, A.T. Bell, Ed; 'Techniques and Applications of Plasma Chemistry'; Wiley: NY (1974)
- 19 B. Chapman, 'Glow Discharge Processes'; Wiley: NY (1980)
- 20 C.L. Foerster, BNL publication 35940
- 21 M.L. Knotek, *private communication*; see also M.L. Knotek, *Rep. on Prog. in Physics*, 47, 1499 (1984)
- 22 A. Mesarwi, A. Ignatiev, *Surf. Sci.* 166, 75 (1986)

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