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AN ELECTRIC DISCHARGE AS A TRAP FOR
DIFFUSION PUMP VACUUM SYSTEMS

by

R. O. Adams

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ROCKY FLATS DIVISION
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ABSTRACT

An electrical discharge is used as a trap for a diffusion pump using DC-704 silicone pump fluid. The discharge cracks the pump fluid vapor thus taking it out of the system. In the process much hydrogen and some other lighter gases are evolved.

SUMMARY AND CONCLUSIONS

Use of an electrical discharge for trapping silicone fluid vapor is shown to be effective only in a limited sense. The pump fluid vapor is apparently trapped by cracking of the fluid. This effectively eliminates the heavy silicone molecules but in the process several lighter gases are evolved. There is a large increase in the partial pressure of hydrogen in the system when the discharge is operating. With a baffle between the diffusion pump and the discharge a slight improvement in operation is observed. However an increase in the hydrogen partial pressure is still appreciable.

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INTRODUCTION

Backstreaming of gases and vapors from oil diffusion pumps is a very important consideration in vacuum technique. It is well known that the ultimate pressure obtainable with a diffusion pump is limited primarily by the outgassing from the walls of the chamber and the backstreaming of vapors from the pump^(1, 2). Sears⁽³⁾ showed that although the net pumping speed of a diffusion pump is large at higher pressures, it effectively drops to zero as the backstreaming of the pump fluid becomes appreciable. Ultra-high vacuum can be attained by preventing backstreaming by proper trapping of the pump fluid vapors⁽²⁾.

To eliminate backstreaming the most common practice is to place some refrigerated walls between the pump and the working chamber. These cold walls are usually situated so that a molecule of pump fluid would have to strike the cold wall before it could get into the chamber. If the wall is cold enough, the probability is high that a pump fluid molecule would stick to the wall and thus be taken out of the system. There are many satisfactory cold-trap designs using a variety of refrigerants, e.g., "Freon" or liquid nitrogen^(1, 2). Biondi⁽⁴⁾ developed a dry-trap which used "Zeolite" or activated alumina pellets to provide effective trapping of organic and silicone pump fluids. Although well designed cold and dry-traps provide satisfactory trapping of pump fluid vapors, they all have certain disadvantages. A cold-trap must be kept cold. A "dry"-trap is only effective on a baked system where the adsorbing pellets can periodically be baked out.

(1) Dushman, S., and Lafferty, J. M., Scientific Foundations of Vacuum Techniques, New York, John Wiley & Sons, 1962, p. 20.

(2) Roberts, R. W., and Vanderslice, T. A., Ultra High Vacuum and its Applications, Englewood Cliffs, New Jersey, Prentice-Hall, 1963, p. 11.

(3) Sears, G. W., "Ultimate Vacuum of Diffusion Pumps", Rev. Sci. Instr., **20**, 458 (1949).

(4) Biondi, M. A., "High-Speed Nonrefrigerated Isolation Traps for Ultra High-Vacuum Systems", Rev. Sci. Instr., **30**, 831 (1959).

At the eighth Vacuum Symposium in 1961, Haefer⁽⁵⁾ reported on a new device which reduced backstreaming of hydrocarbon molecules. This device consists of a cold cathode gas discharge in either a magnetron or an inverted magnetron arrangement. The hydrocarbon molecules are transformed into a solid polymer by ion impact in the gas discharge. This solid polymer is deposited on the walls and acts as an adsorber for gases and vapors. Hydrocarbon molecules are effectively trapped by this mechanism. However the partial pressure of hydrogen increases as a result of the transformation of the hydrocarbons to a polymer.

Haefer worked only with a fore pump in the 10^{-3} torr pressure range. Recently a foreline trap based on this principle has become commercially available⁽⁶⁾.

This report discusses the use of a gas discharge as a trap for diffusion pumps.

EXPERIMENTAL

APPARATUS

The vacuum chamber used in these experiments was a 3/4-inch diameter glass tube 12-inches long. At one end was an ETI omegatron tube and at the other end a Veeco RG-75 ionization gage tube. The ionization gage provides measurement of the total pressure and the omegatron provides continuous analysis, if desired, of the residual gases in the system. This chamber is connected at the center of the tube to the pumps.

The pumping system consists of a 2-inch Veeco air cooled diffusion pump charged with DC-704 silicone fluid, and backed by a Welch Model 1400B mechanical pump. The discharge trap used in these experiments was a modified 5 liter/second Ultek sputter-ion pump. This pump was

(5) Haefer, R. A., "Mechanism and Properties of a New Device to Reduce Back-Streaming of Hydro-carbon Molecules", Trans. 8th Vac. Sym., 2, 1346 (1961).

(6) Bendix-Balzers Vacuum Inc., Rochester, New York.

modified to provide capability for pumping through the pump. Throughput was provided by silver soldering a stainless steel tube into the pump casing opposite the existing opening.

There are actually two groups of experiments done with this basic system. In the first group the discharge trap was placed directly over the diffusion pump. A CVC Type BCR-21 chevron baffle was placed between the diffusion pump and discharge trap in the second group of experiments. Figures 1 and 2 are schematics of these two configurations.

Figure 1. SCHEMATIC DRAWING SHOWING UNBAFFLED SYSTEM.

Figure 2. SCHEMATIC DRAWING SHOWING BAFFLED SYSTEM

Since Haefer reported an increase in the hydrogen partial pressure due to his discharge, the sputter-ion pump was chosen because of its ability to pump hydrogen. In many instances an increase in hydrogen partial pressure would be undesirable. Thus it was thought that discharge in association with the deposition of titanium would be advantageous.

PROCEDURES

The system was pumped to its base pressure without the discharge operating. When the ultimate pressure was reached the discharge was turned on. Analysis of the residual gas was made periodically using the omegatron. The system was unbaked throughout these experiments.

The ionization gage was operated continuously to provide equilibrium conditions. Low emission currents were used to minimize chemical pumping and decomposition of the residual gases⁽⁷⁾.

A permanent magnet provided a uniform magnetic field of 3800 gauss in which to operate the omegatron. A 1 μ -amp current of 90 volts electrons provided the ionization current for the instrument. A radio frequency electric potential of 1.5 volts rms was used. The omegatron gives unit resolution from mass 1 to about mass 55 in this configuration. However, larger mass-to-charge ratios can be observed with loss of resolution.

DISCUSSION

CASE I: NO BAFFLE

When the discharge was turned on after an equilibrium pressure was established in the system, an immediate increase in pressure was noted. Likewise, when the discharge was turned off a decrease in pressure was observed. Figure 3 shows the total pressure in the system changing with time as the discharge is turned off - on - off. This figure is only representative of all such occurrences. The total pressure behaved in this manner with each on-off cycle of the discharge, but of course the peak pressure in each increase was not the same. However there did appear to be a rough pattern to these happenings. As the system continued to run, the magnitude of the pressure-increases appeared to increase. The first time the discharge was turned on, the total pressure increased by only a factor of 3 or 4. However after several weeks of

(7) Winters, H. F., Denison, D. R., and Bills, D. G., "Operation of a Bayard-Alpert Ionization Gauge", Rev. Sci. Instr., **33**, 520-523, (1962).

operation the pressure would increase by several orders of magnitude when the discharge was started.

Figure 4 shows the total pressure and the hydrogen partial pressure in the system as the discharge is turned off. Immediate decreases in both readings can be noted as the discharge is extinguished.

Figure 3. TOTAL PRESSURE IN SYSTEM AS DISCHARGE IS OPERATED

Figure 4. HYDROGEN PARTIAL PRESSURE AND TOTAL PRESSURE IN SYSTEM AS DISCHARGE IS EXTINGUISHED

Representative spectra of the residual gas in the system are shown in Figures 5, 6 and 7.

Figure 5. EQUILIBRIUM MASS SPECTRUM TAKEN WITH DISCHARGE OFF. TOTAL PRESSURE 4.4×10^{-7} TORR

Figure 6. MASS SPECTRUM TAKEN JUST AFTER DISCHARGE STARTED. TOTAL PRESSURE 1.3×10^{-5} TORR

Figure 7. MASS SPECTRUM TAKEN ONE HOUR AFTER DISCHARGE STARTED. TOTAL PRESSURE 2.5×10^{-6} TORR

These three mass spectra were taken after the system had been in operation for approximately 350 hours. However, they are typical of all mass spectra taken under similar conditions. When the spectrum in Figure 5 was taken, the discharge had been off for 24 hours. The system had reached an equilibrium pressure of 4.4×10^{-7} torr, and had remained constant at that value for several hours. The composition of the residual gas had also reached an equilibrium a few hours before this spectrum was taken. Figure 5 shows that carbon monoxide and hydrogen are the principal constituents of the residual gas. The mass-to-charge ratios (m/e) 13, 14, 15 and 16 probably are CH_n ions.

When the discharge was started in the "trap" an immediate large pressure increase resulted (see Figure 3). The composition of the residual gas changes also when the discharge is started. Figure 6 is a mass spectrum taken 5 minutes after the discharge was started. It shows that hydrogen has become the principal component in the residual gas. Also masses are observed which were not present when the discharge was off. Of particular interest is the mass 3 peak. This is a sharp, very definite peak. It is not detectable without the discharge but it is detectable in significant amounts when the discharge is on. This mass has been identified as due to H_3^+ by other investigations⁽⁸⁾.

The gas composition continued to change as the discharge was operated. Figure 7 is a mass spectrum taken one hour after the discharge was started. It is seen that hydrogen and carbon monoxide are still the principal constituents of the residual gas. We notice that the contribution of masses 13, 14, 15, 16 and 17 have decreased by roughly an order of magnitude. Mass 3 has also decreased significantly as has mass 40.

We can also see a few masses appearing, $m/e = 7, 24$ and 78 . The appearance of mass 7 usually indicates the presence of nitrogen in the system. However the 12 and 16 peaks are of sufficient magnitude in

(8) Hartman, T. E., "Anomalous Peaks in the Mass Spectra of H_2 and D_2 Obtained in a Single-Focusing Mass Spectrometer", Bull. Am. Phys. Soc., **10**, 157 (1965).

relation to the 28 peak to indicate that $m/e = 28$ is primarily due to carbon monoxide. It could possibly be that the appearance of $m/e = 7$ is due to the multiple ionization of a CH_n molecule. The appearance of $m/e = 24$ along with the failure of the 26 peak to decrease in proportion to the 28 peak indicates the appearance of molecules of the type C_2H_n .

The peak indicated at $m/e = 78$ indicates that some cracking products from the pump fluid is getting into the omegatron tube. A mass spectrum of DC-704 silicone pump fluid vapor has a strong peak at 78⁽⁹⁾. The omegatron does not have sufficient resolution and sensitivity at the high mass number to say any more than that DC-704 vapor is present in the system.

Mass spectra were taken periodically following that shown in Figure 7. In 24 hours the total pressure had dropped to 1.6×10^{-6} torr and appeared steady at that value. The composition of the residual gas stayed the same as that shown in Figure 7.

After several weeks of operation, the discharge became progressively harder to start. Finally it failed altogether. The system was dismantled and the pump-trap examined and cleaned. Examination revealed large deposits of material upon the internal electrode structure. In some cases the deposits were sufficient to cause shorting between the electrodes. These deposits had the appearance of the residue observed from cracked diffusion pump fluid.

The behavior of the system, as indicated in the discussion above, indicates that the discharge pump-trap acts as a fair baffle and a poor trap and a good gas source. This behavior can be fairly easily explained. The fact that the pressure increases experienced when the discharge is turned on, themselves increase as the system continued in operation, indicates that the vapor from the pump collects on the trap structure and is released when the discharge is started. When the

(9) Hablani, M. H., and Virkus, P. L., "Residual Gases in Ultra-High Vacuum Systems with Oil Diffusion Pumps", Trans. 10th Vac. Sym., 140 (1963).

discharge is started, the pump-trap electrodes are bombarded with energetic ions and electrons. This bombardment causes some heating of the electrodes and some sputtering of the materials deposited on the electrodes. The heavy molecules collected on the internal structure can be decomposed on the surfaces to which they are attached or they may be knocked loose and then decomposed. In either case large quantities of hydrogen and light hydrocarbons are released. These gases along with the gases desorbed due to the ionic bombardment cause the increases in pressure noted.

CASE II: BAFFLED

To eliminate heavy accumulation of pump fluid on the electrodes in the discharge chamber, an air-cooled chevron type baffle was placed between the diffusion pump and the discharge chamber. This baffle is optically dense so that a molecule of pump fluid would have to strike the baffle at least once before entering the discharge chamber. Thus the pump fluid vapor condenses on the baffle surfaces rather than the discharge electrode structure.

The discharge chamber was thoroughly cleaned and baked before it was replaced in the system. Base pressures in the 10^{-7} torr range were achieved without the discharge in operation. Thus the same conditions of vacuum are obtained with the baffle as without.

Figure 8 is a representative mass spectrum of the residual gas without the discharge on. It is seen that hydrogen, carbon monoxide, carbon dioxide and water vapor are the principal constituents of the residual gas. This is common for unbaked oil pumped systems of this type.

Figure 8. MASS SPECTRUM TAKEN IN BAFFLED SYSTEM WITH DISCHARGE OFF. TOTAL PRESSURE 9.4×10^{-7} TORR

When the discharge was started, a small pressure increase occurred and the residual gas composition was as shown in Figure 9. An increase in the hydrogen partial pressure is noticed. Also a large increase in the $m/e = 12$ peak. This would indicate that some heavy molecules are present in the discharge chamber and are being decomposed.

These two spectra were taken a few hours after the initial pumpdown of the system but they are, however, representative. As the system continued to run, the hydrogen partial pressure (occurring when the discharge is started) increased further.

This is similar to the happening in the unbaffled system. However, the pressure increases in the baffled system never reached the magnitude they did in the unbaffled system.

The use of a sputter-ion type discharge appears to be useful for trapping heavy molecules. However, in the process, large amounts of lighter gases are evolved.

Figure 9. MASS SPECTRUM OF BAFFLED SYSTEM WITH DISCHARGE ON. TOTAL PRESSURE 1.2×10^{-6} TORR