GENERAL ELECTRIC
Research Laboratory

DIFFUSION-SORPTION PUMPING

by

P. L. Read and H. H. Glascock

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ABSTRACT

An extremely clean evacuation technique is described that utilizes the selective diffusion of a gas through a thin membrane combined with the persorption of gases by a chilled sorbant. Pressures lower than 1×10^{-8} torr have been achieved in a small system using the diffusion of H through Pd-25% Ag plus sorption pumping by liquid-nitrogen-chilled synthetic zeolite. The technique is especially suitable for the pre-evacuation of getter-ion pumped ultrahigh vacuum systems.

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DIFFUSION-SORPTION PUMPING

P. L. Read and H. H. Glascock

We describe an evacuation technique that utilizes the selective diffusion of a gas through a semi-permeable membrane combined with the persorption of gases by a chilled sorbant. The special virtue of this technique is its extreme cleanliness.

We have been able to reduce the pressure in a small system from 760 torr to 1×10^{-8} torr by using the diffusion of hydrogen through a palladium-25% silver foil combined with the sorption of gas on liquid-nitrogen-chilled synthetic zeolite.

The diffusion-sorption evacuation technique may be visualized by reference to Fig. 1. Initially, valves #1 and #2 are open. The original atmospheric mix of gas (at 1 atm) in the vacuum chamber is displaced by gas A introduced through the membrane, which is permeable only to gas A, by maintaining a partial pressure of gas A greater than 1 atm on the outside of the membrane. When a sufficient purity of gas A in the vacuum chamber is achieved, valve #2 is closed and gas A is then removed from the chamber via the same membrane by maintaining a low partial pressure of gas A in the gas layer adjacent to the outside of the membrane. Finally, residual gas in the chamber is removed by cooling the sorbant. At the end of the evacuation, valve #1 is closed and the main pump (if any) is turned on.

The pressure reduction produced by purging the system with gas A and its subsequent removal will depend upon: (1) the purity of gas A entering the system; (2) the thoroughness of the purge; (3) the geometry of the system; (4) gas evolution from the walls of the system; and (5) the efficiency of the removal of gas A from the system at the conclusion of the purge.

In essence, the two-way diffusion process is an extremely clean means for pre-evacuating the system prior to "turning on" the sorption pump. Several types of sorbant are suitable for the persorption phase of this technique. We chose Linde 13X Molecular Sieve, since the ability of this synthetic zeolite to persorb most gases at low pressures has previously been established.[1]

Although several gas-membrane combinations are available for the diffusion process,[2] the choice of the H-Pd system is dictated by: (1) the high diffusion rate of H through Pd;[2] (2) the extremely high purity of the H diffusion through Pd or Pd-25% Ag; and (3) the ease with which hydrogen can be removed from the system. The impurity content of H diffusing through Pd and Pd-25% Ag has been established by Young[3] to be at most a few parts in 10^{10}, even when the source hydrogen was quite impure. Moreover, Young[4] also demonstrated that essentially all of the H_2 in a closed chamber may be removed by maintaining an O_2 flow over the outside of the hot Pd or Pd-Ag foil. The Pd catalyzes the reaction of hydrogen and oxygen to form water vapor; thus, if the outside of the foil is in an oxidizing atmosphere, the H_2 concentration in the region adjacent to the Pd foil is extremely low.

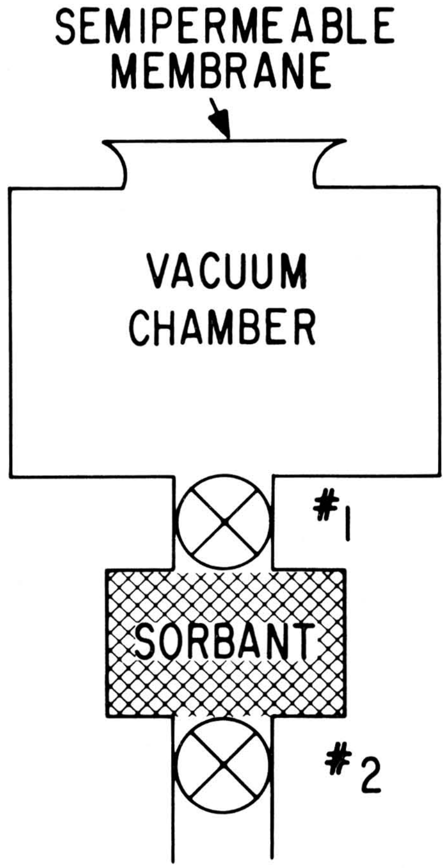

Fig. 1 Configuration of system utilizing diffusion-sorption evacuation technique.

The diffusion-sorption technique has been used to produce pressures in the high vacuum range in a small (0.5 liter) glass vacuum system starting from 1 atm of air. A Pd-25% Ag alloy (Engelhard #1812) is used instead of pure Pd to prevent cracking(5, 6) of the membrane during thermal cycling. The Pd-Ag membrane consists of a circular foil 0.004 inch thick of area 15.5 cm^2. A Nichrome heater is used to heat the foil to 400° to 450° C. The zeolite (9.6 grams) is contained in a stainless steel wire cage. The evacuation technique is as indicated above except that: (1) the system is initially purged with line H_2 through a direct connection in order to reduce the total purge time; and (2) the walls of the system and zeolite chamber are baked at 350°C during the purge.

A typical pump-down curve from atmospheric pressure is shown in Fig. 2. In this test, the system was preflushed for 20 minutes with line H_2 (flow rate 10^{-1} l/sec) admitted through the direct connection. A two-hour flush with hydrogen admitted through the Pd-Ag foil (flow rate 5.5×10^{-4} l/sec) followed the preflush. The source H_2 pressure was 2 atm At the conclusion of the purge the wall heater tapes were turned off and a flow of buffer gas (line N_2) was maintained past the foil for 5 minutes before turning on the O_2 flow. During this 5-minute period, the pressure in the system fell by about five orders of magnitude to 10^{-2} torr. Flowing O_2 past the Pd-Ag foil, plus cooling the zeolite and chamber walls toward room temperature, caused the system pressure to fall to 10^{-5} torr within 30 minutes after initiating the oxygen flow Chilling the zeolite with liquid

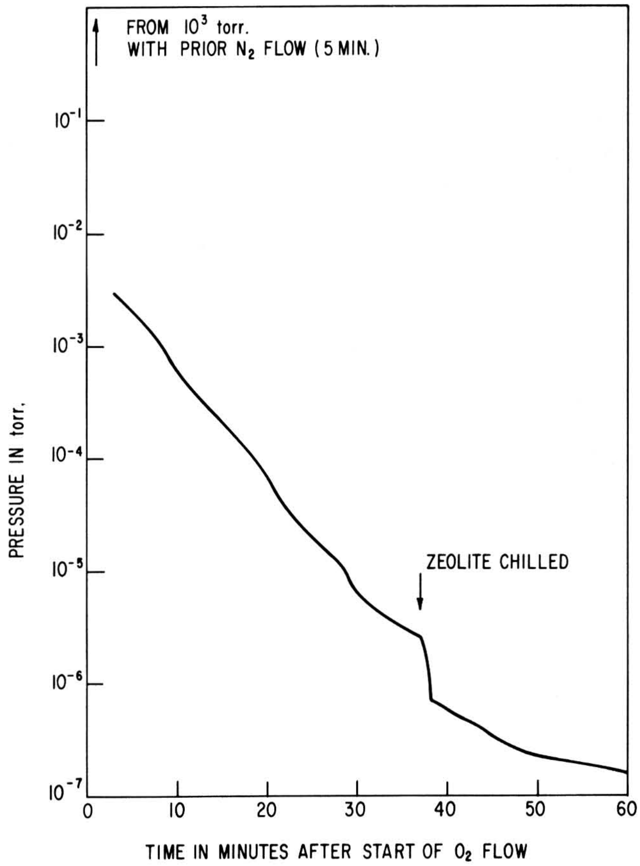

Fig. 2 Typical pump-down characteristic for small (0.5 liter) system evacuated by: (1) purging with H_2 admitted through a Pd-25% Ag membrane; (2) allowing H_2 to diffuse out through the membrane, and (3) sorbing residual gas on liquid-nitrogen-chilled zeolite (Linde 13X Molecular Sieve).

N_2 plus 30 additional minutes of oxygen flow produced a pressure of 1.5×10^{-7} torr. Pressures lower than 1×10^{-8} torr were achieved in this same system by lengthening the purge period.

The diffusion-sorption technique is especially suitable for use as a forepump in getter-ion pumped ultrahigh vacuum systems since it allows the ion pump to be started at quite low pressures. In this way the operating range of the getter-ion pump may be shifted to lower pressures since it is not forced initially to pump large quantities of gas (at high pressures) which then may be re-emitted at low pressures.[7] In addition, the diffusion technique can be used by itself to replace the sorption forepump in getter-ion pumped systems at locales where liquid nitrogen is not readily available.

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TECHNICAL INFORMATION SERIES

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pumping by liquid-nitrogen-chilled synthetic zeolite. The technique is especially suitable for the pre-evacuation of getter-ion pumped ultrahigh vacuum systems.

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