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A LARGE-SCALE GETTER PUMPING EXPERIMENT USING VAPOR DEPOSITED TITANIUM FILMS

R. E. Clausing

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R. E. Clausing

ABSTRACT

The use of titanium getter pumping for large vacuum systems is described. Techniques have been developed which give sorption rates approaching the maximum theoretically possible for many of the chemically active gases. A simple method of determining sticking fractions is described. Sticking fractions for hydrogen, deuterium, oxygen, nitrogen, carbon monoxide, carbon dioxide, and methane are given for various films. The capacity and sorption characteristics for these films are shown. Simple and reliable resistance-heated titanium evaporators are described.

It has been demonstrated that the pumping methods described provide outstanding performance and are both reliable and practical.

INTRODUCTION

The research on controlled thermonuclear fusion at the Oak Ridge National Laboratory requires very large pumping speeds for hydrogen or hydrogen isotopes at very low pressures. The vacuum pumping requirements range between 10^4 and 10^6 liters/sec at pressures from 10^{-4} to 10^{-9} mm Hg. The attainment of these very large speeds with liquid-nitrogen-trapped or zeolite-trapped diffusion pumps is feasible but would require very large and expensive equipment. The space required by such equipment places severe restrictions on the design of experimental apparatus and often would require compromising the most desirable experimental arrangement to permit the necessary vacuum manifolding and equipment.

As a result of these considerations, several other pumping methods were investigated. This report describes the equipment and results obtained thus far on a large-scale getter pumping experiment.

CONCEPT OF COMBINED GETTER AND DIFFUSION PUMPED SYSTEMS

Before describing the experiment in detail, it is necessary to describe some of the limitations on the use of getter pumped systems. (For the purpose of this report, a getter is considered to be an active metallic surface on which sorption of gases occurs. Such surfaces may be

prepared in several ways but in this instance they are deposited on suitable substrates at low pressures by evaporation from a heated source.) Since the getter surface must be prepared at low pressures, a suitable means of bringing the system to this pressure and maintaining it there during getter preparation must be supplied. The getter will not have any appreciable capacity for sorption of inert or chemically inactive gases; therefore, an auxiliary method of removing these gases to very low pressures is also required. These and other considerations lead to the very useful combination of a large getter pumping speed for the bulk of the gas load with a modest size diffusion pump system to provide the initial pump down and the pumping of chemically inert gases. It must be emphasized that the diffusion pump forms an essential part of the resultant system in that it provides the only means of handling the chemically inert gases which are inevitably present to some extent. If the diffusion pump or another means of handling these gases is not provided, they will accumulate in the system and prevent the attainment of very low pressures.

OBJECTIVES OF THE EXPERIMENT

The objectives of the large-scale getter pump experiment were: (1) to determine whether the concepts outlined above could be applied in a practical way to large devices; (2) to procure data on the operation of such a system for the determination of its usefulness; (3) to develop the techniques and equipment necessary for reliable operation of a large system of this kind; and (4) after the first three objectives were accomplished, to improve upon the system, if possible, by developing new or better techniques, materials, or system concepts.

DESCRIPTION OF EQUIPMENT AND PRINCIPLES OF OPERATION

Figure 1 shows the original experimental equipment. The vacuum tank is 36 in. in diameter and 36 in. high. It is made of aluminum and has a removable, water-cooled copper liner which fits loosely on the inside. The liner is in three sections and its original purpose was to permit easy removal of the getter deposits. It has not, however, been necessary to clean the liner during approx 150 days of operation. The 6-in. MCF 700 diffusion pump is separated from the tank by a Freon-cooled baffle (-40°C)

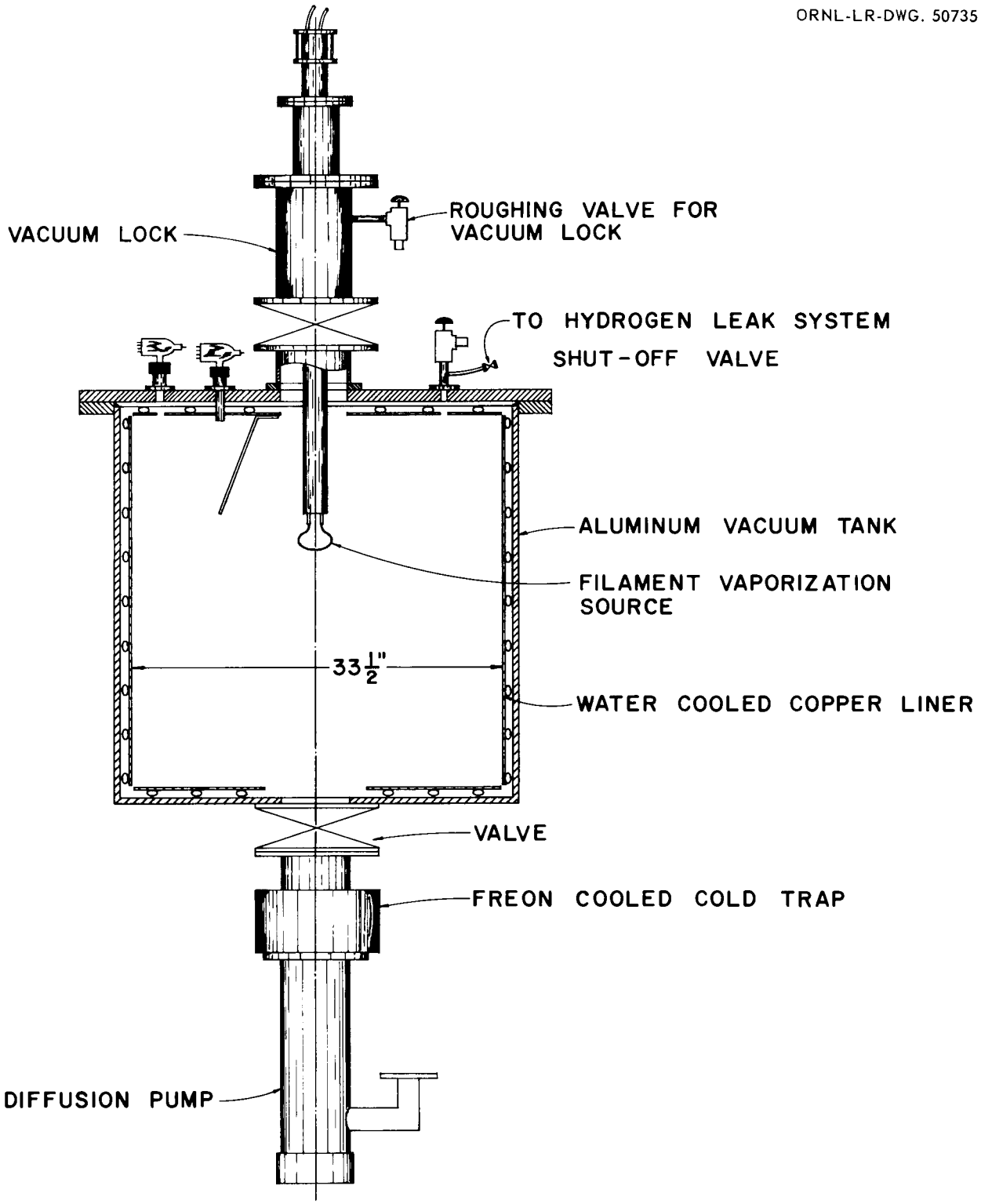

Fig. 1 Large-Scale Getter Pump Test.

and an isolation valve. A vacuum lock is provided to permit changing the getter evaporator assembly. The vacuum seals on both the tank and the lock are neoprene gaskets and O-rings with silicone vacuum grease lubrication.

For measurements of the absorption capacity of the getter films, gas was metered into the space between the tank and liner so that it entered the liner in an unknown but more or less diffused way rather than as a directed beam. Two Veeco RG-75 ion gages were used to monitor the pressure in the system. One gage measured the pressure in the liner while the other measured the pressure in the area between the tank and the liner. Actually, due to the many holes in the top of the liner, both gages normally read very nearly the same pressure. The getter is evaporated from a source near the center of the liner and deposited on the water-cooled liner. Once the deposit is established, gas is admitted at a known rate and the pressure increase noted. The volumetric pumping speed of the system may be defined as the gas input rate divided by the pressure at which the gas is pumped. Provided the sorption rate is not large (that is, only 10% or so of the incident molecules are sorbed on one collision), no serious error is caused by the system geometry. Figure 2 is a photograph of the equipment showing its size and physical arrangement.

The evaporation source used in nearly all of the experimental runs discussed is shown in Fig. 3. It is electrically heated by direct resistance. About 450–500 amp are passed through the filament at a low voltage. Approximately 1000–1500 w are required to produce the required evaporation. The source consists of a 10-in. tantalum rod, 0.170 in. in diameter, the center 7 in. of which is wrapped with one layer of close wound 0.030-in.-diam columbium wire and then two layers of 0.035-in.-diam titanium wire. The wound assembly was bent into the shape shown for convenience in passing it through the vacuum lock. The purpose of the columbium wire is to alloy with the titanium and raise its melting point, thereby permitting faster, more easily controlled evaporation. This source will evaporate about 3 g of titanium per hour and may be used for at least 4 hr at this rate.

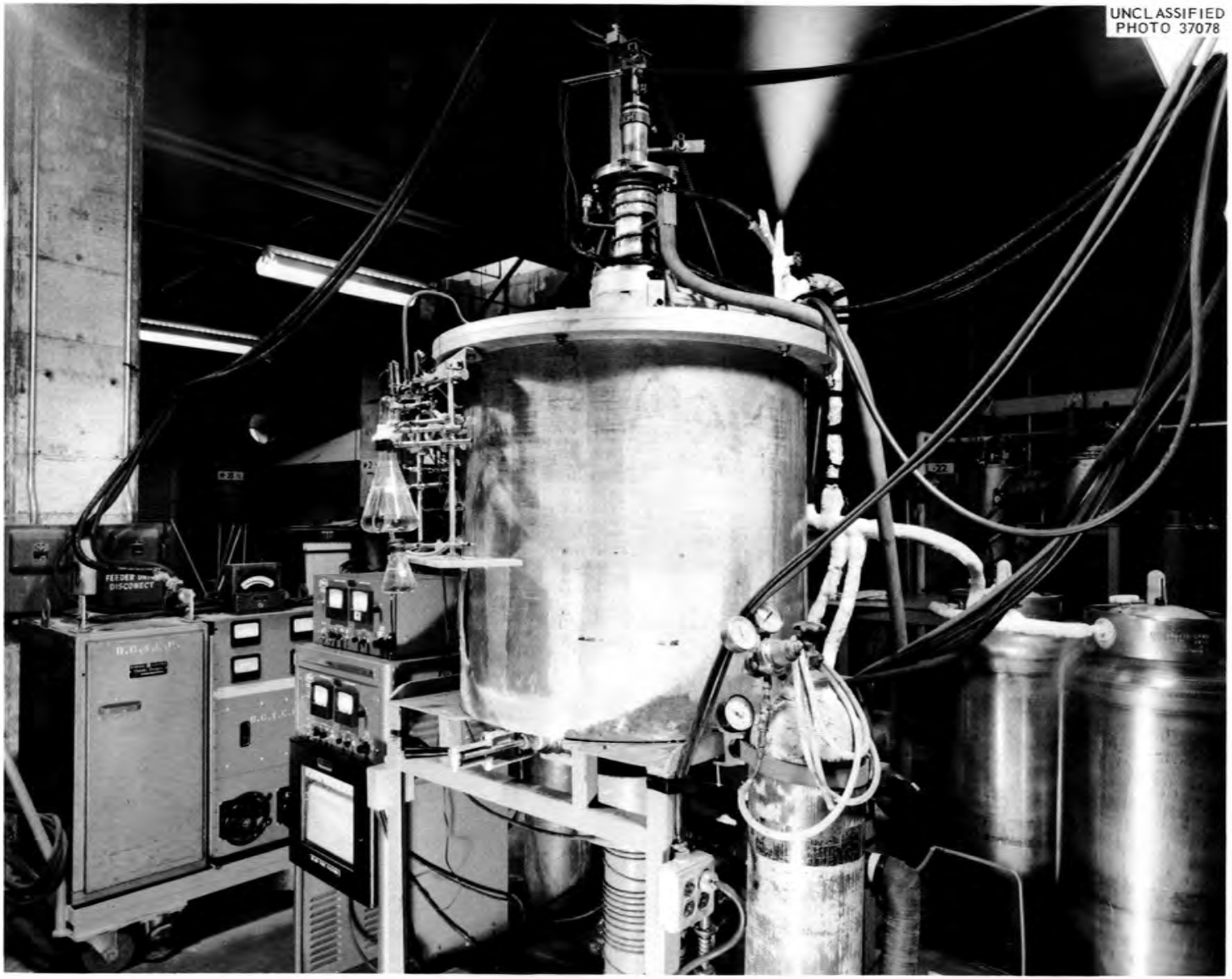

Fig. 2 Photograph of the Large-Scale Getter Pump Test During an Evaporation.

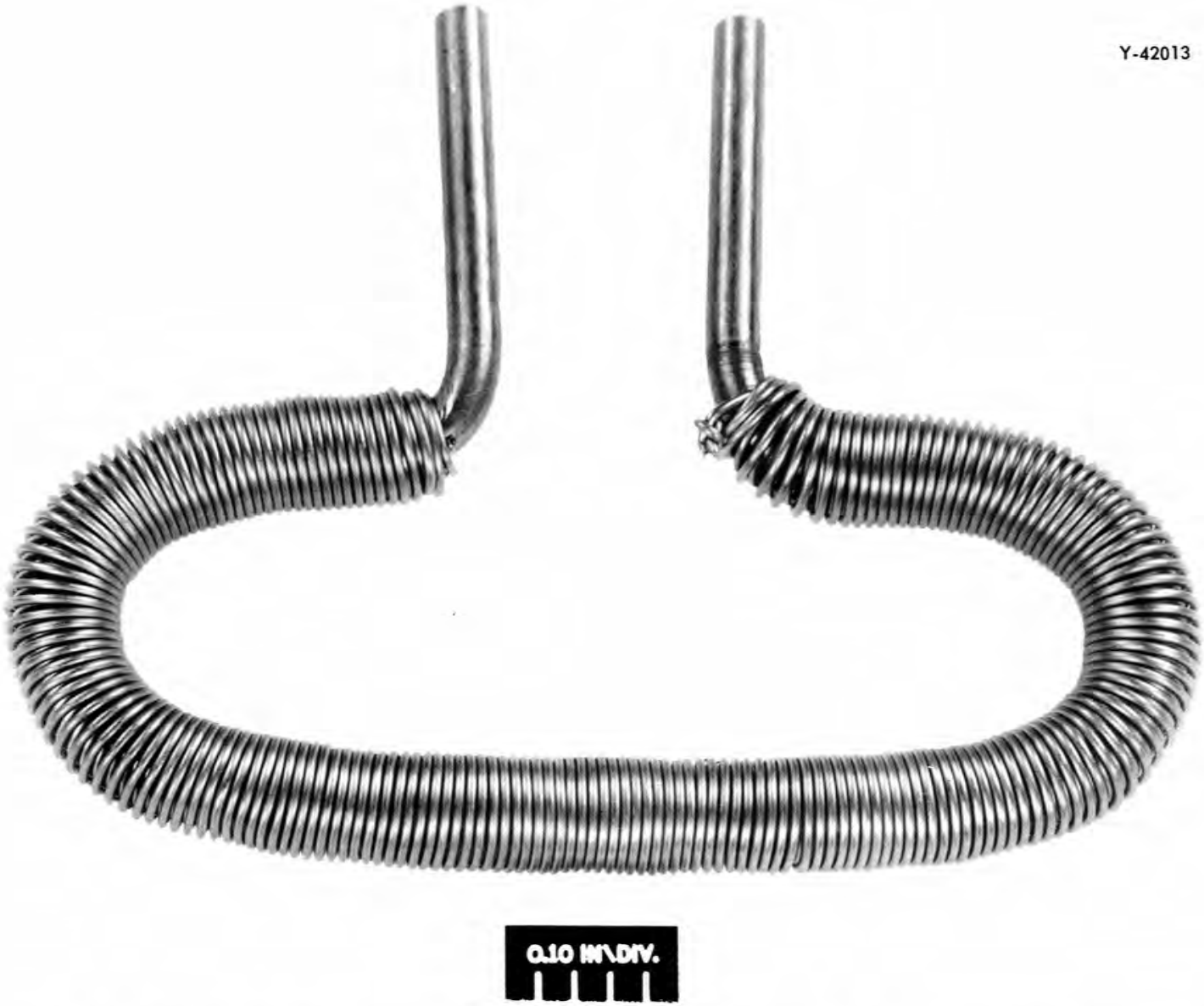

Fig. 3 A Source for Evaporation of Titanium which is Heated by Electrical Resistance.

A larger version of this filament with an active length of approx 35 in., wound into a spiral shape, required approx 7500-w power input at 500 amp and evaporated at a rate of 16 g/hr.

Titanium was chosen for these experiments on the basis of the information in the literature[1,2] and a few simple experiments with several other materials. It is not meant to imply that other materials cannot be found that will produce results as good or better than those reported herein.

Figure 4 shows a homogeneous filament made of an alloy of tantalum and titanium. Evaporation rates from this filament per unit surface area are comparable with those obtained from the previously described sources. These alloy sources are potentially capable of considerably higher evaporation rates than pure titanium wire due to the fact that they can be operated at higher temperatures. The alloys can also be formed into wire and heating power may be supplied directly from a Variac-type auto-transformer. A somewhat more detailed discussion of both of the sources described above has been published.[3]

The feasibility of large-scale getter pumping, many of the operating characteristics, and considerable improvement of techniques were established with the equipment described. There were, however, two major difficulties with this apparatus: There is a serious difficulty in interpretation of very high sorption rates; and, also, base pressures in the original system were limited to about 3×10^{-8} mm Hg due to outgassing of system components. The base pressure limitation is not serious for many experiments and has been recently lowered considerably through the use of a bakable liner inside the water-cooled liner. The bakable liner is isolated from the rest of the system by a zeolite trap. The difficulty in interpretation of high sorption rates has been overcome by some modifications to the gas inlet and measuring systems as suggested by the analysis which follows.

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[1]S. Wagner, J. Phys. Chem. 60, 567 (1956); 61, 267 (1957).

[2]B. M. W. Trapnell, Proc. Roy. Soc. (London) 218A, 569 (1953).

[3]Thermonuclear Proj. Semiann. Progr. Rept. July 31, 1960, ORNL-3011, p 122.

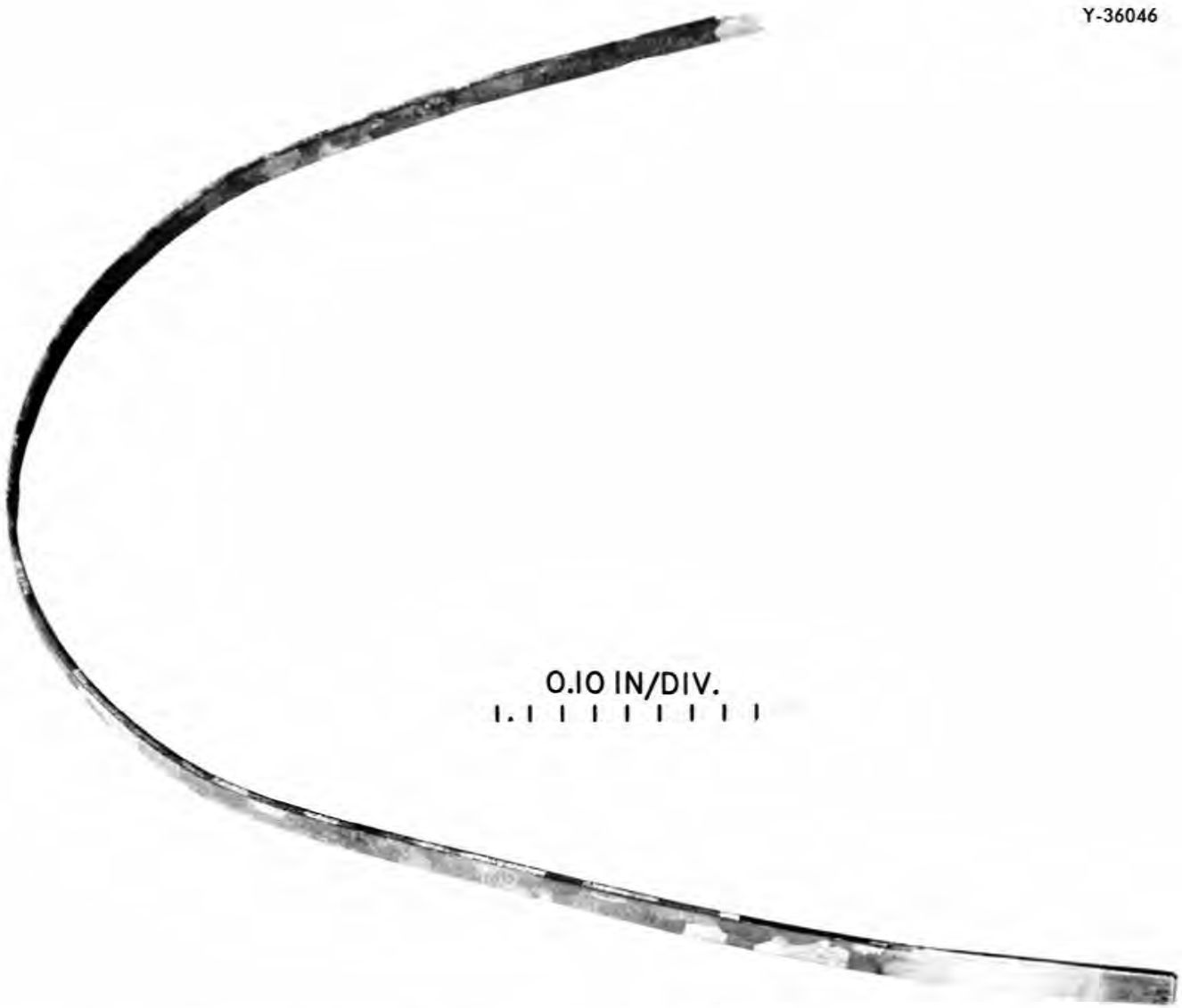

Fig. 4 A Homogeneous Filament Source for the Evaporation of Titanium Made of an Alloy of Tantalum and Titanium.

Let us consider the hollow spherical geometry, as shown in Fig. 5, with gas continuously emitted uniformly in all directions from a source in the center of the sphere. The inside surface of the spherical shell is coated with a material which absorbs a certain fraction (α) of the gas molecules incident upon it each time they strike the wall. One may then write that the total number of particles impacting on the wall per second per unit area (n_T) is equal to

$$n_T = n_e + (1-\alpha)n_e + (1-\alpha)^2 n_e + (1-\alpha)^3 n_e \ldots \tag{1}$$

where n_e equals the number of particles emitted from the source per second divided by the inside area of the sphere and $(1-\alpha)$ equals the ratio of the particles leaving the absorbing surface to those striking it. The term $(1-\alpha)n_e$ then equals the number of molecules that survive the first collision to strike the wall again and $(\alpha-1)^2 n_e$ equals the number of molecules that survive the second collision to strike the wall a third time. This series converges to a finite limit for all values of α between zero and one so that the total flux against the wall per second per unit area is given by

$$n_T = \frac{n_e}{1-(1-\alpha)} , \tag{2}$$

or

$$n_T = \frac{n_e}{\alpha} , \tag{3}$$

and

$$\alpha = \frac{n_e}{n_T} . \tag{4}$$

Equation (4) is quite reasonable since it simply states that the sticking factor, α, is equal to n_e (which in the steady state is the number of molecules introduced into the system per unit time per unit area and must, therefore, also be the number sorbed per unit time per unit area) divided by the total number of collisions with the wall.

Now consider the flux of particles onto a small surface (A) such as that shown in Fig. 5 so that particles in free molecular flow from the source cannot strike this surface except after first striking the sorbent wall of the sphere. Due to the symmetry involved, this surface will intercept particles which have made one or more collisions with the wall

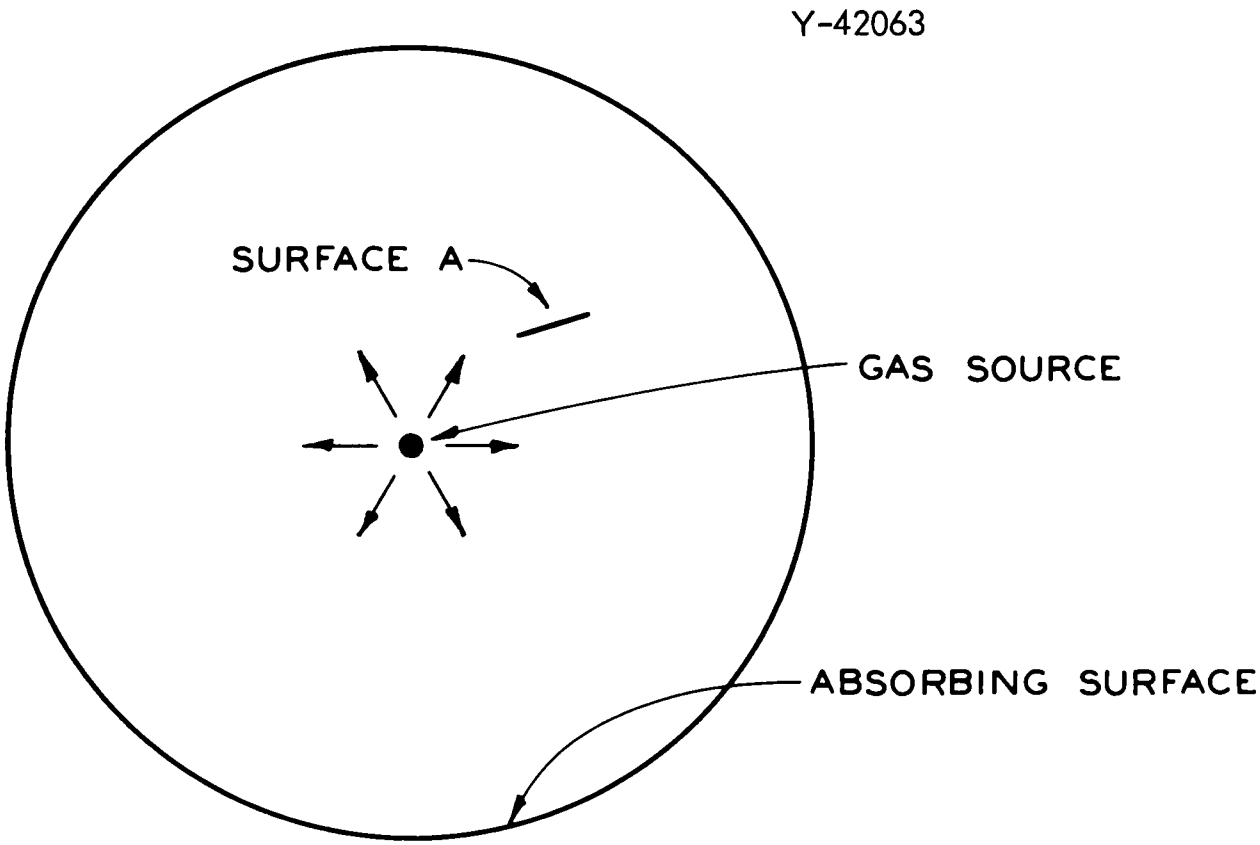

Fig. 5 Ideal Spherical Geometry.

but none of the particles directly from the source. The flux of particles onto this surface per unit area is the same as that onto the sorbent wall if the flux of particles coming directly from the gas source is subtracted. The flux of particles onto this surface per unit surface area is then simply the summation of all of the terms on the right side of Eq. (1) except n_e. If this flux of particles is defined equal to n_g,

$$n_g = (1-\alpha)n_e + (1-\alpha)^2 n_e + (1-\alpha)^3 n_e \ldots\ldots \tag{5}$$

then substituting Eq. (5) into (1)

$$n_T = n_e + n_g \tag{6}$$

and substituting Eq. (6) into (4)

$$\alpha = \frac{n_e}{n_e + n_g} \tag{7}$$

or

$$\alpha = \frac{1}{1 + n_g/n_e} . \tag{8}$$

Now, n_e can be measured if a metered gas leak is used and n_g can be determined from an ion gage connected to an oriface in the plane of the small surface (A). The flux of particles into the gage oriface is assumed equal to the flux out of the gage oriface; therefore, one can determine the flux of particles n_g from classical kinetic theory based on the pressure and temperature in the gage.

The small values of α computed from the original experiment contain only a small error; however, as α becomes larger than 0.1, calculations based on the original geometry contain an increasing error. The numbers reported below are from a modified experiment which is shown in Fig. 6 except where noted otherwise. While the geometry is not the ideal spherical geometry described above, it is considered a good approximation for sticking probabilities up to 0.5 and should not be seriously in error for even larger sticking probabilities.

The gas source consists of many small holes drilled near the plugged end of a 1/8-in. stainless steel tube. There is an arrangement to protect the source from titanium deposition whenever required. The ion gage opens into the system so that it is exposed to a large part of the interior surfaces but not to the primary gas inlet. The ion gage opening is also protected against titanium deposits both by its self-shielding and a mechanical shutter arrangement.

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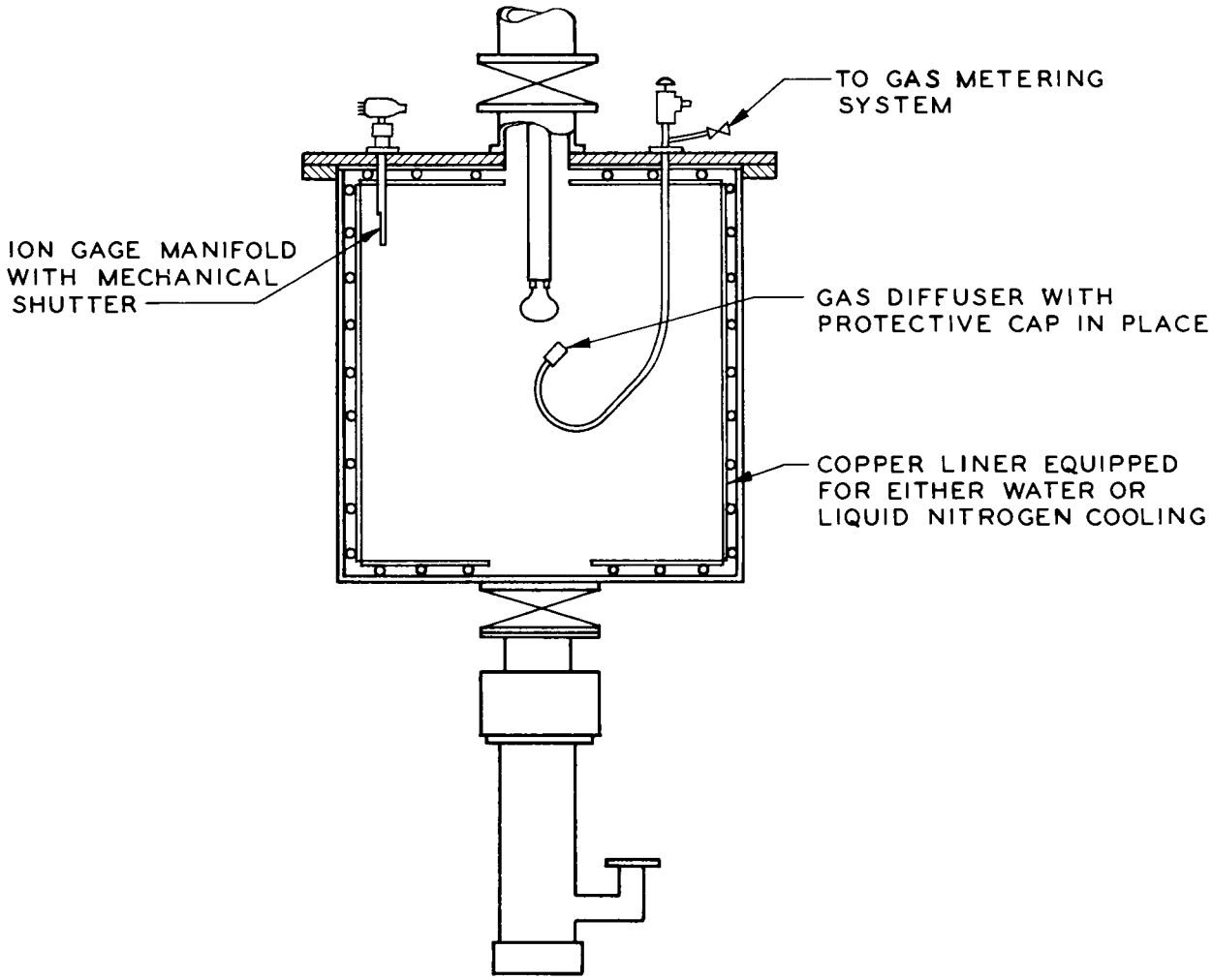

Fig. 6 Modified Large-Scale Getter Pump Test.

DISCUSSION OF EXPERIMENTAL PROCEDURES AND RESULTS

For simplicity in reporting, all of the data are reported in terms of the sticking factor, α. Specific speeds in liters per second per square centimeter at 25°C can be obtained by multiplying α by the constants F_{25} listed in Table 1. Total surface pumping speeds for this system are obtained by multiplying α by the constants, K_{25}, also listed in Table 1.

The ability of evaporated films to sorb gases may be influenced by a large number of variables. The data reported below are quite reproducible and typical for the conditions described. It must be realized, however, that other conditions not fully specified herein may cause significant variations in α. One must therefore be cautious in applying these data to other systems.

Sorption During Continuous Evaporation

The first experiments consisted of evaporating titanium and simultaneously admitting a gas to determine its rate of sorption. A typical curve for the sorption of hydrogen is plotted on this type of deposit and shown in Fig. 7. The sticking factor, α, is plotted as a function of the gas-leak rate for a constant titanium evaporation rate. The ordinate on the right indicates the volumetric sorption rate for the system.

The vertical portion of the curve at high leak rates is due to saturation of the film as it is deposited. For hydrogen under these conditions, approximately one molecule of hydrogen is sorbed for every titanium atom evaporated indicating that TiH_2 is being formed. The portion of the curve at lower leak rates indicates the sticking factor, α, increases as the ratio of hydrogen molecules sorbed to titanium atoms evaporated becomes smaller. A definite sticking factor is established for each titanium-to-hydrogen ratio. This sticking factor seems to approach some limit as the hydrogen-to-titanium ratio approaches 0.02. Due to uncertainties in the base pressure correction for these data, this conclusion is only tentative, however, and will be verified by experiments at lower pressures. Figure 8 is a plot of α vs the ratio of gas-leak rate

Table 1. Constants for Conversion of the Sticking Factor (α) to Specific Speeds and to Surface Pumping Speeds

Gas Species	F_{25} (liters/sec-cm^2)	K_{25} (liters/sec)
Hydrogen	44.2	1,530,000
Deuterium	31.2	1,080,000
Nitrogen	11.9	410,000
Carbon Monoxide	11.9	410,000
Oxygen	11.1	383,000
Carbon Dioxide	9.5	327,000

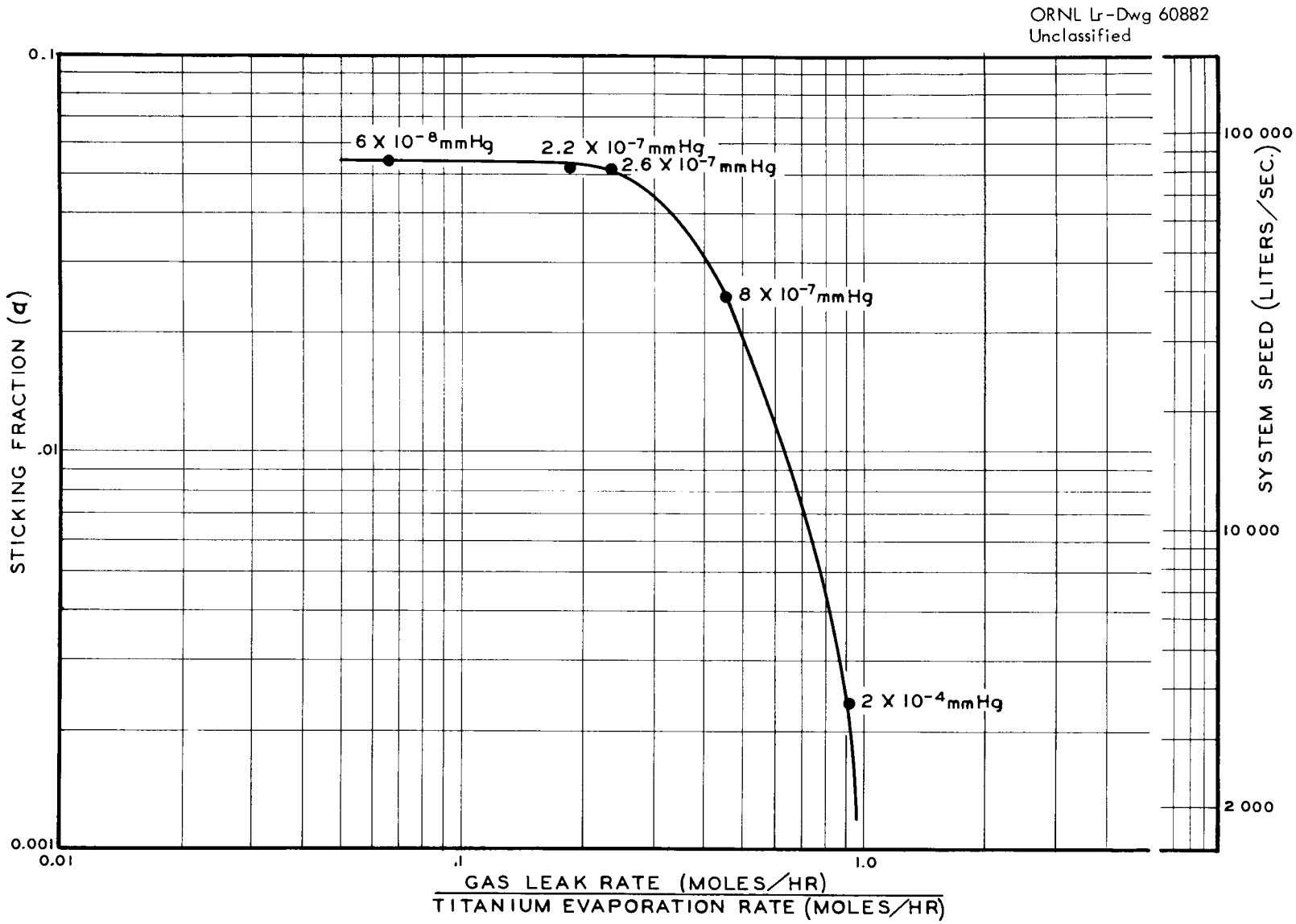

Fig. 7 Sorption of Hydrogen onto the Films Formed During Continuous Evaporation of Titanium onto a Surface at 10°C. Titanium evaporation rate = 0.018 mole/hr.

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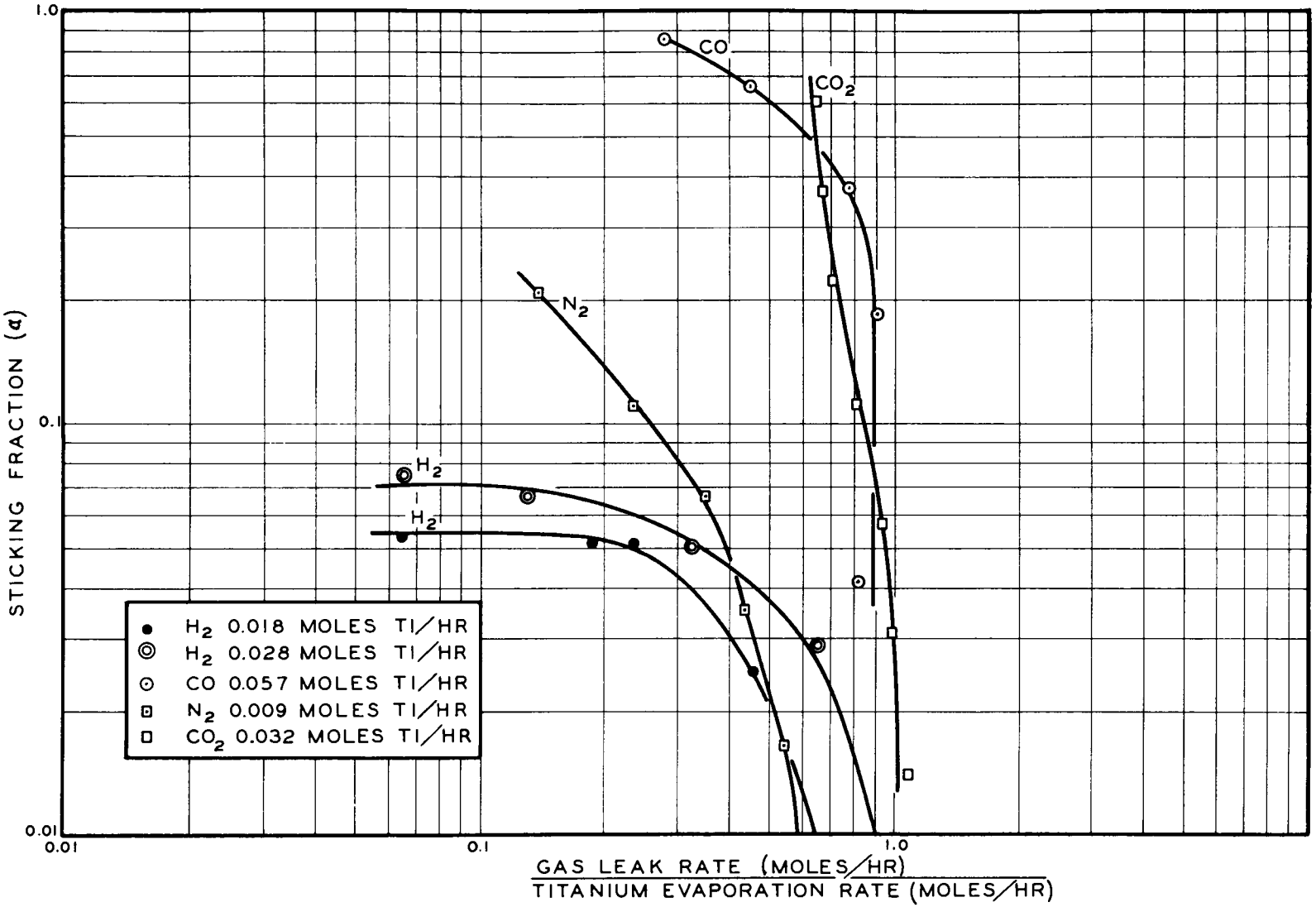

Fig. 8 Sorption of Gases onto Films Formed During Continuous Evaporation of Titanium onto a Surface at 10°C.

to titanium-evaporation rate for nitrogen, carbon monoxide, and carbon dioxide. Methane and several inert gases are not measurably sorbed under these conditions and are pumped only by the diffusion pump.

Sorption onto films produced with higher evaporation rates have somewhat higher sorption rates. These data for hydrogen are also shown in Fig. 8.

The titanium films formed in high vacuum depositions on the water-cooled substrate are metallic in appearance and have a well-developed crystal structure as evidenced by sharp x-ray diffraction patterns.

Sorption onto Films Previously Deposited in High Vacuum

A second useful pumping technique requires the deposition of the film to be completed before the gas sorption is begun. The data from this technique are best plotted as α vs Q, where Q is the quantity of gas sorbed on the film before the data were obtained. Figure 9 presents data for several gases. These data for sorption of nitrogen and carbon monoxide compare favorably with that obtained by other investigators.[4,5] Note that for hydrogen, two curves are given. The curve with the larger sorption rate was obtained on a film formed with a very high titanium deposition rate (16 g/hr). Both hydrogen curves indicate that diffusion of hydrogen into the film occurs readily so that hydrogen has easy access to a very large portion of the deposit. Although no data are presented, the curves for the other gases shown are not expected to be very dependent upon the thickness of the deposited film since these gases are likely to diffuse much more slowly into the film. Because of diffusion, the sticking factors are somewhat influenced by the sorption rates. For most measurements, these rates were approx 10^{-5} or 10^{-6} moles/sec.

If a small amount of oxygen or nitrogen is sorbed onto a film and subsequently the film's ability to sorb hydrogen is measured, it is very drastically reduced from the value obtained when hydrogen is sorbed onto a new deposit.

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[4]M. A. Biondi et al., Ultrahigh Vacuum Techniques, NYO-2904, Annual Report (April 30, 1960).

[5]A. Klopfer and W. Ermrich, Vacuum 10, 128 (1960).

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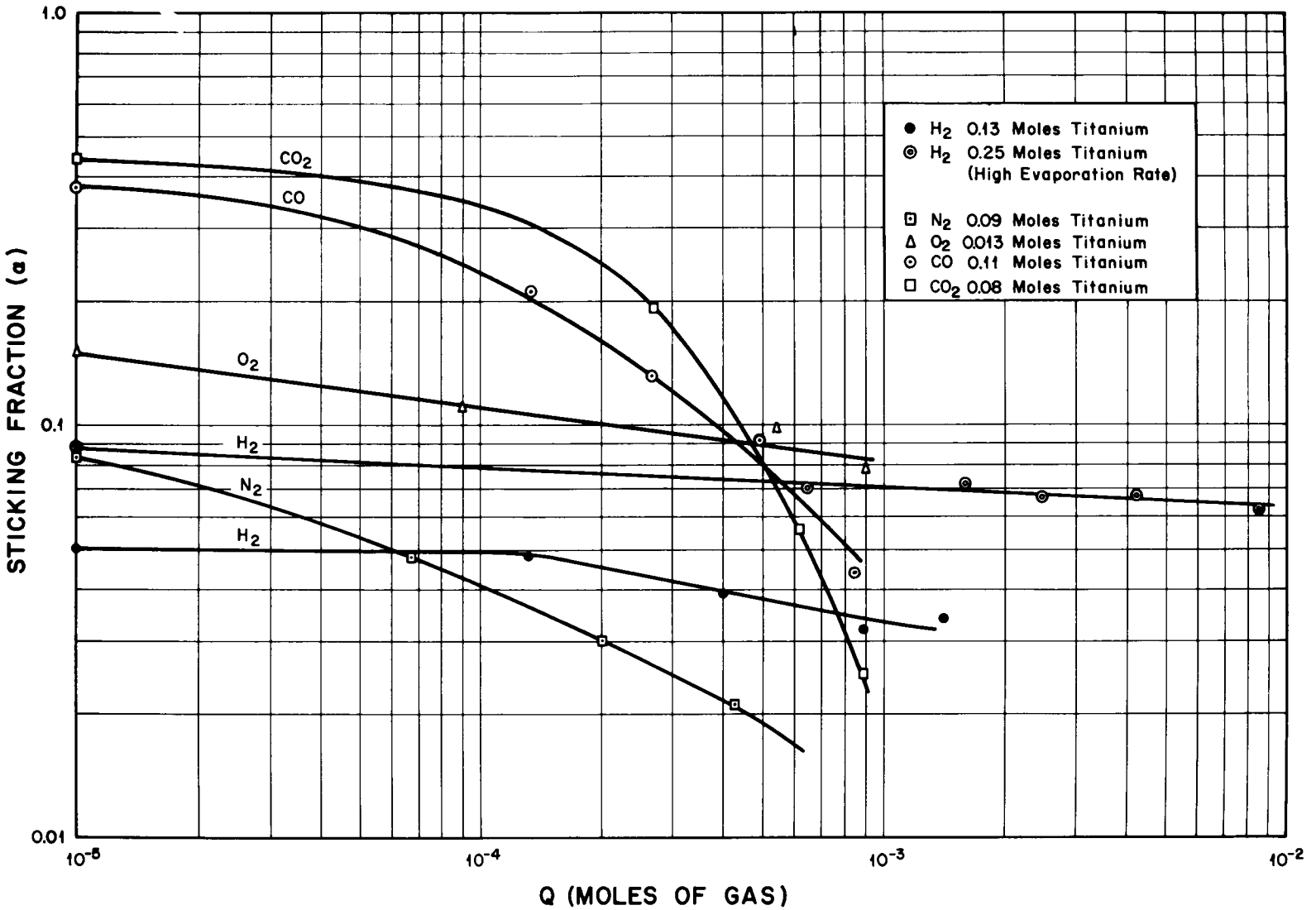

Fig. 9 Sorption of Gases onto Films Formed by Titanium Evaporation in High Vacuum onto a Surface at 10°C.

Sorption onto Films Deposited in the Presence of Inert Gas

Evaporation of titanium films in a pressure of approximately a micron of helium or argon pressure produces deposits, in the system described, which sorb gases much more rapidly than films deposited under similar circumstances in high vacuum. Data obtained from these evaporations are shown in Fig. 10. This improved sorption rate is associated with a very pronounced change in the appearance and structure of the films. The films obtained in these evaporations have a velvety black appearance and are very poorly crystallized. The increased surface area of these films and their increased surface activity undoubtedly favorably affect the sorption processes.[6] It must be noted that the conditions required to obtain this type of deposit may be appreciably different for other system sizes or other geometrical arrangements.

Sorption onto Films Deposited on Substrates at Liquid Nitrogen Temperature (-195°C)

The high vacuum deposition of titanium onto substrates at liquid nitrogen temperatures produces films which have a metallic appearance but have poorly formed crystal structures and high sorption rates. The results of sorption measurements during continuous deposition are given in Fig. 11 (ref 7). Figure 12 is a plot of α vs Q for sorption onto films after their preparation and Fig. 13 shows data for the sorption of hydrogen, deuterium, and nitrogen onto films deposited in a 2.5-μ pressure of helium with the substrate at liquid nitrogen temperature. These latter films had the greatest sorption rates for hydrogen of all of the films tested and had the same velvety black appearance and poorly developed crystal structure as similar films deposited at 10°C.

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[6] O. Beeck, A. E. Smith, and A. Wheeler, Proc. Roy. Soc. (London) 177A, 62–89 (1940–41).

[7] It can easily be shown that no correction for temperature need be made in the α obtained by the method used in this experiment. The temperature of the incident gas must be considered, however, in the calculation of pumping speeds. The numbers in Table 1 are intended for use in room-temperature systems only.

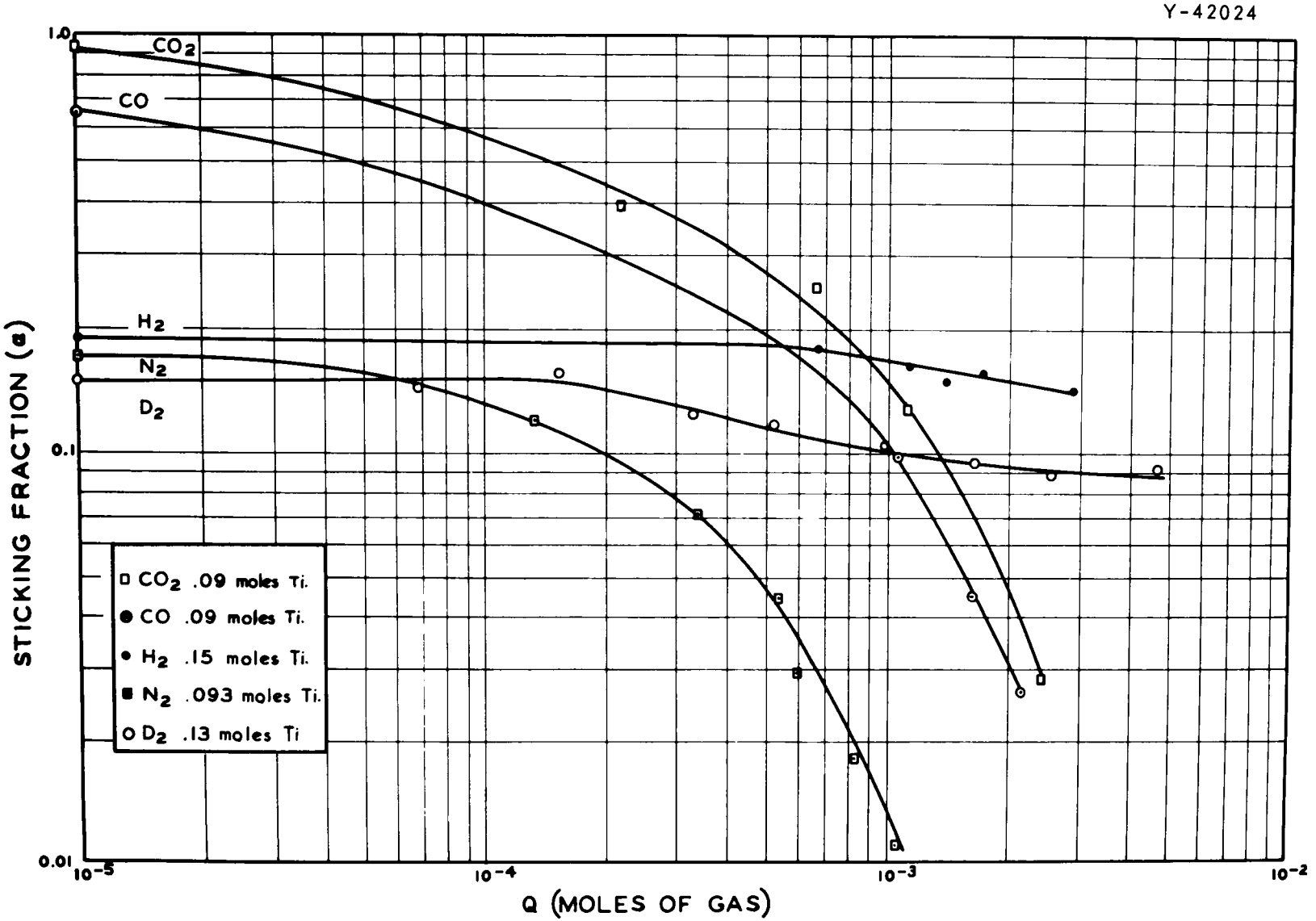

Fig. 10 Sorption of Gases onto Films Formed by Titanium Evaporation in the Presence of 2.5×10^{-3} mm Hg Pressure of Helium onto a Surface at 10°C.

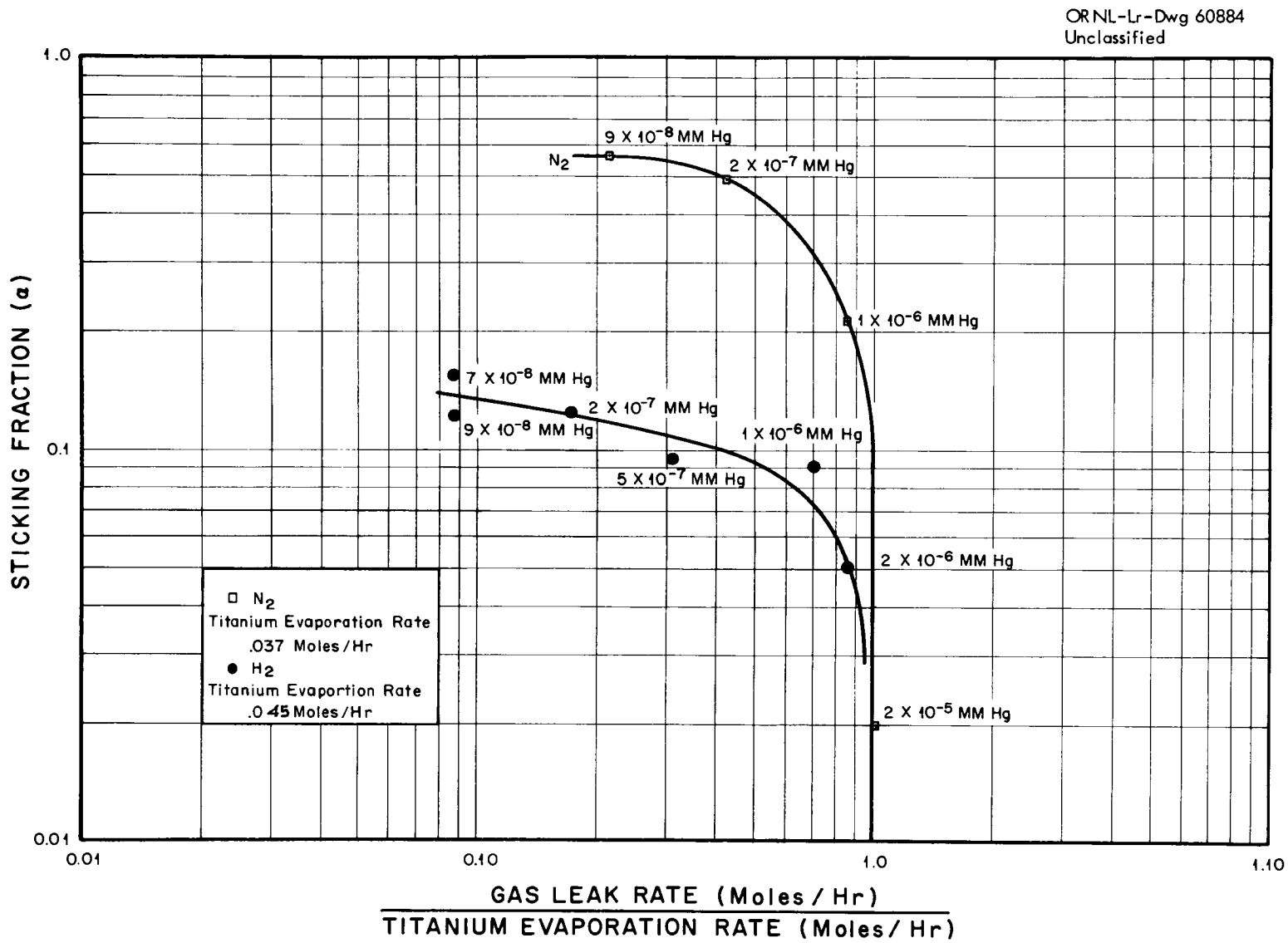

Fig. 11 Sorption of Gases onto Films Formed During Continuous Evaporation of Titanium onto a Surface at -195°C.

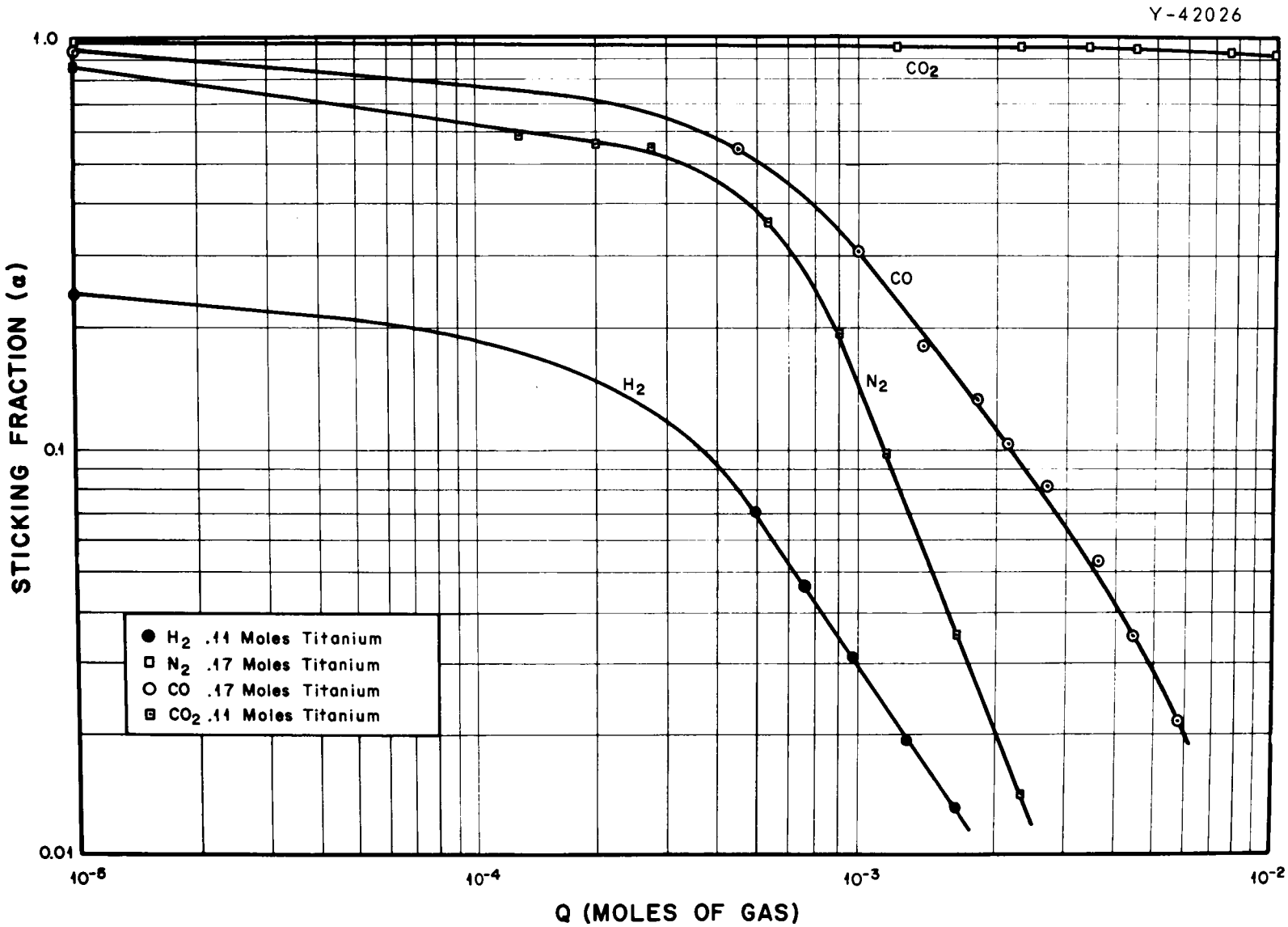

Fig. 12 Sorption of Gases onto Films Formed by Titanium Evaporation in High Vacuum onto a Surface at -195°C.

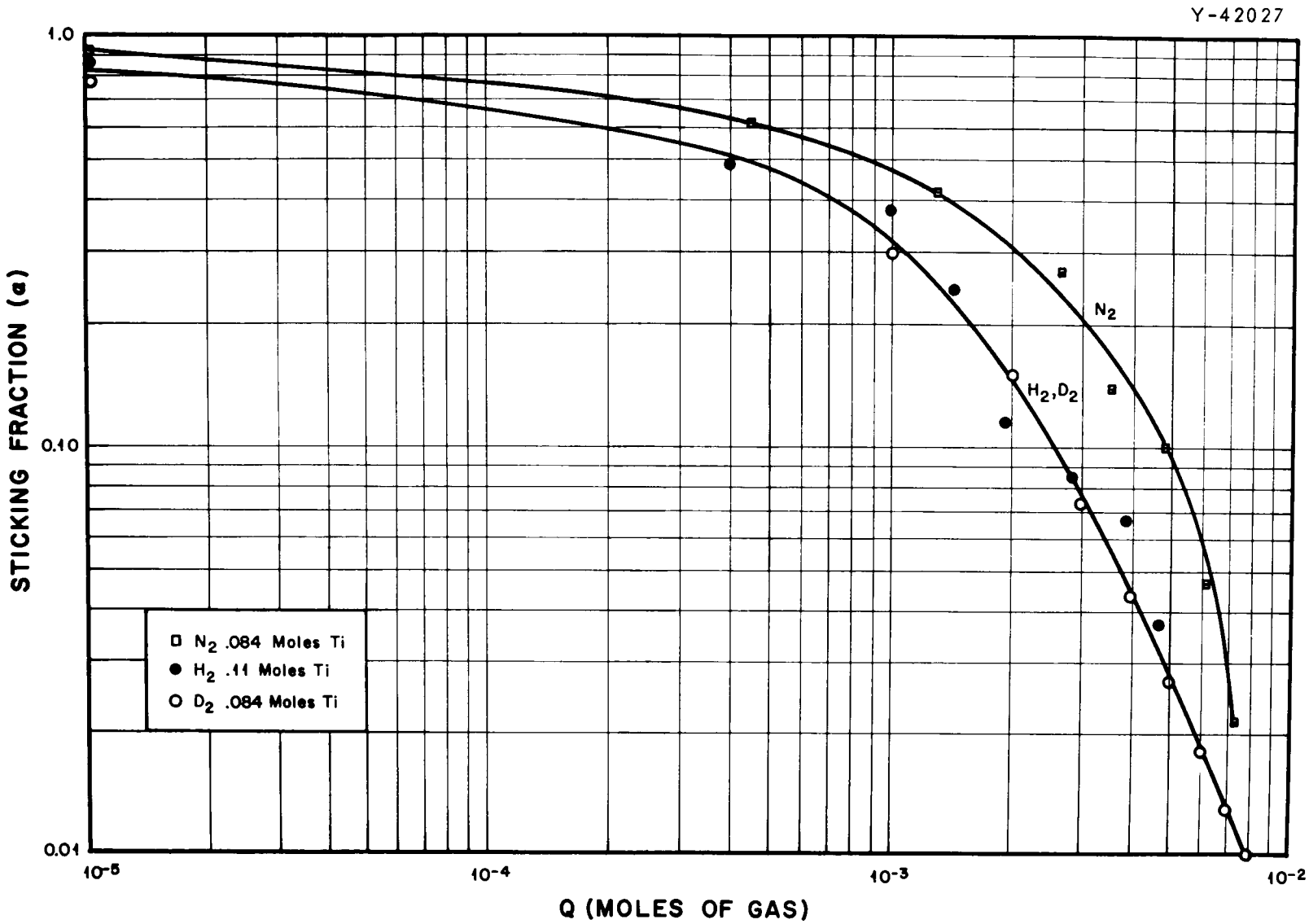

Fig. 13 Sorption of Gases onto Films Formed by Titanium Evaporation in the Presence of 2.5×10^{-3} mm Hg Pressure of Helium onto a Surface at -195°C.

The films deposited at liquid nitrogen temperatures did not retain their high sorption rates when they were allowed to warm up to room temperature nor were the sorption rates on films deposited at room temperature appreciably increased by cooling them to liquid nitrogen temperature after deposition. Films deposited at liquid nitrogen temperature, allowed to warm to 0°C, and then cooled back to liquid nitrogen temperature did not retain their high sorption rates. These results lead to the conclusion that beneficial effects of deposition at liquid nitrogen temperatures are not simply due to more favorable thermodynamics but that the film has a new attribute which anneals out on warming to room temperature.

Figure 14 shows the appearance of the films produced under each of the conditions described in this report. Note the velvety black appearance of the films deposited in the presence of inert gas and the better adherence of films deposited in liquid nitrogen temperatures. X-ray diffraction analysis of these films shows a sharp well-defined pattern only for the high vacuum deposit onto the room-temperature substrate indicating that this is the only film with a well-developed long-range crystalline structure.

SUMMARY

Experimental apparatus has been built and operated to determine the feasibility and practicality of using large-scale getter pumping in combination with a diffusion pump system to obtain high pumping speeds for hydrogen and some other gases at low pressures. Titanium metal was evaporated onto a cooled copper substrate at both 10 and -195°C in high vacuum and under a 10^{-3} mm Hg pressure of an inert gas. Sorption rates of gases onto these surfaces were then measured using a new method of measuring sticking probabilities which was developed for these experiments. Table 2 summarizes the data obtained for several gases on titanium films produced under a variety of conditions. The sorption characteristics of these films are given graphically in Figs. 7 through 13.

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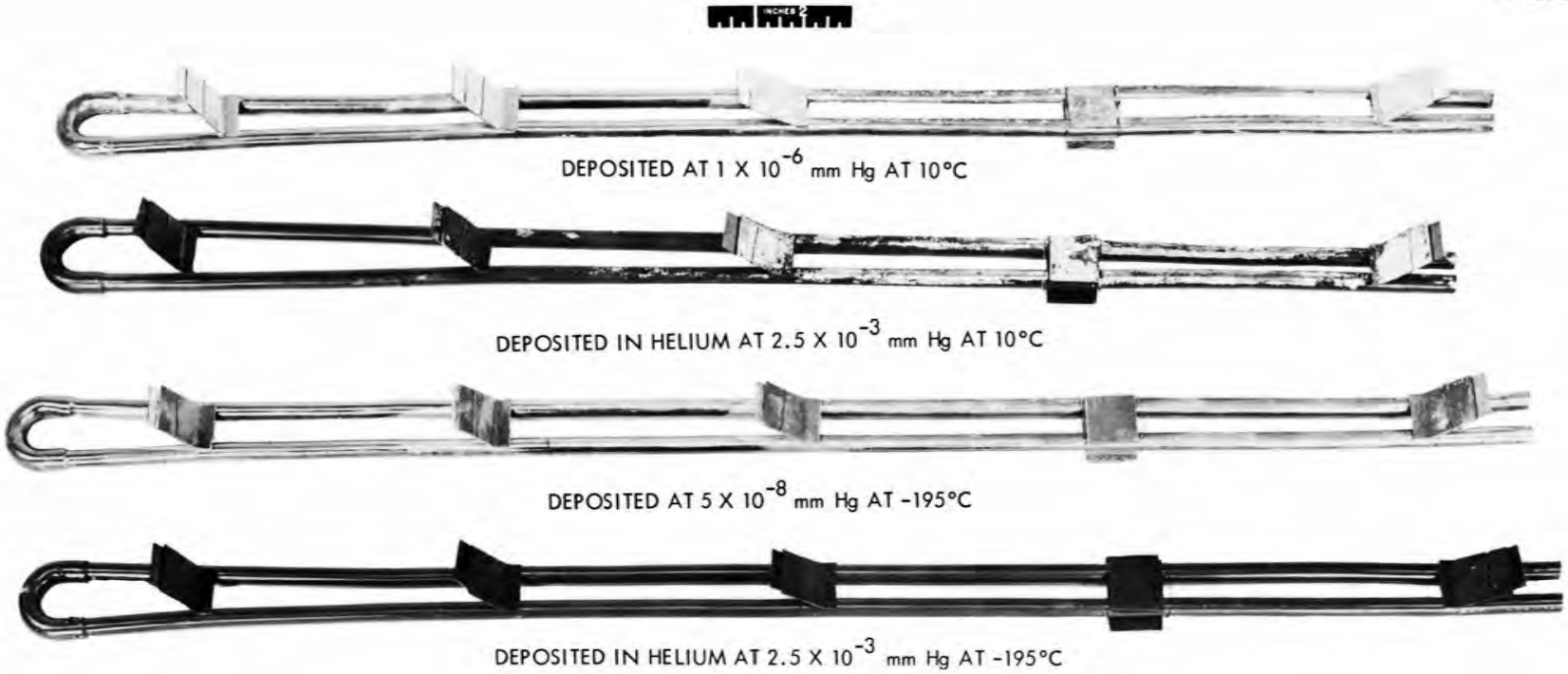

Fig. 14 Photograph Showing the Appearance of Titanium Films Formed by Various Methods.

Table 2. Initial Sticking Factors for Various Gases on Titanium Films Deposited Under Several Conditions.

Gas Species	High Vacuum, 10°C		2-μ Helium 10°C Batch Evaporation	High Vacuum, -195°C		2-μ Helium -195°C Batch Evaporation
	Continuous Evaporation	Batch Evaporation		Continuous Evaporation	Batch Evaporation	
Hydrogen	0.07	0.05	0.19	0.14	0.24	0.85
Deuterium	--	--	0.14	--	--	0.78
Nitrogen	> 0.20*	0.08	0.17	> 0.5	0.85	0.93
Carbon Monoxide	0.86	0.38	0.66	--	0.95	--
Oxygen	--	0.15**	--	--	--	--
Carbon Dioxide	> 0.5*	> 0.4*	0.92	--	0.98	--
Helium	< 0.0005*	--	--	--	--	--
Argon	< 0.0005*	--	--	--	--	--
Methane	< 0.0005*	--	--	--	--	--

*These data are estimated from measurements obtained from the original geometry.

**The oxygen used in this measurement contained appreciable quantities of argon, thus, producing a much lower value of α than is expected from pure oxygen.

As a result of these experiments, the following conclusions can be made:

1. The combination of getter pumping and diffusion pumping is well suited for obtaining high pumping speeds for hydrogen, deuterium, oxygen, nitrogen, carbon monoxide, and carbon dioxide.

2. Simple and reliable resistance-heated titanium evaporators can be easily fabricated and operated to produce suitable evaporation rates. Filament replacement through a vacuum lock is quick and easy.

3. System maintenance is not a problem. New films may be deposited over old ones without detrimental effects to system operation within the limits of these experiments.

4. Sorption rates for hydrogen and other chemically active gases may be increased by altering the structure of the titanium films by both evaporation in the presence of an inert gas and evaporation onto liquid nitrogen-cooled substrates. Sticking fractions for hydrogen as large as 0.8 have been consistently obtained on films deposited in the presence of helium onto substrates at -195°C as compared with sticking fractions near 0.05 on films deposited in high vacuum onto water-cooled substrates.

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