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Effect of Vacuum Processing on Outgassing Within an Orbiting Molecular Shield

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SUMMARY

The limiting hydrogen number density in an orbiting molecular shield is highly dependent on the outgassing rates from the materials of construction for the shield, experimental apparatus, and other hardware contained within the shield. Ordinary degassing temperatures used for ultrahigh vacuum studies (<450°C) are not sufficient to process metals so that the contribution to the number density within the shield due to outgassing is less than the theoretically attainable level (≈ 200 cm^{-3}). Pure aluminum and type 347 stainless steel have been studied as candidate shield materials. Measurements of their hydrogen concentration and diffusion coefficients have been made, and the effects of high-temperature vacuum processing (>600°C) on their resulting outgassing rates have been determined. The densities in a molecular shield due to the outgassing from either metal have been found to be substantially less (<10^{-3}) than the density due to the ambient atomic-hydrogen flux at an orbital altitude of 500 km.

INTRODUCTION

The concept of an orbiting molecular shield and a molecular-beam apparatus suggests the unique possibility of conducting numerous physics and materials experiments which require the simultaneous conditions of extremely low gas density ($\approx 10^3$ cm^{-3}) and microgravity ($\approx 10^{-4}$ g). Past investigations have shown that such conditions, within these orbiting facilities, would be useful in studies of free-jet expansions, atomic-oxygen/surface interactions, metals purification, crossbeam experiments, etc. (See refs. 1 to 4.)

The orbiting molecular shield is presented schematically in figure 1. Trajectories ⓐ and ⓑ are free-stream molecules, and the flux of ⓐ-type molecules is much greater than the flux of ⓑ-type molecules. Trajectory ② shows desorbed molecules from the shield, trajectory ③ shows desorbed molecules from the experiment, and trajectory ④ shows molecules scattered from the orbiter. The shield geometry is shown as a hemispherical shell oriented such that its axis is aligned with the orbital velocity vector U and its open end is aft. In this configuration, the shield interior and the experiments contained within are shielded from molecules in the forward half-space, and only those molecules in the aft half-space with the appropriate spatial coordinates and velocity can overtake the shield. The number of molecules that enter the shield from the aft half-space is dependent on the speed ratio S, where $S = U/\upsilon_m$ and υ_m is the most probable molecular speed. At 500 km, the number density in the molecular shield due to the atomic hydrogen flux ($S \approx 1.9$) is only 200 cm^{-3}. However, in order to achieve the low gas density within such facilities, appropriate vacuum materials must be selected for fabrication of the apparatus. Furthermore, these materials must be extensively processed to provide a minimum outgassing rate. Calculations of the internal density within an orbiting hemispherical shell (molecular shield) have shown that the ultimate internal density ($n_H = 200$ cm^{-3} for an orbital altitude of 500 km) is limited by the atomic-hydrogen flux entering the shield from the aft half-space of the atmosphere. (See ref. 1.) Ideally then, the internal outgassing rate from the shield material should be sufficiently low that its contribution to the density within the shield, compared with that generated by the atomic-hydrogen flux, is negligible. Hueser and Brock (ref. 2) have shown that using degassed stainless steel as a shield material results

in a density contribution of approximately 3.5 times that generated by the atomic-hydrogen flux (Orbit height = 500 km). Also, the outgassing from the experimental apparatus, diagnostic instrumentation, and other hardware contained within the shield could easily increase the operational density by a factor of 10 or more.
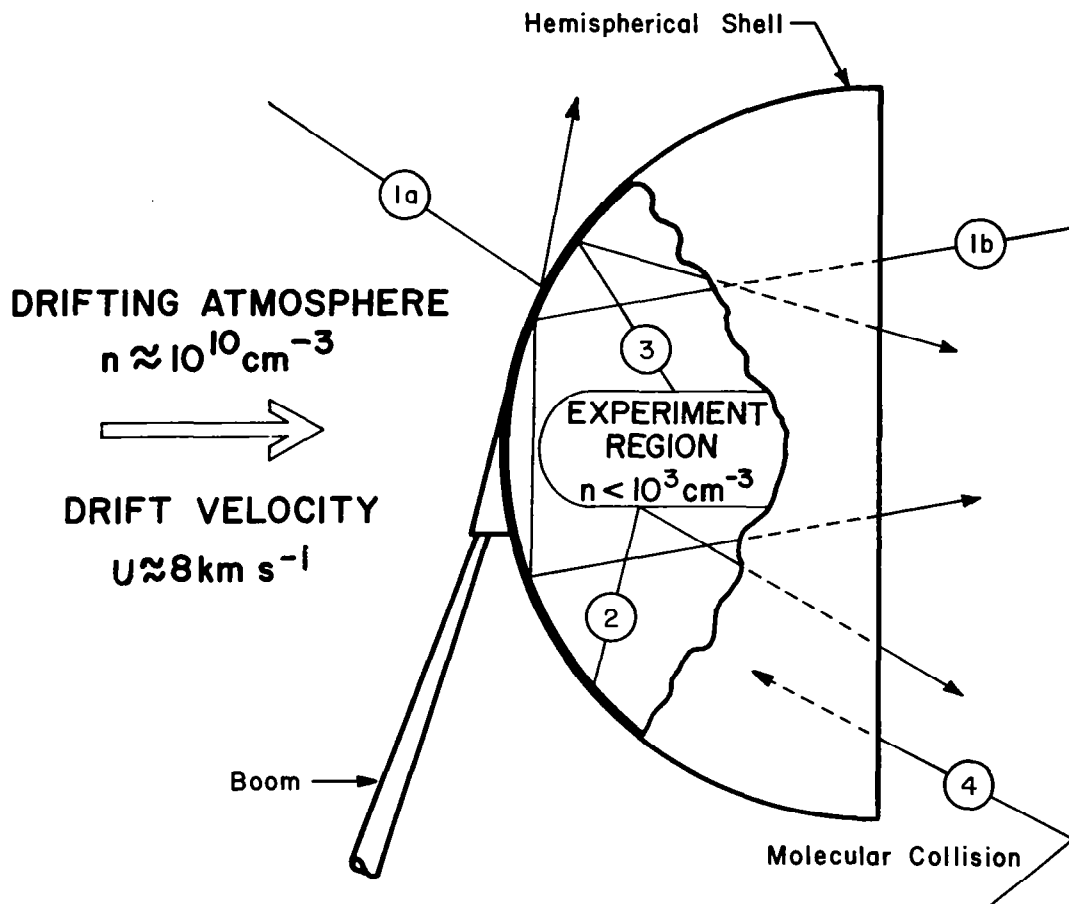

Figure 1.- Schematic of molecular-shield geometry in drifting gas (atmosphere) illustrating typical molecular trajectories. (From ref. 1.)

The purpose of this report is to show that vacuum-processed pure aluminum and vacuum-processed type 347 stainless steel have a much lower outgassing rate than would result from standard degassing procedures. It is also shown that a shield constructed of these materials would result in a density contribution substantially less than the density within the shield due to the atomic-hydrogen flux. (The symbols used in this paper are defined in a list after the references.)

EXPERIMENTAL TECHNIQUE

Measurements of the hydrogen concentration and diffusion coefficient in cylindrical samples of as-received pure cast aluminum (99.995 weight percent), vacuum-melted aluminum (heated to 815°C in vacuo for 4 hours), vacuum-degassed aluminum (heated to 600°C in vacuo for 4 hours), as-received 347 stainless steel, and vacuum-

degassed stainless steel (heated to 900°C in vacuo for 3 hours) have been made by employing a dynamic method in an ultrahigh vacuum (UHV) furnace. The paragraphs which follow are an outline of the experimental approach.

Following a thorough degassing, the major component of outgassing from metals is molecular hydrogen, primarily since atomic hydrogen is the major gas component dissolved in the bulk. The change in bulk concentration of this gas due to outgassing is determined from Fick's second law,

$$\frac{\partial c}{\partial t} = D_H \nabla^2 c \tag{1}$$

where c is the concentration, D_H is the diffusion coefficient independent of c, and t is the time. The solution to equation (1) for a one-dimensional case where a metal sheet of thickness d contained within a vacuum has the boundary conditions $c = c_o$ for $0 < x < d$ at $t = 0$ and $c = 0$ for $x = d$, $x = 0$ at $t > 0$, is (ref. 5)

$$c(x,t) = \frac{4}{\pi} c_o \sum_{n=0}^{\infty} (2n + 1)^{-1} \sin \frac{(2n + 1)\pi x}{d} \exp\left[\frac{-D_H(2n + 1)^2 \pi^2 t}{d^2}\right] \tag{2}$$

The outgassing of hydrogen ν_d from the metal into the vacuum space is then

$$\nu_d(x,t) = -D_H\left(\frac{\partial c}{\partial x}\right)_{x=0} = \frac{4c_o D_H}{d} \sum_{n=0}^{\infty} \exp\left[\frac{-D_H(2n + 1)^2 \pi^2 t}{d^2}\right] \tag{3}$$

The measurement of c_o and D_H for a given material then permits the determination of the bulk outgassing rate.

The dynamic technique employed to determine c_o and D_H involves lowering the cylindrical samples ($\ell = 2.5$ cm, $\delta = 1.25$ cm) into the hot zone of a preheated UHV furnace and monitoring the temporal desorption of gases coming from the sample by a quadrupole mass spectrometer. In addition to separating the gases desorbed from the sample surface from those desorbed from the sample bulk (only hydrogen), a great deal of kinetic information was also obtained. Furthermore, a series of experiments run over the temperature range 400° to 625°C permitted the determination of the temperature dependence of the diffusion constant. Details of this procedure are contained in references 6 and 7.

Figure 2 shows a typical hydrogen desorption plot of as-received aluminum and as-received 347 stainless steel heated to 600°C. The hydrogen concentration was determined from desorption spectra of this type, and

$$c = \frac{2C}{V_s k_B T} \int_\tau p(t)\, dt \tag{4}$$

where C is the annular conductance, V_s is the sample volume, T is the gas temperature, k_B is Boltzmann's constant, $p(t)$ is the pressure above the sample, and τ is the heating interval. Diffusion coefficients were also calculated from the desorption spectra by

$$D = \frac{2.3m}{\left(\frac{\pi}{\ell}\right)^2 + \left(\frac{2\beta_1}{\delta}\right)^2} \qquad\qquad (5)$$

where m is the slope of the $p(t)$ decay, ℓ is the length of the sample, δ is the diameter of the sample, and β_1 is the root of the Bessel function $J_0(\beta_n) = 0$.
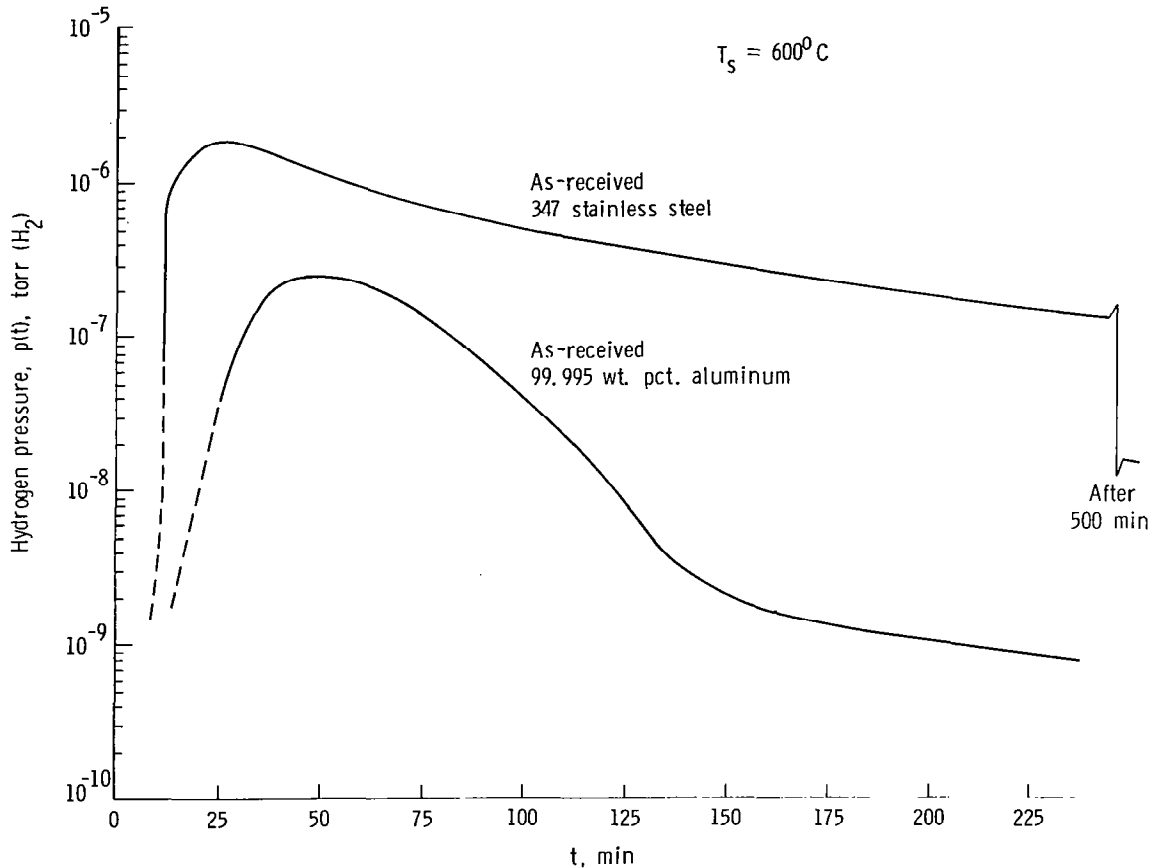
Figure 2.- Hydrogen desorption spectra from 99.995 weight percent aluminum and 347 stainless steel. Dashed lines are estimated leading edge of bulk desorption curves. Stainless steel is still desorbing hydrogen after 8 hours.

The surface treatment for either stainless steel or aluminum is also very important in minimizing the total outgassing rate. Typical spectra representing thermal desorption of surface gases from the electropolished aluminum samples studied herein (exposed to room air at normal temperature and pressure) are shown in figure 3. Carbon monoxide appears to be the primary desorbed gas species with only small quantities of surface H_2 and H_2O observed. Similar behavior was also noted for 347 stainless steel, with the exception that the surface gases desorbed more rapidly. In both cases, however, hydrogen was the only gas detected after surface desorption was completed, and bulk diffusion was the controlling mechanism.
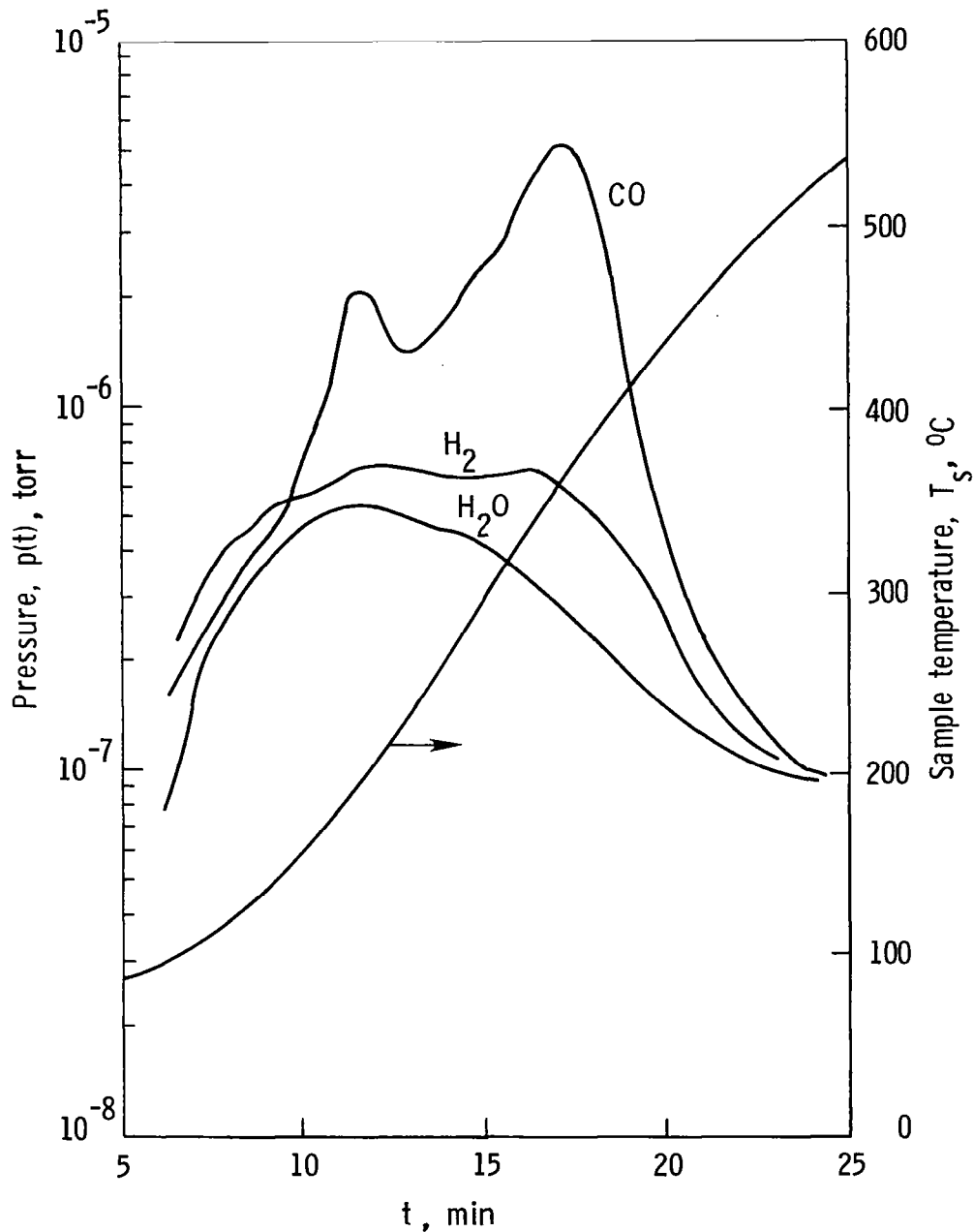
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Figure 3.- Desorption spectra of adsorbed gases on the surface from electro-
polished aluminum exposed to atmosphere as sample temperature increases.

RESULTS

Table 1 shows the initial concentration values along with the appropriate diffu-
sion coefficients (extrapolated to 20°C) determined from equations (4) and (5),
respectively. From these data and equation (3), outgassing rates for 3-mm-thick
aluminum and 347 stainless steel (estimated maximum shield thickness) after 5 days
have been calculated and are shown in table 1.

TABLE 1.- OUTGASSING PARAMETERS FOR ALUMINUM AND TYPE 347 STAINLESS STEEL

Material	c_o, cm^{-3}	$D_H(20°C)$, cm^2-s^{-1}	$v_d(20°C)$, torr-liters $cm^{-2}-s^{-1}$ [a]	Vacuum processing before measurement
As-received Al	6.3×10^{17}	[b]3.2×10^{-10}	8.0×10^{-11}	
Vacuum-melted Al	2.1×10^{12}	3.2×10^{-10}	2.7×10^{-16}	4 hr at 815°C
Vacuum-degassed Al	5.8×10^{10}	3.2×10^{-10}	7.4×10^{-18}	4 hr at 600°C
As-received 347 stainless steel	6.2×10^{18}	2.8×10^{-11}	7.0×10^{-11}	
Vacuum-degassed 347 stainless steel	2.2×10^{12}	2.8×10^{-11}	2.5×10^{-17}	3 hr at 900°C

[a]Units for outgassing presented here are those most often employed.
[b]Assumed for porous aluminum.

The diffusion coefficient for as-received aluminum is substantially higher than for 300-series stainless steel, but the outgassing rates for vacuum-processed aluminum are lower. The reason for this is the inordinately low hydrogen solubility in aluminum. (See ref. 8.) As-received aluminum was determined to have a hydrogen concentration of only 6.3×10^{17} cm^{-3} (10 ppm), but the hydrogen concentration of vacuum-processed aluminum is reduced by as much as seven orders of magnitude. In the as-received aluminum, 99 percent or more of the hydrogen is lodged in the cast-in interdendritic porosity, and the balance is in solid solution. (See ref. 6.) Vacuum processing of the aluminum above the melting point removes all the hydrogen in the pores (since the pores are eliminated) and reduces the amount in solid solution to that in equilibrium with the low hydrogen partial pressure (2×10^{-9} torr (H_2)). Vacuum processing just below melting also removes the hydrogen in the pores and actually lowers the hydrogen in solid solution to much less than that in solid solution above the melting point, since the equilibrium solubility is lower by a factor of 9 at 660°C. Unfortunately, the latter process does not remove the porosity.

Experiments were also conducted to assess the difficulty of recharging the vacuum-melted aluminum samples. The samples were subjected to 1 atm (760 torr) of hydrogen at 600°C for several days. Subsequent vacuum desorption revealed no detectable hydrogen was absorbed, presumably because of the inhibiting Al_2O_3 barrier on the surface. Increasing the charging pressure to 143 atmospheres only resulted in a hydrogen content of $\approx 6 \times 10^{15}$ cm^{-3} (0.1 ppm). Thus, in addition to its extremely low outgassing rate, large-grain aluminum is not susceptible to significant hydrogen replenishment. Other researchers have also indicated that low outgassing rates of aluminum and aluminum alloys can be achieved. Young (ref. 9) measured 4×10^{-13} torr-liters $cm^{-2}-s^{-1}$ after only a 15-hour, 250°C bake. Halama and Herrera (ref. 10) determined the outgassing rate for 6061 aluminum to be less than 1×10^{-14} torr-liters $cm^{-2}-s^{-1}$ after 24 hours at 200°C. Measurements by Moraw and Dobrozemsky (ref. 11) have shown that bakeout of the aluminum in air at 100°C, which most likely increases the thickness of the surface oxide and provides a barrier to the evolving bulk hydrogen, lowers the outgassing rate to 2.8×10^{-14} torr-liters $cm^{-2}-s^{-1}$. In all these experiments, however, the time.

and temperature of the vacuum processing (for samples of equal or greater size) was probably insufficient to desorb all the bulk hydrogen which would accordingly prevent achieving the lowest outgassing rate. The time required for depletion of the hydrogen in the bulk at 600°C for the samples studied in this work is approximately 2 hours (fig. 1). Since the diffusion coefficient decreases exponentially with decreasing temperature, that is, $D_H(600)/D_H(250) = 52$, temperatures substantially lower than 600°C would require much longer times.

Vacuum processing of 347 stainless steel at 900°C for 3 hours was sufficient to deplete the bulk of hydrogen, but more than 10 hours was required at 600°C. The average value of c_o determined for the as-received state was 6.2×10^{18} cm^{-3}. Unfortunately, there are no relatively modern data for the solubility of hydrogen in 347 stainless steel, but since 304 stainless steel is similar in composition, the solubility data of Nelson and Stein (ref. 12) can be used to estimate c_o for vacuum-processed 347 stainless steel. As indicated in table 1, a concentration value of 2.2×10^{12} cm^{-3} was determined for the partial pressure of 2.0×10^{-9} torr (H_2) in the vacuum furnace (900°C). This gives a value of $v_d = 2.5 \times 10^{-17}$ torr-liters cm^{-2}-s^{-1} for the outgassing rate at 20°C, which is within the same order of magnitude as that obtained for the vacuum-processed aluminum. The above estimate is somewhat lower than the measured values of Calder and Lewin (ref. 13) for vacuum-processed U15C stainless steel degassed at 1000°C for 3 hours ($<1 \times 10^{-14}$ torr-liters cm^{-2}-s^{-1}), but is consistent with their theoretical predictions. It is quite likely that their experimental values are near the limit of detectability, primarily because less extensive vacuum processing for different samples in their work and the experimental data of other researchers (ref. 14) gave similar values ($\approx 1 \times 10^{-14}$ torr-liters cm^{-2}-s^{-1}), and because the theory predicts much lower values.

The extremely low outgassing rates of 347 stainless steel determined in this work were achieved by vacuum processing in the solid state at higher temperatures rather than ordinary bakeout temperatures (≤ 450°C). This allows a much greater reduction of c_o. Much of the data obtained by others, as in the aluminum case, were obtained with insufficient processing at lower temperatures and shorter times. This does not allow c_o to approach its equilibrium solubility level. Furthermore, normal bakeouts where only one side is exposed to vacuum, have the inherent problem of resupplying the hydrogen into the bulk from the atmosphere, thus limiting the value of c_o attainable.

MOLECULAR SHIELD GAS DENSITY

The density distribution within a hemispherical molecular shield (ref. 2) is as follows:

$$n_d(r,\mu) = 2v_d\left(\sqrt{\pi}v'_m\right)^{-1} \int_0^{\pi/2} h(r,\mu,\zeta)\ d\zeta \tag{6}$$

where v'_m is the most probable speed of the desorption gas, μ and ζ are azimuthal angles, $h(r,\mu,\zeta) = f[E(k),K(k)]$, $E(k)$ and $K(k)$ are complete elliptic integrals of the first and second kind with modulus k, and r is the normalized radius. The density at the origin of the shield due to outgassing v_d reduces to

$$n_d(0,0) = \frac{2\sqrt{\pi}}{v'_m} v_d \tag{7}$$

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Since the density anywhere within the shield does not vary by more than a factor of 4 from the origin, equation (7) is sufficient to indicate the general effect due to outgassing from the walls of the shield. The calculated densities $n_d(0,0)$ due to outgassing (molecular hydrogen desorbing at 20°C; $v'_m = (2k_B T/m)^{1/2} = 1.56 \times 10^5$ cm-s^{-1}) for the vacuum-processed aluminum and the stainless steel are given in table 2. The value obtained for the vacuum-degassed

TABLE 2.- MOLECULAR-SHIELD GAS DENSITY PARAMETERS FOR VACUUM-PROCESSED

ALUMINUM AND TYPE 347 STAINLESS STEEL

Material	v_d, torr-liters cm^{-2}-s^{-1}	n_d, cm^{-3}	n_d/n_H [a]
As-received Al	8.0×10^{-11}	6.0×10^4	3.0×10^2
Vacuum-melted Al	2.7×10^{-16}	0.2	1.0×10^{-3}
Vacuum-degassed Al	7.4×10^{-18}	5.5×10^{-3}	2.8×10^{-5}
As-received 347 stainless steel	7.0×10^{-11}	1.3×10^5	6.4×10^2
Vacuum-degassed 347 stainless steel	2.5×10^{-17}	4.6×10^{-2}	2.3×10^{-4}

[a]Molecular-shield gas density due to atomic-hydrogen flux is 200 cm^{-3} orbital altitude of 500 km.

aluminum would result in a density due to outgassing 3.6×10^4 times lower than n_H, the density due to the atomic-hydrogen flux. The value obtained for the vacuum-degassed 347 stainless steel would result in a density due to outgassing 4.4×10^3 times lower than n_H. Although the values estimated herein for these materials are very low, they are probably conservative. The outgassing estimated from equation (3) is most likely too high since the values are based on room-temperature extrapolations of the diffusion coefficient. It has been found for stainless steel that hydrogen traps of low potential energy at the lower temperatures (<300°C) substantially reduce the diffusion coefficient below the predicted value from extrapolations, and therefore reduce the outgassing rate. (See ref. 15.) This phenomenon likely applies to aluminum also. It therefore appears that, based strictly on an outgassing argument, either vacuum-processed material would be a suitable candidate construction material for a molecular shield or molecular-shield apparatus. Vacuum-degassed aluminum would still contain the porosity, however, and may result in unwanted pinholes for a very thin shield (≈1 mm). The most desirable processing for the aluminum would be to initially vacuum melt the material, thus eliminating the porosity, and then follow with vacuum degassing in the solid state. This would insure the lowest ultimate outgassing rate and, therefore, the lowest density within the shield. Aluminum is also much lighter, possesses a more stable surface oxide, is far more difficult to recharge with hydrogen, and can be vacuum processed at lower temperatures (<400°C) than stainless steel. Stainless steel, however, does not require vacuum melting and may be more desirable for those applications requiring more material strength.

CONCLUSIONS

Previous calculations of the hydrogen number density within a molecular shield showed that outgassing from the shield and experimental apparatus will limit the density within the shield to a value that is substantially greater than that due to the atomic-hydrogen flux from the atmosphere (Orbital altitude = 500 km). According to good ultrahigh vacuum (UHV) techniques, the materials considered in the above calculations were assumed to be well degassed. In the present work, samples of pure aluminum and type 347 stainless steel were vacuum processed at high temperatures and low pressures for time intervals sufficiently long to minimize their bulk hydrogen concentration. Measurements of the concentration and diffusion coefficients of hydrogen in these metals were made and subsequently used to determine outgassing rates. The values obtained for ν_d were extremely low and imply that in previous research conducted by others, ordinary UHV degassing procedures used were probably insufficient to substantially deplete the bulk hydrogen. Even though this work addresses only two often-used vacuum materials, the procedure of high-temperature vacuum processing to significantly lower c_o, and therefore ν_d, is probably applicable to other vacuum materials as well.

Finally, it was found that a molecular shield constructed of vacuum-processed pure aluminum or 347 stainless steel would yield a contribution to the density within the shield due to outgassing that is less than a factor of 3×10^{-4} that of the contribution due to the atomic-hydrogen flux.

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C   annular conductance of experimental system, $cm^3\text{-}s^{-1}$

c   atomic-hydrogen concentration in solid, cm^{-3}

c_o initial atomic-hydrogen concentration in solid, cm^{-3}

D_H atomic-hydrogen diffusion coefficient, $cm^2\text{-}s^{-1}$

d   thickness of metal sheet, cm

E(k)   elliptic integral of first kind, dimensionless

g   acceleration due to gravity, $1g = 9.8$ m/s^2

K(k)   elliptic integral of second kind, dimensionless

k   elliptic integral, modulus

k_B Boltzmann's constant, 1.38×10^{-16} erg/K

ℓ length of sample, cm

m   slope of hydrogen $p(t)$ versus t plot, $dynes\text{-}cm^{-2}\text{-}s^{-1}$

n   gas number density at orbital altitude, cm^{-3}

n_d hydrogen number density in shield due to outgassing, cm^{-3}

n_H hydrogen number density in shield due to atomic-hydrogen flux, cm^{-3}

p(t)   varying pressure, $dynes\text{-}cm^{-2}$ (1 torr = 1.33×10^3 $dynes\text{-}cm^{-2}$)

r   radius of hemispherical shield, cm

S   speed ratio, dimensionless

T   gas temperature, K

T_s sample temperature, K

t   time, s

U   orbital velocity, $cm\text{-}s^{-1}$

V_s volume of sample, cm^3

x   arbitrary variable in shield material

β_1 root of Bessel function $J_0(\beta_n) = 0$, dimensionless

δ diameter of cylindrical sample, cm

μ, ζ azimuthal angle, deg

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ν_d hydrogen outgassing rate, $cm^{-2}-s^{-1}$
        (1 torr-liter $cm^{-2}-s^{-1} = 3.3 \times 10^{19}$ $cm^{-2}-s^{-1}$)

τ heating-time interval, s

υ_m most probable molecular speed, $cm-s^{-1}$

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15. Supplementary Notes

16. Abstract

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