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HELIUM RELEASE FROM TYPE 304 STAINLESS STEEL

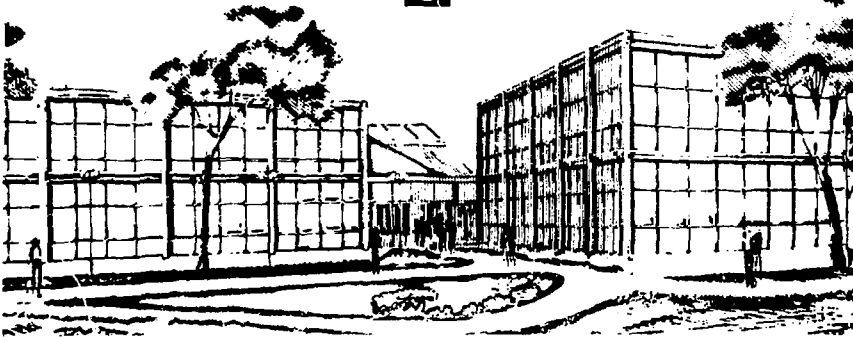
J. R. Cost, R. G. Hickman, J. B. Holt, and R. Borg

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HELIUM RELEASE FROM TYPE 304 STAINLESS STEEL*

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ABSTRACT

Helium in very low concentration (less than atomic parts per billion) has been introduced into type 304 stainless steel by radioactive decay of dissolved tritium. The release of this helium during subsequent annealing has then been monitored with a high sensitivity mass spectrometric gas analyzer. With isochronal annealing, helium is released in two temperature ranges, one near 300°C and the other between 800°C and the melting point. The latter release is interpreted as attributable to helium gas bubbles. The release near 300°C has been studied isothermally between 150 and 300°C and has been analyzed in terms of two stages of exponential decay. The fast and slow release stages have relaxation times near 10² and 10³ s, respectively, and the fast release accounts for roughly 85% of the total release at low temperature. From an analysis of the temperature dependence of the release rate, it is concluded that volume diffusion is the controlling mechanism for the outgassing.

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INTRODUCTION

Because the presence of even small amounts of helium often results in decreased mechanical properties and premature failure in materials for nuclear power applications, there has recently been a large amount of work devoted to characterizing the behavior of helium in these materials. The problems caused by the presence of helium become particularly important for fusion power reactors in which materials will be exposed to helium ion implantation from the plasma and to helium created by beta decay of tritium, in addition to helium created by (n, α) reactions. To deal with materials problems related to the presence of helium, it is important to characterize helium's outgassing behavior, i.e., to determine how such variables as temperature, radiation, material composition, helium concentration, etc., affect the helium release rate. Before such empirical studies are made, however, it is valuable to study a simple metal-helium system at low helium concentrations, without the complications of radiation damage, to determine the basic mechanisms by which helium outgassing occurs.

The present study makes use of the radioactive decay of dissolved tritium as a method for introducing controlled low concentrations of helium into type 304 stainless steel. The subsequent degassing behavior of helium is studied from room temperature to 1300°C.

Use of tritium for the controlled introduction of helium into metals was first described by Blackburn.¹ Tritium undergoes spontaneous beta decay with a half-life of 12.3 y, producing ^3He as it releases an average energy of 5.7 keV per event. Its virtues are that it can dissolve in most metals at moderate temperatures and give a uniform internal concentration of helium. This is to be contrasted with the implantation of energetic alpha particles, which produces very nonuniform concentrations. Furthermore, the radiation damage to the lattice that results from the 5.7-keV electron is very slight compared to other helium introduction techniques (neutron irradiation or direct implantation). In fact, in metals point defects should not even be formed.²

EXPERIMENTAL METHODS

The experimental method has been described previously.³ After pre-annealing at 1000°C in UHV for 1 h, the specimens were exposed to tritium at 300°C and 1.13 atm (114 kPa) for 1 d (864 ks). The samples were 0.406-mm-thick foils of commercial type 304 stainless steel. The tritium was pumped out after sample exposure, and the dissolved tritium was almost all removed by maintaining the specimens at 250°C for about 1 d in a vacuum of 3×10^{-7} Torr (about 40 μ Pa).

After cooling, each specimen was analyzed for residual tritium. This was done by snipping a small corner from the foil, slowly dissolving it in aqua regia, diluting the resulting solution, and measuring the tritium with standard liquid scintillation counting procedures.

The experimental vacuum system used to make the helium release measurements has also been described previously.⁴ During a run helium is collected in the vacuum system for a prescribed time interval while other gases are removed with a titanium sublimation pump. The helium present is determined by sweeping over the $m/e = 3$ peak with a residual gas analyzer.

RESULTS

Helium release was studied for both isochronal and isothermal anneals. The data for the former are shown in Fig. 1 for three different tritiated samples and an untritiated control sample annealed over the temperature range from room temperature to 1300°C. In this figure the height of the mass spectrometer peak in amperes for species with $m/e = 3$ is plotted vs annealing temperature. This species is exclusively ³He, the decay product of tritium, because, as determined by experiments on gas mixtures of helium and hydrogen species in controlled concentrations, the hydrogen species are completely removed from the system by the titanium sublimation pump. Similar calibration experiments with ³He gave a calibration factor of 3.19×10^{22} atoms/A, so that the peak heights can give the release from the sample in atoms of helium.

It may be observed that releases occur in two widely separated temperature ranges, one release showing a relatively narrow peak near 300°C and the other release between 800°C and the melting point. Similar results with a release peak near 300°C and a broad temperature range of release at high temperature have been reported for isochronal annealing of nickel implanted with helium⁵ and also for type 316 stainless steel.³ The helium concentrations released in these two temperature ranges for the three runs shown in Fig. 1 are given in Table 1. Also reported in Table 1 is the concentration of residual tritium made on a small portion of the sample before the helium analysis. It is noted that the total release above 800°C is somewhat larger than that near 300°C; however, the total helium in the samples could not be determined because the furnace did not reach the melting temperature.

One of the goals of this study was to consider the possible mechanisms for these two distinctly different stages of release and, in particular, the release at low temperature. Thus, the remainder of the experimental results study the isothermal annealing of the release peak near 300°C.

Figure 2 shows the release results for four different isothermal annealing temperatures at and below 300°C. These results are plotted semilogarithmically as the amount still to be released vs annealing time.

Table 1. Summary of Isochronal Annealing Results

Run No.	Sample Wt. (g)	Concentration of Tritium, C _T (atom fraction)	Concentration of Helium Released, c (atom fraction)	
			300°C Peak	> 800°C
7	0.6691	7.2×10^{-6}	4.7×10^{-11}	not det.
10	0.5644	5.3×10^{-6}	1.5×10^{-11}	3.7×10^{-11}
13	0.6443	5.4×10^{-6}	7.8×10^{-11}	9.1×10^{-11}

The solid lines in Fig. 2 are least-squares fits of the data at long times (at which the semilogarithmic plot is linear) to the equation for volume-diffusion-limited release from a thin slab of thickness h ,⁶

$$\bar{c}(t) = c_0 \frac{8}{\pi^2} \sum_{x=0}^{\infty} \frac{1}{(2x+1)^2} \exp [-(2x+1)^2 t/\tau], \quad (1)$$

where

t is time,

\bar{c} and c_0 are the average instantaneous and original helium concentrations and are proportional to the summed amplitudes of the ^3He mass spectrometer peaks remaining to be released with the isothermal anneal.

In the above equation the relaxation time, τ , is related to the volume diffusivity, D , by

$$\tau = \frac{h^2}{\pi^2 D}. \quad (2)$$

The least-squares-fit lines shown in Fig. 2 have a constant slope for times greater than 200 s, corresponding to times at which terms with $x > 0$ are negligible in Eq. (1). It may be observed that the data for each temperature show a good fit to simple exponential decay of the concentration for times greater than 10^3 s. The slopes of these lines yield values for τ and thus the volume diffusivity. Values for these parameters are reported in Table 2. Because of the uncertainty due to subtraction of the background amplitude for the mass spectrographic measurement, these values are probably no more accurate than 10%. At short times, when the terms in the solution to the diffusion equation with $x > 0$ are nonnegligible, the least-squares-fit (solid) lines to Eq. (1) show a small region with increased slope. This region includes about the first 20% of the gas release. It corresponds to the time during which the curved concentration profile for helium is established across the thickness of the sample.

Table 2. Summary of Isothermal Annealing Results

Temp. (°C)	Sample Wt. ^a (g)	Slow Release			Fast Release			c_f
		c_s	τ_s (s)	D_s (cm ² /s)	c_f	τ_f (s)	D_f (cm ² /s)	c_f+c_s
150	0.6673	2.7×10^{-11}	2.3×10^3	8.41×10^{-8}	1.7×10^{-10}	1.3×10^2	1.5×10^{-6}	0.86
200	0.3411	2.3×10^{-11}	1.5×10^3	1.35×10^{-7}	1.67×10^{-10}	1.3×10^2	1.5×10^{-6}	0.88
250	0.3522	1.3×10^{-11}	9.2×10^2	2.12×10^{-7}	5.7×10^{-11}	1.4×10^2	1.4×10^{-6}	0.82
300	0.6792	9.7×10^{-12}	7.4×10^2	2.64×10^{-7}	6.5×10^{-11}	7.7×10^1	2.6×10^{-6}	0.83
30 (calculated)	-	-	1.4×10^4	1.37×10^{-8}	-	2.5×10^2	7.8×10^{-7}	-
		$Q_s = 3800$ cal/mole (0.16 eV) $\tau_{0s} = 26$ s $D_{0s} = 7.5 \times 10^{-6}$ cm ² /s			$Q_f = 1200$ cal/mole (0.05 eV) $\tau_{0f} = 34$ s $D_{0f} = 5.8 \times 10^{-6}$ cm ² /s			

* All samples were cut from the same tritiated strip for which the concentration of tritium was measured to be 5×10^{-6} atom fraction.

The kinetics of this early or fast release have been analyzed by subtracting away the contribution due to the later or slow release. The gas release after this subtraction is shown in Fig. 3, again as a least-squares fit to Eq. (1) at each temperature. Because of the short times and thus smaller number of data points for the fast release and the uncertainties involved with subtracting the slow release, the data are not as good as in Fig. 2. However, reasonable fits to exponential decay of the concentration are again obtained. The concentrations, time constants, and appropriate diffusivities for each temperature are also shown in Table 2. One may observe that the fast release accounts for roughly 85% of the total helium release at low temperatures. At this point it should be mentioned that the isothermal helium release data were found to fit this two-stage exponential decay scheme significantly better than other schemes (i.e., mathematical functions) tested, such as kinetics based upon t^n . Although the data could be fit to other considerably more complex multiple mechanism models, the two-stage volume-diffusion-limited model has been chosen because it fits the experimental results and is physically reasonable.

The temperature dependence of the relaxation times from Eq. (1) for both the fast and slow release mechanisms are shown on the Arrhenius plot of Fig. 4. At each temperature measured there is roughly a decade difference in the relaxation times for the two mechanisms. There appears to be less temperature dependence for the faster mechanism, but this probably cannot be stated with certainty because the experimental error is difficult to estimate, especially for the faster mechanism. Table 2 gives experimental values of τ_0 , D_0 , and the apparent activation energies for the equation $\tau = \tau_0 \exp(Q/RT)$. Here, the subscripts f and s refer to the fast and the slow mechanisms. Also given are the extrapolated values for the relaxation times at room temperature.

The various annealing treatments were found to cause release of residual tritium from the samples. Because this element is not measured by the mass spectrometer (the element is trapped by the titanium sublimation pump), liquid scintillation analyses for tritium were made on various samples before and after annealing. It was found that an

isothermal anneal at 300°C for 2000 s reduced the residual tritium by a factor of 6. Also, an isochronal anneal to 1300°C (5 min at 100°C temperature intervals) reduced tritium by a factor of $3,5 \times 10^3$. Mass spectrometric analysis for ^3He was also made after the above anneals. It was found that the sample that had been given the 300°C anneal (which resulted in helium release) did not show helium release when it was remeasured within 1 h after the anneal. This same sample did, however, show a helium release peak, attenuated by roughly a factor of 6, after being held several days at room temperature. The sample given the 1300°C anneal, on the other hand, showed no subsequent helium release, corroborating the results of the scintillation counting experiments.

DISCUSSION

Because of the very low or negligible solubility of helium in metals, there is a strong driving force causing these newly introduced inert gas atoms to interact with defects present. This interaction may be with point defects, or it may be a self-interaction in which inert gas atoms aggregate to precipitate and form bubbles. These two kinds of interactions can occur simultaneously, and thus be competitive processes. Interestingly, the results of these two processes tend to be quite different. Inert gas atoms that interact with impurity atoms or point defects can still be mobile and can ultimately outgas by diffusion to free surfaces. Conversely, the inert gas atoms that form bubbles tend to remain in the metal because, although the small bubbles are mobile and can produce some outgassing, the migrating bubbles tend to coalesce to form larger, relatively immobile bubbles. A general pattern for the behavior of helium in various metals has developed from recent studies. At low annealing temperatures helium atoms interact with point defects to form a complex defect, while at high temperatures they tend to precipitate as bubbles. As previously indicated these two modes of behavior can both occur at some temperatures. Also, in addition to temperature, the helium concentration can be expected to affect which behavior is dominant. The results of this study will now be discussed in terms of these two kinds of behavior.

High Temperature Release

The release we have found in stainless steel at temperatures above 800°C is believed to be caused by bubbles that become mobile in this temperature range. Because bubble mobility decreases with increasing bubble radius, the process of gas release by bubble diffusion to the surface is complicated by the simultaneous process of bubble coalescence in which large, relatively immobile bubbles are formed. The kinetics of the combined processes of bubble diffusion to surfaces and coalescence are difficult to analyze quantitatively; however, it is worthwhile to consider the general shape of the isochronal annealing curve. The release we observed above 800°C did not occur as a classical release peak, but rather is spread over a wide temperature range up to the melting point. This result is in accord with what we would expect for the bubble migration mechanism of gas release. Because of the large dependence of bubble mobility on the radius and the large width of the distribution of bubble radii,⁷ a narrow release peak such as we observed at 300°C is not predicted. Instead, the gas release is expected to occur over a wide temperature range up to the melting point, where the larger bubbles become mobile. Thus our results are consistent with the interpretation that helium bubble diffusion is the high temperature release mechanism. Also, this interpretation is consistent with results from studies of helium bubble formation in numerous other materials.⁸ Obviously, this is not an unequivocal interpretation; thus additional studies are suggested to substantiate the mechanism. Unfortunately, although an attempt was made, it was not possible to confirm the presence of bubbles by electron microscope examination. Such a confirmation would be extremely difficult, however, for helium concentrations in the parts per trillion range.

Low Temperature Release

A model for the helium release near 300°C must fit the isothermal annealing results presented in Table 2. In particular, it should be consistent with the result that the release can best be described by

the sum of two exponential releases with relaxation times differing by a factor of roughly 10.

First, it is useful to consider the kinetics of gas desorption from a surface as an alternative rate-limiting step to that of volume diffusion. If the helium released is considered to have all originated at the sample surface, it will amount to only about 10^{-4} of a monolayer. Thus, we can consider the possibility that helium originally trapped somehow at a relatively low concentration of surface sites was desorbed during the annealing. It should be mentioned that this is not a likely possibility because there is little reason to believe that chemically inert helium will chemisorb on metal surfaces. The weak Van der Waals bond would be expected to only produce physical adsorption and then only at pressures well above and temperatures well below those in this experiment.⁹

In addition to the above arguments, it can be shown by consideration of the experimental and predicted pre-exponential factors for the time constants that simple surface desorption is not the rate-limiting step for the release. Using the classical model for surface desorption, the rate of release from the surface is $dc/dt = c_{\text{surf}}^{-1}$, where τ_{surf}^{-1} is the appropriate rate constant for the release.¹⁰ Ignoring entropy and geometrical factors that are near unity, this rate constant can be written as $\tau_{\text{surf}}^{-1} = \nu_{\text{surf}} \exp(-Q_{\text{surf}}/kT)$, where Q_{surf} is the energy of desorption from the surface, and ν_{surf} is the vibration rate at the surface and can be estimated to be 10^{12} s^{-1} . Comparing the desorption energy and pre-exponential factor with the results given in Table 2, we find first that 4000 cal/mole or less is not an unreasonable value to expect for the desorption energy.⁸ Next, comparing the pre-exponential factor for the rate constant, we find a large disagreement, enough to eliminate the surface desorption model. The value $\tau_{\text{surf}}^{-1} = \nu_{\text{surf}}^{-1} \sim 10^{12} \text{ s}$ is predicted for surface desorption, while a τ_0 value of roughly 30 s was determined experimentally. Although we can now reject the simple surface desorption hypothesis for the fast release in the isothermal anneal, the above analysis may not apply to the complex kinetics expected for a two-stage process such as we have observed. Thus, it is possible

that surfaces may have a role in the mechanism for the slow release. A manner in which a metal surface could trap helium is not known, however.

The experimental value for τ_0 of roughly 30 s is large compared to the literature values obtained for volume diffusion of substitutional (5×10^{-4} s) and interstitial ($5 \cdot 10^{-2}$ s) solutes.¹¹ Our data are in better agreement with prior results for interstitial diffusion than for substitutional diffusion. However, the lack of good agreement suggests that either there is not sufficient accuracy in the data for the extrapolation to obtain τ_0 within two orders of magnitude or the results cannot be interpreted in terms of simple volume diffusion. Some other complicating factor, such as a trapping effect caused by impurities, may be present.

Consider now the steady state predicted to exist in samples held at room temperature for long times compared to the relaxation time at this temperature (see Table 2). The rate of production of helium is

$$\frac{dc}{dt} = C_T \lambda, \quad (3)$$

where

$\lambda = 1.75 \times 10^{-9} \text{ s}^{-1}$ is the radioactive decay constant for tritium,

c is the helium concentration,

C_T is the tritium concentration.

Assuming the surface concentration of helium to be zero, the rate of loss of helium by diffusion is

$$-\frac{dc}{dt} = c/\tau. \quad (4)$$

Therefore, at steady state we have an average concentration of helium, given by

$$c = C_T \lambda \tau. \quad (5)$$

Curves for predicted values of this concentration vs temperature are shown in Fig. 5. They have been calculated using the values from Fig. 4 for the relaxation times for the fast and the slow releases. Also shown in Fig. 5 are the experimental values for the concentration of helium released in the fast and slow stages at each isothermal annealing temperature.

Two comparisons between the experimental results and those predicted by Eq. (5) will now be discussed. First, the gas release predicted for a given temperature should be the difference between the steady-state concentration at 30°C and at the appropriate temperature. For the concentrations calculated using the relaxation time for the slow release, the predicted release is nearly independent of temperature for the anneal temperatures investigated and has a magnitude of 10×10^{-10} atom fraction of helium. This agrees within a factor of 2 with the release observed experimentally at the various temperatures. On the other hand, when the fast release relaxation times are used to calculate the steady-state concentration, the predicted release differs by nearly two orders of magnitude from that observed experimentally. From this it appears that the steady-state concentration at room temperature is controlled by the slow rather than the fast reaction. It is suggested that a reason for this may be that at room temperature most of the helium does not exist as freely mobile atoms, but instead is trapped, possibly at tritium atoms or as a complex defect with vacant lattice sites.^{12,13} For such low concentrations of helium, regions near impurities and the steady-state Schottky defect concentration of approximately 10^{-9} atom fraction at 300°C could play a role.¹⁴

Second, it is noted that the amount of helium released at each temperature by the slow reaction (data points with circles in Fig. 5) is the same within experimental error as the steady-state concentration calculated using the relaxation time for release by that reaction. This agreement suggests that the slow release reaction actually involves release of the steady-state concentration of helium. This would only occur if tritium were also being released. Such a release of tritium is already known to take place because a loss of roughly 80% of the tritium was observed for the sample annealed at 300°C.

What is interesting in the above discussion is the inference that tritium release is the rate-controlling step for the slow reaction of helium release. This seems to be the most probable, although certainly not the only, explanation for the second (slow) stage of helium release. As a part of this explanation there is the requirement that a trapping type of interaction exists between atomic helium and tritium. This does not seem unreasonable. Further studies in which both the tritium and the helium concentrations are monitored will be necessary to properly demonstrate this interaction.

If the slow release stage is governed by diffusion of tritium from the sample, then the fast release stage can be assigned the mechanism of diffusion of atomic helium or a complex defect involving helium from the sample. This explanation is attractive because of its simplicity, i.e., that roughly 85% of the helium will freely diffuse and thus will obey exponential release kinetics, and that the remainder is trapped and becomes mobile only when the trapping species can diffuse. The partitioning of helium indicated above leads to a binding energy to traps of approximately 11,000 cal/mole (0.46 eV).

One final observation is that there appears to be a coincidental similarity between the second term in the infinite series describing the slow mechanism and the first term in the infinite series describing the fast mechanism.

Expanding Eq. (5) for the slow mechanism to two terms, we find

$$\bar{c} = c_0 \frac{8}{\pi^2} \left[\exp\left(\frac{-t}{\tau}\right) + \frac{1}{9} \exp\left(\frac{-9t}{\tau}\right) \right]. \quad (6)$$

If we now restrict our attention to the second exponential term, we can see that it could be written $\frac{1}{9} \exp[-t/(\tau/9)]$ or $\frac{1}{9} \exp(-t/\tau')$. The relaxation time for the second term, τ' , is predicted to be about 10 times smaller than τ . This is very similar to the relationship found between τ_s and τ_f . We can carry the analysis further by inquiring into the relationship of temperature dependence for diffusion coefficients predicted from τ and τ' . Let us expand τ' according to the equations given previously.

$$\tau' = \frac{\tau}{9} = \frac{h^2}{9\pi^2 D_o \exp\left(-\frac{Q_s}{RT}\right)}, \quad (7)$$

$$\tau' = \frac{h^2}{\pi^2 D_o \exp(2.197) \exp\left(-\frac{Q_s}{RT}\right)}. \quad (8)$$

Now let $T = 250^\circ\text{C}$, and

$$\tau' = \frac{h^2}{\pi^2 D_o \exp\left[-\left(\frac{-2283 + Q_s}{RT}\right)\right]}. \quad (9)$$

In Eq. (9) we see that in this form the predicted "activation energy" for diffusion in the second term should be around 2 kcal/mole less than that in the first term, which we used to characterize the slow mechanism. This 2-kcal/mole decrement is again strikingly similar to that observed for the activation energy difference found for the slow and fast mechanisms.

Despite these observations we are confident that two mechanisms do, in fact, exist and that our interpretation of the data is correct. This confidence depends heavily on the fact that the second exponential term in Eq. (6) can contribute only a small fraction of the total release, and we found that 85% of the total release could be attributed to the fast mechanism. This may be seen by comparing the curve of Eq. (1) with experimental data in Fig. 2. A two-mechanism description of the outgassing at low temperature thus retains both its mathematical and physical appeal.

In conclusion we wish to point out that probably the most significant result of this study is the determination that helium degassing occurs by simple exponential decay and thus fits the kinetics predicted for a volume diffusion mechanism. This indicates that the competitive processes of volume diffusion of atomic helium or of a helium complex and of degassing by diffusion of submicroscopic helium bubbles can be studied separately.

In our study this has been possible by working at concentrations low enough that helium agglomeration and precipitation occur in a temperature range well above that for volume diffusion. An additional condition that may be important to this experiment and the observation of exponential degassing kinetics is that helium was introduced by a method not expected to involve appreciable radiation damage. Future studies with the goal of understanding basic mechanisms for the behavior of helium may require use of the tritium decay method of helium introduction. For these studies further work will be needed to characterize the interaction between atomic helium and tritium suggested here.

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FIGURE TITLES

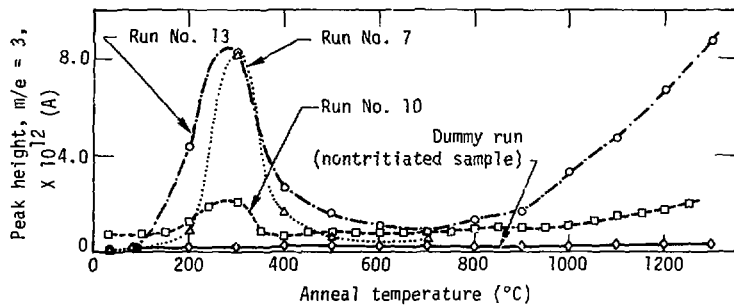
Fig. 1. Isochronal annealing curves for helium release from three different tritiated samples. The time at each temperature was 5 min.

Fig. 2. Semilogarithmic plot of the data for low temperature isothermal release of helium at four different temperatures. The solid lines that fit the data at long times are least-squares fits to Eq. (1), the solution to the diffusion equation for volume diffusion controlled degassing of a thin sheet. The lack of a good fit at short times is because the total degassing fits Eq. (1) for a two-step process. This figure shows the slow release step; the fast release is shown in Fig. 3.

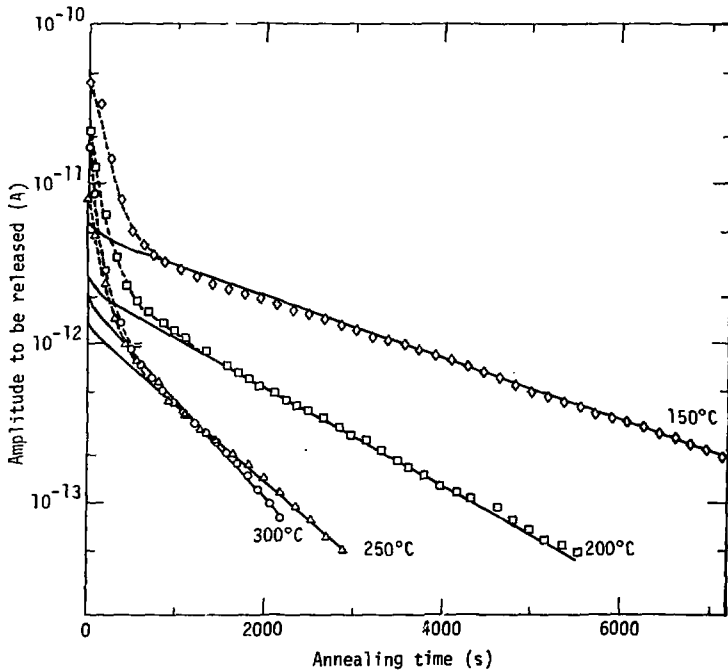
Fig. 3. Semilogarithmic plot of the low temperature isothermal helium release data after the release shown by the solid line in Fig. 2 has been subtracted. The solid lines are least-squares fits to Eq. (1).

Fig. 4. Arrhenius plot of the relaxation times for both the fast and slow release processes. The parameters obtained from the slopes and intercepts are presented in Table 2.

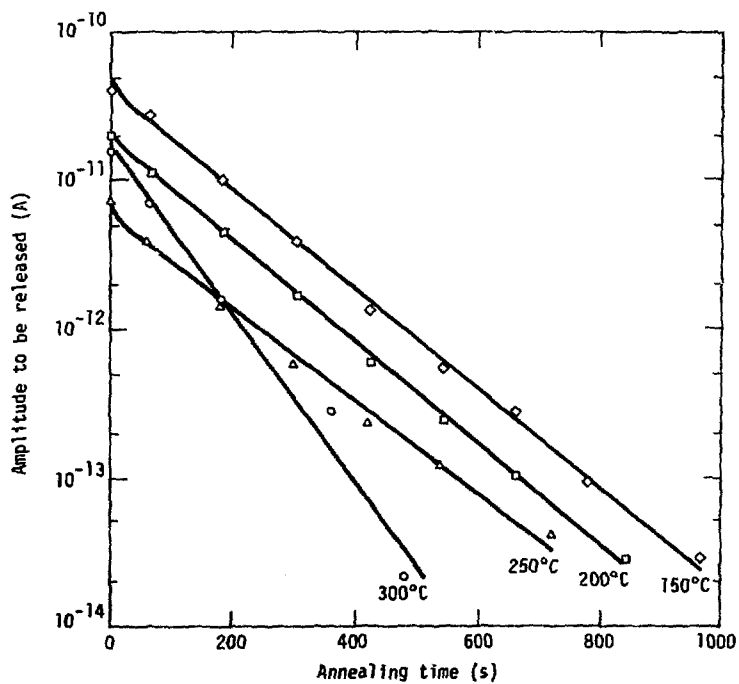
Fig. 5. Calculated and observed steady-state helium concentrations at low temperature.



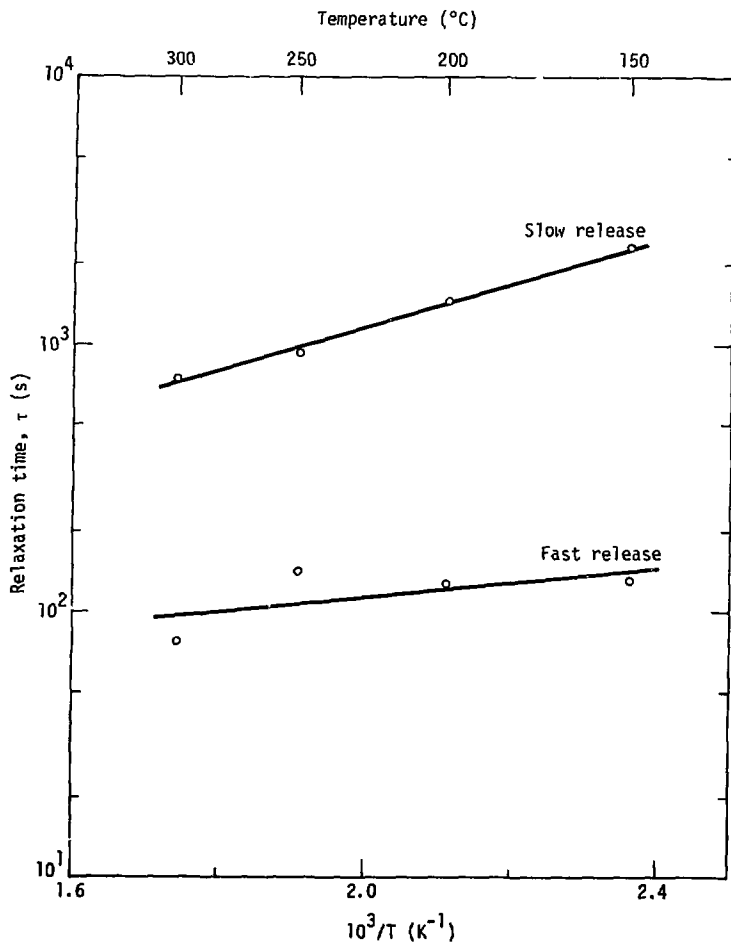
Cost - Fig. 1



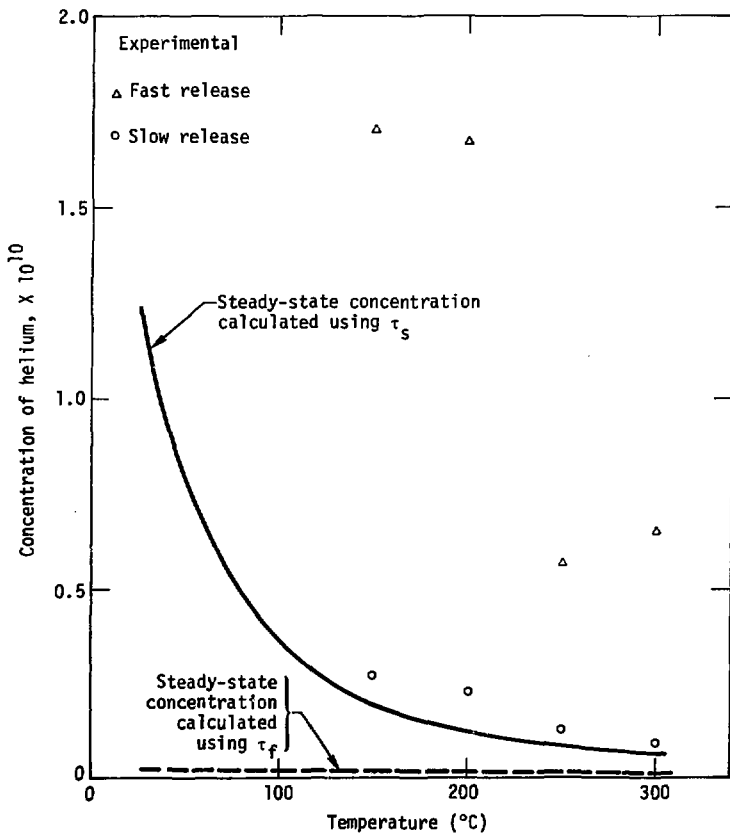
Cost - Fig. 2



Cost - Fig. 3



Cost - Fig. 4



Cost - Fig. 5