

DIFFUSION OF HELIUM IN FCC METALS

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Diffusion constants of helium in gold, silver and aluminium are determined from thermal desorption experiments, giving:

$$\text{Au: } D_0 = 10^{-1.0} \text{ cm}^2/\text{sec}, \quad \Delta H = 1.70 \text{ eV}$$

$$\text{Ag: } D_0 = 10^{-1.2} \text{ cm}^2/\text{sec}, \quad \Delta H = 1.50 \text{ eV}$$

$$\text{Al: } D_0 = 10^{+0.1} \text{ cm}^2/\text{sec}, \quad \Delta H = 1.35 \text{ eV}$$

The results are compared to self-diffusion and to the diffusion of other light elements in metals. Possible diffusion mechanisms are discussed.

INTRODUCTION

Helium is produced in metals by nuclear reactions of energetic particles such as reactor neutrons or light ions. Due to their low solubility¹ the helium atoms agglomerate into bubbles which have detrimental effects on mechanical properties.² Nucleation and growth of the bubbles strongly depends on the mobility of the helium.³ Nevertheless there have been rather few studies of helium diffusion in metals.⁴

Theoretical calculations⁵ indicate that helium is highly mobile as long as it occupies interstitial positions, but it becomes deeply trapped in vacancies or at other lattice defects like dislocations, grain boundaries, impurities or precipitates. Experimental results on interstitial diffusion of helium in nickel^{6,7} are contradictory, while only a few other metals have been investigated.⁸⁻¹⁰

So far three techniques have been applied to study the diffusion of helium in metals at temperatures above room temperature. In early attempts, the release of helium from homogeneously doped aluminium^{11,12} and magnesium¹³ was measured in isothermal experiments. Strong trapping and agglomeration severely hindered the helium mobility and restricted the observation of free migration to very short times.

In a second kind of experiment¹⁴ the decay of tritium was used to homogeneously dope type 304 stainless steel with helium. By this "tritium-trick" helium is produced without lattice damage. On the other hand this "advantage" seems questionable at temperatures above room temperature where interstitial helium atoms are mobile. The retained helium is then necessarily trapped at other sinks which are less well-defined than irradiation produced vacancies.

In a third type of experiment the release of helium from nickel was investigated simultaneously during α -implantation at elevated temperatures.¹⁵ This method also

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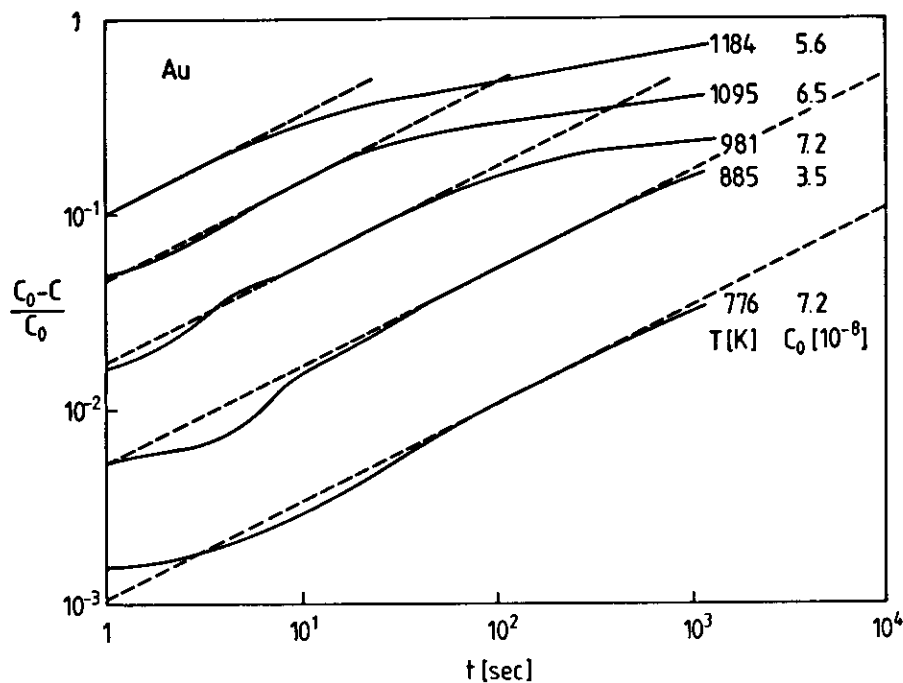


FIGURE 1 Fraction of helium released isothermally from $18 \mu\text{m}$ Au foils as a function of time. The initial helium concentrations c_0 and the temperatures are indicated. The t -axis for the individual curves is corrected by constant values given in Figure 2.

suffered from strong trapping and agglomeration which limited the experiments in nickel to a rather narrow temperature range and caused the method to fail for other metals.

In the present investigation, helium release from foils of various thicknesses was observed in isothermal as well as linear-heating experiments. The helium was introduced homogeneously throughout the foil thickness by α -implantation at variable energy. Investigation of the dependence of the release spectra on time, thickness and concentration made it possible to separate free diffusion from trapping and agglomeration processes.

EXPERIMENTAL DETAILS

Details of the α -implantation and the helium release apparatus have been described earlier.¹⁶ Materials used in the present investigation were 99.999% Al, 99.9999% Ag and 99.999% Au foils which were rolled to various thicknesses ranging from 5 to $50 \mu\text{m}$ and annealed before implantation for 1 h at 600°C (Al), 600°C (Ag) and 800°C (Au), respectively.

The furnace in the release apparatus consisted of an alumina tube of 84 mm length and 6.5 mm inner diameter and a tantalum heater. The specimens were dropped onto an alumina plate in the middle of the tube to which two W-Re thermocouples were attached. In some of the experiments a small molybdenum pot was put

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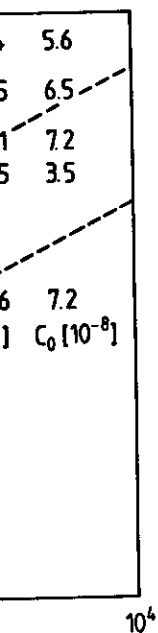
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RESULTS

Gold

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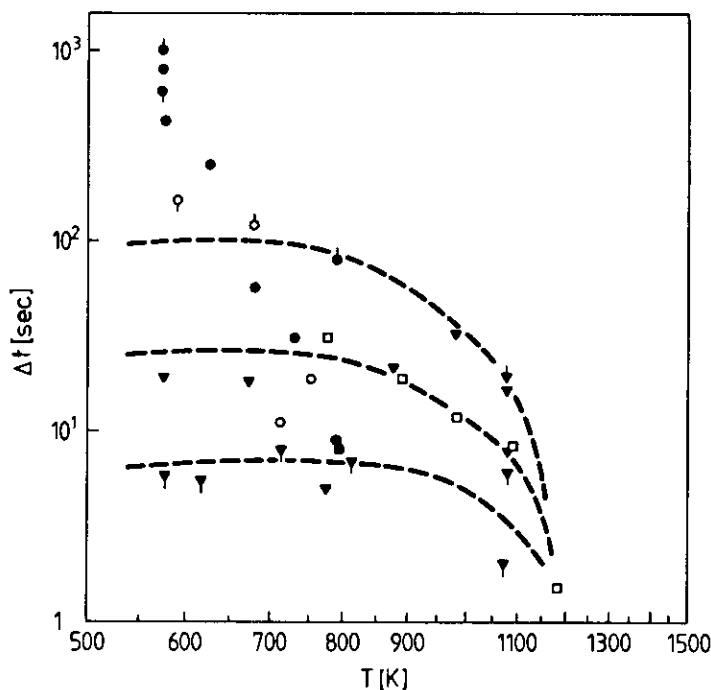


FIGURE 2 Correction values of the t -axis in isothermal release experiments used for Al (○), Ag (▽) and Au (□) as a function of temperature. Filled symbols indicate that a molybdenum pot was positioned in the furnace. Bars below the symbols indicate specimen thickness $\leq 6 \mu\text{m}$, bars on top $\geq 50 \mu\text{m}$, respectively. Symbols without bars represent specimens of 13 to 18 μm . The Δt -values from these three thickness regimes fall roughly on the dashed lines, except the low-temperature Al values.

on the plate in an attempt to improve the heat conduction from the furnace to the specimens.

RESULTS

Gold

Results of helium release from gold during linear heating have been published previously.¹⁶ It was found that helium diffuses freely, unaffected by clustering, in Au specimens for which the product of the thickness $d[\text{m}]$ and initial concentration c_0 [at. parts] is less than $4.6 \cdot 10^{-13} \text{ m}$. Then the total fraction of helium released at a given temperature was roughly proportional to d^{-1} and to $\beta^{-1/2}$ (β =heating rate), but did not depend on concentration c_0 .

The measurements in Ref. 16 have now been supplemented by isothermal experiments shown in Figure 1. Plotted is the released fraction $[c_0 - c(t)]/c_0$ of the initial concentration c_0 . Since the $c_0 \cdot d$ -values of these specimens were slightly above the limit given above in order to increase the sensitivity of the measurements, the curves deviate at the upper end from the straight (dashed) line predicted by diffusion

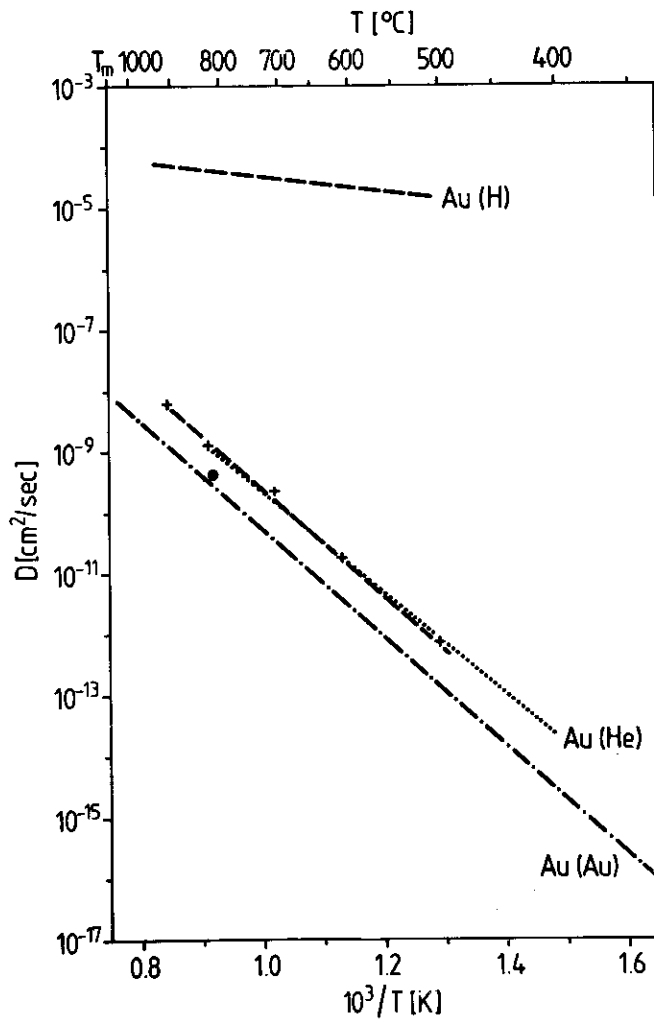


FIGURE 3 Diffusion constants of He in Au derived from the measurements in Figure 1. Included are results from linear heating experiments¹⁶ (dotted line) and a value (●) from Ref. 20. Results from self-diffusion¹⁸ (-·-·-), and diffusion of hydrogen in Au¹⁹ (- - -) are also shown.

theory:^{16,17}

$$\frac{c_0 - c}{c_0} = 2 \left(\frac{4Dt}{\pi d^2} \right)^{\frac{1}{2}} \quad \text{for} \quad \frac{c_0 - c}{c_0} \leq 0.5 \quad (1)$$

Equation (1) shows that free diffusion can only be assumed if $(c_0 - c/c_0)$ is independent of c_0 and is proportional to \sqrt{t} and d^{-1} for $(c_0 - c/c_0) \leq 0.5$.

One problem in these isothermal experiments is that the zero point of the time axis is not exactly known, since it takes some time for the specimen to attain temperature after being dropped into the furnace. Therefore a constant value Δt was subtracted from the time passed after dropping the specimen. Δt -values are given in

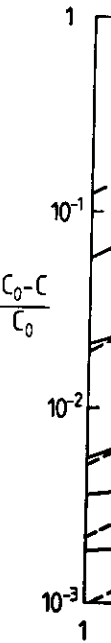


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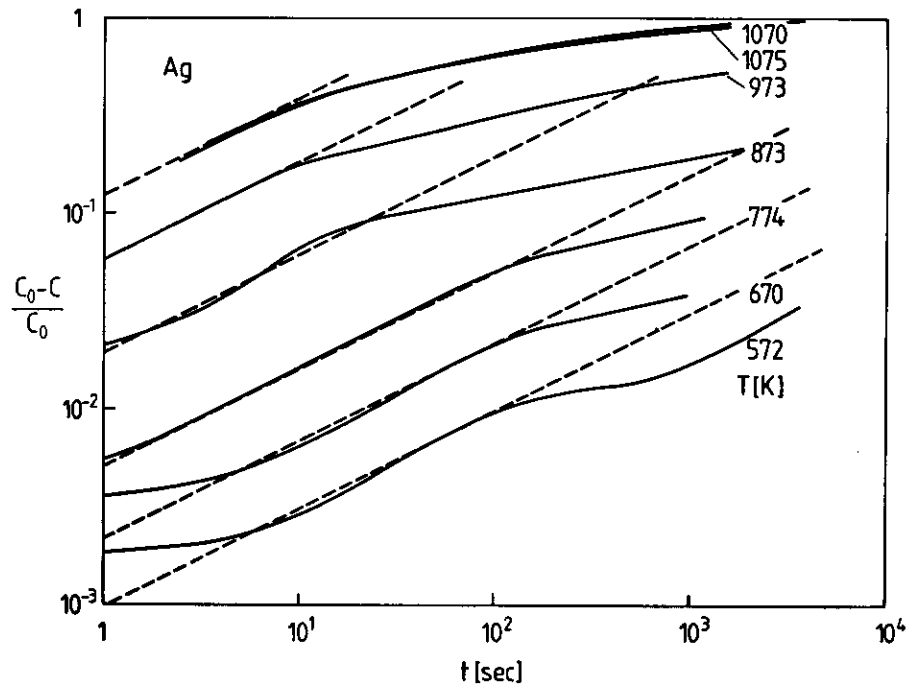


FIGURE 4 Fraction of helium released isothermally from 13 μm Ag foils as a function of time. The initial concentration was about $1.4 \cdot 10^{-8}$ in all specimens. The t -axis for the individual curves is corrected by constant values given in Figure 2.

Figure 2. These values are chosen such that the release curves fit the straight lines at small times as closely as possible. As long as the \sqrt{t} -law in the isothermal release experiment is obeyed over a time regime much larger than Δt , the uncertainty introduced by the choice of Δt seems tolerable. This is the case for Au and to some extent for Ag, but not for the low temperature results of Al (see below). The dashed lines in Figure 2 represent tentatively average delay times Δt for specimens of about 5, 18 and 50 μm , respectively. The almost constant Δt -values at low temperatures and the decreasing Δt values at high temperatures indicate that thermal conduction and heating by radiation are the dominating heat-transfer mechanism, respectively. Again the low temperature data for Al do not fit in this scheme. The molybdenum inset in the furnace had no noticeable effect.

Diffusion constants as a function of temperature are derived from the curves in Figure 1 by applying Eq. (1). In Figure 3 the results are compared to the results from the linear-heating experiments,¹⁶ to self-diffusion data¹⁸ and to diffusion of hydrogen¹⁹ in gold. The isothermal results agree with the linear heating results within the experimental error, giving on the average an Arrhenius behaviour of the diffusion constants:

$$D = 10^{-1.0 \pm 0.4} [\text{cm}^2/\text{sec}] \exp(-1.7 \pm 0.1 [\text{eV}]/kT) \quad (2)$$

A single D -value reported in the literature²⁰ is also in fair agreement.

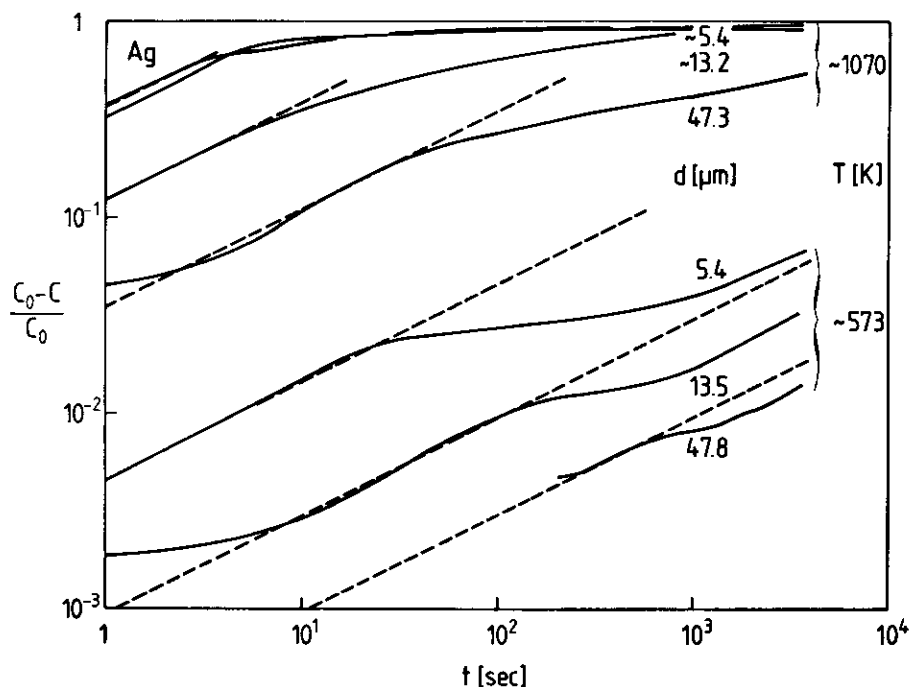


FIGURE 5 Fraction of helium released isothermally from Ag foils of different thicknesses at two temperatures as a function of time. Initial helium concentration was $4 \cdot 10^{-8}$ in the $5 \mu\text{m}$ foils and about $1 \cdot 10^{-8}$ in the other foils. The t -axis for the individual curves is corrected by constant values given in Figure 2.

Silver

Figure 4 shows isothermal release curves of $13 \mu\text{m}$ Ag foils containing $1.4 \cdot 10^{-8}$ atomic parts of helium. The t -axis is corrected by about $\Delta t = 20$ sec (Figure 2). Despite the lower thickness and helium concentration, deviation from the \sqrt{t} -behaviour occurs at lower $(c_0 - c/c_0)$ values than in the case of Au, possibly indicating a stronger tendency to agglomeration.

In Figure 5 isothermal release curves are shown as a function of specimen thickness for two different temperatures. The corrections Δt of the t -axis are included in Figure 2. Obviously the $13 \mu\text{m}$ Ag foils need on the average about twice as long as the $5 \mu\text{m}$ foils to reach the furnace temperature.

In Figure 6 diffusion constants of He in Ag derived from the isothermal release experiments are plotted versus temperature. Above about 500°C the data show a similar temperature dependence as self-diffusion.¹⁸ At lower temperatures a much lower temperature dependence is observed. At these temperatures also the dependence on thickness seems to deviate from Eq. (1) at least for the $5 \mu\text{m}$ foils. Therefore at present only the high temperature data can reliably be ascribed to free bulk diffusion:

$$D = 10^{-1.2 \pm 0.5} [\text{cm}^2/\text{sec}] \exp(-1.5 \pm 0.1 [\text{eV}]/kT) \quad (3)$$

The figure includes results of diffusion of Ar,²¹ Kr²² and Xe²³ in Ag which compare

FIGURE 6 according to Included are Ag-H,²⁸ and

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Aluminium

Figure 7 shows specimens of

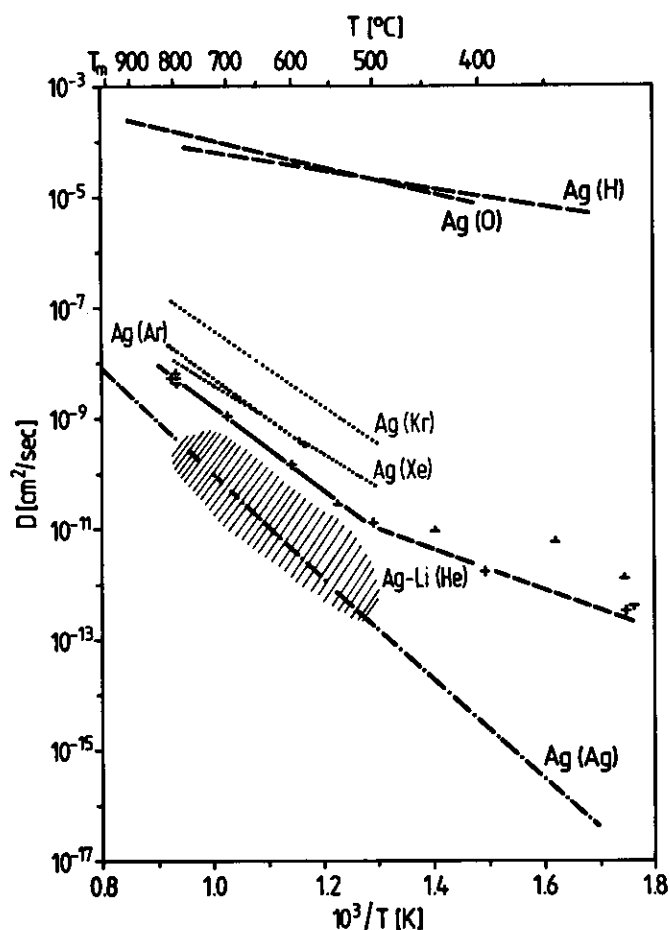


FIGURE 6 Diffusion constants of He in Ag derived from isothermal release experiments according to Eq. (1) for foil thicknesses of 5 μm (\perp), 13 μm (+) and 47 μm (\top), respectively. Included are results for Ag-Ar,²¹ Ag-Kr,²² Ag-Xe,²³ He in Ag-Li (shaded area),²⁵ Ag-O,²⁷ Ag-H,²⁸ and self-diffusion ($-$).¹⁸

reasonably with the present results for helium. Another investigation on the diffusion of Kr in Ag²⁴ reproduced the results of Ref. 22 fairly well but showed deviations for other specimen geometries. Figure 6 also includes diffusion constants on helium diffusion in a Ag-Li alloy²⁵ which lie below the present results. Still lower D -values were obtained in a study on Ag(He)²⁶ with He-concentration above 10^{-6} . These high concentrations were probably the reason why the diffusion constants depend on concentration in Ref. 26.

Aluminium

Figure 7 shows the integral release during linear heating (50 K/min) of two aluminium specimens of different thicknesses with helium concentration of about 10^{-8} . The

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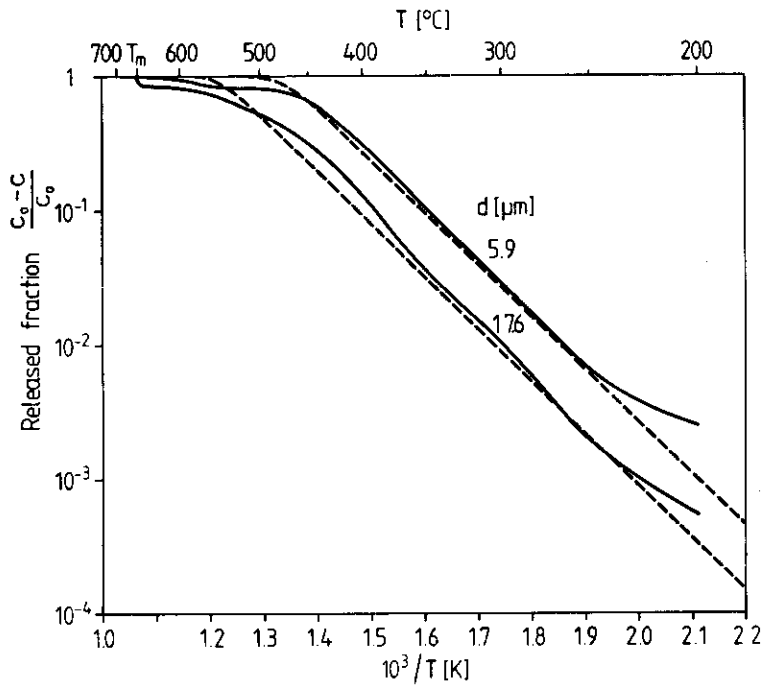


FIGURE 7 Released fraction of helium in Al as a function of reciprocal temperature during linearly heating at a rate of 50 K/min. The initial He concentrations c_0 were $1.8 \cdot 10^{-8}$ in the $5.9 \mu\text{m}$ and $1.4 \cdot 10^{-8}$ in the $17.6 \mu\text{m}$ specimen, respectively.

dotted lines give calculated curves for free diffusion with $D_0=12$ [cm²/sec] and $\Delta H=1.45$ [eV]. Deviations from these curves at the upper ends may be ascribed to agglomeration.

In Figure 8 isothermal release curves of $18 \mu\text{m}$ Al foils are shown. For the three lower temperature curves the corrections Δt on the t -axis which are necessary to match a \sqrt{t} behaviour exceed 100 sec (Figure 2). These high values and the significant deviations from the \sqrt{t} behaviour at small times makes this procedure questionable for these temperatures. Figure 9 further shows that at low temperatures also the dependence on the thickness shows deviations from Eq. (1). Therefore—as was the case for Ag—free diffusion can only be assumed at temperatures above about 650 K.

In Figure 10 diffusion constants derived from the isothermal release experiments are compared to earlier results from other authors.^{11,12} While the results of Ref. 12 seem too low, the results of Ref. 11 show fair agreement with the present results between 450 and 550°C. Below this range the data of Ref. 11 scatter appreciably. The solid line corresponds to

$$D = 10^{0.1} [\text{cm}^2/\text{sec}] \exp(-1.35 [\text{eV}]/kT) \tag{4}$$

which is considered as a compromise to the linear-heating as well as the isothermal results. Included in Figure 10 are self-diffusion data¹⁸ and results for diffusion of hydrogen in Al.²⁹

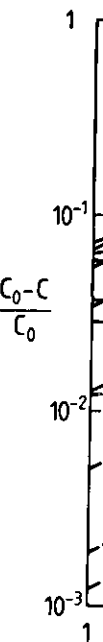


FIGURE 8 Isothermal release curves of $18 \mu\text{m}$ Al foils. The indicated temperature corrections Δt are given in Figure 2.

DISCUSSION

It is assumed that the helium is trapped in vacancies, and that the release is controlled by two diffusion processes:

- 1) The first process is the diffusion of substitutional helium, given by:

$$\Delta H^S = E_V^F$$

where E_V^F is the activation energy of a vacancy of a complex. If the release is controlled by a vacancy, one can write:

$$\Delta H^{S2} = E_V^F + Q_2$$

where Q_2 is the energy of the second process.

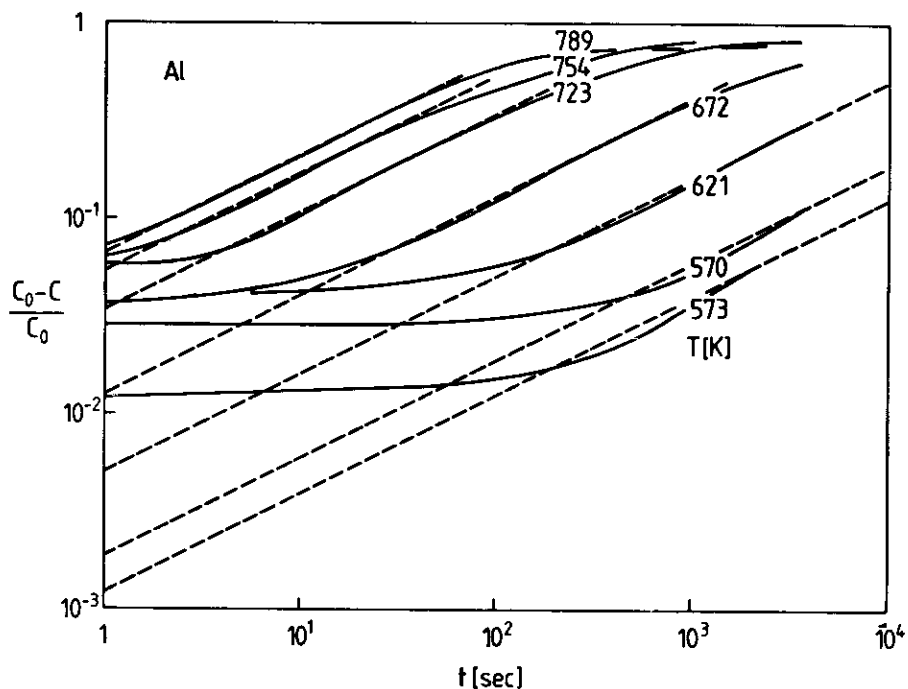


FIGURE 8 Fraction of helium released isothermally from Al-foils as a function of time at the indicated temperatures. All specimens were about 18 μm thick and contained about 1 · 10⁻⁸ atomic parts of helium. The *t*-axis for the individual curves is corrected by constant values given in Figure 2.

DISCUSSION

It is assumed that after room temperature implantation, all helium atoms are trapped in vacancies, that means in substitutional positions. Mobility may be then regained by two diffusion mechanisms.

1) The first is the usual vacancy mechanism which is assumed to operate in most substitutional alloys. The activation energy for this mechanism in the case of He is given by:

$$\Delta H^S = E_V^F - E_{HeV,V^B} + E_{HeV,V^M} \tag{5}$$

where E_V^F is the formation energy of a single vacancy, E_{HeV,V^B} its binding energy of a vacancy to a substitutional helium atom and E_{HeV,V^M} is the migration energy of this complex. As the last two quantities are unknown, only estimates of ΔH^S are possible: If the substitutional-helium-vacancy-complex is approximated by a divacancy, one obtains:

$$\Delta H^{S2} = E_V^F - E_{2V^B} + E_{2V^M} = Q_2 - E_V^F \tag{6}$$

where Q_2 is the activation energy for self-diffusion by the divacancy mechanism. If,

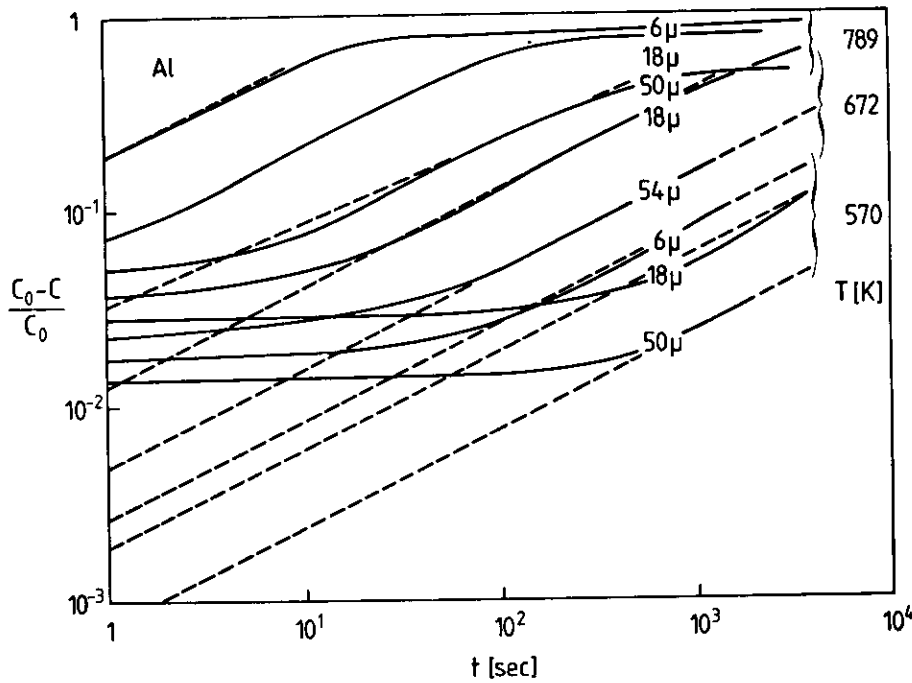


FIGURE 9 Fraction of helium released isothermally from Al foils of different thicknesses at three temperatures as a function of time. He concentrations in the foils were about $1 \cdot 10^{-8}$. The t -axis for the individual curves is corrected by constant values given in Figure 2.

on the other hand, the substitutional helium behaves like a normal lattice atom, ΔH^S is equal to:

$$\Delta H^{S1} = E_V^F - O + E_V^M = Q_1 \tag{7}$$

the self-diffusion energy for the single vacancy mechanism.¹⁸ As $Q_2 - E_V^F < Q_1$, ΔH^{S2} and ΔH^{S1} give lower and upper limits of ΔH^S , respectively.

If the helium is considered as a normal lattice atom, the preexponential factor of the diffusion constant of He would be equal to the preexponential factor D_{10} for tracer self-diffusion:¹⁸

$$D_0^{S1} = D_{10} \tag{8}$$

2) If the vacancy formation energy is high it may be advantageous for the helium to jump out of its substitutional position and migrate interstitially. In this case of dissociation, the diffusion constant D^d is determined by the dissociation frequency of the substitutional helium ν_a , the distance per single interstitial jump, a , and the number of interstitial jumps, n_j , performed until retrapping in another vacancy occurs:

$$D^d = \frac{1}{6} \nu_a \cdot a^2 \cdot n_j \tag{9}$$

FIGURE 10 according to Eq. Included are re of H.²⁹

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$$\nu_a = \frac{c^s}{c^s + c^i}$$

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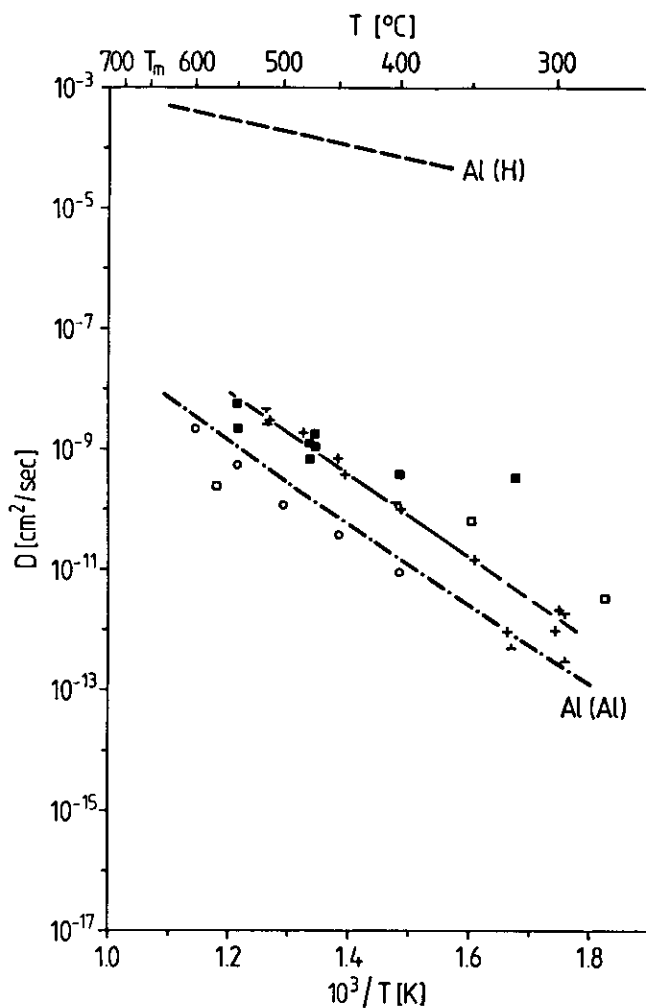
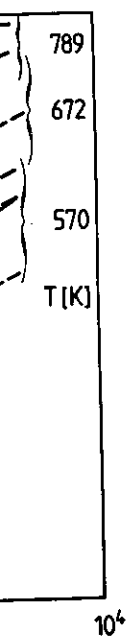


FIGURE 10 Diffusion constants of He in Al derived from isothermal release experiments according to Eq. (1) for foil thicknesses of 6 μm (\perp), 18 μm (+) and 50 μm (∇), respectively. Included are results of helium diffusion (\square \blacksquare)¹¹ and (\circ),¹² of self-diffusion¹⁸ and of diffusion of H.²⁹

For an fcc-lattice (octahedral sites), $a^2 = a_0^2/2$ (a_0 = lattice constant). n_i is inversely proportional to the vacancy concentration and ν_a is given by:

$$\nu_a = \frac{c^s}{c^s + c^i} \cdot \nu_0 \exp[-(E_{\text{He}V^B} + E_{\text{He}^M})/kT] \tag{10}$$

ν_0 is the attempt frequency, $E_{\text{He}V^B}$ is the energy difference between interstitial and substitutional position of a helium atom, and E_{He^M} is the migration energy of interstitial helium. c^s and c^i are the helium concentrations in substitutional and interstitial positions, respectively. For $c^i \ll c^s$ an Arrhenius behaviour is obtained



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TABLE I
Comparison of preexponential factor D_0 [cm²/sec] and activation energy ΔH [eV] to estimates for the vacancy diffusion mechanism and the dissociation mechanism

Metal/Mechanism	D_{10}	Vacancy ^a		Dissociation ^b		Experiment	
		ΔH^{S1}	ΔH^{S2}	ΔH^d	D_0	ΔH	
Al	$10^{-1.33}$	1.28	0.92	0.88	$10^{+0.1}$	1.35	
Ag	$10^{-1.40}$	1.76	1.06	0.84	$10^{-1.2}$	1.50	
Au	$10^{-1.40}$	1.76	1.42	1.35	$10^{-1.0}$	1.70	
Ni	$10^{-0.04}$	2.88	2.12 ^c	0.52 ^d	$10^{-2.2e}$	0.81 ^e	

^a Ref. 18.

^b E_{HeV}^B , E_{He}^M from Ref. 5, E_V^F from Ref. 32.

^c E_V^F from Ref. 33.

^d Experimental values for ΔH from Ref. 34.

^e Ref. 15.

with an activation energy:

$$\Delta H^d = E_{HeV}^B + E_{He}^M - E_V^F \quad (11)$$

In Table I activation energies calculated from Eqs. (6), (7) and (11) are compared to the present experimental data and to results on nickel.¹⁵ Included are self-diffusion data for D_{10} ¹⁸ which may apply for the vacancy mechanism.⁸ It may be estimated that the preexponential for the dissociative mechanism is smaller than for the vacancy mechanism. But this estimate as well as the experimental preexponentials contain sufficient errors to prevent a detailed comparison.

The activation energies clearly favour the substitutional mechanism for Al, Ag and Au and the dissociative mechanism in the case of Ni. On the other hand, these statements strongly rely on the theoretical predictions on E_{HeV}^B and E_{He}^M .⁵ For example, in the case of gold only a small increase in these theoretical values would make such a decision doubtful.

The activation energies for Al, Ag and Au further indicate that the substitutional helium atom is better treated as a host atom (ΔH^{S1}) than as a vacant lattice site (ΔH^{S2}).

It cannot be safely decided whether the results on Ag indicate a change in the dominating diffusion mechanism around 500°C or the existence of easy diffusion paths, like dislocations.

CONCLUSIONS

1) Measurement of helium release in isothermal and linear-heating experiments as a function of time (or heating rate), thickness and initial concentration made it possible to determine diffusion constants of isolated helium atoms in Al, Ag and Au.

2) Consistent results for Al and Ag are obtained only at temperatures above about $0.6 T_M$.

3) Comparison with theoretical estimates favours the vacancy mechanism for helium diffusion in Al, Ag and Au and a dissociative mechanism in the case of Ni. Some evidence exists in Ag for a change of the operating mechanism with temperature.

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