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Enhancement of Sodium Self-Diffusion by Additions of Potassium[†]

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(Received 22 November 1971)

The diffusion coefficient of ^{22}Na has been measured in dilute potassium-sodium alloys (0–1.2 at.% potassium) at temperatures of 0 and 50 °C. Within experimental error a linear enhancement of the diffusion coefficient was observed at both temperatures. The data are analyzed in terms of different pairs of diffusion processes being responsible for the diffusion. While it is not possible to exclude other possibilities, the divacancy-vacancy explanation appears to be the most preferable.

I. INTRODUCTION

The unique determination of the mechanism of self-diffusion in a metal frequently requires the use of a number of different experimental techniques. The vacancy mechanism for diffusion in fcc metals was most clearly established by both measurements of thermal expansion and x-ray lattice parameter as a function of temperature and the isotope effect for self-diffusion. Further confirmation of the vacancy mechanism has come from measurements of the effect of pressure on self-diffusion, measurements of impurity diffusion, and the effect of solute additions to the self-diffusion coefficient. Although all these techniques, except that of influence of solutes, have been applied to sodium, it has not proved possible to uniquely establish the mechanism of diffusion. Indeed, recent measurements[1] show that more than one mechanism is operative in sodium and not one of these has been uniquely identified. The analysis of Neumann[2] of diffusion data in bcc metals indicates that sodium is not unusual and that the data for very few bcc metals truly justify the consideration of only one mechanism. The effect of potassium additions on the sodium self-diffusion coefficient have been measured in an attempt to identify one or more of the diffusion mechanisms.

In most experimental studies, the variation of the self-diffusion coefficient with small solute addition ($< 2\%$) is given by

$$D(c) = D(0)\ (1 + bc), \qquad (1)$$

where c is the atomic fraction of the solute, and $D(c)$ and $D(0)$ are the diffusion coefficients of a solvent tracer in the alloy and pure solvent, respectively. Values of the enhancement factor b have been calculated for fcc metals in terms of three jump-frequency ratios by Lidiard[3] and by Howard and Manning.[4] The same three jump-frequency ratios are needed in the determination of the correlation factor for solute diffusion f_i and the ratio of solute to self-diffusion coefficient $D_i/D(0)$. The three experimentally measured quantities f_i, b, and $D_i/D(0)$ are interrelated by Eq. (1) and by the following equation given by Lidiard[3]:

$$f_i = 1 - \frac{4f_0}{b + 18}\left(\frac{D_i}{D(0)}\right), \qquad (2)$$

where f_0 is the correlation factor for the pure solvent. Measurements of these three quantities allows both a determination of the three jump-fre-

quency ratios and also a comparison of values of f_i determined from isotope-effect experiments and those obtained from Eq. (2). These measurements have been made by Peterson and Rothman[5,6] on the Zn-Ag and Zn-Cu systems and showed that the theories are consistent with the experimental data. Indeed the excellent agreement strongly suggests the essential validity of the theories and the value in applying the same treatment to other systems.

It is in the noble metals where impurity diffusion seems to be well understood in terms of the screened-interaction potential introduced by Lazarus[7] and more fully developed by LeClaire.[8,9] The theory is based on a vacancy mechanism and shows that the addition of more electropositive solutes, which will interact attractively with vacancies at nearest-neighbor distances, will lead to an increase in the total vacancy concentration. Similarly the addition of a less electropositive solute, will lead to a decrease in the total vacancy concentration. The interaction of the solute atoms with neighboring vacancies will perturb the jump frequencies of the solvent atoms in that immediate neighborhood. It is these two effects of solute additions which determine the size and magnitude of b. Values of f_i determined from this screened-interaction model agreed quite well with those determined by Peterson and Rothman[5,6] for the Zn-Ag and Zn-Cu systems.

The above theories need modification when applied to the effect of solute additions to lead self-diffusion. It has been shown by Miller[10] that the theory of Howard and Manning[4] leads to a certain minimum enhancement factor that is compatible with solute diffusion by a vacancy mechanism. The vast majority of observed enhancement factors are much greater than the minimum value. However, measurements of the effect of Cd, Au, and Ag additions to lead[10,11] gave enhancement factors less than this minimum value and provide strong evidence for solute diffusion by a mechanism other than vacancy.

Recently LeClaire[12] has calculated the solute enhancement factor for dilute bcc alloys. The theory takes account of the solute-vacancy interactions at both first- and second-nearest neighbors because the distances are not too different. In the case of a bcc structure there is also a minimum value of b given by LeClaire by the inequality

$$b + \frac{1.53b + 9.18}{b + 9.06} > -15 + 6f_0 \frac{D_i}{D(0)} . \tag{3}$$

Values of $D_i/D(0)$ are available[13] for the Na-K system over a range of temperature from 0 °C to the melting point. This is one of the few systems where the measurements were available and where the solubility of an impurity in sodium[14] was sufficiently high for enhancement-effect measurements to be made.

The low-temperature diffusion process in sodium could be interpreted as being due to an interstitial mechanism.[1,15,16] One would not expect an enhancement effect from solute additions if an interstitial mechanism was responsible for the diffusion. Unfortunately in sodium over most of the measured temperature range, at least two processes appear to be operative. To make measurements of the enhancement factor in a region where over 95% of diffusion was occurring by the low-temperature process it would be necessary to make the anneals at 200 °K. At this low temperature potassium is essentially insoluble in sodium and so a compromise had to be made.

The maximum solid solubility of potassium in sodium (3.1 at.%) is at 289 °K.[14] The phase diagram shows that at both 273 and 323 °K the solubility should be in excess of 2 at.%. Measurements in this range of concentration should allow the determination of b at both temperatures. In pure sodium at 273 °K, approximately 65% of the diffusion occurs by the low-temperature process and at 323 °K this has reduced to 45%. Measurement of b at both temperatures should strongly reflect the nearly zero enhancement effect that would result from an interstitial mechanism, if the low-temperature process is indeed the interstitial mechanism.

The good agreement between the enhancement theories and the experimental data on the noble metals suggests that the effect of neglecting long-range interactions and relaxation around defects is not important. While these effects may be more important in the application of similar enhancement theories to dilute bcc alloys, they have been neglected in the present analysis.

A. Experimental Method

The alloys were prepared from sodium of 99.9995% purity, which had been distilled at Argonne and potassium of 99.97% purity which was supplied by United Mineral and Chemical Corp. The different concentrations were made by adding different volumes of potassium to a given volume of sodium. These volumes were reasonably well defined by extrusion of the alkali metal from a stainless-steel die of known orifice diameter and cutting a measured length defined by two fixed razors. The extrusion was made in a dry box under argon gas. The pieces of sodium and potassium were immediately placed in a stainless-steel chamber which was evacuated and back filled with dry argon to cut down on oxidation of the alkali metal. The stainless-steel chamber was heated to 130–150 °C and vibrated mechanically to promote mixing. After 1 h in the molten state the alloy was forced through a stainless-steel frit (pore size ~10 μ) and cast into a 1.9-cm-diam sample

holder. After cooling, test samples were cut into many pieces and chemical analysis showed that the potassium was uniformly distributed. This method of analysis only shows that with the cooling procedure given below that the distribution of potassium was uniform on a macroscopic level.

The cooling procedure for the cast samples had to be carefully controlled. Too rapid cooling resulting from complete removal of heat sources caused extremely brittle samples for alloys containing greater than 0.6 at.% K. These samples would break apart along well-defined grain boundaries when cut with a razor. The fracture surfaces had a very shiny almost liquidlike appearance. The work of Forty[17,18] on ultraviolet light transmission through dilute sodium-potassium alloys gave strong evidence of segregation of sodium to grain boundaries in the potassium. We did not chemically analyze the fracture surfaces of our potassium-sodium alloys. The observed faster oxidation rate of the boundaries as compared to that in the bulk material would suggest that the brittle nature of fast-quenched alloys was a result of potassium segregation to the grain boundaries causing a molten region at the boundary.

To ensure that the samples had a homogeneous distribution of potassium, the samples were cooled slowly through the solidus line (~0.1 °C/min). The cooling was continued until the temperature was a few degrees below the solidus line as determined from the phase diagram.[14] Using this procedure the grains formed in the samples were 3 mm or more in diameter. The samples were held at the anneal temperature long enough for complete homogenization to take place. These times were of the order of one to three days, depending on the anneal temperature which, in turn, depended on the alloy concentration. Considerable difficulty was found in making alloys of greater than 0.9 at.% potassium. As can be seen from the results, one sample was obtained with greater than 1.0 at.%, but analysis of this sample after the diffusion anneals, indicated a great departure from uniformity.

Diffusion anneals were made at two temperatures 323 and 273 °K. The first temperature was obtained using an oil bath which was controlled with a platinum resistance thermometer to better than ±0.03 °C. The second anneal temperature was obtained with an ice bath. The anneal temperatures were measured with standardized mercury-in-glass thermometers. From the evaporation time through the diffusion anneal and until the sectioning was completed, the temperature of the samples was continuously monitored with calibrated copper-constantan thermocouples. These continuously monitored temperatures were used to determine the heat-up and quench times for each diffusion anneal. Active sodium was prepared from ^{22}NaCl

purchased from the Nuclear Science Division, International Chemical and Nuclear Corp. The evaporation, sample handling, sectioning, counting, and weighing procedures have been described elsewhere.[1,19]

Each alloy was used first for the 323 °K anneal and then the 273 °K anneal. The first evaporation was made with the sample at approximately 283 °K and the sample annealed at 323 °K before quenching to 283 °K for the sectioning. Sections were taken after the 323 °K anneal until no trace of radioactivity was found. The sample was then used for the ice-point anneal for which the evaporation was made on to a sample at approximately 273 °K. While the solid solubility of potassium in sodium below 280 °K is not clearly defined, the quenching experiments of MacDonald et al.[14] indicated a considerable decrease of solid solubility below 280 °K. After the ice-point anneal the samples were rapidly quenched to approximately 250 °K and sectioned. The errors in anneal time using the above procedure were of the order of 0.5% for all the diffusion anneals.

Chemical analysis was made of the middle section of each diffusion anneal, and in a number of cases analyses were made of sections from the end of the anneals. For any alloy the analysis showed homogeneity within plus or minus a few percent with the one exception of the alloy of over 1 at.% potassium. In this alloy, the analysis of the section taken from the 323 °K temperature anneal showed 1.25 at.% K and the 273 °K temperature anneal showed 1.05 at.% K. This large difference in potassium content indicates an inhomogeneous sample. The concentration profiles were accurately linear over two orders of magnitude, and so the data points have been included in the results.

B. Experimental Results

The solution of the diffusion equation for the experimental conditions used in this work is

$$c_x = c_0 \, e^{-x^2/4Dt} \quad , \tag{4}$$

where c_x is the specific activity of the tracer at a distance x from the surface, t is the time of anneal, and D is the diffusion coefficient. The penetration profiles obtained in this work were linear over two or three orders of magnitude in the concentration (see Fig. 1). Lack of homogeneity in the alloys could have led to small regions of high-diffusivity paths. This would have resulted in nonlinear concentration profiles or possible "tails" on the profiles at low specific activity. In not one case was there any evidence of tails.

Values of the diffusion coefficient were obtained by computer from a least-squares fit of the data and are given together with their alloy composition in Tables I and II. As can be seen from a com-
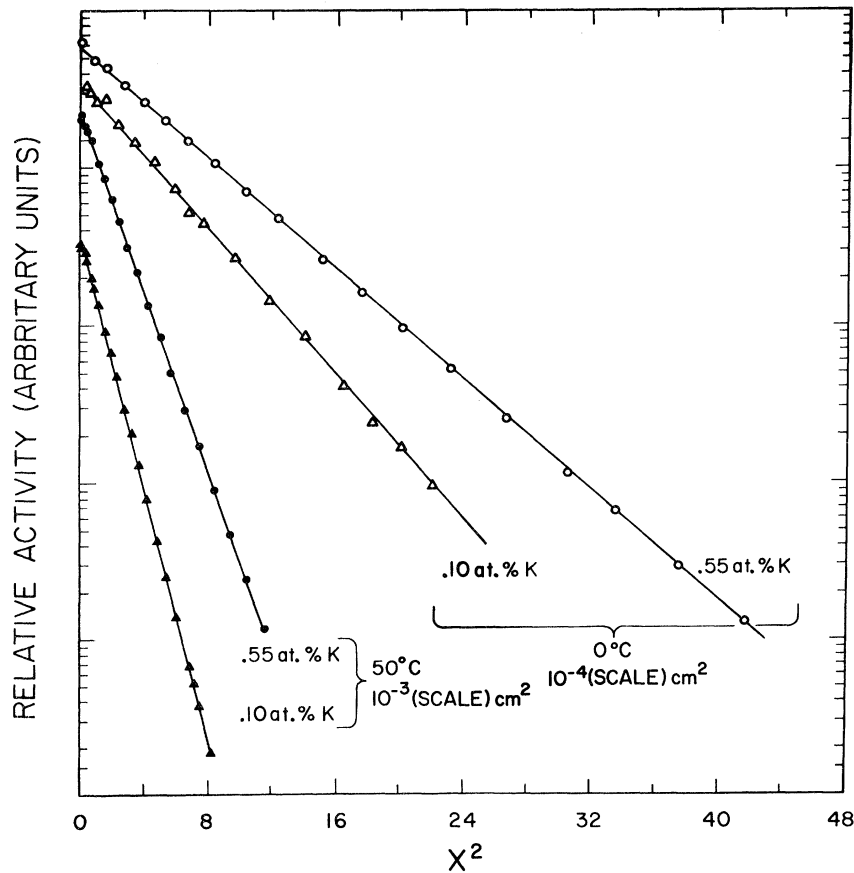
FIG. 1. Typical concentra-
tion profiles for the diffusion
of ^{22}Na into sodium–potassium
alloy.

parison of these two tables, it was not always pos-
sible to obtain diffusion anneals at both temperatures
on one sample. When the experiments were repeated
with another sample, it was not possible to exactly
match the alloy compositions. The values $D(c)/$
$D(0) - 1$ determined in Tables I and II are plotted in
Fig. 2 as a function of composition for each tem-
perature. The lines through the data are drawn
to show the apparent curvature. The least-squares
values of b from Eq. (1) omitting points above

1.0 at.% potassium are 41 at 50 °C and 63 at 0 °C.

II. DISCUSSION

One theory that has been used to explain the en-
hancement of solvent diffusion was that proposed
by Nachtrieb et al.[20] and further developed by Van
den Beukel.[21] This theory relates the rate of self-
diffusion to the slope of the solidus line of the bi-
nary alloy. The values of b determined in this way
have been shown to give a satisfactory fit for a
number of solutes in silver. For close-packed
metals where a single-vacancy mechanism of dif-

TABLE I. Diffusivity as a function of potassium com-
position at 0 °C.

Alloy composition (at.% K)	$D(c)$ (cm^2 sec^{-1})	$\dfrac{D(c)}{D(0)} - 1$
0.00	$1.34 \pm 0.01 \times 10^{-9}$	0.00
0.13	$1.38 \pm 0.04 \times 10^{-9}$	0.034
0.14	$1.44 \pm 0.02 \times 10^{-9}$	0.079
0.19[a]	$1.54 \pm 0.06 \times 10^{-9}$	0.15
0.33	$1.58 \pm 0.01 \times 10^{-9}$	0.18
0.49	$1.81 \pm 0.03 \times 10^{-9}$	0.35
0.56	$1.79 \pm 0.03 \times 10^{-9}$	0.33
0.67	$1.90 \pm 0.02 \times 10^{-9}$	0.42
1.03	$2.12 \pm 0.03 \times 10^{-9}$	0.58

[a]Corrected for deformed sample.

TABLE II. Diffusivity as a function of potassium
composition at 50 °C.

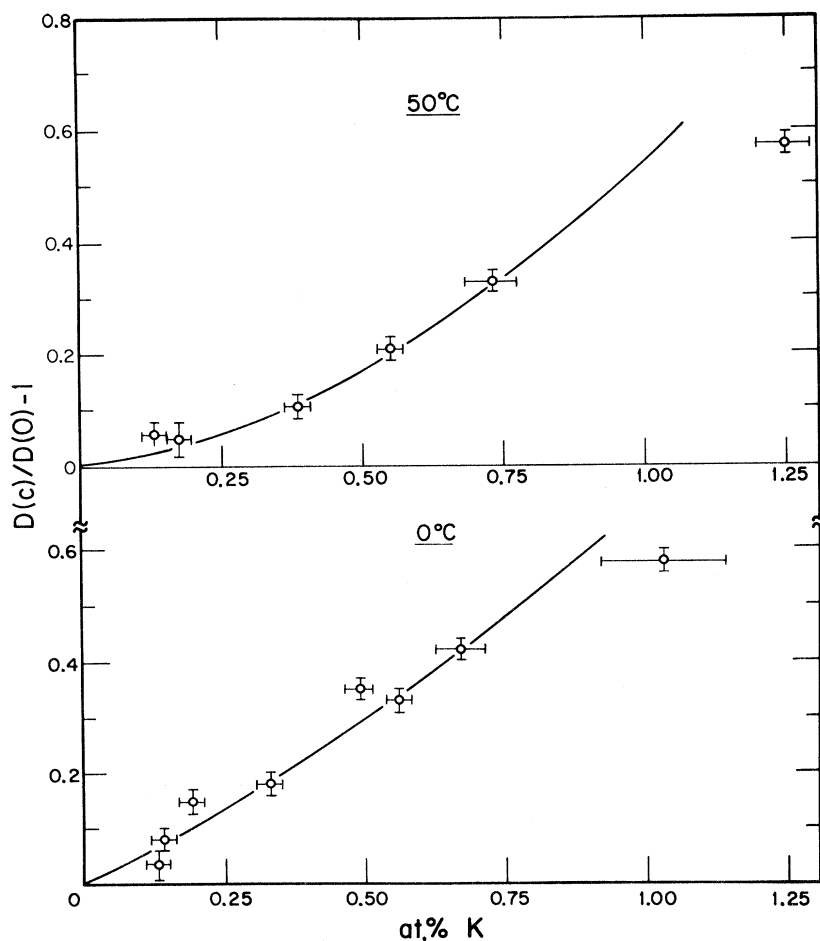| Alloy composition (at.% K)	$D(c)$ (cm^2 sec^{-1})	$\dfrac{D(c)}{D(0)} - 1$
0.00	2.16×10^{-8}	0.00
0.13	$2.28 \pm 0.02 \times 10^{-8}$	0.06
0.18[a]	$2.26 \pm 0.06 \times 10^{-8}$	0.05
0.38	$2.39 \pm 0.03 \times 10^{-8}$	0.11
0.55	$2.61 \pm 0.03 \times 10^{-8}$	0.21
0.73	$2.86 \pm 0.02 \times 10^{-8}$	0.32
1.25	$3.40 \pm 0.03 \times 10^{-8}$	0.57

[a]Corrected for deformed sample.

FIG. 2. Enchancement of ^{22}Na diffusivity in sodium as a function of potassium impurity concentration.

fusion predominates, the theory appears to be in qualitative agreement with the experimental data. In the present potassium-sodium system it might be expected that theory would break down because of the existence of more than one diffusion mechanism in sodium. The value of b calculated from the potassium-sodium phase diagram[14] is indeed 3 to 4 times greater than that determined from the enhancement data. To explain the enhancement data in the above manner would require a considerably smaller slope for the solidus line. It has already been noted that considerable experimental difficulty was encountered when trying to make alloys with greater than 1 at.% potassium. These difficulties imply a greater, rather than smaller, slope for the solidus line than that reported in Ref. 14.

The excellent fit of experimental data on fcc metals to the theories of Lidiard[3] and Howard and Manning[4] presents convincing evidence for the linear enhancement at low impurity concentrations. The simple addition of two processes exhibiting linear enhancement effects would not produce curvature in the enhancement plot. The apparent curvatures that can be seen in Fig. 2 could be explained in terms of the interaction of the different processes responsible for diffusion in sodium. The experimental errors make it difficult to be certain that the enhancement plot truly shows curvature and so the following analysis of the data will be based on the assumption that the plot is linear.

The data of Mundy[1] indicate that there could be three diffusion processes operative in sodium. However, at 50 °C the highest-temperature process would be responsible for only 15% of the diffusion and at 0 °C less than 5%. As these small proportions should not greatly affect the interpretation of the present data, their analysis has been in terms of two diffusion processes. The proportion of diffusion by the high-temperature process D_1^0 and the low-temperature process D_2^0 was determined by the equation

$$D = 0.72 \ e^{-11\,500/kT} + 0.0057 \ e^{-8530/kT} \ .$$

At 50 °C, $D_1^0/D(0) = 0.55$ and at 0 °C, $D_1^0/D(0) = 0.35$.

The two sets of mechanisms most fully discussed by Mundy[1] were that of the vacancy-interstitial and

the divacancy-vacancy mechanisms. If a linear enhancement effect is assumed for each diffusion mechanism and the assumption made that there is no interaction between the different mechanisms, then the measured diffusion coefficient is given by

$$D(c) = D_1^0 (1 + b_1 c) + D_2^0 (1 + b_2 c),$$

so that the measured slope is

$$\frac{1}{c}\left(\frac{D(c)}{D(0)} - 1\right) = \frac{D_1^0 b_1 + D_2^0 b_2}{D(0)} . \tag{5}$$

A. Vacancy-Interstitial Mechanisms

The enhancement effect of solute additions to diffusion is found to be very small in those systems (e.g., C in Fe; Cu, Ag, and Au in Pb) where an interstitial mechanism could be responsible for the diffusion.[11] This strongly suggests that b for diffusion by an interstitial mechanism is very small. If interstitials are responsible for diffusion at low temperatures in sodium and $b_2 \sim 0$, then from Eq. (5) it is found that

$$b_1 = 74.5 \text{ at } 50\,°C$$

and

$$b_1 = 180.0 \text{ at } 0\,°C,$$

where b_1 refers to diffusion by single vacancies. LeClaire's theory[12] for solvent self-diffusion in dilute bcc solid solutions gives the impurity diffusion-correlation factor as

$$f_i = 1 - \frac{D_i}{D(0)} \frac{6 f_0}{b + 15 + (1.53 b + 91.8)/(b + 9.06)} , \tag{6}$$

where D_i is the impurity diffusion coefficient. Equation (6) makes two assumptions. The first is that the solute additions do not appreciably affect the value of the solvent-correlation factor from that in the pure solvent. The only tests of this assumption showed it to be satisfactory in the Zn-Ag system[5] but less so in the Zn-Cu system.[6] The second assumption is that all vacancy jumps from unperturbed sites towards the fourteen sites neighboring the solute occur with the pure-solvent jump frequency. While these assumptions have not been tested for bcc systems, the following analysis of the data would not be possible if these simplifications were not introduced. The work of Barr et al.[13] gives the ratio of the diffusion coefficients as

$$D_i/D(0) = 7.3 \text{ at } 50\,°C$$

and

$$D_i/D(0) = 10.7 \text{ at } 0\,°C .$$

From these data the value of f_i^{IV} (f_i for single vacancies) calculated from Eq. (6) is 0.65 at 50 °C and 0.76 at 0 °C. There are no experimental values to compare with these numbers. Measurements

of the isotope effect for sodium diffusing in potassium showed a value of $f \Delta K = 0.09 \pm 0.05$. Even for $\Delta K \sim 0.5$, the measured value of f_i would be considerably smaller than that found from the enhancement data on the basis of the above vacancy-interstitial analysis. While the isotope effect might not be expected to be comparable for potassium diffusing in sodium, the above comparison may possibly be justified inasmuch as the ratios of $D_i/D(0)$ are very similar in both cases.

There is the possibility that interstitials could diffuse by an interstitialcy mechanism. This has the advantage of explaining the low-temperature self-diffusion isotope effects without having to postulate values of ΔK much smaller than 1. A value of b_2 for an interstitialcy process is not known, but it would appear from the work of Miller[22] that a maximum value of b_2 would be given by

$$b_2 = D_i/D(0) .$$

Using these values of b_2 at the appropriate temperatures, it is possible to determine that at 50 °C, $b_1 = 69$ and at 0 °C, $b_1 = 160$. Again using the theory by LeClaire,[12] one finds $f_i = 0.62(50\,°C)$ and $f_i = 0.74(0\,°C)$. These values are again rather high in comparison with those expected experimentally.

Theoretical values of b have been determined by LeClaire[8,9] in the case of fcc metals. The values obtained compared reasonably well for solutes with small differences in electronegativity. LeClaire writes the difference in the activation energy of impurity and self-diffusion as

$$\Delta Q = \Delta H_2 + \Delta E - C. \tag{7}$$

In this equation ΔH_2 is the difference between the activation energy for the impurity- and solvent-atom jumps; ΔE is the binding energy of the impurity to the vacancy; C allows for the temperature dependence of the correlation factor f_i for impurity diffusion and is given by

$$C = R \frac{\partial \ln f_i}{\partial (1/T)} . \tag{8}$$

There has been some discussion[9,23,24] of the relative magnitudes of ΔE, ΔH_2, and C. Using a Thomas-Fermi approximation for the energy of interaction between the impurity and vacancy, LeClaire[9] found for the noble metals $\Delta H_2 \sim 6 \Delta E$ and C was of similar magnitude and sign to ΔH_2. Corless and March[23] found using a rather more realistic Hartree potential that $\Delta H_2 \sim \Delta E$. An analytical solution for this potential has been determined by Alfred.[25] This solution allowed the determination of ΔH_2 and ΔE for the case of potassium as an impurity in sodium, and it was found that $\Delta H_2 \sim \Delta E \sim -1.3$ kcal/mole. It was found experimentally that $\Delta Q = 1.6$ kcal/mole and so from Eq. (7) it can be seen that C could be expected to be ~ -1.0 kcal/mole. The temperature dependence

of f_i can be determined from the enhancement data on the basis of the vacancy-interstitial and vacancy-interstitialcy mechanisms. The assumption is made that C is constant over the small temperature range considered in this work. The value of C obtained in this manner is half the magnitude and of opposite sign to that assumed on the basis of the Hartree potential.

The above analysis cannot, however, be regarded as conclusive because of the unsatisfactory nature of theories of impurity diffusion when applied to alkali metals. It may be said only that the theoretical and experimental evidence appear to show little support for either the vacancy-interstitial or vacancy-interstitialcy mechanism.

B. Divacancy-Vacancy Mechanisms

The analysis of the data in terms of divacancy-vacancy mechanisms by means of Eq. (5) results in the following two equations. At 50 °C

$$0.55\, b_2 + 0.45\, b_1 = 41 \tag{9}$$

and at 0 °C

$$0.35\, b_2 + 0.65\, b_1 = 63, \tag{10}$$

where b_1 and b_2 are the enhancement factors for diffusion by the single-vacancy and divacancy mechanisms, respectively. The theory of solvent enhancement in bcc metals of LeClaire[12] allows the minimum value of enhancement by single vacancies to be determined from the inequality

$$b_1 + \frac{1.53\, b_1 + 9.18}{b_1 + 9.06} > -15 + 6f_0\, \frac{D_i}{D(0)}\, .$$

The values of $(b_1)_{\min}$ determined in this way are

15.5 at 50 °C and 30.75 at 0 °C. If one assumes these minimum values of b_1 and therefore values of $f_i^{IV} = 0$ at both temperatures, then from Eqs. (9) and (10) one finds the enhancement factor for divacancies is approximately four times that for single vacancies.

If one assumes as in the previous section that $C = -1.0$ kcal/mole, then on the basis of the above interpretation one finds that $b_2 \sim 4b_1$ for $f_i^{IV} \sim 0$ and $b_2 \sim b_1$ when $f_i^{IV} \sim 0.35$. Above this value of f_i^{IV} the enhancement by means of divacancies would be less than that by single vacancies. Unfortunately it is difficult to predict values of b by means of divacancies, and it is not at all clear whether b for divacancies could be expected to be lesser or greater than b for single vacancies. It is clear, however, that the experimental data could be readily interpreted in terms of the divacancy-vacancy mechanism.

III. CONCLUSIONS

On the basis of the above analysis of the enhancement data it is not possible to determine unambiguously the mechanism responsible for diffusion in sodium. However, it would appear that a divacancy-vacancy explanation is preferable to explanations involving interstitials at low temperatures.

†Work performed under the auspices of the U. S. Atomic Energy Commission.

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