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Diffusivity of carbon in Fe–V alloys

S. N. TEWARI
Defence Metallurgical Research Laboratory, P.O. Kanchanbagh, Hyderabad 500258, India

J. R. COST
Los Alamos Scientific Laboratory, MS-734, CMB-8, Los Alamos, NM 87545, USA

Tracer diffusion of carbon in iron–vanadium alloys containing 2.45, 0.5 and 0.1 at% vanadium has been measured in a temperature range from 900 to 1325 K. No effect of magnetic transforms is observed on the C^{14} diffusivity in the α-stabilized Fe–2.4 at% V alloy, as has been reported for α-iron. Trapping behaviour of vanadium in slowing down the carbon diffusivity in iron shows a reasonable fit with the model predicted by Koiwa. However, the effect of these traps appears not to be felt during low-temperature internal friction measurements in Fe–2.4 at% V alloy.

1. Introduction

Measurments of the self-diffusion coefficient, D, [1] of bcc iron in the vicinity of the ferromagnetic to paramagnetic transformation region have shown that the temperature dependence of self-diffusion coefficient may not be properly described by the Arrhenius equation. Stanley and Wert [2] showed that the diffusion in the well-ordered ferromagnetic state is about 100 times slower than what would be expected from extrapolation of the data in the paramagnetic region. They used Fe–18 at% V alloy and, combining radioactivity tracer and anelastic methods, calculated diffusivities over a considerable interval on both sides of the magnetic Curie temperature, T_c. They attributed part of the retardation to an increase in the activation energy, Q, and part to a decrease in D_0, the pre-exponential factor. The increase in the activation energy in the ferromagnetic region [about 3.5 kcal mol^{-1} (14.6 kJ mol^{-1})] would have been either due to an increase in the formation energy of vacancies or to an increase in the motional energy. The part of the change caused by a change in D_0 was thought to be probably due to an entropy effect ($\Delta S/R \approx 2.3$, where ΔS is the change in entropy and R is the gas constant) caused by the ordering of spin. Borg and Lai [3] observed a similar behaviour in an α-stabilized Fe–1.8 at% V alloy. Recently, Hettich *et al.* [4] measured the self diffusivity in α-iron by an accurate microsectioning and counting technique over an extended temperature interval on both sides of the Curie temperature, T_c, and confirmed the existence of this anomaly. They have analysed their data in terms of temperature dependence of the activation entropy and enthalpy (both for the motion and formation) for the self diffusion in the ferromagnetic regime.

Homan [5] investigated the diffusion of carbon in iron using a tracer technique in the temperature range of 889 to 1137 K and reported a deviation from the usual straight line. He observed this deviation occuring at about 1083 K ($T_c = 1043$ K) and postulated an empirical model which included the possibility of a carbon vacancy interaction. This model however concludes that a carbon vacancy pair diffuses faster than either component alone and requires an iron-vacancy to have a migration energy considerably below 10 kcal mol^{-1} (41.8 kJ mol^{-1}) [6], which is quite doubtful. Any interaction with a vacancy would be expected to trap the carbon and slow it down. Borg [6] and Wuttig [7] have attempted to explain this anomaly as a magnetic effect. Recently, da Silva and McLellan [8] have compared the three proposed models, Interstitial-Vacancy Complex [5], Dual Site Occupance [9] and Ferromagnetic Effect [7], and concluded that the Ferromagnetic Effect Model is the more probable one. However, the major objection to this model is the lack of the

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very high-temperature data (due to the bcc to fcc transformation of iron) necessary to establish a complete analogy with the magnetic effect observed for the diffusion of substitutional species. The first purpose of the present investigation was to examine the diffusion behaviour of carbon in α-stabilized iron–vanadium alloy. Diffusivity could thus be measured over a considerable temperature interval on both sides of T_c to assist in the understanding of the mechanism of the abrupt changes in the carbon diffusion and self-diffusion coefficients in α-iron. The residual tracer diffusion technique employing carbon-14 as the diffusing species was selected because it was a relatively fast and simple technique. An Fe–2.4 at% V alloy was selected for this investigation for two reasons. First, it maintains bcc structure up to its melting point [10] and, second, it has been reported that Fe–1.8 at% V alloy has the same D_0 and Q values for the self diffusion of Fe^{59} as pure iron [11].

The diffusivity of interstitial elements through metals, particularly bcc metals, has been known to show a deviation from the Arrhenius relation [12–14]. There is a general belief that the phenomenon is due to an attractive interaction between dissolved interstitials and trapping centres such as impurity atoms. Several investigators have attempted to explain this effect in terms of trapping mechanisms [12, 15–17]. In an Fe–0.9 at% Cr alloy the activation energy for the diffusivity of carbon has been reported to be 33 kcal mol^{-1} (138 kJ mol^{-1}) [18], an increase of 9 kcal mol^{-1} (37.7 kJ mol^{-1}) as compared to that in pure α-iron [5]. Since vanadium addition lowers the carbon activity in the iron alloy much more drastically than does the addition of chromium [19], an even stronger trapping of carbon atoms will be expected in the case of iron–vandium alloys.

The second purpose of this investigation was to examine the trapping behaviour of vanadium in α-iron. Tracer diffusion of carbon was measured in Fe–2.4 at% V, Fe–0.5 at% V, and Fe–0.1 at% V alloys. To provide information concerning the low-temperature diffusivity values of carbon in Fe–V alloy, internal friction studies were also made.

2. Experimental procedure

2.1. Tracer diffusion

Iron–vanadium alloy rod samples* of 6.3×10^{-3} m diameter were swaged down to 4.5×10^{-3} m diameter and several samples of about 2.5×10^{-2} m in length were cut from these. After surface cleaning, these samples were vacuum annealed in quartz capsules at 1073 K for 6 h to remove the cold work. Half of these samples were then placed in an evacuated quartz carburization capsule in which radio-active methane (5.0 millicurie methane capsule from the Radioactive Centre, UK) was transferred by using liquid helium. The capsule was then placed in a furnace at 1123 K for 1.5 h for carburization. The individual carburized cylinders were then vacuum encapsulated in small quartz capsules and homogenized for 72 h at 1123 K. Temperature control of ± 1 K was maintined over the sample length in a constant temperature zone of about 0.127 m in length. The carburized cylinders obtained from this homogenization treatment were bright and shiny in appearance. These were sectioned at both ends by the help of Rockwell Delta TG-3 Tool-maker surface grinder, to an accuracy of 1×10^{-5} m, and radioactivity counted to uniform count to check the homogeneity of C^{14} concentration. Samples were held in a V-block while sectioning and counting. A halogen-quenched Geiger–Muller tube (Number 200 C.B.) of 14 g m^{-2} mica window thickness, was used for counting. The counting assembly was designed such that the sample was always held vertically and sample surface to counting port distance was always kept the same. The counting port was concentric with the sample and only the central 2.5×10^{-3} m diameter region of the sample surface was counted. The entire assembly was kept in a lead-brick structure to decrease the background radiation.

The carburized samples were then electrically butt-welded to the uncarburized iron–vanadium alloy samples with the same composition in a flowing argon atmosphere. The welding current and the contact pressure were so adjusted that this welding technique gave almost perfect welds, as observed by microstructural examination and concentration profile determination, see Fig. 1. In Fig. 1, for plotting the C^{14} profile after welding, the background count has been subtracted from the counts.

To avoid decarburization during the diffusion anneal the welded samples were then carefully copper plated in several steps of precleaning, intermediate cleaning, electrolytic cleaning, precoating

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*Alloy samples were prepared by Materials Research Corporation, USA, from Marz-grade iron and vanadium by arc-melting in an argon atmosphere.

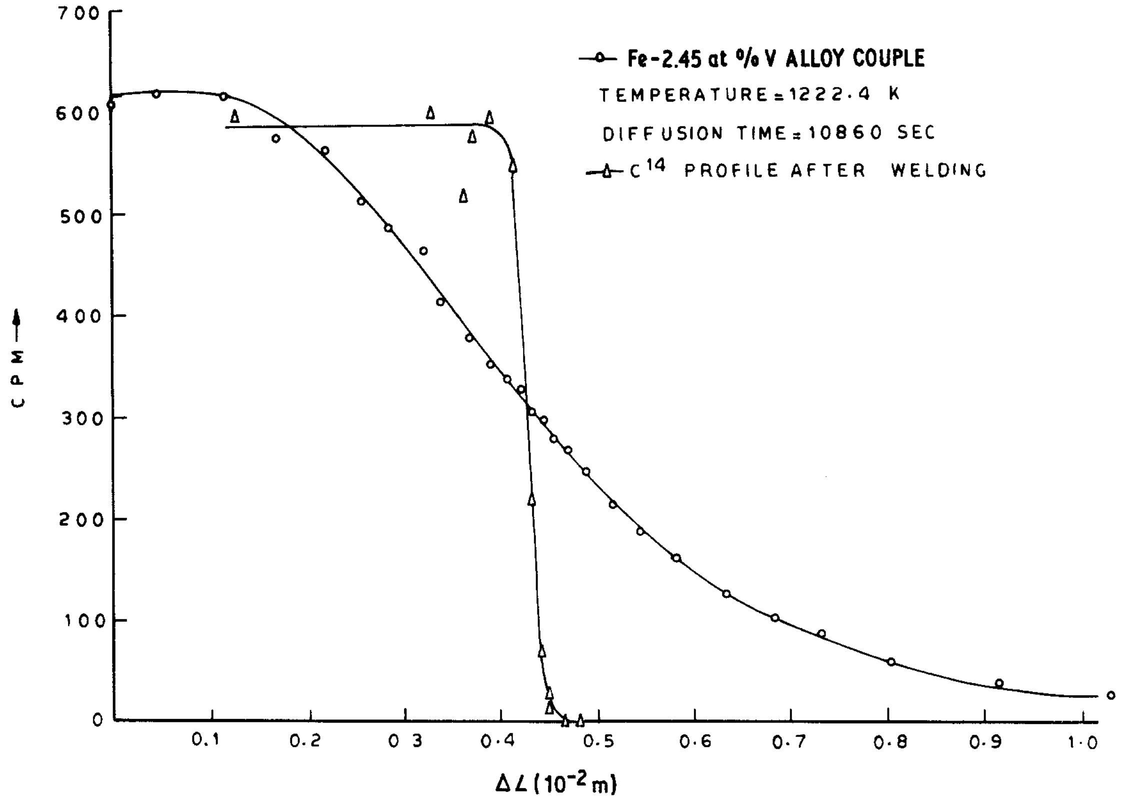

Figure 1 C^{14} concentration profile after welding and after a diffusion anneal in an Fe–2.45 at% V alloy couple. ΔL denotes the distance from an arbitrary plane in the C^{14}-rich side of the couple and CPM denotes the C^{14} activity in counts min^{-1}

and copper plating [20]. An approximately 1.5×10^{-4} m thick copper coating was selected for these diffusion couples because the solubility of carbon in copper is low, diffusivity of copper in α-iron is low as compared to that of carbon and copper is an extremely good getter of oxygen. These diffusion couple specimens were then flamed in a dynamic vacuum for 3 to 4 min to drive off any hydrogen that might have been picked up during the copper plating, and vacuum encapsulated individually in quartz capsules for the subsequent diffusion anneal. The vacuum encapsulated specimens were then annealed in a furnace in which an approximately 0.1 m long zone was kept within ± 0.5 K of the set temperature. After the diffusion anneal the samples were sectioned, the decrease in length was measured by a dial gauge (of accuracy 2×10^{-5} m) and time for 3000 to 4000 counts measured to obtain the C^{14} concentration profile, Fig. 1. Background was also counted for the same number of counts for each diffusion run. However, in Fig. 1, for plotting the count profile for the Fe–2.4 at% V alloy couple the background-count correction has not been made.

2.2. Internal friction

Internal friction measurements have been conducted only on the Fe–2.4 at% V alloy samples. A torsional pendulum [21] was used to measure the amplitude and the decay rate by recording the output of an optical lever. The frequency range used was 1 to 43 Hz. The 0.114 m long specimens were 1.3×10^{-3} m in diameter for the frequencies of 9.9 and 41 Hz and 9×10^{-4} m in diameter for the frequency of 1.45 Hz. The alloy samples were cold-swaged, annealed at 1123 K for 4 h and water-quenched. One 1.3×10^{-3} m diameter sample was also prepared from one of the samples carburized with carbon-14. The furnace in which the specimen was suspended was heated electrically by direct current. The temperature of the sample was measured by two pairs of 1.2×10^{-4} m diameter Cr–Al helically wound wires spot-welded to the specimen, one near the top and the other near the bottom of the specimen. The solenoid coil, to magnetically saturate the sample and to minimize magnetoelastic damping effects, produced a magnetic field of 1 to 1.1×10^{-3} T around the sample.

3. Results

3.1. Tracer diffusion results

The microstructural examination through the welded region of a representative weld performed using conditions indentical to the other diffusion couple weldings used in this investigation showed almost 100% contact across the interface between the two specimens. Some grain coarsening in the immediate viscinity of the weld was also noticed. The concentration profile determination in one of the representative welds just after welding shows negligible carbon-14 penetration due to welding heat generated as compared to that due to a typical duffusion anneal (Fig. 1). Since concentration of carbon-14 is proportional to its activity, the following solution of Ficks second law of diffusion for the case of semi-infinite diffusion couple [22] was used:

$$2\{(C_x - C_b)/(C_o - C_b)\} - 1 = \mathrm{erf}\,[x/2(Dt)^{1/2}], \tag{1}$$

where C_o and C_b are the activities in the high and low side of the diffusion couple before the diffusion anneal, C_x is the instantaneous concentration at a distance, x, from the interface at a time, t. Therefore, from the plot of $\mathrm{erf}^{-1}[(C_x - C_b)/(C_o - C_b)]$ against x or ΔL it is possible to determine the value of the diffusion coefficient, $D(\mathrm{C}^{14})$. One such plot corresponding to the concentration profile in Fig. 1 is shown in Fig. 2. A deviation in the data, $(C_x - C_b)/(C_o - C_b)$, from the straight line can be noticed at the lower and upper end of ΔL values in Fig. 2. The scatter at the higher end of ΔL is due to the more apparent scatter in $(C_x - C_b)$ as C_x approaches C_b in this regime (scatter in C_x and C_b are additive). The apparent scatter at the lower end of ΔL is due to the nature of the $\mathrm{erf}^{-1}\, z$ plot. Small changes in z lead to relatively large changes in the $\mathrm{erf}^{-1}\, z$ at the higher

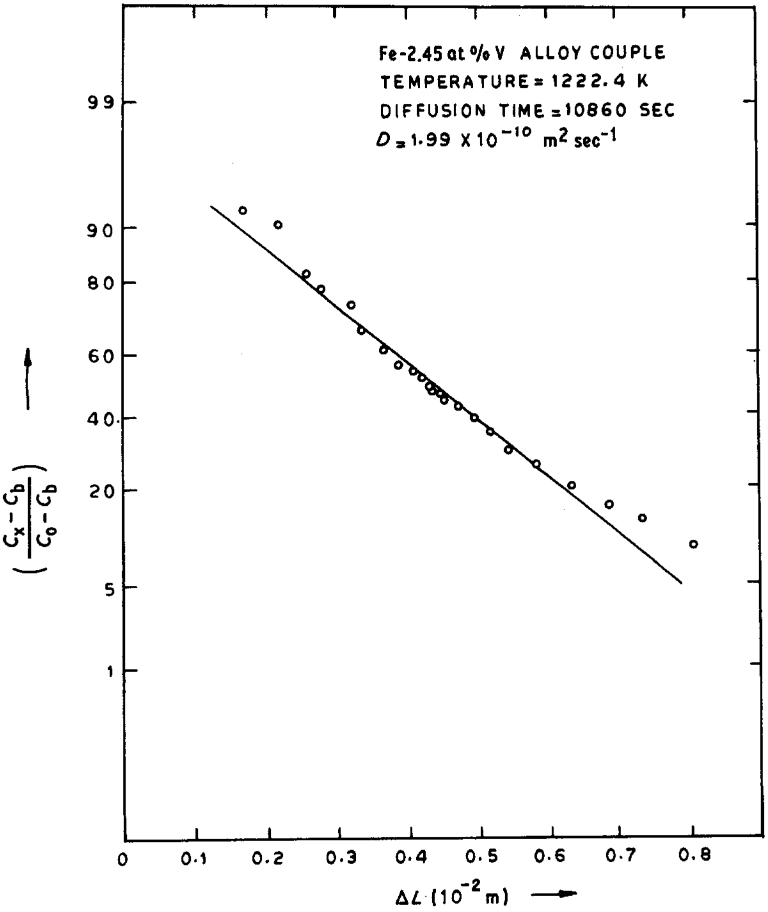

Figure 2 Probability plot of $[(C_x - C_b)/(C_o - C_b)]$ plotted against distance, ΔL, to calculate the carbon diffusivity for the typical diffusion anneal shown in Fig. 1.

TABLE I C^{14} diffusivity in iron–vanadium alloys

Experiment number	Couple type	Anneal temperature (K)	Diffusion coefficient, D (m^2 sec^{-1})	Standard deviation in D, ΔD (m^2 sec^{-1})	Estimated D (m^2 sec^{-1})
1	Fe–2.45 at% V	1141.8	6.98×10^{-11}	6.4×10^{-12}	–
2	Fe–2.45 at% V	998.6	5.23×10^{-12}	7.7×10^{-13}	–
3	Fe–2.45 at% V	1222.4	1.99×10^{-10}	2.9×10^{-11}	–
4	Fe–2.45 at% V	900.1	6.27×10^{-13}	8.8×10^{-14}	–
5	Fe–2.45 at% V	1325.3	8.76×10^{-10}	3.1×10^{-11}	–
6	Fe–Fe	1010.4	1.49×10^{-10}	3.9×10^{-12}	–
7	Fe–0.5 at% V	1136.5	2.07×10^{-10}	1.2×10^{-11}	1.99×10^{-10}
8	Fe–0.5 at% V	990.0	1.27×10^{-11}	1.6×10^{-12}	1.97×10^{-11}
9	Fe–0.1 at% V	1136.5	5.14×10^{-10}	2.4×10^{-11}	3.44×10^{-10}
10	Fe–0.1 at% V	990.0	5.46×10^{-11}	2.9×10^{-12}	5.08×10^{-11}

values of $\mathrm{erf}^{-1} z$. Ideally, therefore, the data at the lower and the higher ends of the ΔL values should therefore be relatively neglected to obtain the straight-line fit in Fig. 2. However, it is not possible to arbitrarily decide on a definite cut-off on the $\mathrm{erf}^{-1} [(C_x - C_b)/(C_o - C_b)]$ plot. Therefore, all the data measured have been given equal weighting in determining the least-squares fit, as shown in Fig. 2.

A computer program was developed and used to obtain a straight-line fit to the $\mathrm{erf}^{-1} [(C_x - C_b)/(C_o - C_b)]$ against ΔL plot and calculate D values. The slope was obtained by a least-squares fit to the above straight line. Standard deviations, ΔD, were programmed to be calculated from the standard deviation in the slope of these lines. A summary of the tracer diffusion experimental data is given in Table I. For calculating D, the observed heat-up time of 2 to 3 min to reach the set temperature in the diffusion anneal for these experiments was neglected, when compared to the total diffusion anneal time of 92 min to 24 h. An experiment conducted on a pure iron specimen to check the accuracy of the measurements gave the D value of 1.49×10^{-10} m^2 s^{-1} at 1010 K (see Table I) as compared to the literature value of 1.2×10^{-10} m^2 s^{-1} [2, 5]. Table I lists the D values obtained for the various Fe–V alloy specimens together with the standard deviation in D observed, ΔD and the anneal temperature. The activation energy, Q, was obtained from the straight line (least-squares fit) obtained from the $\log D$ against T^{-1} (K^{-1}) plot (see Fig. 3). The carbon diffusivity $D(C^{14})$, in Fe–2.45 at% V follows the following tamperature dependence in the temperature range of 900 to 1325 K:

$$D(C^{14}) = 54.7 \times 10^{-4} \exp \frac{(-40\,700 \pm 1\,300)}{1.98\,T} \; m^2\ s^{-1} \quad (2)$$

3.2. Internal friction

Internal friction, Q^{-1}, was calculated using the following formula [23]:

$$Q^{-1} = (1/\pi n) \ln (A_1/A_n), \quad (3)$$

where n is the number of cycles needed for the amplitude to decay from A_1 to A_n. Graphical analysis of Q^{-1} against T plots (see Fig. 4) was used to obtain T_{max} (temperature corresponding to the maximum Q^{-1}) for a particular frequency. In this plot it may be noted that the peaks appear to be composed of two separate peaks. Uncertanity in the background radiation plus the relative uncertainty in Q^{-1} (because it was at low level) made an analytical separation of peaks impossible. The theoretical peak at half-peak height, ΔT^{-1}, was calculated using [24]:

$$\Delta T^{-1} = 2.635\,(R/Q_m)\gamma_2(\beta), \quad (4)$$

where $R = 1.98$ cal mol^{-1}, (8.28 J mol^{-1}), $Q_m =$ is the activation energy for motion, 20 kcal mol^{-1} (8.37 kJ mol^{-1}) and $\gamma_2(\beta) = 1$ (for single relaxation). Graphical analysis was utilized to separate the high-temperature peak with the theoretical width, and the results are shown by the solid line (Fig. 4). A frequency-dependent background damping was observed. The reason for the absence of the peak broadening in the measurements made at 1.45 Hz is not clear.

Diffusion coefficients measured from internal

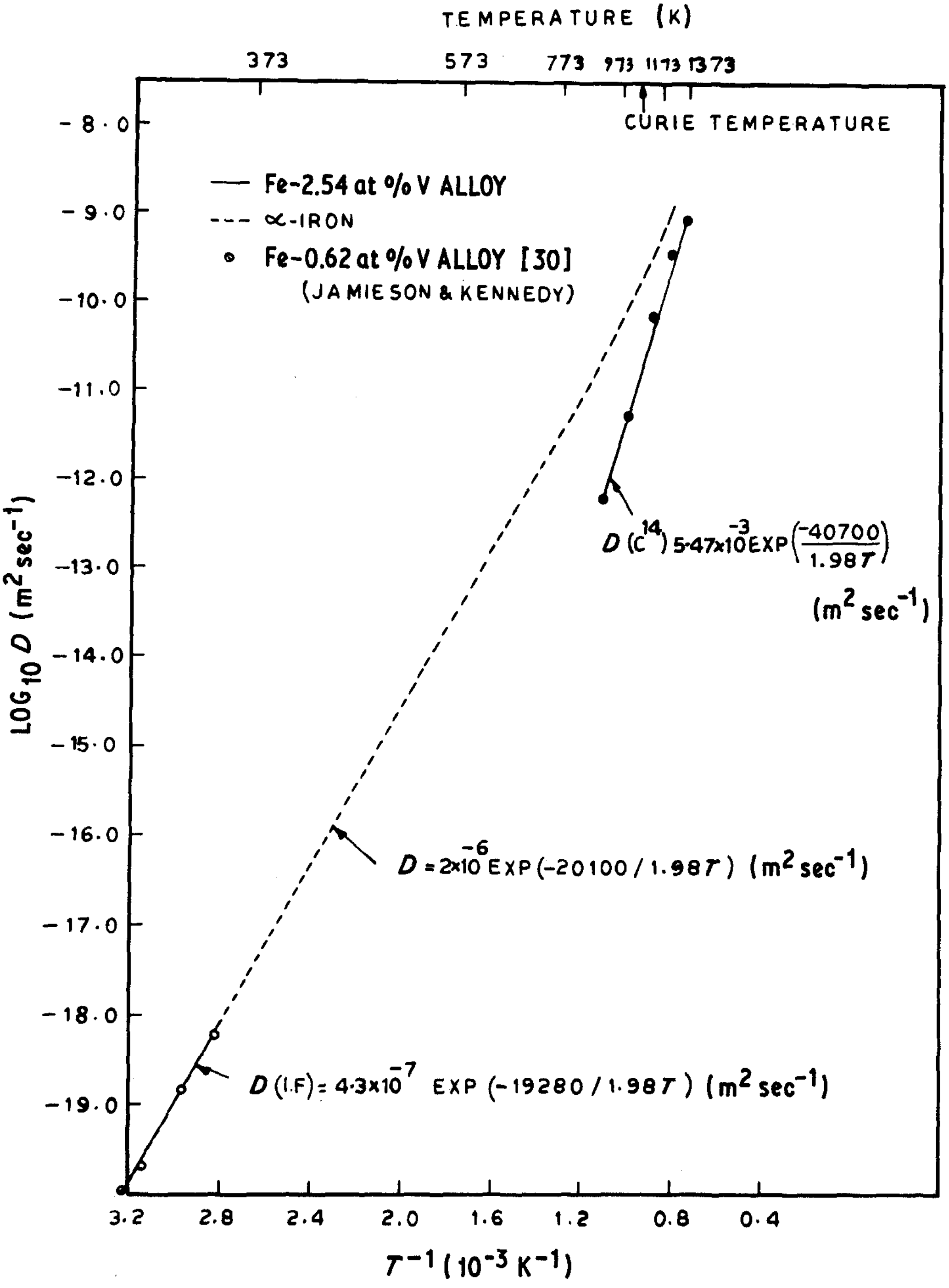

Figure 3 Temperature dependence of the carbon diffusivity in Fe–2.45 at% V alloy as measured by tracer diffusion [D(C^{14})] and internal friction [D(IF)] techniques.

friction measurements, D(IF) corresponding to different frequencies, f, were calculated using [22]

$$D = a_0^2/36\tau, \qquad (5)$$

where a_0 is the lattice parameter of α-iron (2.86 × 10^{-10} m) and $\tau = 1/2\pi f$. The carbon diffusivity (m^2 sec^{-1}) follows the following temperature dependence in the temperature range of 318 to 357 K for Fe–2.45 at% V alloy (Fig. 3):

$$D(\mathrm{IF}) = 4.3 \times 10^{-7} \exp - [(19.28 \pm 0.4) \times 10^3/1.98\,T)].$$

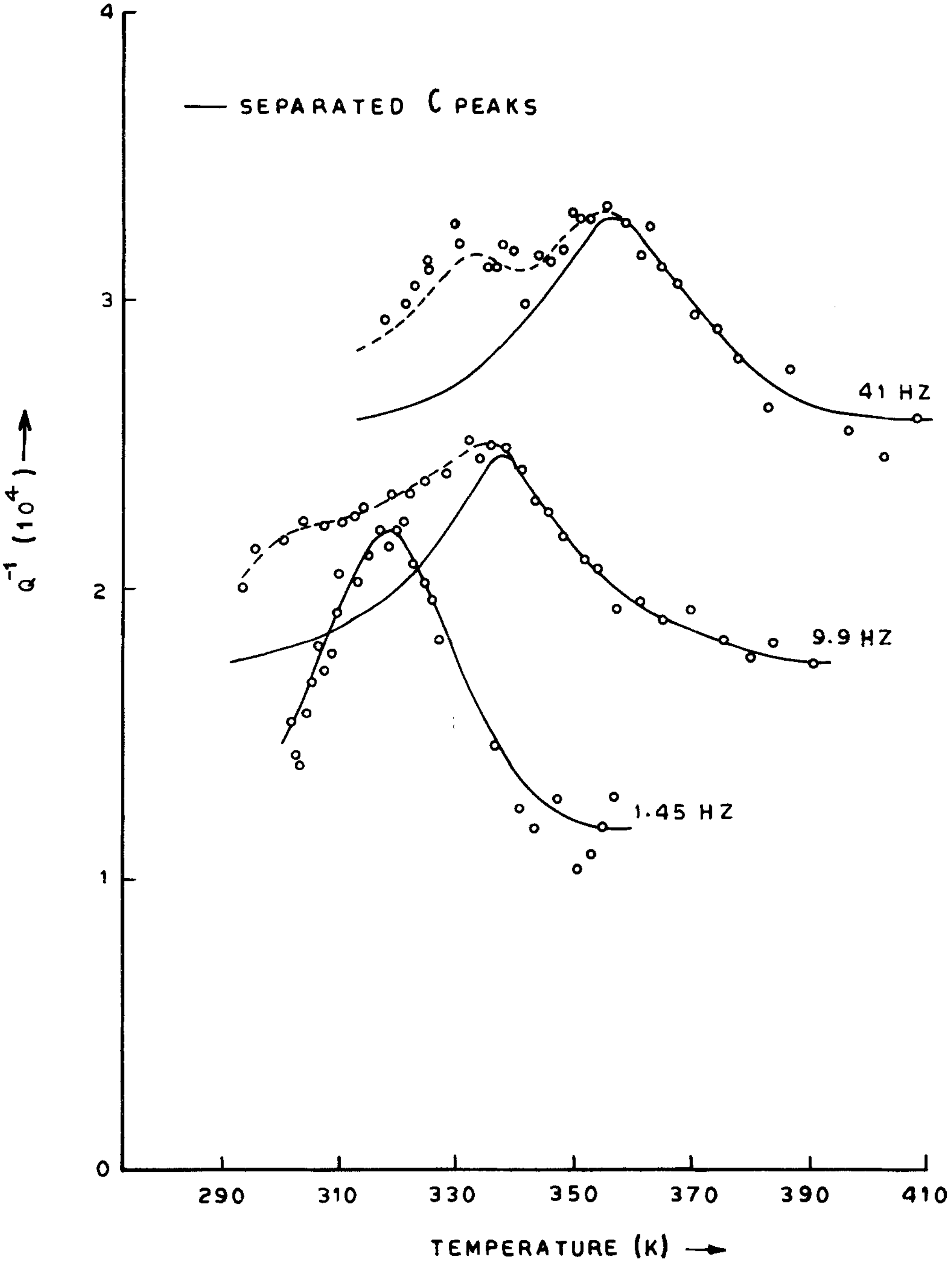

Figure 4 Internal friction peaks due to carbon diffusivity in Fe–2.45 at% V alloy.

(The uncertainty of ± 0.4 kcal mol^{-1} (1.67 kJ mol^{-1}) shown above does not include the contribution from ± 2 K uncertainty in the peak temperature due to graphical analysis.)

4. Discussion

The log $D(C^{14})$ against T^{-1} plot (Fig. 3) for the tracer diffusion experiments in the α-stabilized Fe–2.45 at% V alloy gives a straight line in the temperature range of 900 to 1325 K. No effect due to the magnetic transformation (Curie temperature 1053 K) is observed on the interstitial diffusion of carbon in this alloy. This leads to the belief that the anomaly observed in the Arrhenius plot for carbon diffusivity in α-iron [5] is not due to the magnetic effects.

However, the diffusivity of C^{14} in Fe–2.45 at% V alloy is significantly less than that in α-iron. One can compare the diffusivity value reported in iron in this temperature range, $D = 2.36 \times 10^{-5} \exp - (24600/1.98\,T)$ m^2 sec^{-1} [5], with that observed in Fe–2.45 at% V alloy, $D(C^{14}) = 5.47 \times 10^{-3} \exp - (40700/1.98\,T)$ m^2 sec^{-1}, Fig. 3. It suggests that the vanadium atoms in the iron lattice are acting like very strong traps for carbon. This strong affinity of carbon to the vanadium atoms, as compared to iron, is to be expected, as the heat of formation [25] of VC in this temperature range is about -28 kcal mol^{-1} (117.5 kJ mol^{-1}) and that of Fe_3C is about $+6$ kcal mol^{-1} (25.1 kJ mol^{-1}).

Kiowa [16] has proposed a model to evaluate the trapping effect in diffusion of interstitial impurity in b c c lattices, and predicted the following relationship,

$$D_{\mathrm{app}} = D_{\mathrm{norm}}/[1 - 2C + 2C(\gamma_0/\gamma_1) \exp(E_B/kT)], \qquad (6)$$

where D_{app} is the apparent diffusion constant in the presence of traps, D_{norm} is the normal diffusivity in the absence of traps, C is the atomic fraction of trapping centres (substitutional atoms), E_B is the binding energy, γ_0 and γ_1 are the frequency factors corresponding to the untrapped and the trapped sites; k and T have their usual significance. The above relationship is valid for the octahedral occupancy of carbon atoms, which is generally believed to be the case in the Fe–C system.

Let us now examine the validity of the model of Koiwa for predicting the trapping behaviour of the vanadium atoms observed in this study. We can substitute into Equation 6 the values of D_{app}, D_{norm} and C of $5.47 \times 10^{-3}(-40700/1.98\,T)$ m^2 sec^{-1}, $2.36 \times 10^{-5} \exp(-24600/1.98\,T)$m^2 sec^{-1}, and 0.0245, respectively, observed from our data. The values of E_B and γ_0/γ_1 can now be calculated to be 16.1 kcal mol^{-1} (67.4 kJ mol^{-1}) and 0.088, respectively. Thus, the vanadium traps provide a binding energy of 16.1 kcal mol^{-1} (67.4 kJ mol^{-1}) as compared to iron lattice for carbon atoms. The frequency factor, γ, has contributions from the Debye frequency and the entropy factors, both of which are believed to be strongly affected by the nature of the potential well which the interstitial atom occupies [26–29]. The potential well in an iron lattice must apparently have been modified due to the presence of vanadium atoms (trap sites) to give a γ_0/γ_1 ratio of 0.088.

We can now estimate, from Equation 6, the apparent diffusivities of carbon for Fe–0.5 at% V alloy and Fe–0.1 at% V alloys based on the values of γ_0/γ_1 and E_b, calculated above for the Fe–2.45 at% V alloy. These apparent diffusivities, obtained from the analysis of our data based on the model of Koiwa are shown as dotted lines in Fig. 5 for Fe–0.5 at% V and Fe–0.1 at% V alloys. The apparent diffusivity values thus estimated at 1136.5 and 990 K are also given in Table I. The experimentally-observed carbon diffusivities for the above two alloys at 1136.5 and 990 K are shown as data points in Fig. 5, and are also listed in Table I. A reasonably good agreement can be seen between the observed diffusivity values and those estimated from the Koiwa's model for both these alloys. For the Fe–0.1 at% V alloy however, the D value observed at 1136.5 K (data point) shows the positive deviation from the estimated Arrhenius behaviour (dotted line, Fig. 5), similar to that observed for α-iron [5].

When we compare the activation energy, 19.28 kcal mol^{-1} (80.66 kJ mol^{-1}) and D_0, 4.3×10^{-7} m^2 sec^{-1} obtained from the internal friction measurements at low-temperature range (Fig. 4) in the Fe–2.45 at% V alloy with those reported for α-iron 20.1 kcal mol^{-1} (84.1 kJ mol^{-1}) and 2×10^{-6} m^2 sec^{-1} [20], we observe that the effect of trap-binding energy is not being felt during these measurements. Also, the Arrhenius plot through the diffusivity values measured by the internal friction technique on Fe–2.45 at% V alloy passes through a similar diffusivity value reported for Fe–0.62 wt% V alloy by Jamieson and Kennedy [30]. This is not surprising because, due to the inherent nature of the measurements that we make during internal friction studies, we can only get information about atomic jumps between two neighbouring sites. All this observation indicates is that the carbon atoms within the traps jump with approximately the same activation energy [19.3 kcal mol^{-1} (80.7 kJ mol^{-1})] as the ones in the parent lattice [20.1 kcal mol^{-1} (84.1 kJ mol^{-1})], but do not contribute to macroscopic diffusion unless they jump out of the traps. The internal friction experiment is apparently enabling us to observe the interstitial jump behaviour within only the trapped sites. These internal friction data, however, have considerable uncertainty due to large uncertainty in Q^{-1} caused by

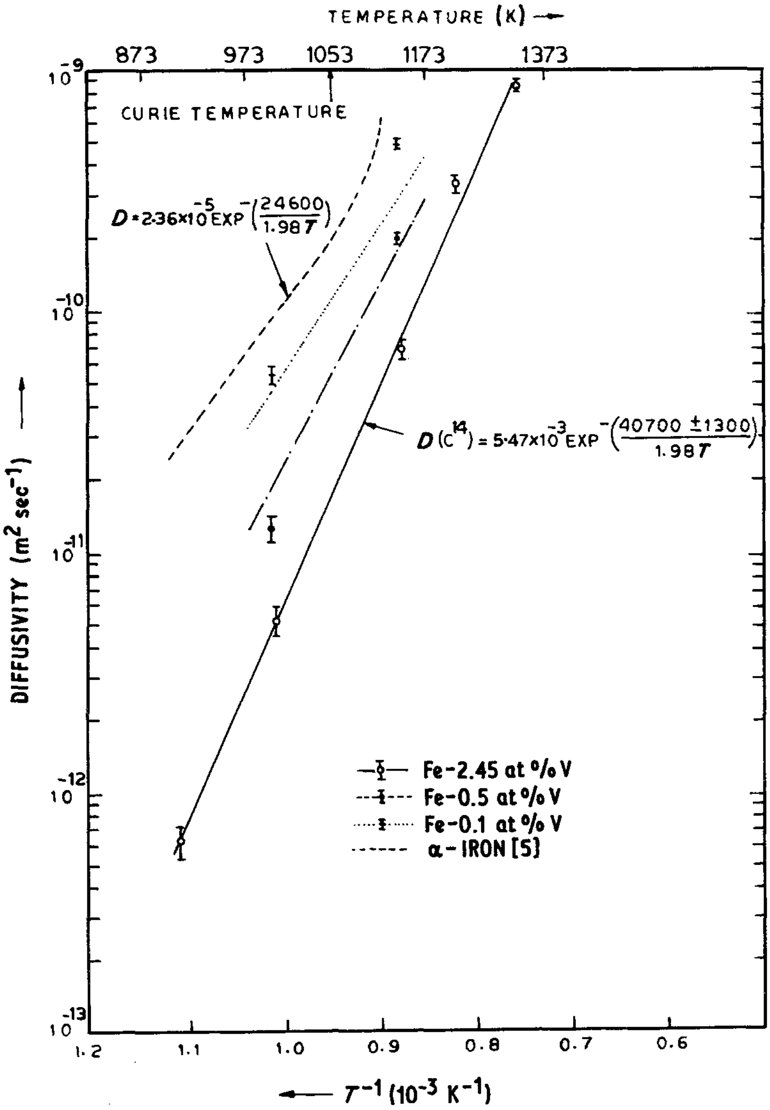

Figure 5 Temperature dependence of the carbon diffusivity in Fe–V alloys. The two dotted lines for Fe–0.5 at% V and Fe–0.1 at% V alloy show the values estimated by Koiwa's model based on $D(C^{14})$ and D. The data shown on these dotted lines are the actual measured values at 1136.5 and 990 K.

the very low carbon concentration in the alloy and the large uncertainty in the background radiation. Careful internal friction measurements on Fe–V alloys (with varying vanadium concentrations), having large interstitial carbon contents, may show the actual vibration frequency and activation energy behaviour of trapped and untrapped interstitial atoms.

5. Conclusions

The ferromagnetic to paramagnetic transformation does not affect the interstitial diffusivity of carbon atoms in α-stabilized Fe–2.45 at% V alloys. Vanadium atoms act as very strong traps in slowing down the carbon diffusivity in the iron–vanadium alloys. The trapping model proposed by Koiwa shows a reasonably good fit with the observed temperature and composition dependence of the tracer diffusivity of carbon in Fe–V alloys. Measurements, however, suggest that the effect of these traps is not being felt during the low-temperature internal friction experiments.

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