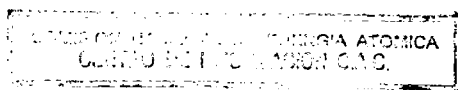


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Diffusion of Nb in Fe and Fe alloys

S. Kurokawa, J. E. Ruzzante, A. M. Hey, and F. Dyment

The diffusion coefficients of Nb have been studied in unalloyed γ Fe over the temperature range 948–1201°C and in Fe–1.5Mn, Fe–0.6Si, and Fe–1.5Mn–0.6Si alloys over the range 1081–1201°C by the direct sectioning method using the isotopes ^{94}Nb and ^{95}Nb . The Arrhenius curves for diffusion into pure γ Fe and γ Fe alloyed with Mn and/or Si are linear in the temperature range 1081–1201°C, and give approximately the same activation energy $Q = 267.4 \text{ kJ mol}^{-1}$. Below these temperatures $\ln D$ v. $1/T$, where D is the volume diffusion coefficient and T the temperature, is non-linear for diffusion into γ Fe. The values of Q and D_0 (the frequency factor) obtained in pure γ Fe are more representative than the values $Q = 344.5 \text{ kJ mol}^{-1}$ and $D_0 = 5.30 \times 10^{-2} \text{ m}^2 \text{ s}^{-1}$ found in the literature. It was possible to examine the influence of the substitutional alloying elements Mn and Si on the behaviour of Nb diffusion in γ Fe and the following effects were observed: the diffusivity of Nb decreases with Mn content but increases with Si content and when both Mn and Si are added the two effects appear to compensate each other. MS/0860

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Important changes in the structure and properties of a metal can be produced by fairly small additions of other elements. Alloys having appropriate properties can be designed by the careful selection of these additions. Solid state diffusion processes are, in many instances, responsible for the alterations in structure and properties. In the case of high strength low alloy (HSLA) steels, an improvement in mechanical properties can be achieved by grain refinement and precipitation hardening. These processes are controlled, in turn, by the suitable manipulation of the recrystallization and precipitation phenomena occurring in the course of industrial processing and, in particular, through the addition of elements such as Nb, V, and Ti. An accurate knowledge of the diffusion behaviour of these elements in the appropriate ranges of temperature and composition is required when these processes are to be analysed fundamentally.

As a preliminary to the present investigation the literature was searched for data concerning the hetero-diffusion of substitutional solutes in α and γ Fe. The data collected are presented in Fig. 1, from which it can be seen that little information is available regarding the elements of principal interest: Nb, V, and Ti. The behaviour of Nb in the γ phase has been investigated only over a narrow range of temperatures. These data will be discussed later. Two sets of results, which differ considerably, have been reported for the diffusion of V in γ

Fe. The data available concerning Ti (Ref. 40) refer only to the solubility and diffusion of Ti in Fe and give coefficients for chemical interdiffusion. There have been no previous studies using the radioactive tracer technique on which the present work is concentrated. By applying the Darken relationship, however, the intrinsic diffusion coefficients can be inferred from the above results. These values were not included in Fig. 1, where only the data obtained by radioactive tracer techniques are represented. For the systems with data obtained by both techniques, the coefficients of chemical interdiffusion and of heterodiffusion as measured with radiotracers differed by a factor of 3–10.

Because of the relative scarcity of reliable tracer data, it was decided to investigate the diffusion of Nb in unalloyed Fe, and in three particular Fe alloys of composition (wt-%): Fe–1.5Mn, Fe–0.6Si, and Fe–1.5Mn–0.6Si. These levels were selected as being typical of the concentrations found in commercial materials. The behaviour of V and Ti in a series of similar Fe alloys will be reported in a further publication.

EXPERIMENTAL METHOD

Materials

The alloys were prepared by Leico Industries Inc. using 99.99% pure starting material. The same firm supplied the 99.99% pure Fe. Rectangular ingots, each weighing approximately 250 g, were cold rolled down to 5 mm thick plates. Cylindrical specimens 10 mm dia. were sparkcut, mechanically polished down to 400 emery paper, and then electrolytically polished. The structure was homogenized with the aid of a long anneal before the diffusion treatment.

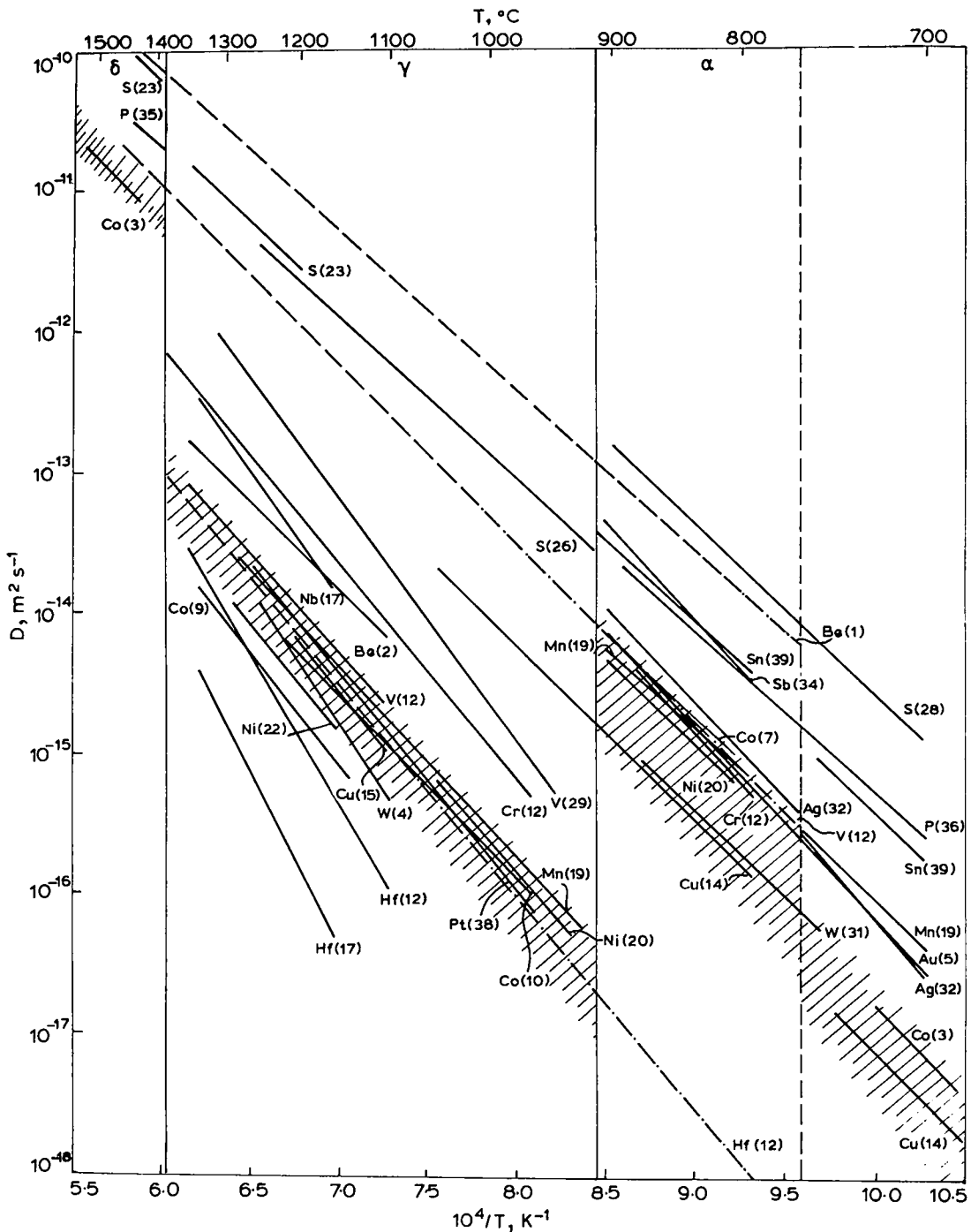
The isotope ^{95}Nb was obtained as a chloride solution in hydrochloric acid or as a complex (oxalate solution in oxalic acid); ^{94}Nb was also obtained as a chloride solution. The characteristics of the ^{95}Nb and ^{94}Nb isotopes used as tracers are described in Table 1.

Annealing

The homogenization heat treatments were performed at 1230°C for 10 days in Chevenard–Journier furnaces which enabled the temperature to be held constant over time within 1–3 K. The specimens were wrapped in Ta foil and sealed in silica tubes under a slight overpressure of 99.998% pure argon. Zr turnings were used as getters. During the diffusion anneals Fe foil was used because of a reaction between the Ta foil and the Nb deposit.

Tracer deposition

The application by electrolytic deposition of the tracer in a dimethylsulphoxide (DMSO) bath, a method currently being employed with success in work on other materials,⁴¹ proved to be difficult. The rate of deposition was poor and only low activity deposits were obtained. Alternative methods, such as the direct deposition of the solution on the specimen and subsequent drying, resulted in a lack of adherence and poor homogeneity of the layer. In addition, chemical attack on the surface occurred under these conditions. Finally, the efficiency of the DMSO electrolytic deposition method was improved by the prior dehydration



literature searched: Be (1, 2), Co (3–11), Cr (4, 12), Cu (13–16), Hf (12, 17), Mn (18, 19), Ni (4, 5, 11, 20–22), S (23–28), V (12, 29, 30), W (4, 31), Au (5), Ag (32, 33), Sb (34), P (35–37), Nb (17), Pt (38), Sn (39)
 --- Be diffusion in Fe–Be, V and Hf diffusion in Fe–V (these alloys retain α phase at high temperature) - - - - - extrapolated values from studied temperature range; shaded areas are Fe selfdiffusion bands

1 Heterodiffusion of substitutional solutes in α , γ , and δ Fe: Ref. nos. in parentheses

Table 1 Characteristics of radiotracers used

Radioisotope	^{95}Nb	^{94}Nb
Half life	35 days	2×10^4 years
Formation reaction	$^{94}\text{Zr}(n, \gamma)^{95}\text{Zr} \rightarrow ^{95}\text{Nb}$	$^{93}\text{Nb}(n, \gamma)^{94}\text{Nb}$
Disintegration reaction	$^{95}\text{Nb} \rightarrow ^{95}\text{Mo}$ 65 days \downarrow 35 days	$^{94}\text{Nb} \rightarrow ^{94}\text{Mo}$ \downarrow 2×10^4 years
Emitted radiation	β^- γ	β^- γ
Energy, MeV	0.160 0.765	0.500 0.700 0.871

of the tracer solution. The homogeneity of the deposited layer was assessed by autoradiography. When difficulties arose in the supply of ⁹⁵Nb by the Amersham Corp., ⁹⁴Nb supplied by the New England Nuclear Corp. was used. Long counting times were therefore required due to the lower activity of the ⁹⁴Nb tracer, coupled with the low efficiency of deposition. The use of ⁹⁴Nb, however, avoids a problem associated with ⁹⁵Nb and with the presence of its radioactive mother, ⁹⁵Zr. The shorter half life of ⁹⁵Nb leads to a gradual increase in the relative concentration of ⁹⁵Zr which, unfortunately, is not resolved when a Na(Tl)I detector is used. Thus, a GeLi detector is required to separate the contribution of ⁹⁵Zr from that of ⁹⁵Nb. Concerning diffusivity, a slight but definite difference, such as that observed⁴² in a matrix of α Zr, is to be expected between ⁹⁵Zr and ⁹⁵Nb in an Fe matrix.

Penetration profiles

The solution of the Fick equation for a semi-infinite medium with a thin layer of radioisotope located on the planar surface is

$$c(x, t) = \frac{K}{\sqrt{\pi Dt}} \exp(-x^2/4Dt) \dots \dots \dots (1)$$

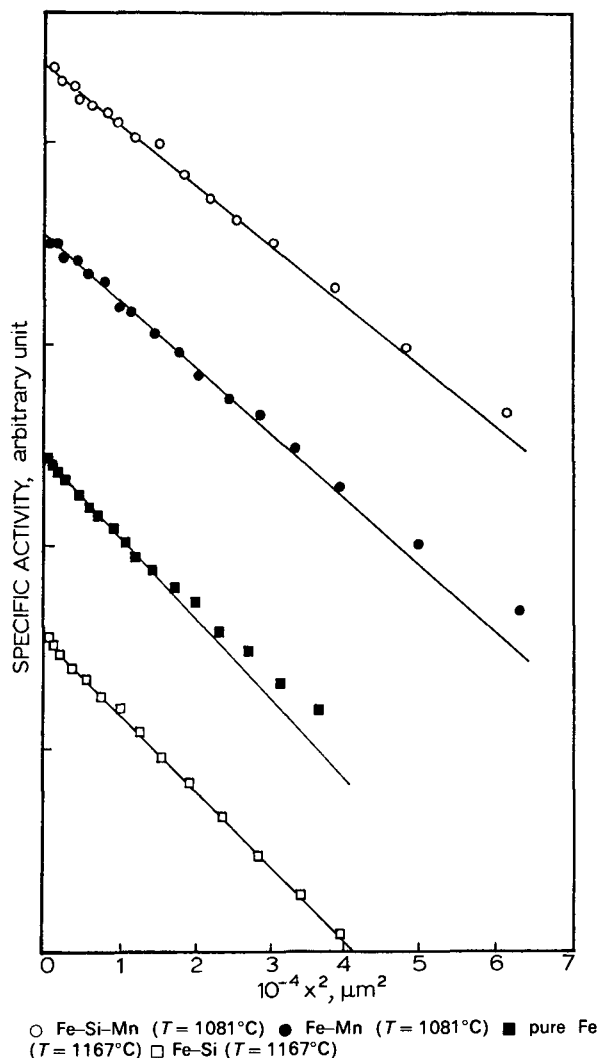
where *c* is the tracer concentration, *x* is the tracer penetration depth, *t* the annealing time, *K* the total mass of tracer deposited, and *D* the volume diffusion coefficient.

The tracer penetration profiles were determined by the direct sectioning method. Since the penetration depths were about 200 μm, it was necessary to use a precision abrading device which is capable of extracting layers to a depth of about 1 μm, the successive faces being kept parallel to within 1/3°. The layer thickness was determined by weight difference and the activity of the extracted layer was measured using a Na(Tl)I detector. Specific activity, which is proportional to concentration, was plotted as a semilogarithmic function of the square of the penetration depth. The coefficients of diffusion were computed from equation (1) by a least squares program, taking into account the errors in both variables. The resultant error in *D* was estimated to be ≤ 5%.

RESULTS

The values obtained for the diffusion coefficients of Nb in the γ phase in Fe, Fe-Mn, Fe-Si, and Fe-Mn-Si are presented in Table 2. These values were determined from the penetration profiles, four examples of which are illustrated in Fig. 2.

From the Arrhenius curves, values of the frequency factor *D*₀ and of the activation energy *Q* were also calculated for the four compositions. These are given in Table 3. It is interesting to note that the values of *D*₀ and *Q* for Nb in unalloyed Fe reported by Sparke *et al.*¹⁷ (see Table 4) are considerably higher than the values established in this investigation. Sparke *et al.* also employed a radioactive tracer technique, but their work



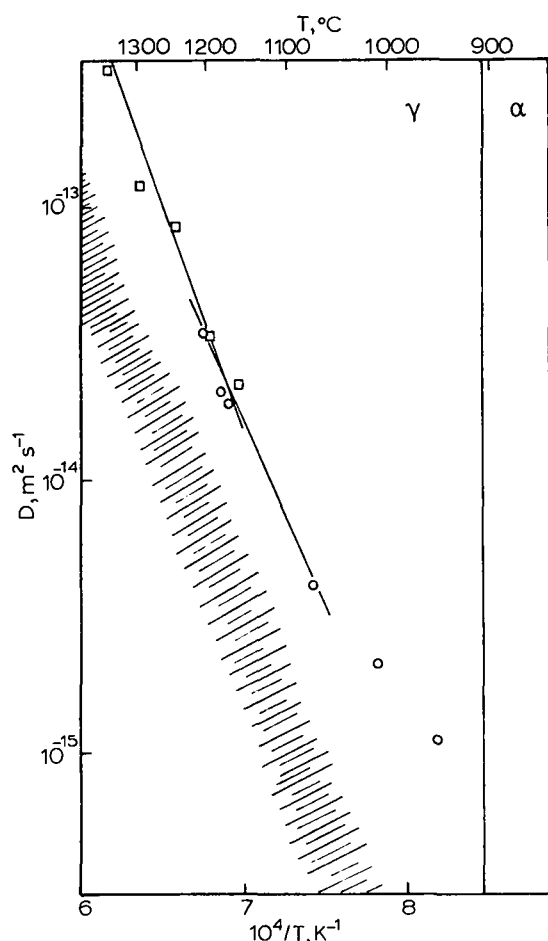
2 Diffusion profiles

covered a higher temperature range, i.e. 1150–1350°C, than that of 948–1201°C used in this study (see Fig. 3).

It has been established that in 'normal impurity diffusion' the impurity diffusion coefficient $D = D_0 \exp(-Q/RT)$ and the pure solvent selfdiffusion coefficient $D_s = D_{0s} \exp(-Q_s/RT)$ do not differ by more than about one order of magnitude, because the differences between *Q* and *Q*_s and *D*₀ and *D*_{0s} are small. It is known that, usually, 0.75 < *Q*/*Q*_s < 1.25 and 0.1 < *D*₀/*D*_{0s} < 10 with the smaller (larger) values of *D*₀ tending to be associated with the smaller (larger) values of *Q* (Ref. 43). The values for *D*₀ and *Q* reported by Sparke *et al.*¹⁷ do not satisfy entirely these relations: *Q*/*Q*_s takes the upper limit value of the range given above, but *D*₀, which generally lies in the range 10⁻⁶ < *D*₀ < 10⁻³ m² s⁻¹, has an unusually high value

Table 2 Diffusion coefficients of Nb in pure Fe, Fe-1.5Mn, Fe-0.6Si, and Fe-1.5Mn-0.6Si alloys

Annealing temperature <i>T</i> , °C	Annealing time <i>t</i> , s	Pure Fe		Fe-Mn		Fe-Si		Fe-Mn-Si	
		<i>D</i> × 10 ¹⁴ , m ² s ⁻¹	Error, %	<i>D</i> × 10 ¹⁴ , m ² s ⁻¹	Error, %	<i>D</i> × 10 ¹⁴ , m ² s ⁻¹	Error, %	<i>D</i> × 10 ¹⁴ , m ² s ⁻¹	Error, %
1201	102780	3.37	2	2.80	3	3.61	1	3.26	4
1185	604800	2.10	5
1167	172440	1.85	3	1.76	1	1.96	1	1.93	1
1081	873960	0.476	4	0.427	2	0.487	3	0.457	4
1007	4019100	0.210	3
948	4844400	0.110	4



○ present work □ Sparke *et al.*¹⁷

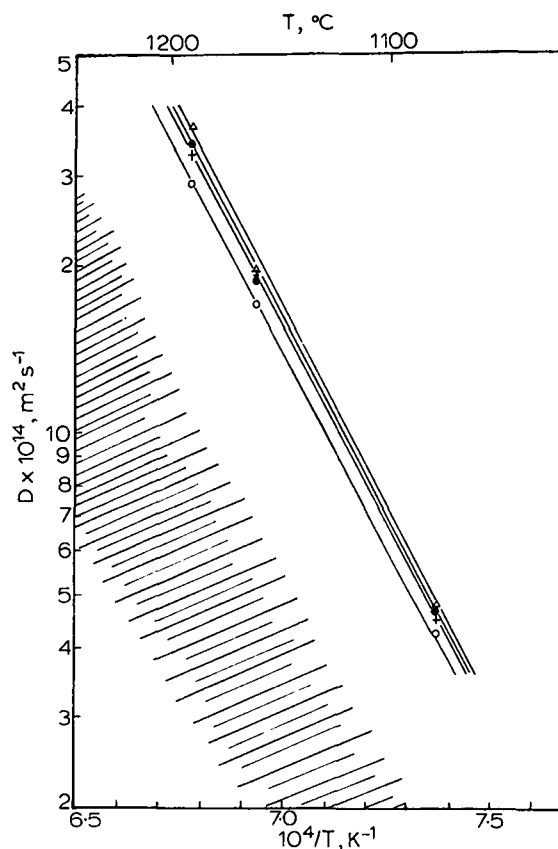
3 Temperature dependence of Nb diffusivity in γ Fe: shaded area is Fe selfdiffusion band

that completely breaks down the relation for D_0/D_0 given above and balances the large value of Q . It is interesting to note that when the diffusion coefficients of Sparke *et al.* and the values obtained in the present work are taken together, within the temperature range 1081–1350°C, the resulting activation energy and frequency factor are normal: $Q = 307 \text{ kJ mol}^{-1}$ and $D_0 = 2.70 \times 10^{-3} \text{ m}^2 \text{ s}^{-1}$. Below 1081°C, the present experimental points do not lie on a straight line (see Fig. 3) and the discrepancies between these and the diffusion coefficient values extrapolated with the parameters of Sparke *et al.* are even greater, and will be discussed in more detail in a future publication. The decrease in slope at lower temperatures is similar to that observed by Gruzin *et al.*⁴⁴ for selfdiffusion of Fe in γ Fe and in Fe–Ni and Fe–Ni–C alloys. It is normally attributed to the influence of either pipe or grain boundary diffusion.

The systematic dependence of the diffusion coefficients displayed in Table 2 on the presence of Mn and/or Si is of greater importance: the trends are illustrated in Fig. 4. It is apparent from both the figure and the table that the diffusivity of Nb in Fe is decreased by the addition of Mn

Table 3 Activation energies and frequency factors for Nb diffusion in pure Fe, Fe–1.5Mn, Fe–0.6Si, and Fe–1.5Mn–0.6Si

	Pure Fe	Fe–Mn	Fe–Si	Fe–Mn–Si
$Q, \text{ kJ mol}^{-1}$	264 ± 11	264 ± 5	270 ± 11	271.7 ± 0.4
$D_0 \times 10^4, \text{ m}^2 \text{ s}^{-1}$	0.75 ± 0.02	0.66 ± 0.11	1.29 ± 0.03	1.40 ± 0.07



△ Fe–Si ● pure Fe + Fe–Mn–Si ○ Fe–Mn

4 Temperature dependence of Nb diffusivity in pure Fe, Fe–Mn, Fe–Si, and Fe–Mn–Si alloys: shaded area is Fe selfdiffusion band

and increased by the addition of Si. The two effects approximately balance each other when Mn and Si are added simultaneously. The addition of Si in Fe–Si alloys is known to have a similar effect in α phase: the diffusivities of Si and Fe increase monotonically with increasing Si content.⁴⁵ The bulk diffusivities of Fe, Cr, and Ni in austenitic stainless steel also increase with increasing Si content.⁴⁶ No systematic variations in diffusivity with addition of Mn have been reported to date in Fe–Mn alloys.

DISCUSSION

The effect of the addition of Mn and Si on the diffusivity of Nb in γ Fe depicted in Fig. 4 requires comment. First, it must be stated that although the changes in the diffusivities are not large, they are clearly greater than the experimental errors. This can be seen from the errors listed in Table 2, which range from 1 to 4% with an average value of about 2.5%. By contrast, the decrease in the diffusivity of Nb associated with the addition of Mn has an average value of 11%, and the increase attributable to the addition of Si has a mean value of 5%. All the increases and decreases evident in Table 2 are consistent and systematic; they are also in qualitative agreement with the effects

Table 4 Comparison of values of Q and D_0 for Nb in pure Fe

$Q, \text{ kJ mol}^{-1}$	$D_0 \times 10^4, \text{ m}^2 \text{ s}^{-1}$	Reference
264	0.75	present work
344.5	530	17
307	27	from both 17 and present empirical data

observed in a preliminary study, where the rate of V tracer diffusion was measured in unalloyed γ Fe and in a series of Fe-Mn and Fe-Si alloys.⁴⁷

The accelerating effect of the addition of Si is perhaps the most easy to rationalize since the diffusion of Si is relatively fast⁴⁸ and its addition, therefore, leads to an increase in the selfdiffusivity of the matrix.⁴⁵ The change in the selfdiffusivity can in turn be expected to enhance the diffusivity of other elements, such as Nb in the present case. The reasons for the decelerating effect of the addition of Mn are perhaps less clear and as has been stated no systematic variations in diffusivity have been reported to date in Fe-Mn alloys.

It is worth noting that alloying with Si leads to increases in both D_0 and Q , whereas the addition of Mn leads to decreases in these two quantities (see Table 3). For both additions the effect on the diffusivity of the change in D_0 overrides the opposite effect of the change in Q the values of which lie, in fact, within the experimental error. Thus, the macroscopic influence of Si and Mn on diffusion in γ Fe can be interpreted as arising largely from the increase and decrease, respectively, that each produces in the pre-exponential factor.

It may be more relevant to speculate on the influence of these effects on other processes, where diffusivity changes might have some bearing such as, for example, diffusion controlled phase transformations. The relevance of these transformations in Fe steel alloys need not be discussed here. The notorious effect of Mn, and to a lesser extent Si, on the transformation kinetics of a wide range of steel compositions is well known. Since Nb is a fairly recent addition to the list of steel components, there is relatively less information available concerning its effect on steel transformations. Precipitated Nb components, i.e. NbC and Nb(CN), are by far the most common phases now being used in commercial steels for C stabilization in stainless steels,⁴⁹ and particularly for grain size control and precipitation hardening in HSLA steels.⁵⁰

In the latter case, precipitation of Nb(CN) in the austenite phase helps to retard recrystallization during rolling and thus, because of the resulting ferrite grain refinement, leads to strengthening. After transformation, further precipitation in the ferrite phase leads to an additional component of strengthening. It is interesting to note that the addition of Mn can reduce the proportion of the γ precipitate and, therefore, produce more precipitation hardening in the ferrite. By reducing the precipitation rate in the austenite, the addition of Mn can also increase the amount of Nb remaining in solution at temperatures in the low γ range and, thus, both increase the hardenability and reduce the continuous cooling transformation temperature. These changes can in turn affect the proportions of γ and α present at low finishing temperatures and, as a result of the change in the transformation temperature, further decrease the final α grain size.

The considerable effect of Nb in delaying static and dynamic recrystallization of austenite is traditionally attributed to boundary pinning by deformation induced precipitation. The authors share the view, which is slowly gaining support, that solute effects by mechanisms not as yet fully described are of similar or greater relevance than precipitates.

Whatever the mechanism, it is obvious that diffusivity of the solute, as in the 'solute drag' mechanism,^{51,52} must be considered. In this context, the relatively higher diffusivity of Nb, confirmed by this work, is of particular interest. However, when very fast processes such as dynamic recrystallization are considered, it is immediately apparent that 'normal' diffusion cannot account for the transport of mass that takes place in such an extremely short time. An alternative would be some defect enhanced mechanism of the type proposed by McLean⁵³ and observed by Esperon

and Hey for MnS in steel.⁵⁴

The reduction in the diffusivity of Nb produced by the addition of Mn described above helps to explain the recent results of Akben *et al.*⁵⁵ concerning the precipitation kinetics of Nb in Fe-Mn-Nb steels. They noted that the rate of precipitation decreased markedly with the addition of Mn, the nose of the C curve moving from about 1 s in the presence of 0.42%Mn to about 20 s when the Mn concentration was raised to 1.90%. They attributed these changes to two effects. Above the nose, the reduced rate of precipitation was associated with the increase in solubility of the carbonitrides, and therefore with the decreased driving force for precipitation, caused by the addition of Mn. The increased solubility is linked to the decrease in the activity of C that occurs when the concentration of Mn is raised.⁵⁶ Below the nose, Akben *et al.*⁵⁵ speculated that the reduced rate of precipitation and of precipitate growth in the presence of higher levels of Mn was consistent with a possible reduction in the rates of diffusion under these conditions. It is clear that the present result (that the diffusivity of Nb in γ Fe decreases with increasing Mn content) lends support to this interpretation. The existence of the decelerating effect of Mn also suggests that investigations similar to that described above could fruitfully be carried out on related compositions of Fe alloys. It would, for example, be of interest to determine whether the presence of Mo decreases the diffusivity of Nb in γ Fe. Such a result would be consistent with the observation that the addition of Mo leads to a decrease in the growth rate of Nb(CN) in deformed austenite.⁵⁷

CONCLUSIONS

The diffusivity of Nb in γ Fe was measured over the temperature range 948–1021°C and the following values of the diffusion parameters for the temperature range 1081–1201°C were obtained: $D_0 = 7.50 \times 10^{-5} \text{ m}^2 \text{ s}^{-1}$ and $Q = 264 \text{ kJ mol}^{-1}$. The Arrhenius plot begins to curve at temperatures below 1081°C.

The addition of 1.5%Mn and/or 0.6%Si to γ Fe has the following effect on the diffusion behaviour. The addition of Mn decreases the diffusivity of Nb in γ Fe by about 11%, a result which coincides with observations on the precipitation kinetics of Nb(CN), the addition of Si increases the diffusivity of Nb in γ Fe by about 5%, and after the addition of both Si and Mn the diffusivity of Nb in γ Fe is approximately equal to that in pure γ Fe.

Nb is one of the fast diffusing metallic elements in Fe and Fe alloys.

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