Diffusion of Aluminum in β -Titanium

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Interdiffusion coefficient $\tilde{D}$ in the β -phase of Ti-Al alloys has been determined by Matano's method in the temperature range 1323 to 1823 K with (pure Ti)-(Ti-8.5 at% Al alloy), (pure Ti)-(Ti-16.5 at% Al alloy) and (Ti-8.5 at% Al alloy)-(Ti-16.5 at% Al alloy) couples. In the whole temperature range the value of $\tilde{D}$ increases gradually with increasing aluminum content. The Arrhenius plot of $\tilde{D}$ up to 6 at% Al shows an upward curvature similar to that recognized in the self-diffusion in β -Ti. The curvature becomes small with increasing aluminum content, and it is nearly linear in the concentration more than 10 at% Al. The activation energies for the impurity diffusion in β -Ti are proportional to the square of radius of the diffusing atom. This suggests that the size effect is dominant in the impurity diffusion in β -Ti.

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1. Introduction

It is well known that the IVb metals (Ti, Zr and Hf) and their alloys in the b.c.c. phase show a so-called anomalous diffusion behavior which can be characterized by a significant upward curvature in the Arrhenius plot of the diffusion coefficients.¹⁾ This behavior has been explained by the model of phonon-assisted diffusion jumps via monovacancies by Köhler and Herzig.^{2,3)} They have proposed a mechanism of temperature dependence of the self-diffusion coefficient taking into account a temperature-dependent migration energy of a vacancy on the basis of the experimental evidence of significant lattice softening of the longitudinal acoustic (LA) phonon in the $\langle 111 \rangle$ direction at the reduced wave vector $2/3$, LA2/3 $\langle 111 \rangle$ phonon, in β -Zr.⁴⁾ The LA2/3 $\langle 111 \rangle$ phonon mode in the b.c.c. lattice is directly related to the nearest-neighbor jump process of the diffusing atom, because its displacement pattern facilitates the promotion of the migrating central atom through the saddle point into the position of the vacancy. Therefore the softening of this phonon mode results in a reduction in the restoring forces in the $\langle 111 \rangle$ direction and in an overall decrease in the free energy G^M of atomic migration. Furthermore, investigations on the phonon dispersion in IVb metals, β -Ti,⁵⁾ β -Zr⁶⁾ and β -Hf,⁷⁾ have revealed a considerable softening of the transverse acoustic (TA) T₁A1/2 $\langle 110 \rangle$ phonon with decreasing temperature in addition to the significant softening of the LA2/3 $\langle 111 \rangle$ phonon with negligible temperature dependence. These observations support strongly the mechanism by Köhler and Herzig.^{2,3)} Besides the atomic displacement in the jump direction the importance of the concomitant displacements of the triangular configuration of the saddle-point atoms has been pointed out.³⁾ An opening motion of the saddle-point atoms perpendicular to the jump direction decreases the potential barrier for the jump. Such a 'breathing' is partly achieved by the LA2/3 $\langle 111 \rangle$ phonon and partly by the T₁A1/2 $\langle 110 \rangle$ phonon.^{8,9)} The degree of softening of

the LA2/3 $\langle 111 \rangle$ and T₁A1/2 $\langle 110 \rangle$ phonons correlates with the activation energy for self-diffusion in the β -phase of IVb metals.

According to Köhler and Herzig,²⁾ G^M in the anomalous b.c.c. metals is expressed by

$$G^M = G_0^M(1 - T_0/T), \quad (1)$$

where G_0^M is the free energy of migration of a monovacancy and T_0 is the hypothetical lowest temperature for the metal to hold the b.c.c. structure. If the lattice is completely softened at T_0 , the frequency of LA2/3 $\langle 111 \rangle$ would diminish to zero, then G^M would diminish to zero. Thus the temperature dependence of the diffusion coefficient D in the anomalous b.c.c. metals should be expressed by

$$D = D_0 \exp(-Q/RT) \exp(G_0^M T_0/RT^2), \quad (2)$$

where D_0 and Q are the preexponential factor and the activation energy, respectively, for the monovacancy mechanism. The extent of deviation from the linearity in the Arrhenius plot of the diffusion coefficient is represented by the term $\exp(G_0^M T_0/RT^2)$ in eq. (2). A similar equation to eq. (2) has been obtained by Sanchez and de Fontaine^{10,11)} on the basis of the ω embryo model where the ω embryo in the transition from the β -phase to the ω -phase has the same structure as the activated complex, and the ω embryo is also regarded as a lattice in significantly softened state of the LA2/3 $\langle 111 \rangle$ phonon.

In our previous studies on the impurity diffusion of transition elements (Cr and Pd),¹²⁾ Ib elements (Cu,¹³⁾ Ag¹³⁾ and Au¹⁴⁾, IIb elements (Ga and In)¹⁵⁾ and IVb elements (Si, Ge and Sn)¹⁶⁾ in β -Ti, eq. (2) has been confirmed to hold well, and it has been recognized that the activation energies for the impurity diffusion and self-diffusion²⁾ in β -Ti are proportional to the square of the atomic radius of the diffusing atom. In the present work, diffusion behavior of IIb element Al in β -Ti has been investigated. Although the diffusion coefficients of Al in β -Ti have been measured by Araki *et al.*¹⁷⁾ and Köppers *et al.*,¹⁸⁾ the diffusion parameters in eq. (2) have not been determined. In all the β -phase of dilute alloys of Ti with Cr, Pd, Cu, Ag, Au, Ga, In, Si, Ge

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and Sn, the interdiffusion coefficient $\tilde{D}$ is independent of concentration of solute.^{12–16)} On the other hand, $\tilde{D}$ in the β -phase up to 2 at% Al of Ti–Al alloys increases with increasing Al content.¹⁷⁾ Then it is interesting to examine whether the curvature of the Arrhenius plot of $\tilde{D}$ depends on the Al content in the β -phase.

In the present work, interdiffusion experiments with the couples of pure Ti and the β -phase Ti–Al alloys containing 8.5 and 16.5 at% Al have been made. The impurity diffusion coefficient of Al in β -Ti has been determined by applying Darken's relation,¹⁹⁾ i.e. the extrapolated value of the interdiffusion coefficient to the infinite dilution of the solute can be regarded as the impurity diffusion coefficient of the solute in β -Ti. This method is effective to avoid some troubles such as chemical reaction on the surface of reactive Ti specimen with radiotracer diffusion experiments. Furthermore, this is especially useful in determining the impurity diffusion coefficient of element, such as Al for which the tracer diffusion experiment with a radioactive isotope is not easy.

2. Experimental Procedure

Pure rods 12 mm in diameter and 100 mm in length were machined from a Ti bar 160 mm in diameter supplied by Kobe Steel Ltd. The main impurities in this material were 0.046 mass% Fe, 0.0032 mass% N and 0.0041 mass% O. The rods were polished chemically, sealed in quartz tube with high-purity Ar gas and then annealed at 1373 K for 172.8 ks (2 days) to cause grain growth. Alumina rings (higher than 99% purity) were fitted at both ends of the rod to prevent reaction with the quartz tube. The resultant grain size was about 3 mm. The rod was cut to make disc specimens 5 mm in thickness. To obtain a fully flat surface, the specimen was set in a stainless steel holder 50 mm in diameter, ground on abrasive papers and polished on a buff with fine alumina paste.

Buttons of Ti–8.5 and 16.5 at% Al alloys were made by Ar arc melting the pure Ti block with Al blocks of 99.999% purity. To homogenize the buttons, the arc melting was repeated a few times. Finally, the buttons were cast into a water-cooled copper boat to make a rod ingot 10 mm in diameter and 80 mm in length. The resultant grain size in alloy rods after the same grain growth treatment as described above was about 2 mm. The alloy rods were cut to make disc specimens 5 mm in thickness. The cut surface of the alloy specimen was ground and polished in the same way as the pure Ti specimen.

To make the semi-infinite interdiffusion couple the pure Ti and the alloy discs were put in a stainless steel holder with two screws, pressed by the screws, wrapped with a V foil, surrounded by Ti sponges and then diffusion welded by heating at 1073 K for 3.6 ks in a stream of high-purity Ar gas. After the diffusion welding, the couple was removed from the holder and put into alumina tube; then the alumina tube containing the couple was sealed in a quartz tube with the high-purity Ar gas. By putting the quartz tube in a furnace, diffusion annealing was carried out at temperatures in the range from 1323 to 1823 K for between 3.6 and 691 ks (1 h to 80 d). At the temperatures higher than 1773 K, the diffusion

time has been corrected taking account of the amount of diffusion occurred during heating the specimen from room temperature to the diffusion temperature.²⁰⁾ For diffusion above 1673 K, the alumina tube containing the couple was sealed in double tubes of quartz, keeping the inner quartz tube from crushing by adjusting the pressure of Ar gas between the inner and outer tubes. The diffusion temperature was controlled to within ± 1 K at 1323–1473 K and to within ± 3 K at 1673–1823 K.

After the diffusion, the couple was cut to parallel to the diffusion direction, and the cut surface was polished on a buff with fine alumina paste to examine the concentration-penetration profiles with an electron probe microanalyzer. The concentration of Al was determined by using the ZAF method. The interdiffusion coefficient was calculated as a function of solute concentration by Matano's method.²¹⁾

3. Results and Discussion

Figure 1 shows the concentration dependence of the interdiffusion coefficient $\tilde{D}$ determined in the temperature range 1323 to 1823 K with (pure Ti)–(Ti–8.5 at% Al alloy) couple and in the temperature range 1423 to 1673 K with (pure Ti)–(Ti–16.5 at% Al alloy) couple and in the temperature range 1473 to 1573 K with (Ti–8.5 at% Al alloy)–(Ti–16.5 at% Al alloy) couple. The interdiffusion coefficients shown in the present work include the experimental error of 10 to 15%. Interdiffusion coefficients determined for different diffusion times are shown distinguishably from each other by different marks. As shown in Fig. 1, at each temperature $\tilde{D}$ is independent of diffusion time and $\tilde{D}$ increases almost linearly with increasing Al content. According to Darken's relation,¹⁹⁾ the extrapolated value of $\tilde{D}$ to the infinite dilution of Al can be regarded as the impurity diffusion coefficient

Fig. 1 Concentration dependence of interdiffusion coefficient $\tilde{D}$ in Ti–Al alloys.

Table 1 Diffusion coefficient of Al in β -Ti.

Temperature/K	Diffusion coefficient/ $\text{m}^2 \cdot \text{s}^{-1}$
1823	$(8.68 \pm 1.02) \times 10^{-12}$
1773	$(5.91 \pm 0.50) \times 10^{-12}$
1723	$(4.26 \pm 0.29) \times 10^{-12}$
1673	$(3.21 \pm 0.42) \times 10^{-12}$
1623	$(1.94 \pm 0.47) \times 10^{-12}$
1573	$(1.33 \pm 0.19) \times 10^{-12}$
1523	$(8.98 \pm 0.13) \times 10^{-13}$
1473	$(5.76 \pm 0.12) \times 10^{-13}$
1423	$(3.61 \pm 0.63) \times 10^{-13}$
1373	$(2.12 \pm 0.31) \times 10^{-13}$
1323	$(1.31 \pm 0.15) \times 10^{-13}$

Fig. 2 Temperature dependence of impurity diffusion coefficients of Al and self-diffusion coefficient in β -Ti.

D_{Al} of Al in β -Ti. The values of D_{Al} determined in this way, using the linear fitting function, are listed in Table 1.

Figure 2 shows the temperature dependence of the impurity diffusion coefficient D_{Al} obtained by the present work along with those by Araki *et al.*¹⁷⁾ and Köppers *et al.*¹⁸⁾ The value of D_{Al} by the latter are taken from the figure given by Mishin and Herzig.¹⁸⁾ The temperature dependence of D_{Al} obtained by these three groups shows excellent agreement each other and can be expressed by a single line as follows;

$$D_{\text{Al}} = (3.03 + 2.85/T - 1.47) \times 10^{-4} \times \exp(-331.6 \pm 16.3 \text{ kJ} \cdot \text{mol}^{-1}/RT) \times \exp(125.6 \pm 11.9 \text{ MJ} \cdot \text{mol}^{-1}/RT^2) \text{ m}^2 \cdot \text{s}^{-1} \quad (3)$$

As shown in Fig. 2, the value of D_{Al} is about one half of the self-diffusion coefficient D_{Ti} of Ti in the whole temperature range of the β -phase. The softening of the LA2/3 $\langle 111 \rangle$ and T1A1/2 $\langle 110 \rangle$ phonons increases the diffusion coefficient D_{Al} of Al by the factor $\exp(125.6 \text{ MJ} \cdot \text{mol}^{-1}/RT^2)$. The values of this factor at the melting temperature of Ti ($T_{\text{m}} = 1943 \text{ K}$) and the β - α transformation temperature of Ti ($T_{\beta-\alpha} =$

Fig. 3 Temperature dependence of interdiffusion coefficient $\tilde{D}$.Table 2 Diffusion parameters, D_0 , Q and $G_0^{\text{M}}T_0$.

Concentration of Al/at%	$D_0/\text{m}^2 \cdot \text{s}^{-1}$	$Q/\text{kJ} \cdot \text{mol}^{-1}$	$G_0^{\text{M}}T_0/\text{MJ} \cdot \text{mol}^{-1} \text{ K}$
0	$(3.03^{+2.85}_{-1.47}) \times 10^{-4}$	331.6 ± 16.3	125.6 ± 11.9
2	$(3.91^{+7.96}_{-2.62}) \times 10^{-5}$	275.3 ± 28.7	82.1 ± 22.1
6	$(5.31^{+2.35}_{-0.43}) \times 10^{-6}$	220.8 ± 43.6	42.3 ± 33.5
10	$(7.02^{+1.67}_{-1.11}) \times 10^{-7}$	163.9 ± 2.7	
12	$(7.46^{+2.08}_{-1.63}) \times 10^{-7}$	162.3 ± 3.2	

1155 K) are 54.7 and 8.3×10^4 , respectively. The corresponding values for self-diffusion in Ti are 61 and 1.1×10^5 , respectively.

Figure 3 shows the temperature dependence of the interdiffusion coefficient $\tilde{D}$ at 0, 2, 6, 10 and 12 at% Al. At 2 and 6 at% Al the temperature dependence of $\tilde{D}$ shows a similar upward curvature to those of D_{Al} and D_{Ti} . At 6 at% Al the curvature in Fig. 3 is small. At 10 and 12 at% Al the Arrhenius plots show almost linearity. Then, the diffusion parameters in eq. (2) are calculated for each concentration of Al and listed in Table 2. The activation energy Q decreases with increasing Al content. Furthermore, the term $\exp(G_0^{\text{M}}T_0/RT^2)$ which represents the extent of deviation from the linearity in Arrhenius plot decreases also with increasing Al content and it becomes nearly zero at 10 at% Al. This suggests that the phonon softening in the β -phase of Ti-Al alloy becomes weak with increasing Al content.

It is interesting to examine the relationship between $G_0^{\text{M}}T_0$ and Q for diffusion in β -Ti. Since Q is the sum of enthalpy H_0^{F} of formation and the enthalpy H_0^{M} of migration of a monovacancy, $G_0^{\text{M}}T_0$ can be written as

$$G_0^{\text{M}}T_0 = (H_0^{\text{M}} - TS_0^{\text{M}})T_0 = (Q - H_0^{\text{F}})T_0 - T_0S_0^{\text{M}}T, \quad (4)$$

where S_0^{M} is the entropy of migration of a monovacancy. According to Sanchez,²²⁾ the term $T_0S_0^{\text{M}}$ is negligible

Fig. 4 Correlation between $G_0^M T_0$ and Q .

compared with $H_0^M (= Q - H_0^F)$. Because we are concerned with β -Ti, $T_{\beta-\alpha} (= 1155 \text{ K}) < T < T_m (= 1943 \text{ K})$, and T_0 is estimated to be 610 K as described below. Thus the second term of the right-hand side in eq. (4) is much smaller than the first term. Then $G_0^M T_0$ increases with increasing activation energy Q . As shown in Fig. 4, this is recognized by the experimental results of Sc,²³⁾ V,²⁴⁾ Ta,²⁵⁾ W²⁶⁾ and Zr²⁷⁾ including results of present authors^{12–16)} and a linear relation

$$G_0^M T_0 (\text{MJ} \cdot \text{mol}^{-1} \text{K}) = 0.610[Q (\text{kJ} \cdot \text{mol}^{-1}) - 129] \quad (5)$$

is obtained, indicating that H_0^F does not depend on the element. The value of H_0^F is estimated to be $129 \pm 14 \text{ kJ} \cdot \text{mol}^{-1}$ by extrapolating $G_0^M T_0$ to zero in Fig. 4. From the slope of the straight line in Fig. 4, T_0 is estimated to be $(610 \pm 26) \text{ K}$. This means that the activation energy Q for the impurity diffusion in β -Ti is controlled by the value of H_0^M rather than by H_0^F . The magnitude of H_0^M should be connected with the ‘breathing’ motion of the saddle-point atoms and thus correlated with the size of diffusing atom.

Now, we examine H_0^M for impurity diffusion in β -Ti. According to Sanchez and de Fontaine,¹⁰⁾ the free energy G_ω of formation of the ω embryo (or the activated complex), is correlated with the transforming parameter ε , from the β -phase to the ω -phase by

$$G_\omega = A(T, p)\varepsilon^2 - B(T, p)\varepsilon^3, \quad (6)$$

where $A(T, p)$ and $B(T, p)$ are functions of the temperature T and pressure p . The transforming parameter ε is proportional to the atomic displacement in the β -phase lattice which participates in the formation of the ω embryo.²⁸⁾ $\varepsilon = 0$ in the b.c.c. structure, but $\varepsilon = 1$ in the transformed lattice (ω -phase). The driving force for the transformation expressed by the second term $B(T, p)\varepsilon^3$ is negligible at high temperatures but increases with decreasing temperature. On the other hand, in a temperature range much higher than the highest temperature T_ω for the ω -phase to exist only the first term

Fig. 5 Correlation between activation energy Q for diffusion against square r^2 of radius of diffusing atoms in β -Ti.

$A(T, p)\varepsilon^2$ is significant, and the normal monovacancy mechanism operates for the diffusion; then

$$G_\omega(T \gg T_\omega) \approx A\varepsilon^2 = G_0^M = H_0^M - TS_0^M. \quad (7)$$

From theoretical considerations,²²⁾ it has been supported that $T_{\beta-\alpha} > T_\omega > T_0$. In the process of formation of the ω -phase of the h.c.p. structure from b.c.c. β -Ti by the lattice displacement in the $\langle 111 \rangle$ direction, the intrinsic strain due to an activated configuration contributing to the transformation to the ω -phase comes about 3.5% in the direction $[121]$ in the β -phase. However, if the atomic radius of one of the two atoms located on the unit cell of the ω -phase is larger by 1.7% than that of the other atom, the intrinsic strain in the ω -phase must be just cancelled out, and ε becomes unity. Furthermore, in the activated configuration the b.c.c. structure becomes unstable, and the migration energy of an activated atom to the nearest-neighbor vacancy becomes a maximum. If the radius r of the diffusing impurity atom is smaller than 1.017 times the radius r_m of the solvent atom, the parameter ε can be represented by

$$\varepsilon = r/1.017 r_m (r \leq 1.017 r_m) \quad (8)$$

and, for the impurity atom larger than $1.017 r_m$,

$$\varepsilon = 1 - (r - 1.017 r_m)/1.017 r_m (r \geq 1.017 r_m) \quad (9)$$

From eqs. (7)–(9), it can be concluded that the migration enthalpy of an impurity in the direction $[121]$ is proportional to the square of the radius of the impurity atom. As the coordination number in the b.c.c. structure of the β -Ti is eight, the atomic radius of the metal of different structure from the b.c.c. is converted for a coordination number of eight by the conversion relation.²⁹⁾ As shown in Fig. 5, for $r \leq 1.017 r_m$, Q increases linearly with increasing r^2 , although the value of Q for W is much higher than those for the

other elements and, for $r \geq 1.017 r_m$, Q decreases with increasing r^2 . This is consistent with the prediction that $H_0^M (= Q - H_0^F)$ is proportional to r^2 , as deduced from eqs. (7)–(9). Thus, it must be emphasized that the size effect is dominant in the impurity diffusion in β -Ti.

4. Conclusions

The present experimental results show the Arrhenius plot of the impurity diffusion coefficient of Al in β -Ti in the temperature range 1323–1823 K exhibit an upward curvature. This can be explained by a monovacancy mechanism with a temperature-dependent migration energy due to softening of the LA2/3 $\langle 111 \rangle$ and T₁Al/2 $\langle 110 \rangle$ phonons in the β -Ti. Further, the weak curvature of the Arrhenius plots of interdiffusion coefficient in the β -phase of Ti-Al alloys less than 6 at% Al has been observed. The activation energy for the impurity diffusion and self-diffusion in β -Ti is proportional to the square of the radius of the diffusing atom.

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