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DIFFUSION ALONG EDGE DISLOCATIONS

IN Ni - Co ALLOYS

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INTRODUCTION

While it has been clearly shown in recent years that enhanced diffusion can occur along grain boundaries and dislocations, the mechanism by which this occurs is not known. Direct measurements of diffusivity along dislocations⁽¹⁻⁴⁾ and measurements of the anisotropy of diffusion along grain boundaries^(5,6) have shown that the enhanced diffusion occurs along the dislocation lines. A number of mechanisms for the motion of solute or solvent atoms along the dislocation lines have been proposed:

(a) Vacancy mechanism.^(4,7-9) If the atomic motion along the dislocation occurs by a vacancy exchange mechanism the activation energy for diffusion can be written:

$$Q_p = E_{m,lv}^p + E_{F,lv}^l + B_{lv} \quad (1)$$

where $E_{m,lv}^p$ is the energy of motion of a vacancy along the dislocation, $E_{F,lv}^l$ is the formation energy of a vacancy in the lattice, and B_{lv} is the binding energy of the vacancy to the dislocation. Since the diffusion measurements determine Q_p and $E_{m,lv}^p$ and B_{lv} are not known it has not been possible to test Equation 1 experimentally. However, in the case of diffusion along edge dislocations in Ni⁽⁴⁾ it was shown that Equation 1 is consistent with reasonable values of B_{lv} and that $E_{m,lv}^p < (E_{F,lv}^l + B_{lv})$. Equation 1

was also shown to be consistent with Q_p derived from internal friction measurements⁽⁹⁾ of Si and Ge solute diffusion along dislocations in copper.

(b) Interstitial motion in the dislocation core. Diffusion could occur by motion of the tracer or solute atoms as interstitials along the dislocation cores. While this appears to be a possible mechanism in those cases where the solute has a much smaller atomic size than the solvent it is not likely to account for substitutional solute or tracer diffusion. Any tracer atom which found itself in an interstitial position at the dislocation core might diffuse as an interstitial until it encountered a jog. At the jogs the interstitial would be adsorbed onto the jog structure and would have a very low probability of being re-emitted unless the interstitial formation energy at the dislocations is very low, i.e., a binding energy between interstitials and dislocations of several eV. This mechanism may, however, be applicable to the cases of interstitial solute diffusion (C, N, O, etc.) along dislocations. Na diffusion along dislocations in LiF⁽¹⁾ has been accounted for by an interstitial mechanism. In the case where the interstitial mechanism applies;

$$Q_p = E_{m,I}^p + B_{I-J} \quad (2)$$

where $E_{m,I}^p$ is the interstitial migration energy at the dislocation

and B_{I-J} is the binding energy of the interstitial to a jog. In the case of self diffusion $B_{I-J} = E_{f,I}^P$, the energy of formation of a self interstitial at the dislocation.

(c) Jog motion. A mechanism related to the interstitial diffusion mechanism is the transport by jog motion along dislocations.⁽⁸⁾ In the case of edge dislocations the solute or tracer atom would move by jog motion along the extra half plane of the dislocation. On encountering another jog, "collision" and jog annihilation occurs reducing the number of jogs by two (in the case of jogs having the same sign) or four (in the case of collision between oppositely signed jogs): The activation energy in this case would be;

$$Q_P = E_{m,J}^P + 2E_{F,J}^P \quad (3)$$

where $E_{F,J}^P$ is the jog formation energy and $E_{m,J}^P$ is the jog motion energy. Since jog formation should be more difficult at dislocations split into partials than at perfect dislocations⁽⁸⁾ the dislocation diffusion activation energy should be higher along partial dislocations if this mechanism applies.

Other mechanisms for diffusion along dislocations based on the ring mechanism⁽¹⁰⁾ or on approximating the dislocation core as a region having liquid character⁽¹¹⁾ can be considered and will be discussed later. In the present paper the dislocation self diffusion studies carried out by Wuttig and Birnbaum⁽⁴⁾

(hereafter denoted as I) on Ni are extended to alloys of Ni-Co to attempt to examine the effect of stacking fault energy on the diffusion mechanism.

EXPERIMENTAL PROCEDURE

The experimental techniques used were similar to those applied in I. A single crystal of high purity Ni (40 wt.%) and Co (60 wt.%) was grown under a dynamic vacuum (10^{-6} torr) using an R.F. induction furnace. Metallographic examination indicated that the crystal was homogeneous in composition and did not contain voids. Specimens having the dimensions and orientations shown in Figure 1a were cut using a spark cutting procedure followed by a chemical polish. These crystals were bent around the $[\bar{1}\bar{2}1]$ to generate about 0.5×10^{-7} dislocations cm^{-2} on the active $(\bar{1}11)[101]$ slip system. The diffusion specimens, cut parallel to the $(\bar{1}\bar{2}1)$ as shown in Figure 1b, were polished and vacuum annealed at 900°C (10^{-7} torr). During the anneal the image forces acting on the dislocations tended to align them perpendicular to the $(\bar{1}\bar{2}1)$ in which orientation they were pure edge dislocations. This was checked by dislocation etch pitting both sides of the specimen and noting that the pattern was essentially identical on both $(\bar{1}\bar{2}1)$ faces.

A thin ^{63}Ni plate (1000 Å) was electrodeposited on one of the $(\bar{1}\bar{2}1)$ faces followed by a plate of nonradioactive Ni producing the specimen shown in Figures c and d. The crystal thickness was of the order of 0.02 cm. Diffusion anneals were carried out in a purified atmosphere of 90% CO_2 + 10% CO and the activity at the non-plated $(\bar{1}\bar{2}1)$ face (exit face) was monitored as a function of the diffusion time. All of the tests discussed in I were carried out to ensure that the tracer on the exit surface arrived by diffusion directly through the wafer specimens.

EXPERIMENTAL RESULTS AND ANALYSIS

The theoretical analysis of pipe diffusion along dislocations is given in I. The dislocation is assumed to be a cylindrical "pipe" of radius r_0 having a diffusivity D_p and extending from the plated surface to the exit surface. The dislocation "pipe" is imbedded in a lattice having $D_l \ll D_p$. Diffusion along the dislocation and loss into the lattice was considered. The amount of tracer $Q(t)$ on the exit surface was calculated for four sets of boundary conditions. The experimentally determined boundary conditions in the case of edge dislocations in pure Ni were a Green's function tracer source at the plated face (corresponding to slow diffusion at the crystal-plated interface) and about zero concentration at the exit face (corresponding to fast surface diffusion on the $(\bar{1}\bar{2}1)$

exit surface). The resulting activity on the exit face for these boundary conditions is:

$$Q(t) = \frac{\alpha \pi r_o^2 \nu}{\cosh(k/\eta)} \left[1 - \pi \cosh(k/\eta) \sum_{n=1}^{\infty} \frac{(-1)^{n-1} (2n-1)}{\xi_n} \exp(-\xi_n^2 t) \right] \quad (4)$$

where α is the Green's function source strength, ν is the dislocation density, $k = 2D_\ell / [r_o^2 \ln(r_m/r_o)]$, $r_m \approx 0.5 \nu^{-1/2}$, $\eta^2 = D_p^2 / \ell^2$, ℓ = specimen thickness, $\xi_n = [(2n-1)/2]^2 + (k/\eta)^2$, and t is the annealing time. $Q(t)$ increases monotonically and approaches an asymptotic value at large t . It may, therefore, be normalized to remove the dependence on α , ν , r_o and the plated area. The normalized $Q(t)$, $Q^*(t)$ depends on two parameters, (k/η) and η^2 where η^2 depends only on D_p and k/η depends also on r_o . Therefore, a two parameter fit of the experimental data to the calculated $Q^*(t)$ results in a determination of the dislocation diffusivity D_p and the dislocation radius r_o .

The solution of the diffusion equation assumed a cylindrical dislocation "pipe". It can be shown, however, that the analysis for a dislocation pipe of elliptical cross section gives the identical result if r_o is interpreted as an "effective radius" i.e., the radius of a circle having the same circumference as the elliptical pipe cross section. The lattice diffusivity D_ℓ for the alloy used was taken from the data of Hässner and Lange⁽¹²⁾ and was given by

$$D_\ell = 0.44 (\text{cm}^2 \text{ sec}^{-1}) \exp -64(\text{Kcal mole}^{-1})/RT. \quad (5)$$

Dislocation diffusion data were obtained for edge dislocations in the 40% Ni 60% Co specimens in the range 620°C to 500°C. The least squares fit of the experimental data reduced to a single curve by proper choice of η^2 and k^2/η^2 at each temperature is shown in Figure 2 for 547.8, 577.5 and 617.8°C. The theoretical curve, Equation 4, is shown as curve A. It should be noted that in addition to the random scatter in the data there is also a significant systematic deviation from the theoretical curve A; the data lie consistently above the curve in the range $0 \leq \eta^2 t \leq 0.3$. This systematic deviation was not observed in the measurements on pure Ni. It appears to be due to a partial failure of the assumption of a Green's function source at the plated surface in the Ni-Co alloy.

The stacking fault energy in pure Ni is of the order of 150 ergs cm² leading to a separation of the partial dislocations of about 3 atomic distances. Tracer atoms can reach the entrance of the dislocation pipe by diffusion along the interface between the specimen and the plated layer. The time dependent flux of the tracer atoms into the pipe entrance can be calculated assuming a circular sink of radius r_o ($C_o = 0$) at the dislocation in a plane of initial concentration C_1 . Under these conditions the flux into the dislocation pipe is given by $J \propto \exp -(D_s t/r_o^2)$ where D_s is the interface diffusivity. If $D_p \gg D_s$ the flux into the dislocation which decreases exponentially, can be closely

approximated by a Green's function source. This appears to be the case for edge dislocations in pure Ni.

In 40% Ni 60% Co the stacking fault energy⁽¹³⁾ is about 30 ergs cm⁻² which corresponds to a separation of the partial dislocations of about 15 atomic distances. Assuming that the stacking faults contribute to tracer flux along the dislocations, the dislocations act as high diffusivity pipes having elliptical cross sections. At the plated surface, therefore, the dislocation can be approximated by a linear sink ($C_0 = 0$) in a plane of initial concentration C_1 . Under these conditions the flux into the pipe is given by $J = C_1 D_s / \sqrt{\pi D_s t}$. The $t^{-1/2}$ dependence of the source strength obtained in this case is a much slower decrease with time than that obtained for the cylindrical pipe. Therefore, a Green's function source is expected to be a poorer approximation at small values of $\eta^2 t$ in the low stacking fault energy material than in those having a high stacking fault energy.

The solution of the diffusion equation which describes dislocation pipe diffusion (as discussed in I) was obtained for the boundary conditions; $C = 0$ at the dislocation exit surface and $J_p = C_1 D_s / \sqrt{\pi D_s t}$, i.e., the flux into the dislocation equal to the flux due to surface diffusion at the dislocation pipe entrance. The time dependent part of the solution for the tracer concentration at the exit surface is given by:

$$Q^*(t) = \int_0^t \left\{ 1 - \pi \cosh(k/\eta) \sum_{n=1}^{\infty} \frac{(-1)^{n-1} (2n-1)}{\xi_n} \exp -\xi_n \eta^2 t' \right\} t'^{-1/2} dt' \quad (6)$$

This expression was numerically evaluated and fitted to the data as shown by curve B, Figure 2. The calculated curve (B) for a time dependent boundary condition appears to fit the data more closely than the calculated curve (A) which uses a Green's function source suggesting that in the Ni-Co specimens surface diffusion at the pipe entrance is more significant than in the pure Ni specimens.

The values of D_p and r_o obtained by fitting the experimental data to the calculated expression are given in Table I. In performing the fit to the calculated curves the greatest weight was given to the points for $\eta^2 t > 0.3$ since these points were least sensitive to the boundary conditions at the dislocation pipe entrance. The dislocation diffusivities obey an Arrhenius expression as shown in Figure 3 with

$$D_p = 3.4 \cdot (\text{cm}^2 \text{ sec}^{-1}) \exp(-30.2 \text{ Kcal mole}^{-1}/RT). \quad (7)$$

TABLE I

Annealing Temperature (°C)	Thickness l (cm)	η^2	k^2/η^2	D_p (cm ² /sec)	r_o (cm)
617.8	0.0220	3.09×10^{-3}	0.01	1.5×10^{-7}	4.3×10^{-6}
577.5	0.0192	2.36×10^{-4}	0.01	8.7×10^{-8}	1.7×10^{-6}
547.8	0.0198	1.13×10^{-4}	0.01	4.43×10^{-8}	10^{-6}
506	0.0142	6.83×10^{-5}	-	1.38×10^{-8}	-

The values of r_o are of the order of 10^{-6} cm and exhibit a significant increase as the diffusion temperature increases. A somewhat smaller increase of r_o with temperature was also noted for pure Ni in I. Using values of the stacking fault energy for the Ni-Co alloy⁽¹³⁾ the spacing of the partial dislocations can be estimated to be 0.5×10^{-6} cm.

DISCUSSION

The dislocation structure of the 40% Ni 60% Co alloy having a stacking fault energy of 30 ergs cm^{-2} can be described as two parallel partial dislocations lying in the $[\bar{1}\bar{2}1]$ and having Burgers vectors of $(a/6)[211]$ and $(a/6)[1\bar{1}2]$. These partial dislocations bound a stacking fault lying on the $(\bar{1}11)$. This configuration is equivalent to an edge dislocation having a Burgers vector $(a/2)[101]$ lying in the $[\bar{1}\bar{2}1]$. Diffusion along these

dislocations can occur either primarily along the cores of the partial dislocations or along the stacking fault joining the partial dislocations as well as along the dislocation cores. In the first case the "pipe diameter," r_o , would be expected to be of the same magnitude as that observed in pure Ni, $\sim 3 \times 10^{-7}$ cm or smaller. In the latter case the "pipe" cross section would be elliptical and the "pipe diameter" would be determined by the spacing of the partial dislocations, i.e., $r_o \approx (1/2\pi) \times \text{circumference of the elliptical cross section of the high diffusion path}$. Using 30 ergs cm^{-2} for the stacking fault energy and 3×10^{-7} cm as the core diameter of the partial dislocations, the "pipe diameter" can be estimated to be $r_o \approx 0.5 \times 10^{-6}$ cm. Experimentally $r_o \geq 10^{-6}$ cm, indicating that enhanced diffusion occurs along the stacking faults as well as along the partial dislocation cores. This conclusion is supported by the previously discussed boundary conditions at the dislocation pipe entrance; the observation that the diffusion data requires the use of a time dependent boundary condition consisting of diffusion along the plated specimen surface into a linear sink is consistent with a dislocation pipe comprised of a stacking fault bounded by two partial dislocations.

It should be noted that the determination of r_o does not depend on any assumptions regarding the mechanism of diffusion along the dislocations. As discussed in I, r_o is determined by

the loss of tracer atoms from the dislocation pipe to the lattice, i.e., by the flux which is approximately normal to the direction of the dislocation pipe.

In the 40% Ni 60% Co alloy the dislocation diffusivity is given by

$$D_p^{\text{edge}} = 3.4 \text{ (cm}^2 \text{ sec}^{-1}\text{)} \exp -30.2 \text{ (Kcal mole}^{-1}\text{)}/RT \quad (8)$$

whereas in pure Ni

$$D_p^{\text{edge}} = 20 \text{ (cm}^2 \text{ sec}^{-1}\text{)} \exp -36.8 \text{ (Kcal mole}^{-1}\text{)}/RT \quad (9)$$

The ratio of the lattice diffusion activation energies in Ni and in the Ni-Co alloy is $(Q_l^{\text{Ni}}/Q_l^{\text{Ni-Co}}) = 1.04$ and in the temperature range of the present experiments $(D_l^{\text{Ni}}/D_l^{\text{Ni-Co}}) \sim 0.6$. The corresponding ratios for the edge dislocation diffusion are

$(Q_p^{\text{Ni}}/Q_p^{\text{Ni-Co}}) = 1.22$ and $(D_p^{\text{Ni}}/D_p^{\text{Ni-Co}}) \sim 0.4$. The ratio $(Q_p/Q_l)_{\text{edge}}$ is 0.55 in pure Ni and 0.47 in the Ni-Co alloy. As shown in

Table II the ratios Q_p/Q_l are greater than 0.5 for high stacking fault energy metals and less than 0.5 for low stacking fault energy metals. While r_o values are not available for the results on materials other than Ni and Ni-Co, the authors suggest that stacking faults may carry the tracer flux in other low stacking fault metals such as Ag or Au.

TABLE II

Metal	Path	Stacking Fault Energy ergs cm ⁻²	Q_p/Q_l	Ref.
Ag	Tilt Boundary	25	0.44	14
Ag	Twist Boundary	25		15
40%Ni 60%Co	Edge Dislocation	30	0.47	<i>this work</i>
Ni	Edge Dislocation	150	0.55	4
Cr	Grain Boundary	high	0.61	16
α Fe	Grain Boundary	~300	0.62	17
γ Fe	Grain Boundary	high	0.61	18
Ni	Tilt Boundary	150	0.40	19
Zn	Grain Boundary	high	0.62	20
Cd	Grain Boundary	high	0.70	21
Pb	Grain Boundary	high	0.65	22

The enhanced diffusion of tracer atoms along the stacking faults between partial dislocations places a severe constraint on the mechanisms proposed for diffusion along dislocations. In an f.c.c. structure, the nearest neighbor configuration of the atoms at the stacking fault is the same as in the lattice while the second nearest neighbor configuration is that of the h.c.p. structure. Self diffusion by an interstitial mechanism would hardly appear more likely along the stacking fault than in the lattice. Similarly mechanisms based on a ring mechanism or on a liquid like dislocation core⁽¹¹⁾ are not suitable. Diffusion by a jog mechanism is possible along dislocations split into partials. However, since the energy to form a double jog increases as the stacking fault width increases,⁽⁸⁾ the diffusion activation energy should increase when the dislocation splits into two partial dislocations. The experimental observations are directly counter to this requirement; D_p is greater and Q_p is smaller in Ni-Co as compared to Ni.

The experimental observations are qualitatively consistent with self diffusion along the dislocation by a vacancy exchange mechanism similar to that which describes diffusion in the lattice. In this case $Q_p = E_{f,lv}^p + E_{m,lv}^p$; the sum of the energies of formation and motion of vacancies along the dislocations.

The equilibrium concentration of vacancies near the dislocations is $C_{lv} = \exp -(E_{f,lv}^l - B_{lv})/kT$, where B_{lv} is the dislocation-

vacancy interaction energy. The binding energy B_{lv} results from interactions between the dilatation stress field of the dislocation and the volume change around the vacancy and between the stress field of the dislocation and the modulus change around the vacancy. Since these contributions to the binding energy vary as r^{-1} and r^{-2} (where r is the distance from the dislocation) an appreciable volume around the dislocation has an equilibrium vacancy concentration in excess of that which characterizes the lattice far from the dislocation. In addition to the excess vacancy concentration which results from the above interactions the stacking fault may have a high vacancy concentration due to "Suzuki segregation".⁽²³⁾ These effects all contribute towards a decrease in the value of $E_{f,lv}^p$ at the dislocation core and along the stacking fault.

The strains at the core of the dislocation can be expected to decrease $E_{m,lv}^p$ below the value in the lattice, $E_{m,lv}^l$ as can be seen using the formalism developed by Girifalco and Grimes.⁽²⁴⁾ While the magnitude of this effect is difficult to estimate, the decrease in the energy of motion should exhibit the r^{-1} dependence of the dislocation strain field. At the stacking fault, the activation barrier for the vacancy diffusion differs from that characteristic of the f.c.c. lattice as can be seen qualitatively in Figure 4. During the diffusion jump 1 → 2 (Figure 4a) in the

f.c.c. lattice the atom must pass through the saddle point configuration which has four nearest neighbors a, b, c, d. Along the stacking fault, a configuration characteristic of the h.c.p. lattice applies as shown in Figure 4b. It is proposed that this difference in the saddle point configuration can lead to a decrease in $E_{m,lv}^P$. Detailed calculations would be required to establish this effect. Although the magnitudes of $E_{m,lv}^P$ and $E_{F,lv}^P$ are difficult to estimate, the vacancy diffusion mechanism appears to be able to qualitatively describe the experimental results.

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Figure Captions

Figure 1a. Orientation of the crystal block

1b. The sliced wafer showing the arrangement of edge dislocations after anneal

1c. The position of the tracer plate on the wafer

1d. Cross section of the completed diffusion specimen

Figure 2. Normalized activity on the exit face vs. diffusion time

■ 617.8°C, ▲ 578.5°C, ● 547.8°C

Curve A - calculated curve using a Greens function source, Equation 4

Curve B - calculated curve using a time dependent boundary condition, Equation 6

Figure 3. Arrhenius plot of the measured edge dislocation diffusivities

Figure 4. Saddle point configurations for diffusion jumps

(a) f.c.c. lattice

(b) along a stacking fault

A

B

C

D

(a)

- Plane 1
- Plane 2
- Plane 3

(b)