Stacking Fault Energy Measurement from Diffusion

R. E. SMALLMAN AND P. S. DOBSON

The annealing kinetics of dislocation loops has been considered from climb theory based on a model involving vacancy diffusion as the rate controlling mechanism. The theory has been applied to fcc metals of high, intermediate, and low stacking fault energy to determine both the intrinsic and extrinsic fault energy using transmission electron microscopy. Reliable values are obtained from metals with $\gamma \gtrsim 70$ erg per sq cm but for low γ metals the rate controlling mechanism is shown to be one of jog nucleation and propagation rather than vacancy diffusion. The technique of loop annealing is also shown to be applicable to hcp metals such as zinc, magnesium, and cadmium, even when the foil surfaces act as vacancy sources. Results of loop shrinkage and loop growth are analyzed to provide fault energy values for those metals with hcp structure.

FOR a detailed understanding of the mechanical and physical behavior of metals it is necessary to have a precise knowledge of the absolute and relative energies of interfaces, such as surfaces, grain boundaries, and stacking faults. In recent years a number of techniques for evaluating the stacking fault energy, γ, of metals have been developed, based on electron microscope observations of defects containing stacking faults. Methods using measurements of extended dislocation configurations such as nodes and extrinsic-intrinsic fault pairs are confined to low fault energy materials, since it is only for these materials that the dislocation extension is large enough to measure. Results have therefore been restricted to silver of the pure metals, and alloys for which $\gamma \lesssim 20$ erg per sq cm. All the analyses so far used to relate the shape of a node to a value of γ have involved considerable approximations and the assumption, difficult to test, that the configuration is in equilibrium. Extrinsic-intrinsic fault pairs only give relative energy values and, like nodes, are subject to possible solute segregation effects. Another method based on the sizes of stacking fault tetrahedra and Frank loops observed in deformed materials has been applied by Loretto *et al.*[1] to metals of slightly higher fault energy, but more recent results obtained by Beeston *et al.*[2] show that the tetrahedra method gives values which are consistently lower than the true value and not in very good agreement with those obtained from node observations. None of the above methods is applicable either to high stacking fault energy materials or to a wide range of fault energies. However, a recent method involving the study of the kinetics of faulted loop annealing, initially pointed out by Edington and Smallman[3] and developed by Dobson and Smallman,[4] is particularly applicable to intermediate and high γ materials and potentially useful over a wide range of fault energies.

In the present paper the kinetics of dislocation loop annealing have been examined for high, intermediate, and low stacking fault energy metals to illustrate the general usefulness of quantitative rate measurements in obtaining intrinsic and extrinsic energies.

R. E. SMALLMAN is Feeney Professor of Metallurgy and Head of the Department of Physical Metallurgy and Science of Materials, The University of Birmingham, Birmingham, England. P. S. DOBSON is Lecturer, Department of Physical Metallurgy and Science of Materials, The University of Birmingham.

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1) ANNEALING ANALYSIS (PERFECT SINK CONDITIONS)

It has been convincingly demonstrated that for intermediate and high stacking fault energy materials at least, the rate of dislocation loop annealing is determined by the rate of diffusion of vacancies away from the loop rather than the emission of vacancies at the loop itself. In a thin metal foil the annealing rate of a loop will therefore depend on the vacancy concentration gradient developed between the loop and the surface of the foil.[5] Providing that the foil surface acts as a perfect vacancy sink the vacancy concentration at the surface is equal to the equilibrium vacancy concentration c_0 and that in equilibrium with the dislocation loop is given by

$$c_L = c_0 \exp(\Delta F/KT) \qquad [1]$$

where ΔF is the change in the free energy of the defect configuration per vacancy emitted ($= dE/dn$) at the temperature T.

For a single, intrinsically, faulted circular dislocation loop of radius r the total energy of the defect E is given by[6]

$$E = (3rG\mathbf{b}^2/4)\{\ln(8r/\mathbf{b}) - (9/8)\} + \pi r^2\gamma_i \qquad [2]$$

where G is the shear modulus, $\mathbf{b}$ the Burgers vector, and γ_i the intrinsic stacking fault energy. In the case of a large loop ($r > 500$Å) in a material of intermediate or high stacking fault energy ($r \gtrsim 60$ erg per sq cm) the term involving the dislocation line energy is negligible compared with the stacking fault energy term and thus ΔF is simply given by

$$\Delta F = \gamma_i B^2 \qquad [3]$$

where B^2 is the cross sectional area of a vacancy in the (111) plane. For large loops the diffusion geometry approximates to cylindrical diffusion and a solution of the time independent diffusion equation ($\Delta^2 c = 0$) gives for the annealing rate,

$$dr/dt = -[2\pi D/\mathbf{b}\ln(L/\mathbf{b})][\exp(\gamma_i B^2/KT) - 1] \qquad [4]$$

where $D = D_0 \exp(-U_D/KT)$ is the coefficient of self-diffusion and L is half the foil thickness. The anneal-

ing rate of a prismatic dislocation loop can be similarly determined, in this case ΔF is determined solely by the line energy,

$$dr/dt = -[2\pi D/\mathbf{b} \ln (L/\mathbf{b})](\alpha \mathbf{b}/r) \quad [5]$$

where the term containing the dislocation line energy has been approximated to $\alpha \mathbf{b}/r$. In principle Eq. [4] affords a direct determination of the stacking fault energy γ by direct substitution, but since $U_D \sim 1.3$ ev in aluminum compared with $\gamma B^2 \sim 0.1$ ev this method is unduly sensitive to small errors in U_D. This difficulty is eliminated by a comparative method[5] in which the annealing rate of a faulted loop is compared to that of a prismatic one at the same temperature.

By slightly rewriting Eq. [5] and combining it with Eq. [4] an expression for the stacking fault energy is obtained independent of the coefficient of self diffusion,

$$2\alpha \mathbf{b}[dr/dt]_F/[d(r^2)/dt]_P = \{\exp(\gamma B^2/KT) - 1\} \quad [6]$$

In addition to prismatic and single faulted dislocation loops, double faulted loops have also been observed in a number of quenched fcc metals.[3,7-9] Such a defect consists of a region of extrinsically faulted material formed by vacancy condensation on two alternate planes which is bounded by second loop of intrinsically faulted material. It is observed, see section 2, that on annealing, the intrinsic loop first shrinks until it meets the inner extrinsically faulted region which then shrinks as one extrinsically faulted loop. The rate of annealing of this extrinsic fault has been determined in a similar way to Eq. [4], the slight numerical differences arising from the fact that two vacancies need to be emitted to reduce the fault area by B^2, giving $\Delta F = \gamma_E B^2/2$ and twice as many vacancies have to diffuse to the surface to give the same decrease in radius as for a single fault, giving rise to the additional factor of two in the preexponential. Thus, the annealing rate for a double extrinsically faulted loop is given by

$$dr/dt = -[\pi D/\mathbf{b} \ln (L/\mathbf{b})][\exp(\gamma_E B^2/2KT) - 1] \quad [7]$$

and the extrinsic stacking fault energy may be determined directly by combining Eq. [7] with Eq. [5] or [6].

The treatment outlined above for the annealing of lattice defects under diffusion-controlled conditions is of general applicability. It has recently been extended[10] to the annealing behavior of small voids of radius r when the corresponding rate equation is given by

$$dr/dt = -(D/r)[\exp(2\Omega\gamma_S/rKT) - 1] \quad [8]$$

where γ_S is the surface energy and Ω the atomic volume.

2) MATERIALS WITH INTERMEDIATE AND HIGH STACKING FAULT ENERGIES

In metals with intermediate and high fault energies the climb behavior is dominated by the force due to the fault, and hence the annealing rate of a faulted loop can be measured to estimate the stacking fault energy using the diffusion-controlled rate equations. In principle the annealing behavior of other lattice defects can be followed to provide an estimate of the driving force from the process, provided the diffusion geometry is sufficiently well defined to develop a meaningful rate equation. To illustrate the applicability of the method a systematic study of the annealing kinetics of single loops, double loops, and voids has been made for the case of aluminum where the foil surface acts as an efficient vacancy sink. Fig. 1 shows typical radius-time curves for these three different lattice defects. In general it is not necessary to make measurements at small r values, see Eqs. [4], [7], and [8], but these are included in Fig. 1 for completeness, and to show that for loops, the dislocation line tension and diffusion geometry cause a deviation in the normal linear relationship.

To obtain accurate measurements it is necessary to carry out sequential-annealing treatments outside the microscope in a temperature-controlled oil bath; Fig. 2 shows Frank loops annealing at 140°C. Because

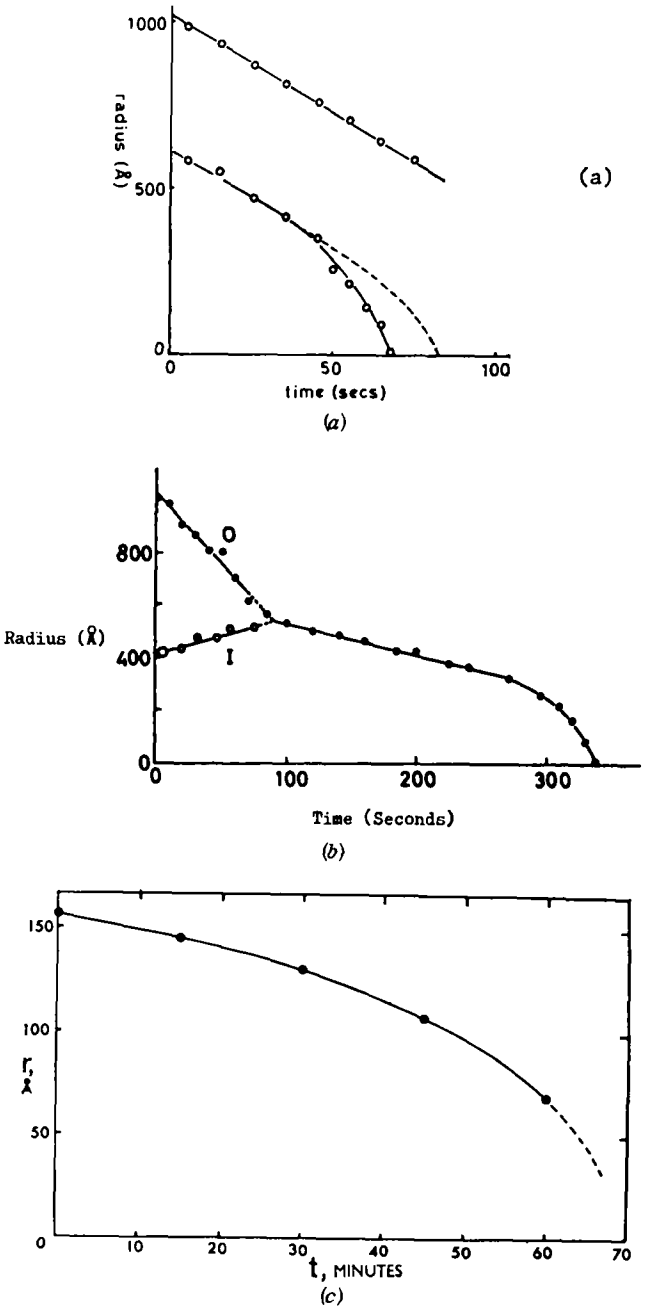

Fig. 1—Annealing curves for quenched aluminum containing (*a*) Frank loops at 175°C, (*b*) double loop at 170°C, and (*c*) void at 175°C.

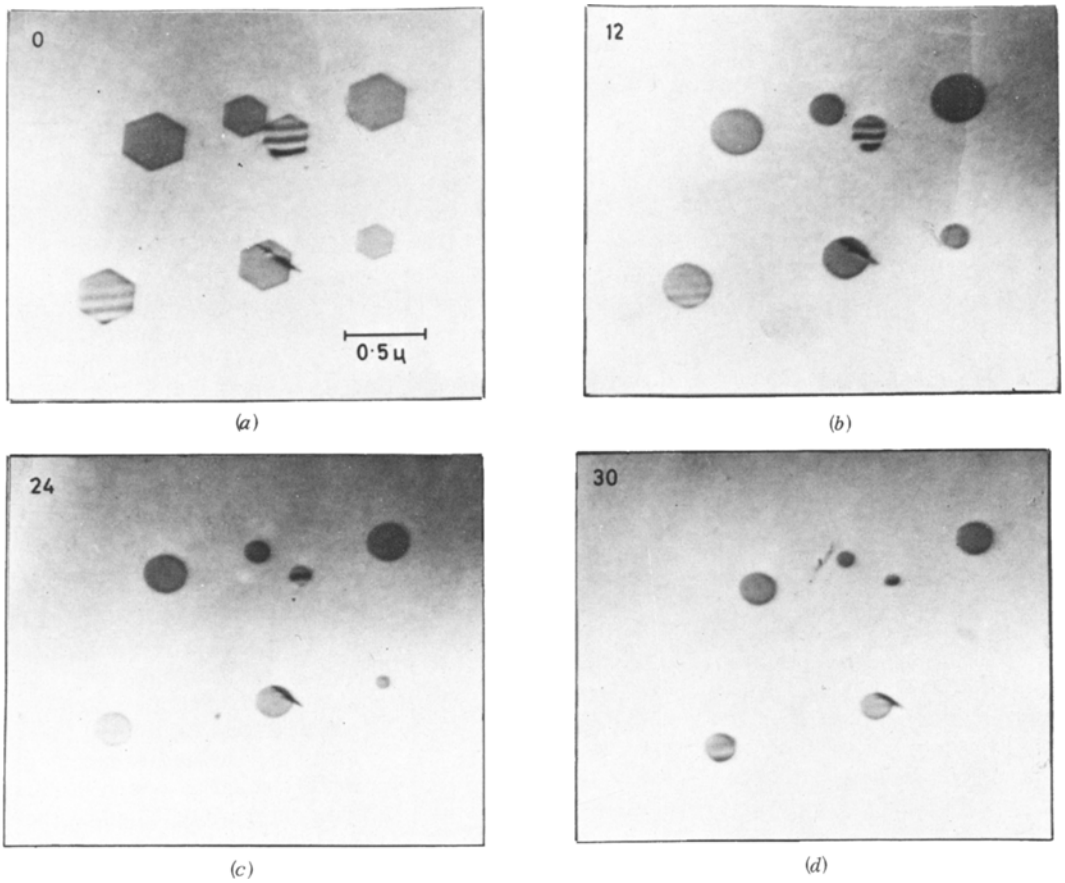

Fig. 2—Annealing of Frank loops in quenched aluminum at 140°C. (*a*) t = 0 min; (*b*) t = 12 min; (*c*) t = 24 min; (*d*) t = 30 min.

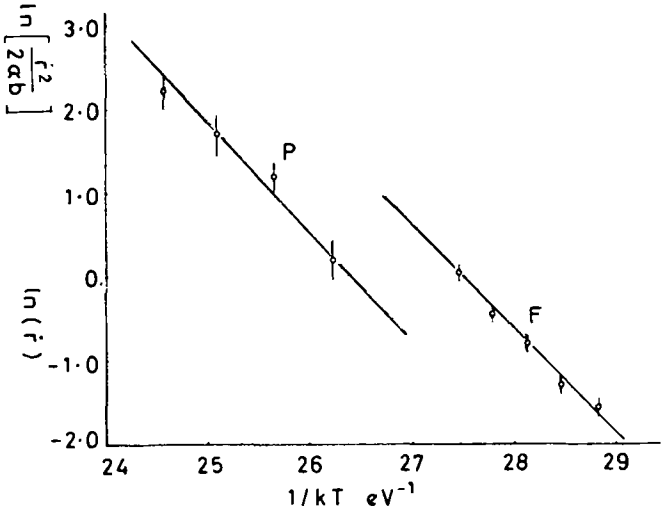

Fig. 3—A plot of the annealing rates for prismatic loops (P) and Frank loops (F) as a function of temperature.

faulted loops anneal much faster than prismatic loops it is not easy to measure a shrinkage rate for both faulted and prismatic loops at the same temperature. It is possible however, to anneal many loops at a series of temperatures and extrapolate the results to a common temperature. Prismatic loops were therefore annealed at 170°, 180°, 190°, and 200°C and faulted loops at 130°, 135°, 140°, 145°, and 150°C. The rates of shrinkage were obtained from linear plots of r vs t for faulted loops and r^2 vs t for prismatic loops, and in all cases the rates were reproducible within about 10 pct at the same temperature. The extrapolation to a common temperature (160°C) was made from a logarithmic plot of the rates, as shown in Fig. 3. Using the rates measured from the plot γ_1 was calculated from Eq. [6] and found to be 135 ± 20 erg per sq cm. To obtain the extrinsic fault energy the annealing behavior of double-faulted loops, which are known to contain an extrinsic fault, can be studied. In general, it is observed that on annealing, the outer intrinsically-faulted loop shrinks, and the inner loop grows slightly by vacancy absorption until they attain elastic equilibrium. Further annealing then results in the double loop shrinking at a reduced rate. Comparison of the rate of shrinkage of the inner double loop with the rate for an isolated Frank loop enables γ_E to be calculated. For aluminum a value of 180 erg per sq cm has been obtained. The value of γ_E and the ratio γ_E/γ_1 may also be obtained by comparing the rate of shrinkage of the outer loops with that for the inner loop when it becomes a double loop, provided the outer loop rate is measured when it is not in close proximity to the inner loop. In this case the outer (single-faulted) loop shrinks at a slightly faster rate than an isolated Frank loop because of the diffusion of vacancies to the inner loop and the increase in rate becomes more pronounced the closer the two loops approach each other.

From the annealing of voids, see Fig. 1(*c*), the surface energy γ_S may be obtained directly from a plot of $\ln[r(\dot{r}/D) + 1]$ vs i/r using the slope of this graph, provided D is known. Fig. 4 shows such a graph obtained by using the 175°C data of Fig. 1(*c*) together with a value of D = 4.5Å² per sec obtained from the annealing of prismatic loops; it can be seen that the

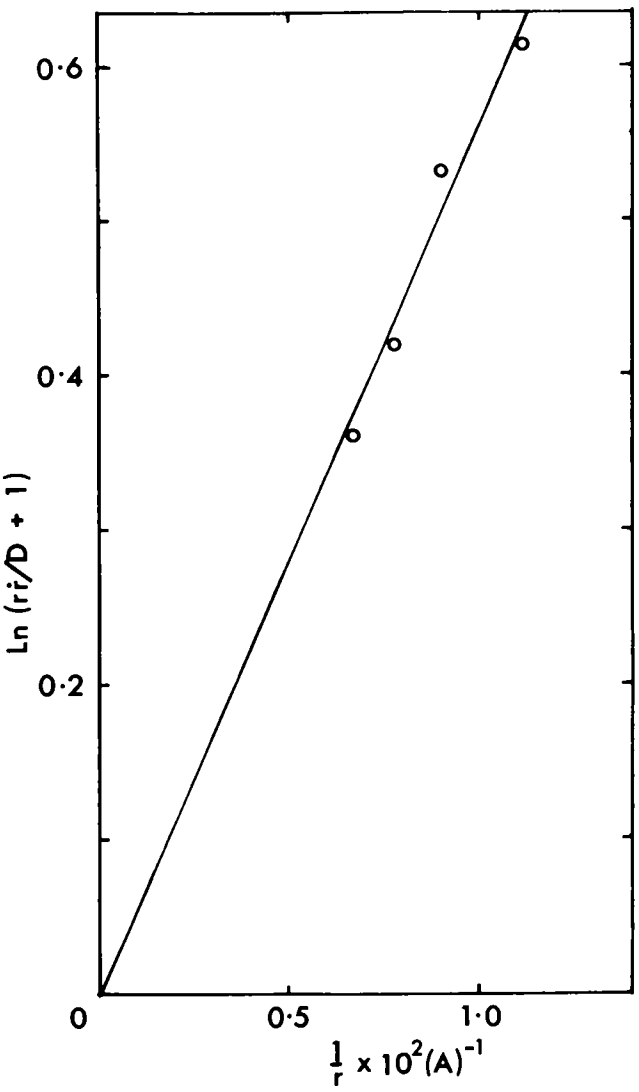

Fig. 4—Plot of $\ln [r(\dot{r}/D) + 1]$ vs $1/r$ for 175°C data given in Fig. 1(*c*).

data obey a reasonably good linear relationship. From the slope of the curve a value of γ_S = 1045 erg per sq cm is obtained. The results from similar analyses carried out on other data derived from annealing voids in the temperature range 150° to 200°C give an average value for γ_S of 1140 ± 200 erg per sq cm. In all cases, the appropriate value of D has been obtained from thin foil prismatic loop annealing studies, see Fig. 7 of the paper by Dobson, Goodhew, and Smallman.[5]

3) NONPERFECT SINK CONDITIONS

The analysis and results presented in sections 1 and 2 apply to the case where the foil surfaces act as perfect vacancy sinks (*i.e.*, $c_S = c_0$). However, in a number of materials, *e.g.* zinc, magnesium, Al-1 pct Mg, this condition is clearly not fulfilled, since similar thin foil annealing experiment show that loop growth rather than loop shrinkage occurs. It has now been demonstrated[11] that the excess surface vacancy concentration necessary to reverse the vacancy flux direction to give loop growth arises from the growth of a thin oxide film on the surface. In these metals oxidation takes place at the oxide-air interface by the transport of metal ions through the oxide, and this flow is maintained by the transfer of metal atoms into the oxide across the oxide-metal interface. Thus, vacancies are created during oxide growth in the surface regions of the foil and an enhanced equilibrium vacancy concentration is developed.

From the point of view of stacking fault energy determinations by the loop annealing technique, this enhanced and indeterminate surface vacancy concentration poses two problems. In the first place loops are rarely observed to shrink, and even in the rare cases where this is observed, presumably due to local cracks in the oxide at which vacancies can be annihilated, it cannot be assumed that the surface vacancy concentration is equal to c_0.

At its simplest, this effect gives rise to a further unknown in the annealing rate equation. In an initial study of loop annealing in zinc, Dobson and Smallman[4] attempted to calculate the surface vacancy concentration from a detailed analysis of faulted loop annealing curves. In principle the acceleration of the annealing rate at small radii, due to the increasing importance of the line energy time, gives additional experimental data from which a further unknown, *i.e.*, c_S, may be calculated. From studies on a number of shrinking loops, it was calculated that for the fastest shrinking loops the surface vacancy concentration was approximately equal to c_0 and the stacking fault energy was then determined[4,12] using the comparative method to be 140 erg per sq cm. It was assumed in this work that the annealing process was emission controlled whereas it is now known to be diffusion controlled. Under these diffusion conditions, the annealing rate at small radius is influenced not only by the line energy term but by the changeover from a cylindrical to a spherical diffusion geometry. However, a study of this geometrical effect on the annealing rate at small radii shows that the original calculations of c_S need to be regarded as an upper limit but since $c_S \simeq c_0$ for the fastest loops investigated, it can be concluded that the influence of the changeover in diffusion geometry had a negligible effect on the stacking fault energy determination.

A more sophisticated method of determining stacking fault energies in foils whose surfaces are acting as vacancy sources can be made by analyzing the growth kinetics of double faulted loops in the hexagonal metals. In this case the precipitation of a second layer of vacancies removes the stacking fault created by the precipitation of the first layer and the defect therefore consists[12] of an annulus of stacking fault surrounding a loop of perfect material, Fig. 5. Under growth conditions it is observed that stacking fault is created by vacancy absorption at the growing outer perimeter of the loop and is destroyed at the growing inner perfect loop. The perfect regions expands faster than the outer stacking fault since the addition of a vacancy to the inner loop decreases the energy of the defect by γB^2 whereas the addition of a vacancy to the outer loop increases the energy by the same amount. This effect is further enhanced as the two loops approach each other due to vacancy transfer from the outer to inner loops. Eventually the two loops coalesce to give a perfect prismatic loop which continues to grow under the vacancy supersaturation.

Thus three sets of independent data can be obtained

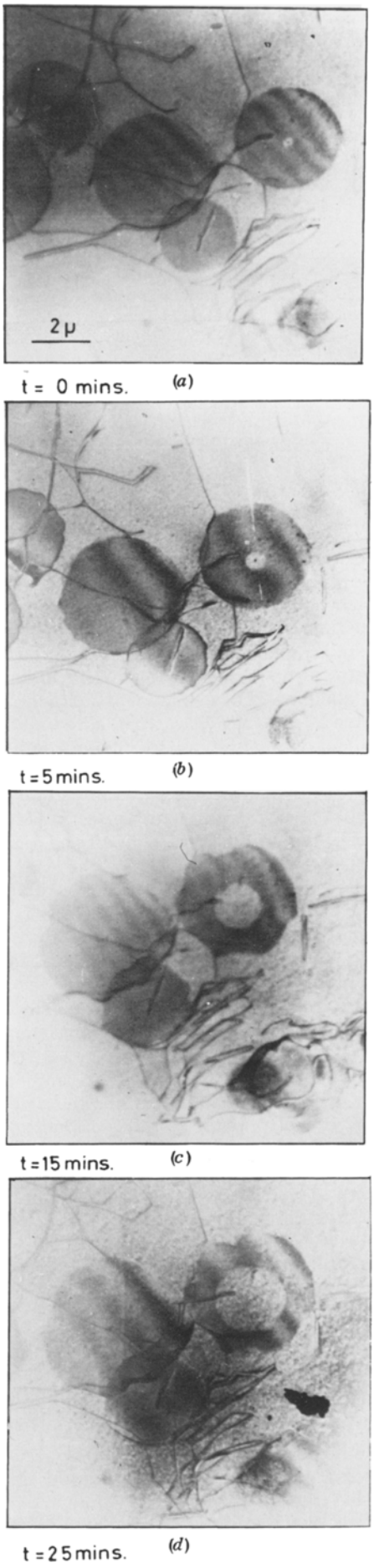

Fig. 5—The growth of single and double faulted loops in magnesium annealed at 180°C. (*a*) $t = 0$ min; (*b*) $t = 5$ min; (*c*) $t = 15$ min; (*d*) $t = 25$ min.

from annealing studies on a single double loop and this provides sufficient information to determine[12] the three unknowns, γ, c_S, and D. The rate of growth of the inner and outer loops are, respectively, given by:

$$\dot{r}_{\text{outer}} = [2\pi D/B \ln (L/\mathbf{b})][(c_S/c_0) - \exp(\gamma B^2/KT)] \quad [8]$$

$$\dot{r}_{\text{inner}} = [2\pi D/B \ln (L/\mathbf{b})][(c_S/c_0) - \exp(-\gamma B^2/KT)] \quad [9]$$

For a high value of γ, which is appropriate for magnesium, $\exp(-\gamma B^2/KT) \ll 1$ and hence Eq. [9] reduces to

$$\dot{r}_{\text{inner}} = -[2\pi D/B \ln (L/\mathbf{b})](c_S/c_0) \quad [10]$$

The growth rate of the final prismatic loop is given by

$$\dot{r}_{\text{prismatic}} = -[\pi D/B \ln (L/\mathbf{b})][(c_S/c_0) - 1] \quad [11]$$

The measured growth rates of the inner loop, Eq. [10], and of the prismatic loop, Eq. [11], allow the vacancy supersaturation c_S/c_0 and the preexponential constant $\pi D/B \ln (L/\mathbf{b})$ to be calculated. Then, by substituting these determined values into Eq. [8] the stacking fault energy was determined to be 125 ± 25 erg per sq cm for magnesium.

Recent work by Rozhanskii[13] *et al.* on the climb of loops in zinc has shown that the nucleation and growth of an oxide film and its related vacancy production can be stopped by chemical treatment of the foil surface. The method consists of removing any oxide film remaining after electropolishing by immersing the specimen in ammonia and then wetting the surface with a weak solution of mercurous nitrate. The surface was wetted by the mercury and after amalgamation it was found that dislocation loops contracted at rates corresponding to the surface vacancy concentration being equal to c_0. This technique has recently been successfully applied to the climb of dislocation loops in cadmium and the stacking fault energy determined to be 175 erg per sq cm. Further details of this work were presented in a separate paper at this Conference.

4) LOW STACKING-FAULT ENERGY METALS

Dislocation loops containing stacking faults have been observed in copper, gold, and silver but, unlike the loops found in aluminum are rarely hexagonal in shape. Instead, many polygonal-shaped loops with edges along ⟨110⟩ containing acute and obtuse corners are formed, Fig. 6 shows typical loops in copper. During annealing at temperatures[14] above 470°C the loops appear to shrink reasonably uniformly consistent with a stacking fault energy of about 70 erg per sq cm. Annealing below 470°C, however, the loops shrink nonuniformly with jog nucleation and propagation somewhat easier at obtuse-angled corners. The loops developed are therefore predominantly acute-angled, presumably due to the fact that the dissociation of the Frank dislocation to form a stair-rod and a Shockley dislocation on an intersecting (111) plane, gives rise to stable configuration involving three stair-rods at acute angles but not at obtuse. At obtuse corners the dislocations are constricted favoring jog nucleation, but to allow loop shrinkage the jogs must subsequently run along the loop sides, which requires further dislocation constriction. Evidently this is provided by thermal activation at temperatures above 470°C but not below for

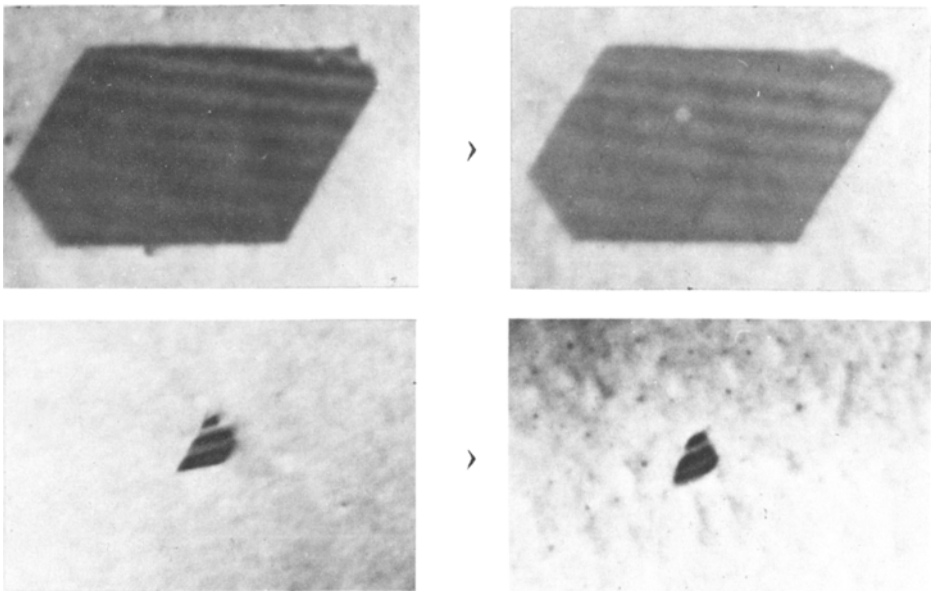

Fig. 6—Dislocation loops in quenched copper developing acute-angled corners during annealing at 447°C. Magnification 70,500 times.

the case of copper with $\gamma \sim 70$ erg per sq cm. This difficulty of jog propagation is also reflected in the formation of irregular-shaped loops containing large superjogs.

Faulted dislocation loops have also been observed in copper after proton irradiation.[15] These loops, which are interstitial in nature and contain extrinsic stacking fault, shrink uniformly by absorbing vacancies generated mainly at the free surface during annealing in the temperature range 370° to 420°C. The value of the stacking fault energy determined was 92 erg per sq cm which, taken together with the results discussed above, gives a ratio $\gamma_E/\gamma_I \simeq 1.3$. It would appear from these experiments with copper that jog propagation becomes difficult for materials with $\gamma \sim 70$ erg per sq cm and hence climb is considerably restricted. The annealing of loops thus changes from diffusion-controlled to emission-controlled conditions and the analysis outlined in section 1 is generally unapplicable to materials with $\gamma \lesssim 70$ erg per sq cm.

The problems of jog nucleation and propagation are even more acute in the pure metals gold ($\gamma \sim 45$ erg per sq cm) and silver ($\gamma \sim 20$ erg per sq cm). The faulted defects observed in quenched gold and silver are similar to those shown in Fig. 6, heavily jogged and angular in appearance. In gold the annealing is irregular at temperatures ~350°C where self-diffusion is rapid and where voids anneal quite readily. The annealing behavior does, however, become more regular at higher temperatures, as observed for copper. In silver, the loops are often found to be stable up to temperatures in excess of 600°C.

5) DISCUSSION

The shrinkage of lattice defects, such as dislocation loops and voids, in the metal foils during isothermal annealing is controlled by vacancy diffusion. A comparison of the measured annealing rate with that predicted by diffusion-controlled rate equation allows a determination of the diffusion coefficient D or interface energy γ. The rate equation generally contains two material constants D and γ one of which may be determined by substitution of a reasonable value for the other. It is unfortunate that the self-diffusion coefficient of most materials is not known with sufficient accuracy for use in such kinetics studies. Nevertheless, it has been demonstrated in this work that D may be eliminated by using a 'comparative' annealing technique and accurate stacking fault energy and surface energy values obtained. Alternatively, a reliable value of γ may be used to determine the self-diffusion coefficient D, as carried out recently by Volin and Balluffi[16] for aluminum. The values obtained of $\gamma_I = 135 \pm 20$ erg per sq cm, $\gamma_E = 180 \pm 20$ erg per sq cm, and $\gamma_S = 1140 \pm 200$ erg per sq cm complete, with $\gamma_{g.b} = 340$ erg per cm for the grain boundary energy and $\gamma_T = 71$ erg per sq cm for the twin boundary energy obtained previously,[17] a consistent set of surface and interfacial energy values for aluminum. The annealing of single-faulted loops in aluminum has also been similarly measured by Tartour and Washburn[18] and a value for γ_i obtained in good agreement with that which Dobson, Goodhew, and Smallman reported, thus confirming the reliability and reproducibility of the technique.

In the hcp metals zinc, cadmium, magnesium, and so forth, it has not been possible to determine the stacking fault energy directly, prior to the introduction of the present vacancy diffusion technique. Dislocation nodes have not been observed in the common hcp metals (although they are measureable in graphite and other layer structures), the texture technique[19] is not applicable since there is no texture stable to further deformation, and other indirect techniques have often led to erroneous estimates. The methods based on diffusion described in this paper involving either the shrinkage of single-faulted loops or the growth of double-faulted loops should enable the influence of electronic factors, alloying elements, c/a ratio, and so forth, on stacking-fault energy (or surface energy) to be determined in future work. It must be remembered however, that the value of γ obtained is for a fault with one next-nearest neighbor violation in the stacking sequence and the fault produced by dissociation in the based plane of the slip dislocation with $\mathbf{b} = a/3\langle 11\bar{2}0\rangle$ has two next-nearest neighbor violations

and a correspondingly higher energy.

A disadvantage of the node and loop-annealing methods is that they are tedious to apply and do not lend themselves readily to rapid surveys across alloy phase fields to study the influence of electronic structure on $\gamma/\mu b$. For this purpose semiquantitative, empirical methods offer great advantages. Possibly the simplest of these is the texture method,[19] which relies on the dependence of rolling texture in fcc metals on $\gamma/\mu b$, as discussed in the paper by Dillamore, p. 2463. A comparison of the various methods for determining γ has been made[2] using Ni-Co alloys where, as shown in Fig. 7, γ varies from the high value of pure nickel to a few erg per sq cm at the phase boundary. The results obtained by the tetrahedra method fall appreciably below the values obtained by the other two methods. Discrepancies in the same sense are found in the results for silver, copper, and gold and for stainless steel and probably arise from the particular mechanism of tetrahedra formation assumed in the analysis. The good agreement between the variation of $\gamma/\mu b$ in the Ni-Co system obtained by the node and texture method provides confirmation of the value for aluminum of 135 erg per sq cm obtained using the loop-annealing analysis, since this value was used as a calibration point for the texture method. It is essential in using the texture technique to have a complete calibration over the full range of γ values. At present, the diffusion analysis outlined in this work is the only direct method available for high γ materials.

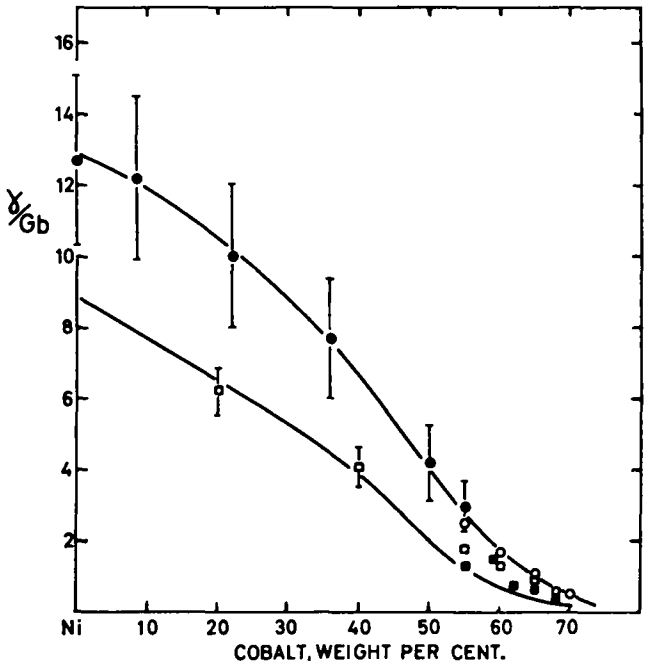

Fig. 7—The variation of stacking fault energy in the Ni-Co system determined from textures ●, nodes ○, and tetrahedra □ measurements.

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