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THE MEASUREMENT OF STACKING FAULT ENERGY

by

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Résumé. — On examine et critique deux méthodes récentes de mesure de l'énergie des défauts d'empilement γ . La première, basée sur une analyse de la cinétique de montée des boucles de défauts, est discutée spécialement dans le cas des métaux f. c. c. et h. c. La seconde, qui utilise la mesure d'un simple paramètre d'orientation privilégié a été comparée à d'autres méthodes plus usuelles d'estimation de γ , et à des mesures récentes de la variation de l'énergie des défauts d'empilement avec la structure électronique des bandes d , qui sont discutées.

Abstract. — Two recent methods for the measurement of the stacking fault energy γ have been critically examined.

The first method based on an analysis of the climb kinetics of faulted loops has been discussed with special reference to f. c. c. and c. p. h. metals. The second technique, which depends on the measurement of a simple preferred orientation parameter, has been compared with other more conventional methods of estimating γ and some recent measurements of the variation of stacking fault energy with electronic d -band structure are discussed.

Introduction. — In the past fifteen years about a dozen methods of measuring the stacking fault energy of a metal γ have been proposed and used. These include the (i) ribbon width, (ii) barrier width, (iii) dislocation node, (iv) fault tetrahedra, (v) loop annealing, (vi) tilt boundary, (vii) low-temperature creep, (viii) τ_{111} , (ix) stacking fault frequency, (x) twin boundary energy, (xi) rolling texture and (xii) stored-energy methods. The most reliable of these is the dislocation node radius method but unfortunately it is only applicable for values of $\gamma/Gb \leq 2 \times 10^{-3}$, where G is the shear modulus and b the Burgers vector of the partial dislocation. Most of the other methods are either theoretically speculative or have serious practical limitations. The most promising alternative to node radius measurements is the fault tetrahedron method — provided the tetrahedra are formed by deformation — but it has not yet been shown in this case that a distribution of suitable jog lengths exists. Even this technique is limited to values of $\gamma/Gb \leq 7 \times 10^{-3}$ and there has been no analytical method which is applicable at high values of γ/Gb .

In this paper we wish to describe two methods, developed at Birmingham, which are most applicable in the intermediate and high γ/Gb range and therefore are complementary to the well established node tech-

nique. The first method is based on analysis of the climb kinetics of faulted dislocation loops, and so far has only been used to estimate γ for Al, Zn and Mg. The second technique depends on the measurement of a simple preferred orientation parameter and even though empirical it is nevertheless useful over a large range of γ/Gb and particularly valuable for comparative purposes.

2. The Loop Annealing Method.

a) THE ANALYSIS. — Dislocation loops containing stacking faults of either an intrinsic or extrinsic nature may be obtained [1] in both quenching and irradiation experiments. On heating a metal foil containing such loops, vacancies are emitted and the loops shrink by climb. Since the driving force for the climb process arises principally from the stacking fault it has recently been pointed out by Edington and Smallman [1] that in principle, the stacking fault energy of the metal γ can be determined from measurements of the climb rate of faulted dislocation loops. The technique is particularly attractive since no direct method is available for the determination of stacking fault energies greater than about 30 ergs/cm², the upper limit of the node method [2].

In experiments with quenched aluminium it was demonstrated that faulted loops annealed out at a much faster rate than prismatic loops and that the increased rate could be attributed to the extra driving force caused by the stacking fault. A mechanism proposed by Friedel [3] and first applied by Silcox and Whelan [4] to the annealing of prismatic dislocation loops in aluminium allows the rate of shrinkage of a loop to be written in terms of the velocity of jogs running along the loop. Jogs can move in a direction either to shrink the loop, under the elastic driving force F_e or to expand the loop under a chemical driving force F_s due to vacancy supersaturation c/c_0 . The net jog velocity is given by

$$v_j = \frac{zvb}{\sin \phi} \exp\left(\frac{-U_D}{kT}\right) \left\{ \exp \frac{F_e B^2}{kT} - \exp \frac{F_s B^2}{kT} \right\} \quad (1)$$

where z is a co-ordination number, v the atomic frequency factor, b the Burgers vector and ψ the angle it makes with the loop, U_D the energy of self diffusion and B^2 the cross-sectional area of a vacancy in the (111) plane. The rate of shrinkage given by this emission-controlled process is

$$\frac{dr}{dt} = -C_j v_j$$

where C_j is the concentration of jogs, which may be taken to have an average value of one-half, and because the annealing experiments are carried out with thin foils, for which c/c_0 may in general be assumed to be unity, the climb rate reduces to

$$\frac{dr}{dt} = -\frac{zvb}{2} \exp\left(\frac{-U_D}{kT}\right) \left\{ \exp \frac{F_e B^2}{kT} - 1 \right\}. \quad (2)$$

The driving force for climb F_e consists of two components, arising from the line tension of the dislocation and the stacking fault energy, and is given by

$$F_e = \frac{Gb^2}{4\pi(1-\sigma)} \frac{1}{r} \ln \left[\frac{r}{b} \right] + \gamma \quad (3)$$

where G is the shear modulus and σ Poisson's ratio. For large loop sizes the driving force due to the fault far outweighs that due to the line tension and with the approximation $F_e \simeq \gamma$ the shrinkage rate

$$\frac{dr}{dt} = C \left\{ \exp \frac{\gamma B^2}{kT} - 1 \right\} \quad (4)$$

indicates a linear relationship between r and t from the slope of which γ can be evaluated by inserting the appropriate values of z , b , U_D etc., into the constant C ,

Figure 1 shows the results obtained by Edington and Smallman in continuous observations of the climb process using a hot stage and a cine camera. From the slope of the r versus t plot a value of

$$\gamma \simeq 280 \pm 50 \text{ ergs/cm}^2$$

was obtained, the error limits being attributed to an uncertainty of $\pm 10^\circ\text{C}$ in the annealing temperature.

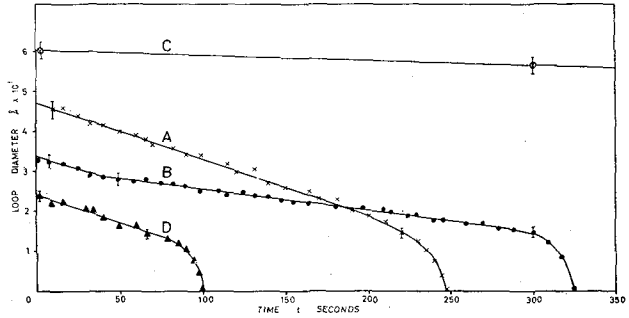


FIG. 1. — Annealing curves for dislocation loops in quenched aluminium (after Edington and Smallman). Diameter versus time plot for the four loops A (Frank sessile), B (double-faulted) C (prismatic) and D (faulted loop within a prismatic).

To overcome the temperature error it is preferable to anneal for specified periods outside the microscope in a controlled furnace using plates rather than cine film to record the image. However, the principle uncertainty in the method, as pointed out by Saada [6] arises from the uncertainty in the value taken for U_D , the activation energy for self diffusion, since $\gamma B^2 \simeq 0.1 \text{ eV}$ and an order of magnitude less than U_D . This error can be eliminated by a comparative annealing experiment in which the rates of annealing of faulted Frank loops and prismatic loops are compared. Retaining the assumption of zero vacancy supersaturation the shrinkage rate of a prismatic loop can be written in a form similar to equation (2) in which the driving force for shrinkage of the loop F_e is given by the first term in equation (3). A difficulty exists however in attempting to compare shrinkage rates in that the shape of the r vs t curve is dissimilar for the two loops and at large loop sizes the rate of shrinkage of a prismatic loop is very slow compared with that for a Frank loop. Dobson and Smallman [5a] have proposed that these difficulties can be avoided by using the integrated form of equation (2) and determining the times taken for loops of initial radius r to anneal out. This requires use of the entire annealing data and the force aiding shrinkage of a Frank loop due to line

tension must be included in the elastic driving force. Silcox and Whelan [4] showed that the term

$$\exp \frac{Gb^2}{4\pi(1-\sigma)} \frac{1}{r} \ln \left[\frac{r}{b} \right] \frac{b^2}{kT}$$

can be approximated by $(1 + \alpha b/r)$ where α is a constant slightly dependent on temperature. For a Frank loop the rate of annealing is then given by

$$\frac{dr}{dt} = -\frac{zvb}{2} \exp\left(\frac{-U_D}{kT}\right) \left\{ \exp \frac{\gamma B^2}{kT} \left(\frac{\alpha b}{r} + 1 \right) - 1 \right\} \quad (5)$$

and for γ values greater than about 100 erg/cm² the final one in the expression may be neglected and the rate equation integrated to give an expression for annealing time as a function of loop radius,

$$t = \frac{2}{zvb \exp\left(\frac{-U_D}{kT}\right) \exp\left(\frac{\gamma B^2}{kT}\right)} \left\{ r - \alpha b \ln \left(1 + \frac{r}{\alpha b} \right) \right\} \quad (6)$$

and γ may be obtained from the slope of a plot of t versus $\{r - \alpha b \ln(1 + r/\alpha b)\}$. The result will still be subject to the inaccuracies in U_D , but a similar procedure for prismatic loops gives

$$t = \frac{2 \sin \psi}{zvb_p \exp\left(\frac{-U_D}{kT}\right)} \left\{ \frac{r^2}{2 \alpha_p b_p} \right\} \quad (7)$$

and the slope of a graph of t versus $r^2/2 \alpha_p b_p$ is then $2 \sin \psi_p / zvb \exp(-U_D/kT)$. It therefore follows that the ratio of the slopes of the annealing plots for the faulted and prismatic loops is

$$\left(\frac{b_F}{b_p} \right) \sin \psi_p \exp\left(\frac{\gamma B^2}{kT}\right)$$

from which γ can be calculated without knowledge of z , v and U_D .

In principle it should also be possible to obtain the advantages of the comparative annealing method from observations on faulted loops alone, by comparing the annealing rates at different radii (i. e. for different contributions from the line tension) when the term containing U_D cancels out as before. Saada [6] has suggested this method and obtained the following expression

$$\exp \frac{\gamma B^2}{kT} = \frac{\rho - 1}{\rho - 1 - (\alpha b/r)} \quad (8)$$

where $\rho = (dr/dt)_r / (dr/dt)_\infty$. However, in the case of a metal with a high stacking fault energy, such as aluminium, the method is insensitive because the driving force caused by the stacking fault is so much greater than that due to the line tension.

For a faulted loop $r = 500 \text{ \AA}$ and $\gamma = 200 \text{ erg/cm}^2$, $\exp(\gamma B^2/kT)$ is 30 and $\alpha b/r$ about 0.33 and consequently from equation (8) ρ is found to be 1.34. However, if the error on the measurement of ρ is 1 % then for $\rho = 1.35$ this means that $\exp[\gamma B^2/kT]$ is 17 whereas for $\rho = 1.33$ $\exp[\gamma B^2/kT]$ is infinity. It is therefore apparent that for high γ -values the Saada method is less reliable than direct substitution of the values for the constants in single annealing rate equation.

An improved method based on a single-annealing analysis would be to compare the linear rate observed at large r , i. e. $(dr/dt)_L$, with the time taken for the loop to anneal out completely. Thus from the general rate equation given by equation (5) and substituting

$$1 = (1 - x) \exp(\gamma B^2/kT)$$

for mathematical convenience, then

$$\frac{dr}{dt} = \frac{zvb}{2} \exp\left(\frac{-U_D}{kT}\right) \left\{ \exp\left(\frac{\gamma B^2}{kT}\right) \left(\frac{\alpha b}{r} + x \right) \right\} \quad (9)$$

which can be integrated to give

$$t = \frac{2}{zvb \exp\left(\frac{-U_D}{kT}\right) \exp\left(\frac{\gamma B^2}{kT}\right)} \times \frac{1}{x} \left\{ r - \frac{\alpha b}{x} \ln \left(1 + \frac{rx}{\alpha b} \right) \right\} \quad (10)$$

or

$$t = \frac{1}{(dr/dt)_L} \left\{ r - \frac{\alpha b}{x} \ln \left(1 + \frac{rx}{\alpha b} \right) \right\}. \quad (11)$$

Thus, the time of annealing and the rate of annealing are related by

$$r - (dr/dt)_L t = \left(\frac{\alpha b}{x} \right) \ln \left\{ 1 + \left(\frac{rx}{\alpha b} \right) \right\} \quad (12)$$

from which x can be calculated and hence γ , since $\exp(\gamma B^2/kT) = 1/(1 - x)$. This method, like Saada's, is not sufficiently sensitive when $x \sim 1$ i. e. for metals with high γ -values, but for materials with low γ values this slope-intercept method should prove more reliable than a direct comparison of slopes.

The Friedel mechanism of loop annealing is based on the assumption of an emission controlled rate process. Balluffi and Seidman [7] have recently pos-

tulated however that the process may be diffusion controlled with a rate equation given by

$$\frac{dr}{dt} = -\frac{4\pi vb}{\ln(t/b)} \exp\left(\frac{-U_D}{kT}\right) \left\{ \exp\frac{F_c B^2}{kT} - 1 \right\} \quad (13)$$

where $2t$ is the foil thickness. This relationship is similar to that derived for the emission-controlled process but gives a rate about a quarter of that for the emission controlled process for typical foil thickness. Clearly the similarity of the two expressions (2) and (13) makes it difficult to distinguish experimentally between the two alternative rate-controlling processes. This postulate should not affect an analysis based on a comparative method, but does emphasize further the limitations of a single-annealing analysis.

b) APPLICATIONS. — The loop annealing method can be applied to determine both intrinsic and extrinsic fault energies, since both types of fault can be obtained either by quenching or irradiation experiments. The main limitation in applying the analysis to f. c. c. metals with low γ -values probably lies in the problems associated with vacancy emission from dissociated dislocation loops, where the initially formed Frank has dissociated into Shockley and Stair-rod dislocations. This limitation does not apply in hexagonal metals, where a $c/2$ [0001] dislocation has no dissociation out of the basal plane.

Apart from the theoretical difficulties associated with the climb kinetic method, practical difficulties have been found in applying the technique to certain metals and alloys. With zinc and magnesium, for example, it is observed [5a], [8] that some dislocation loops grow rather than shrink on heating. Dobson and Smallman [5b] have attributed this effect to the production of vacancies as a result of oxide-film growth, while Harris and Master [8] favour an explanation based on the presence of the oxide film preventing the free surface from acting as an ideal vacancy sink. Other loops are observed to shrink in the normal manner and it is very probable that these lie in the immediate vicinity of breaks in the oxide layer. However, in applying the climb kinetic analysis to the annealing studies in thin foils it is assumed that the foil surface is an ideal sink and as a consequence the vacancy concentration near the loop remains at the equilibrium value. In metals with surface oxide films this condition is not necessarily true and it is therefore important to determine the supersaturation term in the general rate equation

$$\frac{dr}{dt} = \frac{zv b}{2} \exp\left(\frac{-U_D}{kT}\right) \left\{ \exp\frac{\gamma B^2}{kT} \left(\frac{\alpha b}{r} + 1\right) - \frac{C}{C_0} \right\}. \quad (14)$$

This may be achieved in a similar way to that used in deriving equation (12), but in this case putting

$$\frac{C}{C_0} = (1 - x) \exp\left(\frac{\gamma B^2}{kT}\right). \quad (15)$$

Figure 2 shows the annealing behaviour of two different shrinking loops in zinc. Loop B is shrinking

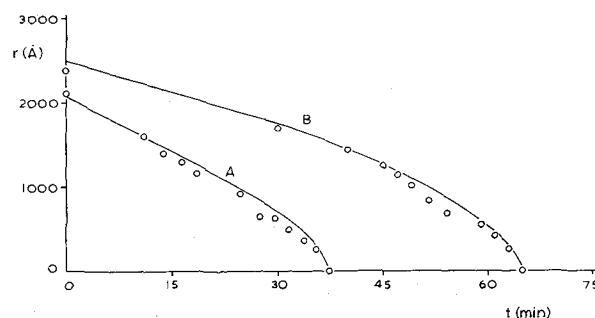


FIG. 2. — Radius versus time plots for two different dislocation loops in quenched zinc (after Dobson and Smallman).

at a much slower rate than loop A because it does so in the presence of a high vacancy supersaturation, i. e. $x = 0.25$ compared to $x = 0.75$ for loop A. In figure 3 the annealing data for loop A is plotted as the function $1/x \left\{ r - \alpha b/x \ln(1 + rx/\alpha b) \right\}$ against time and a value of $\gamma = 290 \text{ erg/cm}^2$ derived from the slope.

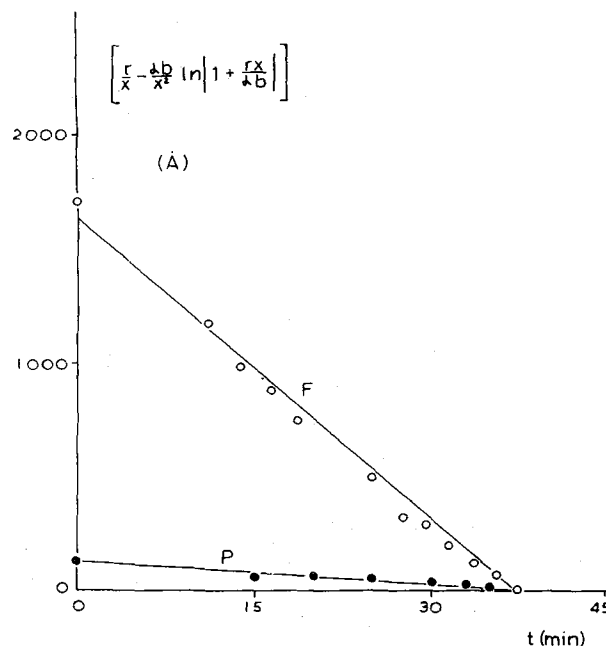


FIG. 3. — Annealing data plotted as a linear function of time for faulted loop (F) and prismatic loop (P) (after Dobson and Smallman).

Curve B shows the appropriate annealing data for a prismatic loop, and a comparison of the slopes of curves A and B, gives a more reliable value for γ of 220 erg/cm². The difference between these two values for γ indicates the magnitude of the inaccuracies in a single-annealing analysis.

In climb kinetic studies of quenched aluminium-magnesium alloys while faulted loops shrink as expected, similar problems of loop growth have been experienced [9] for prismatic loops. In this case the vacancies contributing to loop growth are released from solute atom traps on heating and give rise to a supersaturation. It is therefore necessary to take account of the supersaturation in estimating γ for these alloys.

3. The Texture Method.

This is an empirical method which allows the rapid determination of stacking fault energy at intermediate to high values of γ/Gb . It relies on the observations made by a number of workers [10, 11] that the deformation texture developed in f. c. c. metals on rolling depends on the composition and on the rolling temperature. Brown [12] suggested that the differences in texture between different metals and alloys could be attributed to the variation in the ease of cross-slip and it was later shown [13], using a simple model, that the differences could be described mechanistically by considering the grain rotations in texture development to be due to normal slip together with cross-slip, whose rate of incidence would depend on the amount of thermal activation and the stacking fault energy. In terms of polycrystalline plasticity requirements extremes of preferred orientation relate to the conditions where the Von Mises criterion is satisfied by slip on five independent slip systems or where continuity requirements are met by having three non-coplanar non-perpendicular slip directions together with easy cross-slip. It must be remembered however, that at very low fault energies the correlation between preferred orientation and ease of cross-slip breaks down and this has been shown [14, 15], to be due to twinning and stacking fault formation contributing to the deformation processes.

Extreme types of deformation texture, corresponding to 70/30 brass and to aluminium respectively after 96 % reduction are shown in figure 4. It can be seen from these {111} pole figures that the intensity peak close to the rolling directions is similar in the two textures, while the peak in the transverse direction differs considerably between the two. The ratio of these two intensities thus serves as a sensitive measure of the type of preferred orientation and can be used to

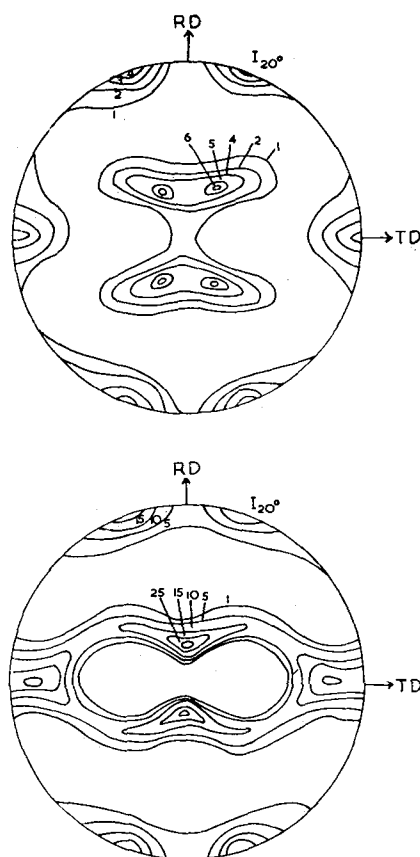


FIG. 4. — {111} Pole-figure of (a) 70-30 brass and (b) pure copper. Intensities in arbitrary units.

follow the transformation, as a function of temperature or composition, from one extreme to the other. In view of the dependence of preferred orientation on the ease of cross-slip, this ratio might be considered as an index of the extent of the contribution of cross-slip to the total strain.

Correlating the incidence of cross-slip with single crystal stress-strain behaviour we can say that a material with a high value of τ_{111} will cross-slip less readily than a material with a low τ_{111} so that the total contribution of cross-slip to a fixed total strain will be lower the higher the value of τ_{111} . This reduces the correlation between texture and stacking fault energy to the same terms as the relation between τ_{111} and fault energy, for which it has been shown that

$$\ln \frac{\tau_{111}(T)}{G(T)} = \ln \left\{ \frac{2}{n} \left(0.056 - \frac{\gamma}{Gb} \right) \right\} - \frac{kT(1 + n/900)}{0.352 Gb^3} (1 + 180 \gamma/Gb) \ln \frac{\dot{\epsilon}}{\dot{\epsilon}_0}$$

where the parameters have their usual significance. Thus comparing the deformation of metals of different fault energy at equal values of kT/Gb^3 and $\dot{\epsilon}$ it only requires the eminently reasonable assumption that n and $\dot{\epsilon}_0$ are smooth functions of γ/Gb to allow the intensity parameter defining the texture to be correlated with γ/Gb . This is most readily done by calibrating the texture variation against values of γ/Gb obtained by the best available methods and in the calibration the node value of silver, the extrapolated node value for copper and the Frank loop annealing value for aluminium were used. This calibration allowed values to be obtained for most of the f. c. c. pure metals [16] and a check on the method is afforded by the fact that the current best value for gold was first obtained by the texture method [14] and has since been confirmed by the node-extrapolation technique and by the fault tetrahedron method [17].

A further check on the method is afforded by measurements on the nickel-cobalt system, where the results from the texture method [19] and from node [20] and tetrahedra measurements [18, 19] lie very well on the same curve, as shown in figure 5. One of the principal sources of difference between the results reported by

different workers lies in the value chosen for the shear modulus G . Jossang et al use the Voigt average, i. e.

$$C_{44} - \frac{1}{5}(2C_{44} + C_{12} + C_{11}),$$

whereas both Seeger and co-workers and Howie and Swann use the shear modulus appropriate for slip on $\{111\}$ planes, i. e. $\frac{1}{3}(C_{44} + C_{11} - C_{12})$. This is the correct value, but in theoretical analyses the use of this value requires that the analysis be rigorously based on anisotropic elasticity. Since single crystal elastic constants are not available for all metals, Dillamore et al used the rigidity modulus G obtained from polycrystalline samples, which is rather larger than $\frac{1}{3}(C_{44} - C_{11} + C_{12})$ and consequently introduces significant error in the value of γ for high fault energy metals. For this reason the value of γ quoted by Dillamore et al should be regarded as comparative for high fault energy metals but within the error limits of $\pm 20\%$ typical for most techniques at values of $\gamma < 100$ ergs/cm². Recalibrating the data of Dillamore et al using $G = \frac{1}{3}(C_{44} + C_{11} - C_{12})$ for Cu, Ag and Al gives the values listed in table 1 for several pure metals.

TABLE I

Comparison of Stacking Fault Energy Values
Obtained by Various Methods

Metal	Node (*)	Tetrahedra	$\tau_3(T)$	$\tau_3(\dot{\epsilon})$	Texture (**)
Cu	85	78	163	50	85
Au	48	56	10-30		55
Ag	21	21	29-43	16-40	21
Ni	345	> 375	300		450
Pd					170
Pt					95
Ir				300	
Rh					330
Ce					> 15
Yb					15
Th					150
Al		> 175			
Pb				25	

The texture method is ideal to evaluate the variation of fault energy with composition for alloy systems.

(*) Corrected as suggested by Brown, Cu and Au and Ni are extrapolated values.

(**) Fitted to $\gamma_{Cu} = 85$, $\gamma_{Ag} = 21$ and $\gamma_{Al} = 250$.

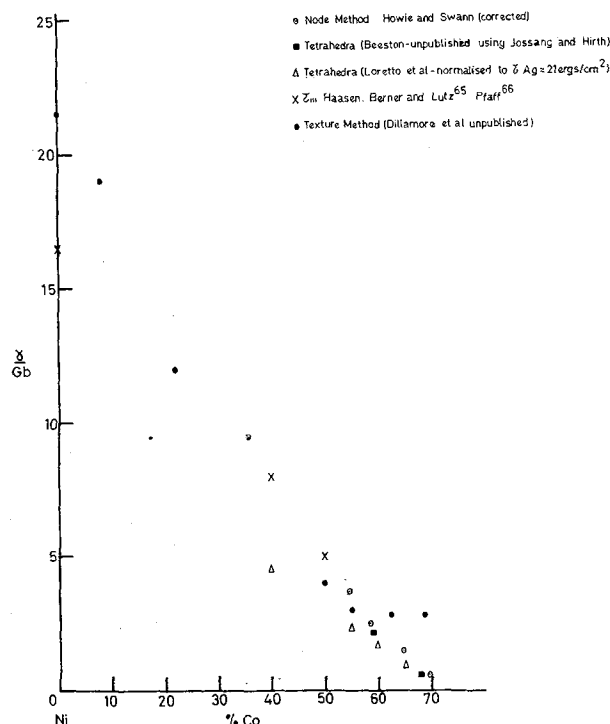


FIG. 5. — Comparison of stacking fault energy values for Ni-Co alloys obtained by different methods.

tems whose fault energy is too high for the node and tetrahedron methods to be useful. Recent work in Birmingham has been carried out using the texture method to study the variation of γ across the Ni-Cu, Pd-Cu and Pd-Ag systems [21]. It has been shown in the Pd-Cu and Pd-Ag systems that decreasing the number of conduction electrons from 1 per atom to 0.6 per atom has no effect on fault energy while the palladium d -band is full, but introducing holes in the d -band sharply increase the fault energy (Fig. 6). For

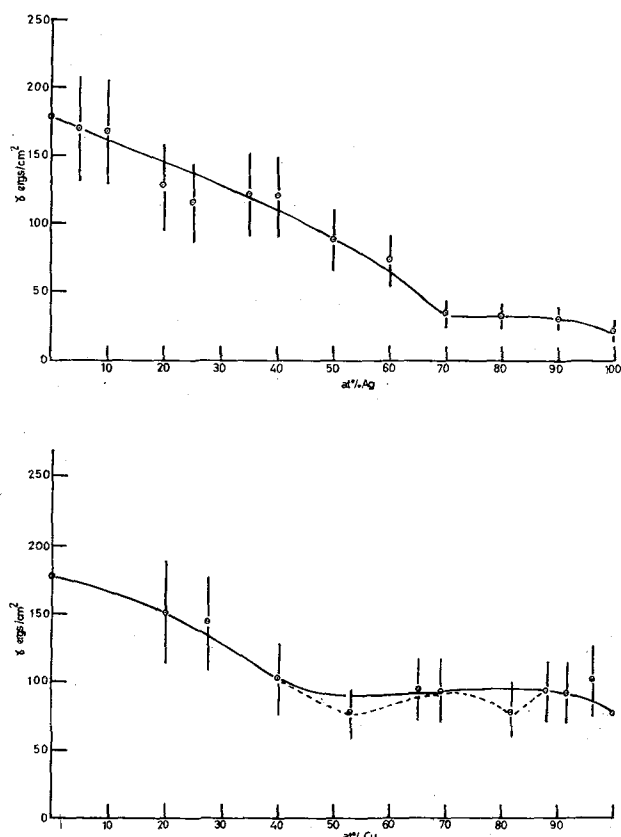


FIG. 6. — Variation of γ with composition for (a) Pd-Ag and (b) Pd-Cu alloys. In (b) the dotted curve exhibits anomalous behaviour as result of ordering (after Harris et al).

the nickel-copper system the results are less simple, probably because a shared band model is not a good approximation to reality. Here reducing the number of conduction electrons causes first an increase and then a subsequent decrease in fault energy with a relative minimum at about 50 atomic %, again the postulate that holes in the d -band contribute to the stability of the f. c. c. structure appears to explain the results at high nickel contents (Fig. 7). (The results on

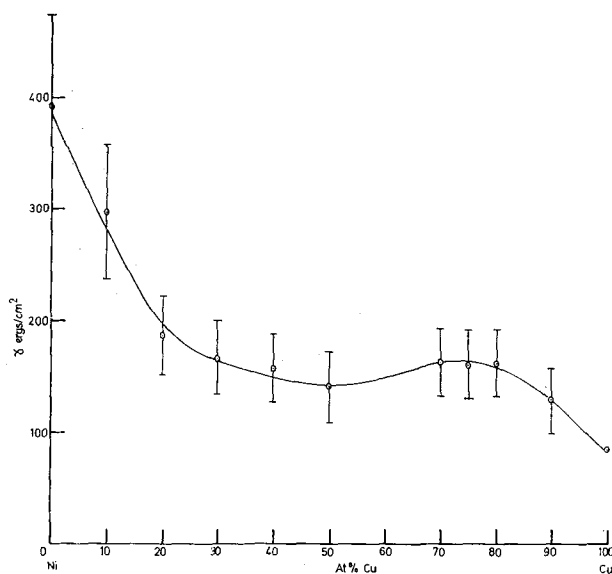


FIG. 7. — Variation of γ with composition in the Cu-Ni alloy system (after Harris et al).

the Cu-Ni system are confirmed by the α -parameter measurements of Henderson [21].)

These results show that the texture method is a most useful empirical method of determining fault energy in a range of fault energy where the best methods are inapplicable and where other methods are tedious or unreliable.

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Note added in proof. — Humble, Loretto and Clareborough (private communication), have recently evaluated γ/Gb for Ni as 8.7×10^{-3} from extrapolation of stacking fault tetrahedra data for Ni-Co alloys. Consideration of their data and additional results on this system (Dillamore and Beeston to be published) leads to a revision of this value to 13×10^{-3} giving $\gamma \simeq 240 \text{ ergs/cm}^2$. Using this value as a fitting point for the texture method lowers the high γ values of Table 1 and gives $\gamma_{\text{Al}} \simeq 130 \text{ ergs/cm}^2$.
