The Temperature Dependence of Stacking Fault Energy in Fe-Cr-Ni Alloys

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The variation of intrinsic stacking fault energy (γ_i) in two austenitic Fe-Cr-Ni alloys has been determined from dislocation node measurements over the range 25° to 325°C by means of high temperature transmission electron microscopy. In both alloys γ_i increases with temperature. Both reversible and irreversible effects have been observed in cyclic heating-cooling experiments. After the first heating to elevated temperature the irreversible component is removed and thereafter cyclic annealing produces essentially reversible changes in γ_i. The large reversible changes are best understood in terms of the variation in the stability of austenite with temperature. The smaller irreversible effect appears to arise from the formation of substitutional solute atmospheres around partial dislocations at elevated temperature.

THE stacking fault energy is an important materials parameter, determining to various degrees such properties as stress corrosion cracking susceptibility,[1] creep resistance,[2] and various mechanical properties. However, judgments of the merits of a particular material in an elevated temperature application can often be made only on the basis of room temperature measurements of the stacking fault energy. It has been shown, in fact, that the intrinsic stacking fault energy (γ_i) of several alloys[3-5] varies with thermal history: A reversible increase in γ_i with increased temperature has been observed in fcc Ni-Co alloys.[3] Irreversible changes on annealing have been reported in fcc Ag-Sn[4] and copper-based alloys.[5] In the case of the commercially important Fe-Cr-Ni system (stainless steels) the twin-density measurements of Cowan and Staehle[6] indicate the importance of considering the temperature dependence of stacking fault energy. They found that the frequency of twinning increased with increasing nickel concentration in a series of these alloys, suggesting that the twin boundary energy γ_t decreased with increasing nickel concentration. Dislocation node measurements,[7] however, had previously indicated an increase in γ_i with nickel content. Since $\gamma_i \propto \gamma_t$ in many materials,[8] these observations are in conflict. Cowan and Staehle emphasized, however, that γ_i had been determined at room temperature whereas twin frequency determinations are made upon microstructures developed at higher (1090°C) annealing temperatures. They suggested that the dependence of γ_i on T may have differed from one alloy to the next, accounting for their observations.

In the present work, the variation of γ_i with temperature for two Fe-Cr-Ni alloys has been determined from dislocation node measurements over the range 25° to 325°C. These temperatures are far below the usual annealing point for stainless steels, yet a significant reversible temperature dependence is observed.

I) EXPERIMENTAL

The two high purity alloys studied were Fe-10.7 wt pct Ni-18.3 wt pct Cr and Fe-15.9 wt pct Ni-18.7 wt pct Cr. The carbon content of each was on the order of 0.005 wt pct. Thin sheets of these materials were annealed in evacuated capsules at 1100°C for 1 hr. The capsules were then removed from the furnace and allowed to cool to room temperature before opening. The sheets were lightly deformed in bending to introduce fresh dislocations. Thin foils were prepared by the Bollman technique using a 60 pct phosphoric-40 pct sulfuric acid electrolyte. The foils were examined in a 100 kv electron microscope equipped with a tilting, furnace-type heating stage designed in our laboratory. The latter was calibrated with a small thermocouple fastened to a specimen and also directly checked by observing the melting points of thin foils of tin and lead in the electron microscope. In view of the uncertainties associated with thermal contact between the furnace and the foils and with electron beam heating, the temperatures are estimated to be accurate to about ± 10°C. Specimen contamination was reduced through the use of a cold finger in the objective lens chamber.

II) RESULTS

A) Room Temperature Measurements of γ_i

The intrinsic stacking fault energy was determined from measurements of the radius y of a circle inscribed within the extended node according to the expression obtained by Brown and Thölén.[9]

$$\frac{\gamma y}{\mu \mathbf{b}^2_p} = 0.055\,\frac{(2-v)}{(1-v)} - 0.06\left(\frac{v}{(1-v)^2}\right)\mathrm{Cos}\,2\alpha + \left[0.018\left(\frac{2-v}{1-v}\right) + 0.036\left(\frac{v}{1-v}\right)\mathrm{Cos}\,2\alpha\right]\log_{10}\frac{R}{\epsilon} \qquad [1]$$

where $\mathbf{b}_p$ is the partial dislocation Burgers vector, α the dislocation character angle, R the outer radius of curvature, v is Poisson's ratio, and ϵ is the cutoff distance related to the dislocation core radius. The use of this expression was described in detail earlier.[10] The effective[11] shear modulus $\mu(6.7 \times 10^{10}$ N per sq m)

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and the effective value of Poisson's ratio (0.43) were calculated from the anisotropic elastic constants given by Salmutter and Stangler[12] for a 12 pct Cr-12 pct Ni alloy, in the absence of information on the particular compositions of interest. The determinations of the Burgers vectors and character angles together with image inclination corrections were conducted in the usual manner.[10]

Over the years several investigators[13-16] have measured γ_i for different stainless steels. Several of these values are given in Table I together with the present results on the basis of the quantity $\gamma/\mu \mathbf{b}$. A more direct comparison is virtually impossible since there has been a striking lack of uniformity in the selection of the values for elastic constants, Poisson's ratio, and the method of calculation. The significance of variations in these parameters has been considered in detail by Ruff.[17] Since the use of effective elastic constants rather than isotropic values is considered to give more meaningful results,[17] we suggest that the values of γ_i reported herein, Table II, are the best available for these alloys.

Table I. Comparison of Reported Stacking Fault Energy Values in Fe-Ni-Cr Alloys for the Indicated Compositions, Wt Pct

Alloy	$10^3\ \gamma/\mu b$	Ref.
9.5 Ni-16.6 Cr	1.03	15
10 Ni-20 Cr	0.98	13
10.7 Ni-18.3 Cr	0.98	This work
14 Ni-18 Cr	1.08	16
15 Ni-20 Cr	1.20	13
15.9 Ni-18.7 Cr	1.41	This work

Table II. Average Values for the Intrinsic Stacking Fault Energy γ_i Determined from Extended Dislocation Nodes. The Number n of Individual Measurements and the 95 Pct Confidence Interval About the Mean are Given. Temperature Corrected Values of the Shear Modulus Were Used.

	γ_i, erg/cm^2 or mJ/m^2			
Alloy	As Deformed	At 135°C	At 325°C	Recovered Nodes, At 25°C
Fe-10.7 pct Ni-18.3 pct Cr	16.4 ± 1.1 $n = 17$	27.6 ± 1.2 $n = 21$	30.4 ± 1.6 $n = 16$	18.2 ± 1.0 $n = 19$
Fe-15.9 pct Ni-18.7 pct Cr	23.6 ± 0.9 $n = 21$	28.8 ± 1.1 $n = 18$	31.8 ± 1.4 $n = 20$	24.9 ± 1.0 $n = 20$

B) Temperature Cycling Experiments

Foils of both alloys were cyclically heated and cooled in the electron microscope, recording extended nodes at temperatures of 25°, 135°, and 325°C. The temperature was allowed to stabilize (approximately 10 min) before measurements were made. Typical results for extended nodes in 15.9 wt pct Ni alloy are shown in Fig. 1. Note that the node shrinks (γ_i increases) with increasing temperature, (*a*) to (*c*), but that this effect is reversible and the node expands again on cooling (*d*). Notice, too, that a significant irreversible change in node size occurs during the first thermal cycle, the recovered node in Fig. 1(*d*) being much smaller than the fresh node in 1(*a*). After the first heating to elevated temperatures the irreversible effects are removed and thereafter cyclic heating produces essentially reversible changes in the node size. Reversible and irreversible heating effects on the node size have also been observed in the 10.7 pct Ni alloy as indicated in Fig. 2. Similar changes in the widths of intrinsic-extrinsic fault pairs[18] in this alloy were observed during the thermal cycles.

A summary of our results is given in Table II and Fig. 3. The data presented therein represent measurements on relatively isolated dislocation nodes. Note

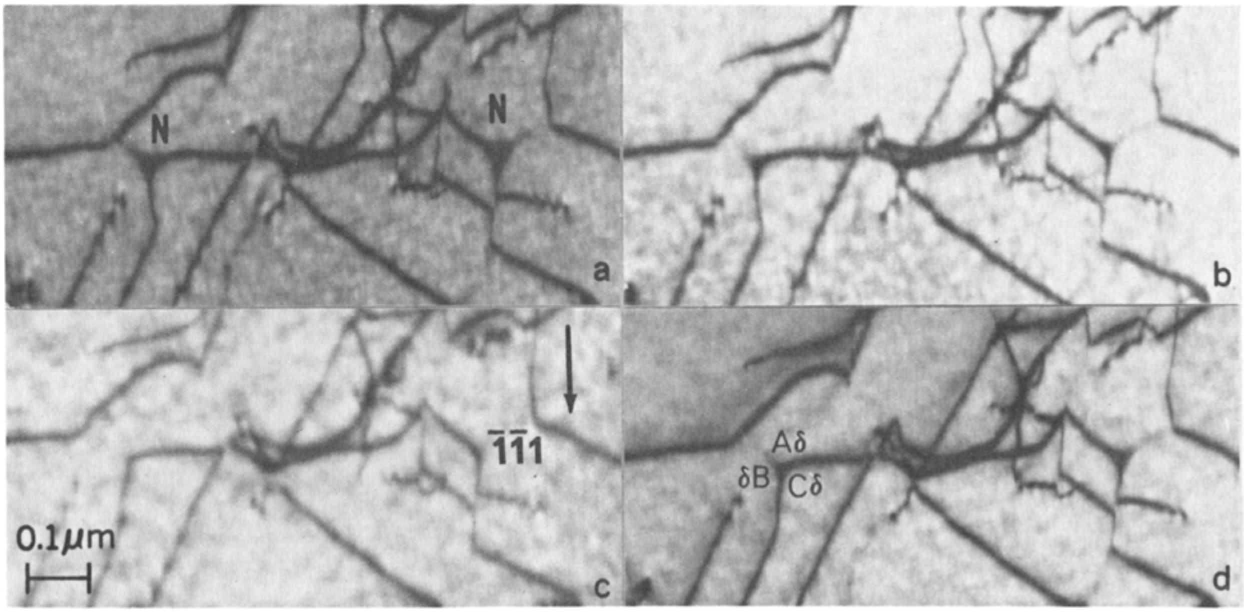

Fig. 1—Thermal cycling sequence of nodes (*N*) in a 15.9 wt pct Ni alloy. (*a*) As-deformed, (*b*) 135°C, (*c*) 325°C, (*d*) 25°C.

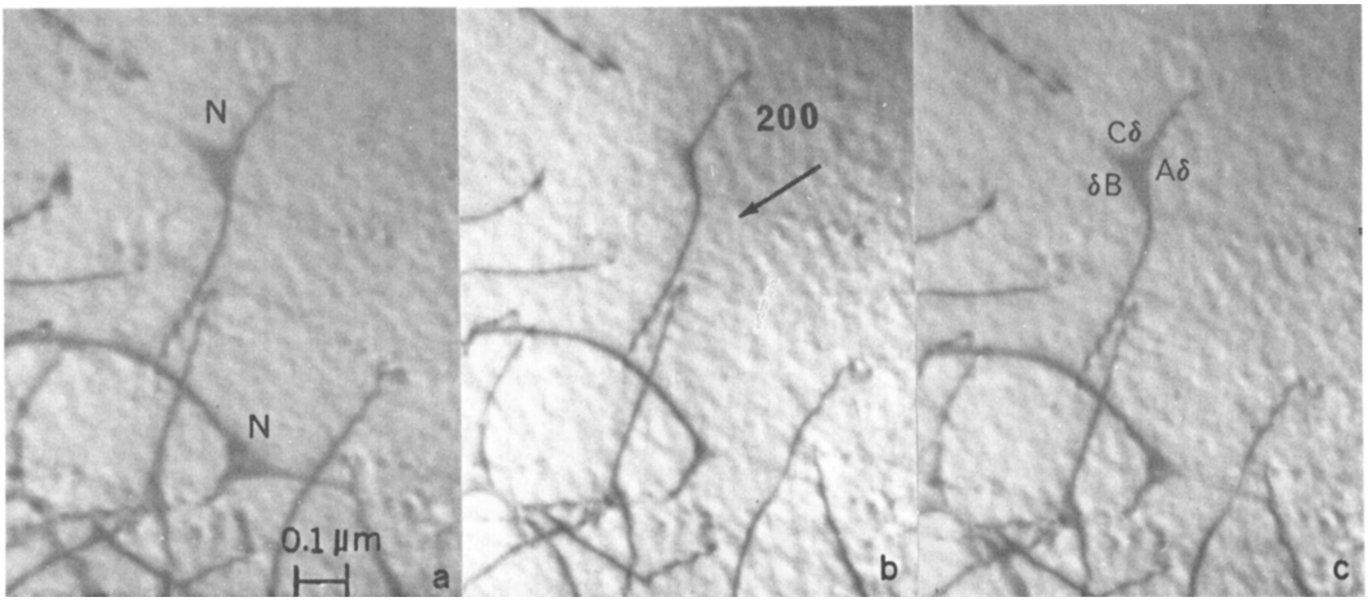

Fig. 2—Thermal cycling sequence of nodes (*N*) in the 10.7 wt pct Ni alloy. (*a*) As-deformed, (*b*) 135°C, (*c*) 25°C.

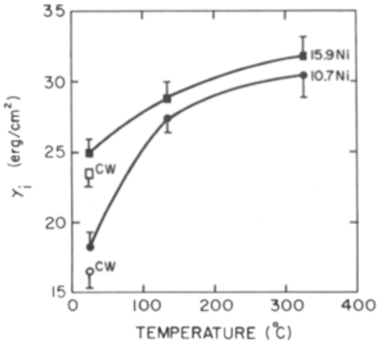

Fig. 3—Stacking fault energy variation with temperature for the two alloys shown. The initial as-deformed values are indicated by CW. The 95 pct confidence half-intervals are indicated.

that γ_i is greater in the nickel-rich alloy, as expected,[13-16] although the difference in γ_i becomes less at the higher temperatures. Node measurements at temperatures above 325°C were not attempted since oxidation of the foils was detected. As a result of the large shear modulus in this material, a reasonable measurement precision of about 5 to 10 pct was obtained at values of γ_i on the order of 30 erg per sq cm, where y was typically ~130Å. The scatter in the determinations of γ_i is indicated by the 95 pct confidence intervals shown in Table II.

III) DISCUSSION

In a recent review, Gallagher[5] has discussed the factors which may affect the size of extended dislocation nodes upon changes of temperature. These factors include the variation in material constants with temperature, deviations from an ideal random solid solution, and a true, reversible temperature dependence of γ_i.

In the first place, one must consider the effect of temperature changes on the elastic moduli and atomic spacing which appear in Eq. [1]. The change in lattice parameter[19] amounts to about 1 pct over the range of temperatures used in this study and $\mathbf{b}_p$ may be assumed constant to sufficient accuracy. Information on the variation of isotropic elastic constants with temperature is available for commercial austenitic stainless steels.[20] Poisson's ratio is relatively insensitive to temperature changes while the shear modulus is decreased by about 6 pct at 135°C and by about 11.5 pct at 325°C. Even allowing for this variation, a substantial temperature dependence still remains in γ_i, Fig. 3. By way of contrast, in pure silver and dilute silver alloys,[5] the temperature dependence of γ_i is small and of the same order and in the same sense as that of the shear modulus.

A) Reversible Changes in γ_i

In the 10.7 pct Ni alloy, γ_i increased from a recovered value of about 18 erg per sq cm (m joule per sq m) at 25°C to about 30 erg per sq cm at 325°C, in a reversible manner. The 15.9 pct Ni alloy showed an increase of about 30 pct in γ_i over this same temperature interval. These changes are believed to represent the actual temperature dependence of γ_i and should be related to the variation in phase stability with temperature. Such a relation is clear in an fcc lattice since an intrinsic stacking fault can be considered a thin region of hcp material. Thus, the variation in faulted area should be related to the variation in phase stability with temperature. For example, in fcc Ni-Co alloys the stability of the fcc phase increases with increasing temperature relative to the hcp phase and one expects, as observed,[3] a positive temperature coefficient of γ_i.

In these fcc Fe-Cr-Ni alloys similar comparisons are less direct for two reasons. First, the two alloys with which we have worked are in metastable equilibrium. According to the equilibrium phase diagram[21] for the present alloys, ferrite (bcc) is stable at room temperature, however, the transformation of austenite is so slow that the fcc phase persists on cooling from the high temperature austenite region to room temperature. In agreement with this, we observe that heating up to 1 hr at 325°C produced neither a significant change in the node sizes with time nor any indication of transforming regions. Secondly, the phase transformation in

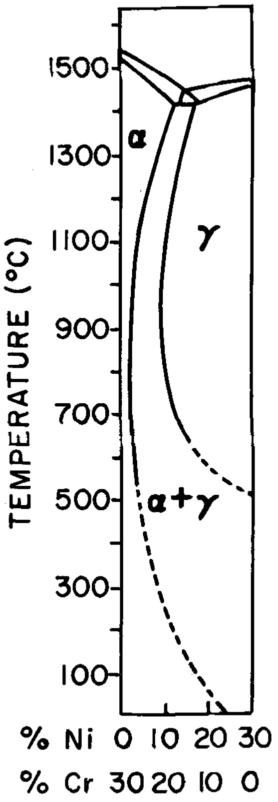

Fig. 4—Pseudobinary phase diagram[22] indicating the austenite-ferrite stability regions.

this system is fcc → bcc (rather than fcc → hcp). However, we note from the pseudobinary phase diagram, Fig. 4,[22] that, in the range of temperatures employed in this study, the stability of austenite should increase with temperature. This increasing stability predicts that the unfaulted matrix region (fcc) surrounding the node should increase in area (corresponding to a decrease in node size) with increasing temperature; this is precisely the observed dependence on temperature. We also note in Fig. 4 that the $(\alpha + \gamma)$ phase boundaries exhibit considerable curvature, accounting for the curvilinear relationship between γ_i and T in Fig. 3.

An hcp epsilon (ϵ) phase has been observed to develop during the martensitic transformation in some metastable austenitic stainless steels.[3,23,24] This transformation may be induced by refrigeration or by deformation. Since the degree of decomposition of the austenite is related to its stability, the likelihood of transformation may be reduced by increasing the concentration of austenite-stabilizing elements, *e.g.*, nickel, carbon. If one postulates that the ϵ phase may be related to the stacking fault regions in these alloys, one again expects that the node size will decrease with increasing nickel content, and with increasing temperature since the probability of the martensite transformation decreases.

It is worth noting that at higher temperatures, *i.e.* greater than about 900°C, one would predict from Fig. 4 that nodes would begin to expand as the slope of the phase boundary becomes positive in contrast to the negative slope at temperatures below 900°C. In alloys more concentrated in nickel and chromium the phase boundaries show only a slight temperature dependence up to about 1100°C. It is therefore conceivable that at these high annealing temperatures, γ_i could be lower in nickel-rich alloys as suggested by the twin density measurements[6] described earlier. We had no opportunity to examine this possibility further, and critical experiments at higher annealing temperatures remain to be pursued.

B) Irreversible Changes in γ_i

As shown in Fig. 4, a small but significant increase in γ_i is observed between the as-deformed and recovered specimens in both alloys. An irreversible change in node size can be due, for example, to the influence of segregation, solute impedance forces[25] or local internal stress variations (see Ref. 5 for detailed references). The role that these factors may play depends on the extent to which these processes can operate at the temperatures involved here.

Information on the effects of solutes in iron alloys of about 18 pct Cr and 13 to 23 pct Ni was reported by Swann and Pickering.[26] They found that stress-aging occurs at low temperatures (75°C) in short aging times (apparently about 2 min). This stress-aging effect was greater in the more nickel-rich alloy. The Portevin-Le Chatelier effect was also observed and became more prominent at higher temperatures (up to 200°C). Both of these effects can be ascribed to the diffusion of solute atoms to dislocations: static dislocations in stress-aging and mobile dislocations in the case of the P-L effect. Deformation-produced vacancies were thought to assist substitutional solute (presumably nickel) diffusion.[26,27] It seems reasonable therefore that solute diffusion could occur at the modest temperatures employed in the present experiments.

It is important next to recognize that in the process of creating isolated extended nodes by the interaction of two ribbons with a common partial,[11] the freshly created node must *expand* by glide from the moment of formation. Typically the node inner radius is of the order of five times the ribbon width of an extended dislocation. The equilibrium node size will be determined by a balance between the fault energy, elastic repulsion between the partials, the line tension of the partials, and local forces due to solute atoms. Therefore, the effect of a solute impedance force if present during room temperature deformation would be to increase the (apparent) γ_i since the nodes would be prevented from reaching their true equilibrium size. Significant impedance forces are not expected during the formation of nodes at room temperature in this system since solute mobility is very low (stress aging[26] has not been observed at 25°C) so that as-deformed nodes probably give a good indication of the fault energy at 25°C.

The as-deformed nodes are observed to shrink during heating, due to a genuine reversible temperature dependence of γ_i. However, the nodes do not expand back to the as-deformed dimensions on cooling. In view of the stress aging results mentioned earlier, we suggest that solute atom atmospheres collect at the partial dislocation at the elevated temperatures (135° and 325°C). At some stage in the subsequent cooling cycle, the expansion of the nodes is impeded by this atmosphere. Thus, the recovered node size at room temperature is actually representative of the equilibrium size at a slightly elevated temperature. The irreversible changes in γ_i observed here are small, and indicate that the solutes are not capable of exerting strong forces on the dislocation nodes in these alloys. We note that this explanation differs from the solute impedance concept described by Christian and Swann,[25] since we predict that the as-deformed node is more likely to be representative of the equilibrium room temperature node size than recovered nodes.

The segregation of solute to faults by a Suzuki mechanism[28] seems rather unlikely to provide a major contribution to the present observations on the irreversible changes in γ_i. Suzuki points out that the equilibrium solute concentration in the faulted region will change relative to the fcc matrix so as to decrease the free energy of the fault. Since both chromium and nickel increase γ_i in Fe-Cr-Ni alloys in the range of compositions used here,[7] the concentrations of these solutes would have to decrease in the fault. If desegretation occurs due to increased mobility of solute at elevated temperatures during a heating cycle one expects γ_i to decrease below the as-deformed value; this is not observed.

CONCLUSION

Both reversible and irreversible changes in γ_i have been observed in the present alloys over the range 25° to 325°C. The large reversible changes seem to be best understood in terms of the variation in the stability of the fcc (austenite) phase with temperature. The smaller irreversible change appears best explained by the formation of substitutional solute atmospheres around node arms.

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