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The Effect of Interstitial Solutes on the Twinning Stress of B.C.C. Metals

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ABSTRACT

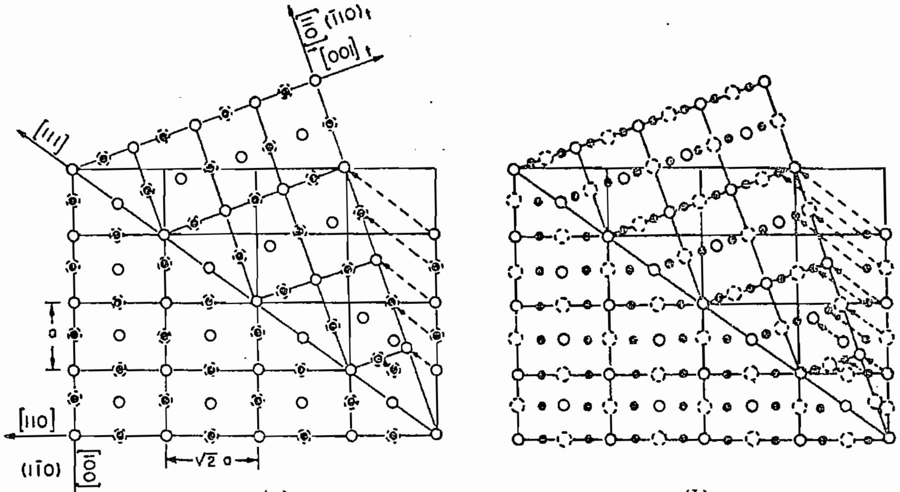
Flow stress measurements at −196°C on Fe-4·8 at. % Sn–C alloys demonstrate that the presence of interstitial carbon markedly increases the stress for deformation by twinning. The flow stress for an alloy with 0·09 at. % carbon is 12·6 Kg/mm^2 higher than the flow stress for a carbon-free alloy. This behaviour, which is an exception to the rule that twinning stresses are relatively invariant to solute content, was anticipated because the normal twinning mode in ferrite does not shear all interstitial atoms to proper octahedral sites. In confirmation of this viewpoint, the experimental results were found to be in reasonable agreement with a theoretical estimate of the change in twinning stress that should result from this crystallographic restraint.

§ 1. INTRODUCTION

THIS paper deals with a quantitative evaluation of the crystallographic restraint which octahedral interstitials impose on deformation twinning in body-centred cubic alloys. The restraint can be deduced from the work of Tamura, Yoshimura, Iwasaki and Ibaraki (1965) on deformation twinning in body-centred tetragonal martensite. They point out (taking [001] to be the axis of tetragonality) that twinning in the $(21\bar{1})$ and (121) modes necessarily gives rise to carbon atom shuffles. On the other hand, because the occupied octahedral sites in such crystals are limited to those having their 'dipole' directions aligned with the given tetragonal axis, no shuffles are required for twinning in the (112) mode. Figure 1 (*a*) shows how a (112) twin carries interstitial atoms in the favoured set of octahedral sites to equivalent sites in the product. Oka and Wayman (1969) recently found that the transformation twins in Fe-1·82 wt. % C martensite are those not requiring shuffles.

Magee and Davies (1971, in press) have established that, in cubic martensites, where all three types of octahedral sites have equal probabilities of occupation, deformation twinning does not occur. One explanation of this behaviour, suggested by D. W. Hoffman (private communication), is that in the cubic martensites (as opposed to tetragonal martensite) *none* of the {211} twinning modes can operate without carbon atom shuffles. Figure 1*b* shows how two-thirds of the carbon atoms are sheared into positions which lie along close-packed directions in the lattice of the twin,

Fig. 1



(*a*) (*b*)

The effect of the (112) twinning shear on the octadehral interstitial sites in a body-centred cubic lattice. (*a*) [1̄10] projection showing only those octahedral sites which are sheared to octahedral sites in the twin. ○: substitutional atoms in plane of projection: ●: possible interstitial atom positions in plane of projection: ◌: substitutional atoms $\sqrt{2}/2a$ above (and below) plane of projection. (*b*) [1̄10] projection showing the octahedral sites which are sheared to sites along the close-packed directions in the twin. ○: substitutional sites in plane of projection; ●: possible interstitial atom positions $\sqrt{2}/4a$ above (and below) plane of projection; ◌: substitutional sites $\sqrt{2}/2a$ above (and below) plane of projection.

e.g. at $[\frac{1}{4}, \frac{1}{4}, \frac{1}{4}]$ in the b.c.c. unit cell. As these positions are where the crystal should least easily accommodate an intruding atom, we shall refer to them as xenohedral sites.

In addition to the martensite work, there are other indications that interstitial solutes suppress twinning in b.c.c. metals (Stiegler and McHargue 1964, Stein, Low and Seybolt 1963). Whether the suppression results from interstitials in solid solution or from precipitates is not established. Moreover, changes in twinning stress were not measured; it was only observed that slip tended to replace twinning as the operative deformation mode when interstitials were added to the alloys.

In the present paper we assess both experimentally and theoretically the increase in the {211} twinning stress caused by the introduction of octahedral interstitials into a b.c.c. lattice. In predicting the change in stress the only unknown parameter is the difference in elastic free energy between a carbon atom in a xenohedral site and one in an octahedral site. A quantitative estimate of this energy is made for the case of carbon in iron using a pairwise interaction model. Measurements of the *change* in twinning stress caused by changes in the amount of carbon in solution in Fe-4·8 at. % Sn alloys are compared with the predicted value.

§ 2. ESTIMATE OF THE CHANGE IN TWINNING STRESS

2.1. *Propagation of a Twin*

As a semi-coherent twin boundary of area A propagates a distance dx (where x is perpendicular to the boundary plane), the interstitial atoms ahead of it will begin to experience the associated twinning shear. An equal number will be left in the completely twinned region behind the boundary. If, for simplicity, one assumes that the unfavourably situated interstitial atoms do not shuffle during the passage of the boundary†, the net effect is the transfer of $\frac{2}{3}C^{\mathrm{v}}A\,dx$ interstitial atoms from octahedral sites to xenohedral sites, where C^{v} is the number of interstitial atoms per unit volume and the factor of $\frac{2}{3}$ arises because one-third of the interstitials are sheared to octahedral sites. Because of the increased energy ΔG^{x-0} of an interstitial atom in a xenohedral site, extra work must be done to enlarge the twin,

$$dG^{x-0}=\Delta G^{x-0}(\tfrac{2}{3}C^{\mathrm{v}}A\,dx), \qquad (1)$$

requiring a force ($f=-\partial G/\partial x$) to push the boundary along:

$$f^{x-0}=-\tfrac{2}{3}C^{\mathrm{v}}A\,\Delta G^{x-0}. \qquad (2)$$

During the incremental propagation, the applied shear stress, τ^{a}, does work of magnitude $dG^{\mathrm{a}}=\tau^{\mathrm{a}}SA\,dx$, where S is the twinning shear. Thus, the force in the direction of propagation on the boundary due to the applied stress is

$$f^{\mathrm{a}}=\tau^{\mathrm{a}}SA. \qquad (3)$$

Twinning will occur when

$$f^{\mathrm{a}}+f^{x-0}+f_0\geqslant 0, \qquad (4)$$

where f_0 is the sum of any other forces opposing twinning. The assumption that such forces depend very little on carbon content is in line with the observation of Bolling and Richman (1967) that the concentration of substitutional solutes does not affect the b.c.c. twinning stress. Substituting eqns. (2) and (3) into (4) and defining $\tau_0\equiv -f_0/SA$, one obtains

$$\tau^{\mathrm{a}}\geqslant\tau_0+\left(\frac{2}{3S}\right)\Delta G^{x-0}C^{\mathrm{v}} \qquad (5)$$

For b.c.c. {211} twins, $S=\sqrt{2}/2$; hence

$$\tau-\tau_0=(\tfrac{2}{3})\sqrt{2}\,\Delta G^{x-0}C^{\mathrm{v}}. \qquad (6)$$

Thus, estimation of the increase in shear stress arising from the presence of octahedral interstitials reduces to the problem of assessing ΔG^{x-0}, the

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† Instantaneously, at least, this involves a change in crystal structure and thus the process could properly be called a martensitic transformation. However, focusing on the substitutional atoms we choose to refer to it as twinning.

difference in free energy of the interstitial in a xenohedral versus an octahedral site.

2.2. *Estimation of ΔG^{x-0} for Carbon in B.C.C. Iron*

Consider three conceptual steps by which a single carbon atom can be transferred from an octahedral site to a xenohedral site. (1) Compute the energy required to move the statically displaced iron atoms in the strain field of the octahedral carbon atom back to their ideal or perfect lattice sites (R^0). (2) Compute the energy required to move the carbon atom from the octahedral site to a xenohedral site, while holding the iron atoms rigidly on the perfect lattice (ΔX^{x-0}). (3) Compute the work done by the crystal as the iron atoms are released and allowed to become displaced in response to the solute forces extending from the carbon atom at the xenohedral site (R^x). Formally then, one writes

$$\Delta G^{x-0} = \{R^0 + \Delta W^{x-0} - R^x\}. \quad \ldots \ldots \quad (7)$$

To evaluate these three quantities, consider the familiar crystalline model in which the interatomic forces are linear functions of the atomic displacements. The interatomic force constants that obtain in such a model for pure b.c.c. iron have been determined to fifth neighbour pairs by Brockhouse, Abou-Helal and Hallman (1967), and we assume that these iron–iron interactions are unchanged by the addition of interstitial carbon. To determine a force constant for the carbon–iron interactions we note that it is possible to estimate the solute force ϕ_i^S at two different radii about the interstitial from existing knowledge of the tetragonal distortions of the average lattice parameter in iron–carbon martensite. As computed recently by Hoffman (1970) the forces on the iron atoms of the first and second shells are $\phi_3^{00\frac{1}{2}} = 7{\cdot}14 \times 10^{-4}$ dynes and $\phi_1^{\frac{1}{2}\frac{1}{2}0} = \phi_2^{\frac{1}{2}\frac{1}{2}0} = 1{\cdot}66 \times 10^{-4}$ dynes. Here the superscripts give the positions of the iron atoms relative to the carbon and the subscripts refer to Cartesian components; also, we note the symmetry property, $\phi_i^{-S} = -\phi_i^S$.

A linear carbon–iron force law can thus be formulated if an estimate can be made of the equilibrium positions of these same iron atoms, displaced as they are by the solute forces. Estimation of the static displacements, U_i^S, is facilitated by use of the lattice Green's tensor $G_{ij}^{(S'-S)}$ attributed by Krivoglaz (1969) to Lifshitz (1948) and discussed in detail by Flinn and Maradudin (1962). Whereas $G_{ij}^{(S'-S)}$ gives the displacement of an atom at S caused by unit components of force on the atom at S', the total displacement of the atom at S, by linear superposition becomes

$$U_i^S = \sum_{S',j} G_{ij}^{(S'-S)} \phi_j^{S'}. \quad \ldots \ldots \ldots \quad (8)$$

Various elements of $G_{ij}^{(S'-S)}$ computed for b.c.c. iron are arranged in table 1. Since the solute forces are assumed zero beyond two shells of iron, eqn. (8)

Table 1. Lattice Green's tensor for b.c.c. iron. Selected variants computed from force constants of Brockhouse *et al.* (1967) by direct-method summation of 16 000 points, see Flinn and Maradudin (1962)

$S'-S$	0,	0,	0	$\pm\frac{1}{2}$	$\frac{1}{2}$,	$\frac{1}{2}$†	0,	0,	1
$G_{ij}{}^{S'-S}$	956	0	0	271	± 69	± 69	203	0	0
	0	956	0	± 69	271	69	0	203	0
(Å/dyn)	0	0	956	± 69	69	271	0	0	225

† Sign of $(S'-S)$ element influences off-diagonal elements in corresponding row and column as indicated. Diagonal elements are always positive.

yields with little effort the desired displacements, $U_3{}^{00\frac{1}{2}} = 0{\cdot}430$ Å and $U_1{}^{\frac{1}{2}\frac{1}{2}0} = U_2{}^{\frac{1}{2}\frac{1}{2}0} = 0{\cdot}022$ Å†. Dividing the difference between the *radial* equilibrium forces $(\phi_3{}^{00\frac{1}{2}} - \sqrt{2}\phi_1{}^{\frac{1}{2}\frac{1}{2}0})$ by the radial spacing of the relaxed shells yields $-24{\cdot}76 \times 10^4$ dyne/cm for the radial force constant ϕ_{rr} in the central force approximation. Having utilized both first and second neighbours to deduce the carbon–iron force law, let us simplify computation of the energetic quantities by restricting attention to nearest neighbours.

The relaxation energy is the work required to reversibly push the displaced iron atoms back to their ideal-lattice sites, in the presence of the interstitial impurity. The work done on each iron atom equals one-half the displacement times the final applied force. At the perfect lattice site, however, the iron–iron forces are balanced, and so this applied force equals in magnitude the solute force $\hat{\phi}_i$, commonly called the 'initial force' (i.e. the force before relaxation) and given by

$$\hat{\phi}_i{}^S = \phi_i{}^S - \phi_{rr} U_i{}^S \quad . \; . \; . \; . \; . \; . \; . \; . \; . \quad (9)$$

when S is $(0\,0\,\frac{1}{2})$, $(\frac{1}{2}\,\frac{1}{2}\,0)$, $(\frac{1}{4}\,\frac{1}{4}\,\frac{1}{4})$, or variants thereof‡. Thus, a sum over the iron atoms about the interstitial yields, for the relaxation energy,

$$R = \tfrac{1}{2} \sum_{S,\,i} \hat{\phi}_i{}^S U_i{}^S. \quad . \; . \; . \; . \; . \; . \; . \; . \quad (10)$$

Considering nearest neighbours we obtain $\hat{\phi}_3{}^{00\frac{1}{2}} = 17{\cdot}81 \times 10^{-4}$ dyne, and $R^0 = 4{\cdot}79$ ev for the octahedral relaxation energy.

The reversible work, ΔW^{x-0}, required to transfer the carbon interstitial from an octahedral to a xenohedral site in the ideal lattice of iron atoms is the same as the work to compress the nearest-neighbour iron atoms from a

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† Estimation and measurement of this dipole displacement field have been the object of numerous investigations. In particular, one can show that the computation of Krivoglaz and Tikhonova (1960), which is tantamount to the Green's tensor method, yielded the oft-quoted negative value for $U_1{}^{\frac{1}{2}\frac{1}{2}0}$ only because of the estimate they were forced to make of the iron-iron force constants in lieu of better data.

‡ In a more general form of eqn. (9), the final term is $\sum \phi_{ij}{}^S U_j{}^S$, where $\phi_{ij}{}^S$ is a tensor derived from ϕ_{rr} by eqn. (2.26) of Flinn and Maradudin (1962).

radius of $a/2$ to a radius of $\sqrt{3}a/4$ against the solute force. As this causes the radial solute force to increase to $\hat{\phi}_r^{\frac{1}{4}\frac{1}{4}\frac{1}{4}} = 22{\cdot}65 \times 10^{-4}$ dynes, the formula $(\hat{\phi}_3^{001} + \hat{\phi}_r^{\frac{1}{4}\frac{1}{4}\frac{1}{4}})(2 - \sqrt{3})a/4$ gives the work done by both neighbours, $\Delta W^{x-0} = 4{\cdot}84$ ev.

For evaluation of the xenohedral relaxation by eqn. (10), the components of the initial force $\hat{\phi}_i^{\frac{1}{4}\frac{1}{4}\frac{1}{4}} = \hat{\phi}_r^{\frac{1}{4}\frac{1}{4}\frac{1}{4}}/\sqrt{3}$ are in hand, but the equilibrium displacements remain to be determined. From eqn. (8) one obtains

$$U_i^{\frac{1}{4}\frac{1}{4}\frac{1}{4}} = \sum_j (G_{ij}^{000} - G_{ij}^{\frac{1}{4}\frac{1}{4}\frac{1}{4}})\phi_j^{\frac{1}{4}\frac{1}{4}\frac{1}{4}}, \quad . \quad . \quad . \quad . \quad . \quad (11)$$

which by symmetry equals $U_2^{\frac{1}{4}\frac{1}{4}\frac{1}{4}}$ and $U_3^{\frac{1}{4}\frac{1}{4}\frac{1}{4}}$. Since the equlibrium solute force is unknown, it is necessary to eliminate it by means of eqn. (9) which becomes

$$\phi_i^{\frac{1}{4}\frac{1}{4}\frac{1}{4}} = \hat{\phi}_i^{\frac{1}{4}\frac{1}{4}\frac{1}{4}} + \phi_{rr} U_i^{\frac{1}{4}\frac{1}{4}\frac{1}{4}}. \quad . \quad . \quad . \quad . \quad . \quad . \quad (12)$$

Solution for the displacement yields $U_1^{\frac{1}{4}\frac{1}{4}\frac{1}{4}} = 0{\cdot}303$ Å, and from eqn. (10) the xenohedral relaxation energy, $R^x = 7{\cdot}40$ ev. Combining the computed energies according to eqn. (7), we estimate the net reversible work done by the nearest-neighbour iron atoms against the solute forces of a single carbon atom during twinning $\Delta G^{x-0} = 2{\cdot}23$ ev.

The contribution of second-neighbour iron–carbon interactions has been assessed by an extension of the preceding method. In place of eqns. (11) and (12), however, two sets of equations are solved simultaneously for the displacements of first and second neighbours about the xenohedral carbon atom. An uncertainty arises in that the interstitial acquires two additional second neighbours as it is transferred to a xenohedral site. Because these iron atoms approach from a relatively large distance, it is unrealistic to assess their contribution to ΔW^{x-0} from the linear force law. Although they must be included in computation of the xenohedral displacements, we lump together their contributions to the energies ΔW^{x-0} and R^x as an undetermined quantity—$2\int \phi(r)\,dr$—which is certainly negative and probably small. Thus, the computation with second-neighbour solute forces included yields $\Delta G^{x-0} = (2{\cdot}48 + 2\int \phi(r)\,dr)$ ev, not far from our simpler nearest-neighbour estimate.

We also note that the errors associated with using a linear force model are not expected to be excessive. Significant non-linear terms are neglected during the large intermediate displacements envisaged in computation, but these terms cancel out in the final result because the net change in the *relaxed* radius of the first iron shell is relatively small. Using the numerical values of the static displacements quoted above, these first shell radii come out to be 1·861 Å and 1·752 Å about carbon interstitials in octahedral and xenohedral sites respectively.

§ 3. Experimental Procedures

In order to study changes in twinning stress, it is necessary that twinning be the dominant deformation mode for all alloys in the condition tested. The comprehensive work of Bolling and Richman (1967) demonstrates that in many iron alloys twinning dominates the deformation behaviour at room temperature and below. It is also important that the major alloying element does not form a carbide more stable than Fe_3C so that the solubility of carbon does not drastically decrease; thus, it was decided to make up a series of alloys based upon Fe-4·8 at. % Sn. Four alloys with varying carbon content were melted and cast in vacuum; all contained 0·06 at. % Al to minimize effects from oxygen and nitrogen in solution. One of the alloys also contained 0·30 at. % Ti which Magee and Paxton (1968) have shown to be sufficient to remove thermal stabilization in iron alloys and thus to restrict carbon in solution to less than 0·01 at. % (Philibert 1955).

Compression samples $\frac{1}{4}$ in. × $\frac{1}{4}$ in. × $\frac{1}{2}$ in. were cut from the ingots that had been annealed at 1000°c for 64 hrs, then annealed *in vacuo* at 725°c for 1 hr and quenched into iced brine. The samples were stored in liquid nitrogen prior to testing with an Instron machine in a self-aligning compression apparatus at a strain rate of 3×10^{-4} sec^{-1} in liquid nitrogen. Deforming at −196°c minimizes deformation by slip. The grain size of the ferrite in the samples did not vary sensibly from alloy to alloy and was approximately 1 mm. Light microscopy showed no evidence of carbides in alloy 4 indicating that all carbon (except in alloy 1) was in solution during the final anneal. Moreover, the iced brine quench should be sufficient to avoid precipitation.

§ 4. RESULTS

Table 2 gives the results of the flow stress measurements (at. 0·2% offset) for the four alloys along with their analysed carbon contents. The salient point is that the flow stress does increase with carbon content. Before considering this result further we note several ancillary findings. (1) Serrations were observed on all stress–strain records; (2) metallographic examination and 'clicks' during deformation also confirmed that twinning was the operative deformation mode; (3) the serrations became smaller as the amount of carbon in solid solution increased. (This is reminiscent

Table 2. Carbon content and flow stress of Fe-4·8 at. % Sn alloys

Alloy No.	At. % C†	Flow stress at 0·2% offset, Kg/mm^2
1	0·019 (also contains 0·3 at. % Ti)	34·0, 34·6‡
2	0·021	38·0, 39·8‡
3	0·054	42·6
4	0·089	45·8, 46·2‡

† Value reported is mean of four measurements by a conductometric method: scatter in individual measurements is about ±20% in carbon content.

‡ Duplicate specimens.

of observations by Bolling and Richman (1965) on Fe–Be alloys when an ordering force was opposing deformation twinning); (4) the alloys became less brittle as the carbon content increased. It was not possible to compress alloy 1 to more than 1% plastic strain without fracture, but alloys 3 and 4 were compressed about 3%. Bolling and Richman (1966 a) observed similar effects of twinning on fracture but in the present case oxygen embrittlement of Fe may well be involved (Low and Feustal 1953).

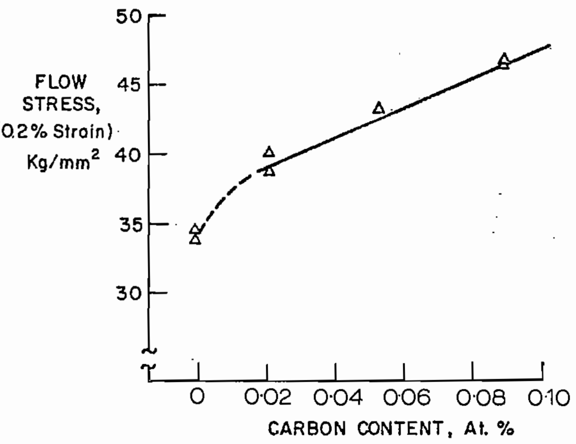
§ 5. DISCUSSION

The change in the shear stress for flow per unit addition of carbon is the relevant parameter to compare to our theoretical estimate. Inserting the estimate of ΔG^{x-0} ($=2{\cdot}23$ ev) in eqn. (6) yields a predicted slope ($d\tau/dC$) of 28 Kg/mm²/at. % C. Unfortunately, the experimental results do not yield a single slope for comparison with this prediction because, when they are plotted as a function of the carbon content, curvature is apparent (fig. 2). Similar behaviour has been observed by Wert (1950) in alloys undergoing slip, the first solute additions likewise leading to more rapid increases in flow stress than at higher concentrations. Since in computing the predicted slope no allowance was made for effects such as the bowing of interface dislocations between widely spaced carbon atoms one might expect our estimate to be in better agreement with the experimental slope at the higher carbon levels. With this in mind, a least squares line (solid in fig. 2) was fit to the nearly linear data from alloys 2, 3 and 4, yielding the following dependence:

$$\sigma(\text{Kg/mm}^2) = 36{\cdot}8 + 104 \times \text{at. \% C.} \quad . \quad . \quad . \quad . \quad . \quad . \quad (13)$$

The experimental slope $d\sigma/dC$ increases to 145 Kg/mm²/at. % C if the results for the lowest carbon alloy are included in the least squares analysis.

Fig. 2



Compressive flow stress (at. 0·2% offset) as a function of the carbon content of the alloys. The results for alloy 1 are plotted at zero at. % C.

For comparison with theory it is necessary to convert the measured compressive flow stresses to appropriate shear stresses. In their consideration of twinning in b.c.c. alloys, Bollman and Richman (1966 b) give a value of 3·1 to the factor $m(\equiv \sigma_{\text{poly}}/\tau_{\text{single}})$ which combines the effects of orientation and constraint. Dividing this number into eqn. (13) provides an experimental determination of 33·5 Kg/mm²/at. % C for the slope $d\tau/dC$, not far from the theoretical estimate of 28 Kg/mm²/at. % C.

Granted that further improvements in both the theory and the experiment are possible, this agreement is adequate to confirm the basic premise concerning the effect of octahedral interstitials on the twinning stress of b.c.c. materials. Indeed, it can be suggested that interstitials in b.c.c. metals represent a notable exception to the rule that twinning stresses are relatively invariant to solute content. The common behaviour of twinning replacing slip at high solute content is not necessarily to be expected in such alloys.

In this respect, it is noteworthy, with reference to the strength of cubic martensite, that for 0·5 wt. % carbon in solution the present results indicate an increase in uniaxial twinning stress of several hundred thousand p.s.i. We thus achieve a quantitative basis for interpreting the finding of Magee and Davies (1971, in press) that twinning is suppressed in cubic martensites at such carbon levels. While twinning (without shuffles) intervenes and limits the strength of tetragonal martensites, the cubic varieties deform by slip at all carbon levels, achieving strengths as much as 200 000 p.s.i. greater than their tetragonal counterparts. It becomes clear, therefore, that the crystallographic restraint investigated here is fundamental to the high hardness attainable in many as-quenched commercial steels.

§ 6. Conclusions

1. Carbon in solution in α iron markedly increases the stress necessary for deformation twinning. Experimental results for three carbon-bearing alloys yield $d\tau/dC = 33{\cdot}5$ Kg/mm²/at. % C.

2. The resistance to twinning apparently arises from the fact that the accompanying lattice shear carried two-thirds of the carbon atoms to improper sites. An estimate of the increase in shear stress for twinning caused by presence of interstitial carbon yields $d\tau/dC = 28$ Kg/mm²/at. % C, in good agreement with experiment.

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