The twinning-to-slip transition

A final point for discussion is the reason for the apparent twinning–slip transition during the growth of a martensite plate. This is at present a matter for speculation but may be related to the temperature increase (at the interface) during the formation of the initial twinned portion of the plate. If conditions are essentially adiabatic during this stage, the local temperature change due to the latent heat of transformation may be significant enough for the preferred mode of martensite deformation (inhomogeneous shear) to be slip rather than twinning. It is well known that twinning is a preferred deformation mode at low temperatures, and slip predominates at high temperatures, implying that a critical or crossover temperature exists. If such a critical temperature exists near room temperature for Fe–Ni alloys, it would be expected that martensite formed near room temperature would be internally slipped, while that formed at quite low temperatures would be internally twinned. At intermediate temperatures a plate may begin its growth by internal twinning to be followed by a transition into slip because of the local temperature increase. The following observations are consistent with this suggestion:

(i) for Fe–Ni alloys containing less than 29%Ni (M_s ∼room temperature) no internal twins are found in the martensite[28]

(ii) for Fe–30%Ni (M_s ∼−30°C) the plates consist of an internally twinned midrib region but the major portion of the plates is untwinned.

(iii) for Fe–33·2%Ni (M_s∼−150°C) the plates are completely internally twinned

(iv) by determining cooling curves (temperature v. time) for Fe–Ni alloys by means of miniature thermocouples[28] the temperature rise during a martensite burst is as much as 40 deg C (theoretical calculations of Nishiyama *et al.*[37] indicate that a local temperature change of as much as 70 degC is possible), which suggests that the initial twinned region of a plate can locally raise the temperature enough to effect a twinning-to-slip transition.

It is relevant to comment on the morphology of martensite v. the mode of inhomogeneous shear. When complete internal twinning is the case, the martensite is definitely plate-like in the classical sense and exhibits very straight, planar austenite-martensite interfaces. On the other hand when no twinning is involved (i.e. Fe–29·0%Ni) it is not clear that plates are formed at all (i.e. needles). For intermediate nickel contents, Fe–30%Ni, the terminus of the transformation twins defines a more or less definite plane, but the interface corresponding to the slip-thickened portion of the plates is highly irregular. Breedis and Wayman[38] found that less habit plane scatter occurred when the midrib plane was taken as the true habit plane, as compared with the mean planar austenite/martensite boundary itself. It may then be that, if the inhomogeneous shear is slip, some restrictions (i.e., dislocation multiplication ability) dictate the final morphology. Thus, when twinning is involved, the shape deformation is an invariant *plane* strain and the martensite takes the form of plates. A more non-planar or needle-like morphology may result when the inhomogeneous shear is slip. In cases where a twinning-to-slip transition occurs the final interface is not planar, and in fact is rather nondescript.[38] It would be a good theoretical problem to consider martensite morphology v. the mode of internal deformation or inhomogeneous shear.

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Discussion 6

Chairman: Mr J. E. Russell (English Steel Corporation Ltd)

Mr J. A. Klostermann (Stichting voor Fundamenteel Onderzoek der Materie) said that he wished to comment on Professor Wayman's paper.

Honma found in iron–nickel alloys containing 20–30% Ni a brick-like, blocky inner structure. By trace analysis the long dimension of the blocks was found to be near {111}. On the surface Honma found a Widmanstätten-like surface relief in the {111} habit and a side-by-side relief which did not reveal any crystallographic consistency.

Mr Klostermann wished to suggest that the blocky inner structure was perhaps closely related to angle profile martensite ('butterfly-martensite') that he had found, with {112} habit. The face martensite was closely related to the surface martensite he had found. He thought that there was only one phenomenon and one habit, namely {112}, and that the difference between Widmanstätten and side-by-side relief could be ascribed perhaps to slight differences in the preparation of the specimen.

He had the impression indeed that there were more than four variants in Figure 10 of Professor Wayman's paper, and this also applied to the micrographs of the very rapidly quenched zone-refined iron (Wayman and Altstetter[1]); but it was rather difficult to decide which needles of the polycrystalline specimen belonged to one crystal. It could therefore be that the rapidly quenched martensite was also surface martensite with a {112} habit, so there was no {111} martensite habit at all in the whole iron–nickel range. Of course, when the habit plane was very badly defined, three {112} habits lying near a {111} pole would be taken for that {111} pole.

Professor Wayman supposed that the habit of surface martensite found by Klostermann and Burgers would be {225}. On this point he could say that the maximum experimental error was estimated to be 2° and that {225} lay about 6° from {112}; even the habit {449} found by Wayman et al.[2] was about 3° from {112}. In fact, he had no reason to assume that the habit was not very near to {112} or exactly {112}.

He did not know what was meant by the cell structure by Speich and Swann, but he assumed that it would perhaps have an appearance like bands of networks of angle profile martensite.

With regard to the straightness of the dislocations arrays in the untwinned region of iron–nickel martensite, he wished to point out that these arrays could possibly be a dislocation reaction product which formed systematically at the martensite/austenite interface. The straightness of the dislocations suggested that they were sessile.

Professor C. M. Wayman (University of Illinois) said that, on Figure 10 of his paper which was taken from Honma, he would agree that, looking at this, one could perhaps see more than three variants.

As to whether it was {112} or {225}, as he suggested in the paper, the reason he had arrived at that conclusion was because he had replotted the results of Klostermann and Burgers and had convinced himself that it could be as close to {225} as it was to {112}.

With regard to dislocations observed in the untwinned regions of the foil they appeared to be straight dislocations in the {110} orientation, but they could be gliding on a {112} plane and they could actually be bowed; in the particular {110} orientation he had shown, in projection they would appear as straight dislocations although in fact they were curved.

**Dr J. W. Christian** (University of Oxford) asked if they were straight in the {111} orientation.

Professor Wayman replied that they were fairly straight.

Mr P. McDougall (University of Leeds) said that Professor Wayman had described the transformations in iron–nickel alloys studied by Honma as having a needle-like form with the needles arranged in blocks having well defined boundaries. He had suggested that the concept of a habit plane might not be appropriate to these structures.

He wished to point out, however, that if the structural unit of the transformation product was formed as laths then these would exhibit both a habit plane and a particular direction of the lath lying in that plane. In this connexion he wished to mention the preliminary results of some recent electron microscope studies of the fine martensite structure produced in the alloy Fe–24%Ni–0.1%C. The structure appeared to consist of laths with the habit plane parallel to $(111)_F$ and the long direction of the lath parallel to $[1\bar{1}0]_F$ for the variant of the orientation relationship $(111)_F \parallel (101)_B$ and $[1\bar{1}0]_F \parallel [11\bar{1}]_B$ (where the subscripts F and B referred respectively to the fcc and bct lattices). These results were thus in general agreement with the earlier findings reported by Bowles[3] that the transformation product in low-carbon iron alloys was formed as laths lying in $<110>_F$ directions, and the later habit plane measurements of Kelly and Nutting[4] and Entwisle[5] which showed that the laths lie parallel to $\{111\}_F$.

Professor Wayman referred to Honma's work and said that, in speaking of the surface relief which Honma observed – provided he had translated it properly – Honma said that the habit {111} associated with the surface relief was obtained by means of a one surface trace, so this ruled out that it would or would not be a plate. This could not be determined on the basis of one surface.

With regard to the interior block or brick-like structure observed by Honma, it would appear that it was strongly similar
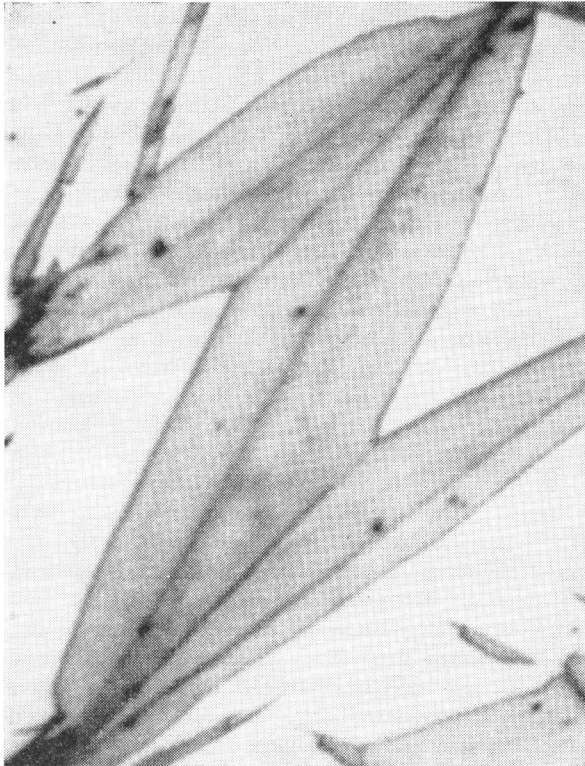
A *Martensite plates of {259}$_\gamma$ habit (0·5%C–24%Ni steel)*
*$\times$6000 (after enlarging $\times$10 in reproduction)*

to what was being observed by Mr McDougall and which he had called a lath.

With regard to the point that there were no martensite needles, Mr. Klostermann and Professor Burgers had reported the needles they had observed to have dimensions 1000:30:40. He did not see that such a ratio could mean anything but a needle.

As far as distinguishing between needles and plates was concerned, he had his own view on that: one could mend a stocking with a needle but if one punched a hole in the edge of a dinner plate and put a thread through that, you could not very well mend a stocking. That was the way he viewed it.

Dr R. Brook (English Steel Corporation Ltd) commented on the nature of the midrib in martensite plates, and the plate junctions.

The burst transformation to martensite was induced in a 0·5%C, 24%Ni steel ($M_b = -50°C$) by cooling to $-70°C$. Metallographic examination revealed that most of the martensite formed in zig-zag arrays of plates on the {259}$_\gamma$ habit. Only a few small plates of {225}$_\gamma$ habit were produced and these were located immediately adjacent to the much larger plates of the
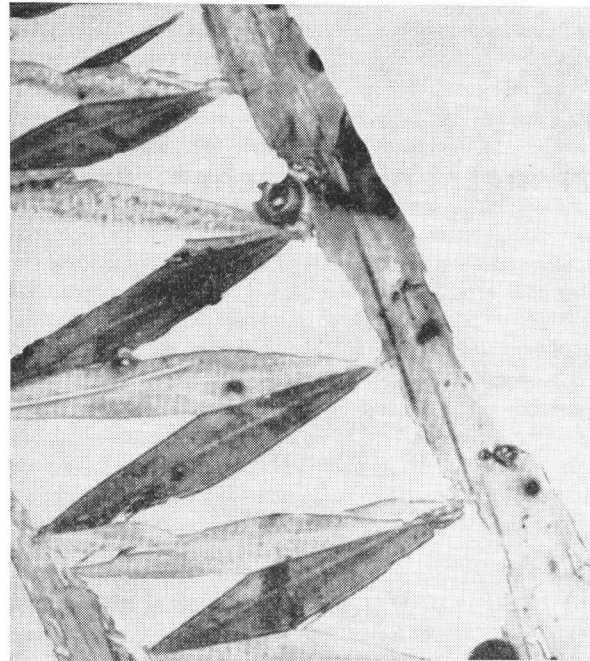
B *Typical zig-zag array of plates produced by the burst transformation to martensite (0·5%C–24%Ni steel)* $\times$500

zig-zags. Where plates of the {259}$_\gamma$ habit met they showed an acute-angled configuration, whereas those of the {225}$_\gamma$ habit were related by obtuse angles.

Figure *A* showed that the interface between adjoining plates of {259}$_\gamma$ habit was straight, and the midribs subtended equal angles about the interface. A number of junctions had been examined and all showed similar features to those described.

Figure *B* was a typical field at a lower magnification, showing that these same features obtained in all of the plates comprising a zig-zag array. This led one to believe that the martensite plates of {259}$_\gamma$ habit propagated first of all in the form of a long thin midrib, which subsequently thickened at a relatively slow rate.

Professor Wayman said he would agree that some of his observations were quite consistent with a midrib region forming first, to be followed by the lateral growth of a plate.[6]

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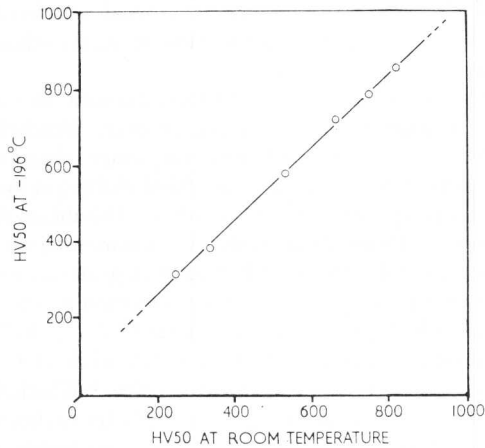
1 *Relationship between HV50 at −196°C and at room temperature for a series of martensitic steels; all the plotted values are the average of at least five indents made at each temperature*

carbon content in solution falls to about 0·2%, then, using Winchell and Cohen's data,[1,2,12] the corresponding hardness due to carbon in solution should be 500 HV at −196°C. Artificial aging of this 0·82%C–16·7%Ni martensite after quenching leads to a maximum hardness of 815 HV at −196°C.[2] This means that precipitation after the quench is responsible for an increase in hardness of 315 HV, which is equivalent to the hardening produced by 0·8% carbon in solution. These results are shown schematically in Figure 2.

The same sort of calculation can be used to estimate the contribution due to aging during the quench in a plain carbon martensite containing 0·8%C. At −196°C this material would have a hardness of 925 HV (see previous section). The strength of a 0·2%C plain carbon martensite with all the carbon in solution can be estimated by subtracting the hardening contribution of nickel (∼75 HV) from the hardness of Winchell and Cohen's 0·23%C–26·8%Ni martensite at −196°C. This gives a value of 425 HV. If, as in the previous example, it is assumed that the carbon content in solution has dropped to 0·2% when the 0·8%C steel aged during the quench, this means that the hardening contribution due to precipitation in the plain carbon steel is 500 HV.

This value is considerably larger than the value computed for aging after the quench. One possible explanation of this difference is that the aging treatments used by Winchell and Cohen did not result in maximum hardness. However, a more likely explanation is associated with the fact that in the plain carbon steel aging occurred *during* the quench, while in the alloy steel aging was carried out *after* the quench. If it is assumed that the precipitation of carbon is dislocation nucleated, then during the quench the dislocations will be moving and continually providing new nucleation sites as they sweep through the material. In the steel aged after quenching, however, the dislocations will be stationary and consequently fewer sites will be available for nucleation. Thus a greater strengthening effect associated with the much higher precipitate density is to be expected in the steel aged during the quench.

(v) Substructure of martensite: role of dislocations and internal twinning

When martensite forms from austenite a macroscopic change of shape as well as an inhomogeneous shear are involved. These two distortions, in particular the inhomogeneous shear, result in the formation of substructure within the martensite grains. When the inhomogeneous shear is compensated by internal twinning the dislocation density within the martensite is not very high, but a large number of narrow twins are produced.[13] In low-carbon steels no internal twinning is observed. This implies that the inhomogeneous shear has been compensated by slip – a conclusion which is supported by the observation that the dislocation density in these low-carbon martensites is high.[13] By comparing the strength of fine grained low-carbon ferrites with the strength of low-carbon martensites the contribution of this dense dislocation substructure to the strength of martensite can be estimated to be 50–100 HV or 10 to 20 tons/in², a relatively small strengthening effect.

The proposal that internal twinning is partly responsible for the strength of martensite was put forward in 1960.[13] This hypothesis has met with considerable opposition and a number of arguments have been advanced to prove that internal twinning has no effect on the strength of martensite.[1,9]

Winchell and Cohen were the first antagonists of the twin hardening model and based their conclusions on the fact that their linear relationship between yield strength and the cube root of the carbon content for twinned Fe–Ni–C martensites extrapolated to a value of less than 20 tons/in² at zero carbon.[1,2] This suggests that internally twinned carbon-free martensite is relatively soft. It is now apparent that the Winchell and Cohen yield stress values should be regarded as varying linearly with the square root and not the cube root of the carbon content.[10] This extrapolates to a more sensible value of about 35 tons/in² at zero carbon. This still indicates that the effect of internal twinning at zero carbon is small or even negligible.

2 *Schematic representation of the hardness changes (at −196°C) on aging a 0·82%C–16·7%Ni martensite artificially after the quench; the data are taken from the results of Winchell and Cohen[2]; ΔHC_1 (=345 HV) is the contribution to the hardness due to 0·82%C in solution, ΔHC_2 (=170 HV) is the contribution to the hardness of the aged martensite due to the carbon still in solid solution; ΔHA (=310 HV) is the contribution to the hardness of the aged martensite due directly to the precipitation or segregation of carbon*

This conclusion is somewhat embarrassing for the Winchell and Cohen theory of hardening by carbon in solution, because these authors use the twin spacing as the factor which limits the length of the dislocation line in their model. This is tantamount to saying that either the twins or the twin boundaries act as barriers to dislocation movement. If Winchell and Cohen insist that the twins do not act as appreciable barriers at zero carbon – and this appears to be correct – then they must either postulate that the twins act as barriers in the presence of carbon or the foundation of their whole analysis collapses.

Radcliffe and Schatz have determined the hardness values of plain carbon steels quenched under a hydrostatic pressure of 42 kb and have used these values to disprove the twin hardening hypothesis.[9] They state that quenching under a high pressure will lead to the formation of a fully twinned martensite at carbon contents greater than 0·4%, whereas from the results of Kelly and Nutting,[13] obtained at 1 atmosphere pressure, fully twinned martensites are not obtained in plain carbon steels until the carbon content exceeds 0·75%. Radcliffe and Schatz then argue that, if the twin hardening model is correct, a 0·4%C steel, quenched under a high pressure to give a fully twinned structure, should have the same hardness as a 0·75%C martensite (i.e., ∼900 HV). Since the 0·4%C steel tested in this way showed an increase in hardness from about 600 HV to only 780 HV, they concluded that the twin hardening hypothesis must be incorrect. Unfortunately Radcliffe and Schatz completely misinterpreted the twin hardening model, which did *not* state that internal twinning was the sole cause of hardening, but instead proposed that twinning, along with other effects such as carbon in solution, contributed to the strength of high-carbon martensite. Correctly interpreted, the twin hardening model predicts that at a given carbon content a twinned martensite will be stronger than an untwinned martensite, and not that all twinned martensites should be of equal strength. In fact these high pressure results provide very convincing evidence *in favour of*, rather than against, the twin hardening mechanism.

The only results which relate directly to the effect of internal twinning are based on hardness measurements of virtually carbon-free iron–nickel martensites.[8–10] There is general agreement that when the martensitic structure changes from laths containing a high density of dislocations at 25%Ni to internally twinned plates at 30%Ni there is no appreciable change in strength. It could be argued that, since the dislocation density in internally twinned martensites is relatively low, then if the twins had no effect on the strength the internally twinned martensite should be weaker (by some 10 to 20 tons/in²) than lath martensite. The similarity in the strength of the two structures then leads to the conclusion that the twins provide a strengthening effect, which approximately balances the loss in strength due to the decrease in dislocation density.

Even if the above argument is correct the effect of internal twinning on the strength of carbon-free martensites cannot be greater than 10 to 20 tons/in². This poses the problem of how an internally twinned martensite plate manages to deform at stresses which are if anything only slightly higher than those required to deform an untwinned martensite lath. No satisfactory explanation of this has been advanced by any of the antagonists of the twin hardening model. The mechanism for

the incorporation of slip dislocations in mechanical twins put forward by Sleeswyk and Verbraak[16] may well provide the answer to this problem. These workers suggest that dislocations with the three $a/2 < 111 >$ Burgers vectors, which are not common to both twins, may pass through twins in bcc metals by dissociation into two dislocations. This process would require very little additional energy and could explain the similar hardness values for twinned and untwinned iron–nickel martensites.

It is now clear from recent work on the internal structure of ferrous martensites that not only is there considerable variation in the twin spacing in a given alloy (contrary to the evidence reported by Cohen[3]) but in addition the internal twins do not always extend throughout a martensite plate.[17–20] Hence, when comparing results from twinned and untwinned martensites it must be remembered that twinned martensite is unlikely to be 100% twinned. As a result any effect due to twinning will be 'diluted' and will never appear to its fullest extent.

COMBINATIONS OF STRENGTHENING EFFECTS

In considering the possible combinations of the various strengthening effects the contributions of grain size and of substitutional solid solution hardening will be neglected since both are relatively small. The contributions to hardening of substructure and of twinning on their own are are also small, but there is the possibility that solid solution hardening by carbon and precipitation hardening may be more effective in twinned as opposed to lath martensite. The effect of substructure will therefore be included in the various hardening combinations to be considered. As there appears to be a difference between aging during the quench and aging artificially after the quench, these two types of aging will be differentiated in the subsequent combinations. The following hardening effects will therefore be considered in their possible combinations:

(1)   carbon in solid solution
(2a) aging during the quench
(2b) aging after the quench
(3a) substructure – dislocations
(3b) substructure – internal twinning.

It is possible, in principle and in practice, to separate the two types of aging process, so combinations involving a mixture of both types of aging will not be considered. For the same reason combinations involving a mixture of the two types of structure will also be omitted. It is impossible for any form of aging to occur without some carbon being present in solution, so all combinations involving aging must include the effect of carbon in solution. Finally, in any combination the type of substructure must always be specified. This leaves the following possible combinations:

(i)	$3b + 1$	carbon in solution in twinned martensite
(ii)	$3a + 1$	carbon in solution in lath or untwinned martensite
(iii)	$3a + 2a + 1$	aging during the quench in lath martensite
(iv)	$3b + 2a + 1$	aging during the quench in twinned martensite
(v)	$3a + 2b + 1$	aging after the quench in lath martensite

Solid solution hardening by carbon and nitrogen in ferrous martensites

M. J. Roberts and W. S. Owen

SYNOPSIS

The variation of the flow stress of a martensitic Fe–21·3%Ni–C alloy with carbon content (up to 0·15%C) and with temperature (20° to −196°C) has been measured. The results are compared with those obtained on an Fe–31%Ni–C series of alloys and on Fe–N alloys (0·10 – 0·12%N) and with the Winchell and Cohen results on Fe–Ni–C alloys with constant Ms temperature and with data relating to the flow stress of ferrite. For all the martensites the relationship between flow stress (τ_f) and atom fraction of carbon (X_c) is represented best by $\tau_f = \tau_i + mX_c^{\frac{1}{2}}$. The effects of changes in the structure of the martensite and the testing temperature on the value of the gradient m are discussed and m is compared with the values predicted by the current theories of solid-solution hardening. The Fleischer theory gives the best agreement. The flow stress of a carbon-free iron, τ_i, is found by extrapolation and it is shown that this stress increases with decreasing temperature. Thus, it is concluded that only a fraction of the variation of the flow stress with temperature is due to thermally activated unpinning of a dislocation line interacting with an interstitial atom. The variation with temperature revealed by the experimental results is compared with the predictions of the theories of Heslop and Petch, Fleischer, and Friedel.   2616

INTRODUCTION

ALTHOUGH solid solution hardening by interstitial solute atoms is seldom the only hardening mechanism contributing to the strength of martensitic iron alloys, it is always a major effect. In this paper only results obtained using alloys and experimental conditions designed to prevent the segregation of carbon and nitrogen atoms are considered and consequently a random distribution of solute atoms will be assumed.

All modern theories of solid solution hardening adopt certain basic concepts first defined by Mott and Nabarro.[1,2] It is assumed that the stress required to bow out a dislocation loop between neighbouring pinning points in the slip plane is greater than that necessary to move a segment of the dislocation line through the algebraic sum of the interaction forces of random

sign acting on the segment. The theories differ in the assumptions made about the magnitude and distribution of the interaction forces and the length of the segment on which they act. All are agreed that an asymmetrical distortion, such as that introduced into an iron lattice by carbon or nitrogen, produces a strong interaction and consequently 'rapid hardening'.[3] Winchell and Cohen[4,5,6,7] proposed a model in which carbon atoms produce 'distortion dipoles', the shear stresses resolved on the slip plane being periodic. They then calculated the root-mean-square force acting on a straight dislocation segment of length L and obtained

$$\tau_f = K\,L^{-\frac{1}{2}}\,X_c^{\frac{1}{2}} \quad\text{...(1)}$$

Where τ_f is the flow stress in shear, X_c the atom fraction of carbon and K a constant which can be evaluated. Comparing equation (1) with experimental data they concluded that L is of constant length $\simeq 10^{-6}$ cm and they suggested that this parameter is determined by the thickness of the twin plates in acicular martensite. The model was not tested against data obtained from martensite with only dislocations in the substructure. Petch,[8,9] considering the solution hardening of ferrite by carbon plus nitrogen, also assumed the dislocation line has limited flexibility and, following Mott,[2] considered the motion of a compound loop containing N^2 single loops of dislocation. They deduced that the frictional stress (the flow stress at zero strain in the absence of dislocation pinning) increases linearly with concentration of interstitial solute, and this contention is supported by experimental results.[8] Both the Fleischer[3,10] and the Friedel[11,12] theories assume a dislocation line which can be bent through a radius of the order of the Burgers vector b, and thus the line is ideally flexible. Since the force between a dislocation and a solute atom is essentially a short range force, two types of forces acting on a dislocation moving in a slip plane can be distinguished: those which are produced by solute atoms lying in the slip plane or adjacent planes and which therefore interact strongly with the dislocation line and those internal stresses arising from atoms located in more remote planes. Fleischer[3] considers the second type to be negligible and this theory predicts a flow stress which increases as $X_c^{\frac{1}{2}}$, at 0°K. It is assumed generally that the Peierls–Nabarro force is negligible, although a stress equal to the internal long range

stress is introduced when considering the specific case of the hardening of ferrite. The Friedel theory predicts also that τf increases as $Xc^{\frac{1}{2}}$ and, in this case, it is stated explicitly that the Peierls–Nabarro stress is neglected.

The increase of the flow stress with decreasing temperature is much more marked in bcc than in fcc metals. Frequently, it has been suggested that this marked effect of temperature is due to the separation of a dislocation from an interstitial atom being assisted by thermal activation.[13] Thus, it is argued that if the carbon and nitrogen are removed from iron the flow stress should be less dependent upon temperature. Although carbon and nitrogen can be reduced to a level below that at which a discontinuous yield or strain aging can occur, to date there is no clear evidence that the rise in flow stress with decreasing temperature is affected significantly. The data available have been reviewed by Low.[14] Heslop and Petch[9] estimated the activation energy for the jump of a dislocation loop pinned by carbon or nitrogen atoms to be 1·5–2·2 eV, and thus the contribution of thermal activation to the unpinning cannot be significant. They concluded that the temperature variation of the flow stress is most likely to be due to a change in the Peierls–Nabarro stress with temperature. This view is not shared by Fleischer and Friedel and both theories assume that the variation of the flow stress with temperature is due to the thermally activated unpinning of dislocation loops. The precise relationship between τ_f and T is dependent upon the assumptions made about the variation of the interaction force with distance close to the dislocation, and the two theories predict rather different relationships. Friedel[11,12] considers a general case but Fleischer[15] analyses the effect of temperature only in relation to the flow of lithium fluoride but, with minor adjustments, the result can be applied to solution hardening by carbon and nitrogen. The object of the present work was to provide experimental data relating to the effects on solid solution hardening of ferrite by interstitial atoms of structural variables such as martensitic substructure and the ordering of the <001> distortions. At the same time data has been collected from which it is possible to test the theoretical predictions about the effects of solute concentration and testing temperature on flow stress. The only data available previously were obtained by Winchell and Cohen[4,5] on internally twinned acicular martensite in Fe–Ni–C alloys containing between 0·02 and 0·82%C and data[8,9,16,17,18] relating to the flow stress of dilute solutions of carbon and nitrogen in ferrite (0–0·03%C). The new experimental results were obtained using a series of Fe–31%Ni alloys containing between 0·20 and 0·30%C, a series of Fe–21·3%Ni alloys with between 0·03 and 0·15%C, and some Fe–N alloys (0·10 to 0·12%N).

EXPERIMENTAL

All the specimens were made from sheet 0·008 to 0·015in thick. The Fe–Ni–C alloys were first decarburized in hydrogen and then recarburized in hydrogen–methane to the required carbon concentration. The Fe–N alloys were prepared by nitriding BISRA iron (number V810) in ammonia–hydrogen.[19] The Fe–Ni–C specimens were annealed at 925°C and cooled rapidly to room temperature. The Fe–N specimens were quenched from 910°C in brine in the nitriding equip-
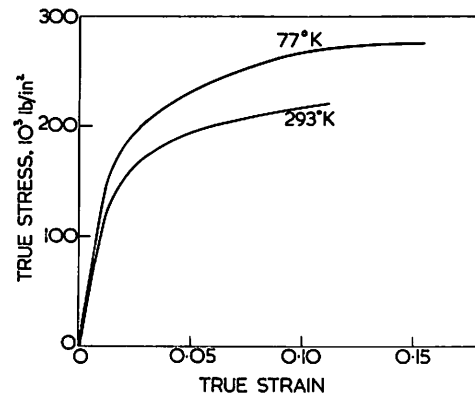
1   *Stress–strain curves for Fe–21·3%Ni–0·11%C martensite tested at 77° and 293°K*

ment. All specimens which could not be tested immediately were stored in liquid nitrogen. Specimens, which were electropolished, were tested at temperatures within the range 20° to −196°C in a Polanyi tensile machine, the load being measured by a Langham–Thompson dynamometer. Although the cross-sectional dimensions were measured with the greatest care, inaccuracies in the values for the area produced the major error in the measurement of the flow stress.

The tensile specimens were examined by optical metallography after test to determine the mode of deformation from the surface markings. All the specimens had appreciable ductility even at −196°C so that no error in the measurements of the flow stress was introduced by premature brittle failure.

Typical load–elongation curves for the Fe–21·3%Ni–C series are shown in Figure 1. In no case did the curves indicate the presence of a discontinuous yield although the machine and testing procedure is known from other experiments to be capable of detecting yield discontinuities in specimens aged only slightly. Thus, it was confirmed that the specimens, although martensitic, were not aged and, consequently, it is very unlikely that any precipitation had occurred. Thus, the hardening must be due entirely to the martensitic structure and the interstitial atoms in solution. The flow criterion adopted was the stress at 0·2% strain (0·2% proof stress) because it was very difficult to determine the elastic limit with sufficient accuracy. The Petch frictional stress σ_i was measured for some of the curves by extrapolation of the strain-hardening curve to the elastic line.[21] Although the agreement with the elastic limit was reasonable, σ_i was generally a little greater and it was decided that the 0·2% proof stress was a more reproducible measure of the flow stress.

The variation of the 0·2% proof stress with carbon content and temperature for the Fe–21·3%Ni alloys is shown in Figure 2 and for the iron–nitrogen alloys in Figure 3. In both cases the flow stress at constant temperature increases with concentration of solute and the flow stress at constant solute content increases markedly with decreasing temperature. For a constant testing temperature, the variation of flow stress in shear (τf) with atomic fraction of carbon (Xc) was represented well by the relationship

$$\tau_f = \tau_i + mX_c^{\,n} \qquad\qquad (2)$$

where τ_i is the flow stress in shear of iron with zero carbon

2 *Variation of the 0·2% proof stress of Fe–21·3% Ni martensite with carbon content between 0·04 and 0·15% and with temperature*

concentration, m is the slope, and the index n is taken to be 0·5. All the known values for the flow stress of martensites and low-carbon ferrite are plotted as a function of the square root of the atom fraction of interstitial solute in Figure 4. All the alloys in the Fe–21·3% Ni series were quenched to 77°K and they did not contain any retained austenite. The Fe–31% Ni series did contain an appreciable volume fraction of austenite which decreased as the temperature of the specimen was lowered. For one carbon content, a series of specimens was prepared covering a range of retained austenite content and it was found that for these specimens the flow stress, measured at 293°K, increased linearly with decreasing volume fraction of austenite. For the other carbon levels a similar linear proportionality was assumed. All the specimens in the main series of tests were quenched to 77°K and the volume fraction of austenite was measured. The flow stress of a specimen with 100% martensite was found by simple proportionality. A

3 *Variation of the 0·2% proof stress of Fe–N martensite with nitrogen content between 0·1 and 0·12% and with temperature.*

4 a *Flow stress of ferritic and martensitic iron alloys as a function of the square root of the atomic fraction of interstitial solute;* **b** *as Figure 4a; data for the Fe–21·3% Ni series plotted separately*

similar correction was made by Winchell and Cohen to the data from the series of Fe–Ni–C alloys with constant M_s temperature. Throughout, the shear stress has been taken as half the tensile proof stress and an allowance of 450 lb/in² per unit weight percent of nickel has been made for the contribution to the flow stress from solid-solution hardening by nickel. The values of the flow stress have been reduced to relate to a base alloy containing 0% Ni. Of course, this datum composition is arbitrary and the stresses are not comparable directly with those for an iron–carbon alloy because the linear variation of proof stress with nickel content used to make the correction does not apply at nickel contents below about 10%. In the interpretation of the results great significance attaches to small differences in the gradient m (Figure 4) and thus it is necessary that this parameter be measured with the best possible accuracy. For the Fe–21·3% Ni series measurements of the flow stress of some alloys at the extremes of the composition range were repeated two or three times and the lines

in Figure 4 were fitted through the experimental points by the method of least-squares.

Reasonable linear plots of the experimental data are obtained if a value of $n = \frac{1}{3}$ is adopted instead of $n = \frac{1}{2}$ and thus, on this basis, it is not possible to distinguish between the Winchell and Cohen and the Fleischer and Friedel theories. However, if the value of $n = 1$ is adopted the deviations from linearity are large and clearly the experimental data are not compatible with the theory of Cracknell and Petch.

At testing temperatures between 20° and —78°C the flow stress of Fe–N and Fe–Ni–C alloys with the same atom fraction interstitial solute was almost exactly the same. At —196°C the results for the Fe–N alloys are a little lower than for the corresponding alloy containing carbon, although there is some scatter in the results. There is very little information about the strength of Fe–N alloys available.[23] The few results on poly-crystalline iron[24] refer to more dilute alloys than those in the present series tested at temperatures above —78°C. They show also that solution hardening by nitrogen is virtually the same as by carbon. Since, in the iron lattice, the two interstitial atoms are almost exactly the same size[25] the result is not surprising but it is contrary to the frequently quoted belief that nitrogen is a less effective hardener than carbon. The only known results on Fe–N alloys tested at —196°C are some single crystals data reported by Edmondson[26] who found that nitrogen is significantly less effective than carbon in raising the yield stress at —196°C. From the present results it appears that the effect occurs also in polycrystalline specimens.

Although the gradient for the Winchell and Cohen alloys is exactly the same as for the 31%Ni series the flow stress of these alloys was some 20 000lb/in² higher than for the present alloys of the same carbon content. This discrepancy can largely, but not entirely, be accounted for by the fact that Winchell and Cohen measured the 0·6% proof stress whereas all the other martensite data refer to a 0·2% proof stress. Also there may have been a difference in the austenite grain size and the size of the martensite plates in the two series.

Values of the gradient m (equation (2)), assuming $n = 0.5$ (Figure 4), are given as a multiple of the shear modulus in
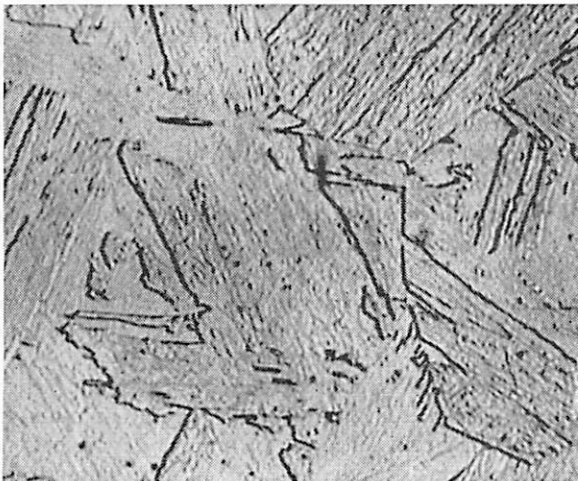
5   *Massive-martensite structure in Fe–21·3%Ni–0·06%C; etched in alcoholic ferric chloride*   ×500
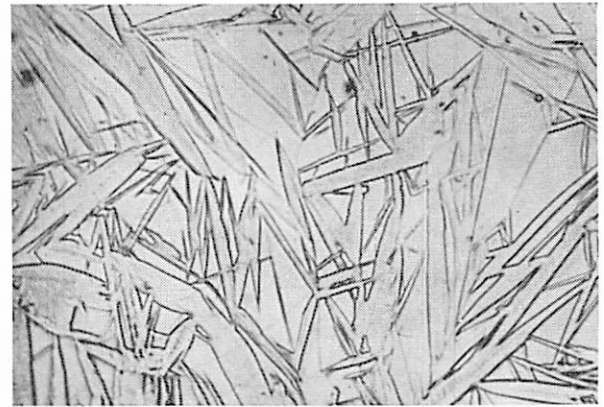
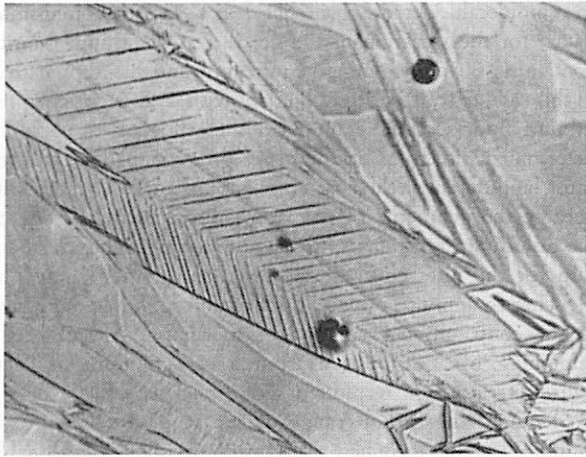6   *Acicular martensite structure in Fe–31%Ni–0·3%C; electroetched in perchloric–acetic acid solution*   ×1000

Table I. The variation of modulus with temperature is taken into account[27]; $10^{-6}\mu = 13.30 - 0.0030T$. The Fe–N series did not cover a sufficiently wide range of nitrogen concentration to enable the gradients to be determined. With the exception of Wert's data[28] at 20°C, which gives roughly the same result as found for the martensites, all the gradients reported for the solution hardening of ferrite by carbon and nitrogen are appreciably smaller at all testing temperatures than those for the martensites. Wert[28] measured the carbon in solution by internal friction, but the other investigators assumed that the dissolved carbon concentration was the same as the total concentration of carbon in the alloy. The latter assumption was probably not justified because carbon was probably removed from solution by the growth of undissolved carbide particles or by precipitation at grain boundaries. Thus, it is concluded that the only reliable value of m for ferrite is that obtained by Wert and that the increase of flow stress with interstitial concentration is about the same in ferrite and martensite.

The difference in gradient m between the 31%Ni and the 21·3% series tested at 273°K and 293°K respectively, reported in Figure 4 and Table I, is difficult to assess quantitatively because of the experimental scatter, but it appears to be real. The 21·3%Ni series was selected because the structure of the transformed alloy is quite different from that of the 31%Ni series and the Winchell and Cohen series. The lower nickel series form massive martensite (Figure 5) which has cubic symmetry, dislocations only in the substructure, and a micro-structure made up of packets of parallel shear plates.[25] The martensite formed in Winchell and Cohen alloys and in the 31% nickel alloys is acicular (Figure 6); the lattice is tetragonal and the substructure is predominantly made up of parallel twin plates.

After deformation at all temperatures between 77° and 293°K wavy slip lines, but no twin traces, were found on the prepolished surfaces of all the specimens of the 21·3%Ni series. However, the predominant mode of deformation of the 31%Ni alloys was by twinning (Figure 7) although some slip markings could also be seen. These observations are in line with Richman's[20] report of the deformation occurring in the Winchell and Cohen alloys. On deforming at 20°C, he found deformation by wavy slip in alloys with 0 to 0·05%C. At

7 *Deformation twinning in acicular–martensite in Fe–31% Ni–0.3% C; electroetched in perchloric–acetic acid solution; for undeformed structure see Figure 6* ×900

higher carbon levels some twinning was found, the proportion increasing with carbon concentration until in alloys containing more than 0.4% C no slip could be detected. All the alloys under consideration here contained less than 0.4% C. It is improbable that the difference in gradient *m* between the massive and acicular martensite is due to a change in the deformation from slip to twinning because the intrusion of twinning is expected to decrease *m*, not increase it. Thus, it must be assumed that, although in some alloys twinning occurs together with the slip, in alloys with less than 0.4% C the first deformation is mainly by slip and that it is this form of deformation which determines the flow stress. This is an aspect of the problem which warrants more detailed investigation. Discounting the influence of deformation twinning, one of two explanations of the slightly larger value of *m* for the acicular martensites seems possible. It may be a result of substructure hardening due to the presence of the twins as proposed by Kelly and Nutting,[29] although if this is so it is not clear why the magnitude of the effect should increase with carbon content. Alternatively, loss of strength in the massive martensites may be due to the <001> distortions adopting randomly the three possible directions in this structure. Considering the effect of disordering similar distortions in an fcc structure, Fleischer[3] has shown that as many as half the distortions may have no interaction with a given dislocation line, but Kelly[30] has pointed out that similar arguments should not apply in the case of a dislocation moving in a bcc lattice. If an effect due to the difference in symmetry of the two types of martensite had been present a much bigger difference in the gradient than that found would be expected. In the massive martensite there are no twin plates and thus the length *L* in the Winchell–Cohen formula (equation (1)) cannot be more than a few atom diameters suggesting that, for the same carbon content, the flow stress should be more than ten times that for acicular martensite. A short extrapolation of the data in Figure 4 shows clearly that this is not so and, consequently, the experimental results provide no support for the Winchell–Cohen concept.

The Fleischer model

The model gives the gradient at 0°K as $\frac{\mu\Delta\varepsilon}{3}$ (Equation 2).

When discussing Wert's results[10] Fleischer introduces an internal long-range stress equal to the flow stress of a specimen with zero carbon content, so that

$$\tau_t = \tau_i + \frac{1}{3}\mu\Delta\varepsilon X_c^{\frac{1}{2}} \quad\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (3)$$

where $\Delta\varepsilon$ is the difference between the longitudinal and transverse strain caused by an interstitial atom. For a carbon atom in the bcc iron lattice $\Delta\varepsilon = 0.38$, using Roberts'[31] data for the variation of lattice parameters with carbon concentration. Then the gradient at 0°K predicted by Fleischer is 0.13μ. The experimental value at −196°C is 0.065μ and the data in Table I suggest that at low temperatures the gradient increases with decreasing temperature. Thus, at 0°K the agreement between theory and experiment is probably quite good.

Data which would enable the temperature dependence of the flow stress of martensite to be compared with the theoretical predictions has not previously been available. Of the present data, only the results from the 21.3% Ni alloy series and the Fe–N alloys cover a sufficient range of testing temperature without a change in the mode of deformation and, unfortunately, the Fe–N alloys cover only a small range of nitrogen content. Thus, the discussion will be concerned mostly with the data relating to massive martensite in low-carbon alloys containing 21.3% Ni. Since the central problem is the extent to which the variation of flow stress with temperature can be attributed to the thermally activated unpinning of dislocations interacting with carbon atoms, it is important to know the variation of flow stress of a carbon-free iron with temperature. It is evident from the data in Figure 4 that the linear plots for the 21.3% Ni alloys tested at 20°, −78°, and −196°C cannot intersect at $X_c = 0$, and consequently τ_i must vary with temperature. In fact, τ_i increases markedly with decreasing temperature (Figure 8) and it is a major part of the temperature-dependent component of the flow stress. The reason for this variation is obscure; the most probable explanation is that the Peierls–Nabarro stress increases with decreasing temperature as proposed by Heslop and Petch.[9]

TABLE I The gradient of the flow stress in shear versus the square root of the atom fraction of interstitial solute in various polycrystalline iron alloys

Alloy	Testing temperature, °K	Gradient		Source
Dilute	293	$\mu/20$	0.05μ	Wert[28]
solutions	293	$\mu/34$	0.029μ	Allen *et al.*[17]
of carbon	293	$\mu/35$	0.028μ	Heslop and Petch[9]
and	213	$\mu/31$	0.032μ	Allen *et al.*[17]
nitrogen	147	$\mu/31$	0.032μ	Heslop and Petch[9]
in ferrite	77	$\mu/25$	0.040μ	Allen *et al.*[17]
	77	$\mu/30$	0.033μ	Heslop and Petch[9]
Fe–Ni–C				
15–31% Ni	273	$\mu/16.1$	0.062μ	Winchell and Cohen[4,5]
31% Ni	273	$\mu/16.1$	0.062μ	This investigation
21.3% Ni	293	$\mu/19.5$	0.051μ	This investigation
21.3% Ni	195	$\mu/17.1$	0.059μ	This investigation
21.3% Ni	77	$\mu/15.4$	0.065μ	This investigation
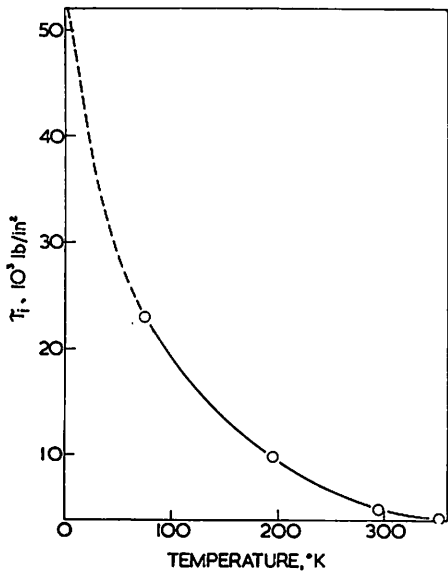
8   *Variation of τ_0 with temperature for the Fe–21·3%Ni–C alloys*

However, because there is a small increase in the gradient *m* (Table I) with decreasing temperature there is also a contribution from thermally activated unpinning. The increase of $(\tau-\tau_0)$ with decreasing temperature is shown in Figure 9 for the 21·3%Ni series and in Figure 10 for the Fe–N alloys. Thus, the present results suggest that in the Fe–Ni–C series roughly two thirds of the increase in flow stress between T_0 (350°K) and 77°K can be attributed to the variation of τ_i with temperature and one third to a thermally activated process involving the carbon atoms. If τ_0 is a shear stress defined as in Figure 12, $(\tau-\tau_0)$ increases more rapidly with decreasing temperature the higher the carbon content, although the effect is not large. Data for the extremes of the carbon range are shown in Figure 9. The experimental determination of the fraction of the increase in flow stress which is due to the increase in τ_i with decreasing temperature is very sensitive to variations in the gradient *m* and consequently it is subject
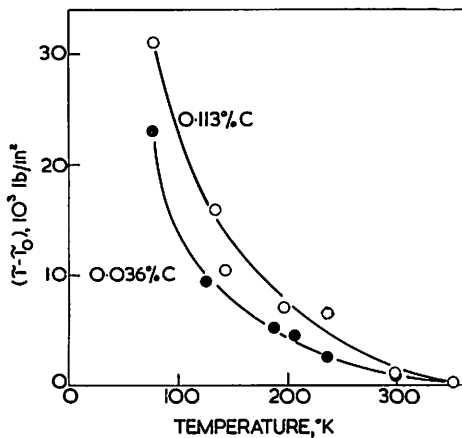
to considerable error. Fleischer[15] has analysed Johnston's data[32] for the variation of the flow stress of lithium fluoride containing 80 ppm of magnesium ions and has shown that, in this case, $(\tau-\tau_i)^{\frac{1}{2}}$ increases linearly with $T^{\frac{1}{2}}$. Since very pure lithium fluoride can be obtained the value of τ_i could be determined directly. Although the proportionality constants must be different if the analysis is applied to Fe–C martensite because the geometry of the lattice distortion in relation to the dislocation is different, if the same force–distance relationship for the interaction is assumed the variation shown by Fleischer to apply to doped lithium fluoride should apply also to the contribution made by the thermal activation of the unpinning of a dislocation from a carbon atom in Fe–C martensite. $(\tau-\tau_0)^{\frac{1}{2}}$ is plotted as a function of $T^{\frac{1}{2}}$ for the extremes of the carbon range of the Fe–21·3%Ni series in Figure 11. The Fleischer relationship is obeyed reasonably well and, as expected, the slope increases a little with increase in the carbon content.
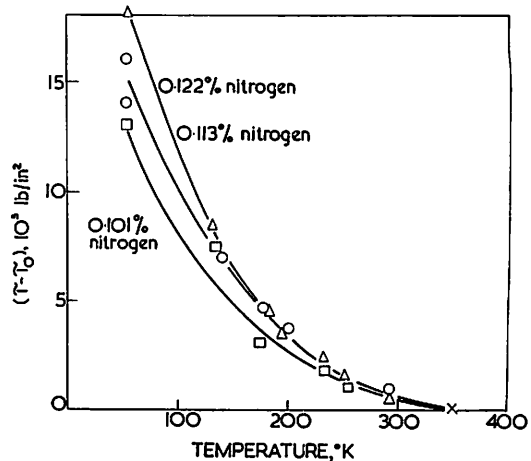
The Friedel model

Friedel assumes that all of the change in flow stress with temperature can be accounted for on the basis of a thermally-activated unpinning model and, for conditions appropriate to the present argument obtains

$$\tau_f-\tau_0 = (\tau_c-\tau_0)\,(1-\frac{T}{T_c})^{3/2}\quad\text{......(4)}$$

where τ_c is the flow stress at 0°K and T_c is the temperature at which τ_f reaches a constant minimum value (Figure 12). In practice, to obtain reproducible values for T_c it is necessary to adopt an arbitrary definition of T_c. Conrad and Hayes[22] define T_c as the temperature at which $\frac{d\tau_f}{dT}=5\times10^{-3}$ kg/mm²/ °K. Applying this criterion to the curves in Figures 2 and 3, $T_c\cong350°K$ which is in good agreement with the previously reported temperature for dilute alloys in iron[22] and with the suggested relationship[22] $T_c=0·22\,T_m$, where T_m is the melting point. According to Friedel[12]
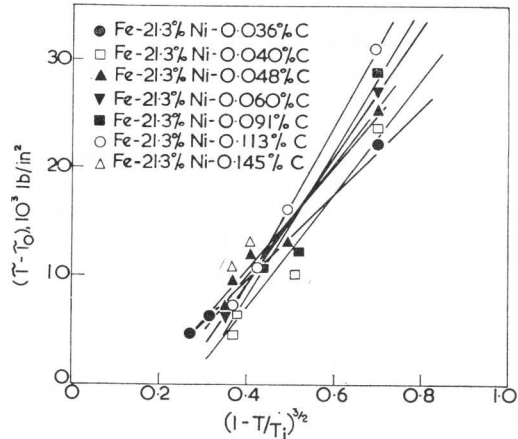
$$kT_c\cong\frac{U_m}{15}\quad\text{.....................(5)}$$

9   *Variation of $(\tau-\tau_0)$ with testing temperature for the Fe–21·3%Ni series of alloys*

10   *Variation of $(\tau-\tau_0)$ with testing temperature for the Fe–N alloys*
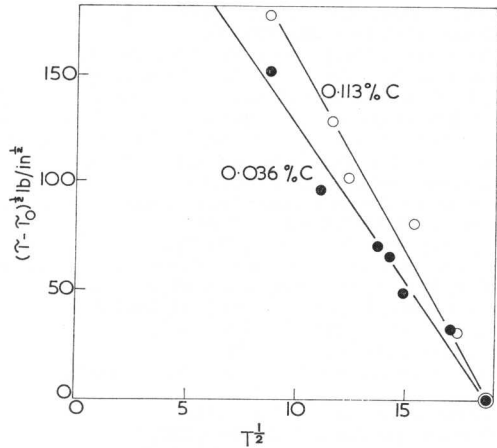
12 $(\tau - \tau_0)^{\frac{1}{2}}$ *as a function of* $T^{\frac{1}{2}}$ *for two Fe–21·3% Ni–C alloys*

13 $(\tau - \tau_0)$ *as a function of* $(1 - \dfrac{T}{T_c})^{3/2}$ *for the Fe–21·3% Ni–C alloys*

which gives values of T_c between 350° and 620°K depending upon which of the possible values of the maximum binding energy between a dislocation and a carbon atom (U_m) is adopted. In this theory τ_0 is assumed to be independent of temperature and to be the value of the flow stress at T_c (Figure 12). Then, assuming the value of T_c derived from the experimental curves to be correct,

$$\tau_f - \tau_0 = \frac{\mu X_c^{\frac{1}{2}}}{30} \left(1 - \frac{T}{350}\right)^{3/2} \dots\dots\dots\dots\dots\dots\dots\dots\dots(6)$$

This predicts correctly that the flow stress at constant temperature is a linear function of $X_c^{\frac{1}{2}}$, but it also predicts that the gradient m at 0°K is $\dfrac{\mu}{30}$ $(0·033\mu)$ which is a smaller value than that predicted by Fleischer and thus further removed from the probable value obtained by extrapolation of the experimental data. At 77°K, the experimental value of m is $0·065\mu$ and that predicted by equation (4) is $0·023\mu$ if T_c is taken as 350°K and $0·027\mu$ if it is taken as 620°K. Thus, there is agreement within an order of magnitude. The variation of the flow stress with temperature predicted by Friedel is tested by the data plotted in Figure 13. Although the predicted trend

12 *Schematic variation of flow stress with temperature illustrating the definition of* T_c, τ_0, *and* τ_c *in the Friedel model*

is found the scatter in the experimental data is accentuated and the plots do not provide convincing evidence for the validity of equation (4). In all respects, the less sophisticated model proposed by Fleischer appears to give better agreement with experimental results. The relative failure of the Friedel model supports further the proposition that at least a substantial proportion of the increase in flow stress with decreasing temperature can be attributed to an increase in τ_i.

CONCLUSION

Analyses of the type discussed in this paper magnify greatly any errors in the experimental data and consequently the quantitative conclusions must be subject to correction when more accurate experimental data are obtained. In particular, the situation would be improved greatly if the variation of the flow stress of pure iron with temperature was known by direct measurement. Qualitatively, it is concluded that;

1. At temperatures between 20° and −78°C the flow stress of Fe–N martensite is about the same as that of Fe–Ni–C martensite with the same concentration of interstitial solute, but there is some evidence to suggest that at −196°C the flow stress of Fe–N martensite is lower.

2. At constant testing temperature, the flow stress of Fe–Ni–C martensite increases as the square root of the concentration of carbon as predicted by the Fleischer and the Friedel models but not in agreement with the theory of Cracknell and Petch. The accuracy was not sufficient to exclude the Winchell and Cohen theory on the basis of the variation of the flow stress with concentration.

3. The increase of flow stress with carbon concentration is nearly the same in massive as in acicular martensite, the increase being just a little greater in the twinned structure. The result for the massive martensite invalidates the Winchell and Cohen theory since this requires the presence of twin plates to restrict the flexibility of the dislocation line.

4. A significant proportion of the increase of flow stress with decreasing temperature can be attributed to the increase of τ_i, the flow stress of carbon-free iron. The remaining increment of stress varies with temperature in the manner predicted by Fleischer.
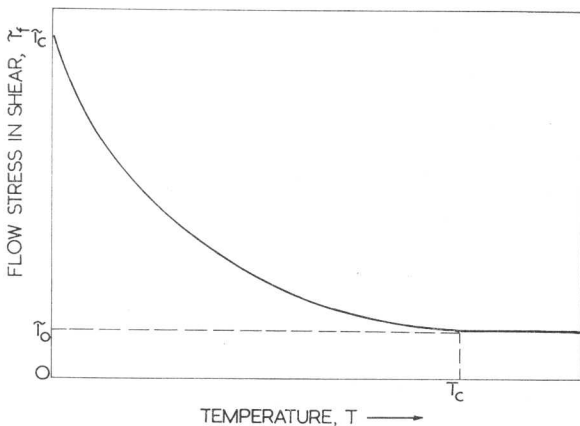
5. Both the Fleischer and the Friedel models predict the gradient of the variation of the flow stress at constant temperature with the square root of the carbon concentration to within an order of magnitude, the Fleischer value being the nearer of the two at $0°K$.

6. The variation with temperature of that part of the flow stress which depends upon thermally activated unpinning is described quite well by the Fleischer model. The Friedel description of the increase of the flow stress with decreasing temperature is not completely satisfactory, probably because it does not permit a variation of τ_i with temperature.

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Discussion 7

*Chairman: Mr J. E. Russell (English Steel Corporation Ltd)*

Professor H. W. Paxton (Carnegie Institute of Technology) said that he had a general question for Professor Owen and Dr Kelly. They had chosen to use the stress for 0·2% plastic strain; Winchell and Cohen, he believed, used that for 0·6% plastic strain.

He wished to mention some experiments carried out which gave him a little pause as to whether these were, in fact, the right kind of stresses to use if one was talking of resistance to dislocation movement. Experiments carried out on iron–nickel–carbon alloys by McEvily *et al.*[1] involved an attempt to prevent aging during the quench together with tensile tests made with a very sensitive extensometer at 77°K.

For martensites containing 0·02–0·6%C, none of which was

precipitated, the initial stages of the stress–strain curve were virtually identical up to *plastic* strains in the range 0·1–0·2%. For specimens allowed to age at room temperature for times of the order of 1 h, the shape of the stress–strain curve was quite different and became sensitive to carbon content (Figures *A* and *B*).

Thus, any measurements on martensite which did not clearly separate the effects of the quenched-in dislocations and their work-hardening response from the effects of aging must be regarded with caution. In particular, the assignment of an arbitrary plastic strain for comparison of materials of different carbon
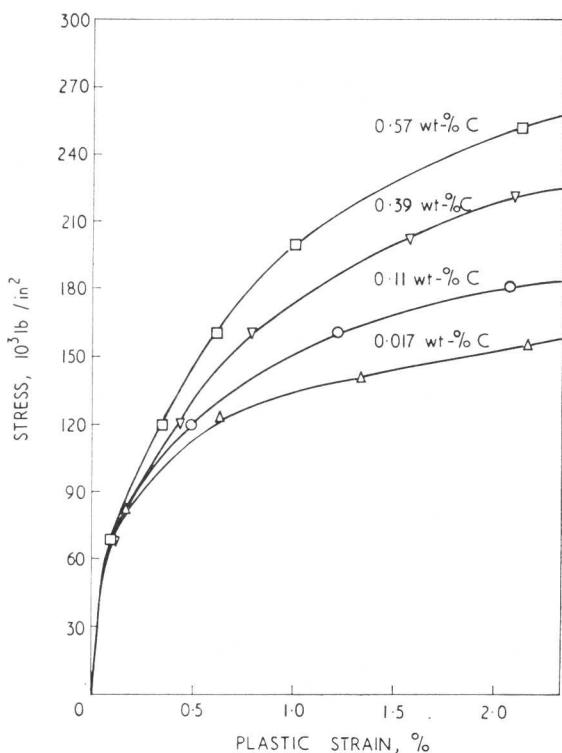
A *Stress–plastic strain curves, obtained at 77°K, of as quenched martensites; the respective carbon contents are shown*
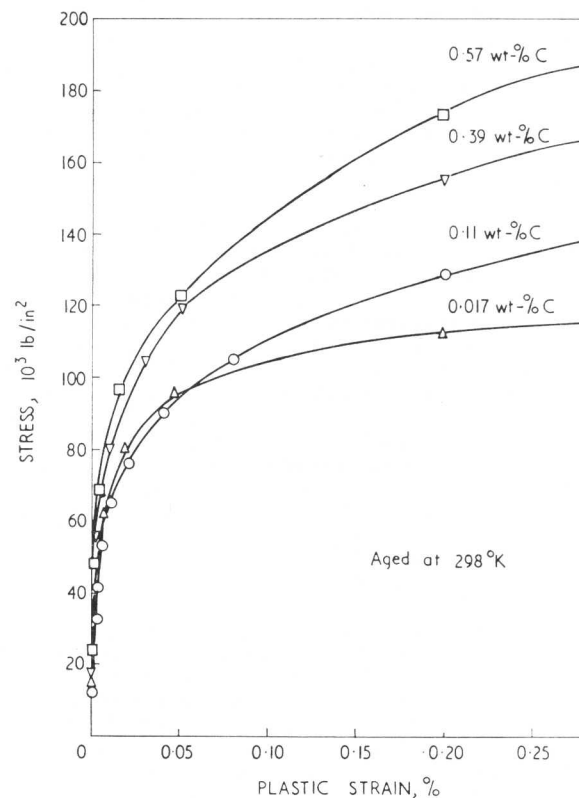
B *Stress–microplastic strain curves, obtained at 77°K, of martensites aged at room temperature*
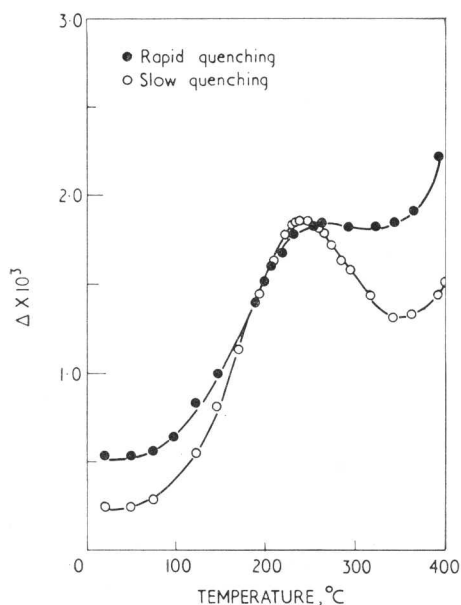
C   Effect of quenching rate on the internal friction peak in a 0·75%C commercial steel; showing that a slow cooling rate produces a large peak, while a rapid cooling rate produces a small peak

contents appeared to be questionable. Hardness measurements were even more unconvincing.

Mr F. B. Pickering (The United Steel Cos. Ltd) said that the results which Dr Kelly had shown, breaking down the strengthening contributions of aging and carbon in solid solution to that of martensite, were of the utmost interest as they indicated that aging played a prominent role in the strength of martensite. He had a similar breakdown of strengthening effects in polygonal ferrite structures, in which 0·01%C in interstitial solution caused an increase in yield stress of 3·6 tons/in² while 0·01%C precipitated to give maximum quench aging at room temperature increased the yield stress by ~6·0 tons/in². Thus carbon precipitated during aging was about 1·7 times as effective as carbon in solution as a strengthening agent. Was this of the same order as the relative effects observed in martensite?

In the aged martensite, could Dr Kelly give any idea as to the nature of the aging; was it as dislocation atmospheres, zones, precipitates? The internal friction work on martensite might help to give some clue to what was happening, because in technological martensites, which were most probably aged, there was no Snoek peak but a well defined Köster peak which was dependent on the carbon content. This suggested that the carbon present was all associated with dislocation, but that there was little present in truly random interstitial solution. Also, if the martensite was cold worked, the Köster peak did not increase in intensity, which indicated that all the carbon was initially associated with the dislocations in the quenched martensite. Did Dr Kelly think that the internal friction technique might be useful in studying aging effects in martensite?

Mr I. F. Hughes (Battersea College of Technology) said that he had been working on the internal friction of pure iron and 0·025% iron–nitrogen alloys and had found that, on quenching pure iron specimens after 2½ days in wet hydrogen, a structure of massive α–grains and block–like martensite was produced which gave a peak at about 250°C. This peak could be tempered away.

He had also been investigating the effect of this 'massive' structure and the effect of tempering it at 570°C on the transient damping of pure iron and the aging characteristics of the iron–nitrogen alloys. Results indicated that the quenched structure

aged at five times the rate of the tempered specimens despite the morphology of the structure remaining the same after tempering.

Mr J. N. McGrath (University College, Cardiff) said that on measuring the internal friction of a commercial 0·75%C steel in the martensitic condition it was found that the peak size depended on the quenching rate. Slow quenching produced a large peak while rapid quenching gave a small peak (Figure C). Subsequent tempering of the rapidly quenched sample did not produce an increase in the size of the 250°C peak. It was suggested that some form of aging or autotempering occurred in the case of the more slowly cooled samples. The observations would then be in line with the hardness measurements reported by Dr Kelly, who found that on increasing the nickel content of his steels and thus reducing the M_s, the as-quenched hardness decreased. These softer steels could not be aged to give the hardness reported for the lower nickel steels. Dr Kelly attributed the higher hardness of the lower nickel steels to precipitation during quenching as a result of their higher M_s temperatures. The similarity of these two sets of observations was striking.

Dr J. W. Christian (University of Oxford) said, on the point about internal friction work and the difference between martensite and ferrite, one thing he noticed from Professor Owen's work was that there was a small temperature dependence due to carbon content, whereas people who had worked with iron containing small amounts of carbon in it found that almost all the temperature dependence seemed to be independent of carbon content. He wondered whether this difference was not in fact due to the way in which the structures had been produced. If one simply took nominally carbon-free iron and cooled it, it was almost impossible not to get some carbide precipitated on dislocations, pinning them too strongly to give a temperature dependence. If one made martensite at low enough temperatures presumably the stresses involved caused some dislocation movement and this could prevent strong pinning. It might well be that the difference in the way in which the strength and the temperature dependence seemed to depend on carbon content might arise partly from differences in the degree of pinning.

Another point he wanted to make concerned the question of the Snoek effect giving an increase in strength. This was discussed by Seeger and Schoeck some time ago. The difficulty seemed to him to be that one expected an effect at temperatures where carbon could move and no effect at low temperatures, but there was no indication of a change of this type in work which had been done at 'metallurgical absolute zero (0° Paxton)', and even at temperatures below this.

His last point was in relation to Professor Paxton's remarks. He thought that one could give fairly convincing reasons for the difference in the results of work at very low strains and the results in which a nominal yield stress was measured. Work on flow stress, as distinct from yield stress, enabled one to eliminate some of the complications which might be due to the initial state of the material. One would be helped in deciding between theories which gave a thermally activated flow stress contribution from carbon and theories which did not if one measured strain rate sensitivity and temperature sensitivity at higher strains rather than at lower strains.

Dr P. M. Kelly (University of Leeds) said that he had no simple answer to Mr McGrath's question concerning the relationship between aging during the quench and the change in the internal friction peak on varying the cooling rate.

In reply to Mr Pickering's comments he agreed that internal friction techniques could well be used as another method of studying the aging effect in martensite. With reference to Mr Pickering's figures for the strength increase caused by carbon as precipitate in ferrite and the strength increase due to carbon in solution in ferrite, a similar ratio (about 2) was also observed in the case of martensite. However, it was now fairly certain that the solid solution hardening by carbon in martensite was proportional to the square root of the carbon content and did not vary

linearly with carbon content as Mr Pickering had implied in the case of ferrite. The nature of the precipitate in the aging of martensite was still not known and the only new item of information appeared to be that there might be two types of aging – aging during the quench and aging of a twinned martensite after the quench.

Dr L. Kaufman (ManLabs Inc.) asked what was the difference between a lath and a plate.

Dr Kelly said that he could answer Dr Kaufman's question by reference to a rectangular block of dimensions a, b, and c. When a was very much smaller than b and b was approximately equal to c the block was a 'plate'; when a was smaller than b and b was smaller than c the block was a 'lath'; and finally when a and b were approximately equal and both were considerably smaller than c the block was a 'needle'.

Professor Owen said that the lack of sensitivity of what could reasonably be called the elastic limit (i.e. the stress to produce a plastic strain of about 10^{-6}) to the composition of the martensite had been well established by the MIT work of 15 years ago and confirmed by the recent work at the Ford Laboratories. As Dr Christian had said, this effect could be explained satisfactorily by the generation of dislocations from locations of high local stress, giving a pre-yield strain sufficiently large to be measured.

Mr R. Phillips (Colvilles Ltd) wrote that the hypothesis that the prior austenite grain size of a martensitic structure should control the tensile properties in much the same way that grain size controlled the tensile properties of polygonal structures was implicit in much of the literature on thermomechanical treatments of martensitic steels. Kelly and Nutting had tried to make an empirical estimate of this 'grain size' contribution and he wished to offer some further thoughts on the subject.

The first point was that there was a distinct danger that the methods used to alter the grain size in the work from which Kelly and Nutting took their data also changed the friction stress. Using controlled rapid reaustenitizing to vary the grain size of En30B, Mr Fussell, Mr Storey, and he, at BISRA, obtained an apparent K of 1·9 tons/in².mm$^{\frac{1}{2}}$ compared with the value of 3·3 tons/in².mm$^{\frac{1}{2}}$ used by Kelly and Nutting. Furthermore, they were able to show that only half of this figure was really due to microstructural refinement, the remainder being a lattice hardening effect.

The next point concerned the application of a theory of the Petch type to martensite. The yield stress σ_y of an impure, slowly cooled or tempered polygonal bcc metal was related to the grain size by

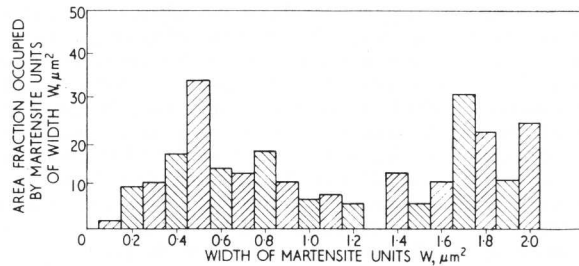$$\sigma_y = \sigma_i + k_y d^{-\frac{1}{2}} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (1)$$

For ferrous materials k_y was about 1·3 tons/in².mm$^{\frac{1}{2}}$. This equation was applicable when an extension at more or less constant stress and Lüders front phenomena were observed. Such phenomena were not observed in ferrous martensites unless they were of very low strength; it must therefore be regarded as very doubtful if the k_y effect could be expected in them and if equation (1) could be applied to high-strength martensites.

Because of the stress–strain characteristics, it was more consistent to regard the yield strength of martensite as a flow strength which for polygonal structures was represented by equation (2)

$$\sigma_f = \sigma(\epsilon) + k_f d^{-\frac{1}{2}} \dots\dots\dots\dots\dots\dots\dots\dots\dots\dots\dots (2)$$

where σ_f was the flow stress and $\sigma(\epsilon)$ a function of strain. For ferrite k_f was about 0·6 tons/in².mm$^{\frac{1}{2}}$. If a grain size effect of this type (usually attributed to the work which must be done to transmit strain across the misorientation at a grain boundary) obtained in martensite the question arose of the microstructural parameter to take for d.

The structure of a number of industrially important martensitic steels might be described as long, thin units collected into bundles, the units being parallel in each bundle and the bundles being in small groups defined by the prior austenite grain boundaries. Thus grain size was a much more complex consideration in martensite than in polygonal structures, both because of the different types of microstructural unit to be considered and the acicularity of some of them. The martensitic microstructure did not, therefore, appear to be adequately described by the prior austenite grain size.
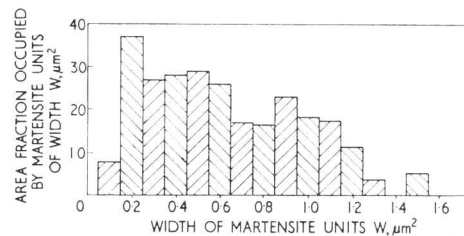
Mr Fussell of BISRA had been investigating the effect of prior austenite grain refinement on the microstructure of quenched En30B in some detail. By controlled rapid reaustenitizing, prior austenite grain sizes of 34 and 7 μm were obtained. This fivefold decrease produced a fourfold decrease in the length and a threefold decrease in the width of bundles. The effect on the individual units was more difficult to describe. In the material with the 34 μm prior austenite grain size the unit widths had the distribution curve shown in Figure D, while that with 7 μm prior austenite grain size had the unit width distribution curve shown in Figure E. The distribution of lengths of units might be similarly described. However, although the percentage change of length was greater than that of width, the bulk of the slip planes were limited by the unit width and it was the unit width, primarily, which determined the density of unit boundaries; the width was considered more important, therefore, than the length in determining the mechanical properties. Comparison of Figures D and E showed that the effect of refinement in martensite was one of cutting off the end of a spectrum of sizes rather than of changing the mean of a Gaussian distribution, which was the effect of refining a polygonal structure.

Since the prior austenite boundaries were all included in a consideration of the bundle boundaries, grain size effects in martensite might be described entirely in terms of bundles and units. Crussard had rewritten Petch's flow stress equation as

$$\sigma_f = \sigma(\epsilon) + k_1 d^{-\frac{1}{2}} + a k_2 d^{-\frac{1}{2}}$$

where k_1 corresponded to the influence of grain size on the increase in density of dislocation tangles with deformation (the finer the grain size, the higher the density of tangles for a given strain) and k_2 corresponded to the dislocations stored at boundaries in the process of transmitting slip through them; a was related to the shape defined by the boundaries.

k_1 was then most probably related to the width of units and k_2 was related to both bundles and units, and crude calculations indicated that in refinement the greater contribution to the $a k_2$ term was from the change in bundle dimensions. A detailed

D *Distribution of area occupied by martensite units of a given width for conventionally heat treated En30B steel*

E *Distribution of area occupied by martensite units of a given width for rapidly reaustenitized En30B steel*

model would be required to obtain the relative values of k_1 and k_2 and an accurate calculation of the effect of microstructural change on the flow stress of martensite. However, upper and lower estimates of the grain size contribution to the strength of martensite could be made more easily. Thus the grain size contribution to the strength of the 34 μm prior austenite grain size of En30B was calculated to be between 5 and 19 tons/in^2 and at 7 μm was between 11 and 25 tons/in^2; all four figures were relatively small contributions to the total yield stress of about 90 tons/in^2. For either extreme, the difference in grain size contribution upon achieving a refinement of the prior austenite grain size from 34 to 7 μm is only 6 tons/in^2, which was also the observed difference due to refinement.

Thus, although he would write a section on grain size in a paper on the strength of martensite somewhat differently from Kelly and Nutting, he agreed with them that the contribution from this source was small. It followed that refinement of grain size had made little contribution to increases in strength in previously published work and that it was an unpromising avenue for further developments. The effects of refinement on toughness might, however, be a different story.

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Investigation of transformation plasticity during martensite formation in a medium alloy steel

O. A. Ankara and D. R. F. West

SYNOPSIS

An investigation has been made of the plastic deformation (transformation plasticity) of a nickel–chromium–molybdenum medium alloy steel undergoing the martensite transformation under various tensile stresses, ranging from about 200 to 53 000 lb/in², and for various austenitizing conditions in the temperature range 800°–1 200°C. Transformation plasticity occurred for stresses much below the normal yield stress of austenite and in a temperature range of about 40 degC below the M_s temperature. For a given temperature of stressing the amount of deformation increased with increase in applied stress, a linear relationship being found to exist up to a stress corresponding to the austenite yield stress. For a given stress and temperature of stressing the amount of transformation plasticity was smallest for short-time, low-temperature austenitizing treatments, and increased with increase in austenitizing temperatures or time. The results are in agreement with the predictions of a macroplasticity approach, the extent of the transformation plasticity depending linearly on the magnitude of the applied stress and the transformation volume change, and inversely on the austenite yield stress, for applied stresses less than the austenite yield stress.

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INTRODUCTION

WHILE undergoing phase transformation metals and alloys are mechanically weak, and plastic deformation can occur under stresses much below the normal yield stress of the parent phase; the term 'transformation plasticity' is sometimes used to denote this phenomenon. The literature relevant to this type of plastic behaviour has been reviewed recently under the title of 'super-plasticity',[1] and it appears that only a few studies have been made with reference to martensite formation.[e.g.2-6]
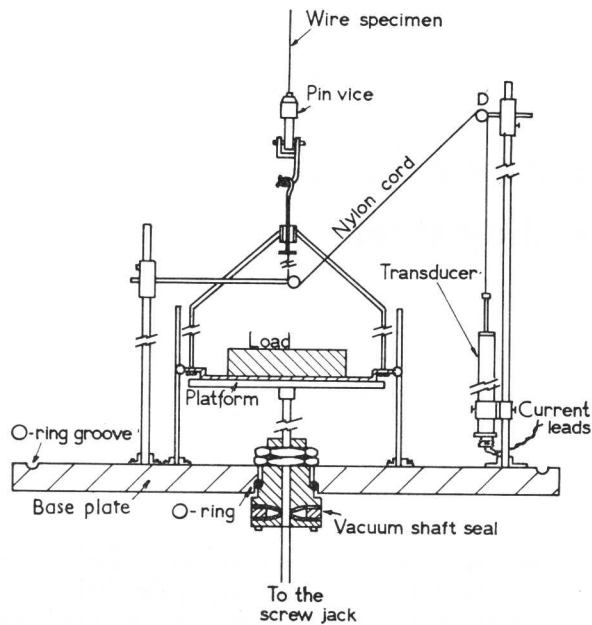
Investigations of transformation plasticity associated with allotropic transformations have shown that the main factors controlling the extent of the plastic flow are the magnitude of the applied stress, the specific volume change during transformation, and the yield stress of the phase that deforms plastically.[7,8]

Recently Johnson and Greenwood, using macroplasticity concepts, have developed an expression relating the plastic flow to these factors; this approach takes into account the internal stress developed as a result of the transformation volume change and its effect on the yielding of the system when an external stress is superimposed.[9]

The investigation reported here concerns transformation plasticity during martensite formation in a medium alloy nickel–chromium–molybdenum steel with an M_s temperature of about 300°C. This steel was selected on the basis of its high hardenability, which enabled martensite to form during free cooling of wire specimens in vacuum; the TTT diagram shows a fairly lengthy incubation period for bainite formation, which is desirable in experiments where austenite is stressed above M_s, since bainite formation can be accelerated by applied stresses.[10]

The investigation was intended to throw light on the basic mechanisms of transformation plasticity, and the influence of several factors was studied. Thus, the extent of transformation plasticity was determined for a range of stresses applied at various temperatures and for various structural conditions of the austenite obtained by control of the austenitizing conditions. The austenitizing conditions used, ranging from high-temperature treatments (e.g. 1 200°C) to short-time, relatively low-temperature treatments (e.g. 850°C), were similar to those used in an investigation of martensite transformation kinetics in the same steel.[11] In the work on kinetics, it was found that relatively low M_s temperatures were obtained with short-time, low-temperature austenitizing (e.g. for 1 s at 850°C the M_s was 283°C), while higher M_s values were obtained as the time and temperature of austenitizing were increased (e.g. 100 s at 1 200°C gave an M_s of 322°C). It was proposed that dislocations introduced into the austenite during heating the steel from room temperature, as a result of the volume change accompanying austenite formation, tend to anneal out to a degree dependent on austenitizing temperature and time; the final dislocation density achieved influences the yield stress of the austenite and the M_s temperature. In the present investigation, measurements of the yield stress of austenite were made in the range from about 330° to 650°C for austenite produced by various austenitizing treatments to study the relationship between the

1   *Schematic diagram of transducer and loading system*

2   *Temperature v. transducer readings for specimen stressed above M_s with a stress greater than the austenite yield stress*

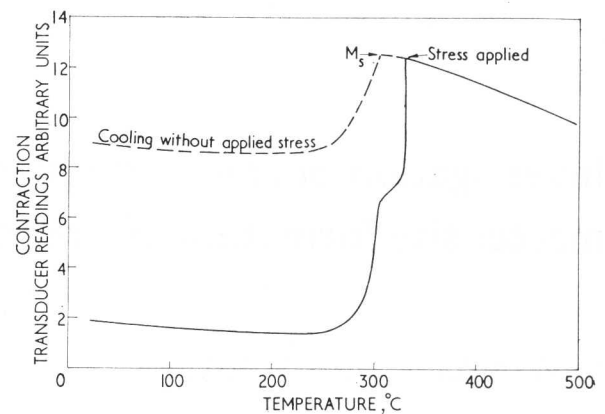extent of transformation plasticity and the yield stress of the parent phase.

EXPERIMENTAL

Wire specimens of 0·02in diameter of an En30A steel (BISRA Code Number HD) were used in the investigation; the steel composition (wt-%) was: 0·31C, 4·13Ni, 1·33Cr, 0·12Mo, 0·13Si, 0·49Mn, 0·012S, 0·011P.

The apparatus used was an adaptation of that described in a previous paper.[12] It enabled measurements to be made either of the temperature and electrical resistance, or of the temperature and length of a specimen during continuous cooling, and it incorporated a device for applying a tensile stress to the specimen at any required temperature (Figure 1).

The wire specimen, about 6in long, was vertically mounted, held in small grips in a vacuum chamber, and heated in a vacuum of about 10^{-4} mmHg to the austenitizing temperature by passage of an alternating current. The rate of heating and the time of austenitizing at a selected temperature were controlled by means of an electrical circuit incorporating a variac transformer and two process timers in series. After austenitizing, the ac was replaced by a small dc (required for the measurement of electrical resistance) and the specimen was allowed to cool in vacuum. Chromel–alumel wires (0·002in in diameter) were welded to the centre of the specimen length to form a thermocouple. Also, pure iron wires of the same diameter were welded to the specimen at points 2in apart, and equidistant about the thermocouple, to form potential leads for measuring electrical resistance. A continuous record of temperature and electrical resistance was obtained during cooling using an X–Y recorder, and from such a curve the M_s, and also the percentage of martensite existing at various temperatures below M_s, could be determined.

Stressing of the specimens during cooling could be achieved by means of a deadweight loading system, the load being contained within the vacuum system and applied as illustrated in Figure 1. The load was placed on a scale pan which rested on a platform; a rod from the platform passed through a vacuum seal to a screw jack operated by a reversible ac motor. Lowering of the platform caused the load to be suspended from the actual specimen via a hook, and in this way a specimen could be stressed at a selected temperature to within ±2 degC. The design of the system was such as to obviate any shock loading effects.

Two methods were used for the measurement of length changes occurring during the cooling of either stressed or unstressed specimens. One enabled measurements to be made continuously during cooling and the other measured only the difference in length of a specimen between the initial condition and that following a cycle of austenitizing and cooling under stress.

The principle of the former method, which was the more important of the two, is illustrated in Figure 1. The extension of the specimen was transmitted via a pulley system to a transducer unit contained in the vacuum chamber; the electrical output from the transducer represented the length change and was fed together with the thermocouple output to the X–Y recorder, so that a continuous plot of temperature v. specimen length was obtained (e.g. as in Figure 2). A length change as small as 0·0005in could be detected. Generally, only one X–Y recorder was used and curves of either temperature v. length or temperature v. electrical resistance were plotted, but in some experiments two recorders were used so that both curves could be plotted simultaneously.

A particular feature affecting the accuracy of the transducer technique was that the measurement obtained represented the total length change occurring along the whole length of the specimen between the grips. As a result of the chilling effect of these grips on the specimen, there was a region at each end of the specimen that did not reach the Ac_1 temperature during austenitizing and there was also a region between Ac_1 and Ac_3 where only partial austenitizing was achieved. Also, because of the temperature gradient existing during cooling, martensite formation and transformation plasticity did not occur completely uniformly along the wire. Measurements were made of
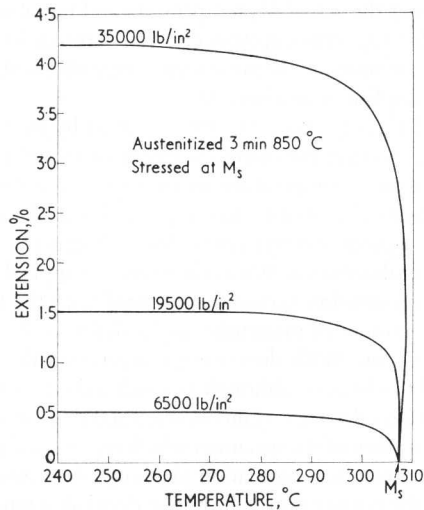
3 *Actual extension occurring during transformation under stress*

4 *Effect of magnitude of applied stress on total extension*

the temperature distribution along the wire at 850°C and also at a lower temperature near M_S; it was found that a length of about 3in was uniformly heated, and each of the non–uniformly austenitized regions was about ¾in in extent. The error introduced from this cause was taken into account by using an 'effective' gauge length in calculations of the percentage extension, as described below.

The second method of measuring length changes was not subject to any 'end-effect' error since a gauge length of only 2in (i.e. the distance between the potential leads) was employed. A cathetometer was placed outside the vacuum chamber and sighted on to the specimen through a window in the chamber wall. The distance between the potential leads was measured at room temperature before and after a given cycle of austenitizing and cooling under stress. The gauge length could only be measured to an accuracy of about 0·02in so that this method was less sensitive than that using the transducer. By comparison of the results of the two methods, an 'effective' gauge length for austenitizing at 850°C was estimated for the transducer method; length changes measured by the transducer were then expressed as a percentage of this estimated gauge length.

The total extent of the transformation plasticity in a given experiment was obtained by noting the difference in length at room temperature between a specimen transformed to martensite without and with stressing respectively. The term 'total elongation, %' as used in the graphical presentation of the results (e.g. Figure 4) refers to transformation plasticity measured in this way and expressed as a percentage of the effective gauge length.

In experiments involving austenitizing at 850°C measurements were made by both the cathetometer and transducer methods. The results for other austenitizing temperatures were obtained by using the cathetometer method only.

In the measurements of the strength of the austenite, the yield stress was taken as the stress corresponding to about 0·1% strain. To make the required measurements a dynamometer was mounted within the vacuum chamber in place of the dead-weight loading assembly. The cooling of the specimen could
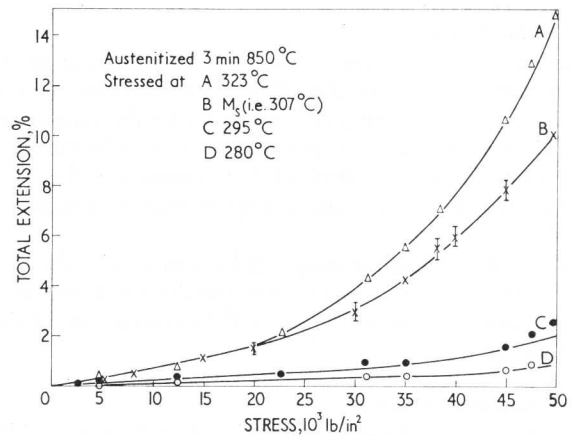
be stopped at the required temperature by passing an ac of sufficient magnitude to maintain the temperature constant. The movement of the rod attached to the screw jack then gave continuous straining, and the outputs from the dynamometer and transducer were fed to the X–Y recorder which thus gave a stress–strain plot from which the yield stress value was measured. For measurements of austenite yield stress at temperatures in the range, say, 350°–400°C, the cooling of the specimen was relatively slow and little accuracy was lost by carrying out the test as the specimen cooled. The results are not of high absolute accuracy in view of the limitations of the experimental procedure, but they are nevertheless useful for comparing the effects of various austenitizing treatments, and for the quantitative interpretation of transformation plasticity.

The as–received structure of the wire was tempered martensite, and experiments to determine a suitable initial homogenizing treatment were made, as previously reported.[11] On the basis of these experiments samples were initially treated for 5 min at 1 000°C and were then cooled to produce martensite. For the experiments on transformation plasticity, martensitic samples were rapidly heated at about 100–200 degC/s to the required austenitizing temperature. Measurements of length changes occurring during heating for austenitizing indicated that the major part of the transformation to austenite occurred in the range of about 695°–715°C.

Standard austenitizing times of 3 min were used for temperatures in the range 800°–1 000°C and 100 s for temperatures above 1 000°C; these times were considered to produce an essentially homogeneous austenite from the standpoint of chemical composition. The values of M_S agreed closely with those previously reported for the same steel.[11] Also, the reproducibility and accuracy of the M_S values were similar to those of the previous work; thus, from specimen to specimen, the M_S for a given treatment agreed to within ±2 degC, while for a given specimen a reproducibility of ±1 degC was obtained. An indication of the scatter obtained in the measurements of the total extension is shown in Figure 4; for example, for a stress of 20 000 lb/in² applied at M_S the results of replicate experiments lay between 1·3 and 1·8%, while at higher stress levels the scatter was nearly twice as great.

RESULTS
Austenite yield stress
Measurements of austenite yield stress were made at several temperatures in the range 330°–650°C. A temperature of 300°C was taken as representing an average M_s for the various austenitizing treatments, and approximate yield stress values for this temperature were obtained by extrapolating the 330°–650°C data for various austenitizing treatments as shown in Table I.

The highest strength corresponded to austenitizing for 1 s at 850°C, and the strength decreased progressively with increasing austenitizing time at 850°C and with increasing austenitizing temperature.

Transformation plasticity
The effects on transformation plasticity of tensile stresses within the range 200–53 000 lb/in² were studied for selected temperatures of stress application, either above, below, or at M_s, for a standard austenitizing treatment at 850°C. It was found that even when no stress was applied during martensite formation, a small permanent extension occurred (up to about 0·06%) when a martensitic specimen was austenitized and then cooled to produce martensite. When stresses less than 1 000 lb/in² were applied during cooling the extension was not more than 0·06%, and it was therefore not possible to be certain whether any transformation plasticity occurred with such stresses. Stresses of 1 000 lb/in² or more, when applied at or above M_s, gave detectable transformation plasticity; for example, for a stress of 1 000 lb/in² applied at M_s, approximately 0·1% permanent extension occurred. With increasing values of stress, the amount of deformation increased.

When stresses greater than the yield stress of the austenite are applied above M_s, and maintained during cooling through the martensite range, the total extension is made up of the plastic deformation that occurs above M_s due to the stress and the transformation plasticity associated with the main bulk of martensite formation. Interpretation of the results is then rather complex since the application of such stresses between M_s and M_d induces some martensite formation, which may assist yielding by virtue of a transformation plasticity effect. Also, although the total extension observed during cooling under stress to room temperature is greater than that found when the same stress is applied at M_s, experiments reported below showed that the extent of the main transformation plasticity is somewhat reduced by plastic deformation of the austenite occurring above M_s. Thus, when studying the effects of stresses greater than the yield strength of the austenite, it is desirable to apply

TABLE I   Austenitizing treatment and yield stress

Austenitizing treatment		Austenite yield stress at 300°C, lb/in²
850°C	1 s	35 000
850°C	6 s	30 000
850°C	12 s	27 000
850°C	3 min	20 000
900°C	3 min	17 000
950°C	3 min	16 000
1 000°C	3 min	14 000
1 100°C	100 s	13 000
1 200°C	100 s	12 000

the stresses at the actual M_s temperature and not above it, and this was done in some experiments. With stresses less than the yield stress of austenite the same results were obtained when the stress was applied at or above M_s.

Figure 2 gives the type of curve obtained by the transducer technique, showing the change in length of a specimen during cooling to room temperature when a stress greater than the yield strength of austenite was applied above M_s and maintained throughout cooling; a curve for cooling in the unstressed condition is also shown. When the stress was applied a marked and sudden extension occurred, associated with the formation of a small amount of martensite, and a slight recalescence was found to occur. With decreasing temperature the specimen continued to elongate, although at a reduced rate. On cooling through M_s recalescence again occurred and there was a further sudden extension of the specimen which represented the beginning of the main transformation plasticity; the extension continued as the temperature fell, but at decreasing rate. It is important to note, when interpreting the length changes that occur during cooling, that the measured changes include contributions from thermal contraction and from the volume expansion due to martensite formation, as well as from the transformation plasticity.
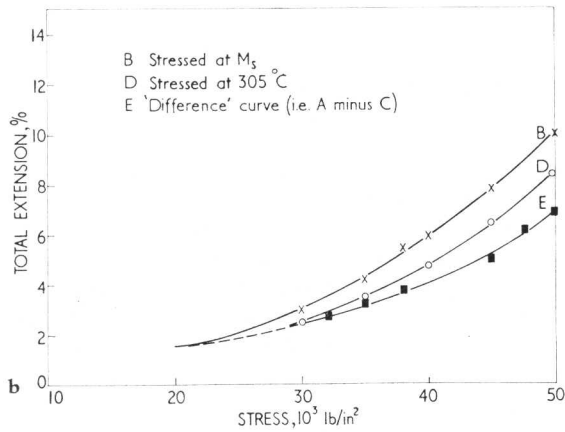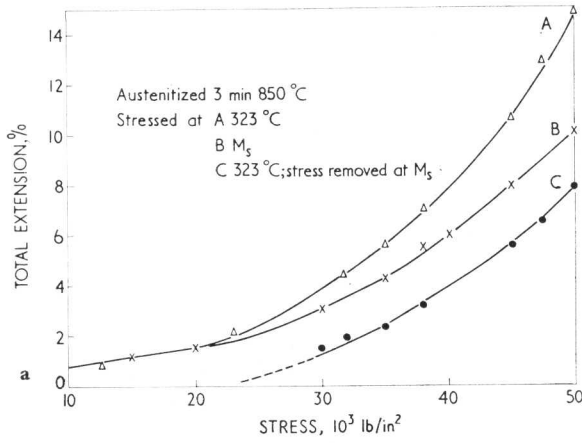
The actual progress of transformation plasticity with decrease of temperature below M_s is illustrated by Figure 3. These curves have been derived from the transducer results by making corrections for thermal contraction and for the volume expansion due to martensite formation. They show that the transformation plasticity was practically completed when the temperature had fallen about 40 degC below M_s. During normal unstressed cooling approximately 70% of martensite forms within this range of 40 degC.

Figure 4 shows that the total amount of transformation plasticity increases with increase in applied stress. Thus, for example, for stressing at M_s after austenitizing at 850°C, as the stress was increased up to 20 000 lb/in² the extension reached a value of about 1·5%. When the stressing temperature was decreased below M_s the amount of plasticity decreased. The results of Figure 4 for a given temperature of stress application (e.g. curve A) indicate a straight line relationship between the total extension and the magnitude of the applied stress in the lower range of stresses. Although the slope of the lines decreased as the temperature of stress application was decreased from M_s (307°C) down to 280°C, no increase of slope was observed when the stressing temperature was increased to 323°C. The plotted data for the various temperatures showed a departure from linearity beyond a certain stress level; the slope of the curves then increased progressively, the effect being particularly marked when the stresses were applied at or above M_s. A distinct departure from linearity occurred at about 20 000 lb/in² for stresses applied at or above M_s, and this stress corresponds closely to the austenite yield stress at M_s. The departure from linearity occurred at higher stress levels when the stressing temperature was below M_s, e.g. at about 30 000 lb/in² for 295°C.

When the stress was applied at temperatures below about 270°C (approximately 40 degC below M_s), no transformation plasticity was detected, which agrees with observations on the temperature range within which plasticity occurs (Figure 3).
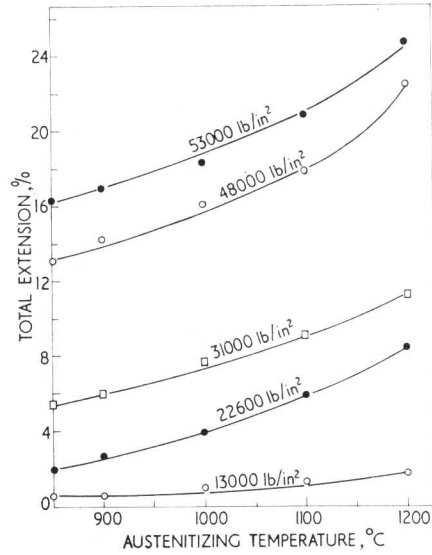
a

b

5 *Effect of stressing temperature on total extension*

6 *Effect of austenitizing temperature on total extension; stressed at 330°C*

As shown in Figure 4 (curves *A* and *B*), when stresses in the non-linear portion of the graph were applied above M_S the total amount of deformation that occurred was greater than when the same stresses were applied at M_S; this was due to the occurrence of plastic deformation above M_S. Experiments were made to investigate the effects of such plastic deformation on the subsequent transformation plasticity that begins at M_S, and the results are shown in Figure 5*a* and *b*. Curve *A* shows the total extension that occurred when stresses ranging up to 50 000 lb/in² were applied at 323°C (i.e. 16 degC above M_S) and were maintained down to room temperature. Curve *B* shows the values of the extension when the specimens were stressed at M_S. Curve *C* represents the extension resulting from applying the stress at 323°C and removing it at M_S. The difference between curves *A* and *C* gives the amount of transformation plasticity associated with martensite formation below M_S under conditions when the specimen was stressed at 323°C with stresses greater than the austenite yield stress. This 'difference curve' is curve *E* of Figure 5*b*, and is seen to lie significantly below curve *B*; this shows that when stresses that cause plastic deformation of the austenite are applied above M_S the effect is to reduce the transformation plasticity occurring on cooling through M_S. The effect of the existence of a relatively small amount of martensite (about 7%) in reducing transformation

plasticity is illustrated by curve *D* which shows the amount of plasticity following the application of a stress 2 degC below M_S. This amount of martensite is comparable to that formed at 323°C when a stress of 50 000 lb/in² is applied.

Experiments were made to determine the effect of austenitizing temperature on transformation plasticity. Austenitizing treatments using the standard times were carried out at various temperatures in the range 850°–1 200°C. Stresses up to 53 000 lb/in² were used with the same stressing temperature in each case (330°C), bearing in mind that the M_S temperature increases as the austenitizing temperature is increased. 330°C was chosen as being above the M_S temperature for all the austenitizing treatments.

Measurements of total extension for the various austenitizing temperatures and stresses are shown in Figure 6. It is seen that, for a given stress, the extension increases with austenitizing temperature; also for a given austenitizing temperature the extension increases with stress. The increase in plasticity with increase in austenitizing temperature is particularly marked at the higher stress levels; thus, for a stress of 53 000 lb/in², increasing the austenitizing temperature from 850°C to 1 200°C gave an increase in extension from approximately 16% to 25%. The results of experiments in which the stress was applied at the actual M_S temperature corresponding to each particular austenitizing treatment are shown in Figure 7; the trends are similar to those of Figure 6. The recalescence effect occurring at M_S increased with increasing austenitizing temperature and stress, being very pronounced for high austenitizing temperatures and large stresses applied at, or just above, M_S; when a stress of 50 000 lb/in² was applied at M_S after austenitizing at 1 200°C, the temperature rise at M_S was about 50 degC.

Experiments were made using austenitizing times much shorter than the standard times; a typical 'short-time' treatment was about 1 s at the actual austenitizing temperature following rapid heating from the martensitic condition. The results for short-time and normal austenitizing treatments at 850°C for various
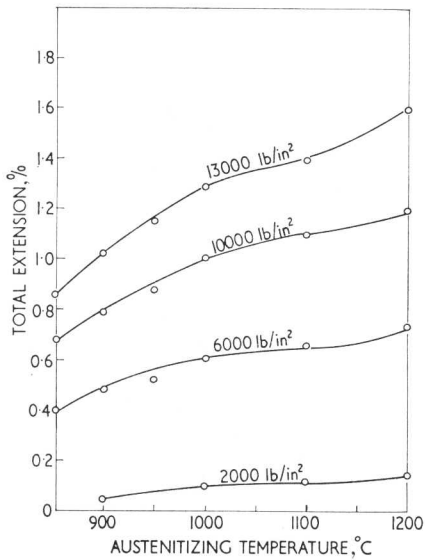
7   *Effect of austenitizing temperature on total extension; stressed at M_s*
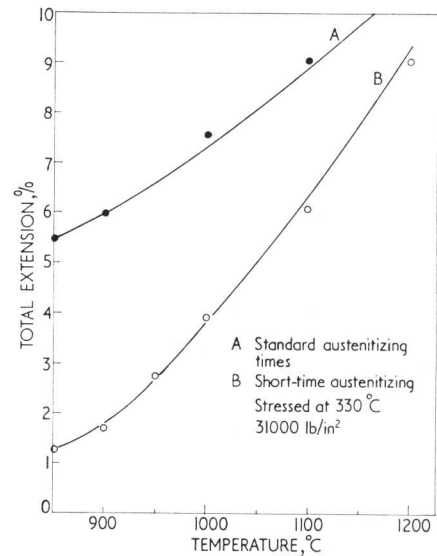
9   *Effect of austenitizing temperature on total extension*

stresses applied at the M_s are shown in Figure 8. For the relatively low stress values the extension and stress show a linear relationship, but the extension is much less for the short-time than for the normal treatments, the difference being particularly marked at the higher stress levels where the linear relationship does not hold. The results of Figure 9 show that the difference between the two types of treatment decreases as the austenitizing temperature increases.

Other experiments were made to study the effect of 'double-austenitizing' treatments and of heating rate for austenitizing along the lines previously reported in a study of transformation kinetics.[11] In the first type of experiment specimens were austenitized at 1200°C and then held for several minutes at some lower austenitizing temperatures before final cooling; the extent of the transformation plasticity was found to correspond to the value normally found for the higher temperature treatment. In the other series of experiments, comparison was made between the extent of the transformation plasticity obtained with the normal rapid heating for austenitizing and with slow heating

through the critical range, using an austenitizing temperature of 800°C. The results (Figure 10) show a linear relationship of extension and stress in the lower part of the stress range. The M_s was found to be higher with slow heating for austenitizing, as previously reported,[11] and the extent of the transformation plasticity was greater for slow than for rapid heating; for example for a stress of 20000 lb/in² the plasticity is 1·8% as compared with 1·3% for the rapid heating austenitizing.

Microstructural observations

Some metallographic work, using the Greninger–Troiano quench–temper technique, was carried out to study the martensite formed under various stressing conditions.

In one series of experiments with a standard 850°C austenitizing treatment a selected stress was applied at M_s and the specimen was allowed to cool 5 degC below M_s; the stress was removed and the specimen rapidly heated to 500°C and held for 1 min to temper the martensite. Stress levels of 8000, 23000, 35000, and 50000 lb/in², respectively, were used. For
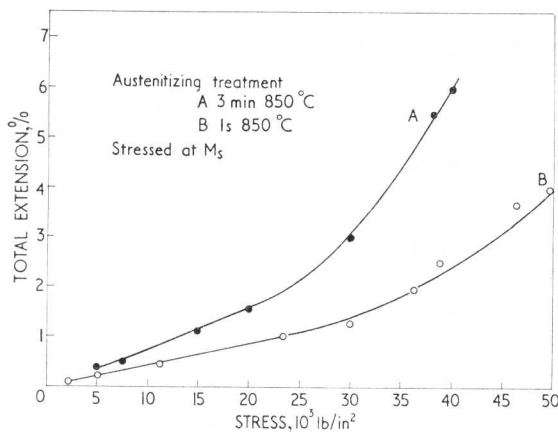
8   *Effect of short-time austenitizing on total extension*
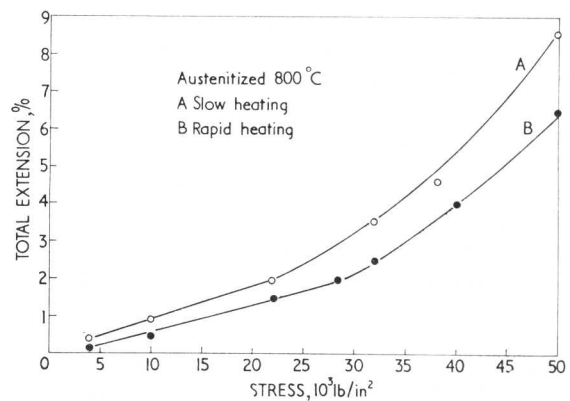
10   *Effect of heating rate through critical range on total extension*

the first of these no significant difference was observed between the martensite formed under stress and that which formed in experiments when no stress was applied, and no preferred orientation of the martensite plates was observed. With the stress of 23 000 lb/in² it was found that some of the martensite plates showed a preferred orientation effect, being inclined at about 40° to the tension axis. With the larger stresses a greater number of martensite plates showed the orientation, and the plates tended to become finer with increase in stress. It is possible that some bainite may have formed, particularly where marked recalescence occurred.

Experiments were also made in which a stress was applied 10 degC below M_S and maintained during cooling through a further 5 degC; in this case the martensite which formed while the stress was applied was contained within a structure in which some martensite plates were already present. Microscopical examination did not reveal any significant preferred orientation effect in these experiments.

Some work was done on the effect of applied stress on the kinetics of martensite formation. Following standard austenitizing at 850°C various stresses were applied at M_S (307°C) and removed at 305°C. The amount of martensite formed was determined by quantitative metallography and compared with that formed in an unstressed specimen. The amount of martensite increased progressively with increasing stress; thus, in an unstressed sample approximately 7% of martensite formed, while for stresses of 10 000, 20 000, and 50 000 lb/in² respectively the amounts of martensite were approximately 10, 20, and 40%. It should be noted that the actual range of temperature over which the martensite forms depends on the extent of the recalescence effect; with the largest stress used this amounted to about 3–5 degC.

DISCUSSION

The main features requiring interpretation are the dependence of the magnitude of transformation plasticity on the magnitude of the stress, on the temperature of stress application, and also on the nature of the austenitizing treatment. The main observed trends are as follows:

(i) for a given temperature of stressing the total extension due to transformation plasticity increases with the magnitude of the stress, a linear relationship being shown up to a certain stress level

(ii) the extent of the plasticity decreases as the temperature of stress application is decreased below M_S, and for temperatures lower than about 40 degC below M_S no plasticity is observed with the range of stresses used

(iii) for a given stress and stressing temperature the total extension is smallest for short-time, low-temperature austenitizing, and increases markedly with increase in austenitizing temperature.

Two complementary approaches may be used to discuss the results. The first of these is based on concepts of macroplasticity and involves three parameters: the applied stress, the austenite yield stress, and the volume change associated with the transformation. The second approach is to consider structural factors and possible mechanisms involved in the plasticity.

The first approach has been developed with reference to the general phenomenon of transformation plasticity by de Jong

and Rathenau[7,8] and Johnson and Greenwood,[9] but hitherto does not appear to have been applied specifically to martensite formation. Johnson and Greenwood have proposed a quantitative relationship based on the fact that when a region of product phase forms in a weaker parent phase the associated volume change produces a stressed region in the parent phase at the phase interface; if the volume change is great enough this stress will reach the yield stress of the weaker phase. If now an external stress is applied to this internally stressed system while the transformation is proceeding, plastic deformation will occur for stresses very much less than the normal yield stress of the weaker phase.

The following relationship was derived by Johnson and Greenwood:—

$$\varepsilon = A\frac{\sigma}{Y}\left(\frac{\Delta V}{V}\right) + B\left(\frac{\Delta V}{V}\right) \qquad \text{...............................(1)}$$

where ε is the plastic strain occurring during the transformation from one phase to another, σ the applied stress, Y the yield stress of the weaker phase at the transformation temperature (i.e. austenite in the present case), $\Delta V/V$ the fractional (or percentage) difference between the specific volumes of the two phases, A a constant (value about 2/3), and B a constant whose value is sensitive to the geometry of the transformation front.

The term $B(\Delta V/V)$ represents the plastic deformation due to the transformation alone, without stress application; in the present case this term is found experimentally to be very small, less than 0·06%, and is neglected.

The linear relationship of equation (1) is not expected to apply for stresses greater than the yield stress of the weaker phase; there will be a departure from linearity in the direction of greater amounts of plasticity.

The present results may be used to test the applicability of the above relationship to transformation plasticity during martensite formation for the range of stresses up to the austenite yield stress. Several preliminary points should be noted. First, as described, transformation plasticity occurs within a range of some 40 degC below M_S, during which range approximately 70% of martensite forms. Thus, the value of $\Delta V/V$ used in equation (1) is taken to be 70% of that associated with complete transformation to martensite. The value for complete transformation is about 2·7% referred to the M_S temperature, and the relevant volume change for the transformation plasticity calculations is therefore taken as approximately 1·9%. It is assumed that the transformation volume change occurring during transformation plasticity is independent of austenitizing and stressing conditions. Another point to note is that since transformation plasticity occurs over a range of temperatures the austenite yield stress is not constant. Apart from the variation of austenite yield stress with temperature, there is the added complication that the formation of martensite strengthens the remaining austenite. However, a very large proportion of the plasticity occurs in the range of some 10 degC below M_S, and the change in yield stress, due to simple temperature dependence, over a range of 40 degC is only about 1000–2000 lb/in²; the austenite yield stress at 300°C was used as a representative value in all calculations.

The validity of equation (1) may first be checked by consideration of the effect of stresses of various magnitudes, applied

at or above M_S, for a given austenitizing treatment, which determines the value of the austenite yield stress. For example, reference to Figure 4 (curve *A*) shows that the initial portion, at low stresses, is in agreement with the predicted linear relationship between the extent of the transformation plasticity and the magnitude of the applied stress. For the standard austenitizing treatment at 850°C the yield stress of the austenite at M_S is approximately 20 000 lb/in² and this value agrees well with that at which the graph departs significantly from linearity. A calculation from equation (1) gives a value of 1·3% for a stress of 20 000 lb/in², which is in good agreement with the experimental value of 1·5% (curve *B*). In the higher stress range (above the yield stress) the amount of transformation plasticity increases at an increasing rate as the stress is increased.

As discussed below, the amount of transformation plasticity is found to decrease as the austenite yield stress increases; the reduction in the amount of transformation plasticity occurring on cooling through M_S, following plastic deformation of the austenite above M_S (Figure 5), may therefore be attributed to an increase in yield stress of the austenite due to strain hardening and the formation of some stress-induced martensite.

With reference to Figure 4 the smaller slope of the linear portion of the extension v. stress graphs, and the greater extent of the linear portion associated with stress application below M_S, can be attributed to two factors. In the first place, since some martensite has already formed when the stress is applied, there is a correspondingly smaller volume change occurring between the temperature at which the stress is applied and that at which transformation plasticity ceases; the effect of this is to reduce the amount of plasticity that occurs for a given stress. Secondly, the yield stress of the austenite is expected to be increased by the formation of some martensite. This is attributable to the introduction of dislocations into the austenite, and also to the presence of the martensite plates which act as barriers to dislocation movement; this factor also reduces the transformation plasticity, and moreover should increase the stress range within which the linear relationship holds. It is difficult, in the case of Figure 4, curves *C* and *D*, to determine accurately the stress level at which the departure from linearity occurs, but the values seem to be about 30 000 and 40 000 lb/in² respectively. Assuming these values to be the respective yield stresses applicable in equation (1), and taking appropriate $\Delta V/V$ values corresponding to the amount of martensite formation that occurs between the temperature of stress application and that at which plasticity ceases, it is calculated that the total extension at the end of the linear range should be approximately 0·6% and 0·2% for curves *C* and *D* respectively. These values agree reasonably with the experimental data.

The dependence of transformation plasticity on the austenite yield stress can be studied by using the results of the experiments on various austenitizing treatments. The variation of yield stress with austenitizing conditions is considered to be controlled by the dislocation concentration and austenite grain size, which are themselves determined by the austenitizing conditions. A high concentration of defects is introduced into the austenite when a steel is rapidly heated into the austenite range, as a result of the transformation volume change; these defects then anneal out as a function of austenitizing temperature and time. The high strength of austenite, produced by a short-time
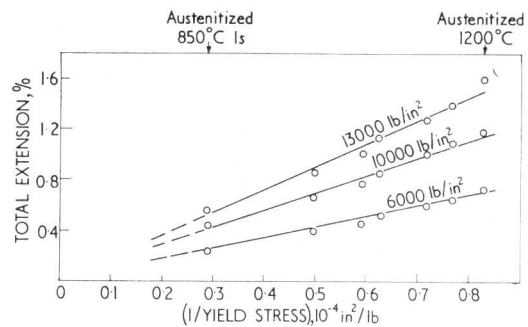
11 *Total extension v. reciprocal of austenite yield stress*

austenitizing treatment at, say, 850°C, is dependent on the high defect content of the lattice and on the fine austenite grain size: the contributions of defect content and grain size to the strength are not yet known accurately, but it is considered likely that the defect content provides the main strengthening contribution. With high-temperature austenitizing treatments the decrease in defect content and the increase in grain size give a much lower austenite strength and an increase in the transformation plasticity.

Equation (1) predicts that the extent of the transformation plasticity varies inversely with the austenite yield stress. Figure 11 shows values of total extension (%) for several levels of applied stress plotted against values of the reciprocal of the austenite yield stress at 300°C, corresponding to various austenitizing treatments. Data are shown for a 1 s austenitizing treatment at 850°C (obtained by interpolation from Figure 8) and for normal austenitizing treatments in the range 850°–1200°C. For each of the levels of applied stress a reasonable straight line may be drawn; each line is drawn assuming that it extrapolates to the origin of the graph in agreement with equation (1) (if the assumption is made that the $B(\Delta V/V)$ term can be neglected).

The slopes of the lines (i.e. total extension/reciprocal of yield stress) may be used together with the value of $\Delta V/V$ and the appropriate yield stresses to calculate the value of the constant *A* in equation (1). It is found that for each of the levels of applied stress the calculated values are between 0·7 and 0·75, and thus agree well with the value of $\frac{2}{3}$ used by Johnson and Greenwood in the equation.

The good agreement with theory of the experimental results for the various austenitizing treatments may also be illustrated by calculating the extent of transformation plasticity from equation (1) for particular cases, using the known values of yield stress. Thus, for example, for a 1 s austenitizing treatment at 850°C, with an applied stress of 20 000 lb/in² the transformation plasticity is calculated as 0·7%, which agrees closely with the measured value in Figure 8. Agreement with theory is also found in that the plot of total extension v. applied stress for short-time austenitizing in Figure 8 deviates from linearity at about 30 000–35 000 lb/in², which is close to the austenite yield stress value.

The greater amount of transformation plasticity shown by specimens slowly heated for austenitizing as compared with those rapidly heated (Figure 10) may also be interpreted on the basis of austenite yield stress. The results of a few measuremens

showed that normal austenitizing at 800°C gave an austenite yield stress of about 25 000 lb/in² at 300°C as compared with a value of 20 000 lb/in² when slow heating was used. Values of transformation plasticity calculated using these figures agree reasonably with the experimental values of Figure 10, although the agreement is not quite as close as for other austenitizing treatments discussed above.

With regard to the double-austenitizing treatments, no measurements of austenite yield stress were made but, since the grain size and dislocation density, which control the yield stress, will be determined by the first (i.e. higher) austenitizing temperature, the transformation plasticity corresponds to that characteristic of the higher austenitizing temperature.

On the basis of the evidence discussed above, it appears that the application of the macroplasticity approach gives good quantitative agreement with the observed results, and the applied stress, austenite yield stress, and transformation volume change appear to be the dominant factors controlling the phenomenon of transformation plasticity.

With regard to the complementary approach involving considerations of structure and mechanisms, two particular aspects may be considered: the dislocation structure of the austenite and its relevance to the plastic flow that occurs during transformation; and the preferred orientation of martensite plates formed under stress.

In terms of dislocation theory, it has been suggested[3] that the energy to move dislocations to the grain boundaries from their sources may be supplied by the phase transformation, and that very little additional stress might then be needed to initiate deformation in adjacent grains. However, a comparison of transformation plasticity in single crystal and polycrystalline samples of an iron–nickel alloy has shown that dislocation pile-up at grain boundaries is not an essential part of an explanation.[6] An alternative proposal may be based on the well-established observation that a high concentration of dislocations exists in austenite adjacent to martensite plates.[13] This feature may be related to the stressed region postulated in the macroplasticity approach by assuming that, under the influence of an applied stress during the transformation, dislocation movement could readily occur in favourable oriented slip systems, thus producing the large extension during transformation. As the amount of martensite present in the structure increases with falling temperature, the dislocation content and hence the yield strength of the residual austenite will increase progressively; a strengthening effect will also be operative due to the martensite plates acting as barriers for dislocation movement. These effects, together with the reduction of the amount of untransformed austenite as the temperature decreases, provide an explanation of the cessation of detectable transformation plasticity at a temperature some 40 degC below M$_S$.

The formation of martensitic plates with a preferred orientation as observed in the present experiments and in previous work[e.g.14] may be interpreted on the basis of Le Chatelier's principle; the plates which form initially are expected to be those most favourably oriented with respect to the applied stress to relieve the applied stress. The tendency to a 'directional' effect in the martensite formation would mean that the transformation volume change might lead to extension of the specimen under quite low stresses. However, the evidence from the present results does not support the view that this is a major factor in causing transformation plasticity in the alloy steel studied. Thus, no significant preferred orientation was observed for a stress of 8 000 lb/in² even though transformation plasticity occurred. Also, there seemed to be no significant preferred orientation occurring below about 295°C, whereas some transformation plasticity occurred below this temperature. Further evidence against the preferred orientation explanation is provided by the results showing that austenitizing conditions affect the extent of transformation plasticity; no experiments were made to see whether the preferred orientation effects varied with austenitizing conditions, but it seems unlikely that there would be a large difference. In fact, the effect of austenitizing conditions seems to be better explained in terms of the yield strength of the austenite.

To summarize the discussion on both approaches to the problem, the good quantitative agreement of the macroplasticity approach with the observations suggests that the transformation plasticity is dominated by the three factors of applied stress, austenite yield stress, and transformation volume change. In terms of structural considerations, various possible mechanisms may be invoked to explain the observed phenomena; it is suggested, however, that the main mechanism is the high concentration of dislocations in austenite adjoining martensite plates, and the movement of these dislocations in favourably oriented slip systems during transformation.

CONCLUSIONS

1. During the transformation of austenite to martensite, plastic deformation occurs under stresses much less than the normal yield stress of the austenite; thus, deformation was detected with a stress of only 1 000 lb/in².

2. For a given temperature of stress application, the amount of deformation increased with increase in applied stress, and a linear relation existed between extension and stress up to a stress corresponding to the austenite yield stress.

3. Transformation plasticity was detected only in a range of about 40degC below M$_S$, in which about 70% of martensite formed. Within this temperature range the extent of transformation plasticity decreased with decreasing temperature of stress application; when the stresses were applied below this range no transformation plasticity was detected.

4. Plastic deformation of the austenite above M$_S$ reduced the amount of transformation plasticity that occurred during subsequent cooling through M$_S$.

5. The extent of transformation plasticity varied with the conditions of austenitizing. The lowest values of plasticity were associated with short-time austenitizing treatments, and the extent of the plasticity increased considerably with increase in austenitizing temperature or time. Values of transformation plasticity following rapid heating during austenitizing were lower than those obtained when slow heating was used for austenitizing.

6. The yield stress of the austenite was found to vary with austenitizing conditions; relatively high strengths were associated with short-time treatments at relatively low temperatures, and lower strengths with higher austenitizing temperatures.

7. The results can be interpreted on the basis of macroplasticity considerations and are in good quantitative agreement with

a relationship that predicts that the extent of transformation plasticity varies linearly with the magnitude of the applied stress and the transformation volume change, and inversely with the austenite yield stress, for relatively low applied stresses.

8. Martensite plates were found to form with a preferred orientation in the initial stages of transformation under some conditions of stress, but this does not appear to be a major factor in relation to the mechanism of transformation plasticity.

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