Strain Age Hardening in the Range 150~350°C of Carbon Steel*

By Kazuyoshi Nishino** and Kiyoharu Takahashi***

Cold worked carbon steel hardens in two stages on annealing. The primary hardening below 150°C may be explained by Cottrell's mechanism, so the secondary hardening in the range 150~350°C was chiefly treated in this investigation. Experiments on hardness, stress-strain relationship and the Bauschinger effect showed the following results. The secondary strain age hardening becomes marked with increased carbon content, and in addition, it is not accompanied by any rise of the yield point upon measuring under a stress of the same kind and direction as those of prior working. It is likely that the hardening is associated with the dispersion of cementite, since it is not pronounced within the ferrite phase unlike the case of α -brass. This presumption may be supported by the fact that the metals in which alumina particles finely disperse also show a similar hardening. Higher carbon steels exhibit a marked Bauschinger effect, its removal on annealing showing, as in the case of α -brass, a similar trend to that of hardening. In addition, hardening in a given steel is chiefly affected by the arrangement, shape and size of the cementite particles. The cementite particles or platelets distributed in the ferrite act as an obstacle against dislocations during deformation, generating pile-ups of dislocation. On unloading, or after unloading, the dislocations are under the full action of the back stress, by which the Bauschinger effect is induced. It is inferred that the strain age hardening of carbon steels is associated with the hindrance of motion back of the dislocations.

(Received May 31, 1961)

I. Introduction

A phenomenon which is called strain age hardening or anneal hardening can be clearly observed in face centered cubic solid solutions containing relatively high concentration of solute, such as α -brass or austenitic stainless steel. While with poly-phase alloys, there are some cases where the hardening is remarkable only when the second phase finely disperses⁽¹⁾⁽²⁾, even if their matrix alone does scarcely show.

Cold worked carbon steels harden generally in two stages with low temperature annealing. The primary hardening below 150°C takes place within the ferrite phase and it has been reasonably explained by Cottrell's mechanism, while the secondary in the range of 150° to 350°C has been considered to be

due to precipitation or dissolution of cementite⁽³⁾⁽⁴⁾ but, since a Fe-Al₂O₃ alloy shows a similar hardening, it would be expected that the hardening is associated with the internal stress⁽⁵⁾ or the dispersion of the second phase as an obstacle against dislocation.

In copper alloys, as reported previously⁽⁶⁾, the α solid solution shows marked elastic after- and Bauschinger effects, leading to the presumption that strain age hardening is generally marked only in such alloys which strongly show both of the above effects. On the other hand, in aluminum alloys and carbon steels, their solid solution alone exhibits little or no such effects or strain age hardening⁽²⁾, but both these effects and strain age hardening are still more prominent in increasing the amount of finely dispersed second phase⁽¹⁾⁽⁷⁾. Therefore, it may be expected

*** Faculty of Engineering, Yamagata University, Yamagata, Japan.

** Research Institute for Iron, Steel and Other Metals, Tohoku University, Katahira-cho, Sendai, Japan.

* This paper was published in the Journal of the Japan Institute of Metals, 24 (1960), 514.

(1) Y. Imai and H. Hirotsu: Sci. Rep. Inst. Tohoku Univ. Japan, 12 (1960), 168.

(2) I. Gokyu: J. Japan Inst. Metals, 7 (1943), 7. (In Japanese)

(3) F. Garofalo and G. V. Smith: Trans. ASM, 47 (1955), 957.

(4) M. Kawasaki: Sect. Rep. Japan Inst. Metals, IX (1952). (In Japanese.)

(5) F. H. Andrew and H. Lee: J. Iron Steel Inst., 165 (1950), 149, 369.

(6) K. Nishino: J. Inst. Metals, 20 (1956), 115, 704; 22 (1958), 50; 23 (1959), 232, 236.

(7) N. N. Davidenkov and G. M. Shevandin: Z. Metallkunde, 26 (1934), 193.

that the hardening has a close relation with the Bauschinger effect in a manner similar to that of α brass⁽⁶⁾, and also with the dispersion of particles. Plastic deformation of two phase alloys such as age hardenable Al-Cu and alumina dispersed alloys, has recently been studied by many investigators, and discussions were made from the view point of the interaction between dislocations and dispersed particles. The present investigation was performed to make the nature of the secondary strain age hardening of carbon steel more definite from the standpoint of the dispersion of cementite particles, and also by comparison with α -brass.

II. Specimens and Methods

Contents of impurities in the carbon steels used were in the range as listed in Table 1. Alumina dispersed copper was prepared by internal oxidation

Table 1

Element	Si	Mn	P	S
%	0.20~0.38	0.35~0.60	0.011~0.035	0.013~0.038

of Cu-0.52 % Al alloy ($1.3 \times 5 \times 100 \text{ mm}^3$) heated at 900°C for 14 days in a manner similar to Martin's method⁽⁸⁾. Alumina dispersed iron was prepared by sintering pure iron powder (270 mesh) and fine alumina powder (about 1μ) in a hydrogen stream. The alumina particles were somewhat larger than internally oxidized copper which was proven by microscopic observation.

Anneal treatment of carbon steels for softening was performed by heating in the range of 30° to 50°C beyond the A_3 and A_1 points for 1 hour and furnace cooling. Low temperature annealing after cold working was made by using both oil and Pb-Sn baths, and the heating time was 3-4 minutes, depending upon the heat capacity of the specimen.

Experiments on the Bauschinger effect were carried out by torsion, which was adapted to a reversal of stress. The specimens were in the form of thin walled hollow cylinders with the dimensions shown in Fig. 6, in order to homogenize the distribution of the applied stress. Tensile tests were made with Amsler's testing machine with specimens 7 mm in diameter and 50 mm in gage length.

III. Results

1. Strain age hardening of carbon steel

Strain age hardening curves for cold worked carbon steels are shown in Fig. 1. The hardness increased in two stages, i.e., the primary hardening below 150°C and the secondary in the range of 150-350°C. The degree of the primary decreases with the increase of carbon content, leading to the confirmation that since the decrease corresponds to that in the volume fraction of ferrite, the primary takes place within the ferrite phase by Cottrell's mechanism. The softening observed at about 200°C with lower carbon steels, is more marked the larger the primary hardening. In Fig. 2 are

revealed annealing time-hardness curves at the illustrated temperatures with a 0.9 % C steel cold rolled to 50 % reduction. On the assumption that the degree of the primary hardening is identical to that of the

Fig. 1 Strain age hardening curves of carbon steels. Annealing time : 3 mins.

a : 0.02 % C steel 55 % rolled
b : 0.11 % C steel 55 % rolled
c : 0.90 % C steel 65 % rolled

softening at 200°C, and that with higher carbon steels the softening is not apparent although it occurs actually, it may be acceptable that the degree of hardening obtained from the curves in Fig. 2 are equal to that of secondary strain age hardening. The activation energy for the secondary hardening obtained from the results was about 27000 cal/mol. This value is

Fig. 2 Strain age hardening curves of 0.9 % C steel 50 % rolled. a : 215°C, b : 280°C, c : 320°C, d : 350°C

much higher when compared with that of the primary, say, about 18000 cal/mol, and it seems to be almost irrespective of the carbon content.

The degree of the secondary hardening is largely affected by the carbon content as shown by curve (a) in Fig. 3. Here, the degree was determined from $H_1 - H_0/H_0 \times 100$, in which H_0 and H_1 are the hardness in the as-worked state and the maximum hardness in strain age hardening curves as shown in Fig. 1, respectively. The degree of the secondary hardening is the larger the higher the carbon content unlike the primary, strongly supporting the view that the secondary hardening is associated with the dispersion of cemen-

tite or the phase boundaries between ferrite and cementite rather than the ferrite itself, since the cementite itself is unlikely to induce the hardening. In addition, the degree is low and remains constant

Fig. 3 Secondary strain age hardening, inelastic effect and carbon content in carbon steels.

a : 55 % rolled
b : 6 % prestrained

below 0.2 % C. It is noteworthy that the carbon content above which the secondary hardening becomes abruptly marked is in general agreement with the critical carbon content at which the stress concentrated around cementite particles during deformation mutually interacts⁽⁹⁾.

Fig. 4 shows the relation of the degrees of cold

Fig. 4 Degrees of secondary strain age hardening and cold rolling in 0.9 % C steel.

a : Lamellar pearlite
b : Globular pearlite
(720°C for 10 hours tempered)

work and secondary hardening in a 0.9 % C steel. The degree of the hardening is much less in a coarse globular pearlitic structure than in a fine lamellar, and it increases significantly beyond about 40 % reduction, being probably due to the fact that cementite particles are markedly fragmented by heavy working. In addition, it may be noted from Fig. 4 that the secondary hardening is pronounced from the start of the straining unlike in the case of α -brass, in which the strain age hardening becomes steeply marked beyond a certain degree of cold working, that is, about 15 %⁽⁶⁾.

2. The Bauschinger effect of carbon steel

(1) Relation between yield stress and hardness

Hardening would be expected to be accompanied by a rise in yield stress, but in α -brass, the rise corresponding to the strain age hardening could not be

detected upon measuring under the same kind of stress and direction as those of prior working. To be certain of this relation in carbon steel, tension yield stress measurements were made.

Fig. 5 Change of yield stress of extended carbon steels with annealing temperature.

Curves (a) in Fig. 5 show the changes of yield stress on 0.1 % C and 0.9 % C steels in the same direction as the primary tension of about 5 % strain with annealing temperature. Here, the annealing time was 4 minutes, and the change in yield stress is given by the ratio of yield stress τ' after annealing at the temperatures illustrated on the abscissa to the flow stress τ of pre-tension. The yield stress is raised markedly within the temperature range of the primary strain age hardening. While, it shows a dissimilar trend to that of hardness (curve b) beyond 200°C, and especially in low carbon steel, there is no rise in yield stress corresponding to the secondary hardening, but rather a lowering of yield stress similar to α -brass. With 0.1 % C steel, since the hardness distribution is non-uniform in lower ranges of tensile working and is difficult to measure accurately, it was necessary to measure a specimen rolled to a high reduction. The effect of the kind of cold working on the change in hardness with low temperature annealing is slight unlike α -brass as will be stated in a later paper. Accordingly, it may be allowable to consider that the change in hardness shows a similar trend both in cold rolled and extended specimens. In high carbon steels, the yield stress exhibits only a slight rise, and its temperature is appreciably lower than that of the secondary hardening, leading to the presumption that the degree of the secondary hardening varies with the directions. The Vickers hardness is regarded as being nearly independent of the direction. Thus, when the secondary strain age hardening is measured by unidirectional stress, the condition of cold working must be considered.

(2) Bauschinger effect

To obtain further information on the directionality above stated, the experiments of Bauschinger effect

(9) G. Mima and S. Hori : J. Japan Inst. Metals, 16 (1952), 361. (In Japanese.)

were carried out. In the experiments, specimens were first twisted followed by low temperature annealing at zero load, and then re-twisted in a direction opposite to the prior working. The results with a 0.9

Fig. 6 Torsion stress strain diagram of 0.9 % C steel.

- a : Untreated
b : 200°C annealed
c : 300°C annealed

% C steel are given in Fig. 6. As shown by curve (a), which concerns a specimen without low temperature annealing, it shows a marked Bauschinger effect. However, the yield stress in the opposite direction is raised by low temperature annealing and the Bauschinger effect is removed (curves b and c). The change of the ratio of yield stress λ/λ_0 with annealing temperature is revealed by curve (a) in Fig. 7. Here, λ and λ_0 are noted in Fig. 6 and the ratio λ/λ_0 is

Fig. 7 Removal of Bauschinger effect in 0.3 % C steel.

- Prestraining
a : 1.4 % shear strain
b : 50 % rolling

smaller the more marked the Bauschinger effect. The shape of the curve bears a close relation to that of the strain age hardening (curve b), it differing widely from that of the change of yield stress in the work direction (curve a in Fig. 5). It may be noteworthy that the higher the carbon content in carbon

steels the less the increase of λ/λ_0 below 150°C and the more that in the range of 250-320°C⁽¹⁰⁾, showing a similar trend to that of the change in hardness.

As shown in Fig. 8, the higher the degree of prior

Fig. 8 Degrees of Bauschinger effect and prior working in 0.9 % C steel.

- a : Lamellar pearlite
b : Globular pearlite

torsion the more marked the Bauschinger effect, and in addition, the degree of this effect λ_0/λ' is much less for a coarse globular pearlitic structure (curve b) than for a fine lamellar structure (curve a), the tendency being in good agreement with that of the secondary strain age hardening (Fig. 4). Here, since the degree of the Bauschinger effect is hard to measure in such alloys as showing a marked effect, it was obliged to represent by the ratio of the flow stress λ_0 of prestraining to the stress λ' corresponding to permanent strain 0.03 % in the reverse direction as shown in the upper part of Fig. 8.

For obtaining more exact information on the subject, the inelastic effect in extensively worked specimens was studied as in the case of α -brass⁽⁶⁾. It is more convenient to measure in the range of higher degrees of cold working than in the case of the Bauschinger effect. The inelastic deformation being considered to commence upon unloading after prior working at the point where the stress strain curve departs from the straight line as shown in Fig. 6, the ratio λ_1/λ_0 was taken to represent the degree of inelastic effect. Here, λ_0 and λ_1 were noted in Fig. 6. Experiments were made by tension at room temperature. Strain rates on loading and unloading were about $4 \times 10^{-4} \text{ sec}^{-1}$ and $4 \times 10^{-5} \text{ sec}^{-1}$, respectively. As given in Fig. 9, the inelastic effect of carbon steels becomes marked by increasing the working degree, and the shape of the curve is generally conformable to that shown in Fig. 4. Furthermore, the ratio increases with increasing carbon content, and the trend of this increase at 6 % tensile strain (curve b in Fig. 3) is in

(10) S. Kumakura: Meeting of Japan Soc. of Mech. Eng., (1958). (In Japanese.)

good agreement with that of the secondary hardening (curve a in Fig. 3) as in the case of α -brass⁽⁶⁾. Hence, it may follow that the more marked the inelastic effect the larger the secondary strain age hardening.

Fig. 9 Inelastic effect and tensile strain.
a : 0.3 % C steel
b : 0.5 % C steel
c : 0.7 % C steel
d : 0.9 % C steel

(3) Strain age hardening of alumina dispersed metals

In the case of carbon steels, the diffusion of carbon atoms is relatively active in the temperature range of the secondary hardening, and the cause of the hardening has been considered to be on the basis of a chemical change such as precipitation or dissolution of cementite. To clarify the above effect, the strain age hardening of metals in which thermally stable particles disperse was investigated. Although there was an observation with reference to the subject⁽¹⁾, the experiments were done in detail in the temperature range of the strain age hardening. The result on sintered Fe-5 vol % Al_2O_3 alloy is shown by curve (c) in Fig. 10, in which marked strain age hardening is observed in the range of 150-300°C, the result being generally conformable to that already found⁽¹⁾. The degree of the hardening is about 10 %, which is appreciably higher than that of a sintered iron without dispersion of alumina particles⁽¹⁾. Since dispersed alumina particles are thermally stable, there may be no chemical change during low temperature annealing, in addition, the primary strain age hardening below 150°C could hardly be detected. It may, therefore, be relevant to say that the contents of carbon and nitrogen are appreciably low.

Alumina dispersed copper which was obtained by internal oxidation of a Cu-0.52 % Al alloy, also shows a marked hardening (curve b), and its degree is about 14 %, this value also being considerably higher than that of pure copper prepared by melting electrolytic copper (curve a). A similar result was also obtained

in a sintered Cu- Al_2O_3 alloy. It may be pertinent to mention from these results that dispersed particles affect the strain age hardening, and thus that the secondary hardening of carbon steel must be considered

Fig. 10 Strain age hardening of alumina dispersed metals.
a : Pure copper 70 % rolled
b : Cu- Al_2O_3 alloy 16 % rolled
c : Fe- Al_2O_3 alloy 45 % compressed

from the view point of the dispersion of cementite particles as an obstacle against dislocation during deformation rather than the movement of carbon atoms within the ferrite during low temperature annealing.

IV. Consideration

Since the primary strain age hardening can be reasonably explained by the Cottrell mechanism, secondary hardening will be chiefly treated in this paper.

1. In carbon steels, the ferrite phase alone shows only a slight Bauschinger effect and some strain age hardening. The fact that these are both more marked in higher carbon steels, may lead to the presumption that they are related to the distribution of the cementite particles or platelets as an obstacle against dislocation. The theory based on a chemical reaction such as dissolution or precipitation of cementite may be unacceptable for the reasons that the iron alloy in which thermally stable alumina particles finely disperse shows a hardening similar to that of carbon steels, and that the increase in hardness does not necessarily need low temperature annealing as will be reported in a later paper. The internal stress theory has already been proposed by Andrew and Lee⁽⁵⁾, but it does not clarify the relationship between the internal stress and hardening. It is also felt from the present investigation that the internal stress has a close relationship to the hardening. However, as further investigations are required on this subject, details will be described in a later paper.

2. The trend of the removal of the Bauschinger effect on low temperature annealing is in good agreement with that of strain age hardening, and the more marked the Bauschinger effect the larger the strain age hardening as shown in Fig. 3.

The cementite particles distributed in the ferrite act as an obstacle against dislocation during deformation, generating pile-ups of dislocation and stress concentration around them. On unloading or after unloading, the dislocations are easy to move back under the influence of the back stresses, by which the Bauschinger effect is induced. The back stress is generated by various causes, but in this case it is considered to originate chiefly in the pile-up dislocations around the particles.

The removal of the Bauschinger effect may take place by the prevention of motion back of dislocation under the influence of the back stress. The prevention is considered to concern a dislocation-dislocation interaction. A mechanism based on a locking of dislocation by the solute atom may not be significant in the case of the secondary strain age hardening of carbon steels for the reasons stated above. Thus, a possible mechanism by which dislocations in pile-up groups become difficult to move back is a rearrangement of dislocations, such as formation of sessile dislocation⁽¹¹⁾ or a mutual reaction of dislocation groups⁽¹²⁾ around the obstacle, or climb of dislocations. These and a comparison with the strain age hardening of α -brass will be discussed in detail in a later paper.

The fact that a rise in yield stress corresponding to strain age hardening was not apparent (Fig. 5), may be explained in the following manner. The rearrangement of dislocations occurs at the expense of the back stresses, by which the yield stress in the work direction will be lowered. Even if it is raised by the rearrangement, it hardly increases as a whole since the two cancel out each other, but rather decreases.

3. The degree of the strain age hardening differs widely depending upon the measurement method. This may be readily recognized from the fact that nearly no rise of yield point corresponding to hardening could be detected upon measuring under a stress of the same kind and direction as those of prior working, and that the trend of the elimination of the Bauschinger effect, in other words, the trend of the increase of yield stress in the opposite direction, was closely similar to that of the increase in hardness.

Fig. 11 gives a schematic polar representation of the yield stress in various directions after unidirectional straining. Since the yield stress of carbon steels extended or compressed varies widely with the direction and it is highest in the work direction and lowest in the opposite direction. On the basis of Saito's⁽¹³⁾ and Kumakura's⁽¹⁰⁾ results, the yield stress distribution after unidirectional working would be expected to be as shown by the solid line in the figure. Point A reveals the yield stress in the work direction, and points B or D and C are those in the transverse and opposite directions, respectively. In a strain age hardened state, the change in the work direction is slight but marked in the other directions, the yield stress being considered to show a distribution as

represented by the broken line. As understood from this model, the degree of the increase in yield stress by low temperature annealing varies with the direction of measurement. The Vickers hardness is considered

Fig. 11 Schematic polar representation of yield stress.
Solid line: As worked state
Broken line: Strain age hardened state

to be insensitive to the direction, and thus it would show a mean value of the yield stresses in all directions. Although the hardness is a complicated property, it may, to some extent, be beneficial for the measurement of the strain age hardening rather than a unidirectional property.

The anisotropy in the strain age hardening mentioned above concerns cold working unidirectionally applied. While, when cold working is made by rather complicated stress as is the case of rolling or drawing, an anisotropy in strain age hardening is less marked although it is also observed. In addition, the degree of the hardening differs with the measurement method. The increase in the Vickers hardness by low temperature annealing of a 0.9 % C steel cold rolled to 50 % reduction is about 20 %, and that in ultimate tensile strength is 5 %, being appreciably smaller. While the increase in proportional limit is very large and in the range 50-300 %, varying with the directions. In general, an alloy showing marked strain age hardening gives only a slight increase in proportional limit by cold rolling, whereas by low temperature annealing it exhibits a marked increase. This may be readily recognized from the fact that the more marked the Bauschinger effect the larger the strain age hardening.

V. Conclusion

A study on the strain age hardening of carbon steels in the range of 150° to 350°C was carried out and the results obtained are as follows.

(1) Strain age hardening occurs in two stages on annealing after cold working. Primary hardening below 150°C takes place within the ferrite phase and it may be explained by Cottrell's mechanism. While, the secondary in the range of 150-350°C becomes marked with increasing carbon content, showing that

(11) M. J. Makin: *Phil. Mag.*, **3** (1958), 287.

(12) H. K. Birnbaum: *Acta Met.*, **9** (1961), 320.

(13) K. Saito: *Trans. Japan Soc. Mech. Eng.*, **20** (1954), 771, 777.

this hardening is associated with the dispersion of cementite particles or platelets as an obstacle against dislocation. This was also confirmed by the results with reference to the metals in which alumina particles finely disperse.

(2) As shown in Fig. 5, an increase of yield stress corresponding to the secondary strain age hardening could hardly be detected upon measuring under a stress of the same kind and direction as those of prior working. In addition, higher carbon steels exhibit marked Bauschinger effect as in the case of α brass, and it was confirmed that its removal during low

temperature annealing is considered to be associated with the secondary strain age hardening.

(3) The cementite particles or platelets distributed in the ferrite act as an obstacle against dislocation during deformation, generating pile-ups of dislocation. On unloading or after unloading, the dislocations in the groups are easy to move back under the influence of the back stresses, by which the Bauschinger effect is induced. It is inferred that the strain age hardening of carbon steel is associated with the hindrance of motion back of the dislocations, and this will be discussed in a later paper.