Equivalent Rule of Strain Aging in Carbon Steel*

By Takenobu TAKEDA**, Yasuo NASU***

and Eiryo SHIRATORI****

The rate equation and the equivalent rule of strain aging in carbon steel were derived from age hardening curves taken in the temperature range from 170°C to 250°C after a compressive prestrain. The maximum increment in hardness increased as the temperature rose and the prestrain became larger. The process of aging could be expressed by a rate equation of homogeneous and first-order reaction. The equivalent rule of strain aging was formulated in consideration of both the maximum value of hardening and the prestrain. It was shown that the applied rule permitted a precise estimate of equivalent aging time at the aging temperature range of second stage.

Key words: Heat Treatment, Strength, Carbon Steel, Strain Aging, Equivalent Rule, Rate Process, Activation Energy.

1. Introduction

When a prestrained carbon steel is aged at the temperature range of second stage^{(1),(2)} and then reloaded, the yield point reappears and in the reverse direction to prestraining the Bauschinger effect is partially removed. Strain aging is considered effective for a qualitative improvement of static⁽³⁾ and fatigue strengths^{(4),(5)} in structural steels.

When strain aging is used for the improvement of materials, the equivalent rule of aging decided in consideration of the amount of prestrain, temperature and time is necessary for the quality control of materials. However, it appears that the rate equation^{(6),(7)} and the equivalent rule⁽⁸⁾ of aging previously reported, derived from the Cottrell-Bilby theory⁽⁹⁾, can not precisely express the aging phenomenon at the second stage, and they hold only at the first stage⁽¹⁰⁾. Furthermore, it appears that they are not useful in practical application because the maximum value of hardening is not taken in the rate equation of aging and the effect of the amount of prestrain is not taken into account in the equivalent rule.

In this paper, taking note of the above mentioned problem, the rate equation and the equivalent rule of aging were formulated in consideration of the maximum value of hardening and the amount of prestrain respectively. The applied rule was compared with the experimental results and discussed.

2. Specimen and Experimental procedure

The experimental procedure is as follows. The compressive prestrained specimen is cut in two at the cross section of central part. A pair of cut specimens are aged at a desired temperature and then hardness of each piece is measured at intervals of suitable aging time. Repeating the process of aging, interruption of aging and hardness measurement, until the increment in hardness reaches the saturation value, the age hardening curve is determined.

2.1 Specimen

The material used is a carbon steel supplied in the form of rolled bar of 25 mm in diameter. The chemical compositions and the mechanical properties are shown in Tables 1 and 2 respectively. The specimens are columns of 14 mm outer diameter and 28 mm height. They are fully annealed for 1 hr at 910°C in a vacuum furnace before testing.

2.2 Compression test

A compressive prestrain was given to the specimen using a 50 ton universal testing machine of hydraulic type. In order to reduce friction between the end surface of specimen and the platen, a compression test was performed using a teflon sheet and MoS₂.

Table 1 Chemical compositions (%)

C	Si	Mn	P	S	Nb	N	Al	Fe
0.24	0.23	0.49	0.022	0.017	0.029	0.0031	0.002	Re

Table 2 Mechanical properties of annealed material

Lower Yield Point	Tensile Strength	Elongation	Reduction of Area	Hardness
MPa		%		Hv
270	455	35.8	59.6	128

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** Associate, Faculty of Engineering, Yamagata University, 4-3-16, Jonan, Yonezawa, Yamagata.

*** Associate Professor, Faculty of Engineering, Yamagata University.

**** Professor, Faculty of Engineering, Saitama University, 255, Shimo-okubo, Urawa, Saitama.

as lubricant. By means of this method, the bulging of specimen was made very small and the maximum value of bulging was only 0.6 % when the compressive strain was 20 % in height. In addition, as the barrel-shaped bulging didn't occur at the central part of specimen, a uniform compressive strain during 6 mm in height was given to the specimen. The strain was obtained from the change in diameter by means of a snap gage with an accuracy of $1\mu\text{m}$ which directly contacted with the specimen. The amount of prestrain was chosen to be 1.83%, 3.32%, 7.65% and 11.87% in a perfectly unloaded state.

2.3 Hardness test

As an inhomogeneous deformation occurred at a place close to the end of specimen, hardness measurements were made on the cross sectional surface of central part. The prestrained specimen was cut in two at the central part with a precision cutter and the cutting surfaces were finished with a surface grinder. Cutting and grinding were performed spraying cold water on the specimen to avoid the influence of aging. As it was reported that the affected part due to cutting was removed by abrading above $0.1\text{ mm}^{(11)}$, the grinding surface of specimen was abraded about 0.15 mm with emery papers and a buffing machine. Hardness measurements were made using a Vickers hardness tester at 98 N load. The measured value was the average for ten measurements.

2.4 Aging procedure

The specimens were aged for a desired time at each temperature of 170°C , 200°C ,

225°C and 250°C in an oil bath. Subsequently they were quenched in an oil bath maintained at $0 \sim 3^\circ\text{C}$ and then cooled in a water bath of room temperature for the interruption of aging. The first oil cooling was made to prevent the influence of quench hardening. After hardness measurement, aging was continued again.

3. Experimental results and Discussions

Figures 1 (a)~(d) show the age hardening curves, where H_0 is hardness after the prestrain. The increase in hardness, ΔH , grows faster with a rise of temperature. This means that the age hardening phenomenon occurs in a kind of rate process. The maximum increase in hardness due to aging, ΔH_{max} , changes as shown in Fig. 2. The value of ΔH_{max} becomes larger with an increase of prestrain ρ_0^* and with a rise of temperature. The degree of process of aging, y , is represented by the ratio of ΔH at each aging time to $\Delta H_{\text{max}}^{(12)}$,

$$y = \Delta H / \Delta H_{\text{max}} \dots \dots \dots (1)$$

Assuming that the Johnson-Mehl equation ⁽¹³⁾ holds between the degree of process of aging

(a) Prestrain $\rho_0^* = 1.83\%$ ($H_0 = 129$)

(b) Prestrain $\rho_0^* = 3.32\%$ ($H_0 = 137$)

(c) Prestrain $\rho_0^* = 7.65\%$ ($H_0 = 152$)

(d) Prestrain $\rho_0^* = 11.87\%$ ($H_0 = 162$)

Fig. 1 Age hardening curves

Fig. 2 Relation between maximum increase in hardness and prestrain

y and the aging time t , the aging rate dy/dt is expressed as

$$dy/dt = k t^{n-1} (1-y), \dots \dots \dots (2)$$

where k : a rate constant and n : a time exponent. The integrated form of Eq.(2) is as follows;

$$\log \cdot \log \{1/(1-y)\} = n(\log k + \log t) + \log(\log e/n), \dots \dots \dots (3)$$

where e is a base in the natural logarithms. It is found that the assumption expressed by Eq.(2) is valid when the results in Fig. 1 lie on a straight line after plotting on the co-ordinate system of $\log \cdot \log \{1/(1-y)\}$ vs. $\log t$. In Figs.3 (a)~(d), an almost linear relation is obtained in all cases of prestrain. The slope of straight line is $n = 0.90 \sim 1.05$. Therefore, the age hardening phenomenon can be considered almost homogeneous and first-order reaction of $n=1$. Namely, the degree of process of aging is expressed as

(a) $\epsilon^* = 1.83 \%$

(c) $\epsilon^* = 7.65 \%$

(b) $\epsilon^* = 3.32 \%$

(d) $\epsilon^* = 11.87 \%$

Fig. 3 Relation between degree of process of aging and aging time

$$y = 1 - \exp(-kt) \dots\dots\dots (4)$$

Assuming that the rate constant k is controlled by aging temperature and aging occurs in a single thermally-activated process, k varies according to the Arrhenius equation,

$$k = A \exp(-Q/RT) \dots\dots\dots (5.a)$$

where

A : a frequency factor,
 Q : an apparent activation energy*,
 R : a gas constant (8.28 J / mole°K),
 T : an absolute temperature.

Taking logarithms of Eq.(5.a), the following equation is derived,

$$\log k = \log e (\ln A - Q/RT) \dots\dots\dots (5.b)$$

The relation between $\log k$ and $1/T$ is shown in Fig.4. The value of Q is deduced from the slope of straight line, $\tan \alpha$, and it is calculated by the following equation,

$$Q = R \tan \alpha / \log e \dots\dots\dots (6)$$

The relation between the apparent activation energy and the prestrain is shown in Fig.5. The value of Q decreases at first with an increase of prestrain and then becomes effectively constant.

Applying the relationship between hardness and flow stress, given by $H = C \sigma^{(14),(15)}$, where C is a constant and σ (MPa) is a tensile stress corresponding to 8 % strain, to the material used for this experiment, C varies from 0.26 to 0.29 depending on the amount of prestrain. Although it is difficult to estimate exactly the increase in flow stress from the increase in hardness due to aging, it is considered that Q almost corresponds to the activation energy gained in the process of the flow stress increasing after the end of yield point elongation.

On the activation energy in age hardening process in carbon steel, at temperatures below 100°C, Kumagai⁽¹⁶⁾ reported that

the activation energy Q , obtained from the process of the tensile yield stress increasing in a low carbon steel, varied from 87.9 kJ/mole {21 kcal/mole} to 146.4 kJ/mole {35 kcal/mole} with an increase of prestrain. At temperatures from 100°C to 250°C, on the other hand, Yamada⁽²⁾ measured the activation energy of 117.2 kJ/mole {28 kcal/mole} from the process of the tensile yield stress increasing in a drawn eutectoid carbon steel wire and also Rashid⁽¹⁷⁾ measured 144.3 kJ/mole {34.5 kcal/mole} from the process of the tensile yield stress increasing in a 2.8 % tensile prestrained high strength steel. In addition, Kawasaki et al.⁽¹²⁾ reported that the activation energy of 38.5 kJ/mole {9.2 kcal/mole} was obtained from the process of the hardness increasing in a 9% tensile prestrained low carbon steel. According to the experimental results and the study of Kawasaki et al.⁽¹²⁾, the activation energy obtained from the process of the hardness increasing has a tendency to be smaller than that obtained from the process of yield stress increasing. As to aging for a relatively short time, age hardening which occurs at temperatures below about 100°C is due to the formation of Cottrell atmosphere controlled by the diffusion of carbon and nitrogen atoms. This is termed the first stage of aging by Wilson et al.⁽¹⁰⁾. At temperatures from 100°C to 250°C, the Cottrell atmosphere is quickly formed and age hardening is mainly due to the precipitations of carbide and nitride. This is termed the second stage of aging by Kumakura⁽¹⁾ and Yamada⁽²⁾.

As described above, when the effect of prestrain on aging is evaluated by separating the age hardening into two stages, the results in Fig.5 show that the activation energy at second stage apparently decreases with an increase of prestrain. The case for a decrease of Q is considered as follows. It seems that the precipitation rates of carbide and nitride are essentially con-

Fig. 4 Relation between rate constant and aging temperature

Fig. 5 Relation between apparent activation energy and prestrain
 (1 cal/mole = 4.184 J/mole)

* As the maximum increase in hardness is dependent on aging temperature, the size and the volume fraction of aging precipitations may differ according to temperature. In this paper, the degree of process of aging, y , is introduced and the rate equation is expressed by an equation from which the term of maximum value of hardening is eliminated. In addition, assuming that the same aging reaction occurs within the temperature range of experiment, Q is determined from the change of hardness. Therefore, Q is termed an apparent activation energy.

trolled by the diffusion of carbon and nitrogen atoms⁽¹⁸⁾. The dislocation density increases with an increase of prestrain and cell walls are formed⁽¹⁹⁾. As such a place is easy to make a short-circuiting-diffusivity path of carbon and nitrogen atoms, it is expected that the diffusion coefficient increases through a mechanism of grain-boundary or pipe diffusion⁽²⁰⁾. It appears that the precipitations of carbide and nitride are promoted by an increase of the diffusion coefficient and Q decreases. A detailed examination on this problem will be reserved for a future study.

4. Formulation of Experimental results and Equivalent rule

Figure 6 shows the relations between the maximum increase in hardness and the temperature. Within the temperature range of experiment, these relationships are approximated by straight lines in all cases of prestrain.

$$\Delta H_{\max}(\rho_0^*, T) = F(\rho_0^*)T + G(\rho_0^*) \dots (7)$$

Parameters F and G are expressed as a function of prestrain from the results in Fig. 2: namely,

$$\left. \begin{aligned} F(\rho_0^*) &= f_1/(\rho_0^* + f_2) + f_3 \\ G(\rho_0^*) &= g_1/(\rho_0^* + g_2) + g_3 \end{aligned} \right\} \dots (8)$$

In Eq. (8), $f_1 \sim f_3$ and $g_1 \sim g_3$ are material constants. For the material used in this experiment, they are as follows;

$$\left. \begin{aligned} f_1 &= -9.79 \times 10^{-2} \\ f_2 &= 3.05 \times 10^{-1} \\ f_3 &= 3.19 \times 10^{-1} \end{aligned} \right\} \left. \begin{aligned} g_1 &= 6.50 \times 10 \\ g_2 &= 4.19 \times 10^{-1} \\ g_3 &= -1.50 \times 10^2 \end{aligned} \right\}.$$

The calculated results are shown by solid lines in Figs. 2 and 6.

The relation between the rate constant and the prestrain is shown in Fig. 7. The value of $\log k$ becomes larger with a rise of temperature, while it decreases with an increase of prestrain and becomes effectively constant. As shown in the foregoing chapter,

Fig. 6 Relation between maximum increase in hardness and aging temperature

the relationship between k and T is expressed by the Arrhenius equation, which is written as

$$k(\rho_0^*, T) = A(\rho_0^*) \exp\{-Q(\rho_0^*)/RT\} \dots (5.c)$$

The frequency factor and the apparent activation energy change as shown in Figs. 8 and 5, respectively. Both $\log A$ and Q decrease with an increase of prestrain and then tend toward respective limiting values. These changes are approximated by the following equations,

$$\log A(\rho_0^*) = p_1 \exp(p_2 \rho_0^*) + p_3 \dots (9)$$

$$Q(\rho_0^*) = q_1 \exp(q_2 \rho_0^*) + q_3 \dots (10)$$

In Eqs. (9) and (10), $p_1 \sim p_3$ and $q_1 \sim q_3$ are material constants, which are determined as follows;

$$\left. \begin{aligned} p_1 &= 3.54 \\ p_2 &= -35.10 \\ p_3 &= 1.81 \end{aligned} \right\} \left. \begin{aligned} q_1 &= 29.70 \times 10^4 \\ q_2 &= -35.84 \\ q_3 &= 19.50 \times 10^4 \end{aligned} \right\}$$

The curves in Figs. 4, 5 and 8 show the calculated results.

According to the above-mentioned method, $\Delta H_{\max}$ and k are obtained by performing experiments with three kinds of prestrains at respectively two temperatures, and the age hardening curves can be determined from Eq. (2). The curves in Fig. 1 show the calcu-

Fig. 7 Relation between rate constant and prestrain

Fig. 8 Relation between frequency factor and prestrain

Table 3 Equivalent aging times

ρ_0^* %	t min ($\Delta H_{\max}$)		
	150°C	200°C	250°C
3	14.3 (6.2)	2.6 (7.6)	0.9 (9.0)
5	7.8 (7.0)	2.0 (9.2)	0.8 (11.4)
10	5.5 (8.0)	1.5 (11.9)	0.6 (15.8)
15	5.2 (8.2)	1.3 (13.4)	0.5 (18.6)
Hundy's equation	22.5	2.0	0.3

lated results. There is a fairly good agreement between the calculated and the experimental results.

As the method of determining the age hardening curve has been established, the strain aging condition corresponding to the same amount of age hardening can be predicted. Now let us assume that the increase in hardness due to aging is ΔH_i when the specimen given the prestrain ρ_{0j}^* is aged for the time t_i at temperature T_i . The time t_j required to produce the same amount of age hardening under the condition of prestrain ρ_{0j}^* and temperature T_j , can be predicted by the following equation,

$$t_j = \ln \left\{ \frac{1}{1 - \Delta H_i / \Delta H_{\max}(\rho_{0j}^*, T_j)} \right\} / k(\rho_{0j}^*, T_j) \quad (11)$$

where $\Delta H_{\max}(\rho_{0j}^*, T_j)$ is a maximum increase in hardness under the condition of prestrain ρ_{0j}^* and temperature T_j . The strain aging conditions required to produce $\Delta H_i = 6$ are shown in Table 3 for an example. The value in parentheses is the maximum increase in hardness under each condition of prestrain and temperature. The time required to produce the same amount of age hardening becomes shorter with a rise of temperature and with an increase of prestrain. In order to compare with this study, the results calculated from Hundy's equation⁽⁸⁾, assuming that the mechanism of aging is controlled by the diffusion of carbon atom, are shown in the under side of Table 3. In Hundy's equation, since both the prestrain and the maximum value of hardening are not taken into account, the equivalent time to the aging condition of prestrain 5 %, time 2 min and temperature 200°C in the upper side of Table 3 is shown. Hundy's equation gives a longer estimated time in the case of low temperature and a shorter estimated one in the case of high temperature.

5. Conclusions

The rate equation and the equivalent rule of aging were derived from the age hardening curves for a compressive prestrained carbon steel. The following results were obtained.

- (1) At the aging temperature range of second stage, the maximum value of hardening becomes larger with an increase of prestrain and with a rise of temperature.
- (2) The process of aging at the second stage can be expressed by a rate equation of

homogeneous and first-order reaction.

(3) The apparent activation energy decreases with an increase of prestrain and then becomes effectively constant.

(4) The equivalent rule of aging was formulated in consideration of both the maximum value of hardening and the prestrain from six age hardening curves. It was shown that there is a fairly good agreement between the calculation and the experiment.

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