Sub Zero Treatment of Quenched Steel. III : Effect of Aging on the Stabilization of Retained Austenite

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Sub-Zero Treatment of Quenched Steel. III
Effect of Aging on the Stabilization of Retained Austenite*

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Synopsis

The stabilization of retained austenite after partial transformation to martensite has been investigated using 1.12 % C, 4.98 % Ni steel. The stabilization degree was expressed by the temperature lag θ of transformation point or the amount of the stabilized austenite, δ. It has been observed that the temperature lag θ is a function of aging time. It has been found that the recovery of martensite transformation occurred during subsequent cooling after aging in the early stage of stabilization, but did not occur in the last stage of it. Accordingly the correlation between θ and δ is not a linear relation. The stabilization proceeded very rapidly when the previously transformed martensite increases to more than 65 per cent.

I. Introduction

Generally, when cooling is interrupted at the martensite transformation temperature range and after holding isothermally at that temperature or at the higher temperature, cooling is subsequently continued, transformation occurs not immediately at the temperature where cooling has been interrupted but at the lower temperature where the super-cooling has a certain degree proceeded. In the case of the interrupted temperature being fixed, the higher the holding temperature (below critical temperature) and the longer the holding time (within critical time), the larger the super-cooling degree is. Accompanying this, the retained austenite becomes more stable and the transformation occurring point falls, furthermore, the amount of austenite retained to below the temperature where the transformation stops, so call M_f temperature gradually increases. The stabilization degree is expressed by the temperature lag of the transformation point caused by the super-cooling, or by the increasing amount of retained austenite. If there existed a linear relationship between the temperature lag and the amount of retained austenite it would be very convenient to derive one from the other, but it seems that there is not always such a simple relationship between them from the results of the present authors' previous works[(1)~(4)]; that is, in the early stage of stabilization, *Ms*' point fell and it seemed as if the stabilization proceeded, but as soon as the transformation resumed a rapid recovery of martensite transformation occurred[(1),(5)], and when the sub-zero cooling temperature reached M_f point the amount of martensite was similar to the case without aging

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The present experiment was carried out to clarify this stabilization phenomenon more in detail.

II. Experiments

The chemical composition of the specimens used for this experiment was C 1.12 per cent, Si 0.27 per cent, Mn 0.33 per cent, Ni 4.98 per cent, P 0.003 per cent and S 0.017 per cent. The size of the specimens was 5 mm in diameter and 70 mm in length. The specimens were held for 20 minutes at 1000°C in vacuum of 10^{-4} mm Hg, and water-quenched at the room temperature. The *Ms* point was determined by Greninger-Troiano(6) method, and for the determination of *Ms*' point and the amount of retained austenite a low temperature dilatometer, a low temperature ballistic galvanometer type apparatus for magnetic analysis and a microscope were used. To obtain the martensite transformation curve in continuous cooling, specimens, to avoid the interruption of cooling, were quenched into alcohol in which preliminary dropped liquid nitrogen to hold it at the desirable temperature, and by this method it was able to hold down to about −80°C.

III. Results

As mentioned in the introduction, for the measurement of the stabilization degree, two methods are known, that is, by the temperature lag, θ, of the transformation point due to the super-cooling, and the amount of the austenite, δ, retained to below M_f point. θ is expressed as follows:

$$\theta = T_h - Ms' \quad \text{......(1)}$$

where T_h refers to the holding temperature after the cooling was interrupted. Consequently, θ expresses the temperature range of the super-cooling due to the stabilization. On the other hand, the amount of the retained austenite increased by the stabilization, δ, is expressed by Cohen(7) as follows:

$$\delta = K(\sigma_s - T_h) \quad \text{......(2)}$$

where σ_s refers to the uppermost critical temperature within which the stabilization can occur, and K is the constant. And σ_s is obtained by the next equation.

$$\sigma_s = 0.57(Ms) + 26°F \quad \text{......(3)}$$

In the present experiment, it is necessary to measure θ which varies with the progrees of aging. For this purpose it will be very convenient if a correlation between θ and the aging time can be obtained, and the same can be said as for δ.

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(7) M. Cohen, *Phase Transformatiou in Solids*, (1951) 615

First, as for θ, as shown in Fig. 1, it is seen that θ has a functional relationship to the aging time. θ becomes large with the prolongation of the aging time, and approaches a certain fixed value. When this critical value of θ is put as

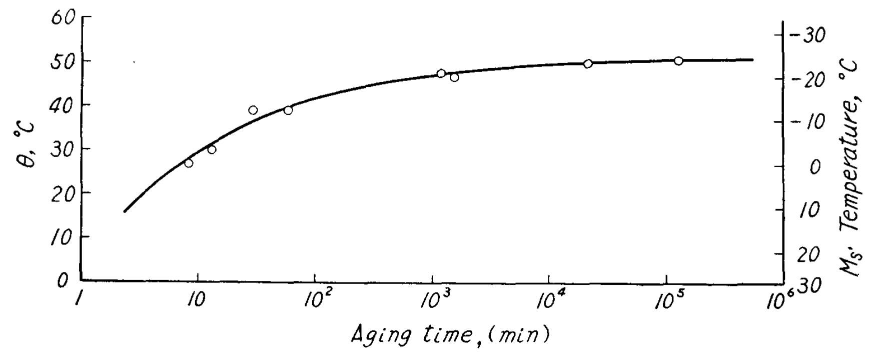

Fig. 1. Effect of aging time at room temperature on *Ms*' point and the temperature lag θ in 1.12% C, 4.90% Ni steel, austenitized at 1000°.

θ_{max}, the relation between P, the ratio between θ and θ_{max}, and the aging time can be expressed by the following equation:

$$\frac{1}{1-P}=kt^n \qquad (4)$$

where k is the constant. The value of θ_{max} was obtained by the experiment using a specimen tempered at 80°C. In the case of tempering at 80°C for 1 hour, *Ms*' point became lowest, however, in the case of further tempering, *Ms*' point rose. Thus, from the lowest value of *Ms*' point, 52°C was obtained as the value of θ_{max}. Putting the values of this θ_{max}, θ and t in Fig. 1 into the equation (4), the correlation between $\log\frac{1}{1-P}$ and $\log t$ was obtained as the line shown in Fig. 2, from which the following was obtained.

$$\theta=52-48t^{-0.34} \qquad (5)$$

Eq. (5) can be applied to every cases unless the aging time is very short. Therefore this may be enough to the present experiment.

The relationship between *Ms*' point and the aging time could be obtained from Eqs. (1) and (5).

$$Ms'=48t^{-0.34}-25 \qquad (6)$$

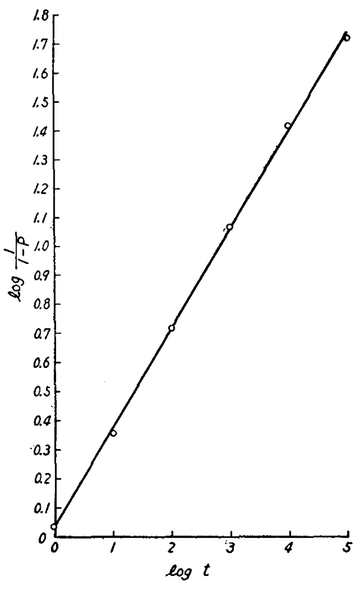

Fig. 2. Correlation between $\log\frac{1}{1-P}$ and $\log t$

Fig. 3 shows the martensite transformation curves, that is, the relationship between the amount of matensite or retained austenite and the cooling temperature. The outermost curve in the figure is the martensite transformation curve of the specimen continuously cooled from the quenching temperature down to M_f point, and that is the not stabilized normal transformation curve. The *Ms* point of this specimen was about 84°C. When cooling was interrupted at the room temperature, about 52 per cent of austenite retained. After this, when subsequent aging at room temperature was carried out, for 1 hour, *Ms'* point fell and the transformation

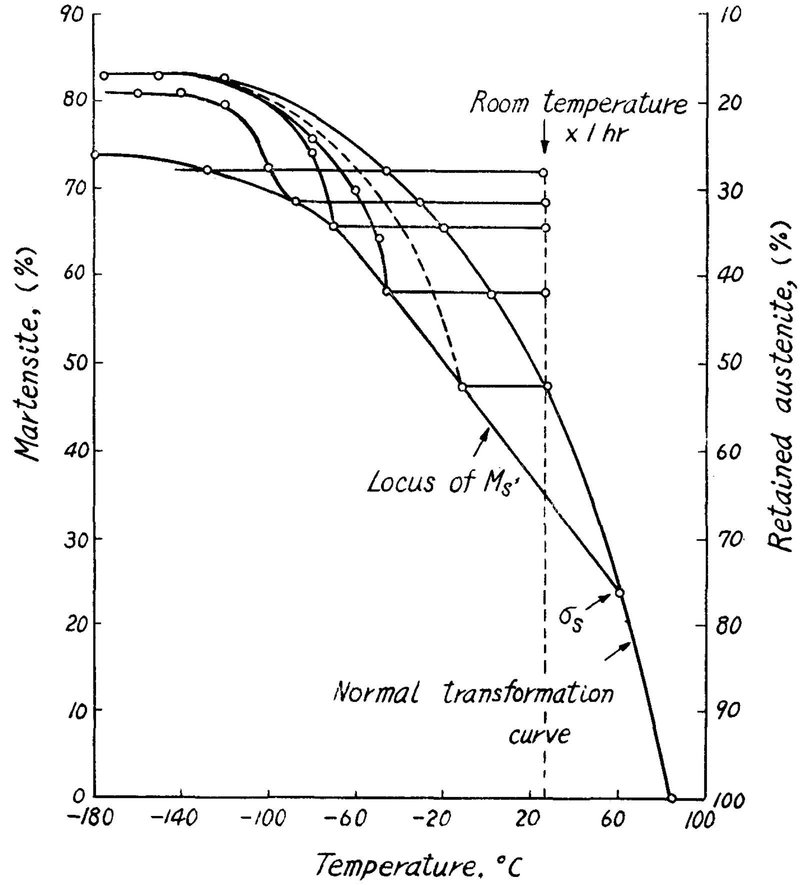

Fig. 3. Martensite transformation curves after quenching from 1000° to various temperature and subsequent aging at room temperature for 1 hr.

did not occur down to −12°C. When it reached *Ms'* point, transformation immediately occurred and proceeded as shown by the dotted line in the figure.

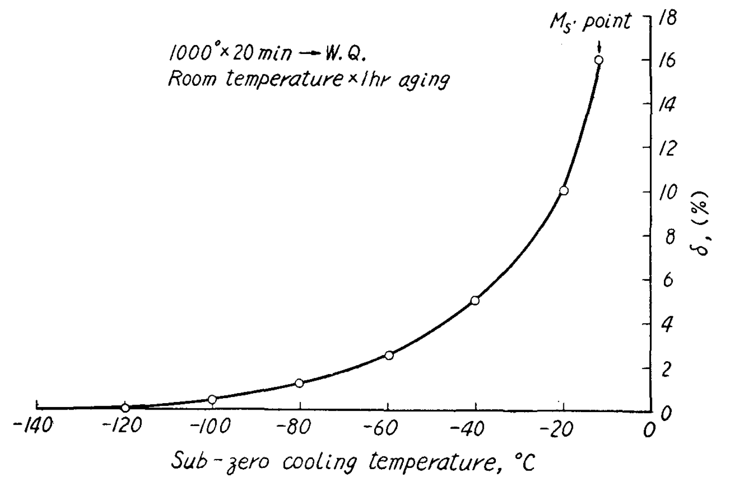

Fig. 4. Effect of sub-zero cooling temperature on the amount of stabilized austenite δ.

Here it should be noticed that the difference between this curve and the normal transformation curve, δ, decreased with the fall of the temperature and at about −120°C there was little difference between them, that is martensite transformation completely recovered.

Fig. 4 shows the effect of sub-zero cooling temperature on the amount of stabilized austenite, δ. In the figure δ is zero at −120°C, this is the phenomenon in the early stage of stabilization, that is, the phenomenon of within 1 hr's aging, (as will be mentioned later) with the further prolongation of aging time the amount of stabilized austenite gradually increased. After cooling was kept down to −90°C, aging was carried out for 1 hr at room temperature, the stabilized austenite, δ, also retained to below M_f point as shown in Fig. 3. This seems to be due to the progress of stabilization with the increase of the amount of martensite produced by the initial quenching. Generally it has been observed that the larger the amount of the prior martenste, the easier the stabilization is.[(5),(7)~(17)]

Fig. 5 shows the relation among the amount of the martensite produced by the initial cooling, the temperature lag. θ, and the amount of the stabilized austenite, δ. As for θ, as seen in the figure, the relation between the prior martensite and θ seems to be approximately linear, being slightly change at the point where the amount of martensite is about 65 per cent, while as for δ, it is almost completely recovered up to about 65 per cent of the prior martensite but above this point austenite retained to below M_f point immediately increases. Concerning this, it has been reported[(18)] that in the case of 0.8 per cent C steel, when the amount of martensite exceeds 75 per cent, the stabili-

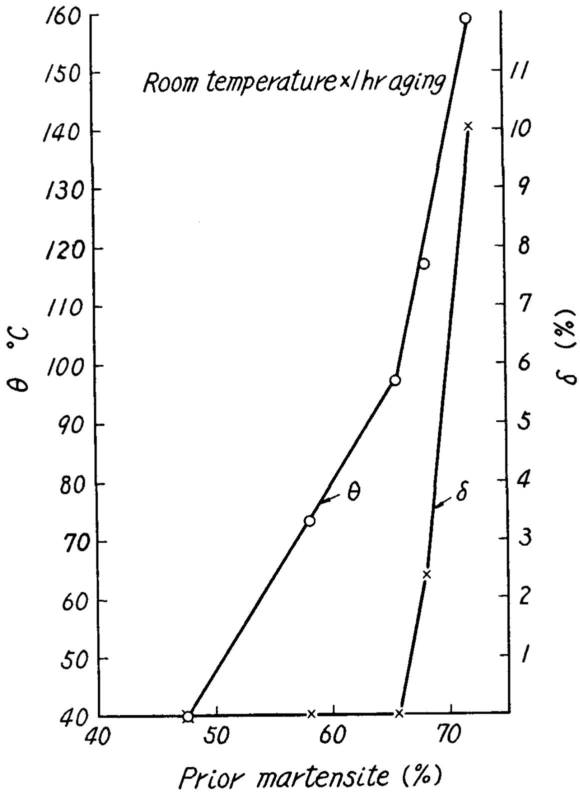

Fig. 5 Effect of the prior martensite on the temperature lag θ and the amount of stabilized austenite δ.

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zation extremely proceeds.

Next, the critical temperature for stabilization, σ_s, can be expressed by Eq. (3), and the calculated value of σ_s from this equation is in good coincidence with the intersecting point of the locus of *Ms*' point and the normal transformation curve,as shown in Fig 3. The value is approximately 60°C. However, it have been reported [(19)~(27)] that in the case of isothermal holding above *Ms* point, stabilization can occur, and the stabilization makes *Ms* point fall. The present authors too reported in the previous paper[(3)] that *Ms* point fell caused by the isothermal holding above *Ms* point. From this fact the following can be said that σ_s, obtained from Eq. (3) and shown in Fig. 3, may be the upper critical temperature within which the stabilization can take place in the range below *Ms* point. However, if the stabilization below *Ms* point occurs by the same mechanism to that above *Ms* point, that is, the two are a series of a phenomenon; σ_s becomes to be not so important and it is merely apparent critical temperature in the range below *Ms* point. To clarify this, it is necessary to make the comparative investigation of the stabilization mechanisms at above and below *Ms* point, and the detail will be mentioned in the following paper.

Fig. 6 shows the martensite transformation curves of specimens aged for various times at room temperature. As far as the aging time is short, the recovery of martensite transformation occurs and the curve coincides to the normal transformation curve at about M_f point, while with the prolongation of aging time the amount of retained austenite δ increases. This tendency, as seen in Figs. 3 and 5, is the same to the case of increase of the amount of prior martensite.

Fig. 7 shows the relationship between the amount of retained austenite, δ, and aging time. As far as the aging time is 1 hr, the recovery of martensite transformation is seen and δ is zero, while with the prolongation of aging time a part of the stabilized austenite begins gradually to retain to below M_f point.

From the above-mentioned results it is seen that when the amount of prior martensite is large or when the aging time is long, the stabilized austenite, δ, retains, however, when the amount of prior martensite is small or when the aging time is short, that is, in the early stage of stabilization, if cooling is carried out down to M_f point, martensite transformation recovers nearly completely, and the amount of martensite becomes to be as similar to the case not stabilized.

Next, it is investigated whether there is a linear relationship between δ and

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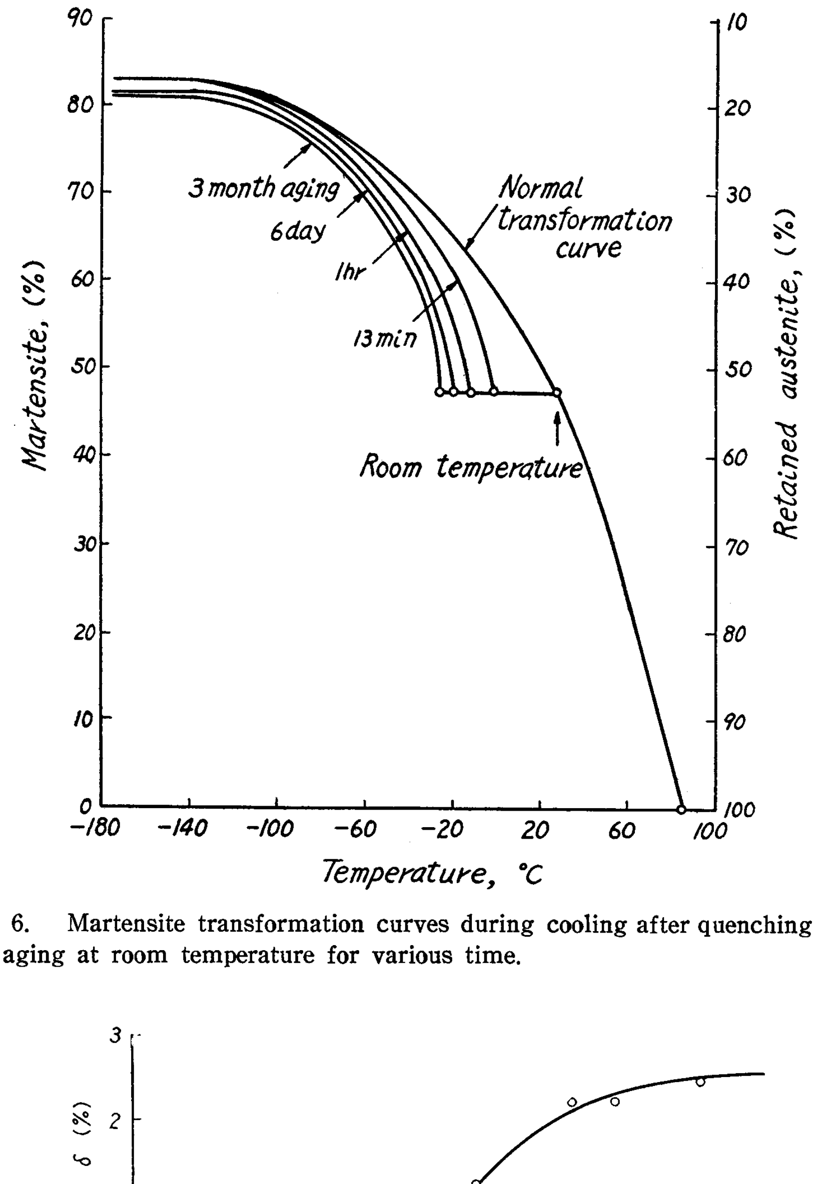

Fig. 6. Martensite transformation curves during cooling after quenching and aging at room temperature for various time.

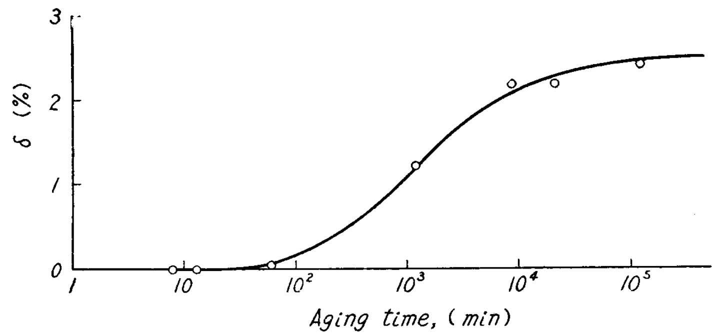

Fig. 7. Effect of aging time at room temperature on the amount of stabilized austenite δ.

θ, if a linear relationship exists between them, it will be very convenient to estimate one from the other. However, from the experimental results it is likely not to exist such a simple relationship between them. Fig. 8 shows the correlation between the temperature lag, θ, and the amount of stabilized austenite, δ, of the specimens hardened in water and aged at room temperature for various periods of time. The relationship between δ and θ at Ms' point is nearly linear, while that at M_f point is not linear, δ decreases by the occurring of the recovery of martensite transformation, and when θ is within about 35°C the transformation completely recovers and δ becomes to be zero. Then, retained austenite gradually increases. This fact indicates that the relationship between δ and θ is not simple.

From the above results, it can be concluded that the stabilization phenomenon is not expressed by a simple figure, but by a complicated figure (with curved plane) as shown in Fig. 9, that is, when the stabilization temperature is constant the amount of transformed martensite by subsequent sub-zero cooling after aging is represented by the curved plane produced by the axes which refer to cooling temperature, line OA, and aging time, line DO. Consequently the curve AE indicates *Ms'* point which varies by the difference of aging time. Since point A refers to the interrupt temperature of cooling, GE, the difference of line AG (parallel to line OD) and curve AE,

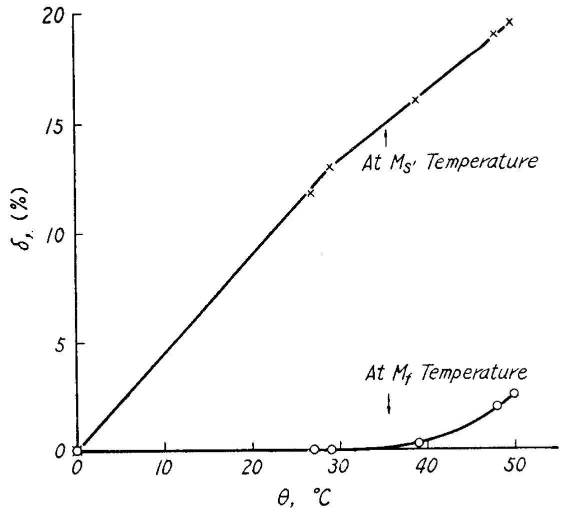

Fig. 8. Correlation between the temperature lag θ and the amount of stabilized austenite δ.

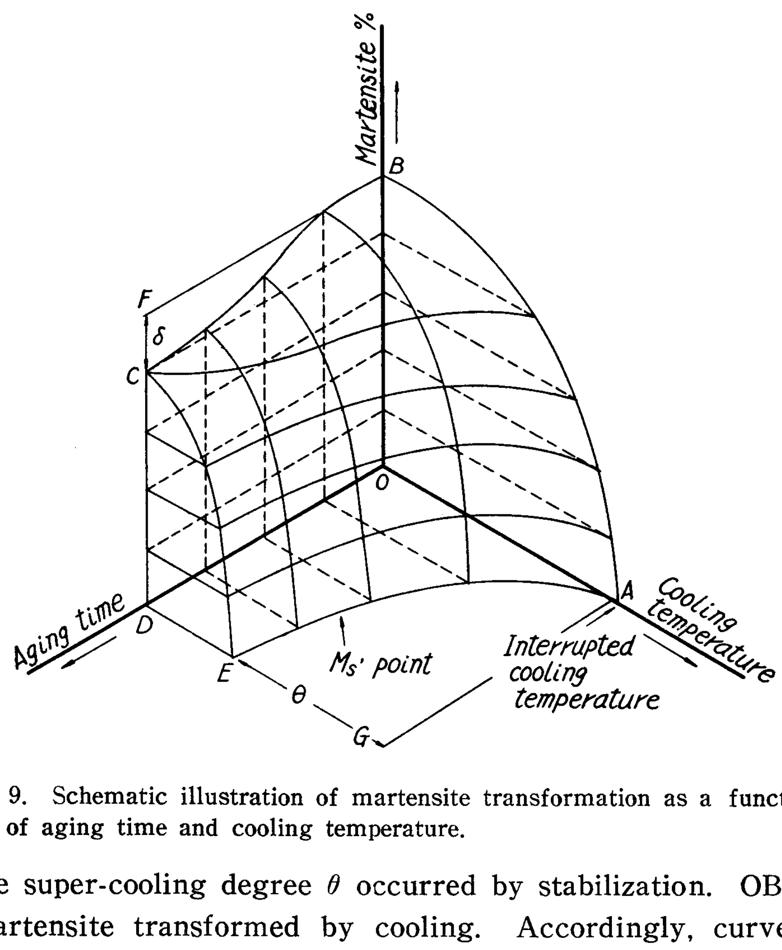

Fig. 9. Schematic illustration of martensite transformation as a function of aging time and cooling temperature.

represents the super-cooling degree θ occurred by stabilization. OB refers to the amount of martensite transformed by cooling. Accordingly, curve AB is the martensite transformation-temperature curve. Curve BC represents the relationship between aging time and the amount of martensite, and it shows that with the prolongation of aging time the amount of martensite transformed by the subsequent cooling decreases due to the occurring of the stabilization. The difference between line BF, parallel to OD, and curve BC represents the increasing amount of stabilized austenite, δ. Consequently, the curved plane ABCE represents the amount of the martensite transformed by the subsequent cooling, after aging for some time.

Summary

The stabilization phenomenon of the retained austenite of 1.12 per cent C and 4.98 per cent Ni steel was investigated and obtained the following results.

(1) The temperature lag, θ, of the transformation point and Ms' point is expressed as the function of aging time.

(2) In the early stage of the stabilization, the amount of the retained austenite decreased with the lowering of sub-zero cooling temperature and stabilization completely recovered at about M_f point. However, the amount of not recovered retained austenite increased with the increase of the stabilization.

(3) There was not a simple linear relationship between θ and δ. Consequently it was impossible to derive one from the other.

(4) The larger the amount of prior martensite, the easier the stabilization was, especially the stabilization remarkably progressed when the amount of martensite was more than 65 per cent.