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Effects of preheating treatment on repeated martensitic transformation under cyclic heating and cooling

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Hot-worked die steel is usually used under conditions requiring repeated heating and cooling. The repeated transformation of austenite to martensite or vice versa may occur on the surface of the hot-worked die. The characteristics of repeated martensitic transformation and the effects of preheating treatments have been investigated using a Formaster-F fully automatic transformation dilatometer. The results show that the volume fraction of martensite, f_m, formed in 750 °C→ 20 °C repeated cycling is changed with the cycling number, (N), and the preheating treatment has an important influence on the kinetics of martensitic transformation.

1. Introduction

The maximum temperature at which a hot-worked die steel can be used for prolonged periods is determined, among other factors, by its strength and resistance to softening at elevated temperature. The maximum possible strength of the hot-worked die steel is required both to resist plastic deformation and to minimize heat checking which is associated with thermal fatigue. Malm and Norstrom [1] have shown that increasing the yield strength at the elevated temperature will result in greater resistance to both crack initiation and propagation by thermal fatigue.

While a variety of martensitic hot-worked die steels, such as AISI H13 and H19, is available which maintain high strength at temperatures up to 650 °C, there is none which can retain the same strength levels when the working temperature is above 650 °C. In contrast with martensitic hot-worked die steel, austenitic hot-worked die steel has excellent strength at elevated temperature. In particular, when the temperature is above 650 °C, the strength of this type of steel is much higher than that of martensitic type [2], which can retain the 600 MPa strength level at 750 °C. However, the large thermal expansion coefficient of austenitic steel causes very high thermal stresses on the surface of the hot-worked die during thermal cycling, which then leads to easy heat checking [3].

To obtain the desired high strength and low thermal expansion coefficient, a new kind of die steel with martensite–austenite duplex microstructure has been developed [4]. This steel with high nickel and chromium equivalents, however, has a lower critical transformation temperature, that is, $A_{c1} \simeq 560\,^{\circ}\mathrm{C}$. The hot-worked dies made of this steel will undergo repeated martensitic transformation during thermal cycling, if the working temperature is above the A_{c1} temperature. Unlike the conventional martensitic transformation, very little is known of the repeated martensitic transformation under cyclic heating and cooling. The objective of this work was the investigation of the characteristics and effects of preheating treatment on repeated martensitic transformation in a martensite–austenite dual-phase steel.

2. Experimental procedure

The chemical composition (wt%) of the steel tested is: C 0.486, Mn 5.65, Ni 1.93, Cr 2.21, Mo 2.01, V 1.95, Si 0.42, S 0.018, P 0.019. The steel was melted in a 100 kW induction furnace and cross-forged at 1200 °C to bars with 10 mm diameter and 300 mm length and then air cooled. The specimens for dilatometric experiments were machined to 3 mm diameter and 10 mm length from pieces of bars which had been softened by annealing at 880 °C for 3 h and furnace cooling to 700 °C with a holding time of 2.5 h, and furnace cooled to room temperature.

Dilatometric experiments were carried out on a Formaster-F fully automatic transformation dilatometer. The process of cyclic heating and cooling is shown in Fig. 1. Four different preheating treatments were taken to evaluate their effects on repeated martensitic transformation. The preheating treatments were as follows:

(1) P1: austenitized at 1100 °C for 5 min, and then cooled to room temperature by nitrogen flushing;

(2) P2: As P1, and then reheated at 650 °C for 3 h, cooled to room temperature and reheated to 600 °C for 2 h, cooled to room temperature again;

(3) P3: As P1, and then tempered at 400 °C twice, each for 1 h;

(4) P4: As P1, and then tempered at 400 °C twice, each for 3 h.

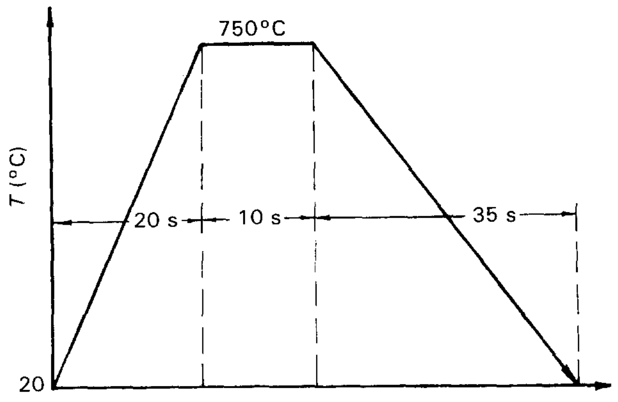

Figure 1 Schematic diagram of the repeated heating and cooling treatment.

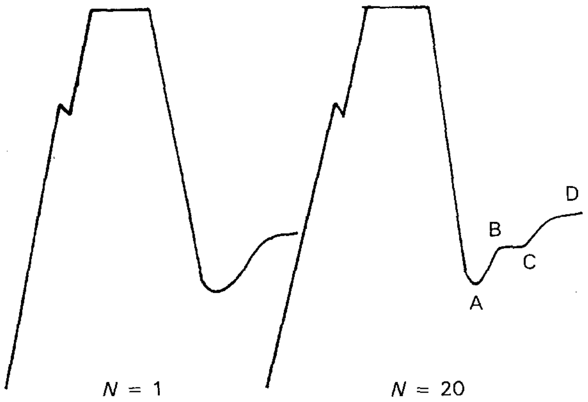

Figure 3 Schematic diagrams of the dilatometric change curve with $N = 1$ and $N = 20$, respectively.

Both the process of cyclic heating and cooling and the preheating treatments were carried out in Formaster-F dilatometer. The length change, time and temperature were automatically recorded. The length change of a specimen, ΔL, obtained in the dilatometric experiments can be related to the volume fraction of martensite, f_m [5]

$$\Delta L = 0.07214 f_m \qquad (1)$$

From Equation 1, the volume fraction of martensite under different cyclic numbers can be calculated.

3. Results and analysis

3.1. Characteristics of repeated martensitic transformation

The change in the volume fraction of martensite, f_m, with the thermal cyclic number, N, is shown in Fig. 2 for process P4. The result shows that in the initial stage of 750 °C → 20 °C repeated cycling, f_m increases with increasing cyclic number, N, then in the following stage, f_m decreases with increasing N, until finally, a stable value of f_m about 50%, is approached.

The most important experimental result was that as $N \geqslant 9$, the length change of the specimen exhibited a discontinuity during cooling, as shown in Fig. 3. When the temperature was below M_s the length increased owing to volume expansion in the austenite to martensite transformation, as shown in the AB stage. In the following stage, BC, however, the length of the specimen remains unchanged. Finally, with the temperature below a certain value, the length is obviously increased again, as shown in stage CD.

The critical temperature, A_{c1}, is relatively low (about 560 °C for the steel tested), because of the addition of nickel and manganese. There is no obvious change in microstructure except for tempering of martensite when tempered at 400 °C. Because the heating temperature, 750 °C, is above A_{c1} in the repeated heating and cooling cycle, martensite with 47 % volume fraction, obtained during quenching and tempering at 400 °C will transform into austenite, and about 100 % austenite is obtained at 750 °C. The temperatures related to the points A and C in the dilatometric line shown in Fig. 3 are M_s and M_c; they change with cyclic number, N. The results are given in Fig. 4. It can be seen that M_s is dramatically increased with increasing cyclic number, N, then in the following stage, a stable value of M_s is obtained. In contrast, M_c are decreased first and then remain unchanged with N.

The resistant force of martensitic transformation depends upon the yield strength of austenite [6]. The yield strength of austenite, in turn, is related to its crystal defects, such as dislocation, grain boundary, etc. It has already been shown that the introduction of

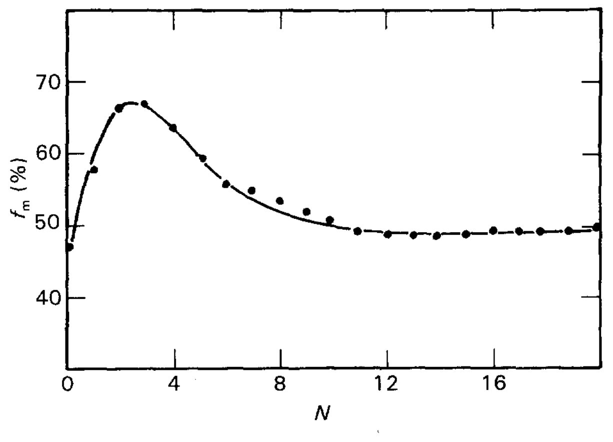

Figure 2 Change of martensite volume fraction, f_m, with cyclic number, N, in the sample treated by process P4.

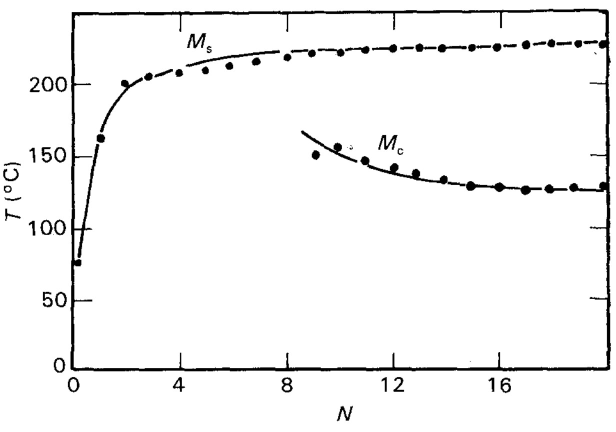

Figure 4 Changes of M_s and M_c with cyclic number.

dislocation, dispersion strengthening and the formation of a solute atmosphere in austenite will increase the yield strength of austenite and, therefore, decrease the M_s temperature and the volume fraction of martensite transformed. However, the tested steel containing a suitable level of vanadium and carbon is easily precipitated with finer V_4C_a carbide in the ranges from 600–800 °C [7]. Therefore, two different factors affecting martensitic transformation exist: on the one hand, the carbon content in the austenite will be decreased and the stability of austenite decreased by the precipitation of V_4C_a, and on the other hand, the yield strength of austenite will be enhanced by precipitation hardening of V_4C_3, and the resistant force of martensitic transformation will be increased. The V_4C_3, carbide is dynamically precipitated at 750 °C during 750 °C → 20 °C cycling and it is unchanged during cooling from 750 °C to 20 °C. Thus the amount of V_4C_a is increased with increasing cyclic number, N. Meanwhile the carbon content of the austenite matrix in the vicinity of V_4C_a particles is decreased, and the M_s temperature is dramatically increased in the initial stage of cycling, as shown in Fig. 4. However, on further increasing the cyclic number, the transformation from austenite to martensite becomes monotonic, as shown in Fig. 3, and the second accelerative transformation of martensite appears, which is represented by the M_c temperature. Because the increase in the amount of V_4C_a carbide with cyclic number enhances the resistance to martensitic transformation, the transformation of the untransformed region will be carried out at lower temperature. From the results shown in Figs 2 and 4, we can conclude that in the initial stage of the cycling, the main factor controlling the martensitic transformation is the decrease of carbon content in the austenite, which increases f_m and M_s whereas with increasing cyclic number, the main factor is the effect of V_4C_a precipitation hardening, which makes f_m decrease and M_c appear.

The changes of f_m with temperature below the M_s points for various cyclic numbers are given in Fig. 5 for process P4. When $N = 0$, i.e, before the cyclic heating and cooling in 750 °C → 20 °C is carried out, f_m is monotonic, increasing with decreasing temperature. However, during the cyclic heating and cooling, the curve representing the amount of martensite formed with decreasing temperature is not monotonic. The change of f_m with temperature exhibits three stages with different slopes. In the stage at which the cooling temperature is slightly lower than the M_s point, f_m is greatly increased with decreasing temperature, then, in the next stage, f_m increased slowly; finally, f_m dramatically increased in the last stage. Obviously, the kinetic behaviour of the martensitic transformation in repeated heating and cooling is different from that of the conventional martensitic transformation due to the dynamic precipitation of V_4C_a.

3.2. Effects of preheating treatment on repeated martensitic transformation

The samples exhibit various microstructures after preheat treatment as mentioned above. After process P1, the microstructures are mainly composed of austenite and martensite with a volume fraction 47%. Process P2 is a conventional quenching and tempering process for hot-worked die steel. The fine V_4C_a particles will precipitate in the matrix phase during tempering. Because of the V_4C_a precipitate, the carbon content in austenite is decreased and the M_s temperature is increased from 99 °C for the quenching condition to 196 °C for the tempering condition. The final microstructure is composed of austenite, precipitated V_4C_a and martensite with a volume fraction of 65%.

After treatments P3 and P4 procedures, the microstructures consist of austenite and tempered martensite with volume fractions of 47 % and 44 %, respectively, without the precipitation of V_4C_a.

From Fig. 6, it is apparent that the preheating treatments have an important effect on the tendency of f_m to change with cyclic number. If the pre-treatments,

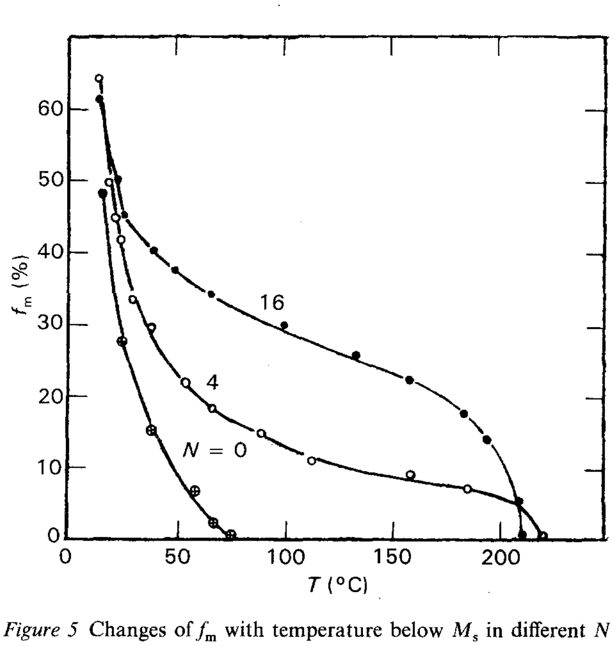

Figure 5 Changes of f_m with temperature below M_s in different N for the sample treated by process P4.

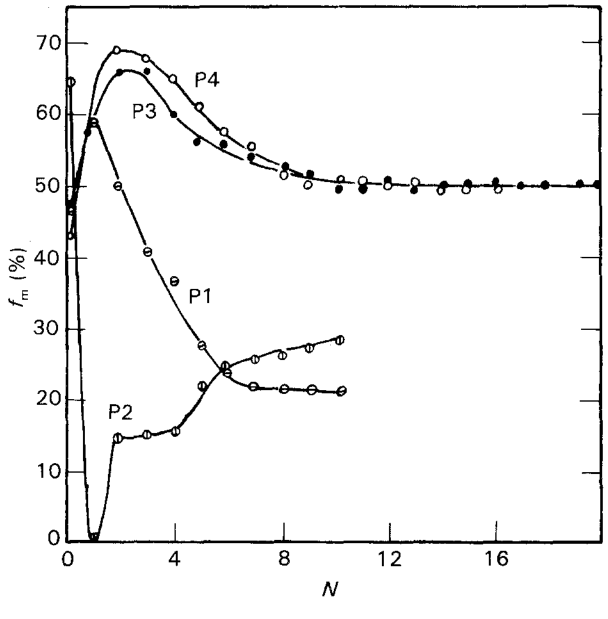

Figure 6 Changes of f_m with N for different preheat treatments.

such as P1, P3, and P4, do not cause the precipitation of V_4C_a, in the initial stage of repeated heating and cooling, f_m is increased with increasing cyclic number N, and then, after reaching its maximum, f_m tends to decrease and finally remain at a stable value. In contrast to the above tendency, the f_m in the sample treated by procedure P2 is first decreased and then increased with cyclic number, N.

The V_4C_a carbide has already precipitated in the sample treated by process P2 before the cycling continues. The great resistance of V_4C_a to martensitic transformation can be seen from the first cycling, in which there is no martensite formed. However, because the tempering temperature of P2 is lower than that of the repeated heating, i.e. 750 °C, the V_4C_a precipitated during process P2 will partly dissolve in austenite and its amount will be decreased, so that f_m will increase with increasing cyclic number.

In contrast to P1, tempering occurs at 400 °C in the P3 and P4 procedures. This tempering causes the supersaturated carbon content in austenite and martensite to decrease. Therefore, the amount of V_4C_a precipitated during cycling is decreased, and f_m is larger than that obtained by process P1.

The influence of the preheating treatment on the kinetics of martensitic transformation in a given cyclic number, i.e. $N = 4$, is shown in Fig. 7. It is apparent that the preheating treatments have a great effect on the kinetics of martensitic transformation.

3.3. Kinetic analysis

The common kinetic equation of martensitic transformation proposed by Koistinen and Marburger [8] is

$$f_m = 1 - \exp[-\alpha(M_s - T_q)] \qquad (2)$$

where T_q is the instantaneous temperature, α a constant which was found to be equal to $0.011\ K^{-1}$ for most steel [8], and M_s the martensitic transformation start temperature.

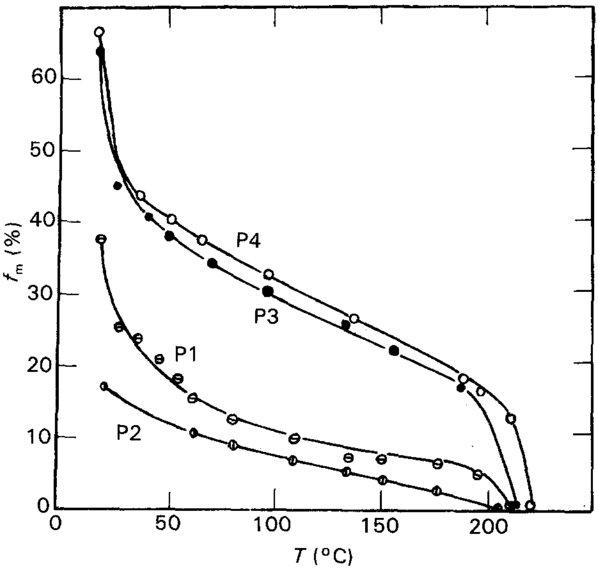

Figure 7 Changes of f_m of different preheat treatments with temperature in $N = 4$.

Typical examples of the common martensitic transformation and the transformation under repeated cycling are schematically illustrated in Fig. 8. The difference between the two is that the rate of the common transformation is contrary to that under repeated cycling. It is probable that because of the dynamical precipitation of V_4C_a carbide, the rate of martensitic transformation is lowered, and once the driving force of transformation is sufficiently high, which can be obtained by lowering the temperature, the rate of transformation will be enhanced again.

Equation 2 can be rewritten

$$\ln(1 - f_m) = \alpha(M_s - T_q) \qquad (3)$$

This equation indicates that the term $\ln(1 - f_m)$ will linearly change with $(M_m - T_q)$ for common martensitic transformation. However, the change of $\ln(1 - f_m)$ with $(M_s - T_q)$ for the repeated cycling, for example, $N = 4$, is divided into two parts with different slopes as shown in Fig. 9. For Parts I and II of process P4, the changes of $\ln(1 - f_m)$ with $(M_s - T_q)$ can be described, respectively, as follows

$$\text{Part I} \quad \ln(1 - f_m) = -0.1511 - 0.002(M_s - T_q) \qquad (4)$$

$$\text{Part II} \quad \ln(1 - f_m) = 2.585 - 0.018(M_s - T_q) \qquad (5)$$

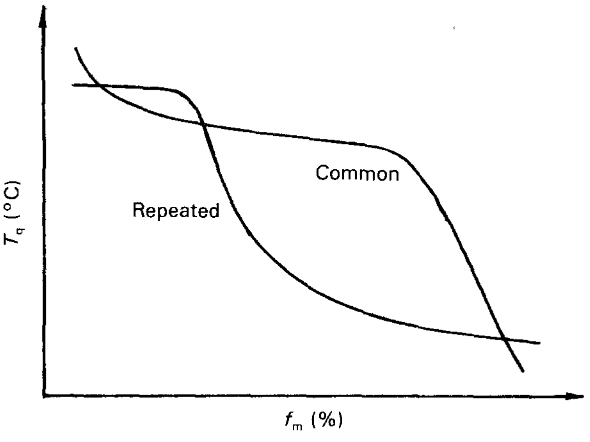

Figure 8 Schematic diagram of the change of f_m with temperature for conventional and repeated martensitic transformation.

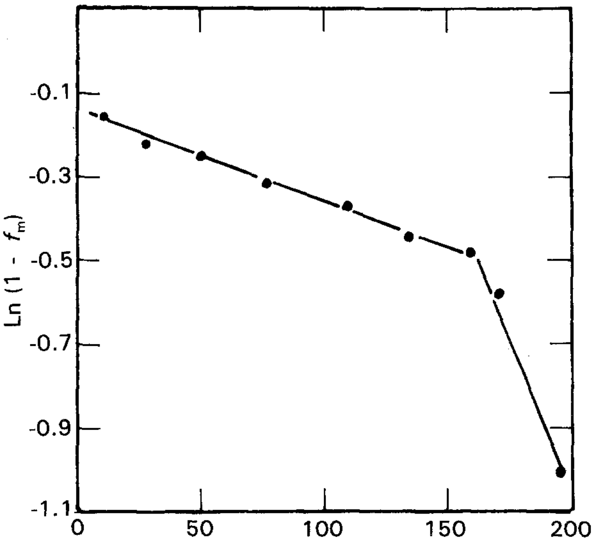

Figure 9 Change of $\ln(1 - f_m)$ with $(M_s - T_q)$.

The linear correlation factors for Equations 3 and 4 are 0.9988 and 0.9422, respectively.

4. Conclusions

1. The volume fraction of martensite, f_m, formed in 750 °C → 20 °C repeated cycling changes with the cyclic number. In the curve shown, of the change of f_m with temperature, there are critical points at which f_m begins dramatically to increase, that is, M_s and M_c. With increasing cyclic number, M_s is increased and M_c decreased.

2. The preheating treatment has an important influence on the kinetics of martensitic transformation in repeated cycling. If the pre-treatments do not cause the precipitation of V_4C_a carbide, during the initial stage of the repeated cycling f_m is increased with increasing cyclic number, N, and then after reaching its maximum, f_m tends to decrease. If the sample already contains precipitates from the pre-treatment, there the opposite tendency to change exists.

Acknowledgement

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