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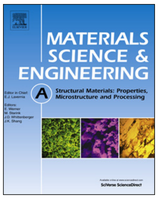

Effects of quenching and tempering on the microstructure and bake hardening behavior of ferrite and dual phase steels

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ABSTRACT

The effects of quenching and tempering on the microstructure evolution and bake hardening (BH) behavior of both ferrite and dual phase steels were investigated. The C–Mn steels were heated to the soaking temperature, quenched in water and then tempered in the 100–500 °C range. After pre-straining, the baking treatment (180 °C for 20 min) was carried out to measure the BH values. It was found that increased quenching temperature reduced the BH value. Furthermore, the BH value turned to be negative when the quenching temperature exceeded 670 °C and 710 °C for the steels annealed at 800 °C and 900 °C, respectively. The ferrite aging and the martensite tempering played key roles in the bake hardening behavior during the tempering process. In the present study, three stages were identified during tempering of the above steels: (1) the relief of residual stresses in the ferrite; (2) the precipitation of carbides in both ferrite and martensite; (3) the dissolution of carbides in the ferrite. The BH values of both ferrite and dual phase steels had a complex variation with the tempering temperature. After an apparent increment, the BH value reduced, and then was slightly enhanced when the tempering temperature increased from 25 °C to 500 °C.

1. Introduction

Recently, there has been a growing interest in the dual phase (DP) steel due to the combination of continuous yielding, low yield strength, high tensile strength, good ductility and high bake hardening value [1–3]. The DP steel normally produced by quenching and tempering is used in the automotive industries extensively because of the low cost and high strength [4]. The quenching of the DP steel results not only in the formation of martensite, but also in the high residual stresses. Consequently, the quenched DP steel is generally reheated to suitable temperature to improve the mechanical properties. Three stages are reported in the DP steel during tempering [5]: (1) below 225 °C, formation of Cottrell atmosphere around the mobile dislocations and relief of residual stresses in the ferrite, (2) 225–535 °C, precipitation of ε-carbides in both ferrite and martensite and (3) above 535 °C, recovery of martensite and formation of cementite. It has been reported by many authors that the yield strength and tensile strength decrease, while the elongation and yield point elongation increase with increasing the tempering temperature [6–8]. However, the microstructure control by quenching and tempering to enhance the bake hardening behavior has not been addressed in detail.

The bake hardening treatment has been extensively used for the automotive steel sheets to enhance the yield strength after pre-straining. The mechanism of bake hardening is an interaction process of the mobile dislocations and interstitial carbon atoms in the ferrite grain interior [9,10]. Consequently, the bake hardening behavior mainly depends on the mobile dislocation density and the amount of solute carbon atoms in the low carbon and ultra low carbon steels. It is well known that the microstructure of the DP steel consists of a soft ferrite matrix and hard martensite islands [8,9]. Thus, the microstructure evolution of the DP steel during paint baking is different from low carbon and ultra low carbon steel. In the DP steel, there are three stages during baking [11]: (1) formation of Cottrell atmosphere in the ferrite, (2) carbon clusters or carbides precipitation in the ferrite and (3) low temperature tempering of the martensite phase, including martensite decomposition and the relief of residual stresses.

The present work is focused on the static strain aging phenomenon in both ferrite and DP steels. The specific aims of this work are (1) to analyze the effects of quenching and tempering on the BH value and (2) to provide the fundamental understanding of the influence of the solute carbon atoms on the bake hardening behavior.

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2. Experimental procedure

Cold rolled low carbon steel with chemical composition (in wt%) of 0.083C, 0.033Si, 0.322Mn, 0.021 P, 0.006S was used in this research. After reheating to 1200 °C for 1 h and hot rolling to 3 mm, the steels were coiled at 720 °C, and then cold rolled to 0.4 mm thickness.

The steels were heated to soaking temperature (Ts: 700–900 °C) for 90 s (ts), cooled to quenching temperature (Tq: 600–800 °C) in air, and then quenched to room temperature in cold water. After quenching, the specimens were tempered in the range of 100–500 °C for 180 s and subsequently cooled in air. The steels were temper rolled with a 0.5% reduction ratio on a cold rolling mill after quenching and tempering cycles.

Optical and SEM microscopic observations were carried out after etching in 4% nital solution. Tensile samples were cut from the quenched and tempered sheets according to JIS 5# with 50 mm gauge length. Tensile tests were carried out in an Instron tensile machine at a constant cross-head speed of 2 mm min^{-1}. The martensite volume fraction was calculated by the point counting method. At least three samples were tested for every condition and the average value was calculated. To study the influences of quenching and tempering on the bake hardening behavior, the specimens were pre-strained to 2%, and then baked at 180 °C for 20 min. According to Fig. 1, the BH value is defined as the difference between the flow stress at the end of prestraining and the lower yield stress after aging treatment [9,12].

3. Results and discussion

3.1. Influence of quenching on the bake hardening behavior

Fig. 2 shows the tendency of the BH value with the quenching temperature. As seen in Fig. 2, with an increase in the quenching temperature, the BH value decreases for the steels without tempering. For example, the BH values are 46 MPa and 52 MPa for the steels annealed at 800 °C and 900 °C, respectively, at the quenching temperature of 600 °C. The BH values decrease to −30 MPa and −23 MPa when the quenching temperature reaches 800 °C. The positive BH values remain until quenching up to about 670 °C and 710 °C for the steels annealed at 800 °C and 900 °C, respectively.

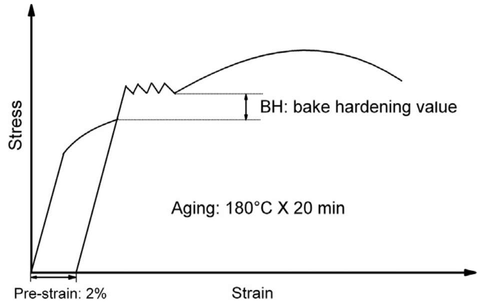

Fig. 1. Definition and measuring method of the BH value.

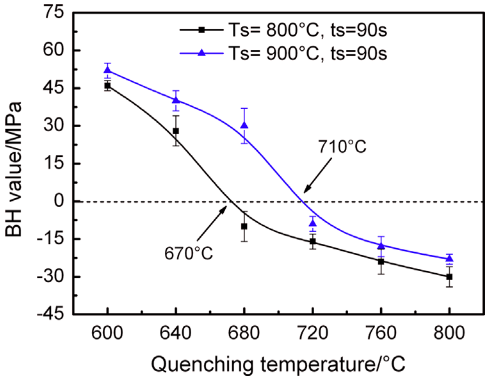

Fig. 2. Effect of quenching temperature on the BH value.

In the DP steel, the microstructure evolution during baking includes three stages [11]. Therefore, the BH value can be calculated by the following equation:

$$BH = \Delta\sigma_{\text{Cottrell}} + \Delta\sigma_{\text{precipitation}} + \Delta\sigma_{\text{tempering}} \quad (1)$$

where $\Delta\sigma_{\text{Cottrell}}$ is the hardening caused by the formation of Cottrell atmosphere; $\Delta\sigma_{\text{precipitation}}$ is the hardening because of the precipitation of carbides in the ferrite; $\Delta\sigma_{\text{tempering}}$ is the variation due to the tempering of martensite phase including martensite decomposition and relief of residual stresses. Usually, $\Delta\sigma_{\text{Cottrell}}$ and $\Delta\sigma_{\text{precipitation}}$ are positive, while $\Delta\sigma_{\text{tempering}}$ is negative. In addition, the residual stresses include thermal stresses due to the temperature variation and structural stress because of the martensite formation [13]. The residual stresses have a pronounced influence on the mechanical properties of the samples, even leading to distortion [14]. The residual stresses mainly depend on various parameters, including quenching temperature, martensite volume fraction, cooling rate and thermal conductivity [13].

Fig. 3 shows the elongation variation before and after baking treatment. The elongation rises after pre-straining and aging treatment when the quenching temperature is increased to about 670 °C and 710 °C for the steels annealed at 800 °C and 900 °C, respectively. This can be mainly attributed to the relief of residual stresses during baking. The formation of a small amount of martensite leads to lower residual stresses when the steels are quenched at lower temperature. The summation of $\Delta\sigma_{\text{Cottrell}}$ and $\Delta\sigma_{\text{precipitation}}$ is higher than $\Delta\sigma_{\text{tempering}}$. Thus, the BH value is greater than zero and the elongation decreases after aging treatment. It is well known that the residual stresses are enhanced with increasing the quenching temperature and martensite volume fraction [13]. The tendency shown in Fig. 4 is a significant enhancement in the martensite volume fraction when the quenching temperature increases. This can be attributed to the higher volume fraction of the untransformed austenite. A large number of martensites start to form at the quenching temperatures of 670 °C and 710 °C, resulting in higher residual stresses during quenching. After the relief of residual stresses in the ferrite during baking [6,8], the summation of $\Delta\sigma_{\text{Cottrell}}$ and $\Delta\sigma_{\text{precipitation}}$ is lower than $\Delta\sigma_{\text{tempering}}$. Consequently, the BH value becomes negative. This is in agreement with the result obtained by Gunduz and Zhang [9,15]. In brief, with an increase in the quenching temperature, there is a decrease in the BH values due to the relief of residual stresses introduced during cooling.

Another important reason responsible for the reduction in the BH value in the case of the high quenching temperature can be the high dislocations density in the ferrite. The mobile dislocations cannot be pinned completely by the interstitial atoms because of the limited solute carbon atoms and baking time. Consequently, higher quenching temperature results in a significant amount of martensite and unpinned dislocations [16]. The low yield strength of the DP steel is attributed to the mobile dislocations in the ferrite [2]. Thus, increasing the amount of unpinned dislocations leads to lower yield strength after baking treatment. By increasing the quenching temperature the BH value decreases because the free dislocations are pinned by the solute carbon atoms incompletely.

As clearly shown in Fig. 2, the BH value of the steel annealed at 900 °C is higher than that of the steel annealed at 800 °C. Generally speaking, when the annealing temperature exceeds A_{c1}

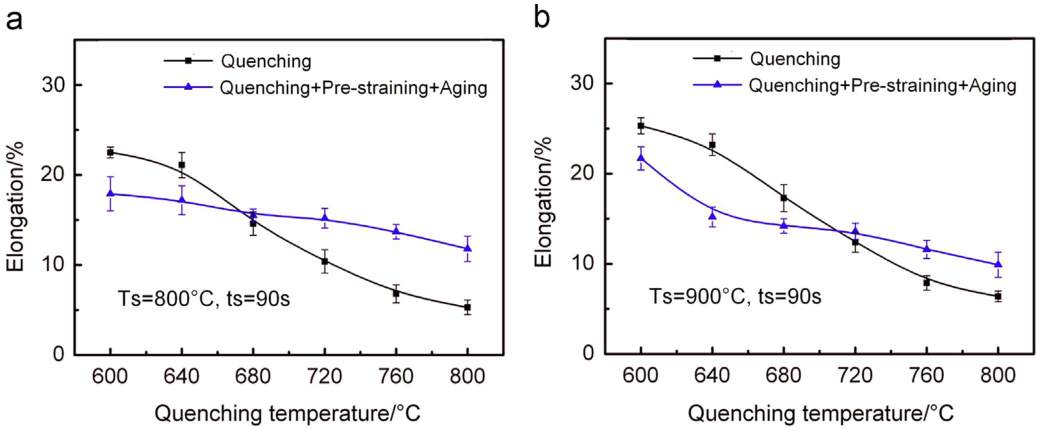

Fig. 3. Effect of pre-straining and baking treatment on the elongation: (a) Ts=800 °C and (b) Ts=900 °C.

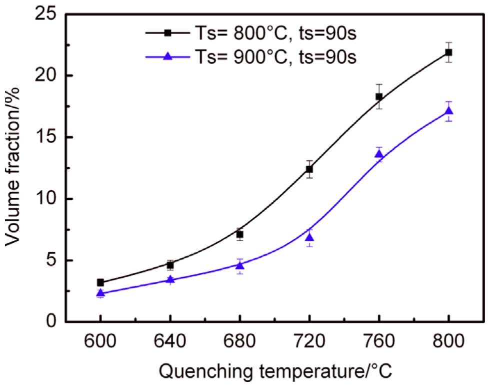

Fig. 4. Effect of quenching temperature on the martensite volume fraction.

the carbides gradually dissolve during austenitizing, increasing the amount of the solute carbon atoms in the matrix. The dissolution of the solute carbon atoms in the ferrite and austenite was investigated by Yong [17], and the expression of the solute carbon concentration was derived

$$\lg w([C]_\alpha) = 2.38 - 4040/T \quad (2)$$

$$\lg w([C]_\gamma) = 1.36 - 1480/T \quad (3)$$

where $w([C]_\alpha)$ is the mass fraction of solute carbon in the ferrite; $w([C]_\gamma)$ is the mass fraction of solute carbon in the austenite and T is the annealing temperature. According to Eqs. (2) and (3), the mass fractions of solute carbon in the ferrite and austenite are 0.04% and 0.96%, respectively, at the annealing temperature of 800 °C. The mass fraction of solute carbon in the austenite is 1.26% at the annealing temperature of 900 °C (above A_{c3}). With increasing the annealing temperature from 800 °C to 900 °C, the mass fraction of the solute carbon atoms in the matrix is obviously enhanced. Therefore, the carbide particles dissolve completely in the matrix at 900 °C (Fig. 5(b)), while a large number of undissolved carbides are retained in the ferrite at 800 °C (Fig. 5(a)). Therefore, higher annealing temperature leads to greater solute carbon atoms and thus to higher BH values.

In addition, the grain size plays an important role in the bake hardening behavior [12]. Increasing the annealing temperature from 800 °C to 900 °C favors an increase in the grain size. The grain boundaries provide segregation sites for the interstitial atoms. Thus, diffusion of the solute carbon atoms to grain boundaries during soaking results in lower solute carbon concentration in the grain interior. With the grain size increase, the amount of the solute carbon atoms segregated to grain boundaries decreases. A similar result has been reported by Alshalfan [18]. Furthermore, the diffusion distance of carbon atoms is limited due to the slow diffusion rate and short soaking time. Therefore, the finer grain leads to shorter diffusion distance between the interstitial atoms and grain boundaries and thus to greater segregation carbon atoms. In conclusion, the amount of segregation atoms decreases with increasing the grain size, resulting in greater solute carbon atoms in the grain interior and higher BH value.

3.2. *Influence of tempering on the bake hardening behavior*

In order to understand the effect of microstructure evolution on the carbon content and bake hardening behavior during tempering, the steels were heated to 700 °C and 780 °C for 90 s and then quenched in cold water. The microstructure of both ferrite and DP steels, developed by annealing at different heating temperatures, is shown in Fig. 6. In Fig. 6(a), the microstructure displays the fine ferrite and a small amount of carbides in the ferrite steel. In addition, the carbides are aligned along the rolling direction. It is observed that the microstructure of the DP steel annealed in the intercritical region mainly consists of the martensite phase dispersed in the ferrite matrix (Fig. 6(b)). The volume fraction of martensite islands distributed randomly in the ferrite matrix is about 20.6%.

Fig. 7 shows the influence of the tempering temperature on the BH values of both ferrite and DP steels. As seen in Fig. 7, the effect of the tempering temperature on the BH value can be divided into three stages. Below 300 °C, the BH value of the ferrite steel quenched at 700 °C is enhanced significantly with the increasing tempering temperature. The maximum BH value is 22 MPa at the tempering temperature of 300 °C. However, with an increase of the tempering temperature from 300 °C to 400 °C, the BH value makes an apparent reduction, and then improves again when the tempering temperature is increased to 500 °C. In the DP steel, the varying tendency of the BH value is similar to ferrite steel. The BH value reaches the maximum value (57 MPa) at the tempering temperature of 200 °C. In addition, the BH value of the steels quenched at 780 °C is higher than that of the steels quenched at 700 °C. This is thought to be due to the increment in the carbon content and martensitie volume fraction [2,11].

According to the previous discussion, in the as-quenched ferrite steel, the BH value mainly consists of three parts. Between 25 °C and 300 °C, the tempering treatment has no apparent influence on $\Delta\sigma_{\mathrm{Cottrell}}$ and $\Delta\sigma_{\mathrm{precipitation}}$, because the number of mobile dislocations and the amount of solute carbon concentration keep almost constant during the short tempering stage. However, higher tempering temperature results in the relief of residual stresses during tempering [6,8]. After the relief of residual stresses before

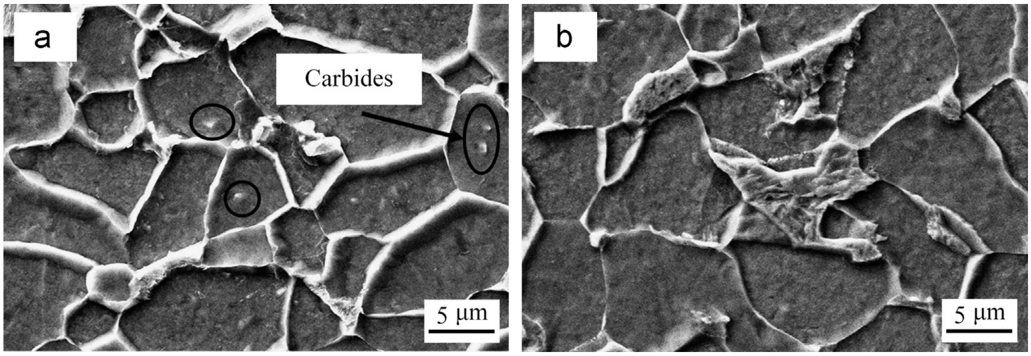

Fig. 5. SEM micrographs of the specimens annealed at (a) 800 °C and (b) 900 °C.

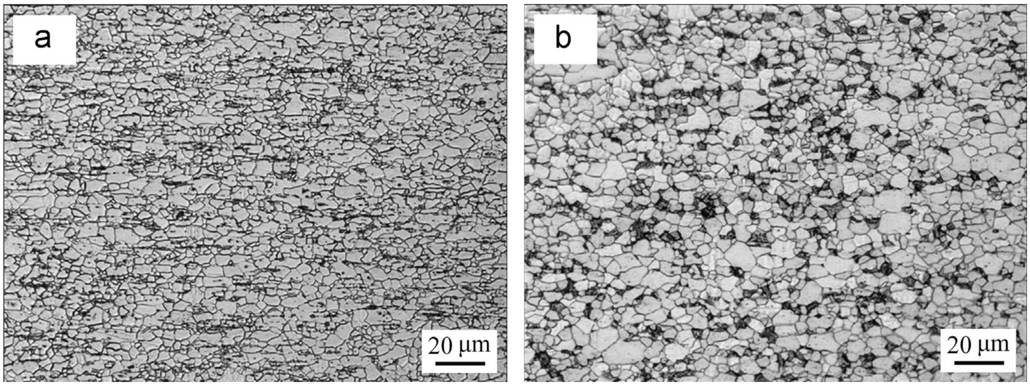

Fig. 6. Microstructure of (a) ferrite steel annealed at 700 °C and (b) DP steel annealed at 780 °C.

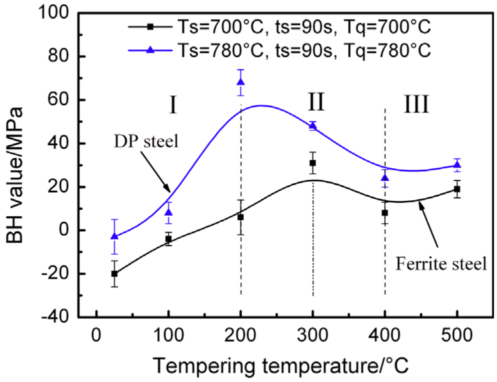

Fig. 7. Effect of tempering temperature on the BH value.

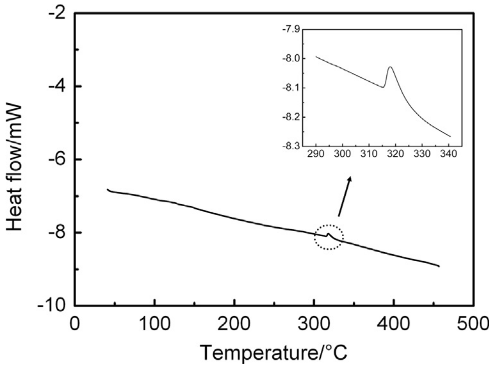

Fig. 8. Differential scanning calorimetry (DSC) curve of the steel quenched at 700 °C.

baking, $\Delta\sigma_{\text{tempering}}$ becomes smaller and smaller, and can be even neglected. Consequently, with increasing the tempering temperature in the 25–300 °C range, the BH value of the ferrite steel annealed at 700 °C obviously increases.

Fig. 8 shows the heat flow variation of ferrite steel during heating. It is clear that an exothermic peak occurs between 310 °C and 330 °C. In other words, a large number of carbides precipitate during tempering when the tempering temperature is increased to about 320 °C. A similar tempering temperature (328 °C) for the carbides' precipitation in the ferrite has also been reported by Chang in an earlier work [5]. Thus, the amount of carbon in solid solution reduces due to the precipitation of carbides. According to the Cottrell model [19], the bake hardening behavior depends on the amount of the solute carbon atoms and mobile dislocations density. Therefore, the BH value decreases due to the reduction in the carbon content in the matrix when the tempering temperature increases to 400 °C.

As seen in Fig. 7, increasing the tempering temperature from 400 °C to 500 °C leads to higher BH value. Meanwhile, the yield strength increases slightly while the tensile strength stays almost constant in the present study. This interesting phenomenon can be attributed to the pinning of mobile dislocations by Cottrell atmosphere [5]. Merlin [20] suggested that higher tempering temperature favored an increase in the mass fraction of solute carbon in the ferrite. After tempering at 500 °C for 180 s, the specimens are cooled rapidly in air, resulting in more solute carbon atoms in the matrix [5]. Consequently, the bake hardening behavior is enhanced again at the tempering temperature of 500 °C.

The influence of the tempering temperature on the microstructure evolution of the DP steel is shown in Fig. 9. As seen from Fig. 9(a), the microstructure of the quenched DP steel consists of a ferrite matrix and pure martensite islands. Meanwhile, the carbides cannot be observed in the martensite islands. As seen in Fig. 9(b), the morphology of the DP steel tempered at 100 °C or 200 °C is not significantly

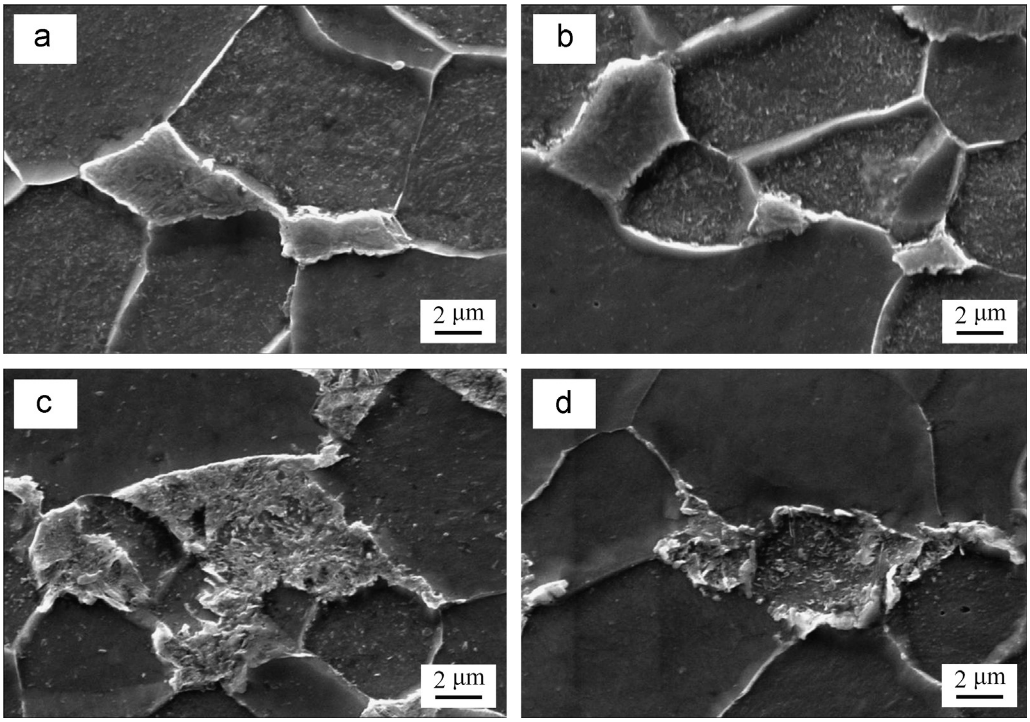

Fig. 9. SEM micrographs of the samples quenched at 780 °C and tempered at (a) 25 °C; (b) 200 °C; (c) 400 °C and (d) 500 °C.

different from the as-quenched one. This is consistent with the results obtained by Fang [6], Gunduz [7] and Sayed [8]. However, the residual stresses and free dislocations density surrounding the martensite islands are reduced during tempering [6,8]. Therefore, the BH value rises at the tempering temperature ranging from 25 °C to 200 °C.

When the tempering temperature is increased to about 300 °C, the surfaces of martensite islands are smoothened. This result can be explained by the fact that the martensite phase starts transforming into carbides and ferrite [7]. Kamp [21] pointed out that the carbides precipitated in the martensite when the DP steel was tempered at 300 °C for 2 s. This is in agreement with the result obtained in the present study. Therefore, the carbon concentration in the tempered martensite decreases due to the carbides' precipitation [8,21].

In addition, the cementite particles in the martensite coarsen with increasing the tempering temperature from 300 °C to 400 °C. The carbon concentration in the martensite further reduces due to the diffusion of carbon atoms to the cementite [7,8,21]. Furthermore, according to the previous discussion, the carbides begin to precipitate in the ferrite grain interior when the tempering temperature is increased to about 320 °C. The ferrite is covered with a large number of white carbides particles (Fig. 9(c)) when the tempering temperature reaches 400 °C. This could eventually lead to lower solute carbon concentration in the ferrite. In summary, the carbon content in the matrix decreases obviously due to the precipitation of carbides in both ferrite and martensite [7,8,21]. Consequently, with an increase in the tempering temperature from 200 °C to 400 °C, there is a significant decrease in the BH value.

As clearly seen in Fig. 9(d), when the tempering temperature reaches 500 °C the martensite decomposes into cementite and ferrite totally. Moreover, the cementite particles in the ferrite dissolve or become finer. Similar to ferrite steel, increasing the tempering temperature from 400 °C to 500 °C results in greater solute carbon atoms due to the dissolution of carbides in the ferrite. Therefore, the BH value increases at the tempering temperature of 500 °C.

4. Conclusions

(1) The residual stresses, carbon content and free dislocations density play an important role in the bake hardening behavior in the DP steel. Increasing the annealing temperature results in higher BH values due to the dissolution of carbides.

(2) With an increase in the quenching temperature, the BH value decreases due to the relief of residual stresses during baking. The BH value of the steels annealed at 800 °C and 900 °C turns to be negative when the quenching temperature increases to 670 °C and 710 °C, respectively.

(3) The microstructure evolution of both ferrite and DP steels during tempering clearly consists of the three stages: the relief of residual stresses, the precipitation of carbides in both ferrite and martensite and the dissolution of carbides in the ferrite.

(4) In both ferrite and DP steels, the BH value has the same tendency with the tempering temperature. After a significant increment, the BH value apparently decreases, and then is enhanced slightly with increasing the tempering temperature in the 25–500 °C range.

Acknowledgments

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