The Influential Factors of Baking Time in Internal Friction and Bake-Hardening Properties on Low Carbon Steel

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Abstract. This paper aims at exploring the distinctive ways of baking time affecting the hardening degree of the cold-rolled steel. Therefore, with different time span, the annealed sample was baked to test its baking hardening properties. The sample was analyzed microstructurally after being annealed, pre-deformed and baked. The internal friction was simultaneously measured in terms of different processing conditions during experiments. The following findings are the significant conclusion of the experiments: The important experiment result indicated that when the degree of pre-deformation setting at 5% and the baking temperature at 170℃, the BH value increased but remained unchanged afterwards. Furthermore, when the baking time span was set at 1000 minutes, the BH value reached the maximum but would remain unchanged afterwards. When the degree of pre-deformation was set at 5%, and the baking temperature was set at 170℃ with baking time 20 minutes, the SKK peak reached the maximum. When the baking time was 1000 minutes and constantly extended, the height of SKK peak rarely changed. The above experiments are designed to better illustrate and understand the significance of the hardening steel properties for future application in the automobile industry.

1. Introduction: Aims and Objectives

Due to its processability required by the automobile production industry, bake hardening steel (BH steel) is currently one of the most innovative and widely adopted automobile steel. Lightness of the steel properties has its cutting-edge advantages as it can largely reduce the weight of automobile, and more importantly, its strong concave resistance. This being so, BH steel is most applicable for producing automobile exterior panel of a car body, and is thus one of the most advanced steel types. The research of bake hardening value is a hot topic now as it is crucial for developing automobile steel. It is widely regarded as the interaction of the interstitial atom and dislocation from the stamping process, i.e., the formation of the Cottrell Atmosphere which is closely related to the degree of pre-deformation and the content of dissolved carbon. The experiment results of the BH value affected by the pre-deformation and baking time differ, and at times, differ significantly. A.K.De et al.[1,2] suggested that there would be 1%-10% pre-deformation and it was irrelevant to its BH value or its pre-deformation. Jiao[3], Li[4] and Guan[5] conducted similar researches that the BH value would increase and then decrease aligning the uprise of pre-deformation. Other researches indicated that the BH value decreased in accordance with the increase of pre-deformation[6,7]. Due to its strong applicability, intriguing nature and controversial suggestions by different researchers. At present, in relation to the variations of BH value with different baking time, only a limited number of researchers have considered such possibilities and
thus investigations in this field are scarce. So this paper aims at experimenting the low-carbon steel affected by the baking time via internal friction. The main objective is to investigate its affect on the hardening properties of steel. The mechanism of the BH value will be illustrated micro-structurally.

2. Experimental materials and methods
Low carbon bake hardening steel in annealing was maintained as the main experimental material. Table 1 listed its chemical composition as follows:

|   | C   | Si  | Mn  | P   | S   | Al  | N   |
|---|-----|-----|-----|-----|-----|-----|-----|
|   | 0.21 | 0.01 | 0.21 | 0.011 | 0.007 | 0.019 | 0.002 |

The gauge length of 20mm as standard tensile specimen was used to be tested for the experimental material. The following procedure was utilizing UTM5305 to perform various pre-deformation on uniaxial tension. The adjustment of the degree of pre-deformation was: 5%, with 5mm/min being the tensile speed. 101-1 oven was the baking equipment, and the baking temperature was set at 170℃×10, 20, 100, 500, 1000, 2500, 5000, 7000, 10000 min.

The BH value was tested in accordance with GB/T24174-2009, and was analyzed by the means value of the 5 sample experiments. The average value of these 5 samples was obtained and observed. The micro-structure of the samples was observed through ZEISS optical microscope, and the dislocation morphology by JEM-2100 TEM. The internal friction measurement was conducted by MFP−1000 multifunctional internal friction instrument. The size of internal friction sample was: 50mm×1mm×1mm. The heating rate was set at 3℃/min. The room temperature was set at 700℃. The strain amplitude was 20×10−6. The frequency testing was: f=1. A corresponding fitting software was adopted to reflect and reveal the nature of internal friction.

3. Discussion of experiment results

3.1. Micro-structure
Fig. 1 demonstrated the size of average grain was 160μm subsequent to annealing, as 850℃×30min was used in annealing the experimental steel. Carbon offset[8] occurred with the grain boundary due to the size of crystalline grain being larger than 16μm.

Fig.1 Micro-structure of experimental steel after annealing

3.2. The effect of baking time on internal friction
In Figure 2 the pre-deformation was at 5% with baking degree at 170℃. The BH value and time was in the curve relationship of: 10, 20, 100, 500, 1000, 2500, 5000, 7000, 10000 min.
From Figure 2 it clearly showed that with the extension of the baking time, the BH value initially increased. It meant from 10 - 1000min the BH value increased from -16.4MPa to -11.5MPa. After that the BH value almost remained unchanged. It meant from 1000 - 10000min, the BH value stayed at -11.5MPa. The BH value increased utmost in the 10 - 20min.

Figure 3 showed the experiment results of different timing frequency f=1 deducting the internal friction, Its baking time was 20 min with internal friction curve. The uppermost curve was the internal friction curve after measuring. The middle one was the background internal friction curve. The lowest was the real internal friction curve.

In order to research further on the relationship of BH value with the baking time, the relevant data was listed in Table 2. And with different comparison of the BH value with the SKK peak under different times, it was listed in Figure 4. Figure 4 clearly showed that the Skk peak value changed with the time with the trends of increase first and then remained unchanged. But while at 20min an exception occurred. Table 2 demonstrated that with long enough baking time, the Cottrell Atmosphere always existed with the clusters formed by the interaction of carbon atoms with dislocations. the baking time from 10-10000min, SKK peak reached the maximum at 20min. It was at 3.644×10⁻³. In 10-20min the SKK peak also increased significantly, i.e., from 0.489×10⁻³ to 3.644×10⁻³. The increase was about 9 folds.

From Figure 4 it could be seen that the baking time was 20min, the BH value was not at maximum. Zhang[9] suggested that the affect of baking process on BH value had two sides: one caused the formation of the Cottrell Atmosphere and increased its flow stress. On the other hand, while the baking released internal stress release would thus decrease flow stress. Therefore, while the baking time was 20min, baking would increase the density of Cottrell Atmosphere of which it would be slightly bigger.
than the degree of internal stress release. As a result, the BH value increased with a limited degree. So in 20min the BH value could not reach its maximum. And in 20-100min the SKK peak decreased significantly from $3.644 \times 10^{-3}$ to $0.558 \times 10^{-3}$. The decrease was approximately 6.5 folds. In 100min the BH value was still larger than that of the 20min.

Fig. 4  The comparative curve of SKK peak and BH value during baking time

This was because at 100min, baking increased the density of Cottrell Atmosphere and the increase range was much higher than that of the internal stress release. This being so, the BH value increased more. It could be seen that while the baking time was at 20min, baking released the most internal stress. From 1000-10000min, the SKK peak value did not show significant variation. It remained between $0.6-0.7 \times 10^{-3}$. Figure 4 showed that in this period of time, the changes of BH value and SKK peak value were in accordance with each other. It was demonstrated that the interaction between carbon atoms and dislocation was at its balance. The density of the Cottrell Atmosphere formed by baking and the difference between the density of atmosphere and the release of internal stress stabilize.

| Baking time/min | $T_{\text{Snoek}}/K$ | $Q_{\text{Snoek}}^{-1} \times 10^{-3}$ | $T_{\text{SKK}}/K$ | $Q_{\text{SKK}}^{-1} \times 10^{-3}$ | $(T_{\text{Snoek}}+h_{\text{SKK}})$ /K |
|----------------|---------------------|-------------------------------|---------------------|-------------------------------|-----------------------------------|
| 10             | 325                 | 0.760                         | 509                 | 0.489                         | 1.249                             |
| 20             | -                   | -                             | 483                 | 3.644                         | 3.644                             |
| 100            | -                   | -                             | 513                 | 0.558                         | 0.558                             |
| 500            | 325                 | 0.338                         | 504                 | 0.592                         | 0.920                             |
| 1000           | 336                 | 0.590                         | 505                 | 0.702                         | 1.292                             |
| 2000           | -                   | -                             | 503                 | 0.722                         | 0.722                             |
| 5000           | -                   | -                             | 502                 | 0.695                         | 0.695                             |
| 7000           | -                   | -                             | 501                 | 0.554                         | 0.554                             |
| 10000          | -                   | -                             | 504                 | 0.627                         | 0.627                             |

4. Conclusions
One significant result from the experiment showed and proved that the BH value would change with the increase of the baking time. It first increased but then remained unchanged. While the baking time was at 1000min the BH reached its maximum. With the continuous increase of the baking time, the BH value did not vary much. While the pre-deformation was at 5% and baking temperature 170℃ with baking time of 20min, the SKK peak reached its maximum. When the baking time was at 1000min with the continuous increase of the baking time, the SKK value peak did not change much.
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