

# Effects of Annealing Conditions on Bake Hardenability for ULC Steels

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The influences of annealing conditions on bake hardening properties (BH) in the continuous annealing route were studied in this paper with Ti+Nb bearing ultra-low carbon bake hardening (ULC-BH) steels. The effects of the cold rolling reduction rate, soaking temperatures, slow cooling conditions and rapid cooling conditions were evaluated to clarify the complex metallurgical interactions. The soaking temperature has a significant effect in increasing the solute carbon content and enhances the BH property. Slow cooling was found to slightly promote the BH values with higher cooling rates as the re-precipitation was prevented and the supersaturated solute carbon could remain in the solid solution. The effects of rapid cooling starting temperatures were also investigated in this study but no obvious tendency was observed. The corresponding grain sizes were calculated and analyzed to help evaluate the influence of cold-rolled reduction and grain boundaries. It was concluded that a greater BH was achieved by larger grain size with less cold-rolled reduction.

**Keywords:** Annealing, Bake-hardening, Solute carbon

## 1. INTRODUCTION

Automobile bodies are highly expected to be both stronger and lighter due to more and more attentions are paid to global environmental concerns and safety requirements. Many lightweight materials, such as aluminum, magnesium, and plastics, have been suggested and successfully used in some novel components in new types of automobiles. However, there are still several concerns like cost, formability, reliability and recyclability that limit these applications in automobile evolution. One optimum combination can be achieved in the outer body parts of automobiles with ultra-low carbon bake hardening (ULC-BH) steels as they initially provide lower yield strength and excellent formability<sup>(1-3)</sup> in delivery condition and show a remarkable increase of yield strength during paint baking which results in a high dent resistance of the painted sheet.

The mechanism of bake-hardening is a kind of strain aging<sup>(4)</sup> resulting from the segregation of interstitial solute carbon and/or nitrogen atoms to the mobile dislocations generated by press-forming. A discontinuous yielding can be observed in stress-strain curve due to the interstitial solute atoms, carbon and nitrogen, were thermally activated then migrated to form Cottrell atmospheres to pin mobile dislocations<sup>(2,5,6)</sup> during 170°C/20min paint baking treatment. Usually bake-

hardening steels are designed to have a range approximately between 15 to 20 wt ppm of carbon in solid solution in the ferrite to obtain a minimum BH value of 30 MPa<sup>(2)</sup>.

In order to obtain the proper amount of solute carbon for the bake-hardening effect, interstitial atoms are fully or partially stabilized by NbC and TiC<sup>(7-8)</sup> and the related dissolution and/or precipitation are also utilized during the subsequent annealing and cooling process in Ti+Nb bearing ULC-BH steels. In the full stabilization cases, the solute carbon atoms come from NbC dissolution during the subsequent high temperature annealing process and are retained in the coming rapid cooling process. The solute carbon atoms are easier to produce via the continuous annealing process for the advantages of high temperature annealing and a high cooling rate.

The main objective of this paper is to study the influence of the annealing conditions on BH property in the continuous annealing route. Therefore, the effects of the cold rolling reduction rate, anneal temperature, slow cooling condition and rapid cooling condition were evaluated and discussed to determine their effects on the bake-hardening property and to clarify the complex metallurgical interactions.

## 2. EXPERIMENTAL METHOD

The grade of steel investigated in this study is JAC 340H and its mechanical properties are shown in Table

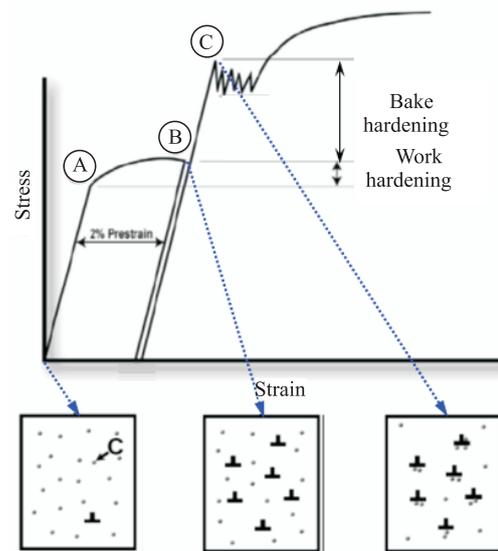
1. The chemical composition contained 0.0021wt% of C, 0.008wt% of Nb, and had some Ti and Mn additions in solid solution. Samples were taken from hot-rolled strips in an industrial production line. Two cold-rolled reductions of 77% and 60% were given to get a final thickness of 0.7mm. Annealing, slow cooling, rapid cooling and gas-jet quenching were implemented with samples of a size of 75mm×22mm, in a protective atmosphere consisting of a mixture of 7vol.% H<sub>2</sub> and 93vol.% N<sub>2</sub> with a Hot Dip Process Simulator (Iwatani and Surtec). Samples for the annealing test were soaked at four temperatures of T<sub>A1</sub>, T<sub>A2</sub>, T<sub>A3</sub> and T<sub>A4</sub> within a temperature range of 700°C to 900°C for a short time, followed by gas-jet quenching to room temperature. Samples after T<sub>A2</sub> annealing without quenching were prepared for slow cooling experiments. Three slow cooling rates (R<sub>sc1</sub>, R<sub>sc2</sub> and R<sub>sc3</sub> < 20°C/s) were adopted in a temperature range from T<sub>A2</sub> to T<sub>A4</sub> and also followed by gas-jet quenching. The rapid cooling test used samples with T<sub>A2</sub> annealing and R<sub>sc3</sub> slow cooling. Five rapid cooling starting temperatures, T<sub>rc1</sub>~T<sub>rc5</sub> within T<sub>A4</sub> to 500°C, were selected to cool the samples down to room temperature by gas-jet. All the annealing conditions are summarized in Table 2.

In this study, all the quenching steps were performed by gas-jet with a high cooling rate exceeding 100°C per second. This high cooling rate is to prevent carbide precipitation and to try to reflect the real supersaturated solute carbon concentration in every annealing condition. Bake hardenability is determined by measuring the flow stress increment between the values at 2% elongation and the higher yield strength after aging at 170°C for 20min, as shown in Fig.1. In this work, OM and Internal Friction Measurement (IFM) were also used to help investigate the influence of grain size and solute carbon concentration on BH.

### 3. RESULTS

#### 3.1 Effects of soaking temperatures

The effects of soaking temperature were evaluated in materials with 77% cold rolled reduction. Samples



**Fig.1.** Schematic diagram showing the definition of evaluating bake hardening (BH) strength and the interactions of carbon and dislocations.

were annealed at temperatures T<sub>A1</sub>, T<sub>A2</sub>, T<sub>A3</sub>, and T<sub>A4</sub> for a short time to perform recrystallization then were rapidly cooled to room temperature by gas jet. The microstructure evolution reached fully recrystallized status, as shown in Fig.2 indicating that all the samples had a nearly equiaxed ferritic structure. With such a fully recrystallized status, the variations of bake hardenability and solute carbon concentration, measured by tensile test and internal friction for different soaking temperatures, are shown in Fig.3. It is obvious that a strong influence of soaking temperatures on BH property can be observed; as the higher the soaking temperature, the higher the BH was. In addition, the solute carbon content has the same influence on BH. In Fig.3 the BH values increase greatly with a growing solute carbon content. This tendency is totally similar to the effect of the soaking temperature.

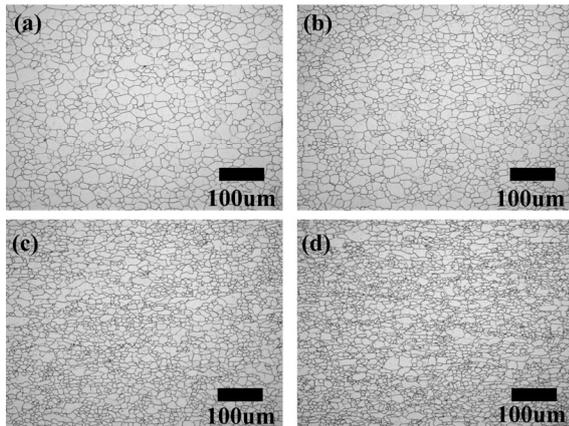
The effect of the soaking temperature on the yield stress is shown in Fig.4. Unlike Fig.3, a negative slope of the yield stress appears. The yield strength decreases with an increasing soaking temperature.

**Table 1** Mechanical properties of JAC340H grade BH steel (wt.%)

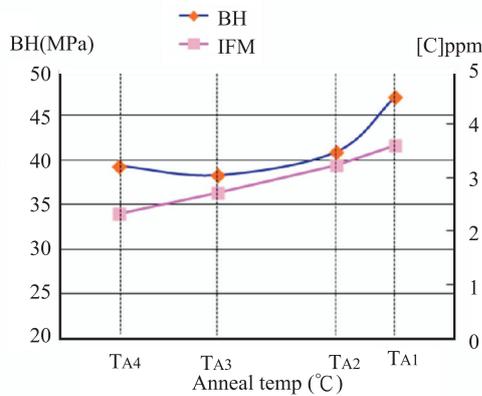
| Steel    | Yield strength (MPa) | Tensile strength (MPa) | Total elongation (%) |
|----------|----------------------|------------------------|----------------------|
| JAC 340H | 219.26               | 349.8                  | 41.98                |

**Table 2** Annealing conditions

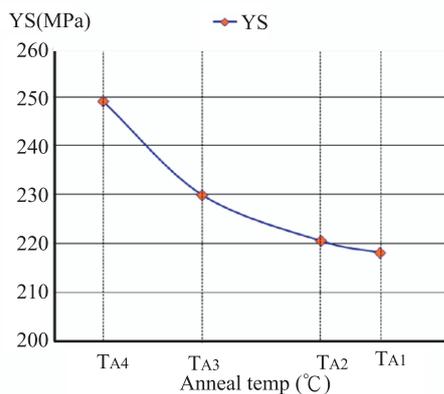
|      | Annealing temperature | Slow cooling     | Rapid cooling | Quenching  |
|------|-----------------------|------------------|---------------|------------|
| No.1 | TA1, TA2, TA3, TA4    |                  | GJ to R.T.    |            |
| No.2 | TA2                   | Rsc1, Rsc2, Rsc3 | GJ to R.T.    |            |
| No.3 | TA2                   | Rsc3             | Trc1~Trc5     | GJ to R.T. |



**Fig.2.** Microstructures of samples after annealing at temperatures of (a) $T_{A1}$ , (b) $T_{A2}$ , (c) $T_{A3}$ , (d) $T_{A4}$ . Their corresponding grain sizes are 13.61 $\mu\text{m}$ , 11.95 $\mu\text{m}$ , 8.72 $\mu\text{m}$  and 7.74 $\mu\text{m}$ .



**Fig.3.** Bake hardenability and solute carbon concentration measured by tensile test and internal friction for different soaking temperatures.

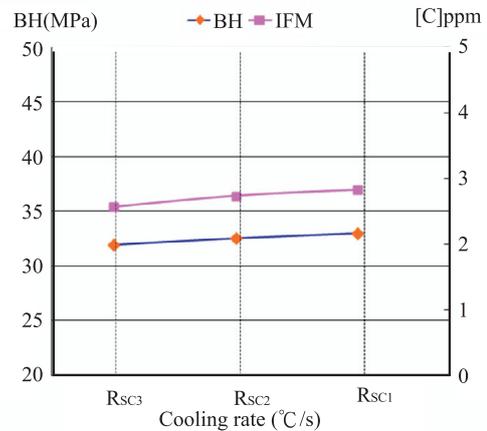


**Fig.4.** Yield stress variation after soaking at different temperatures.

### 3.2 Effects of slow cooling and rapid cooling

The variations of BH properties in a slow cooling rate test are shown in Fig.2 for samples annealed at  $T_{A2}$

for a short time followed by gas-jet quenching. Three slow cooling rates  $R_{sc1} > R_{sc2} > R_{sc3}$ , that are all smaller than 20°C/s, were used to verify the effect of slow cooling in a temperature range of  $T_{A2}$  to  $T_{A4}$ . From Fig.5, it can be seen that the BH value slightly increases with an increasing slow cooling rate. This promotion is not as obvious as the effect of soaking temperature. In addition, the solute carbon content measured by IFM also exhibits the same small enhancement on the BH. In Fig.5 the small positive slope of solute carbon content implies that still more free solute carbon remained in matrix with a higher slow cooling rate.

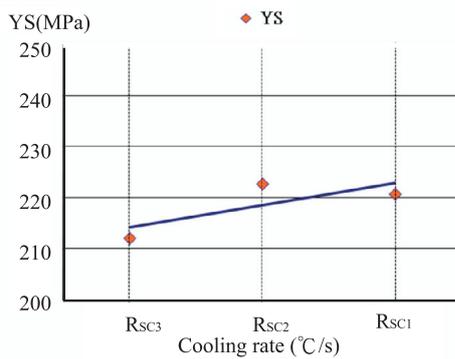


**Fig.5.** Variations of bake hardenability and solute carbon concentration with different slow cooling rates.

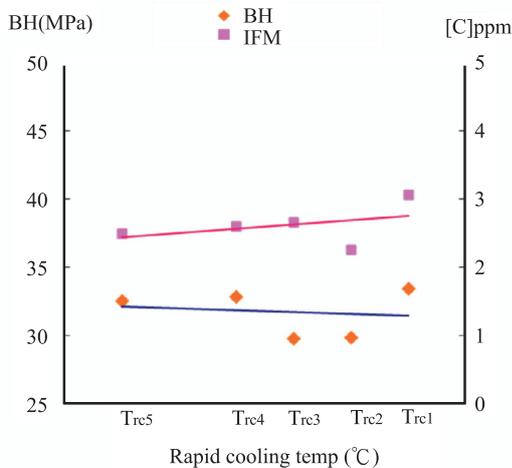
Figure 6 shows the yield stress variation versus the different slow cooling rates. The yield strength slightly increased when a higher slow cooling rate was adopted. This is consistent to the small improvement of the BH during the slow cooling process. All the findings mentioned above indicate a clear but small promotion of slow cooling rate on bake hardenability and yield strength. Figure 7 shows the variations of the bake hardenability and solute carbon concentration with different rapid cooling starting temperatures. Samples were annealed at a temperature of  $T_{A2}$  with a slow cooling rate of  $R_{sc3}$  followed by rapid cooling from  $T_{rc1} \sim T_{rc5}$  to room temperature to study how rapid cooling starting temperatures affect the BH property. However, no strong correlation between bake hardenability and rapid cooling starting temperatures was found.

### 3.3 Effects of cold-rolling and grain size

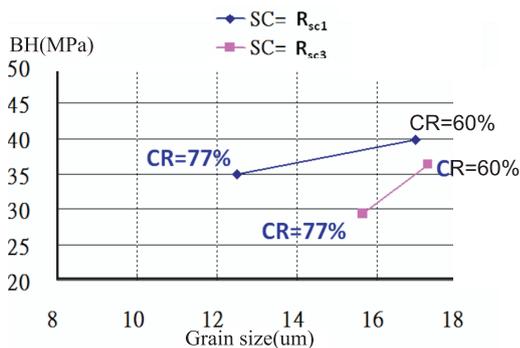
The effects of cold-rolling and grain size on the BH were investigated in this work. Two cold-rolled reduction rates of 60% and 77% were applied in slow cooling and rapid cooling experiments. Figure 8 shows the variations of bake hardenability and grain size with



**Fig. 6.** Yield stress variation for different slow cooling rates.

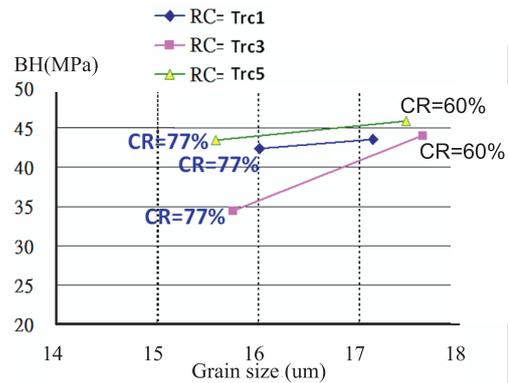


**Fig. 7.** Variations of bake hardenability and solute carbon concentration with different rapid cooling starting temperatures.



**Fig. 8.** Variations of bake hardenability and grain size with different cold reduction and slow cooling rates.

different cold-rolled reduction rates and slow cooling rates. From Fig. 8, it can be noted that more BH was achieved with a larger grain size by less cold-rolled reduction in whatever slow cooling conditions. Figure 9 also shows the same characteristic, i.e. the BH was improved with large grain size by less cold-rolled



**Fig. 9.** Variations of bake hardenability and grain size with different cold reduction rates and rapid cooling starting temperatures.

reduction at every rapid cooling starting temperature. These results show the great importance of grain size to the BH property and will be discussed in the following sections.

## 4. DISCUSSIONS

### 4.1 Solute carbon content variation during soaking process

The strengthening mechanism of bake hardening is attributed to strain aging by interstitial solute atoms. In the Nb-Ti IF steel we investigated, theoretically all of the nitrogen is assumed to form aluminum or titanium nitrides at higher temperatures and only small amounts of carbon remained in solid solution as solute carbon to control the BH property. During soaking at the annealing temperatures of  $T_{A1}$ ,  $T_{A2}$ ,  $T_{A3}$  and  $T_{A4}$ , the individual equilibrium solubility of carbon can be reached. Figure 3 documents the solute carbon content increases with increasing soaking temperature. This phenomenon resulted from more dissolution of niobium carbide<sup>(10-11)</sup> with an increasing soaking temperature and the solute carbon partially remaining in the subsequent cooling process. Therefore, more free carbon was available to pin dislocations during the 170°C aging treatment and the BH property is therefore improved.

### 4.2 Solute carbon content variation during cooling process

It is well known that diffusion strongly depends on temperature and time. The solute carbon dissolved from niobium carbide during high temperature soaking has a tendency to re-precipitate during the cooling process<sup>(12-13)</sup>. Figure 5 illustrates the variation of solute carbon concentration in the cooling process. More solute content was observed with a higher cooling rate because the rapid cooling prevents the reprecipitation of niobium carbide as the solute carbon has less time to

diffuse and produces a supersaturated solute carbon concentration in matrix. On the other hand, with slower cooling rates, the dissolved solute carbon during soaking had more time to return to the niobium clusters and to re-precipitate as niobium carbide in the lower temperature ranges. All gas-jet quenching in this study was therefore intended to produce the least reprecipitation of NbC upon cooling and to maintain the highest value of solute carbon content.

#### 4.3 Grain boundary

Higher soaking temperatures not only enhance the dissolution of niobium carbide but also enlarge the grain size and reduce the ratio of grain boundaries. After continuous annealing some solute carbon might segregate to grain boundaries during the cooling process. L. Storjéva proposed<sup>(14)</sup> that these interstitial solute atoms which diffused to low energy position, grain boundaries, might exhibit different strain aging characteristics than those distributed in the grain interior. The carbon atoms which segregated along the grain boundaries were more stable and required higher temperatures for strain aging compared to those inside the grain interior. S. Hanai and N. Takemoto<sup>(15)</sup> reported that in the same solute carbon concentration, sheet steels with fine grain sizes shows higher the BH values than those with coarse grains. S. Hanai and N. Takemoto also conclude that solute carbon along grain boundaries can influence the BH property through high temperature (170°C) strain aging. Moreover, these solute carbons along grain boundaries were not affected by room temperature strain aging, unlike the solute atoms found within the grain interior.

However, different points of view were taken by other researchers. A significant increase of BH values was observed in ULC-BH steels with a coarse grain size<sup>(16)</sup>. Both the solute carbon content measured by IFM and the increase of yield stress support the view that the larger grains, i.e. those with less grain boundaries, promote the BH property. This suggests that the grain boundaries can be seen as the sink of interstitial atoms and decrease the amount of solute carbon content that would contribute to BH values. The same phenomenon was observed in this study as shown in Fig.8 and 9. All the experimental data obtained in this work showed the tendency that a greater BH is accompanied by a larger grain size. The root causes of the different results mentioned above are not well understood. A further study focusing on the BH property variation along the grain boundaries and in the grain interior is suggested to clarify this difference.

### 5. CONCLUSIONS

This paper studies the annealing conditions in a continuous annealing process with BH steels of

JAC340H grade taken from the China Steel industrial production line. Bake-hardening was found to be strongly influenced by solute carbon content and grain size. The final solute carbon content and the grain size were influenced by the soaking temperature and the following cooling procedures. The conclusions are summarized as follows:

- (1) Annealing performed in a particular temperature range shows a strong influence on the BH property and the solute carbon content. Higher soaking temperatures promote the dissolution of the solute carbon content and thereby enhance the bake hardenability.
- (2) Cooling rates also affect the solute carbon content but not as obviously as the tendency of soaking temperature. The diffusion of solute carbon to form niobium carbide clusters can be suppressed by a higher cooling rate and the solute carbon can partially remain in the matrix.
- (3) Experimental results show the correlation between grain size and BH property. The BH value increases with a larger grain size.

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