Influencing Factors of Nb-Ti Treated ULC-BH Steels' Bake Hardening Property

P. Tian

State Key Laboratory of Electronic Thin Films and Integrated Devices, University of Electronic Science and Technology of China Chengdu 610054, China Chengde Branch, Hebei Iron & Steel Co., Ltd. Chengde 067102, China

Y. Cui

School of Metallurgical and Energy, Hebei United University Tangshan 063009, China

Abstract—The precipitate in ULC-BH steels was analyzed by the phase analysis, the expressions for calculation of solution C content in the ULC-BH steel treated by Ti and Nb was developed. The test result indicates that the BH2 value was improved with the increasing of solution C content. The BH2 value can be improved by enhancing the temperature of annealing and cooling rate. The steel has higher BH2 value when the coiling temperature was 640°C compared with 710°C and the annealing temperature and cooling rate have more effect on bake hardening value than coiling temperature.

Keywords-ULC steels; bake hardening; solute carbon content

I. INTRODUCTION

Ultra Low Carbon-Bake Hardening (ULC-BH) steel is common to use ULC-steel (C \leq 0.005%, N \leq 0.004%) containing small amounts of titanium and/or niobium to partially stabilize the steel. Small amounts of carbon atoms are preserved after annealing and temper rolling. Bake hardening (BH) effect are caused by the diffusion of carbon to dislocation (segregation) and the pinning of dislocations impairing a subsequent deformation. The solution strengthening can be also be achieved by adding P. Bake hardening does not significantly affect production costs, and can also allow a greater final strength level to be achieved in higher formability grades.

At present a small amount of big steel manufactories in China can produce ULC-BH steel, but the product's capability stability need to be improved. The problem can be solved by controlling the component and adjusting the technological parameter. The amount of solute carbon affects the desired BH-effect, so controlling the component and adjusting the technological parameter are the key points to produce the high grade ULC-BH steel. Research on the influence of the annealing process was less for the mechanical properties of V or V-Ti ULC-BH Steel [1-3]. In this article, we found the way to calculate the solution carbon content, analyzed effect of R.G. Bai, X.L. Zhang, H. Gao Chengde Branch, Hebei Iron & Steel Co., Ltd. Chengde 067102, China

Z.Y. Zhong State Key Laboratory of Electronic Thin Films and Integrated Devices, University of Electronic Science and Technology of China, Chengdu 610054, China

coiling temperature and annealing technological on BH2 value, expecting giving reference for producing ULC-BH steel.

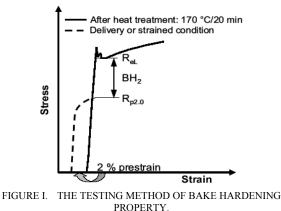
II. MATERIALS AND EXPERIMENTAL PROCEDURE

The experimental production of ULC-BH steel is smelting by vacuum inductance furnace, the final steel sheet production about 0.8mm thickness was got after forging, hot rolling, cold rolling, annealing, and temper rolling. The finishing temperature is 910°C, the simulation coiling is keeping the temperature (640°C and 710°C) for one hour and furnace cooling. Table 1 shows the composition analysis of experimental steels. To get the effect of technological parameter on BH2 value, the continuous annealing simulation furnace, which simulated the effect of different annealing temperature and cooling rate on the experiment production's capability, was used. The experiment annealing steel for phase analyze is gotten by oil quenching after salt bath annealing.

TABLE I. COMPOSITION ANALYSIS OF EXPERIMENTAL STEELS (MASS FRACTION, %).

| No | С | Nb | [C] | N | Ti | Nb/C | Ti/N |
|-----|--------|-------|------|--------|-------|-------|-------|
| 1# | 0.0036 | 0.012 | 20.5 | 0.0033 | 0.009 | 0.431 | 0.780 |
| 2# | 0.0030 | 0.012 | 14.5 | 0.0042 | 0.012 | 0.517 | 0.835 |
| 3# | 0.0039 | 0.020 | 11.4 | 0.0036 | 0.013 | 0.663 | 1.056 |
| 4# | 0.0023 | 0.010 | 10.1 | 0.0039 | 0.010 | 0.562 | 0.750 |
| 5# | 0.0024 | 0.011 | 9.8 | 0.0042 | 0.011 | 0.593 | 0.766 |
| 6# | 0.0023 | 0.008 | 6.3 | 0.0035 | 0.014 | 0.461 | 1.203 |
| 7# | 0.0020 | 0.012 | 4.3 | 0.0032 | 0.011 | 0.776 | 1.005 |
| 8# | 0.0038 | 0.012 | 3.9 | 0.0028 | 0.017 | 0.408 | 1.775 |
| 9# | 0.0019 | 0.012 | 3.5 | 0.0032 | 0.007 | 0.817 | 0.658 |
| 10# | 0.0030 | 0.010 | 0 | 0.0031 | 0.018 | 0.414 | 1.670 |
| 11# | 0.0019 | 0.014 | 0 | 0.0031 | 0.012 | 0.953 | 1.132 |

After the metallographic specimen whose surface parallel to the rolling direction (including full thickness) was grinded, polished, the microstructure and grain size were observed in optical microscope. The metallographic analysis and measurement software was used to analyze the ferrite grain equivalent diameter. In order to test the precipitation solution behaviour of hot rolled and annealed steel, physical and chemical methods of quantitative determination was used to analyze the phase composition. The BH2 of test plate was menstruated by the room temperature tensile test, the testing method [4] of bake hardening property shown in Fig.1.



III. RESULT AND DISCUSSION

A. The Chemical Formula of Solution Carbon Content

To get the formula for calculating solution carbon content through analysis. The experiment annealing steel for phase analyze was gotten by oil quenching after the cold-rolled steel's salt bath annealing (830°C×60s). The second-phase in the annealing steels and corresponding hot rolling steels was analyzed, the results of the phase analysis shows the hot-rolled plate and annealed sheet have the same types of precipitate: TiN, TiC, NbC, Nb(C, N), AlN, MnS, etc. TiS and Ti4C2S2 in the Ti treated ULC-BH steels which reported in the literature [4-6] was not found. Compared to hot-rolled plate, (Nb, Ti) (C, N) is only a slight decrease in annealed sheet as table 2, the solute carbon content of three steels only increase 2, 4, 6 ppm, so only a slight (Nb, Ti) (C,N) is dissolve. If you do not consider the annealing of (Nb, Ti) (C, N) precipitates dissolved, assuming that (Nb, Ti) (C, N) is total precipitated, so the chemical formula of the solution carbon content of ULC-BH steel was shown in Fig.2. The solution carbon content (Table 1) was calculated by the chemical formula in Fig.2.

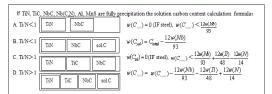


FIGURE II. THE PRECIPITATED PHASE FORM WITH DIFFERENT TI AND NB CONTENT.

B. Effect of Different Parameters on BH2 Value

1) The effect of solution carbon content on BH2 value: Select cold rolled steel sheet of No.1-No.11 in table 1 to simulate the continuous annealing $(830^{\circ}C \times 60s, \text{ cooling rate}: 50^{\circ}C / s)$. The relationship between solution C content calculated and BH2 value have been tested. Fig.3 showed that BH2 value increased with the increasing of solid solution carbon content. So controlling the solution carbon content in 12-23ppm ensure the BH2 value being about 30-50MPa when the annealing temperature is 830 °C and every 1ppm solution carbon can provide about 2MPa BH2 value.

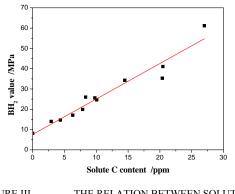
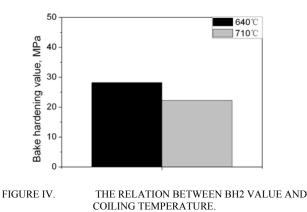


FIGURE III. THE RELATION BETWEEN SOLUTION C CONTENT AND BH2 VALUE.

2) The effect of coiling temperature of hot-rolled plates on BH2 value: Select the cold-rolled plate which coiling temperature was 710 °C and 640 °C to simulate continuous annealing (830 °C ×60s, cooling rate was 50 °C/s). Fig.4 showed the relationship between BH2 values and coiling temperature. Select cold rolled steel sheet of No.5 in table 1, the BH2 value is higher 5 MPa at 640 °C coiling temperature than at 710 °C coiling temperature.



3) The effect of annealing temperature on BH2 value: By using the continuous annealing simulation furnace to simulate the different annealing temperature (810° C, 830° C, 850° C), after kept heating for 60s, and then cooling at 50 °C /s.

Select No. 6, No.7, No.8 steel to study the effect of annealing temperature on bake hardening property. Fig.5 shows that BH2 value increased 7-14 MPa with the annealing temperature increased from 810 $^{\circ}$ C to 850 $^{\circ}$ C.

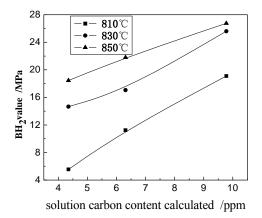
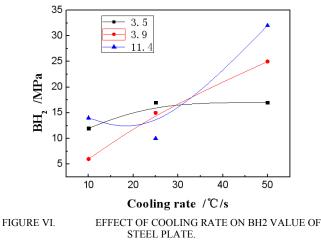


FIGURE V. EFFECT OF ANNEALING TEMPERATURE ON BH2 VALUE OF STEEL PLATE.

4) The effect of cooling rate on BH2 value: Put No. 7, No.6, No.4 steels whose solution carbon content were 3.5ppm, 3.9ppm and 11.4ppm respectively into continuous annealing simulation furnace to simulate the different annealing cooling rate on bake-hardening properties. It can be seen from Fig.6 that the bake-hardening properties of steels gradually increased 5-20MPa as cooling rate increased from 10 to 50° C/s.



IV. ANALYSES

From the above results we can see that BH2 value depends on the control of solid solution C content. Phase analysis showed the type and the amount of precipitates is basically the same in hot-rolled and annealed steel sheet, in 830°C heat preservation and 60s anneal process, NbC dissolved so little, almost could be negligible. Therefore we may estimate the content of solid solution carbon content by calculating the state of (Nb, Ti) (C, N) completely precipitated. Experiment proved that corresponding relationship exists between the calculated value of solid solution C and BH2 value. The establishment of this relationship provides a good reference for the practical production. The solution carbon content calculated and in the actual lattice with the changing trends in the composition is basically the same, but there is a certain degree of deviation. Because the solution C content in the lattice is not only concerned with the composition, but also be impacted by coiling temperature of hot-rolled plates, cold-rolled sheet annealing temperature and cooling rate. Therefore, by summarizing experience we find the relationship between the solution C value calculated and BH2 value would be beneficial in different process parameters in actual production.

The steel has higher BH2 value when the coiling temperature was 640°C compared with 710°C. The reason [5] may be that with the lower coiling temperature, Nb with slower diffusion velocity result in plentiful, small and scattered NbC second-phase particles precipitated in hot-rolled steel plates. Because Nb atom's diffusion distance becomes shorter in annealing process, with more NbC particles dissolved in a limited time, the increase of solution carbon content makes BH2 value higher. Because NbC dissolve less in annealing process, so coiling temperature has little effect on the BH2 value. It can be supposed that the influence of coiling temperature may increase with the increase of annealing temperature because more NbC could be dissolved in high annealing temperature.

When the annealing temperature increased, BH2 value significantly increased, this are concerned with two factors. The first factor is that partially solution of NbC produced a small amount of solid solution C in annealing process. But phase analysis showed that NbC dissolved little in annealing process. The second factor is that with the increase of annealing temperature, the grain size become bigger and the grain boundary area decreased which lead to the decrease of total amount of carbon in grain boundary, so the solution carbon content in matrix and BH2 value are increased [7].

Improving the cooling rate after heating is good for reduce precipitation of NbC, so the solution of carbon content can be improved, bake hardening property can be obtained easily. So improve annealing cooling rate is good for getting enough BH2 value. Taking into account of the actual annealing production lines with limited cooling rate, it selected higher cooling rate about $50^{\circ}C/s$ is useful.

V. CONCLUSION

1) BH2 value of Nb-Ti treated ULC-BH steel is mainly relevant to the solution carbon content. The BH2 value can be estimated through the establishment of the solution carbon content formula.

2) Higher annealing temperature results in improving of solution carbon content, so BH2 value is increased. It's useful to improve the cooling rate after annealing to prevent the Nb(C, N) particles precipitation, so the bake hardening property can be improved.

3) Compared with 710 $^{\circ}$ C, when coiling temperature was 640 $^{\circ}$ C, the BH2 value was slightly higher, but had little effect. Annealing temperature and cooling rate after annealing have greater impact on BH2 value.

REFERENCES

- S.W. Ooi, G. Fourlaris, A comparative study of precipitation effects in Ti only and Ti-V ultra low carbon strip steels, Materials Characterization, 56(3), pp.214-226, 2006.
- [2] S.W. Ooi, G. Fourlaris, D.A. Tanner, et al, Precipitation in vanadium bearing ultra low carbon strip steels, Materials Science and Technology, 22(5), pp.525-536, 2006.
- [3] A.S. Waleed, G.S. Tohn, Kip Findley, et al, Effect of annealing time on solute carbon in ultra low carbon Ti-V and Ti-Nb steels, Metallurgical and Materials Transaction A, (37), pp.206-216, 2006.
- [4] Christine Escher, Volker Brandenburg, Ilse Heckelmann. Bake hardening and ageing properties of hot-dip galvanized ULC steel grades. International Symposium on Niobium Microalloyed Sheet Steel for Automotive Application, pp.383-395, 2006.
- [5] L.J. Baker, Metallurgy and processing of ultralow carbon bake hardening steels, Materials Science and Technology, 18 (4), pp.355-368, 2006.
- [6] Xiaojun Guan, Jiajuan Zhou, Wei Pan, et al, Research on precipitates in Ti-treated ELC-BH sheet, Iron and Steel, 37(7), pp.50-53, 2002.
- [7] A. Soenen, B.D. Cooman and S. Vandeputte, Carbon distribution between matrix, grain boundaries and dislocations in ultra low carbon bake hardenable steels, 42nd mechanical working and steel processing conference proceedings, 28 (9), pp.31-37, 2001.