Research Article

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Improvement of toughness and hardness in BR1500HS steel by ultrafine martensite

<https://doi.org/10.1515/htmp-2020-0069>

received December 1, 2017; accepted June 3, 2018

Abstract: To obtain the ultrafine martensite for BR1500HS ultra-high-strength steel, a new preparation process of cyclic heat treatment (CHT) with descending heat temperature and holding time along with cycle steps was developed. First, a series of thermal dilation tests were conducted with the temperature range of 420–730°C and the heat rate of 5 K/s on a Gleeble-3800 thermomechanical physical simulator. According to the experimental data, the temperature ranges and the optimal holding time to completely austenitize BR1500HS were determined. Then, to confirm the optimal parameters of CHT for BR1500HS, several tests with various temperatures and cycle steps were conducted and analyzed by optical microscope and scanning electron microscope. Subsequently, the CHTs with decreasing heating temperature and holding time were studied due to the increasing internal energy of steel along with the CHT process. The lath width was measured as a criterion to evaluate the refinement degree in this article. After several loops of heat treatment, the lath width is reduced to 0.268 μm . Finally, the hardness evolution of the specimens subjected to CHT in this study was analyzed and compared with the lath width test results, which justified the effectiveness of the new developed process.

Keywords: ultrafine martensite, ultra-high-strength steel, cyclic heat treatment, phase transformation

1 Introduction

In recent years, an ultra-high-strength steel (UHSS) BR1500HS has been developed to meet the needs of increasing strength and reducing weight of automotive [1]. BR1500HS has a yield strength higher than 550 MPa and a tensile strength higher than 700 MPa at room temperature [2,3]. Components made of BR1500HS are always prepared by the hot forming process coupled with synchronous quenching, in which the non-diffusive phase transformation from austenite to martensite occurs and brings about the improvement of hardness and strength [4,5].

For impact-resistant components, not only higher hardness and strength but also a certain degree of toughness is pursued. In fact, high hardness and strength are always determined by the martensitic transformation level, while the toughness is ensured by the refinement degree of martensite [6]. A cyclic heat treatment (CHT) containing several loops of solution and quenching can be an effective approach to achieve the fine-grained or even ultrafine-grained martensite phase by controlling the austenite grain size in the solution process before quenching [3,7,8]. In the material field, the refined grain (with a grain size of $d \leq 10 \mu\text{m}$) was divided into three classes, which are micrometer ($1 \mu\text{m} \leq d \leq 10 \mu\text{m}$), ultrafine grained ($500 \text{ nm} < d \leq 1 \mu\text{m}$) metals and nanocrystalline (with a grain size of $10 \text{ nm} \leq d \leq 500 \text{ nm}$) [9,10]. In order to obtain the ultrafine-grained martensite, it is a major issue to determine the two vital parameters of this CHT, i.e. austenitic solution temperature and holding time [11,12]. Too much attention has been paid to the trial-and-error experiments that lack measurement of the actual phase transformation curves and analysis of the relative characteristics in actual production, while in traditional research, solution temperature and holding time are always designed as constants in each cycle due to the lack of the study in non-diffusive phase transformation kinetics [13–17], which makes it almost impossible to find the optimal CHT routine. Consequently, in order to obtain the ultrafine martensite phase, it is significant to find an optimal CHT routine for UHSS BR1500HS.

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Table 1: Chemical composition of the studied BR1500HS steel

Chemical composition	C	Si	Mn	Cr	Mo	B	S	P	Fe
% by weight	0.23	0.25	1.35	0.19	0.04	0.03	0.006	0.01	Balance

Over the last few decades, CHT has been employed in many steels and alloys to refine the grain and obtain an enhancement mechanical property. Saha et al. [14] conducted the CHT with 1–8 cycles for a 0.16% carbon steel and then analyzed the morphology features and dislocation distribution. The results showed that the grain size of martensite decreased first and then increased, i.e., too many cycles caused grain growth. It was summarized that more cycles were not preferable, and the loops of a CHT were a vital parameter. Kiran et al. [15] designed a CHT routine with the same soaking temperature and time in each cycle for a tungsten heavy alloy containing cobalt and molybdenum and achieved a marginal increase in tensile property and impact toughness. Wang et al. [16] achieved the refined grains of TiAl alloys by three CHT routines with the same soaking temperature and time in each cycle and concluded that the grains with an average size of 1 mm could be refined to 10–30 μm . Saha et al. [14] pointed that the required energy to generate new grains and form martensite in a cycle was decreasing with the cycle number increasing in actuality. That is to say, if the energy in each cycle, which is afforded by the temperature and holding time of the heated process, is the same and exceeding the critical value, the refined grains will grow up and the refinement will be counteracted to a certain degree. Thus, in order to obtain the ultrafine martensite grains and further enhance their mechanical properties, it is significant to redesign the routine of CHT with varying temperature and holding time.

In this work, a new preparation process of CHT with varying temperature and holding time in each loop of solution and quenching was designed to acquire the ultrafine-grained martensite with the grain size smaller than 1 μm . The thermal dilation tests were conducted on a Gleeble 3800 thermal physical simulator so as to identify the complete austenitizing temperature (A_{c3}) and the optimal holding time at austenitizing temperature. Thereafter, the solution temperature range of $A_{c3} + (30-50)^\circ\text{C}$ was determined. Then, a series of CHTs were conducted to determine the optimal solution temperature and cycle steps to achieve optimal CHT routine. Subsequently, the CHTs with degressive solution temperature and holding time were conducted and the lath width was analyzed by optical microscope (OM) and scanning electron microscope (SEM). Finally, the lath width and hardness of each specimen were measured and compared, which indicated the effectiveness of the newly developed preparation process.

2 Materials

BR1500HS has low austenitic grain boundary energy and high hardenability due to additional boron. The chemical compositions and the original microstructure of BR1500HS analyzed in this study are given in Table 1 and Figure 1(a), respectively. The as-received steel contains massive pro-eutectoid ferrite and pearlite areas which mainly form at the triple point junction on the grain boundary [18]. Then, the

Figure 1: Microstructure of the BR1500HS (a) as-received and (b) treated with solution treatment (ST).

Figure 2: Schematic of lath martensite structure of low carbon steel.

specimens are heated to 1,100°C with a fixed heating rate of 5°C/s and kept at this temperature for 7 min, which is followed by air cooling for solution treatment. The microstructure of the specimen after such solution treatment is exhibited in Figure 1(b). A mass of retained austenite exists and the martensite is extremely large.

In martensitic steel BR1500HS, a grain often comprises primary austenite grains, packets, blocks and laths, as shown in Figure 2. It is well accepted that the microstructure of martensite is affected largely by the primary austenite grains [19]. Morito et al. [17] pointed that a primary austenite grain consisted of several packets with different orientations, which were composed of either one block (shown in Figure 2c) or two blocks separated by high-angle boundary (shown in Figure 2b). Each block included a mass of slightness martensite laths that were approximately paralleled to each other with the same or similar orientation. As the minimum unit in the martensite, lath width is a critical criterion for refined martensite and it is measured in this work to evaluate the refinement degree.

3 Experiment

3.1 Principles of CHT

During a heat treatment process, the martensitic steel undergoes a phase transformation of martensite-to-austenite ($\gamma \rightarrow \alpha$) in the heating period and austenite-to-martensite ($\alpha \rightarrow \gamma$) in the cooling period. The $\alpha \rightarrow \gamma$ transformation is a diffusionless massive polymorphic transformation which occurs enormously fast with different orientations between martensite and original austenite. The crystal structure of steel that underwent $\alpha \rightarrow \gamma$ transformation converts from face-centered cubic to body-centered

Figure 3: The mechanism of CHT for BR1500HS.

cubic with no composition variation, hence the variation of atom position is not more than one atomic spacing [20]. On the contrary, the transformation of $\gamma \rightarrow \alpha$ is regarded as the typical diffusion transformation. As the temperature increases, the supersaturated carbon atoms dissolve from martensite and cementite forms in the matrix, then the cementite dissolves gradually and new austenite grains nucleate. Moreover, the refined grain is prone to nucleate at the high-energy sites, such as grain boundaries of primary austenite and the interface of ferrite and cementite [21]. During the transformation of $\gamma \rightarrow \alpha$, the higher the nucleation rate is, the smaller the austenite grains are.

The evolution of the microstructure during CHT for BR1500HS is illustrated in Figure 3. During a heat treatment cycle, new austenite grains nucleate first on the grain boundaries of primary austenite and martensitic packets at the heating period. Then, martensite generates at the cooling period along with water quenching. The CHTs promote nucleation in non-nucleated regions, and thus refinement grains can be obtained. This is mainly due to the fact that $\alpha \rightarrow \gamma$ is a shear transformation, during which a large number of microdefects involving dislocations, twins, stacking faults etc. generate and enhance the stored energy. Such microdefects are inherited in the latter transformation of $\gamma \rightarrow \alpha$ and the stored energy is increasing through CHT with the fragmentation of microstructure and the increase in dislocation density. Likewise, the internal stored energy and the driving force of phase transition increase as the CHT times increase.

3.2 Identification of solution temperature range and holding time

The optimal solution temperature is significant for the CHT procedure, which is well accepted to be $Ac_3 + (30-50)^\circ\text{C}$ for hypoeutectoid steel. When the solution temperature is between Ac_1 and Ac_3 , a portion of ferrite would be reserved

Figure 4: Processing route for CHT with decreasing quenching temperature.

addition to martensite and the strength and stiffness will be decreased greatly. On the contrary, if the quenching temperature is higher than Ac_3 too much, the austenite grains will grow to a large extent and thus reduce the toughness.

Thermal dilation test is widely used to identify the phase transformation point of metal and alloys with the tangent method because phase transformations always cause volume expansion or shrinkage [11,12]. The density order of different phases of steel is as follows: austenite (γ -Fe) > pearlite (P) > ferrite (α -Fe) > martensite [13]. Hence, the transformation of other phases to austenite always brings about the volume shrinkage while the dissolution of austenite leads to the volume expansion. To measure the thermal dilation tests, the specimen length, which represented the volume changes of specimens, was monitored continuously by a personal computer and eventually converted into the dilatometric curves through an automatic data acquisition system. Figure 4 illustrates the heat dilatometric curve of BR1500HS at the holding temperature of 640°C. It is found that the length of specimen is variational with the temperature. During the expansion test, the temperature was heated to 1,100°C with a fixed heating rate of 5°C/s first, and kept in this temperature for 7 min to austenitize the specimens completely. Then, the specimen was cooled to 640°C with a fixed cooling rate of 50°C/s and held for sufficient time (15–20 min).

Obviously, the heat dilatometric curve can be separated into the following three parts: heating period, holding period and cooling period. During the first heating stage, the specimen length increased linearly due to simplex thermal expansion effect before the occurrence of phase transformation from ferrite + pearlite (bcc) to austenite (fcc), which brought about a distinct inflection point. Moreover, the temperature corresponding to the inflection point was just

the initial temperature of phase transformation from ferrite + pearlite to austenite (Ac_1). Based on such characteristic, the tangent method was employed to find out the temperature of the inflection point. As the phase transformation proceeded, the decreasing trend of the length continued to increase. When the specimen was austenitized completely, the other inflection point appeared and the corresponding temperature was considered as the complete austenitizing temperature (Ac_3). According to the experimental results, the mean temperature Ac_3 was measured as 871°C. Then, the optimal temperature region of quenching was determined to be 900–960°C in this article.

Thereafter, the specimen length kept its linear expansion again with increasing temperature and then arrived at a peak value when the temperature reached 1,100°C. However, during the following temperature holding period, the specimen length reduced due to the dissolution of interstitial compounds, secondary phases and alloying elements. Such trend got alleviated gradually. The holding time was vital in the heat treatment, which should make the dissolution process occur to a large extent and prevent the generation of coarse austenite grains in the meantime. Under such premise, several tests were conducted with different holding times and 5 min was employed in this article after a number of analyses.

3.3 Experiment schedule design

To determine the optimal solution temperature, an experiment schedule which contained four different temperatures of 900, 920, 940 and 960°C; a fixed holding time of 5 min; and a fixed cycle step was designed. In each cycle, the specimen was heated to a certain temperature of 1,100°C with the heating rate of 10°C/s and then held for 5 min in the electric resistance furnace. The optimal temperature was determined by the analysis results of the martensite morphology and packet size in these CHT specimens.

Moreover, to confirm the optimal cycle steps, an experiment schedule which contained four different steps of one cycle, two cycles, three cycles and four cycles, a fixed solution temperature of T_{best} , a fixed holding time of 5 min and water cooling was designed. The optimal cycle steps were obtained from the analysis results of these CHT specimens.

As the optimal solution temperature (T_{best}) and cycle steps were recognized, the experimental procedures of the CHT with descending temperature and holding time along with the cycle steps were designed and are shown in Figures 5 and 6.

Figure 5: Testing procedures for CHT with decreasing holding time.

Figure 6: The diagram of the measured lath width.

3.4 Experimental procedures

The as-received BR1500HS steel was machined into cylindrical specimens with a length of 25 mm and a diameter of 6 mm by wire-electrode cutting. To austenitize the specimens completely and to reduce anisotropy as well as internal temperature gradient simultaneously, the specimens were heated to 1,100°C with a fixed heating rate of 5°C/s and kept at this temperature for 7 min, which was followed by air cooling for solution treatment. Thereafter, to determine the temperature and holding time in CHT, a series of thermal dilation tests on the basis of volume expansion and shrinkage induced by phase transformations were conducted. Subsequently, the CHT tests designed in Section 4.1 were conducted, and all the tested bars were sectioned to small pieces. After being polished with successive grades of emery papers and diamond paste, these specimens were etched by 4% Nital and observed with metallurgical OM and SEM in a secondary electron image mode. After observations, the SEM images were used to measure the lath width of martensite to evaluate the refined effects. The diagram of the measured lath width is illustrated in Figure 7. In each image, ten blocks were selected and the widths of five laths in each block were measured to achieve the mean value of lath width.

Finally, three points that distributed uniformly along with the diameter of these specimens were selected and Vicker's hardness was measured. Then, an even value of hardness was obtained to estimate the property of these specimens.

4 Results and discussion

4.1 Determination of the optimal solution temperature and cycle steps

The CHT tests with a fixed holding time of 5 min, a fixed one cycle step, the same water cooling and four different

solution temperatures of 900, 920, 940 and 960°C were conducted to identify the optimal solution temperature. The metallographs of these specimens were observed and demonstrated in Figure 8. As the solution temperature increased from 900 to 960°C, the grain size of specimen was decreasing, the amount of martensite was increasing, the distribution of martensite became more homogeneous and the retained austenite was reduced. The grain size of specimen quenched from 900°C was refined. However, there were still many retained austenite with various sizes as shown in Figure 8(a). When the solution temperature was 920 and 940°C, the grain refinement was more prominent, the fraction of martensite augmented and the volume of retained austenite was degressive than that at the lower quenching temperature. Figure 8(d) shows that the specimen quenched from 960°C displayed a remarkable refinement and an evenly distributed martensite. Moreover, the volume of martensite increased and the lath width of martensite

Figure 7: The dilatometric curves of BR500HS (heat preservation at 640°C).

Figure 8: Microstructure of the specimens subjected to single quenching treatment at the austenitizing temperature of (a) 900°C, (b) 920°C, (c) 940°C and (d) 960°C.

decreased at 960°C. Thus, the optimal solution temperature was determined to be 960°C.

Based on the above analysis, four CHT tests at the solution temperature of 960°C with four different steps of one cycle, two cycles, three cycles and four cycles were conducted to obtain the optimal cycle step. In order to observe the distribution and morphology of martensite precisely, SEM was employed and the secondary electron images are demonstrated in Figure 9. As the cycle step rose, the amount of retained austenite was degressive and the quantity of martensite increased. To analyze the refinement degree of martensite, the lath width was measured in this section. The microstructures of the specimens that underwent one and two cycles are shown in Figure 9(a and b), where the martensite is dispersive and the remaining austenite is also bulky. The corresponding lath widths of these two specimens were 0.88 and 0.94 μm. Figure 9(c) shows that when the cycle steps increased to three, the quantity of martensite tended to increase, though the martensite remained disordered. The lath width of the specimen decreased to 0.8 μm. Furthermore, after four cyclic treatments (as shown in Figure 9(d)), the lath width of the specimen was measured to be 0.52 μm and the martensite was homogenized.

As the cycle step rose, the volume of martensite increased and the lath width decreased, thus four CHT

cycles are determined to be the optimal choice in these tests. Nevertheless, there were still a large number of remained austenite around the martensite packets. Figure 9(e) shows one grain of the specimen which was subjected to four CHT tests and observed with an SEM in 5,000 times magnification. The laths in a packet were uniform and parallel to each other, which also proved the martensite composition depicted in Figure 2.

4.2 Microstructure analysis of BR1500HS steels subjected to CHT with decreasing temperature and holding time

Figure 10 exhibits the SEM images in 1,000 and 5,000 magnification of the specimen subjected to CHT with decreasing temperature and three cycle steps. Because the optimal solution temperature was determined to be 960°C, the temperature arrangement was designed as 960, 940 and 920°C. In Figure 10, we can see that the specimen subjected to CHT with decreasing temperature shows unobvious refinement and larger distance of martensite laths compared with that subjected to the three-cycle quenching process. The purpose of decreasing-temperature CHT was to restrain the further growth of recrystallization

Figure 9: SEM images of the specimens quenched from 960°C with the cycle steps of (a) one cycle, (b) two cycles, (c) three cycles, (d) four cycles and (e) four cycles, 5,000×.

grains in the premise of sufficient recrystallization. Although the energy demand during the grain refining process in the second and third cycles decreased gradually

due to the increasing internal energy, this test had not clarified such assumption. From the above analysis, we can know that the results may be affected by the large

Figure 10: SEM images of the specimen subjected to CHT with decreasing temperature: (a) 1,000×; (b) 5,000×.

temperature gradient, which decreased to the non-ideal temperature for the martensite generation.

The SEM images of the specimen subjected to CHT tests with decreasing holding time of 5, 3.5 and 2 min and three cycle steps at the temperature of 960°C were observed in 1,000 and 5,000 magnification and are illustrated in Figure 11. In Figure 11(a), we can see that there was nearly no retained austenite and the distance of martensite laths decreased enormously. Moreover, the lath width was measured to be 0.268 μm in Figure 11(b), which proved the ultrafine martensite. This is largely due to the fact that the CHT will reduce the energy demand for recrystallization and the redundant energy will result in the growth of grains. Meanwhile, the CHT tests with descending holding time in austenitizing temperature can provide more reasonable energy for austenitizing. Such phenomenon indicates that the CHT with descending holding time in austenitizing temperature

along with cycle steps is effective in avoiding the grain growth and reducing retained austenite.

4.3 Relationship between hardness and lath width

The results of bulk hardness are shown in Table 2. Solution-treated steel possessed the lowest hardness (169 HV) due to the coarse grain microstructure. As the cycle steps rose from 1 to 4 with the same solution temperature of 960°C and held for 5 min, the hardness of specimen was improved gradually. This can be attributed to the decreasing packet size with increasing cycle steps. Compared with the specimen treated with solution treatment at the solution temperature of 960°C, the hardness of sample with one cycle is 198 HV. However, the hardness of the specimen that underwent the CHT with two cycles was

Figure 11: SEM image of the specimen subjected to CHT with decreasing holding times at temperature of 960°C: (a) 1,000× and (b) 5,000×.

Table 2: Bulk hardness for different heat treatment conditions

CHT conditions	Hardness/HV			Average/HV
	1	2	3	
Solution treatment (ST) at 1,100 for 1 h followed by air cooling	184	167	158	169
ST + holding 5 min at 960°C followed by water cooling for one cycle	166	173	165	168
ST + holding 5 min at 960°C followed by water cooling for two cycles	188	229	226	214
ST + holding 5 min at 960°C followed by water cooling for three cycles	212	222	210	215
ST + holding 5 min at 960°C followed by water cooling for four cycles	245	271	283	266
ST + holding 5 min at descending temperature of 960°C–940°C–920°C followed by water cooling for three cycles	226	237	197	220
ST + holding at 960°C with descending holding time of 5 min–3.5 min–2 min followed by water cooling for three cycles	249	279	240	256

improved to 214 HV and the hardness finally reached its maximum with four cycle steps (266 HV). Thereafter, the hardness of specimen treated under decreasing temperatures with three cycle steps (3-temp, 220 HV) was similar to that of the specimen executed under one single temperature with three cycle steps. However, the hardness of specimen treated with decreasing holding time (3-time, 256 HV) was much higher than that of specimen executed under three cycles with the same solution temperature (cycle 3) and it was approximate to that treated with four CHT tests with one single temperature due to its refined martensite and nearly no retained austenite.

Figure 12 illustrates the bulk hardness and lath width of specimens studied in this article. Figure 12(a) shows that the hardness of specimen subjected to CHT at the temperature of 960°C and the fixed holding time of 5 min increased and its lath width decreased with the increase in cycle step. In Figure 12(b), we can see that with the same cycle step of three, the hardness of specimen subjected to CHT with descending temperature and holding time was higher than that of

specimen subjected to CHT with single temperature and holding time. Meanwhile, as the test proceeded, the lath width was obviously decreased and finally reduced to 0.268 μm , which indicated that the martensite was ultrafine effectively. Subsequently, the negative correlation between hardness and lath width was recognized. In conclusion, the holding time at austenitizing temperature had a remarkable influence on the hardness of specimen during CHT. Furthermore, treatment with decreasing holding time could also provide a better economy and a higher production efficiency.

5 Conclusions

- (1) A series of thermal dilation tests were conducted on a Gleeble 3800 thermal physical simulator. Then, the phase transformation point of complete austenitizing (Ac_3) and the optimal holding time at austenitizing temperature of BR1500HS were identified.

Figure 12: The bulk hardness and lath width of specimen vibration with (a) cycle steps and (b) the same three cycle steps with different temperatures and holding times.

- (2) The tests with various temperatures and cycle steps were conducted and analyzed by OM and SEM, and the lath size of martensite decreased with an increase in cycle steps.
- (3) The CHT tests with decreasing heat temperature and decreasing holding time were designed and executed due to increasing internal energy of steel along with cycle steps. The results illustrated that the specimen treated with decreasing holding temperature (3-Temp) showed a coarser microstructure compared with that treated with one single temperature (cycle 3). Meanwhile, the specimen subjected to treatment with decreasing holding time (3-Time) obtained a ultrafine martensite with the lath width of 0.268 μm .
- (4) The hardness development of the specimens that underwent CHT in this study was measured and compared with the lath width. Then, the negative correlation between hardness and lath width was revealed. The specimen subjected to CHT under three cycle steps with decreasing holding time exhibited higher hardness and more refined martensite than that subjected to CHT under three cycle steps with one single temperature, which justified the effectiveness of the developed preparation process for ultrafine martensite.

Acknowledgments: This work was supported by National Natural Science Foundation (51605035), National Defense Science and Technology Project (JCCPCX201705, 4140-2050202), Qin Xin Talents Cultivation Program of Beijing Information Science & Technology University (QXTCPA-201903, QXTCPB201901), Project of State Administration of Foreign Affairs (G20190201032).

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