Effect of boron and boron-nickel on low-temperature impact toughness of hot-rolled

Nb-added HSLA H-beams

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Nb-treated HSLA steels alloyed with 11ppm of B and 11ppm of B + 0.5mass% of Ni were studied to find an effective method to improve the low-temperature impact properties of HSLA H-beams. The mechanical properties and microstructure of the experimental steels were investigated by uniaxial tensile test, Charpy impact test (V-notch), transmission electron microscopy (TEM) and X-ray diffraction (XRD), respectively. The results indicated that the absorbed energy at -50°C can reach up to 44.4 J from 13 J with the addition of 11ppm of B, and to 121 J with the addition of 11 ppm of B + 0.5 mass% of Ni. The precipitate of Nb carbonitride and the grains can be significantly refined by B addition, and precipitates with diameter up to 30 nm were detected. The $Fe_{23}(C,B)_6$ precipitates were suppressed by the simultaneous addition of B and Nb. No Fe-Ni intermetallic was detected by using XRD method. It was assumed that the B in solid solution state and the homogeneously distributed Ni have beneficial effects on the low-temperature impact properties of Nb-treated HSLA steels.

Key words: boron, nickel, niobium, low-temperature impact toughness, H-beam

INTRODUCTION

H-beams are widely used in various fields of modern society because of their economical features. Currently, there is increasing interest in the study of high-grade hot-rolled H-beams, especially, with advanced low-temperature impact properties, due to the intensive exploration of scientific and energy resources in high-latitude and cold regions.

Presently, the properties of high-strength lowalloy (HSLA) steels are mainly improved by grain refinement [1], as the decrease in grain size is accompanied by an improvement in mechanical properties, as can be represented by the Hall-Petch relation [2]. Thermo-mechanical control processing (TMCP) consisting of controlled rolling [3] provides a powerful method to refine the grains of steels. plastic HSLA Generally, intensive deformation and rapid cooling are required in TMCP to obtain a refined microstructure. However, TMCP is improper to produce H-beams, because rolling parameters of H-beams the are predetermined and not easy to change [4], thus, intensive deformation which is required in control rolling is hard to realize. Moreover, it is very complicated to prevent cracks and distortion in a rapid cooling process, because of its complex cross-section.

The behavior of small amount of B in steels has been elucidated by many researchers. Boron addition can slow down the coarsening of austenite grains during reheating [5], and the segregated B on moving new austenite boundaries can suppress the grain growth and retard the recrystallization of austenite after high- temperature deformation to a significant degree [5-8]. Thus, a fine-grained austenite structure can be obtained. The segregated B in austenite boundaries can suppress the ferrite transformation [9], because the segregated B can occupy the preferential sites of ferrite and reduce the grain boundaries energy [5,10]. However, when B is added alone, coarse precipitates like $Fe_{23}(C,B)_6$ will appear and the positive effects of B addition will be reduced [11,12]. For this reason, the synergistic effects of B and Nb have drawn attention. It is reported that when B is added to Nb-treated steel, the Nb carbonitrides can be refined and the total density of the precipitates increased as compared with steels with only Nb added [13]. Also the formation and coarsening of $Fe_{23}(C,B)_6$ are suppressed by the simultaneous addition of B and Nb [11]. Thus, the segregated B along the γ grain boundaries increases and an enhanced pinning effect and solute drag effect on grain boundaries can be obtained by fine precipitates and solute atoms. Lee et al. [14] have researched the L1₂-type Cu-added zirconium

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trialuminide intermetallics and found that the segregated B in grain boundaries can result in an increase of the grain boundary cohesion by forming strong interatomic bonds, which can sharply increase the ductility and effectively suppress intergranular fracture.

Ni can stabilize in the steel and form substitutional solid solution with ferrite. The comprehensive mechanical properties of the steel can benefit a lot from the addition of Ni. The steel containing 0.5 mass % of Ni is widely used to produce low-temperature pressure vessel steel, which can be used in the range from -40° C to -60° C. However, subsequent heat treatment is generally required, which is impracticable for the production of hot-rolled H-beams.

Based on our previous research [4,15-18], the objective of this article was to characterize the effects of B and Ni on the low-temperature impact toughness of hot-rolled Nb-treated HSLA H-beams. Particular attention was paid to link the B addition to the refinement of precipitates and microstructure. Also, the Ni species in the steels was investigated by using XRD method.

MATERIALS AND EXPERIMENTAL PROCEDURE

The investigation involved three different steels, a carbon-manganese structural steel conforming to GB/T 1591-2008, grade Q345E was selected as the reference material. In order to find out the effects of B and of the combined addition of B and Ni, the other two steels were designed to be of the same composition as the reference steel alloyed with 11 ppm of B and 11 ppm of B + 0.5 mass% of Ni, respectively. The chemical composition of the steels is listed in Table 1. All steels were prepared by convert steelmaking process. The H-beam blanks were firstly reheated at 1250°C for 1 h and rolled in universal H-beam rolling mill with a final rolling temperature of 910°C, then air-cooled to room temperature.

Tensile tests were performed using a universal testing machine at room temperature. The standard

Charpy V-notch impact test was carried out over the temperature range from -10°C to -70°C. V-notch Standard Charpy specimens of 10mm×10mm cross-section were machined with the longitudinal direction parallel to the rolling direction of the H-beams, and the V-notch was machined through the thickness direction perpendicular to the rolling plane.

All samples for metallographic observation were mechanically polished and etched with a 4% nital solution. The microstructure was characterized using a Su-70 thermal field emission scanning electron microscope (FE-SEM).

The transmission electron microscopy (TEM) investigations were carried out on an H-800 microscope with X-ray energy dispersive spectroscope (XEDS) operating at an accelerating voltage of 150kV. High resolution TEM was also used to observe the very fine particles in the steels. The X-ray diffraction analyses of the mechanically polished samples were performed on а DMAX-2500 X-ray diffractometer using Cu Ka radiation, operating at V=50 kV, I=40 mA. A scanning rate of 0.02°/s was applied to record the patterns in the 2θ range from 10° to 100° .

RESULTS AND DISCUSSION

Mechanical Properties

Table 2 shows the mechanical properties of the experimental steels. It can be seen from Table 2 that all steels exhibit good comprehensive mechanical properties. It can also be seen that the yield strength of the experimental steels increases from 385 MPa to 420 MPa with the addition of 11ppm of B, and to 440 MPa with the addition of 11ppm of B + 0.5% mass of Ni. This may due to the grain refinement effects of the B addition and the solid solution strengthening effects of Ni addition. No changes of tensile strength and elongation the experimental steels were registered.

Figure 1 shows the variations of absorbed energy as a function of test temperature for different experimental steels.

Steels	С	Si	Mn	Р	S	Ni	Nb	В
1#	0.18	0.24	1.26	0.012	0.005	0.01	0.024	_
2#	0.18	0.23	1.31	0.013	0.004	0.01	0.023	0.0011
3#	0.18	0.21	1.32	0.012	0.005	0.51	0.020	0.0011
Table 2. Mechanical properties of experimental steels								
Steels		Yield strength/MPa		MPa	Tensile strength/MPa		Elongation/%	
1#		385			530		30.0	
	2#		420		530		29.0	
3#		440			570		29.5	

 Table 1. Chemical composition of the experimental materials (mass %)



Fig. 1. Charpy V-notch absorbed energy *versus* temperature

As can be seen, the absorbed energy of 1# steel at -10 ℃ reaches 102.3 J, but a sharp ductile-to-brittle transition was observed as the temperature decreased, and absorbed energy is 15J when the temperature drops to -40°C. In fact, the average absorbed energy of H-beams for preventing brittle fracture at -40 °C specified in GB/T 1591-2008 is 34 J. Compared to 1# steel, 2# steel shows better impact toughness with decreasing temperature down to -50° C, and when the absorbed energy is at 34 J the temperature decreases by 20° C. It is suggested that the addition of trace B strongly enhances the impact toughness of the steels, especially the low temperature impact toughness. 3# steel displays the highest toughness among the experimental steels, more precisely, the absorbed energy of 3# steel at -10°C is 190.2 J, and at -50°C it still maintains most of its impact toughness with the absorbed energy of 121 J, even at -60°C the absorbed energy of 3# steel remains at 52.4 J. Compared to 1# and 2# steels, the temperature at absorbed energy of 34J decreases by 30°C and 10°C, respectively. The fracture surfaces of the experimental steels at -40° C were also analyzed by scanning electron microscopy, utilizing a Hitachi Su-70 FE-SEM. The area of the fracture surface towards the middle of the width of the specimen and a few millimeters below the notch was the main area of investigation. The fracture surfaces are shown in Figure 2. We can see that the fracture mode of 1# steel at -40 °C was entirely brittle fracture, and the fracture surface mainly contained trans-granular cleavage-like fracture zones. The fracture surface of 2# steel consisted mainly of shallow microvoids with some regions of larger dimples. The fracture surface appearance of the 3# steel tested at -40°C was ductile and consisted of large dimples.

Microstructure observation and XRD results

To reveal the effects of B and Ni, the microstructure and precipitation characteristics were investigated. Figure 3 shows the SEM graphs

of the experimental steels. It can be seen that the microstructures of all three alloys consist of ferrite and perlite. While the average grain diameter of 1# steel was the largest one (9.66 μ m), followed by 2# steel (8.17 μ m), it decreased to 6.05 μ m in 3# steel. As all three experimental steels were produced with the same process, it indicates that the B addition to Nb-added HSLA steels has a beneficial effect on grain refinement, and this effect seems to be enhanced by the combined addition of B and Ni.



Fig. 2. Fracture surfaces of experimental steels tested at -40 $^{\circ}$ C. The CVN impact energy is shown for reference. (a) 1# steel; (b) 2# steel; (c) 3# steel.

Absorbed Energy (J)

The TEM graphs (Figure 4) of extraction replica for the experimental steels reveal that the precipitates can be significantly refined by the simultaneous addition of B and Nb. The total density of the precipitates of 2# and 3# steels increased as compared with that in 1# steel (only Nb-added).



Fig. 3. SEM graphs of the experimental steels. The average grain diameters are shown for reference. (a) 1# steel; (b) 2# steel; (c) 3# steel.

Thus, the grain boundaries pinning effects in recrystallization and the phase transition process can be enhanced by the fine precipitates, and finally a finer grain structure can be obtained.

The precipitates were identified by EDS and TEM methods (Figure 5). Considering the Nb-containing precipitates, only Nb(C,N) and Nb(C,N) were detected. No Nb(C,B) particles, which were reported in many studies [7,12,13], were detected. This may due to the very fine particles of Nb(C,B) and the incapability of the equipment used in this study. And no coarse

 $Fe_{23}(C,B)_6$ particles which were reported in B-containing steels [7,11,19] were detected. It indicates that the simultaneous addition of B and Nb can suppress the precipitation of $Fe_{23}(C,B)_6$, which consumes a large amount of B and has adverse effects on fulfilling the potential of B. Based on the characterization results of the particles in the steels, we can conclude that the existence form of B in the steels is mainly solid solution status. It can segregate in the moving new boundaries during recrystallization and phase transition process, and produce solution dragging effects to decrease the motion of the boundaries. Thus, a fine microstructure can be obtained. Moreover, the boundary cohesion is supposed to be enhanced by the segregated boron as reported in other studies [14].







Fig. 4. TEM images of extraction replica of experimental steels. (a) 1# steel; (b) 2# steel; (c) 3# steel



Fig. 5. Selected area diffraction pattern and the key diagram of Nb(C,N) and Nb(C,N). a-SDP of Nb(C, N); b-SDP of NbC $\$



Fig. 6. XRD patterns of 2# and 3# steel



Fig. 7. The distribution of Ni in 3# steel

To reveal the effects of Ni in this study, the existence form of Ni was observed using XRD and the distribution of Ni was investigated using EDS, the results are shown in Figure 6 and Figure 7. The

XRD results of 2# steel and 3# steel, showed that only α -Fe diffraction peaks were detected. It indicates that the effects of 0.5 mass% of Ni on improving low-temperature impact toughness are not due to the formation of Fe-Ni solid solutions which are considered as a main reason for Ni to enhance the toughness of steels. Figure 8 shows that Ni is distributed homogeneously in 3# steel. However, the interaction of B and Ni needs further investigation in the following studies.

CONCLUSIONS

The effects of B and of the simultaneous addition of B with Ni on the low-temperature impact toughness of industrial Nb-added HSLA H-beams were investigated, and the following conclusions can be derived:

(1) Compared to the single addition of Nb, finer grain structure can be obtained by the simultaneous addition of B and Nb under the same production conditions. The microstructure can be further refined when 0.5 mass% of Ni was added to the Nb, B-added steels. This can be attributed to the grain boundaries pinning effects by the fine precipitates which are obtained by the simultaneous addition of B and Nb and the solute dragging effects of boundary segregated B and homogeneous distributed Ni atoms.

(2) Besides the pinning effects and the solute dragging effects of boundary segregated boron, the boundary cohesion is supposed to be increased by the segregated boron.

(3) No Fe-Ni solid solution was detected in this study. The effects of Ni are supposed to be pinning effects of the homogeneous distributed Ni atoms.

(4) Practical manufacture shows that it is an economical method to improve the low-temperature impact toughness by adding 11 ppm of B and 0.5 mass% of Ni to the investigated H-beams. The production process does not need to be adjusted compared to the steels with only Nb-added.

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ВЛИЯНИЕ НА БОР И БОР-НИКЕЛ ВЪРХУ ЯКОСТТА НА УДАР НА ГОРЕЩО ВАЛЦУВАНИ НИСКО ЛЕГИРАНИ ГРЕДИ С ВИСОКА ЯКОСТ, СЪДЪРЖАЩИ ДОБАВЕН НИОБИЙ

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(Резюме)

Nb-съдържащи ниско легирани стомани са сплавени с 11 ppm B и 11 ppm B + 0.5 мас.% Ni и са изследвани с оглед намиране на ефективен метод за подобряване на нискотемпературните якостни свойства на ниско легирани греди с висока якост. Механичните свойства и микроструктурата на стоманите са изследвани съответно чрез едноосов тест на опън, Charpy ударен тест с V-бразда, TEM и XRD. Установено е, че абсорбираната енергия при -50°C от 13 J може да достигне 44.4 J с добавка от 11 ppm B и 121 J с добавка от 11 ppm B + 0.5 мас. % Ni. Утайката от ниобиев карбонитрид става значително по-фина с добавка на бор – измерени са зърна с диаметър до 30 nm. Утаяването на Fe₂₃(C,B)₆ се подтиска от едновременното добавяне на бор и ниобий. Не е установено наличие на твърди разтвори от Fe-Ni чрез XRD. Направено е предположение, че борът в твърдия разтвор и хомогенно разпределеният никел имат благоприятно влияние върху нискотемпературната якост на удар на Nb –съдържащи ниско легирани стомани.