Effect of thermal deformation and Nb element action on the organization and performance of steel

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Abstract

The influences of Nb element and hot deformation on the strength of steels were systematically investigated. The microstructures and mechanical properties of the steels are determined through optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and tensile tests. The nanometer sized carbide particles precipitated in the grain boundary are attributed mainly to severe deformation at high temperature. Of the two steels with different content of Nb element and deformation, the strength of 2# steel is more lager due to more content of Nb can form more precipitates to limit grains growth. Furthermore, the results also shown that adding Nb can refine the microstructure of the steel and effectively improve the mechanical properties, but only thermal deformation has no obvious effect on the improvement of the properties of the steel.

1. Introduction

In recent years, the high strength low alloy steels (HSLA) have attracted significant interest in applications with excellent formability for the bridge and building industries [1–3]. Refining the pearlite interlamellar spacing and grain size of steels can effectively improve the mechanical properties and elongation rate of steel [4–6]. In engineering structure, HSLA steels are the key point that ensures structural safety of earthquake-resistant frames during major earthquake [7]. For the HSLA steels, the precipitation of microalloying carbides contributed is of significant interest. Therefore, it is very important to consider the alloy design and thermomechanical processing parameters. The strength of HSLA steels can be enhanced by nanoscale precipitates, Which is consist of the microalloying elements such as NbC, VC or (Ti,Nb)C [8–10]. Improve the HSLA steels yield strength, it is often accompanied by decreased plasticity. It is very necessary to develop steels with both high strength and good toughness to meet it safety and reliable requirements. At present, China mainly uses V microalloy to improve the HSLA steels performace, high cost, which is not conducive to microalloy extensive promotion and used of high-strength reinforcement. Through Nb, the reinforcement and fine crystal in microalloy strengthening the effect and improving the yield strength of the HSLA steels has an important theoretical research value and a practical application strength. Hyzak et al. [11] studied the relationships between the microstructures and the mechanical properties of pearlite. The results shown that refining the interlamellar spacing of pearlite and grain size effectively enhances the strength and fracture toughness of the steel. The microstructure control of low temperature ultra-low carbon steel has a good effect on the mechanical properties [12–15]. Adding Nb element content is the most efficient way to develop HSLA steels with microstructure [16–19]. Microalloying elements are usually utilized singly or in combination to control grain-size and provide precipitation-hardening [20].

In this study, we compare and analyze the different tensile intensities caused by the different Nb content in the steels and precipitation after heavy hot deformation. And then the precipitation is examined in the terms of size, morphology and crystallography. Simultaneously, analyzing the strengthening mechanisms of precipitates. The refinement degree of microstructure after the hot deformation and the influence of refined microstructure on mechanical properties are studied in order to study the improvement of properties of steels after adding Nb. Nb content contributes to the increase of yield strength and elastic energy of the steels. The current research is
helpful to add Nb element to the steels to improve its mechanical properties, which is conducive to the low-cost large-scale production of seismic reinforcement.

2. Experimental materials and procedures

The two steels with the chemical compositions of 0.24C-0.61Si-1.54Mn-0.2Nb-0.21 V (wt%) and 0.25C-0.6Si-1.54Mn-0.32Nb-0.2 V (wt%) respectively, then the steels were smelted by 50 kg intermediate frequency induction furnace. And then hot forged into a size of 500 mm (L) × 30 mm (W) × 30 mm (H) plate. The specimen was cut into a block sample with a size of 100 mm (L) × 30 mm (W) × 18 mm (H). The pieces of steels were process according to figure 1. According to the effect of Nb on pearlite transformation, the carbon content of 0.2% low-carbon steel pearlite is easier to generate at 650 °C. Therefore, at 650 °C insulation for 30 s, not only conducive to the generation of pearlite, but also prevents the long insulation time caused by grain growth. The temperature range of recrystallization is calculated base on the following equations:

\[
778 + 464C + (1774Nb - 738\sqrt{Nb}) + (732V + 230\sqrt{V})
\]

Where 'C', 'Nb' and 'V' are the mass fraction of corresponding elements in steel (wt.%). The temperature of possible recrystallization is calculated as 950 °C–1100 °C, at which the grain size of the steels can be further refined by the rolling method. Rolling in the non-recrystallization region of 850 °C–950 °C allows dislocation and stress release, reduce stress concentration and avoid crack production. The steels can be hardened to improve its strength. The deformed specimens were cooled to room temperature at a rate of 30 °C min⁻¹, as shown in figure 1, and the chemical compositions of the steels are listed in table 1. These 1 # steel samples were hereafter labeled as 1 # steelD5, and 1 # steelD4, respectively. And then 2 # steel samples were hereafter labeled as 2 # steelD5 and 2 # steelD4, respectively.

The steels samples were polished and then etched with a solution of 4% Nital, and then mainly characterized by scanning electron microscopy (SEM), optical microscopy (OM), transmission electron microscopy (TEM). Tensile test were performed as per the Chinese code (GB/T228-2002). Figure 2 shows that the dimension of standardized steel bar sample for tensile tests.

| Samples | C    | Si   | Mn   | P    | Ti   | N    | V    | Nb   | Fe   |
|---------|------|------|------|------|------|------|------|------|------|
| 1 # steel | 0.240 | 0.610 | 1.540 | 0.029 | 0.08 | 0.007 | 0.210 | 0.200 | Balance |
| 2 # steel | 0.250 | 0.600 | 1.540 | 0.027 | 0.08 | 0.008 | 0.200 | 0.320 | Balance |

Figure 1. Schedule diagram of deformation and thermal treatment.
3. Results and discussion

3.1. Effect of Nb element and deformation on ferrite and pearlite microstructures

The optical microstructure (OM) image of 1\textsuperscript{st} steel and 2\textsuperscript{nd} steel are shown in figure 3. All the micrographs reveal ferrite and pearlite microstructure with grain boundary (GB). Dotted lines have been used to increase the visibility of the GB. The pearlite is a hierarchical structure, which is composed of several colonies into one prior austenite grain. The hierarchical structures of pearlite is consists of alternating layers of ferrite and cementite. Figures 3(a), (b) shows optical micrograph of 1\textsuperscript{st} steel\textsubscript{D5} and 1\textsuperscript{st} steel\textsubscript{D4}, respectively. Figures 3(c), (d) shows optical micrograph of 2\textsuperscript{nd} steel\textsubscript{D5} and 2\textsuperscript{nd} steel\textsubscript{D4}, respectively. From these OM micrographs (figure 3), the white-etched phase is ferrite, which nucleates in the isothermal region of ferrite. The dark-etched phase is pearlite, which is nucleated in the remaining austenite. After deformations of two steels, a two-phase structure composed of ferrite and pearlite was detected in the figure 3. However, the microstructures of two steels cannot be distinguished at the limited resolution of OM. Since Nb element, a strong strengthen element, has the ability to
refine the grains. The deformations result in the significant refinement of ferrite and pearlite microstructures in coarse grains.

The typical SEM micrographs of the uniform deformation at the cooling rates of 0.5 °C s⁻¹ are presented in figure 4. In the process of slow cooling, Nb element carbide will precipitate at the grain boundary. From these SEM micrographs (figure 4), it can be detected that the spheroid pearlite microstructure is composed of sphere-like cementite particles within a ferrite microstructure. All the SEM micrographs (figure 4) reveal two types of phases, the main phase is pearlite of brighter contrast and the other one is ferrite of darker contrast along the grain boundaries. Thus, the long shape microstructure of the pearlite mass will change into spherical pearlite. The reason is that the driving force behind the long shape of spherical pearlite will reduce the surface free energy [21], the spherical pearlite process reduces the interface area between the ferrite and cementite phase, and the further reduces the surface free energy through the isothermal holding process.

At the same cooling rate of two steels, and then the sizes of ferrite gradually decrease with more times of deformation. Hence, the sizes of 1# steelD5 are smaller than 1# steelD4, and the tensile strength is larger. Previous studies have shown that the pearlite strength and interlamellar spacing conform to Hall-Petch relationship. In the present study, the precipitation of NbC or (Ti,Nb)C in the boundary of ferrite may refinement sizes of grains and the interlamellar spacing of pearlite. The ferrite as a soft phase, and plastic deformation is associated with the dislocation multiplication and slip. In addition, dislocation move freely in the ferrite zone and aggregate along the interlamellar spacing of pearlite, and then resulting in greater deformation until fracture. Hence, partial dislocation is difficult when the interlamellar spacing of pearlite is smaller, therefore, the tensile strength is larger. According to the statistical results of the Nano Measurer, the interlamellar spacing of pearlite of steels is S₁(0.182 μm), S₂(0.191 μm), S₃(0.124 μm) and S₄(0.14 μm), respectively. The interlamellar spacing of pearlite of 2# steelD5 steel is minimum. Since the steels is performed during the hot rolling process, as the Nb content increases, the Carbon-nitride of Nb are precipitated on the austenite boundary, dislocation and defects, pinning the austenite boundary, which hinders the austenite growth, refines the interlamellar spacing of pearlite during the phase transition. The same content of Nb element, Compared with the coarse-grained 1# steelD4 (figure 4(b)) and 2# steelD4 (figure 4(d)), much finer lamellae were obtained for the fine-grained 1# steelD5 (figure 4(a)) and 2# steelD5 (figure 4(c)), respectively. Also, the content of Nb elements and the times of deformation led to the refinement the lamellae. The micro-strain is caused by the residual stress caused by the reduction of pearlite interlamellar spacing. It must be mentioned that interfaces between the ferrite and cementite have two strain

Figure 4. SEM micrographs obtained for (a) 1# steelD5, (b) 1# steelD4, (c) 2# steelD5, (d) 2# steelD4.
areas. When the interlamellar spacing of pearlite is large, the overlap region between the areas is too smaller to the ferrite hardening is unsaturated. When the interlamellar spacing of pearlite is small, the two strain regions overlap, and the ferrite hardening reaches saturation, leading to the easy formation of micro-cracks between the ferrite and the cementite interface. The progressive decreases in the grain size and the interlamellar spacing of pearlite is observed for Nb-added steels (1# steel and 2# steel), which may be related to the effect of the multiple deformation. It was suggested that the refinement of grain sizes and interlamellar spacing can increase tensile strength.

3.2. Mechanical properties

Figure 5 shows that the variation of engineering stress and the engineering strain of the 1# steelD5, 1# steelD4, 2# steelD5 and 2# steelD4, respectively. The engineering stress-strain curves exhibits very similar shape features, and the strength peak appears at the strain of about 11%. The 1# steelD4 and 2# steelD4 shows a tensile strength of about 786 MPa and 968 MPa, respectively, and then while only a minor increased in strength (about 40 MPa) was obtained for the refinement 1# steelD5 and 2# steelD5, respectively.

In the present study, the same cooling rate of 1# steelD5 and 2# steelD5 and it has been observed that the tensile strength differs for the different Nb contents addition. It can be hypothesized that more Nb contents added in the steels (1# steel and 2# steel), the interlamellar spacing of pearlite and the grain size are finer. It is well known that the element of Nb being a strong carbide influences the diffusion of carbon, which could suppresses the thickness of cementite and leading to thinner cementite. Enhancement of the tensile strength of the steels were mainly due to the formation of small grains sizes during rolling. It is well known that deformation of steel at high temperature can improve the mechanical properties, which is that high temperature deformation can refine the grain size and provide more nucleation points for pearlite transformation. The tensile strength of steel is closely related to the size of ferrite and the interlamellar spacing of pearlite.

Figure 6 shows the relationship between tensile strength and the ductility of steels (1# steel and 2# steel). The change tendency of the engineering stress is obviously the opposite as that engineering strain. With 2# steels increased the engineering stress, engineering strain is reduced by 2%. It can also be noticed that the tensile strength of 2# steels with less ductility reduction. It was observed that the tensile strength of 2# steels are improved but the ductility did not decrease significantly. The main reason is that the increase of Nb elements can make the grain size of steel widely refined. It must be mentioned that the interlamellar spacing of pearlite and the grain refinement are a general strategy to enhance the strength of steels without much sacrificing their ductility. It is to be noted that the grain sizes of 1# steel is larger than 2# steel, but the ductility is greater than 2# steel. The reason is that the large size ferrites grains can retaining the ductility. Therefore, it can be concluded that good combinations of ductility and strength are obtained by 2# steelD5.

After tensile testing, the fracture surface were examined under SEM, as illustrated in figures 7(a)–(d). Deeper dimples and shallow dimples rupture, can clearly be observed on the fractured surface of the steels. Compared with the 1# steels (figures 7(a), (b)), the 2# steels undergo a substantial shrinkage with rougher at the central
These evidence that 1# steel may carry much higher plastic strain than 2# steel. The fracture micrographs at the 2# steel show cleavage facets with numbers of secondary cracks. And the dimples of the 2# steel are very deep, it can be described that the strength of 2# steel is increased. Bright gleaming fractured surface of the 2# steel clearly indicates the increasing in the brittleness. It can be correlated with the least amount of ductility of the 2# steel. It is clearly noticed that with Nb elements of the 2# steel is increased, the size of the dimple marks increased, and then the strength increased. In conclusion, the size of ferrite and the interlamellar spacing of pearlite increase, and then the strength gradually increase with the same deformation.

Figure 6. The relationship between engineering stress and engineering strain.

Figure 7. Fractured surface morphology of tensile: (a) 1# steel15, (b) 1# steel14, (c) 2# steel15, (d) 2# steel14.
3.3. Analysis of precipitations

The typical TEM micrographs of the precipitation after uniform deformation are presented in (figures 8, 9). Mechanical behavior of thermo-mechanical controlled rolled 1\# steels and 2\# steels were investigated by tensile tests. The precipitates of steels are spherical or ellipsoidal in shape. According to the two-dimensional fast Fourier transformation, In figure 8 are identified as (Ti, Nb)C carbides with a NaCl-type crystal structure. It can be found that the lattice parameter of 1\# steels (1\# steel\textsubscript{D5}, 1\# steel\textsubscript{D4}) and 2\# steels (2\# steel\textsubscript{D5}, 2\# steel\textsubscript{D4}) are 0.432 nm and 0.447 nm, respectively. For EDS analysis of the precipitate of 1\# steel\textsubscript{D5} and 1\# steel\textsubscript{D4}, as shown in

Figure 8. TEM micrographs and corresponding diffraction patterns images of the nanoscale carbides in 1\# steel (a)-(c) 1\# steel\textsubscript{D5}, (d)-(f) 1\# steel\textsubscript{D4}.

Figure 9. TEM micrographs and corresponding diffraction patterns images of the nanoscale carbides in 2\# steel (a)-(c) 2\# steel\textsubscript{D5}, (d)-(f) 2\# steel\textsubscript{D4}.
figures 8((c),(f)), the TEM (figures 8(a), (d)) indicates that the main precipitate is (Ti, Nb)C. It is well known that Ti and Nb are interchangeable in (Ti, Nb)C carbide lattice that can lead to a small change in lattice parameter compared with Ti and Nb. Figures 8((a), (d)) shows the precipitate of (Ti, Nb)C, which is due to the low content of Nb, and Ti can replace the Nb absent in the carbide. The previous studies reported that the lattice parameter of (Ti, Nb)C carbide is about 0.44 nm for precipitates of composition (60% Nb and 40% Ti). The existence of Ti elements in carbides of Nb elements can reduce the parameter of the NbC. Therefore, the nanoscale carbides (Ti, Nb)C and NbC can be found in 1steels (figures 8(a), (d)) and 2steels (figures 9(a), (d)), respectively. As described above, the Ti atoms are involved in the precipitation of NbC. And the lattice parameter of carbide will be smaller than the theoretical lattice parameter of NbC and larger than the theoretical lattice parameter of Ti, which may reduce the chemical interface energy of (Ti, Nb) C carbide. Consequently, this effect will cause the decrease of nucleation energy barrier. And reducing the content of Nb will enhance the nucleation of (Ti, Nb)C carbide during the early stages of precipitation. Based on the EDS (figures 9(c), (f)) results, the precipitate in 2steels mainly contain Nb, C and Fe as the main peak, indicating that the precipitate in steels are mainly NbC, and the shape is circular, with an average grain size of about 50 nm, and it is precipitated at the ferrite boundary, electron diffraction pattern of the precipitate (figures 9(b), (e)) analysis shows that these precipitates have a FCC crystal structure. Figures 9(d) and (e) shows the orientation relationships between the NbC and the ferrite matrix for the thermomechanical processing schedules revealed that some fine precipitates exhibited the Baker-Nutting orientation relationship (001)_{NbC}//(000)_α, [010]_{NbC}//[110]_α (figure 9(b)), which indicates they precipitated in ferrite, whereas others did not show these orientations (figure 9(e)). It was observed that the increased Nb content of 2steels (figure 9) formed more precipitation at the grain boundary, and the grain size of steel can be efficient refine, and the mechanical properties are improved. When the steel is pulled, due to the soft ferrite, plastic deformation occurs first, and the surrounding pearlite still maintains elastic deformation. Smaller interlamellar spacing of pearlite and refinement ferrite provide more space for dislocation accumulation, so dislocation accumulation is gradually replaced by confined layer slip. Through the comparison between the two steels, it can be found that the improvement of mechanical properties is mainly provided by microstructure refinement, rather than single high-temperature deformation, i.e., (1steel_1D3, 1steel_1D4) or (2steel_1D3, 2steel_1D4) with the same content of Nb, but the increase of Nb content can effectively improve the two steels strength.

4. Conclusions

(1) Severe deformation of the steel can reduce ferrite grain size effectively, and then enhance the strength of two steels (1steel and 2steel).

(2) The microtissue of 1steel and 2steel are ferrite and pearlite, with the content of Nb increasing, ferrite grain size and the interlamellar spacing of pearlite are refinement. Increasing the content of Nb element can effectively improve the two steels strength, and result in numerous randomly dispersed precipitates of NbC carbide particles in the grain boundary matrix. And then the tensile strength of 2steel is 20% higher than 1steel.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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