Microstructural evolution and mechanical properties of a low alloy high strength Ni-Cr-Mo-V steel during heat treatment process

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Abstract. In order to obtain an optimal heat treatment for a low alloy high strength Ni-Cr-Mo-V steel, the microstructural evolution and mechanical properties of the material were studied. For this purpose, a series of quenching and temper experiments were carried out. The results showed that the effects of tempering temperature, time, original microstructure on the microstructural evolution and final properties were significant. The martensite can be completely transformed into the tempered lath structure. The width and length of the lath became wider and shorter, respectively with increasing temperature and time. The amount and size of the precipitates increased with temperature and time. The yield strength (YS), ultimate tensile strength (UTS) and hardness decreased with temperature and time, but the reduction in area (Z), elongation (E) and impact toughness displayed an opposite trend, which was related to the morphological evolution of the lath tempered structure.

1. Introduction

Recently, with the rapid development of ship building, drilling platform of offshore oil & gas, and aerospace manufacture, the low alloy high strength Ni-Cr-Mo-V steels have been used widely due to their promising mechanical properties [1]. In these fields, many components have to be possessed with proper microstructures, excellently mechanical properties and good dimensional stability [2]. In order to satisfy these requirements, the materials usually have to be subjected to a series of heat treatments. It is well-known that the microstructure takes a complex evolution during the processes. Moreover, the microstructural evolution directly affects the mechanical properties. Therefore, it is necessary to investigate the effects of heat treatment parameters on the microstructural evolution and mechanical properties.

In the past years, many authors have paid attention on this subject. Tao et al [3] investigated the effect of cooling method on the microstructural characteristics and mechanical properties of a as-cast high-strength low-alloy steel. Peng et al[4] studied the microstructural evolution and strength of 9Cr-3W-3Co steel under different aging conditions by experiments. Shin et al [5] investigated the microstructural evolution and the tensile behavior of precipitation hardened martensitic steel after aging treatment. Maji et al [6] carried out some experiments to ascertain the effect of cooling rate on microstructure and mechanical properties of an eutectoid steel subjected to a heat treatment. Although these studies investigated the microstructural evolution and mechanical properties of steels, some conclusions, obtained from these studies, may not be valid for the present material. Therefore, it is necessary to investigate the microstructural evolution and its effects on the mechanical properties of the Ni-Cr-Mo-V alloy during heat treatments.
2. Material and experimental procedure

A 10Ni5CrMoV alloy was used in this study. The chemical composition of the steel is listed in Table 1. Its initial microstructure was tempered martensite and the grain number was 8.5, as shown in Fig.1(a). The material was subjected to quenching and tempering treatments. This procedure has been showed in Fig.1(b).

The heat-treated material was subsequently subjected to a standard metallographic grinding, polishing and etching (by a solution of 97% alcohol and 3% nitric acid). The material was shot in the center zones by an optical microscope (Axiovert 200 MAT) with magnifications from 50 to 1000 times. Three to five micrographs at each condition were used to determine the grain size, area percentage and lath width (ASTME112-10).

The hardness, tensile and impact tests were performed. A Vicker’s hardness testing machine (Tukon 2100B) using 3-kgf load was used. The standard tensile specimens were prepared and conducted on a tensile testing equipment (CSS-44300) with a load of 10 kN and loading strain rate of 10^-3 s^-1. The Charpy V-notch impact specimens with 10 mm × 10 mm × 55 mm size were prepared according to GB/T229-2007 [10]. The impact specimens were performed at temperatures of -120°C and -80°C on an impact equipment with a capacity of 500 J.

![Material and experimental procedure](image)

(a). The initial microstructure  
(b) heat treatment procedure

3. Results and discussions

3.1 Microstructural evolution during quenching

The material was subjected to a quenching (including water-quenched, oil-quenched and air cooled) and tempering process. During the process, the microstructure had a complex evolution, as shown in Fig.2. It can be seen that a complete lamellar martensitic structure can be obtained through the water-quenched treatment (as shown in Fig.2(a)). For the lamellar structure, the length and width were very sensitive to the cooling rate. The sizes of primary lamellar structure were larger than that of the secondary structure, because the former had more time to grow than the latter. When the material was subjected to the oil quenched, the original structure was replaced by a large amount of lath martensite and a little of upper bainite, as shown in Fig.2(b). The sizes of the structure were obviously larger than those of lamellar

![Table 1. The chemical composition of the material](image)
structure Fig.2(c) showed the optical micrograph of the material subjected to air cooled. The final microstructure comprised a little martensite and much bainite.

3.2 Microstructural evolution during tempering

After the water-quenched treatment, the material was subjected to a tempering treatment. The tempering temperatures were 600°C, 620°C, 640°C and 660°C and the time was from 1 to 8 hours. As shown in Fig.3 (at 600°C), a tempered structure can be obtained. Clearly, the tempered structure gradually became coarsening with the increasing time. According to Dong’s results [7], this phenomenon can be attributed to the dislocation annihilation and recombination of two subgrain boundaries. At 620°C, as shown in Fig.4, the size of the tempered structure was larger than that at 600°C. When the tempered temperature increased to 640°C, as shown in Fig.5, a large amount of carbide precipitates can be observed in the microstructure at 3 hours. The needle-like precipitates were much tiny (less than 0.1 um). The size and amount of the precipitates increased with the time. The tiny needle-like structure gradually developed into the short rod-like or equiaxed. The length of the tempered lath decreased while its width increased with the temperature and time, which resulted in the decreasing of length-width aspect ratio of the lath. This phenomenon has been explained by Guimarães et al [8]. They believed that the aspect ratio was proportional to the elastic strain energy of the lath. With the increasing of temperature and time, the elastic strain energy was gradually relaxed by the plastic deformation within and the transformation of self-accommodating groups of martensite units. When material was tempered at 660°C, as shown in Fig.6, a large number of precipitates were formed in the tempered lath since 2 hours.
Figure 3. The microstructural evolution of the material tempered at 600°C for different time

Figure 4. The microstructural evolution of the material tempered at 620°C for different time.

Figure 5. The microstructural evolution of the material tempered at 640°C for different time.
3.3 Mechanical properties

3.3.1 Hardness. The variation of hardness value of the material with the tempering temperature and time was shown in Fig. 7. It can be seen that the value was sensitive to the time. A general trend was that the value decreased with the increasing tempering temperature and time. At 600°C, the value was always larger than the original value (310 HV), but the hardness value became smaller than the original value at 660°C. This indicated that it was not proper for the material to be tempered at these temperatures. At 620°C and 640°C for holding 2-3 hours, the hardness value (located in the red dotted rectangle) was close to the original value of the material. Thus, the proper tempering conditions for the material may be at 620-640°C for holding 2-3 hours.

Figure 6. The microstructural evolution of the material tempered at 660°C for different time.

3.3.2 Tensile properties. The variation of the ultimate tensile strength (UTS) and yielded strength (YS) with the tempering time was displayed in Fig. 8(a). Clearly, both the YS and UTS decreased with the time. Zhang et al.’s [9] study had shown that the martensitic lath was the key factor controlling the strength. The martensitic lath width and dislocation density decreased with the time, which led to the decrease of strength. In addition, the solution strengthening effect became weaker with time. However, the precipitation strengthening effect was minor. Therefore, the strength of the material decreased with the time.

Figure 7. The variation of hardness of the material at 620°C with tempering time.
Fig. 8(b) showed the variation of reduction of area (Z) and elongation (E) with time. It can be seen that both of the Z and E increased with the time. It has been reported that the decrease of ductility was attributed to the decrease of aspect ratio of martensite lath [10]. During the tempering process, the aspect ratio of the tempered lath decreased with the time. Therefore, the values of Z and E increased with the holding time.

3.3.3 Impact toughness. The variation of impact toughness of the material with time was showed in Fig. 10. It can be seen that the impact toughness increased with the time. Two reasons can explain this phenomenon. The first was the softening of the base metal due to the precipitation of interstitial atoms [11]. The second was the change of crystallographic orientation and crack direction. Therefore, in order to ensure a good combination of strength and plasticity, the temper condition should be selected as 620°C + 2~3 hours.

4. Summary
The width and length of the tempering lath became wider and shorter with the temperature and time. The amount and size of carbide precipitates increased with the temperature and time. The beginning time of the precipitation was shorter for a martensite microstructure a mixed microstructure. The yielded strength, ultimate tensile strength and hardness decreased with the tempering temperature and time, while the reduction of area, elongation and impact toughness increased with the temperature and time. The variation of these properties was closely related to the variation of lath width and length. The optimal quenching and tempering treatment was obtained. The material was subjected to the water quenched and then was tempered at 620°C for 2-3 hours, which can make sure a good combination of strength and toughness.

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