Effect of cryogenic-tempering treatment on the performance of high ni gear steel

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Abstract. Effects of cryogenic-tempering hybrid heat treatment on the microstructure and mechanical properties of 18Cr2Ni4W gear steel after carburizing-quenching were investigated. The performances of the fully carburized and non-carburized samples were compared and analysed. The results show that the effect of cryogenic treatment on the microstructure and mechanical properties of 18Cr2Ni4W steel without carburization is negligible. The carburizing layer with high carbon content results in a large amount of residual austenite after the direct quenching to ambient temperature. After cryogenic-tempering, the microstructure of surface layer is notably homogenized, and further transformation of retained austenite into martensite is promoted. The residual stress can be relaxed and the microstructure is stabilized after tempering at 240°C, along with an increase of the hardness of surface layer.

1. Introduction

18Cr2Ni4W steel is a typical kind of high-alloy carburizing carbon steel, which can acquire high strength, excellent toughness, hardness and wear resistance through appropriate heat treatment, and can be used for engineering applications barely through quenching-tempering treatment after carburization. It is widely employed for the important manufacturing engineering components of metallurgy, mining, machinery and other industries [1-2]. Generally, it is used to fabricate important components with bulk cross section, good toughness and low notch sensitivity, working under large load [3]. The wear resistance and mechanical properties of components can be optimized through carburizing or nitriding treatment combined with subsequent heat treatment after quenching-tempering treatment [4-5]. The austenite phase region can be enlarged and the austenitization is retarded by chromium (Cr) and tungsten (W). The Ms point of the surface layer with a higher carbon content after carburization is further decreased [6-7], and the subsequent quenching results in more retained austenite in the surface layer, which lead to the mechanical properties of the material inferior to the demands and surface properties requiring further improvement [8-9]. Therefore, it is significant to study the effect of heat treatment after carburization on the microstructure and mechanical properties of 18Cr2Ni4W steel. In this paper, a cryogenic-tempering hybrid heat treatment process is carried out for the carburized 18Cr2Ni4W steel, and the effect of cryogenic treatment on the hardness of surface carburizing layer was systematically investigated, as well as the evolution of microstructure after treatment.
2. Material and methods

The high nickel (Ni) gear steel used in this study is 18Cr2Ni4W, low carbon alloy steel. The corresponding specimens are in cylindrical shape with a diameter of 80 mm and a height of 60 mm. The main chemical compositions are shown in Table 1.

The specimens were first polished and cleaned to eliminate the oil stain and oxide-scale on the surface. The conditioning heat treatment of normalizing at 920°C and tempering at 650°C was conducted on the specimens to eliminate the defects introduced in casting and processing, refining the microstructure and removing the structure inheritance. Subsequently, the specimens were carburized at 940°C for 30 h. The carburized specimens were then quenched with different mediums, cryogenically treated at -196°C for 1 h. And finally low temperature tempering at 240°C was performed on the specimens (Figure 1). The treated samples were then polished to obtain a mirror finish and etched in a weak solution of nitric acid and alcohol about 4% (volume fraction). The microstructure was characterized using Zeiss Axiovert200MAT optical microscope. The surface hardness and cross section gradient hardness were measured by THV-1MDT micro-hardness tester with a load of 1 kg, a holding time of 10 s. The average value of hardness was taken from 3 indentations at the same distance from the surface. The tensile specimens were processed with a thickness of 3 mm and a gage length of 20 mm and a width of 4 mm. Uniaxial tensile test was performed at ambient temperature using CMT5105 universal testing machine at a strain rate of 2 mm/min and three tensile specimens for the same treatment were tested for reproducibility.

![Figure 1. Heat treatment process diagram of 18Cr2Ni4W.](image)

3. Results and discussions

3.1. Effect of quenching medium on microstructure and properties

As shown in Figure 2, the tensile curves of specimens quenched with varying cooling mediums after heating at 850°C for 0.5 h are identical, which indicates that quenching medium has negligible effect on tensile strength and plasticity of 18Cr2Ni4W steel with 0.5 h holding time. Figure 3 shows that the microstructures of specimens quenched with oil and 10% NaCl aqueous solution are coincident, containing fine martensite lath, residual austenite and a small amount of carbide. The microstructures of the specimens are gradually finer from center to surface, especially quenched with water. This is

### Table 1. Chemical composition of the tested 18Cr2Ni4W steel (wt%).

| C   | Si  | Mn  | P   | S   | Cr  | W   | Ni  | Fe  |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.163 | 0.239 | 0.41 | 0.002 | 0.003 | 1.51 | 0.98 | 4.05 | Bal |

![Table 1. Chemical composition of the tested 18Cr2Ni4W steel (wt%).](image)
because the surface of the specimen contacting directly with the quenching medium, resulting in larger temperature gradient and cooling rate.

Figure 4 shows the representative tensile stress-strain curves of the specimens quenched with different mediums, which exhibits a considerable ultimate tensile strength of 1400 Mpa. The elongation of specimens by oil-quenching and water-quenching is similar, slightly inferior to that of the specimen quenched with 10% NaCl aqueous solution. This is because the cooling capacity of 10% NaCl aqueous solution is superior to oil and water, which gives rise to a quicker phase transformation and a more homogeneous microstructure, resulting in better plasticity and higher strain rate.

Figure 2. Stress-strain curve obtained by quenching with different medium at 850 °C for 0.5 h.

Figure 3. Microstructure diagram of samples obtained by quenching with different medium at 850 °C for 0.5 h: (a) oil, (b) water, (c) 10% NaCl.

Figure 5 shows the microstructures of the 18Cr2Ni4W steel specimens quenched after heating at 850 °C for 1h in an argon atmosphere with different mediums, which are primly martensite. The microstructure of the specimen quenched with 10% NaCl aqueous solution is finer and more homogeneous than quenched with water. The specimen quenched with water contains more martensite in center than surface of the microstructure, due to a decrease of thermal transmission efficiency between specimen and medium which induces an insufficient cooling rate of the specimen, stem from the bubbles adhere to the surface of specimen generated by the water evaporation caused by the heat conduction between water and sample during quenching process, leading to a surface with relatively low martensite content and hardness value [10].

As shown in Figure 6, the engineering stress-strain curves of the specimens quenched with different mediums are quite different. The specimens held for a long time and subsequently cooled in oil contained relatively more residual austenite, resulting in a low tensile strength. The plasticity of the specimen quenched with 10% NaCl aqueous solution is inferior to oil-quenched or water-quenched samples and the specimen quenched with water obtained an elongation of more than 25%, showing excellent plasticity.
Figure 4. Stress-strain curve of sample obtained by quenching with different mediums at 850 °C for 1 h.

Figure 7 shows the microstructure of the specimen held for 2 h is distinctly coarser than that held for 1 h, due to a more sufficient diffusion of carbon atoms which gives rise to coarsen-grained austenite and thicker lamellar martensite obtained from phase transformation [11], resulting in decrease of the plasticity of material.

3.2. Effects of cryogenic treatment on micro-structure and performance

As shown in Figure 8, the hardness of the specimens through different heat treatment increases from surface to subsurface to the maximum value and decreases from subsurface to center. Through carburizing, quenching and deep cryogenic treatment with liquid nitrogen, the hardness of the specimen was significantly improved and the martensite was obviously refined (shown in Figure 9), along with the carbide precipitated from residual austenite gradually transforming into martensite and hindering the dislocation migration within grains [12-13], resulting in the matrix being strengthened.

Figure 5. Microstructure of samples obtained by quenching with different medium at 850 °C for 1 h (a) oil, (b) water, (c) 10% NaCl.

Figure 6. Stress-strain curves obtained by quenching with different medium at 850 °C for 2h.
3.3. Effects of low tempering temperature on microstructure and properties

3.3.1. Effect of tempering on microstructure and performance of non-cryogenic/ cryogenic samples.

Figure 7. Microstructure of samples obtained by quenching with different medium at 850 °C for 2 h: (a) Oil, (b) Water, (c) 10% NaCl.

Figure 8. Comparison of gradient hardness of (a), (b): (a) oil quenching; (b) oil quenching + cryogenic treatment.

Figure 9. The microstructure comparison of (a), (b): (a) oil quenching + cryogenic treatment; (b) oil quenching.

Figure 10. The microstructure comparison of (a), (b): (a) oil quenching; (b) oil quenching + 240°C tempering 1.5 h.
Figure 11. Comparison of gradient hardness of (a), (b): (a) oil quenching; (d) oil quenching + 240 °C tempering 1.5 h.

Figure 12. Comparison of gradient hardness of (a), (b): (a) oil quenching + cryogenic treatment; (b) oil quenching + cryogenic treatment + 240 °C tempering 1.5h.

Figure 13. The microstructure comparison of (a), (b): (a) oil quenching + cryogenic treatment; (b) oil quenching + cryogenic treatment + 240 °C tempering 1.5 h.

Figure 10 shows the microstructure of the specimen quenched with oil, subsequently tempered at 240°C and held for 1.5 h, resulting in fine carbide particles dispersed in the tempered martensite [14], of which the hardness is distinctly inferior to that barely oil-quenched without tempering, due to the residual stress introduced from quenching was eliminated after tempering (shown in Figure 11).

According to Figure 12 and 13, the hybrid process of cryogenic treatment with tempering at 240°C for 1.5 h promoted the tempered martensite producing and further eliminated residual stress derived from the quenching, accelerating the dispersive distribution of initial carbide within the grain boundaries into martensite [15-16]. The tempering temperature can improve the toughness of the specimen but apparently reduced the hardness of material.

3.3.2. Effects of cryogenic treatment on microstructure and properties of tempered specimens. As shown in Figure 14 and 15, carbides in the martensite will precipitate along grain boundary during the tempering, the supersaturated cubic lattice will be transformed into cubes, which release space for the
transformation of residual austenite, release residual stress, and reduce the compressive stress of residual austenite. Residual austenite has a tendency to transform to tempered martensite, resulting in a slight increase in hardness, but its effect is less than that caused by the release of residual stress under the thermal drive [17-18], so the hardness decreased after tempering. Decomposition of retained austenite left without martensite transformation can make the martensite structure more stable in the subsequent cryogenic treatment as well as carbide precipitation from retained austenite, which prompt the transformation of retained austenite to martensite. The existing martensite shrinks in volume at extremely low temperature and the lattice constant of Fe atom has a tendency to shrink, which promotes martensite to precipitate more superfine carbide to distribute in the matrix. And, the dislocation movement between grains can be inhibited, which has the effect of dispersion strengthening [19-20].

![Figure 14. Comparison of gradient hardness of (a), (b): (a) oil quenching + 240 °C tempering 1.5 h; (b) oil quenching + 240 °C tempering 1.5h + cryogenic treatment.](image)

![Figure 15. The microstructure comparison of (a), (b): (a) oil quenching +240 °C tempering 1.5 h; (b) oil quenching + 240 ° C tempering 1.5 h + cryogenic treatment.](image)

According to Figure 16, the hardness at the center of the sample before and after cryogenic treatment is compared which shows that the surface of the material is further strengthened, but the hardness at the center of the sample has no changes and still maintains good toughness after a series of heat treatment processes.

![Figure 16. Change of central hardness of 18Cr2Ni4W steel after different heat treatments.](image)
3.3.3. Effects of secondary cryogenic treatment on microstructure and performance. Figure 17 and 18 show that after two different cryogenic treatment, the hardness of sample has no distinct changes as well as the microstructure. There is no obvious precipitation of carbide, the microstructure is uniform and tend to be stable. The results also indicate that after the first cryogenic treatment, the content of residual austenite was effectively reduced, consisting with the previous work and the remaining residual austenite after secondary deep cold treatment has no effect on the hardness.

After two cryogenic treatment and 240°C low-temperature tempering treatment, there exist the carbon partition period and the carbon precipitation. Especially the carbon partition from martensite to austenite is conducive to stabilizing the residual austenite and plays a greater role in the matching of plastic and toughness without decreasing the hardness and strength of the microstructure.

![Figure 17. Comparison of gradient hardness of (a), (b): (a) oil quenching + cryogenic treatment + 240 °C tempering 1.5 h; (b) oil quenching + cryogenic treatment + 240 °C tempering 1.5 h+ cryogenic treatment.](image)

![Figure 18. Microstructure comparison of samples (a), (b): (a) oil quenching + cryogenic treatment + 240 °C tempering 1.5h;(b) oil quenching + cryogenic treatment+ 240 °C tempering 1.5 h+ cryogenic treatment.](image)

4. Conclusions
(1). The microstructure of the high Ni gear steel is more uniform and fine with a higher hardness when oil-quenched after heating at 850°C for 0.5 h. Under the working condition that the steel can be used, the scheme can provide sufficient tensile strength, and the plasticity of this set of samples is also good, which will help to extend the service life of the material.

(2). The samples were deep cryogenically treated in liquid nitrogen. The residual austenite in the microstructure gradually precipitates carbides, which promotes the transformation of the residual austenite into martensite and reduces the content of the residual austenite, refining the grain and improving the strength or toughness of the material. The precipitated carbides concentrate at the grain boundary, which impedes the movement of dislocations among grains and plays a role of grain boundary strengthening. After deep cooling, the surface hardness of the sample increased obviously, while the core remained good toughness.
After quenching and cryogenic treatment, the test samples are tempered at a low temperature of 240 °C, which is conducive to improving the stability of the residual austenite of the test samples and optimizing the service life of the component.

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