

***THE EFFECTS
OF DEEP
CRYOGENIC
TREATMENT ON
M2 HIGH SPEED
STEEL***

The effects of deep cryogenic treatment combined with various tempering times on the mechanical properties and the microstructural evolution of M2 high speed steel show that deep cryogenic treatment can increase the steel hardness.

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Deep cryogenic treatment has been reported to be a beneficial process for improving the properties and microstructure of steels. Generally, deep cryogenic treatment should be combined with tempering to achieve the optimal effects on steels. However, the microscale relationship between deep cryogenic treatment and subsequent tempering is unclear. Therefore, the effects of deep cryogenic treatment combined with various tempering treatments on the mechanical properties and the microstructural evolution of M2 high speed steel are investigated in this work.

The results showed the hardness was increased by deep cryogenic treatment. Furthermore, deep cryogenic treatment improved the thermal stability of steel as the red hardness was maintained to be relatively higher after multiple times of tempering. Deep cryogenic treatment induced the transformation of retained austenite to martensite, which provided more nucleation sites for the precipitation of carbides during the subsequent tempering, and consequently promoted the dispersedly distribution of ultra-fine carbides in the microstructure. The strengthening effects of phase transformation and carbides precipitation were the main reasons for the improvement of mechanical properties of M2 high speed steel.

1 INTRODUCTION

Deep cryogenic treatment (DCT) is the process of subjecting materials at ultra-low temperature (generally below minus-100) for certain time to optimize the service performance through changing the microstructure irreversibly. It has been well proved that deep cryogenic treatment is beneficial to the mechanical properties, wear resistance, and dimensional stability of ferrous metals, including tool steels, carburized steels, alloy structural steels, and stainless steels. Benefiting from the progress of cryogenic technology and test methods, the application of deep cryogenic treatment has been extended to nonferrous metals and compound materials [1-9].

For many steels, DCT is usually conducted after quenching to reduce the content of retained austenite [10-12]. Tempering is generally necessary to achieve a more stable and equilibrium state for the iron-based alloys after quenching. The main microstructure changes of a quenched steel during tempering are concluded to be the decreased tetragonality of martensite, increased precipitation of carbides, and decomposition of retained austenite. In many researches, the conduction of DCT is usually combined with tempering to obtain better effects on steels than single DCT or tempering, where DCT most of the time is conducted prior to tempering [13-15]. Enhanced carbide precipitation during tempering of cryogenically treated samples has been found in a lot of research works. As for the mechanisms, it is assumed the martensitic transformation during DCT occurs in connection with a noteworthy transformation strain and may introduce lattice defects and a new state of stress in

the material. However, current interpretations of the enhancement of DCT on tempering remain phenomenological. The mechanism responsible for the modification of the precipitation characteristics and process is still not clarified.

Therefore, the effects of deep cryogenic treatment combined with various tempering times on the mechanical properties and the microstructural evolution of M2 high speed steel are investigated in this article.

2 EXPERIMENTAL PROCEDURE

2.1 Material preparation

M2 HSS was chosen for studying the effect of deep cryogenic treatment in conjunction with tempering. The chemical composition of specimens as analyzed by spark emission spectrometer (Model DV-6 Baird USA) is as follows (wt.%): C-0.88, Mn-0.22, Si-0.45, Cr-4.50, Ni-0.20, Mo-5.45, W-6.55, V-2.10, Fe-balance. Commercially available M2 HSS tool bits of size 10 × 10 × 50 mm were procured and used as experimental material specimens for the study.

2.2 Heat and deep cryogenic treatments

High temperature quenching (Q) treatment was conducted by using a vacuum quenching furnace. Partial pressure method was used for preventing the volatilization of some alloying elements at high temperature. After being held at 1,200°C for a certain time, specimens were quenched to room temperature with water as shown in Figure 1a. The DC-B15/13 type high temperature resistance furnace was employed for tempering (T). As presented in Figure 1b, tempering temperature was 540°C and the holding time was 2 hours. After that, specimens were taken out for natural recovery at room temperature.

Deep cryogenic treatment was carried out in a program-controlled SLX-150 cryogenic system. As shown in Figure 1c, various temperature gradients were employed to ensure specimens had been cooled down thoroughly during the cooling process. Specimens were cooled down to minus-80°C with 20 minutes of soaking time and then cooled down to minus-120°C with 30 minutes of soaking time, respectively. Finally, the minimum cryogenic temperature was minus-160°C and the holding time was 2 hours. The cooling rate in the whole cooling process was 2°C/min constantly.

In order to investigate the effects of deep cryogenic treatment on the process of tempering, different sequences between deep cryogenic treatment and tempering with various tempering times were adopted. All 12 combinations were divided into five groups based on the tempering times as shown in Table 1. “Q,” “C,” and “T” refer to the quenching treatment, deep cryogenic treatment, and tempering treatment respectively. It can be clearly indicated that the sequence of these characters is the practical order in which the treatments were conducted.

Tempering Times	Index
0	Q/QC
1	QT/QTCT/QCT
2	QTT/QTCT/QCTT
3	QTTT/QTCTT/QCTTT
4	QTTTT

Table 1: Different combinations of deep cryogenic treatment and traditional heat treatment.

2.3 Mechanical property tests

The hardness measurement was conducted in an SHBRV-187.5 Digital Brinell Rockwell & Vickers Hardness Tester with an error range of $\pm 2\%$. The loading force for the test was 200 N, and the duration time was 10 seconds. Five points were tested for each sample, and the average values were recorded. Impact toughness was tested by a JB-30A impact test device with standard Charpy U-notch (CUN) specimens at room temperature, according to the standard of GB/T 229-2007. Three specimens were used for each process, and the average values were calculated as the final result.

Specimens were heated to 600°C and then cooled down by air-cooling after 2 hours holding time. After that, the hardness of specimens was measured in complying with the method mentioned above, whose value was defined as the red hardness of the M2 high speed steel.

2.4 Microstructure analysis methods

For the purpose of investigating the changes in microstructure, specimens were ground in abrasive papers with meshes from 400 to 2,000, and then conducted mechanical polishing. After that, the polished surfaces were etched in a 6% nital solution for 10 seconds. An optical microscope (OM) (Olympus BX53M) and scanning electron microscope (SEM) (Hitachi SU1510) were used for detecting the microstructure morphology. The content of retained austenite was measured via magnetization method.

3 RESULTS AND DISCUSSIONS

The hardness measurement results of M2 HSS specimens subjected to different processes are shown in Figure 2a. It can be seen that hardness is decreased with the increase of tempering times in most processes, which accords with a general law that multiple tempering would be detrimental to the hardness of steel. We also found the changes of hardness have a close relation with the sequence between deep cryogenic treatment and tempering at certain tempering times. For instance, deep cryogenic treatment can increase the hardness when there is no tempering treatment participating in, as presented in the results of Q and QC; when there is a single tempering, the addition of deep cryogenic treatment can also improve the hardness, and specimen treated by QTC shows higher hardness than QCT; when the tempering times are increased twice, however, the introduction of deep cryogenic treatment has reduced the hardness as comparing QTT with QTCT and QCTT, and there is no obvious difference between the results of QTCT and QCTT; when the tempering times are elevated to three, whether deep cryogenic

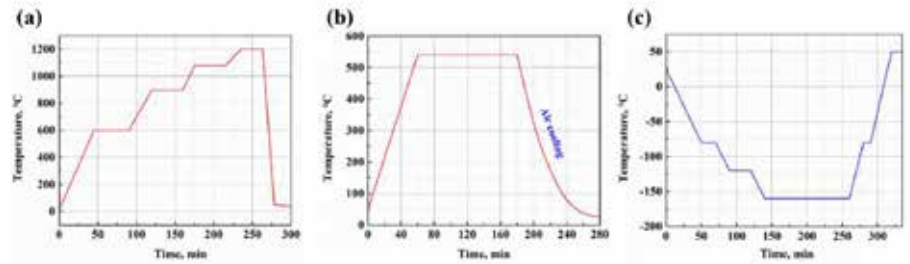


Figure 1: Schematic diagram of (a) quenching treatment, (b) tempering treatment, and (c) deep cryogenic treatment.

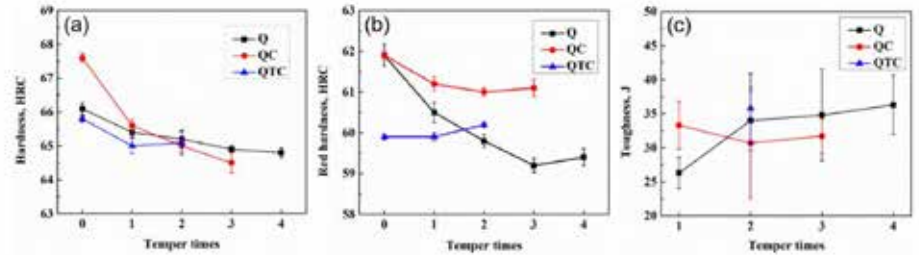


Figure 2: Measurement results of (a) hardness, (b) red hardness, and (c) impact toughness.

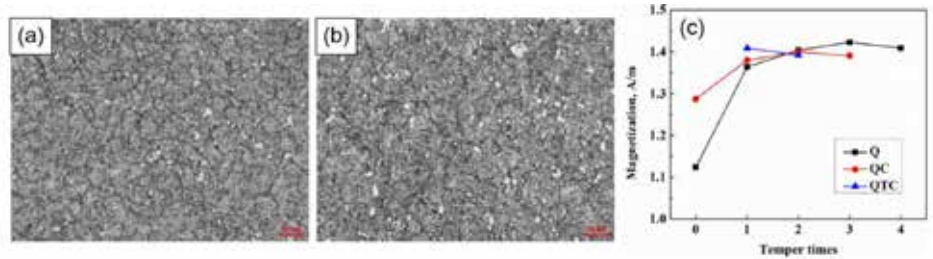


Figure 3: SEM graphs of (a) QT specimen and (b) QCT specimen. (c) is the result of magnetization for retained austenite measurement.

The induction of martensitic transformation after deep cryogenic treatment is also a reason for increasing the hardness of the M2 HSS specimens.

treatment is beneficial for the hardness depends on the sequence between deep cryogenic treatment and tempering. Conducting deep cryogenic treatment after tempering can increase the hardness of specimen while implementing deep cryogenic treatment prior to tempering produces poor effect, as comparing the results of QTTT with QTCTT and QCTTT.

The red hardness of M2 HSS specimens treated by different processes are exhibited in Figure 2b. There are significant reductions in red hardness with increasing the tempering times. However, inflection points of red hardness values can be found with the increase of tempering times, which are also related to the sequence of between deep cryogenic and tempering. For instance, without conducting deep cryogenic treatment, the inflection point appears at three tempering times; as conducting deep cryogenic treatment after quenching and prior to tempering, red hardness is decreased first

with the increase of tempering times and starts increasing after two tempering times; when deep cryogenic treatment follows with tempering, the inflection point can be found after only one tempering time. Furthermore, despite the tempering times, the execution of deep cryogenic treatment after quenching and prior to tempering has an obvious effect on improving the red hardness as compared to the simple quenching and tempering, and the effects show better than that of conducting deep cryogenic treatment after tempering. These results indicate the addition of deep cryogenic treatment makes the contribution to maintain a relatively high red hardness after multiple tempering times. In order to ensure a positive effect, deep cryogenic treatment should be conducted after quenching directly, and the tempering times should not exceed twice that in the meantime.

It can be seen from Figure 2c that deep cryogenic treatment can also improve the impact toughness of M2 HSS as the toughness value of QCT is higher than that of QT. This result implies that deep cryogenic treatment has the potential to enhance the hardness and toughness concurrently, whose effects are closely relevant to the tempering times and the processing sequence combined with tempering.

SEM graphs of the microstructure evolution of M2 HSS specimens after QT and QCT are shown in Figure 3a and 3b. It can be observed that the original austenite grain boundaries are clear and nano-acicular martensite is in the austenite grains. Carbides are precipitated from quenched martensite as shown in white particle morphology. By comparison, it can be found there exists more nanoscale superfine carbide particles with disperse distribution in the QCT specimen.

The promotion of carbide precipitation makes contribution to enhance the hardness and red hardness of M2 HSS. Figure 3c shows the changes in retained austenite content after deep cryogenic treatment and different tempering times. It can be found that the magnetization of the deep cryogenically treated specimen is higher than that of the untreated one, which indicates a fact that deep cryogenic treatment can promote the transformation of retained austenite to martensite.

The induction of martensitic transformation after deep cryogenic treatment is also a reason for increasing the hardness of the M2 HSS specimens. Furthermore, the new formed martensite after cryogenic treatment will increase the lattice defects such as distortion and internal stress, which helps to form more nucleation site for the precipitation of carbides. Consequently, deep cryogenic treatment promotes the formation of carbide precipitates in the following tempering process and improves the precipitation strengthening effects for M2 HSS material.

4 CONCLUSIONS

The effects of deep cryogenic treatment combined with various tempering times on the mechanical properties and the microstructural evolution of M2 high speed steel are investigated in this work. Deep cryogenic treatment can increase the hardness of the steel. Especially, deep cryogenic treatment can improve the thermal stability of steel as cryogenic steel maintains relatively higher red hardness after multiple times of tempering. Deep cryogenic treatment triggers the transformation of retained austenite to martensite, which provides more nucleation sites for the precipitation of carbides during the subsequent tempering, and consequently promotes the dispersed distribution of ultra-fine carbides in the microstructure. The strengthening effects of phase transformation and carbide precipitation are the main reasons for the improvement of mechanical properties of M2 high speed steel. ❄

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