Effect of multiple deep cryo-treating and tempering on microstructure and property evolution of high carbon bearing steel

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Abstract

Interest has been shown in cryogenic treatments over the last seven decades mainly due to the transformation of retained austenite and the precipitation of fine carbides. By changing the microstructure, cryogenic treatments can improve wear resistance and hardness to some extent. In this work, the effect of multiple deep cryo-treating and tempering (DCTT) on microstructural evolution in 52100 bearing steel has been investigated using x-ray diffraction (XRD) and scanning electron microscopy (SEM). The Rockwell hardness tests were also conducted to verify the microstructure evolution. Moreover, the dilatometry was used to estimate the dimensional stability. The results demonstrated that DCTT not only contributes to the precipitation of nano-size carbides, but also leads to a more homogeneous size distribution. It was also proved that the variation of carbides and hardness could be attributed to the synergy of decrease of retained austenite and movement of carbon atoms brought in DCTT. Furthermore, the sample subjected to DCTT exhibits the highest wear resistance property.

1. Introduction

The 52100 bearing steel has been adopted as a key alloy in the manufacture of bearings for more than a century [1]. Conventional heat treatment process for this 0.1C–1.5Cr type alloys is austenitization, quenching, and low-temperature tempering [2]. Quenching from the austenitizing temperature leads to a microstructure containing martensite, \(\sim 6-12\) vol\% of retained austenite [3, 4] and \(\sim 4\) vol\% of carbide particles, which are undissolved during austenitization. The subsequent tempering at approximately 160 °C brings specific microstructural evolution, which include precipitation of transition carbides and decomposition of retained austenite [5]. A variety of transition carbides [6–8], including \(\varepsilon\)-carbide [9], precipitates from the supersaturated martensite and improves toughness and wear resistance. However, the behavior of incomplete decomposition of retained austenite may set obstacles for higher hardness and good dimensional stability [10].

Cryogenic treatment was reported to help remove retained austenite and had beneficial effects on steel performance dating back to the 1940s. A typical cryogenic treatment or deep cryogenic treatment (DCT) [11, 12] consists of cooling down as low as possible [13] from ambient temperature, soaking the components at a relatively low temperature approximately at \(-196\) °C [14–17], maintaining at this temperature for an appropriate time (generally \(~24–36\) h) [18] and then warming up back to room temperature in ambient air [19]. To maximize the benefit obtained, cryogenic treatment should occur after quenching and before tempering [20].

The general interpretations of enhanced mechanical properties brought by cryogenic treatment could be attributed to the transformation of retained austenite into martensite [21], formation of fine and homogeneous...
transition carbides [22] and modification to a more favorable macro-stress state [23]. The phenomenon is usually assumed to occur during the cryogenic treatment process, while post tempering is considered to be insignificant on mechanical property [24, 25]. However, carbide precipitation, which plays a significant role in the improvement of hardness and wear resistance, can be induced by the subsequent tempering [26, 27].

Moreover, microstructure evolution is modified during tempering when the steel is cryogenically treated beforehand. The presence of fine $\eta$-carbides in the cryogenically treated steels [22, 28], which can substitute the usual $\varepsilon$-carbides [29] and the precipitation of transition carbides is also enhanced during subsequent tempering [30]. Low temperature tempering behind DCT can also be used to relieve residual thermal stresses [31]. While the compressive strain in austenite is relaxed incompletely [32].

As an inexpensive one-time permanent treatment [33], interests have been shown in cryogenic treatments over the last seven decades. The ring rolling and heat treatment are general method to improve the performance of bearing ring. The microstructure is improved by ring design, ring rolling and alloy design [34–39]. Ring rolling can further enhance the properties of the ring [40]. However, the application of cryogenic treatment in bearing industry is not as pervasive as quenching and tempering. The interaction of cryogenic treatment and conventional heat treatment gives a complex mechanism responsible for the improved mechanical properties. Cryogenic treatments can improve the wear resistance, toughness fracture resistance and hardness [41, 42].

While multi-pass cryogenic treatment rare reported. Hence the multiple DCTT is used in this paper to analyze the beneficial effects of this process. The influence of multiple DCTT is studied on the microstructure and property of 52100 bearing steel.

### Table 1. Chemical compositions of 52100 bearing steel (wt%).

| Steel | C     | Cr    | Si    | Mn    | P    | S    | Fe    |
|-------|-------|-------|-------|-------|------|------|-------|
| 52100 | 0.93–1.05 | 1.35–1.60 | 0.15–0.35 | 0.25–0.45 | ≤0.025 | ≤0.015 | bal   |

### Table 2. The heat and cryogenic treatment cycles for the samples.

| Samples | Quenching | Deep cryogenic treatment | Tempering | $\gamma$ (vol%) |
|---------|-----------|--------------------------|-----------|-----------------|
| A (group QT) | 15 min at 840 °C and oil quenching | None | 1 h at 160 °C | 9.8 ± 0.2 |
| B (group DCTT1) | 6 h at –196 °C | | | 3.6 ± 0.2 |
| C (group DCTT2) | 6 h at –196 °C on the basis of sample B | | | 3.3 ± 0.2 |
| D (group DCTT3) | 6 h at –196 °C on the basis of sample C | | | 3.2 ± 0.2 |

2. Material and experiments

2.1. Material and specimen preparation

52100 bearing steel bar used in this study was received with a size of $\phi 50 \times 100$ mm. The nominal chemical composition of the material was shown in table 1. Four samples were selected to analyze multiple DCTT on microstructure and property evolution. The samples were first preheated at 12 °C min$^{-1}$ to 650 °C, warmed up to 840 °C and heated preservation for 15 min, then placed in oil. Quenching was carried out in a vacuum furnace at 20 Pa. After conventional quenching, the samples were divided into four groups.

The sample A was tempered immediately after quenching and it was named as group QT (quenching and tempering), whereas sample B (group DCTT1) was given DCT immediately. The sample B was cooled to approximately –196 °C with the cooling rate of 1 °C min$^{-1}$ to avoid thermal shock and soaked for 360 min. The refrigerating medium was liquid nitrogen, then the sample was taken out and put in the air until the surface temperature of the sample reached room temperature. Finally, it was treated in furnace for 1 h at 160 °C. The time interval between quenching and DCT was restricted to 30 min. The sample C (group DCTT2) was subjected to another DCTT based on the sample B, while sample D (group DCTT3) was subjected to triple DCTT altogether after quenching. The process of these treatments was listed in table 2.

The parameters of treatment process at DCTT were the same. The heat treatment routes were presented in figure 1.

2.2. Microstructural analysis

X-ray diffraction (XRD) analyses were performed on polished samples using the D8 Discover x-ray diffractometer with Mo radiation from 35° to 115° at a 0.01°/min of scan rate [43]. The volume fraction of
retained austenite was measured from the XRD analysis in accordance with ASTM standard E975-00. The carbon concentration within the matrix was calculated according to [44].

Microstructure characterization of carbides was carried out using scanning electron microscope (SEM; JEOL JSM-7100F). To obtain a reliable measurement results, an accelerating voltage of 15.0 kV was used, which makes carbides appear in white and the matrix in black/grey. The size and distribution of carbides were quantitatively analyzed using image pro plus analysis software. Approximately 5000 particles from different regions of each sample were analyzed.

Cylindrical dilatometer specimens (12 mm in length and 5 mm in diameter) were machined from the sample A to D. Dilatometry was performed in a Netzsch DIL402C dilatometer and the length changes of the samples was measured during isochronal heating at 1.5 °C min⁻¹ to 600 °C.

2.3. Mechanical property test
The hardness values were measured using a Rockwell’s hardness tester at a 150-kgf load. A total of 15 indentations were considered in each sample for estimating the average value of the hardness. The dry sliding wear tests were conducted on an HT-1000 ball-on-disc high temperature tribometer. The samples with a diameter of 25 mm were slid against a ZrO₂ ball with a diameter of 3 mm and the surface of the samples was polished to Ra = 0.5 mm. The test parameters were as follows: normal load 20 N, sliding speed 0.236 m s⁻¹, and sliding distance 3393 m. To ensure the accuracy of the experiment results, each wear test was repeated three times. The wear rate was calculated with the following method (1).

\[
\text{Wear rate} = \frac{\text{Worn volume}}{(\text{normal load} \times \text{sliding distance})}, \quad \text{mm}³\ \text{Nm}⁻¹
\]  

3. Results

3.1. Quantification of retained austenite based on XRD
Figure 2 shows the typical XRD data of the A to D samples. The volume fraction of retained austenite is listed in table 2. The peaks of martensite and retained austenite are clearly visible. The volume fraction of retained austenite in sample A is 9.8% and B is 3.6%. The decrease of peak \{002\} and \{022\}, indicates the transformation of retained austenite to martensite during the DCTT in sample B. While the retained austenite is not completely transformed [45]. There is still 3.3% and 3.2% left in sample C and D. It portends increasing the cycle times has little influence on the retained austenite content.

The tetragonality of martensite decreases concomitantly with the precipitation of carbon element during each cycle, as shown in table 3. The C concentration in the matrix is 0.26% after quenching and tempering for 1 h. Which is in agreement with the report in [29, 46]. The tetragonality of martensite in sample B is calculated to be lower than the sample A. The FWHM of each peak of the samples are listed in table 4.

The C concentration in the matrix declines successively along with the increase of cycle times. The broadening and shifting of peak \{011\} \(\alpha'\) are attributed to variation both in the tetragonal structure and strain state of the matrix, which is under the influence of dislocations and precipitations in each cycle.
3.2. Quantification analysis of carbide

SEM images of sample A-D show the similar microstructure consisting of martensite matrix and dispersive spherical carbides, as shown in figure 3. The amount of carbides in figure 3(a) is significantly less than that of other samples. The samples all present a trace of original martensite structure while that of the samples B, C, and D are smaller and more indistinct, as seen in figures 3(b)–(d), respectively. Primary and secondary carbides cannot distinguish effectively. Hence all the visible carbides are uniformly analyzed together. The different parameters of carbides are shown in figure 4 and table 5.

The carbide size of the samples ranges from 0.1 to 0.8 μm, and the distribution in the DCTT and QT samples is similar. It can be seen an obvious increase of fraction of carbide particles (figure 4) in the range from 0.1 to 0.25 μm in samples B, C, and D compared with sample A. While the mean particle size decreased in the order of samples A, B, D, and C, rather than with the increase of cycle times. With respect to sample A, the mean particle size of sample B is lower by 11.5%. The value of the sample C is 19.2% and sample D is 15.4%. Moreover the area fraction [47] of the carbides is higher by 49.4% than sample A. That of the sample C is 66.7% and sample D is 68.6%.

3.3. Mechanical properties

3.3.1. Hardness

In figure 5, the macro-hardness result of samples B–D is higher than the sample A. The obvious improvement in sample B can be attributed to the transformation of a large amount of the retained austenite to martensite. The hardness result presents that the second cycle increases the hardness of the sample by approximately 1 HRC than
sample A. However, hardness declines slightly after the third cycle compared with the second, but still higher than the QT sample. This continuous decrease of macro-hardness as the increase of cycle times can be explained by the interaction of the depletion of interstitial carbon from the matrix \[48\] and the formation of small sized carbides.

Table 5. Summary of different stereological parameters of carbides.

| Parameters               | Specimens     |
|--------------------------|---------------|
|                          | Sample A      | Sample B      | Sample C      | Sample D      |
| Mean size (μm)           | 0.26 ± 0.11   | 0.23 ± 0.12   | 0.21 ± 0.09   | 0.22 ± 0.10   |
| Area fraction (vol%)     | 8.95 ± 0.34   | 13.37 ± 0.52  | 14.92 ± 0.75  | 15.09 ± 0.53  |

Figure 3. SEM images used for the quantitative determination of carbide: (a) Sample A treated with QT, (b) Sample B treated with DCTT1, (c) Sample C treated with DCTT2, and (d) Sample D treated with DCTT3.

Figure 4. Effect of heat treatment cycle on carbide size distribution.
Hardness in the range 59–66 HRC has been reported to be positive correlation with rolling contact fatigue life [49]. Generally, the hardness is determined by the supersaturation of the matrix of interstitial carbon, which hardens the matrix through tetragonal distortion. The intervention of DCT changes the main cause of the hardness. The interaction of retained austenite elimination, more homogenized nano-sized carbide precipitation and decreasing supersaturation of the martensite matrix determine the hardness. The improvement of hardness in sample B should mainly be attributed to the transformation of retained austenite to martensite. A dual DCTT results in slight decrease in hardness and little variation on the amount of retained austenite in sample C compared with B. The enhancement effect brings by nano-sized carbide precipitation exceeded the impairment result from the depletion of interstitial carbon, the increase of overall carbide size and reduction of supersaturation become the obstacle to a higher bulk hardness in sample D.

Fine carbides are produced during deep cryogenic treatment. The newly formed fine carbides and the original larger carbide form a denser, more coherent, and much tougher matrix. These fine carbides improve the hardness and wear resistance without significantly affecting the toughness [41]. While the hardness of martensite decreases with the decrease of carbon in the martensite. The hardness of the DCTT samples is higher than the QT sample under the combined influence of these two factors. It shows the influence of the former is greater than that of the latter.

3.3.2. Wear resistance
Figure 6 shows the friction coefficient curves and wear rate of the samples with different heat treatment. It is found that friction coefficient values of samples decrease as the cycle times increase. While the amplitude of the variation is unconspicuous. The mean friction coefficient under the 20 N load is found to vary between 0.38 and
0.27 as the cycle times increase. At the apply load, the highest wear rate is observed in the sample A, whereas the lowest wear rate is the sample C.

The fine carbide particles and reasonable distribution are beneficial to improve wear resistance. It was observed that the DCTT samples have a higher wear resistance than the QT sample. This can also be related to the decrease of retained austenite and its transformation to martensite under the DCTT process. The better distribution of martensite laths, along with the more uniform and finer distribution of carbides, increased the wear properties. The report [46] investigated the effects of deep cryogenic treatment (DCT) on the wear resistance of AISI 52100 bearing steel. After conducting the experimental studies, 36 h was found to be the optimal holding time at cryogenic temperature. At this holding time, the wear rate and friction coefficient were decreased, while the hardness reached to maximum values. But in this study, overall consideration the best phenomenon was obtained from the dual DCTT cycles. Even though mean friction coefficient of sample D is minimum, the wear rate is a bit higher than the sample C.

Micrograph of the worn surface of the samples is listed in figure 7. In sample A, the big groove is clearly visible. The coarse carbides embedded in the matrix peeled off. When the cyclic shear stresses are enough to peel the primary carbides from the matrix, the coarse carbides will peel off to act as the special abrasive particle during the sliding process. This leads to an increase in the wear rate of sample A. While no significant grinding crack and groove are found on the worn surface of sample C. The size and quantity of wear debris in samples A, B, and D are more than sample C.

3.4. Dimensional stability using dilatometry

Figure 8 shows the dilatometry measurement of the samples during isochronal heating periods. The dilatometric curves present two different slope changes over two temperature ranged from 100 °C to 200 °C and 200 °C to 300 °C, respectively. The lower temperature range corresponds to the precipitation of a transition carbide and the higher temperature range corresponds to the decomposition of retained austenite. The lack of a flat changes [18] on the measured curves in figure 8 is probably due to the incomplete segregation of transition carbide during the short tempering [32] and the slow heating rate. During the higher temperature range, the strain of cryogenic treated samples is lower due to the transformation of retained austenite. The amount of remaining retained austenite is nearly the same in samples B, C, and D hence the dilatometric curves exhibit the same variational trend. The slight decrease in samples C and D might be related to the formation of cementite due to the tempering process during multiple cryo-treated and tempering.

Dimensional change is considered to be related to the precipitation of transition carbides, the decomposition of retained austenite and the precipitation of cementite during tempering and the subsequent
aging. Reducing the amount of retained austenite and improving its thermal/mechanical stability is a major means of improving dimensional stability.

4. Discussion

4.1. Variation of the retained austenite
It has been widely confirmed DCT can facilitate the decomposition of retained austenite [14, 15, 21, 24]. The present work showed the retained austenite could be effectively reduced in the first DCTT. The supercooling brought by DCT is usually thought to be the driving force for transformation of retained austenite to martensite. However, the retained austenite could not be converted completely even after a threefold DCTT, which indicated a higher stability of retained austenite.

For high carbon steel, the precipitation of cementite is kinetically hindered for temperatures below 227 °C in the retained austenite [47]. The carbon atoms in solid solution in the retained austenite are inclined to remain stable during tempering at 160 °C.

4.2. Precipitation of carbides
As shown in table 5, the area fraction of carbides is higher in the DCTT samples than in the QT sample, implying the precipitation process of secondary phase could be accelerated by cryogenic treatment. The lattice distortion in combination with supersaturation of the matrix could act as driving force for the carbide precipitation [50], bringing markedly amount of secondary phase after DCT. During the low temperature tempering followed, parts of transition carbides transform to θ-carbides [29] and grows up along with the existing ones to the mean size ranged from 0.1 μm to 0.8 μm, as shown in figure 3. All the visible carbides were taken into statistical analysis, as shown in figure 4 and table 5.

The carbides could still precipitate during the DCTT in the second cycle. The strain-induced martensite formed during the cryogenic treatment in the previous cycle also provided carbon source for segregation [51]. The dislocation sites, grain boundaries, and the carbides produced during tempering in the previous cycle could act as nuclei sites of carbides during the cryogenic treatment in the next cycle [52, 53]. The evolution of particle size distribution in sample C also indicates that carbides precipitated after in the second cycle.

The average size of carbide particles in sample D increases and the peak value of the particle size distribution moves to the right in figure 4. The cryogenic treatment in the third cryo-treating and tempering still produces lattice distortion but the significantly decreased supersaturation of the matrix may restrict the carbon segregation. The change of particle size distribution in sample D indicates cryogenic treatment modified the tempering response, not only by enhancing the formation of transition carbide, but also by causing a tendency for over tempering [32].

When the samples are placed in deep cryogenic environment, part of the retained austenite will transform to martensite. Although newly formed martensite and quenched martensite have some differences in crystal structure, they have high saturation at deep cryogenic temperatures. The martensite becomes more supersaturated with decreasing temperature [41]. The lattice distortion of martensite increases and becomes more unstable. At this time, carbon and alloying atoms has the tendency to move towards nearby defects. These
positions will become the nuclei for the formation of carbide on subsequent warming up or tempering. Hence, although the area fraction of carbide increases, the mean size decreases.

4.3. Beneficial effect of multiple cryo-treating and tempering

The most prominent findings brought by DCTT include unique mechanism of martensite formations, time-dependent size of precipitated carbide particles according to current researches [52]. A large proportion of retained austenite converts into martensite by DCTT whereas there is still almost 3% in the steel. Wear resistance can be further enhanced by multiple DCTT while too many cycles are not conducive to the improvement of wear performance. Multiple DCTT can shorten the time of cryogenic treatment, thus decreasing the power consumption. Furthermore, by extending of the soaking time of cryogenic treatment, reducing the tempering temperature in one cryo-treating and tempering appropriately and increasing the cycle times, a microstructure with better wear-resisting properties and dimensional stability may be obtainable.

5. Conclusions

In this work, the effect of deep cryo-treating and tempering on the transformation of retained austenite, evolution of carbide and dry wear behaviors of 52100 bearing steel has been investigated. The significant conclusions are drawn as follows:

1. Retained austenite could not be converted completely through cryogenic treatment. The compressive strain as well as the thermal and mechanical stability of retained austenite increased along with the cryo-treated and tempered cycles.

2. Cryogenic treatment modified the tempering response by increasing lattice parameter, enhancing the formation of transition carbide and relieving compressive strain state to the matrix. Cryo-treating and tempering accelerated the reduction of supersaturation of the matrix and optimize the carbide precipitation. Multiple cryo-treating and tempering offered high dimension stability.

3. The results showed that, for the present investigation conditions, a dual cryo-treating and tempering obtains a suitable distribution of carbides and dimension stability, better hardness and wear resistance. Increasing the cycles to more than three times may deteriorate the carbides distribution, decrease hardness and reduce the wear-resisting properties.

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