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Effect of Cryogenic Time on Wear Resistance of M2 High Speed Steel

Qiang Li, Xiaoyang Huo*

School of Materials Science and Engineering Henan Polytechnic University, Jiaozuo city 454003, China
* Corresponding author

Abstract: The wear resistance, residual austenite volume and carbon content in martensite of M2 steel were studied by means of universal vertical friction and wear testing machine, X-ray diffractometer (XRD), scanning electron microscope (SEM) and digital Rockwell hardness tester. The results show that a large number of fine carbide particles in M2 steel martensite are precipitated by cryogenic treatment and distributed uniformly and diffusely in the structure. With the increase of cryogenic time, the residual Austenite volume of M2 steel gradually decreases, while the Martensite volume increases, resulting in the wear resistance and Rockwell hardness increasing first and then decreasing. After 24 hours of cryogenic treatment of M2 steel, its wear resistance increased by 310%, hardness increased by 3.6%. Therefore, 24 hours is confirmed as the best cryogenic treatment time for the wear resistance and hardness of M2 steel.

Keywords: Cryogenic treatment, Friction and wear properties, M2 high speed steel, Carbide, Rockwell hardness.

1. Introduction

High speed tool steel M2 is widely used and is a kind of cold working die steel. The steel has the characteristics of high hardness, wear resistance, high temperature stability and high strength [1], and is often used in hard cutting materials tools, cold extrusion convex die, concave die, cold heading die, plastic die and mechanical parts [2]. M2 steel, that is, the national standard steel number is W6Mo5Cr4V2, the steel number in the American ASTM standard is M2, and the Japanese JIS standard is called SKH10. Tool steels are often tempered after quenching by Conventional Heat Treatment (CHT). This treatment method helps to transform austenite into martensite, release internal stress, and improve the hardness and toughness of steel. However, a large amount of austenite and thick slab martensite are often left in the steel after conventional treatment, which is not ideal for improving wear resistance. The instability of austenite makes it prone to martensitic transformation under heat and mechanical stress, resulting in the deformation of workpieces (such as molds, tools, etc.), which further affects its accuracy and use effect, and may even lead to the failure of mechanical parts and even cause safety accidents [3-4]. In the industry, the economic loss caused by parts failure is considerable, so it is very important to improve the wear resistance of die steel to extend its service life and save production costs [5-7]. In order to reduce losses and increase efficiency, the wear resistance must be improved.

Cryogenic treatment is a method that can significantly improve the wear resistance of materials, thereby extending the life of tools and drills. Compared with heat treatment, cold treatment has the advantages of simple operation, no damage to the workpiece, no pollution, low cost. The effect of cold treatment on the material organization is permanent and acts on the entire part. Depending on the treatment temperature, cold treatment can be divided into Sub-zero Treating (ST) and Cryogenic Treating (CT). Shallow cooling treatment is generally carried out above -100 ° C, and dry ice with a temperature of about -72 ° C is usually used as the treatment medium. The cryogenic treatment is carried out in the range of -100℃ to -190℃, and liquid nitrogen with a temperature of about -198℃ is often used as the cooling medium [8-9].

Studies in recent years have shown that the introduction of cryogenic treatment can significantly improve the wear resistance of tool steel, improve the hardness and dimensional stability of steel, and reduce the internal stress [10-20]. Therefore, it is widely used in aerospace, automobile, electronics and other industries in developed countries, as well as in medicine, oil and gas, Musical Instruments and other fields. For example, some Swiss watch manufacturers place key parts under snow mountains for cryogenic treatment to improve the wear resistance and dimensional stability of watch parts. However, the effect of cryogenic treatment time on the wear resistance of tool steel has not been fully discussed in the current literature. It is important to determine the optimal cryogenic treatment time, which can not only ensure the improvement of material properties, but also reduce the cost of cryogenic treatment and improve production efficiency. The study of the effect of cryogenic treatment time on the wear resistance of M2 steel is helpful to promote the development and application of cryogenic treatment technology in the industrial field and save energy at the same time. Therefore, this study conducted 0-36 hours of cryogenic treatment on M2 steel through experiments, aiming to explore the influence of cryogenic treatment on the properties of M2 steel under different time lengths, and then determine the best cryogenic treatment time to improve the properties of M2 die steel.

2. Experimental Materials and Methods

2.1. Material and cryogenic treatment

The steel used in the experiment is M2 steel rolled, softened and annealed by the manufacturer. Table 1 shows its main chemical composition (mass fraction, wt %). 5 cuboid samples of the same size (10mm× 10mm×55m) were processed using EDM wire cutting.

Table 1. Chemical composition of M2 steel (mass fraction, %)

C	W	Mo	Cr	Si	Mn	S	P	Fe
0.85	6.38	5.37	4.34	0.228	0.28	0.02	0.067	remain

The sample heat treatment process curve is shown in Fig.1 : ①Austenitizing and quenching: The sample is heated in the furnace to 800℃ for 10min, and then heated to 1200℃ for 10min before oil quenching. ② Cryogenic treatment: The sample is placed in liquid nitrogen (-196℃) for soaking, holding time is 1, 12, 24 and 36 h, respectively. After the cryogenic treatment, the sample is taken out and placed in the air to restore its temperature to room temperature. ③ Tempering: Heat the heat treatment furnace at 600℃, and then put the sample into the vacuum furnace, hold the heat for 1 h, air cool to room temperature and remove the surface oxide, and grind off the decarbonized layer with sandpaper.

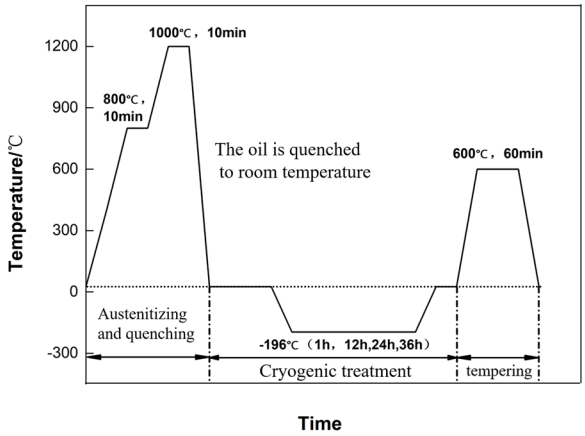


Figure 1. Heat treatment process diagram

Table 2 shows the number and grouping of heat treatment samples. The number represents the holding time at -196℃. CT0 indicates no cryogenic treatment. As the control group of the experiment, CT1, CT12, CT24 and CT36 indicate cryogenic treatment for 1, 12, 24 and 36h respectively.

Table 2. Numbers of heat treatment samples

Sample number	Heat treatment procedure		
	(1) Quenching	(2) cryogenic treatment	(3) tempering
CT0	1200℃，10min	0h	600℃，1h
CT1		1h	
CT12		12h	
CT24		24h	
CT36		36h	

2.2. Characterization experiment

After grinding and polishing the sample, the phase composition was analyzed using the SmartLab 9kw X-ray diffractometer, and the phase retrieval function in MDI Jade 6.5 analysis software was used to determine the residual austenite content. The X-ray diffraction test 2θ has a range of 30° to 90° and a scanning speed of 10° per minute. The test was performed under CuKα radiation (wavelength λ=0.15406 nm) with a current set at 250 mA and a voltage of 40 kV. The surface morphology of the fracture was observed using Merlin Compact scanning electron microscope. The hardness of the sample is tested by an electric Rockwell hardness tester (model SHBRV-187.5). Each sample is tested 5 times and the hardness value is averaged.

2.3. Wear test

The wear test was carried out by using MMW-1 vertical universal friction and wear testing machine using pin-disk method. In the test, a cylindrical specimen with Φ4.8mmx12.7mm was selected as a rotating pin with a rotating radius of 24cm. 45# quenched steel is used as the material for rotating grinding surface, and the ring size is Φ54mm×Φ38mm×10mm, its hardness is 52~55HRC, and the surface roughness Ra is less than 0.5m. The sliding speed of the rotating pin is 2m/s (corresponding speed is 800rpm), and the wear test is carried out under the condition of normal load (FN) of 30N (3kgf). Each set of experiments was repeated three times at room temperature, the weight of the sample (m1, m2) before and after wear was recorded, and the mass loss was calculated using an electronic scale with an accuracy of 0.1mg. The formulas of wear rate (WR) and wear resistance (W_R) are shown in (1) and (2):

$$WR = \frac{m_1 - m_2}{L} \tag{1}$$

$$W_R = \frac{F_N \times V_S}{WR \times H_r} \tag{2}$$

Where: WR is the wear rate of composite material, unit mg/m; m1 represents the mass of the sample before wear, in milligrams (mg); m_2 represents the mass of the sample after wear, also in milligrams (mg); L is the sliding distance of the sample in the experiment, in m. W_R is wear resistance, F_N is

positive load, unit N; V_S is the sliding speed in m/s; It is the pin wear rate in mg/L, and Hr is Rockwell hardness [6].

3. Result and Analysis

3.1. Residual Austenite volume

The X-ray diffraction (XRD) patterns of the samples after different heat treatments are shown in Fig.2. It can be observed from the figure that the (220), (200), (111) crystal faces and (110), (200), (221) crystal faces of the residual austenite and martensite of the sample show sharp peaks in the annealed state, indicating that the grain sizes of austenite and martensite are large. After cryogenic treatment, the half-height width of these peaks decreased, suggesting that the grain size became finer. In Fig. 2, the diffraction peak intensity of residual austenite (111) and (221) of the cryogenically treated sample is reduced, indicating that the content of residual austenite is reduced, and cryogenically treated sample is helpful to promote the phase transition of austenite.

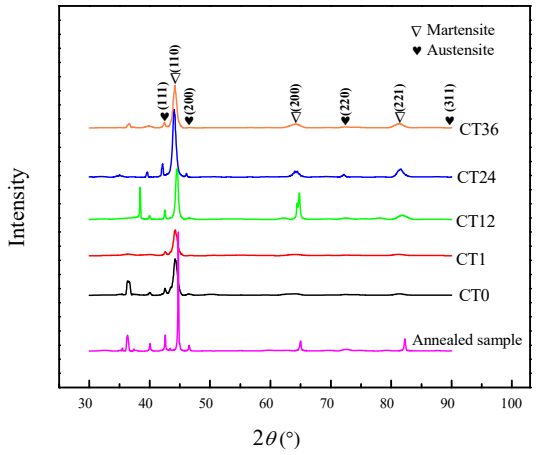


Figure 2. XRD patterns at different cryogenic times

According to industry standard YB/T5338-2019, the formula for the volume fraction of residual austenite in steel is shown in formula 3:

$$V_\gamma = \frac{1 - V_c}{1 + G\dfrac{I_{\alpha(hkl)i}}{I_{\gamma(hkl)j}}} \tag{3}$$

Where: is the total integral number of carbide phase in the sample, is the cumulative diffraction line intensity of martensite crystal face, is the cumulative diffraction line intensity of austenite crystal face, G is the ratio of martensite to austenite strength factor. The cumulative diffraction intensity of austenite (200) (220) (311) crystal face and martensite (200) (211) crystal face is selected as the calculated value.

The residual austenite volume fraction calculated according to formula (3) is shown in Fig. 3. The figure clearly shows that with the increase of cryogenic time, the content of residual austenite in tissues gradually decreases. This is consistent with previous research results [21-23], indicating that cryogenic time has a significant impact on organizational transformation. The content of residual austenite is the highest in the initial annealing state, which can be attributed to the fact that M2 steel is a high alloy steel, which is rich in alloying elements to help the stable existence of austenite at room temperature. In contrast, CT0 showed a slight decrease in residual austenite content compared to annealed samples. This is due to the relatively low martensitic transition temperatures Ms and Mf of high alloy steels, which retain a large amount of residual austenite even after quenching and tempering treatment. It can be observed from the figure that the austenite content of CT1 sample decreased sharply, while the austenite content of CT12, CT24 and CT36 samples decreased slowly. This is because as the amount of austenite transformed into martensite increases, the stress existing in the organization also increases, and the compressive stress state is expected to stabilize austenite, thus making it difficult for the residual austenite to further transform [5]. The increase of cryogenic time gradually promoted the organizational transformation, but the transformation efficiency gradually decreased. For the samples with more than 24 hours of cryogenic treatment, the austenite content was significantly reduced to less than 5%. Cryogenic treatment helps to promote the transformation of austenite, and the longer the cryogenic soaking time, the number of transformations also increases.

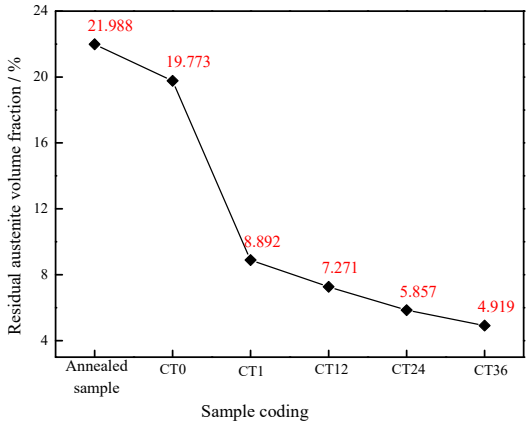


Figure 3. Residual Austenite volume (volume fraction, %)

The amplified XRD pattern of the sample M (110) side is shown in Fig. 4. Relative to the annealed steel, the peak Angle of the other samples shifted to the left between 0.26˚ and 0.72˚. After cryogenic treatment, the peak Angle of M (110) surface of the sample is obviously shifted to the left, which indicates that the spacing d of the crystal surface increases, which causes the change of martensite crystal structure. It may be that the precipitation of susaturated carbon atoms in martensite causes the shift of the peak angular position of martensite M (110).

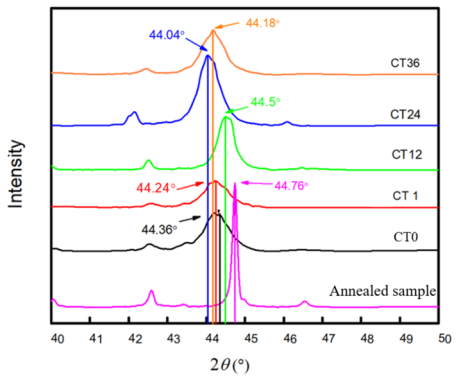
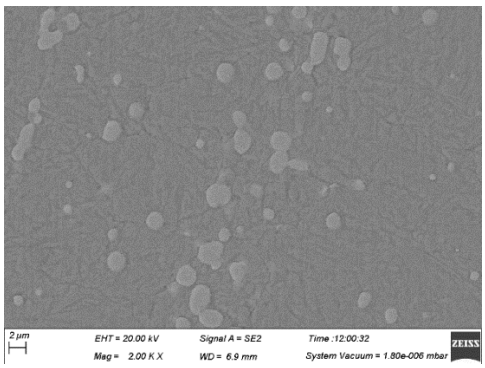


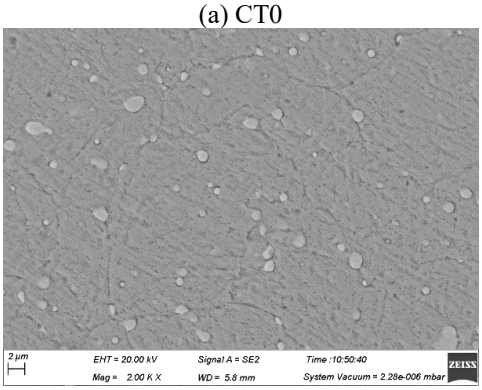
Figure 4. Local XRD patterns of different samples

3.2. Microstructure analysis

The electron microscope scanning images of CT0 and CT24 samples are shown in Fig.5. Comparing the two images, it is found that the number of carbide particles in CT0 is small, and the distribution is not uniform, and most of them are distributed in the grain boundary. However, the carbide particles in CT24 are smaller and more numerous, and the fine carbide is dispersed at the grain boundaries and inside the grain. The results show that the cryogenic treatment promotes the precipitation of carbides in M2 steel and makes them uniformly dispersed in the structure. This is conducive to improving the mechanical properties of the material. In addition, the grain boundaries of CT0 are clear, while those of CT24 are blurred. This may be because cryogenic treatment promotes the transformation of austenite to martensite, resulting in martensite appearing inside the grain, thus blurring the grain boundaries.



(a) CT0



(b) CT24

Figure 5. Electron microscope scanning of CT0 and CT24 samples

3.3. Carbon content in martensite

The carbon content formula (6) of martensite is derived by the formula (4)(5)(6) of the lattice parameter relation of martensite, and the carbon atom content of M2 steel martensite after cryogenic treatment is calculated.

$$d_{(hkl)} = \frac{1}{\sqrt{\frac{h^2+k^2}{a^2} + \frac{l^2}{c^2}}} \tag{4}$$

$$c = \frac{\sqrt{2}\, d_{(110)}\, d_{(211)}}{\sqrt{2d^2_{(110)} - 5d^2_{(211)}}} \tag{5}$$

$$a = \sqrt{2}\, d_{(110)} \tag{6}$$

$$p = \frac{c-a}{0.129} \tag{7}$$

Where, a_0 is the lattice constant of a-Fe; p is the percentage of carbon content in martensite; a and c are the lengths of the martensite axis. $d_{(hkl)}$ is the distance between the crystal faces.

The carbon content of martensite samples with different cryogenic time is shown in Fig.6. With the increase of cryogenic treatment time, the content of carbon atoms in martensite decreases gradually, which indicates that cryogenic treatment can promote the precipitation of carbon atoms in martensite matrix. CT0 treatment results in a sharp decrease in the carbon content of annealed samples, which indicates that tempering can effectively promote the diffusion and precipitation of carbon atoms. The carbon atomic weight of CT1, CT12, CT24 and CT36 decreased slowly and tended to be stable. As can be seen from the figure, susaturated carbon atoms are basically precipitated from martensite before and after 12 hours of cryogenic treatment, while longer cryogenic treatment has little effect on the precipitation of carbon atoms.

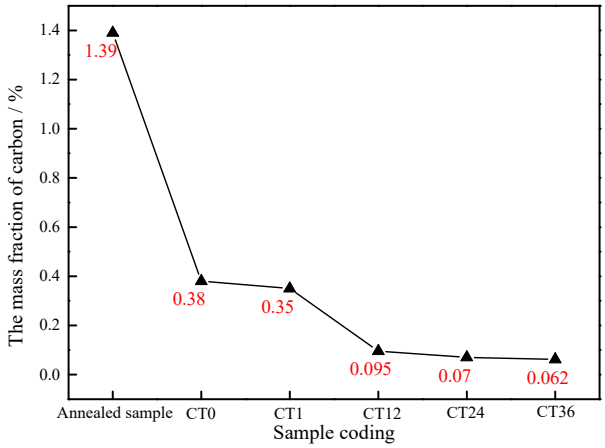


Figure 6. Carbon content in martensite samples with different cryogenic time

Although in the extremely low temperature liquid nitrogen immersion environment, the activity of carbon atoms in martensite matrix is reduced, and it is not easy to diffuse and move. However, plastic deformation occurs during the martensitic phase transition, which causes a sliding dislocation to trap the immobile carbon atom and carry it to a defect, such as a grain boundary or dislocation. This process reduces the number of carbon atoms in martensite and promotes the aggregation of carbon atoms [24]. With the extension of cryogenic time, the degree of phase transition and plastic deformation increase, so the number of carbon atoms driven by dislocation increases, and the content of carbon atoms is further reduced.

3.4. Hardness

The hardness values under different cryogenic times are shown in Fig.7. With the extension of cryocooling time, Rockwell hardness of M2 steel first increased and then decreased, and the hardness of CT24 was the highest, reaching 67.5HRC. Hardness is affected by a number of factors, including: (1) the number, nature and size of

martensite; (2) The amount of austenite; (3) The type, quantity, distribution and size of carbides. According to the previous analysis, cryogenic treatment promotes the transformation of austenite to martensite in M2 steel, thereby increasing the amount of martensite in the tissue and the dislocation density in martensite. At the same time, the amount of soft austenite is reduced, and the distribution of carbides is more uniform and the number is increased. With the increase of cryogenic time, the influence of these factors gradually increases, resulting in a gradual increase in the hardness value. However, the hardness of CT36 is reduced, which may be affected by the following factors.

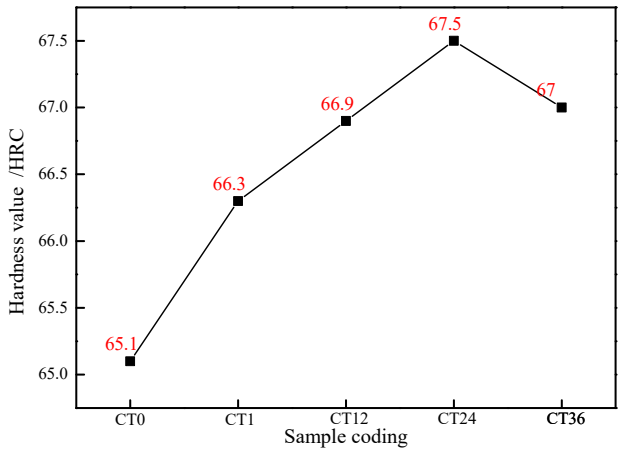


Figure 7. Hardness values at different cryogenic times

Cryotreatment causes the secondary hardening peak to move to the lower tempering temperature [25-26], forming a state of "over-tempering" [27], resulting in the hardness first rising and then decreasing. The excessively long cryogenically cooling time of the CT36 sample is equivalent to being in the state of "over-tempering", which makes the carbide in the tissue gradually accumulate and increase, and cannot effectively nail the dislocation, resulting in a decrease in hardness. In addition, long-term cryogenic treatment will also cause M3C carbides to become coarser, further reducing hardness. Cryogenic treatment promotes the precipitation of carbides in large quantities and intensifies the decomposition of martensite, so the hardness also decreases slightly [28-30]. In the structure of CT36, the alloy carbide increases and martensitic decomposition increases, resulting in a slight decrease in hardness compared with CT24. In addition to the above influencing factors, the content of austenite also affects the hardness. Although the austenite content of CT36 decreased by 1% compared to CT24, the hardness decreased when it should have increased, indicating that the hardness was more significantly influenced by martensite and carbide. The final hardness curve is the result of the competition of the above three factors.

3.5. Wear rate and wear resistance

According to formula (1) and (2), the calculated wear resistance and wear rate of the sample are shown in Fig.8. Compared with the samples of conventional treatment, the wear of the samples of cryogenic treatment first decreased and then increased with the extension of holding time. When the cryogenic treatment time reached 24 h, the wear rate of the sample was the lowest. There is a critical value of 24h for the effect of the deep cooling time on the wear rate of M2 steel. The wear rate of CT36 samples increased significantly, indicating that the improvement of wear resistance by cryogenic cooling time is limited, and too long treatment is not conducive to the improvement of wear resistance. The wear resistance of D2 tool steel decreases after 36 hours of cryogenic cooling[31], which is similar to the results obtained in this paper. It can be seen from the wear resistance diagram that the wear resistance of M2 steel first increases and then decreases, which is exactly the opposite of the change diagram of wear rate.

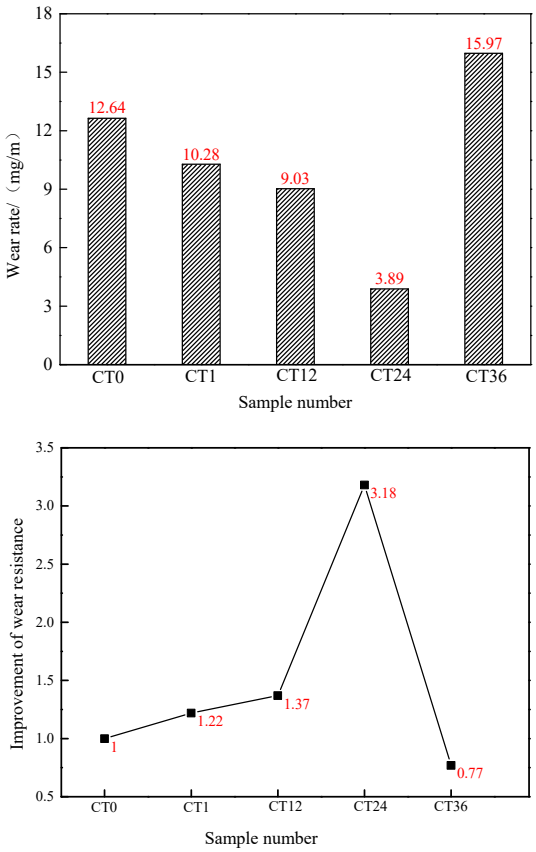


Figure 8. Wear rate and wear resistance of samples

During the wear process, a contact layer is formed between sliding surfaces, and the structure and properties of the contact layer determine its wear behavior [32-33]. The cryogenic treatment increases the hardness of M2 steel, increases the carbide and decreases the residual austenite volume, thus reducing the degree of friction thermal softening, improving the resistance to thermoplastic deformation and improving the wear resistance.

The amount of austenite in the tissue of CT36 sample is small (only 4%), indicating that there is more martensitic transformation, and theoretically the wear resistance should be further improved. However, contrary to the experimental results, the wear resistance of CT36 was significantly reduced. There may be several reasons for this phenomenon: First, the austenite volume in CT36 sample is too low, which may not be enough to effectively inhibit crack growth, but lead to reduced wear resistance. Secondly, long-term cryogenic treatment leads to the most significant refinement decomposition of tempered martensite twins, which is unfavorable to the improvement of wear resistance. In addition, the large-sized secondary carbides precipitated in the CT36 sample are easy to be peeled to form pits in the wear process, resulting in abrasive wear, which further intensifies the wear amount and reduces the wear resistance. Finally, too

long cryogenic cooling time may cause microscopic internal stress in the martensitic structure, making the material more vulnerable to damage during friction, resulting in reduced wear resistance [35].

4. Conclusions

1) The residual austenitic transition of M2 steel is positively correlated with the cryogenic time. In the initial stage of cryogenic treatment, the volume of Austenite decreased significantly, and the transformation rate gradually slowed down in the later stage. Cryogenic treatment significantly reduces the residual austenite volume in M2 steel to less than 5%.

2) With the extension of cryogenic time, the Rockwell hardness of M2 steel does not continue to increase, but first gradually rises, and then slightly decreases. After 24 hours of cryogenic treatment, the hardness of the steel reached the maximum value (67.5HRC), which was 3.6% higher than that of conventional treatment.

3) Cryogenic treatment promotes a large number of carbide particles in M2 steel to precipitate, resulting in a uniform distribution of fine carbide particles in the organization. With the extension of cryogenic time, the carbon content in M2 steel martensite decreased significantly, almost making the martensite without carbon atoms.

4) Cryogenic treatment improves the wear resistance of M2 steel, and the wear resistance gradually increases with the increase of cryogenic time. When the cryogenic time reaches 24 hours, the wear resistance of M2 steel reaches the highest point, which is 3.1 times that of the sample without cryogenic treatment. Therefore, the experimental results show that 24 hours of cryogenic treatment in liquid nitrogen environment is the best cold treatment process.

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