The International Journal of Materials and Engineering Technology (TIJMET) (2024) 7(2)
http://dergipark.gov.tr/tijmet
ISSN: 2667-4033

RESEARCH ARTICLE https://doi.org/ 10.70858/tijmet.1499365

Effect of deep cryogenic treatment on microstructural and mechanical properties of high-performance CPOH and WP7V tool steels

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Article Info

Article history:
Received 11.06.2024
Revised: 24.12.2024
Accepted: 26.12.2024
Published Online: 30.12.2024

Keywords:
Tool steel
CPOH
WP7V
Deep cryogenic treatment
Wear test

Abstract

In recent years, deep cryogenic treatment (DCT) has been applied to steel materials as an additional heat treatment to improve various material properties such as wear resistance, impact resistance and hardness. In this study, the mechanical properties of CPOH cold work tool steel with high wear and impact resistance and Dörrenberg Edelstahl patented WP7V cold work tool steel used in applications requiring high hardness were improved by deep cryogenic heat treatment. The materials subjected to deep cryogenic heat treatment were subjected to a conventional austenitizing and quenching process. After quenching, the materials were subjected to deep cryogenic treatment and then tempered. The microstructure, hardness, wear and impact properties of cryogenically heat treated materials were investigated in comparison with those obtained after conventional heat treatment. It was found that the wear rate of CPOH steel increased by 76.22% and WP7V steel increased by 88.95% with deep cryogenic heat treatment. At the same time, as a result of microstructure examinations, it was determined that the amount of residual austenite decreased and finer secondary carbide distribution was realized. It was found that deep cryogenic heat treatment of tool steels in addition to conventional heat treatment can improve material performance and provide high performance tool steels required by the industry.

1. Introduction

CPOH steel material; It is a new generation cold work tool steel that combines high wear and impact resistance with 8% chromium and 2.5% molybdenum. This tool steel is used in bolt rolling rollers and combs, cold rolling mill rollers, precision cutting dies for sheet metal up to 10 mm. WP7V tool steel is a special tool steel with high chromium, molybdenum and vanadium alloy patented by Dörrenberg Edelstahl. It is used in molds subjected to high stresses due to its high hardness depth, engraving molds with flat shapes such as forks and spoons, cold or hot cutting molds or punches, sheet metal cutting molds of 7 mm and above [1].

In hard various applications such as precision cutting, stamping and punching applications, tool steels are subjected to high loads, contact pressures, contact temperatures and conditions that cause wear of the materials. Tool steels exhibit mechanical properties such as high hardness, wear resistance and impact strength due to their tempered martensite microstructure and carbides in their structure. In order to further improve these properties under harsh conditions of use, deep cryogenic treatment has attracted much attention in recent years [2].

In the last decade, the application of cryogenic heat treatment to metallic materials has led to significant advances in various industries, such as medicine, aerospace, robotics, materials science, nanotechnology and mining. This technique, 19. it evolved over the century from the first attempts of James Dewar and Karol Olszewski to process materials at low temperatures using liquefied gases (nitrogen and hydrogen). Scientific foundations of cryogenic heat treatment, 20. it was further reinforced by the first observations made by NASA in the middle of the century. NASA has detected a noticeable increase in hardness and wear resistance in the materials used on space shuttles returning from space, especially aluminum components. These findings have revealed the potential of cryogenic heat treatment to improve the properties of metallic materials. Since then, cryogenic heat treatment has been adapted by various methods to improve the macroscopic and microscopic properties of metallic materials and significant achievements have been achieved in industrial applications. This process improves the microstructure of the materials, increases their durability and enables the emergence of longer-lasting and efficient products that meet the performance requirements [3-4]. However, a study by Hong et al. [5] indicated that cryogenic heat treatment reduces the cost by 50%. Therefore, it is recognized as an environmentally friendly and economical approach to improving materials.

Deep cryogenic processing (DCT) involves holding materials at very low temperatures, such as -125 to -196 °C, for 12 to 48 hours [6]. In deep cryogenic processes, materials are gradually cooled from room temperature to cryogenic temperatures of -196°C. After maintaining this temperature for a certain period of time, the temperature is raised to room temperature at the same rate as the cooling rate. The use of liquid nitrogen or nitrogen gas as the cooling medium avoids thermal stress problems during slow cooling [7].

In general, in tool steels, the purpose of deep cryogenic treatment is to remove residual austenite and distribute fine carbides evenly. The transformation from austenite to martensitic phase begins at the martensite initial temperature (M_s) and is completed at the martensite ending temperature (M_f). However, the martensite terminal temperature (M_f) is considered

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How to cite this article:
Karakurt, V., Öztürk, F., Çitrak, T., Gözüak, G.N., Zığındere, O., Effect of deep cryogenic treatment on microstructural and mechanical properties of high-performance CPOH and WP7V tool steels, The International Journal of Materials and Engineering Technology (TIJMET), 2024, 7(2): 77-84

to be below room temperature. This results in the partial transformation of austenite to martensite during rapid cooling (Q). Deep cryogenic treatment accelerates the conversion of retained austenite to martensite by reducing the temperature to extremely low temperatures (-196 °C). At the same time, deep cryogenic treatment contributes to the formation of finer secondary carbides [8–10]. These changes in the microstructure after cryogenic treatment of tool steels help improve the mechanical properties of the material, including hardness, impact strength and wear resistance [11]. Marcos Perez et al., [12] in their study, investigated the effect of deep cryogenic heat treatment applied at -196°C for 12 hours on the mechanical properties of AISI H13 steel. They reported that the cryogenic heat treatment significantly improved the fracture toughness of H13 steel, attributing this improvement to the homogeneous distribution of fine carbides resulting from the cryogenic process. In another study, D. Das et al., [13] investigated the effect of deep cryogenic heat treatment performed at -196°C for 36 and 84 hours on carbide precipitation and tribological behavior of D2 steel. The samples subjected to both conventional and deep cryogenic heat treatment were tested for wear under sliding conditions at three different loads (49.05 N, 68.67 N, and 78.48 N) using a constant sliding speed. Their findings demonstrated that deep cryogenic heat treatment promotes the formation of refined secondary carbides, increases their volume fraction, and leads to a more homogeneous distribution. They also reported that the wear resistance of the material improved due to a reduction in retained austenite, an increase in carbide content, and the homogeneous distribution of carbides.

This study investigates the effects of deep cryogenic treatment (DCT) on the next-generation tool steels, CPOH and WP7V, in comparison to conventional quenching and tempering (QT) processes. DCT is applied to enhance the performance of these innovative steels, which are subjected to challenging industrial conditions. To date, no studies in the literature have examined the application of cryogenic treatment on these patented steels, making this research a valuable contribution to achieving superior performance in industrial applications.

In this context, samples treated with and without DCT were analyzed for hardness, microstructure, and mechanical properties (wear and impact resistance). The findings of this study provide a comprehensive evaluation of how DCT affects the performance of steels compared to traditional heat treatment methods.

2. Materials and methods

2.1. Materials

Table 1 shows the chemical compositions of CPOH and WP7V tool steels. The tool steels were obtained from SAĞLAM METAL A.Ş.

Table 1. Chemical composition of CPOH and WP7V tool steels

Tool Steel	Fe	C	Cr	Mo	V
CPOH	Balance	1.00	8.00	2.50	0.30
WP7V	Balance	0.50	7.80	1.50	1.50

2.2. Heat treatment of the materials

Quench and tempering heat treatments of tool steels were carried out in a vacuum environment in the Schmetz furnace at Sağlam Metal. Both tool steels were gradually pre-annealed at different temperatures after vacuum was applied and then the furnace was raised to process temperature. After the austenitic transformation was achieved at 1040 °C for 45 minutes, the materials were cooled with nitrogen gas and the martensitic transformation (hardening) of the tool steel was carried out. For comparison purposes, some samples were taken into a deep cryogenic heat treatment cabinet and subjected to deep cryogenic heat treatment at -150 °C for 24 hours. In this process, nitrogen gas was used as the cooling medium. Then, tool steels without deep cryogenic heat treatment (conventional quench) and with deep cryogenic heat treatment were completed with two-stage tempering under the same conditions at 520°C for 5 hours. The heat treatment process applied to CPOH and WP7V tool steels is shown in the graph in Figure 1.

2.3. Microstructure investigations of materials

For metallographic examinations, the tool steels were first sanded with sandpaper at 320, 600, 800, 1200 and 2500 grit intervals and the surface cleaning was completed by polishing with 3 µm diamond paste. Chemical etching of the samples was carried out by immersion in 100 ml of distilled water containing 3% Nital (HNO_3). Microstructure characterizations were performed using a Nikon LV150N flat metal microscope.

2.4. Hardness measurements

Hardness measurements of tool steels were carried out by the Vickers method using the Future-Tech FM800e device with a diamond pyramid tip under a load of 500 gf. Measurements were made with a sinking time of 10 seconds and five separate measurements were taken for each material and the average value was reported.

2.5. Impact Test

Impact test samples with dimensions of 75x10x10 mm were prepared from tool steels with and without deep cryogenic treatment. Charpy Impact Test was carried out in Karabük University Iron and Steel Institute MARGEM Laboratory according to ASTM E23-16b standard. Charpy Impact Test results of CPOH and WP7V tool steels were calculated by taking the average of 2 tests.

2.6. Wear test

Wear tests of tool steels were carried out using the linear wear test device within Sağlam Metal. Tool steels were tested using aluminum trioxide (Al_2O_3) balls in a dry environment, at a speed of 175 rpm and under a load of 20 N for a distance of 500 meters. After the test, volume losses were determined by examining the surface profilometry of the wear marks on the materials.

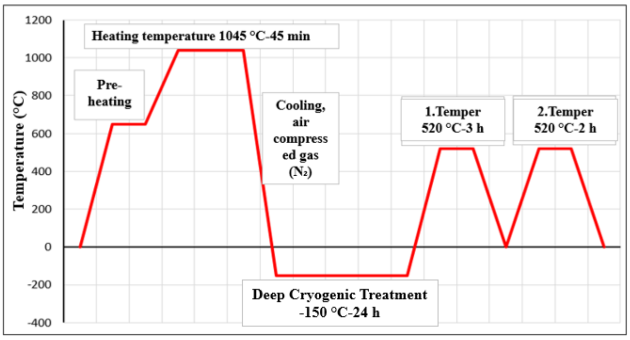

Figure 1. Process graph of heat treatment of CPOH and WP7V quality tool steels

3. Results and discussion

3.1. Microstructure investigations

Figures 2 and 3 show the microstructure images of CPOH and WP7V tool steels with conventional (QT) and deep cryogenic heat treatment (DCT). The microstructure of both steels consists of matrix and carbides. In both cases, the matrix consists primarily of tempered martensite. The irregular, spherical, white structures seen in microstructural images are alloyed carbide phases, which are primary and secondary carbides [14]. In Figures 2(a) and 3(a), micrographs of CPOH and WP7V tool steels without cryogenic treatment reveal an uneven distribution of primary carbides. Additionally, the presence of austenite retained in the microstructure of WP7V tool steel without deep cryogenic treatment is observed more clearly in Figure 3 (a).

When the cryogenically treated microstructure images of CPOH and WP7V tool steels presented in Figure 2(b) and Figure 3(b) are examined, it is seen that both steels have a tempered martensite structure, primary carbides are not observed and secondary carbides are smaller than 5 µm. The formation mechanism of secondary carbides is attributed to thermal stresses generated during the deep cryogenic process, leading to the fragmentation or refinement of existing large primary carbides into smaller secondary carbides [15–16]. There are also effects of deep cryogenic treatment such as (i) homogeneous redistribution of fine alloy carbide, (ii) precipitation of very small size (<1 µm) carbide alloy elements. [17].

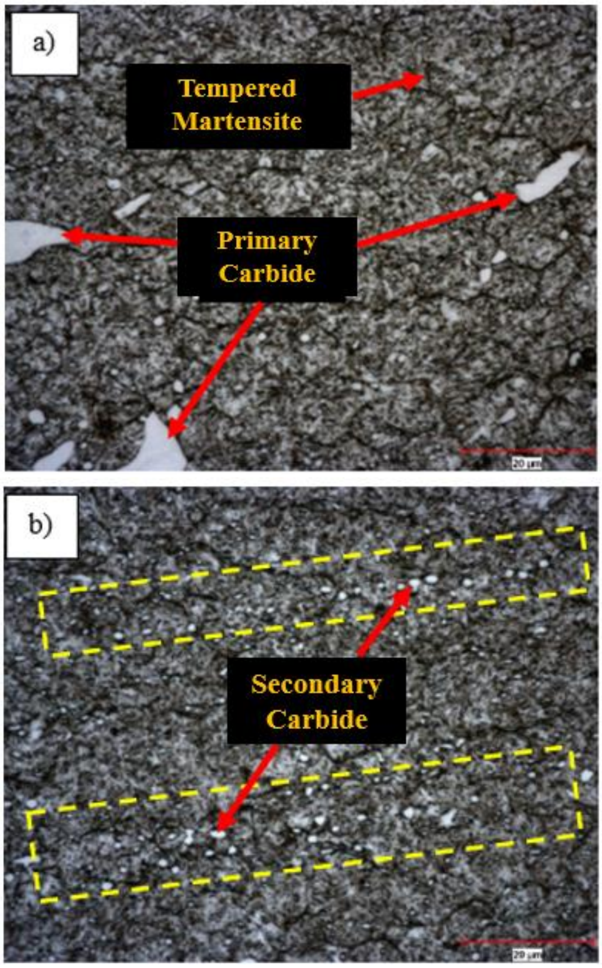

Figure 2. Optical microstructure image of CPOH tool steel at 1000X magnification a) QT, b) Q+DCT+T

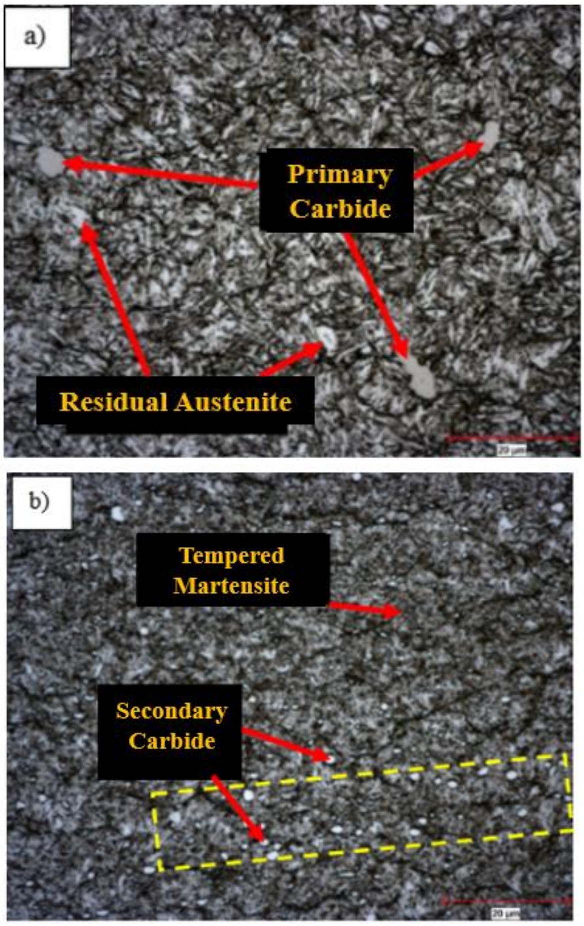

Figure 3. Optical microscope image of WP7V tool steel at 1000X magnification a) QT, b) QT+DCT+T

3.2. Hardness test results

Vickers hardness results of CPOH and WP7V tool steels without deep cryogenic treatment (Conventional (QT)) and with deep cryogenic heat treatment (DCT) are shown in Figure 4. The hardness of CPOH and WP7V tool steels with conventional heat treatment was determined as 788.8 HV and 714.9 HV, respectively. The hardness of CPOH and WP7V tool steels subjected to deep cryogenic heat treatment was measured as 803.1 HV and 724.7 HV, respectively. After deep cryogenic heat treatment, an increase in the hardness of the tool steel was observed by 15 HV for CPOH and 10 HV for WP7V. This increase in hardness after deep cryogenic heat treatment increases the hardness strength by causing the remaining austenite, a softer phase seen in the microstructure image, to transform into martensite, a harder phase with a higher carbon content [18]. Moreover, hardness increased in deep cryogenic heat treated samples due to continuous austenite removal, more uniform carbide distribution, and higher secondary carbide content [19–20]. It is reported in the literature that the presence of secondary fine carbides significantly increases the mechanical strength of steel due to their small and hard properties [21].

In both cases, CPOH tool steel was observed to be harder than WP7V. This is due to the fact that CPOH tool steel contains 0.5% more carbon than WP7V tool steel in the chemical composition given in Table 1. The higher amount of carbon in CPOH tool steel increases the solid solubility in the steel crystal structure, causing the crystals to bond more tightly to each other and increasing the hardness [22].

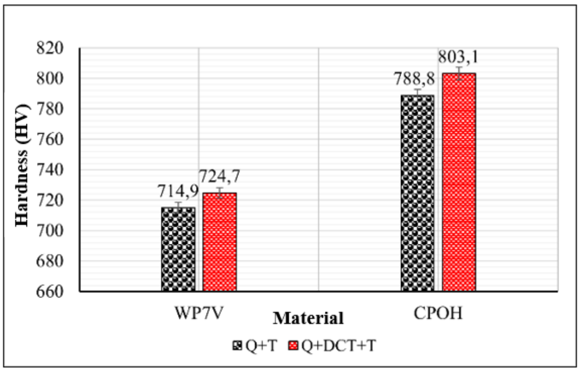

Figure 4. Hardness test results of QT and DCT heat treated CPOH and WP7V tool steels

3.2. Impact test results

Table 2 shows the data obtained from the Charpy impact test. While the average impact energy of CPOH and WP7V tool steels subjected to conventional heat treatment was calculated as 2.11 J and 5.8 J, respectively, the impact energy of CPOH and WP7V tool steels subjected to deep cryogenic heat treatment was calculated as 3.77 J and 8.76 J. It was observed that the impact energy increased when both tool steels were subjected to deep cryogenic processing. The notch impact strength of the material was calculated using the notch impact strength calculation formula (1) [23]. The impact strength of CPOH tool steel was found to be 26.38 kJ/m^2 after conventional heat treatment, while it was 47.13 kJ/m^2 after deep cryogenic heat treatment. The impact notch strength of WP7V tool steel under conventional heat treatment and deep cryogenic heat treatment was found to be 72.5 kJ/m^2 and 109.5 kJ/m^2, respectively. It was observed that the impact notch strength of both tool steels increased after deep cryogenic heat treatment.

$$\text{Impact strength} = \frac{absorbed\ energy}{\text{A}} \quad (1)$$

A = Cross-sectional area in the notch area

This increase in impact toughness resulting from deep cryogenic treatment is believed to be an improvement due to finer and more uniform carbide precipitation seen in microstructural images [24]. In both conditions, CPOH tool steel was found to have lower impact notch strength compared to WP7V tool steel. The higher the carbon content in tool steels, the more brittle the material becomes, thus reducing its ductility and making the steel more brittle. Based on this mechanism, it was concluded that the impact notch strength of CPOH tool steel was lower than that of WP7V tool steel.

Table 2. Charpy Impact Test Results of CPOH and WP7V Tool Steels

Tool Steels	Average Impact Energy (J)		Impact Strength (kJ/m²)	
	QT	Q+DCT+T	QT	Q+DCT+T
CPOH	2.11	3.77	26.38	47.13
WP7V	5.8	8.76	72.5	109.5

3.4. Wear test results

Table 3 shows the average friction coefficients of CPOH and WP7V tool steels obtained from the dry wear test for 500 m. While the friction coefficient of CPOH tool steel with conventional heat treatment was 0.478μ, the friction coefficient after deep cryogenic heat treatment was 0.389μ. While the friction coefficient of WP7V tool steel was 0.545μ after conventional heat treatment, it decreased to 0.401μ after cryogenic treatment. Both materials showed a decreasing trend in friction coefficient after deep cryogenic treatment.

Table 3. Coefficient of friction values of CPOH and WP7V tool steels.

Tool Steel	Coefficient of Friction (μ)	
	QT	Q+DCT+T
CPOH	0.478	0.389
WP7V	0.545	0.401

Figure 5 shows the 3D profile topographies of the wear scars of (a) conventional (b) deep cryogenically treated CPOH tool steel after the wear test. Considering the color concentration, it was observed that the depth color concentration of the deep cryogenically treated material was lower than that of the conventionally heat treated material.

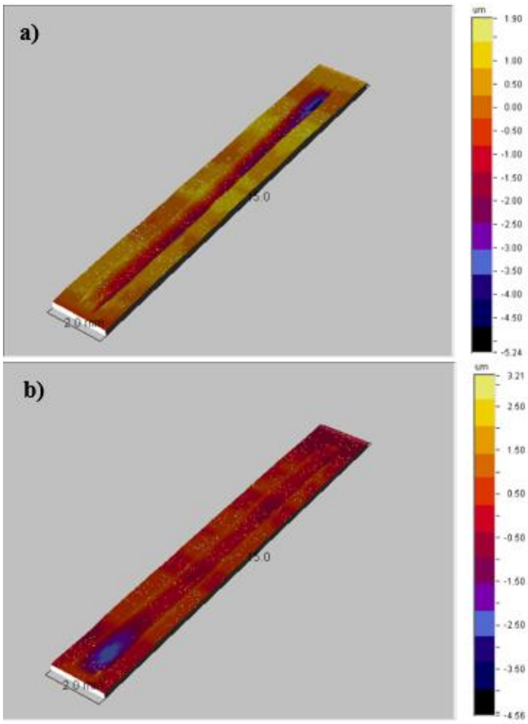

Figure 5. 3D surface topography images of CPOH tool steel a) QT, b) Q+DCT+T

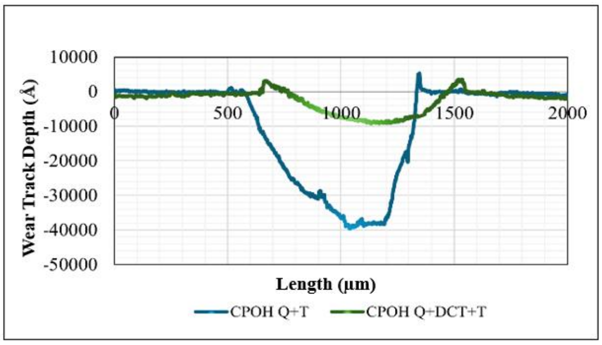

Figure 6. Two-dimensional profilometer images of the worn surfaces of conventional and deep cryogenic heat treated CPOH tool steel

Figure 6 shows the 2D profilometer image of conventional (QT) and deep cryogenic heat treated (DCT) CPOH tool steel after the wear test under 20 N load. Detailed examination of the profilometer images shows that deep cryogenic treatment reduces the pit depth and wear area.

Figure 7 shows the 3D surface topographies of (a) conventional and (b) deep cryogenic treated WP7V tool steel after the wear test. It is observed that the depth color concentration is higher in the conventional heat treated 3D topography of WP7V tool steel compared to the deep cryogenic treated material.

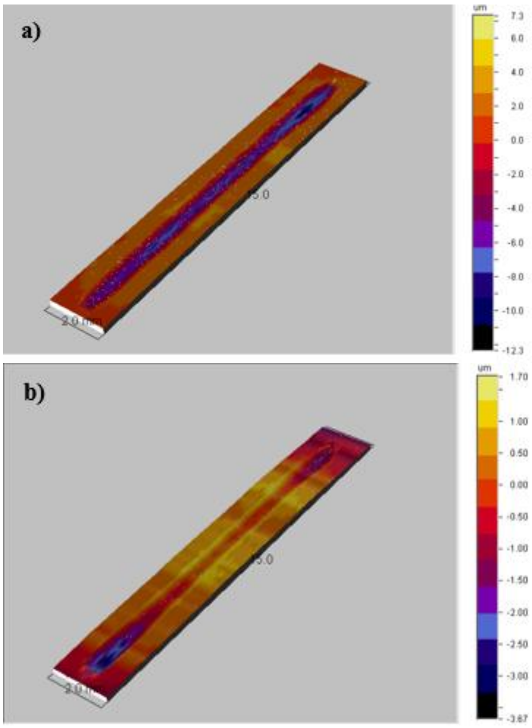

Figure 7. 3D surface topography images of WP7V tool steel a) QT, b) Q+DCT+T

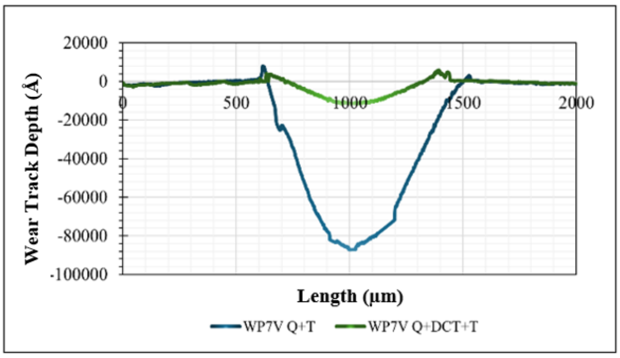

Figure 8. Two-dimensional profilometer images of the worn surfaces of conventional and deep cryogenic heat treated WP7V tool steel

Figure 8 shows the 2D profilometer image of conventional (QT) and deep cryogenic heat treated (DCT) WP7V tool steel after the wear test under 20 N load. The deep cryogenic heat-treated WP7V tool steel was observed to exhibit less wear depth and better wear properties compared to the conventionally heat-treated WP7V tool steel.

In Figure 9, the wear depths of conventional and deep cryogenic heat treated CPOH and WP7V tool steels are compared. CPOH tool steel with conventional and deep cryogenic heat treatment showed better wear resistance compared to WP7V tool steel with conventional and deep cryogenic heat treatment. In both cases, it is thought that the fact that CPOH tool steel has a lower pit depth than WP7V tool steel is due to the alloying elements it contains. The high carbon content in CPOH tool steel increases the hardness resistance, making the interatomic bonds in the material stronger during wear as the hardness increases. This makes it difficult for abrasive particles to dislodge the material. This reduces the effectiveness of mechanisms such as microscraping and notching as the material hardens.

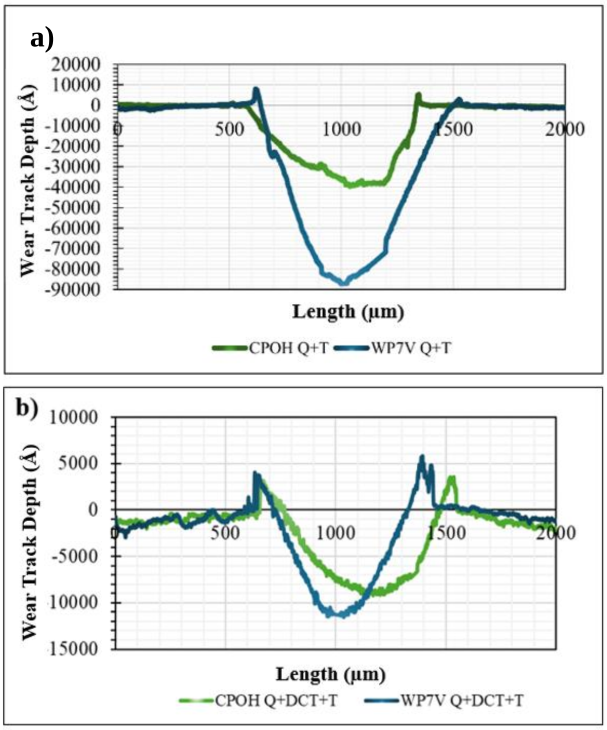

Figure 9. Comparative wear depth plot of CPOH and WP7V tool steel a) QT, b) Q+DCT+T

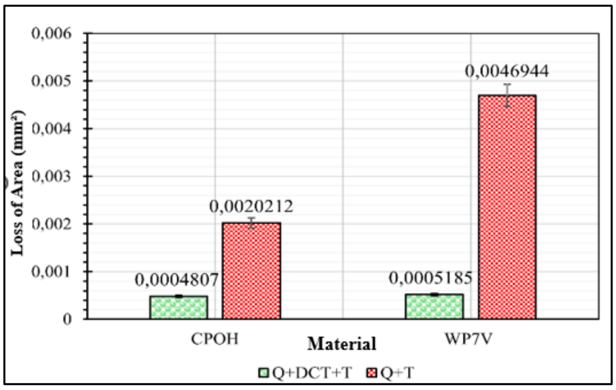

Figure 10. Area measurements calculated from two-dimensional profilometer images of the worn surfaces of CPOH and WP7V tool steel

Figure 10 shows the area loss diagram of the material, consistent with the wear scars obtained from two-dimensional profilometer analyzes of CPOH and WP7V tool steels. It is observed in Figure 10 that the highest area loss occurs in conventionally heat-treated WP7V tool steel.

Although area loss after deep cryogenic heat treatment decreased compared to conventional heat treatment for both tool steels, for WP7V tool steel, deep cryogenic heat treatment resulted in a more significant improvement in area loss compared to CPOH tool steel. This observation can be explained by the fact that the alloying elements in WP7V tool steel promote secondary carbide formation. It is thought that the high content of vanadium alloying element in WP7V tool steel allows the precipitation of vanadium-rich secondary carbides [25].

Figure 11 shows the wear rate graphs of WP7V and CPOH tool steels. Wear rates were calculated with the formula given below [26].

$$\text{Wear rate} = \frac{loss\ of\ area}{\text{load} \times \text{distance} \times \text{density}} \qquad (2)$$

The wear rates of CPOH and WP7V tool steels subjected to conventional heat treatment are 2.57478E-08 and 5.98013E-08, respectively. The wear rates of CPOH and WP7V tool steels subjected to deep cryogenic heat treatment were 6.12357E-09 and 6.6051E-09, respectively. A significant reduction in the wear rates of both tool steels subjected to deep cryogenic heat treatment was observed. The effect of deep cryogenic heat treatment to improve wear resistance is thought to be due to the increased density of uniformly dispersed fine secondary carbides formed by deep cryogenic heat treatment and the removal of retained austenite, a soft phase [27-28]. After deep cryogenic treatment, an improvement in wear resistance of 76.22% for CPOH tool steel and 88.95% for WP7V tool steel was observed.

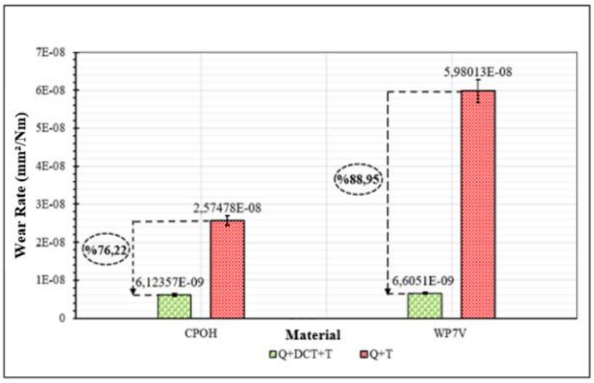

Figure 11. Wear rate graphs of CPOH and WP7V tool steels

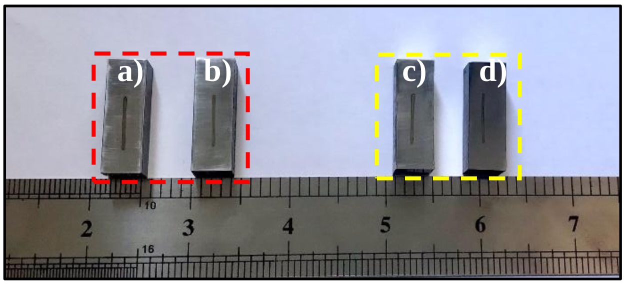

Figure 12. Macro images of WP7V and CPOH tool steels subjected to wear testing, a) WP7V QT, b) WP7V Q+DCT+T, c) CPOH QT, d) CPOH Q+DCT+T

According to the wear test results applied over a distance of 500m under 20N load, macro and micro images of the wear marks of conventional and cryogenically treated WP7V and CPOH tool steels are presented in Figure 12 and Figure 13. As seen in the 50X microstructure images of the wear scars in Figure 13, it was determined that conventionally heat-treated WP7V and CPOH tool steels had wider scar depths compared to their cryogenically treated forms.

When the wear traces of (a) conventional (Q+T) and (b) cryogenically treated (Q+DCT+T) states of WP7V tool steel are examined in Figure 13, it is seen that in the case of conventional heat treatment, the parts broken off from the surface along the wear trace are removed from the system without adhering to the surface again. It is considered to be moving away. At the same time, it is seen that they do not interact much with the surfaces of the sample and the alumina ball. This indicates that it exhibits intense abrasive wear. In the case where cryogenic treatment was applied, particles adhering to the surface as well as intense abrasive wear were observed. Adhesive wear type was also occasionally exhibited in cases where cryogenic treatment was applied.

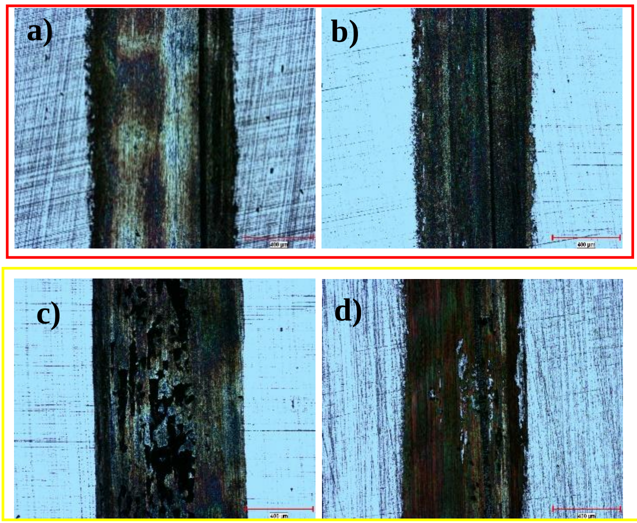

Figure 13. 50X optical microscope images of WP7V and CPOH tool steels subjected to wear testing, a) WP7V QT, b) WP7V Q+DCT+T, c) CPOH QT, d) CPOH Q+DCT+T

Figure 13 shows the wear marks of both conventional (Q+T) and cryogenically heat-treated (Q+DCT+T) forms of CPOH tool steel. In both (c) conventional and (d) cryogenic heat treated forms, it is observed that adhesive wear is dominant, with the particles that break off at the beginning of wear sticking to the surface again.

The predominance of adhesive wear in CPOH tool steel is considered to stop or slow down the progression of wear during sliding compared to WP7V tool steel. As seen in the wear rate graphs in Figure 11, CPOH tool steel supports this assessment by exhibiting a lower wear rate compared to WP7V tool steel.

Figure 14 illustrates the schematic wear mechanism based on carbide size and distribution. As shown in the mechanism, homogeneously distributed secondary carbides create a uniform hardness and surface during wear, resulting in stable wear across the entire surface. In contrast, the wear mechanism of tool steel with primary carbides reveals that irregularly distributed primary carbides lead to instability on the tool steel surface, causing more wear in areas lacking carbides due to lower hardness.

This mechanism highlights the wear characteristics of materials subjected to both conventional and deep cryogenic heat treatments. The research findings indicate that the presence of primary carbides in conventionally heat-treated materials increases the wear rate, whereas the wear rate decreases after deep cryogenic treatment due to the formation of secondary carbides, enhancing wear resistance.

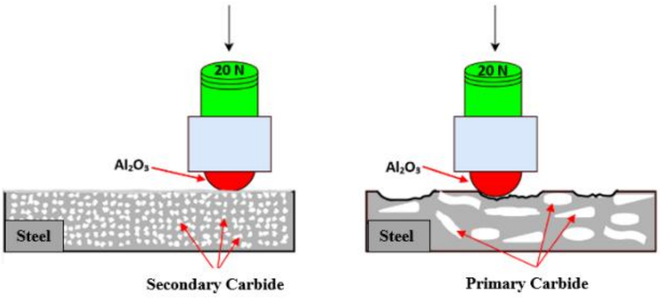

Figure 14. Schematic illustration of the wear mechanism according to carbide type (Primary-Secondary) and distribution

Additionally, it is believed that the deep cryogenic treatment in CPOH and WP7V tool steels induces martensitic conditioning, leading to the formation of defects such as dislocations, where carbon atoms accumulate. Deep cryogenic treatments also generate residual compressive stresses within the structure, particularly at interfaces such as the carbide-matrix interface. These compressive stresses can mitigate the tensile stresses that occur during wear, thereby reducing crack initiation and propagation and enhancing the adhesion of carbides to the matrix. This results in improved wear resistance during wear [29-30].

4. Conclusions

- In this study, the hardness of CPOH tool steel treated with conventional heat treatment was measured at 788.8 HV, whereas the hardness of CPOH tool steel subjected to deep cryogenic treatment was 803.1 HV. It was found that the deep cryogenic treatment increased the material's hardness by 1.8%.
- While the hardness of WP7V tool steel with conventional heat treatment was measured as 714.9 HV, the hardness of WP7V tool steel with deep cryogenic treatment was measured as 724.7 HV. Deep cryogenic treatment increased the hardness of WP7V tool steel by 1.4%.
- According to the Charpy impact test results, the average impact energy of CPOH tool steel increased by 78.67% after deep cryogenic treatment, while the average impact energy of WP7V tool steel increased by 51.03%.
- According to the wear test results, the application of deep cryogenic treatment reduced the area loss of CPOH tool steel by 76.22% and the area loss of WP7V tool steel by 88.95%.
- Compared with conventional heat treatment, deep cryogenic treatment reduced the wear rate of CPOH tool steel by 76.22% and the wear rate of WP7V tool steel by 88.95%.
- As a result of the study, it was evaluated that deep cryogenic treatment, when used in conjunction with conventional heat treatment in both new generation tool steels and other tool steels, can improve mechanical and wear properties, thereby extending the service life in applications where wear and impact resistance are critical, such as in the aerospace, automotive, defense, and metalworking and machinery industries.

Author contributions

Volkan Karakurt: Investigation, writing, review, explanation of wear mechanisms

Talip Çitrak: Performing of hardness measurements, heat treatment of samples according to specific parameters

Feyzanur Öztürk: Performing of wear tests

Orçun Zığındere: Interpretation of impact tests, review & editing, conceptualization

Gamze Nur Gözüak: Microstructure characterization

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