

Comparison of Tribological Behavior of Deep Cryogenic Treated Hot Work Tool Steel at Room and High Temperatures

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Abstract

The deep cryogenic treatment is a complementary operation that is done on a variety of tool steels aimed at improving their abrasion resistance and hardness. In the case of the H13 hot-work steel, which is widely used at high temperatures as a hot-deformation tool, it is needed to determine the efficiency of subzero treatment on it at the working temperature. In this regard, this paper focused on effect of deep cryogenic treatment on the wear behavior of H13 hot-work steel at the working temperature. Two types of quench-tempered and quenched-subzero-tempered samples were compared in this study. The microstructure of the samples was determined by scanning electron microscopy and their structure was determined by X-ray diffraction device after the cryogenic treatment. The wear test was performed at the working temperature of 600 °C (die temperature in the iron-based alloys forging process). The results indicated that the highest hardness rate has occurred in the quenched-subzero-tempered conditions, which it is about 26% higher than that of the quenched-tempered in the oil. Applying quenched-subzero-tempered operations has reduced the percentage of residual austenite by 10%. In addition, the fine, dispersed, and uniform sediments in this sample are more observed than the quenched-tempered samples. The improved weight lose were respectively achieved by 50% and 44% at temperatures of 25 and 600 °C respectively. The evaluation of the wear surfaces indicated that the abrasion dominant mechanisms were close-fitting and tribochemical.

Keywords: Deep cryogenic treatment; High-temperature abrasion; AISI H13 hot-work steel; Residual austenite; Hardness.

1. Introduction

Cryogenic treatment or subzero cooling includes the cooling of a part to low temperatures to change the austenite phase as a soft phase to martensite as a steel

hard phase. This operation is used as a permanent treatment, which includes the whole piece, at the end of the conventional thermal treatment and before the tempering operation ¹⁾. The cryogenic treatment is used as a complementary operation to extend the lifetime of the parts in many manufacturing industries such as cutting tools, cold-working tools, and drilling ^{2,3)}.

The hot work tool steels, which are symbolized as H-group steels in the AISI classification system, usually are able to withstand softening during prolonged or repeated exposure to high temperatures that is required for hot-working or die casting of metals ⁴⁾. Due to the frequency and efficiency, the tool steels of H10, H11, and H13 are further used, which the main usage involves the making of hot-working tools like forging molds, etc.

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The research on the deep cryogenic treatment of hot-working steels has so far been concentrated on the H13 steel ⁴⁾. Koneshlou et al. ⁵⁾ have studied the effect of deep cryogenic treatment on the microstructure, mechanical properties, and the abrasion behavior of the hot-working H13 steel. This operation has been done at a temperature of -72 °C and -186 °C. The results indicated that the application of the subzero thermal operation has many effects on the microstructure and causes the transformation of remaining austenite into martensite. With lowering the subzero operating temperature, the amount of martensite formed has increased. The greatest effect of the cryogenic treatment is on the wear behavior of the alloy. By deep cryogenic treatment, the more appropriate distribution of the martensite packs along with more and finer carbide particles and with a more uniform distribution, the more improved tribological behavior of the alloy at room temperature ^{4,5)}. The investigation of the machining behavior of the H13 hot-working steel under the cryogenic treatment has been done in another study ⁶⁾. The results of this study indicated that the minimum abrasion of the tool and suitable smoothness have been observed in the conditions of the cryogenic-tempered sample. The study done by Péreza et al. ⁷⁾ on the effect of the cryogenic treatment on the increased fracture toughness of the hot-working tool steels revealed that the best properties of the fracture toughness have been obtained during the cryogenic treatment at -186 °C and then the thermal annealing treatment. Makarian et al. studied the wear behavior of the H13 hot-working steel after cryogenic treatment, based on which, the abrasion behavior of this alloy has increased by 50% at room temperature after the cryogenic treatment and tempering ⁸⁾.

In the area of hot-working tools, the surveys show that an increased durability and a good wear resistance have been seen due to applying cryogenic treatment on them at room temperature. The remarkable thing about the hot-working tool steels is that they are used at high temperatures as their name suggests. For example, in the iron-based alloys forging operation the die temperature reaches about 550 to 650 °C. This is while all the studies done on the subzero operations of the H13 hot-working steel have not paid attention to this issue and examined the wear behavior of these steels at room temperature. In this paper, the aim is to evaluate the efficiency of cryogenic treatment on the H13 hot-working steel at its working temperature (600 °C) to examine the effectiveness of this operation at the forging temperature of the iron-based alloys.

Table 1. The chemical composition of the H13 steel used in this study (wt. %) obtained by emission spectroscopy.

C	P	S	Si	Mn	Cr	Mo	V	Fe
0.40	0.025	0.006	1.03	0.38	4.98	1.23	0.92	Rest

2. Materials and Methods

The H13 hot-working steel, which is widely used as a forging die for shaping iron and non-ferrous alloys in forging operation, was used in this study as the raw material. The chemical composition of this steel was determined using the emission spectroscopy method, which results are presented in Table 1.

The schematic of the applied heat treatment and the samples selected for microstructural and micro-hardness examinations are presented in Fig. 1. Initially, the samples were austenite-treated up to 1040 °C, then kept for 30 minutes at this temperature and quickly quenched in the oil. After this stage, they were exposed to liquid nitrogen (-186 °C) for 24 hours. The samples were then exposed to a temperature of 560 °C for 2 hours for annealing operation followed by cooling in the furnace environment. The points A, B, C, and D shown in Fig. 1 were used for microstructural and micro-hardness evaluations of the samples obtained from the operations. Finally, the wear behavior of the quenched-subzero-tempered samples was compared with the quenched-tempered samples at room temperature and 600 °C.

The X-ray diffraction experiments were performed with a Philips X-Ray Diffraction Spectrometer. The single-wavelength Cu K α beam with a wavelength of 54.21 Å⁰ was used by all the experiments. The time of each step, the step size, and the study range of 2 θ were selected as one second, 0.05 degrees, and 30 to 120 degrees, respectively.

The hardness of the samples was measured in HV30 Vickers scale according to ASTM E92 standard. The average of 15 measurements was considered as the sample

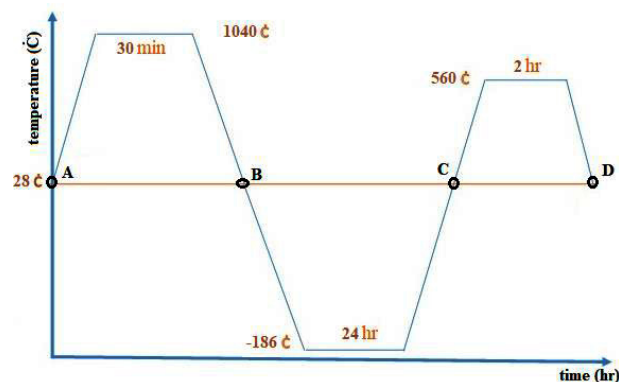


Fig. 1. The schematic of the heat treatment cycle with the specified points A, B, C, D used for microstructural and micro-hardness tests.

hardness. The microstructural studies were performed using an optical microscope, Olympus PGM3 model, and a scanning electron microscope, LEO 435 VP model. The wear behavior and the coefficient of friction of the samples were studied at room temperature and 600 °C. The criterion for choosing the test temperature for pin-on-disk wear testing was based on the fact that the die temperature is between 550 °C and 650 °C in the forging operations of iron-based alloys⁹⁾. First, the roughness of the discs was reduced by Abrasive paper of 1200, and then, the wear test was performed under the conditions of an applied load of 25 kg resulting from the load ability test on the quenched-tempered sample, the velocity of 0.03 m/s, and the wear distance of 500 m. A scanning electron microscope was used to determine the wear mechanism of wear traces and wear products after the test.

3. Results and Discussion

3.1. Variations of microstructures after heat treatment

Fig. 2 shows the microstructure of the samples in the

conditions of (A) primary raw material, (B) Quenched-subzero hot-working steel, (C) quenched-tempered, and (D) quenched-subzero-tempered. As shown in Fig. 2A, the structure of this steel without treatment includes perlite and carbide. The carbides are in a raw state as coarse and generally continuous carbides in the grains boundary. The carbides are marked as a white phase. It should be noted that the austenitization temperature is about 800 to 900 °C in the simple carbonated steels. This temperature in the H13 hot-working steel increases to above 1000 °C due to the solution of strong carbides in the austenite structure resulting from the presence of alloying elements such as Si, Cr, and Mo.

3.1.1. Quenched-subzero

The scanning electron microscopy image of the quenched-cryogenic treatment sample (Point C in Fig. 1) is shown in Fig. 2B. The structure consists of martensite packs. All the sediments are dissolved in the underlying structure due to dissolution at high temperature. Applying cryogenic treatment to the H13 hot-working steel has caused a large number of changes in its structure so that

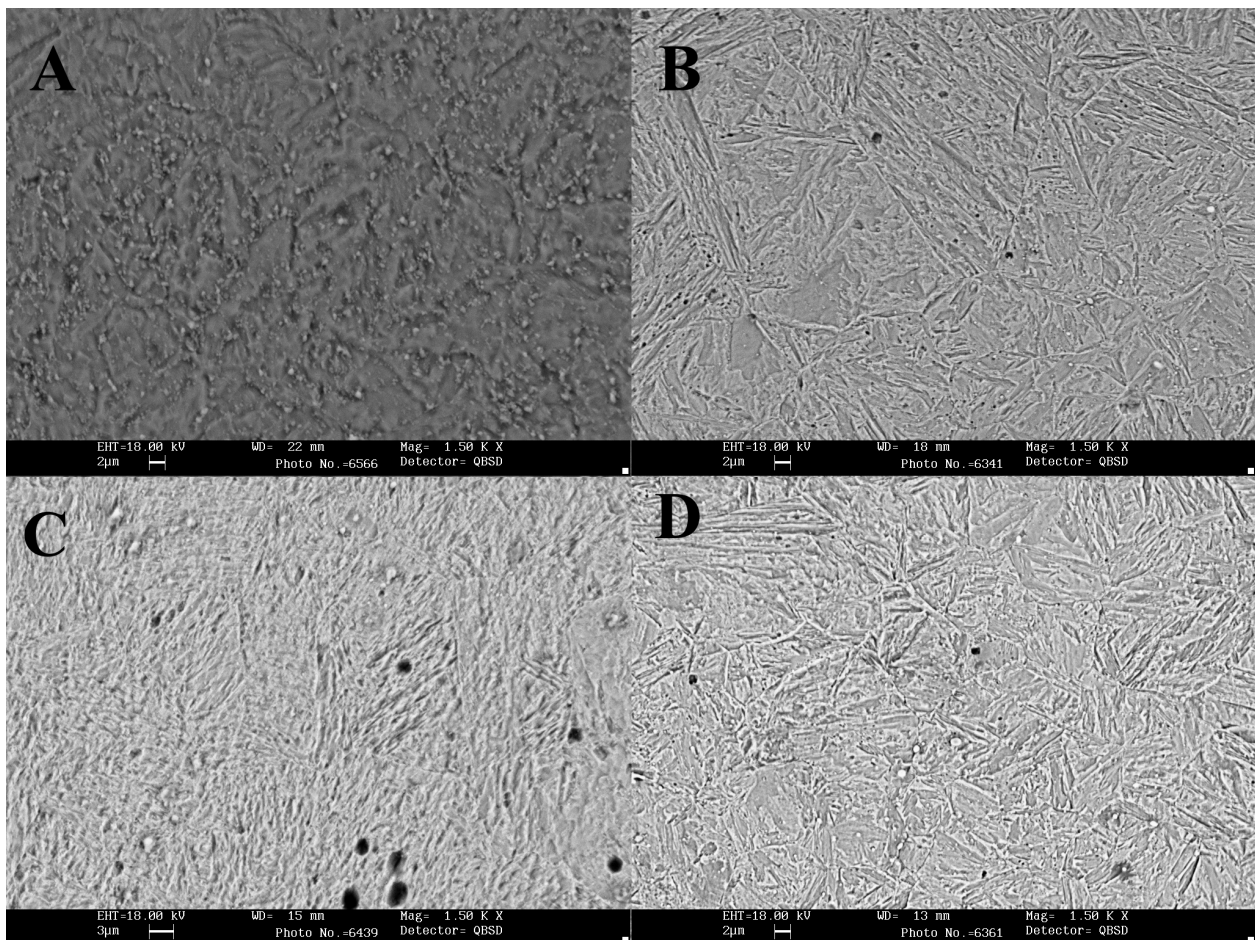


Fig. 2. The microstructure of H13 hot-working steel; A. Primary raw material, B. Quenched-subzero hot-working steel, C. Quenched-tempered, and D. Quenched-subzero-tempered.

the perlite structure and carbides have transformed into martensite packs in the original sample. The results of this research are consistent with the results of the proposed research worked by Koneshlou et al.¹⁰. According to the above mentioned conditions, the highest content of martensite has formed in the structure after cryogenic treatment.

3. 1. 2. Quenched-tempered

The structure of quenching-tempering treatment is shown in Fig. 2C. The resulting microstructure consists of the tempered martensite associated with alloyed carbides; however, the size of these carbide particles is also relatively large. A number of martensitic packs can be still seen that have remained unchanged during the tempering process. The presence of the residual austenite in this situation is unavoidable since it remains still at high temperature of M_f with quenching operation and the martensite cannot be formed at a high percentage. The presence of austenite in the tool steels is recognized as one of the major factors in reducing the hardness and wear resistance, which can limit the efficiency of these steels¹¹⁻¹³.

3. 1. 3. Quenched-subzero-tempered

Fig. 2D shows the microstructure of the quenched-cryogenic-tempered sample in accordance with point D in Fig. 1. Small sediments are found in the structure, which are located alongside the tempered martensite. One of the most recognizable cases in this Figure is the presence of the sediments morphology and the uniformed martensite tempered phase. This is highly important in the uniformity of mechanical properties and improving the behavior of the alloy against the impact loads. As stated, the absence of residual austenite in the quenched-subzero-tempered conditions (in accordance with Figs. 2B and 2D) is due to the exposure of the sample to the temperature conditions below M_f , which is determined more precisely by XRD analysis.

In addition to eliminating or reducing the amount of the residual austenite, highly fine carbides also precipitate in the cryogenic treatment, which have a suitable distribution compared to the quenched-tempered carbides (Figs. 2D and 2B). When the precipitation of these fine carbides occurs as the internal stresses increase due to the cooling of martensite to a very low temperature, the transformation of the austenite to martensite, and the difference in the thermal coefficient of contraction of different phases, and thus, crystalline defects such as dislocation or twin modes will appear. On the other hand, by decreasing the temperature to $-186\text{ }^\circ\text{C}$, the martensites crystalline lattice parameter decreases, and therefore, the super-saturation increases in the martensite with the decreased temperature. Hence, the lattice distortion and thermodynamic instability would increase in the martensite. Both of the above factors act as a driving force for

carbon atoms leaving the martensite and replace around the crystal defects to reduce the final energy of the system. Due to the very low diffusion rate of the carbon atoms at the subzero temperature, the placement of carbon atoms occurs so hard around the crystalline defects and the deposition of fine carbides is impossible. However, the carbon diffusion coefficient increases during heating of the samples from a subzero temperature to the room temperature. Thus, the carbon atoms induce the formation of fine clusters around the crystalline defects by being rejected from the martensite crystal lattice and a low-range diffusion, which will then form highly fine carbides. The deposition process in the deep cryogenic treatment involves the martensitic transformation and the creation of fine carbides similar to the tempering process in the quenched-tempered samples. However, there is a difference that they have finer particles and a more suitable distribution due to the lower temperature. It occurs owing to low diffusion rate resulting from the lower temperatures in the conditions of subzero operation.

The comparison of precipitates in Figs. 2C and 2D in two conditions of quenched-tempered in oil and quenched-cryogenic-tempered condition indicated that the sediments are finer and more dispersed in the quenched-cryogenic-tempered sample. In addition, the adhesion of precipitate to the ground is higher in the quenched-cryogenic-tempered treatment operations. In the deep cryogenic treatment, if enough time is provided, the low-range operation will lead to the formation of carbon and alloying elements near the imperfections. On the other hand, as the temperature decreases, the super-saturated carbon increases in the martensite, and thus, the lattice distortion and thermodynamic instability will increase in the martensite. Both of these factors act as a driving force for the moving of carbon atoms and the alloying elements around the crystalline defects¹⁴⁻¹⁶, which cause the formation of highly fine nuclei and lead to the formation of very fine carbide deposits even in nano-size scale in the process of tempering¹⁵⁻¹⁶.

There are two steps in the quenching-cryogenic-tempering operation. The first step is carried out up to $-130\text{ }^\circ\text{C}$, causing the increased hardness. In the second stage, which is done up to a temperature of $-186\text{ }^\circ\text{C}$, the isothermal decomposition of the initial martensite occurs in which nucleation is accrued for the precipitation of nano-scale carbides that caused the structure softening. In addition to their effect on increasing the hardness, the mentioned carbides also improve the abrasion resistance. In the following section, the elemental analysis of these sediments was discussed¹⁷.

3. 2. The precipitations analysis in the quenched-cryogenic-tempered treatment sample

The existence of fine precipitate with uniform

dispersion formed in the quenched-cryogenic-tempered treatment sample is shown in Fig. 3 with a higher magnification. Two types of precipitates are visible, the A precipitate is white and the B precipitate is dark. The results of the EDS analysis of A and B precipitates are presented in Fig. 4. According

to the results of the spot analysis, precipitate type A is in the form of complex consisting of vanadium, iron, chromium, and molybdenum elements.

The B-type precipitates consist of vanadium, chromium, iron, molybdenum, and titanium elements according to Fig. 3. The type "A" precipitates generally have a

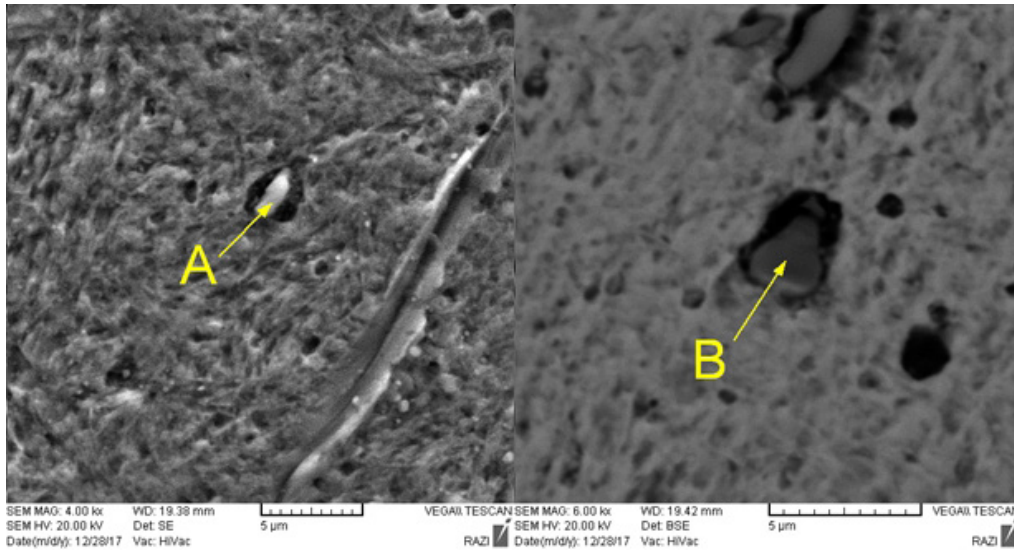


Fig. 3. The sediments observed in the quenched-subzero-tempered treatment sample, A-White sediment, and B- Dark sediment.

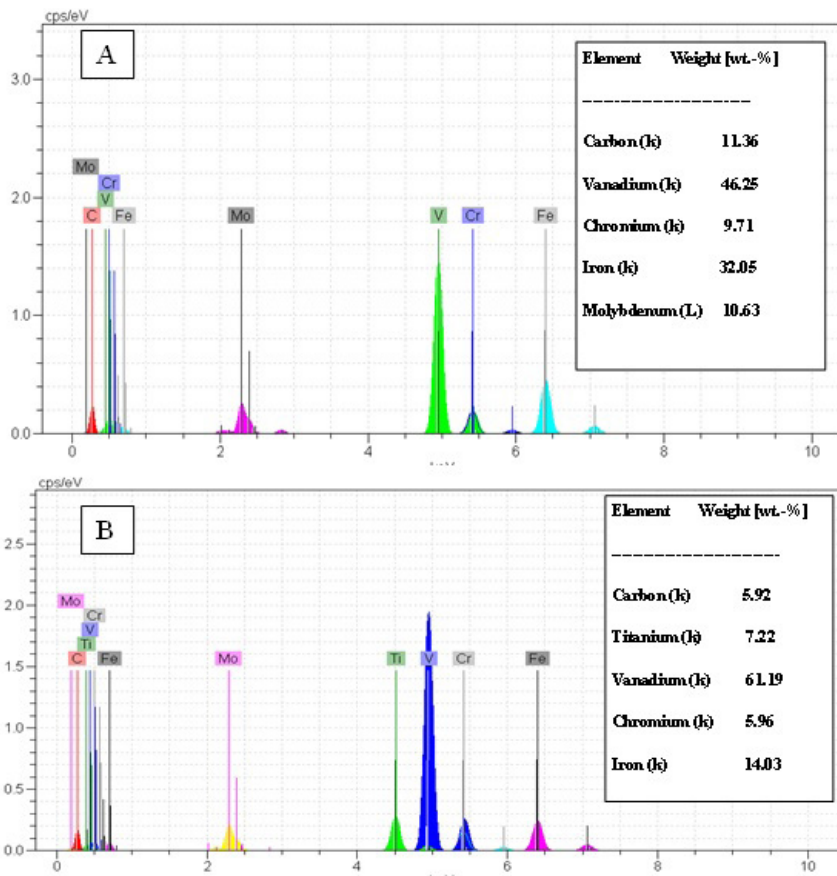


Fig. 4. The EDS analysis of sediment A and sediment B in Fig. 3.

smaller size with a more uniform dispersion compared to the B-type sediments.

3. 3. The structural analysis and study of the quenched-tempered samples and the quenched-cryogenic-tempered samples

The x-ray diffraction patterns of the quenched-tempered samples and the quenched-cryogenic-tempered sample are presented in Fig. 5. The percentage of the residual austenite has decreased by applying the cryogenic treatment for 24 hours. The percentage of the residual austenite was calculated according to Eq. (1). In this equation, $I^{hkl,\gamma}$ and $I^{hkl,M}$ are respectively the peak intensity (hkl) of austenite and martensite and $R^{hkl,\gamma}$ and $R^{hkl,M}$ are the relative intensity factors related to the crystallographic planes, respectively.

$$\gamma (\%) = \frac{(I^{hkl,\gamma}/R^{hkl,\gamma})}{[(I^{hkl,\gamma}/R^{hkl,\gamma})+(I^{hkl,M}/R^{hkl,M})]} \quad \text{Eq. (1)}$$

By calculating the residual austenite percentage, its rate has reduced from 10% in the quenched-tempered sample to below 5% in the quenched-cryogenic-tempered sample, which this amount cannot be detected by the XRD method. The reduction in the residual austenite by the cryogenic treatment has been also observed by other researchers^{15, 18, 19}.

3. 4. The evaluation of the quenched-tempered samples and the quenched-cryogenic-tempered sample

The hardness rates of the H13 hot-working steel samples in different thermal conditions are given in Fig. 6. The maximum hardness changes observed in the quenched-cryogenic sample as 750 HV, which has dropped to 170 HV by applying the tempering operation on this sample. The hardness differences have increased at minimum as 150% in the quenched-tempered sample and at maximum as 276% in the cryogenic treatment sample with no annealing operation compared to the initial sample. The highest hardness changes have

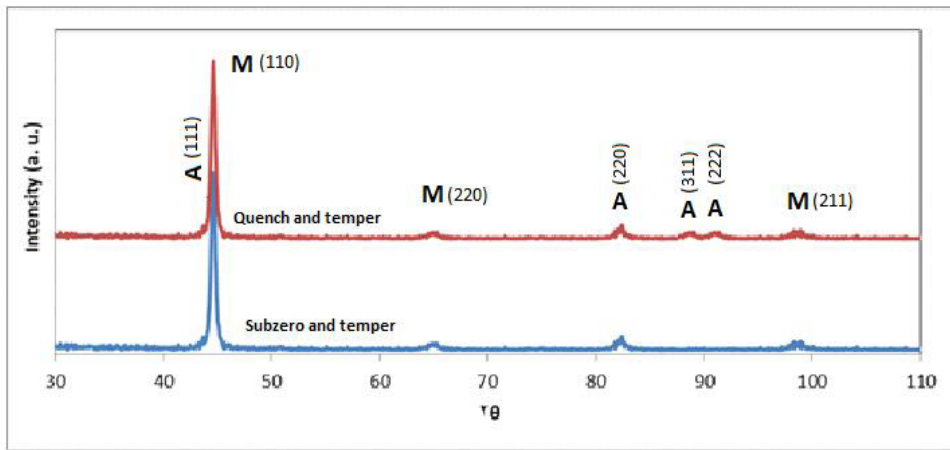


Fig. 5. The X-ray diffraction pattern of the quenched-tempered sample and the quenched-cryogenic-tempered sample.

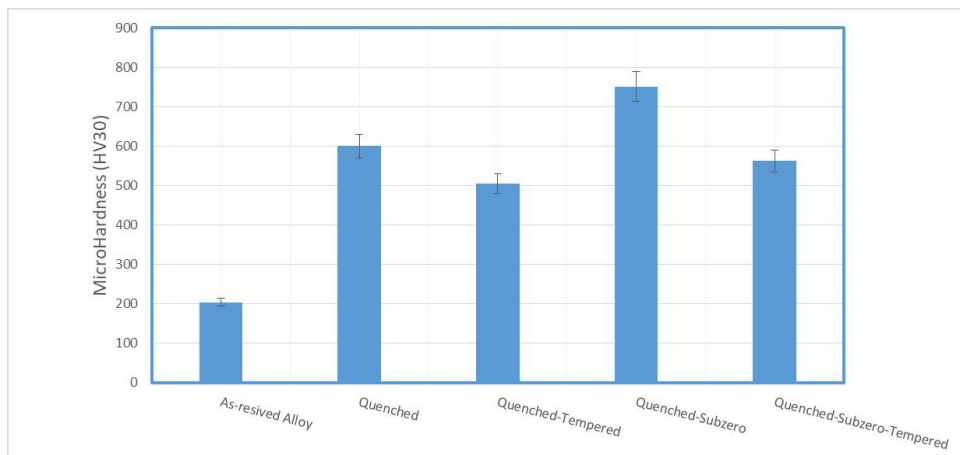


Fig. 6. The micro-hardness rates of the H13 hot-working steel samples in different thermal operation conditions.

been caused in the quenched-cryogenic-tempered treatment conditions, which it is about 26% higher than the quenched-tempered conditions in the oil. The reason for this increase can be attributed to the different percentage of the remaining austenite in these two conditions according to the results of the X-ray diffraction pattern (Fig. 5) and the calculations derived from Eq. (1). In other words, the increased hardness in terms of quenched-cryogenic-tempered treatment is due to the percentage of lower residual austenite in this sample. The comparison of two samples with final thermal treatment conditions, including tempered samples and quenched-cryogenic-tempered treatment indicated that the sample hardness with the quenched-cryogenic-tempered treatment conditions is about 11% higher than the hardness of the quenched-tempered sample in the oil. According to the microstructural analysis of the samples (Fig. 2C and 2D). The reason for this increased hardness can be attributed to a higher percentage of fine precipitates and a more uniform dispersion in the quenched-cryogenic-tempered sample. In summary, the increased hardness of the quenched-cryogenic-tempered sample compared to the quenched-tempered sample can be attributed to a decrease in the amount of residual austenite (Fig. 5), the increase in the volume fraction of carbides, their more appropriate distribution, and their fragmentation (Figs. 2C and 2D). The results obtained are consistent with the results achieved by other researchers^{15, 20, 21}.

3. 5. Examining the wear behavior of the quenched-tempered sample and the quenched-cryogenic-tempered sample

After examining the microstructure and the hardness of the samples, two samples were selected with a final operation, including the quenched-tempered sample and the quenched-cryogenic-tempered sample for tribological test at room temperature and 600 °C (die temperature in steels forging operation).

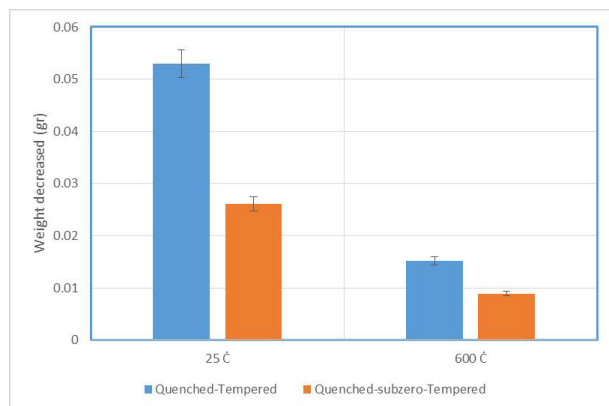


Fig. 7. The weight loss rate in the wear test for the quenched-tempered sample and the quenched-cryogenic-tempered sample at temperatures of 25 °C and 600 °C.

The amounts of weight loss in the wear test are given in Fig. 7 for the quenched-tempered sample and the quenched-cryogenic-tempered sample at temperatures of 25 °C and 600 °C. Performing the subzero operation on the H13 steel has increased the wear resistance of the sample by 50% and 44% at temperatures of 25 °C and 600 °C, respectively. The reason for this improvement involves the removal or reduction of the residual austenite, a more appropriate distribution, size reduction, and the increase in the volume fraction of carbides^{15, 20, 21}. In addition to removing the residual austenite as a soft phase, increasing the percentages of alloying elements and more uniform and homogeneous distribution of precipitates are among the reasons (Fig. 2D). The precipitation of fine carbides reduces the percentage of carbon and the alloy elements in the underlying phase. Therefore, the substrate phase toughness increases. The precipitation of hard fine carbides and the increased substrate toughness (martensite) reduce the wear rate in the deep cryogenic treatment^{20- 21}. In a research conducted on steel D6, an increase in the wear resistance in the deep cryogenic operations is respectively 5-11% and 39-68% compared to the conventional thermal operations. The reasons for the increased wear resistance are reported to be as the association with the transformation of residual austenite to martensite in the superficial cryogenic operations, the complete conversion of the remaining austenite to martensite, and the deposition of secondary carbides in the deep cryogenic operations²².

The changes in the friction coefficient in terms of distance during wear test in the quenched-tempered sample and the quenched-cryogenic-tempered sample

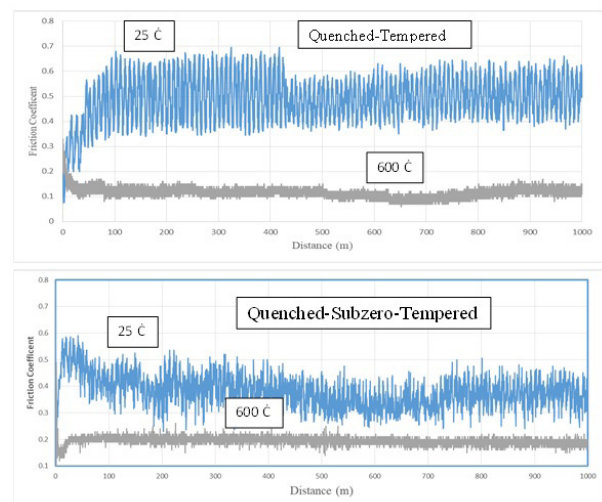


Fig. 8. Changes in the coefficient of friction in terms of distance during the wear test in the quenched-tempered sample (upper image) and the quenched-cryogenic-tempered sample (lower image) at temperatures of 25 °C and 600 °C.

at temperatures of 25 °C and 600 °C are shown in Fig. 8 respectively.

Also, Table 2 shows the coefficient of friction values with their fluctuations in two heat treatment conditions used in this study and at the temperatures of 25 °C and 600 °C. In accordance with Fig. 8 and Table 2, the coefficient of friction at a temperature of 600 °C is lower than the room temperature in both thermal treatment conditions. The reason for this can be attributed to the oxidation of the wear surface and slipping on the oxides, which reduce the friction coefficient. The greater fluctuation rate in the quenched-tempered sample than the quenched-cryogenic-tempered sample is shown in Table 2. This factor can be attributed to the three-body wear that occurs by placement of precipitates in the abrasion zone. Due to the larger precipitates in the quenched-tempered sample (Fig. 2C) compared to the quenched-cryogenic-tempered sample (Fig. 2D) and their lower adhesion to the substrate, the possibility of existence of coarser precipitates in the abrasion path has become higher in the quenched-tempered samples. This has led to an increase in the friction coefficients fluctuations in these samples.

In general conditions, the value of coefficient of friction in the quenched-tempered sample is slightly lower than the quenched-cryogenic-tempered sample. This can be attributed to the lower hardness of the quenched-tempered sample surface, which has caused more wear and a lower coefficient of friction in these specimens.

The surface wear of the samples was studied by SEM. Fig. 9 shows the wear trace of the quenched-tempered and quenched-cryogenic-tempered samples at 25 °C. The wear mechanism in the samples is an adhesive wear one, which has reduced due to the deep cryogenic treatment. The studies by

researchers have shown that the increased volume fraction and the reduced spacing between the carbides in the tool steels have reduced the adhesive wear rate ²³⁾.

Fig. 10 shows the wear surfaces of the quenched-tempered and the quenched-cryogenic-tempered samples at 600 °C. The wear mechanism in the samples is an

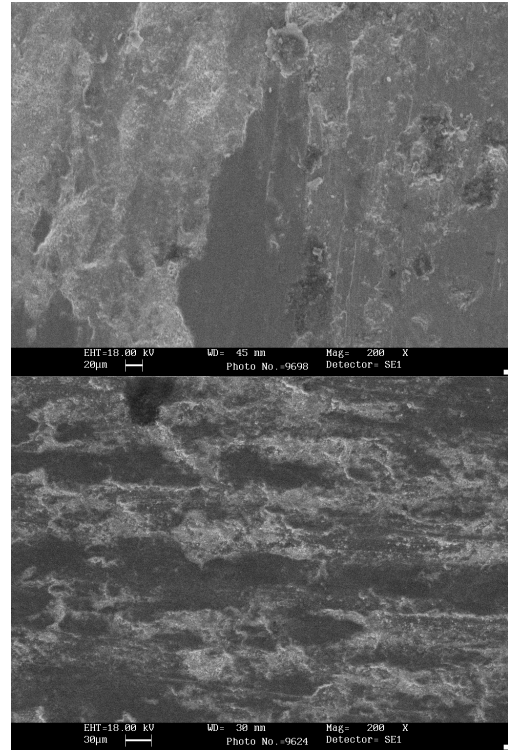


Fig. 9. The wear surfaces of the quenched-tempered sample (image above) and the quenched-cryogenic-tempered sample (image below) at 25 °C.

Table 2. The value of coefficients of friction with their fluctuations in the two thermal operation conditions used in this study and at temperatures of 25 °C and 600 °C.

600 °C	25 °C	
0.02 ± 0.15	0.1 ± 0.5	quenched-tempered sample
0.01 ± 0.2	0.05 ± 0.4	quenched-cryogenic-tempered sample

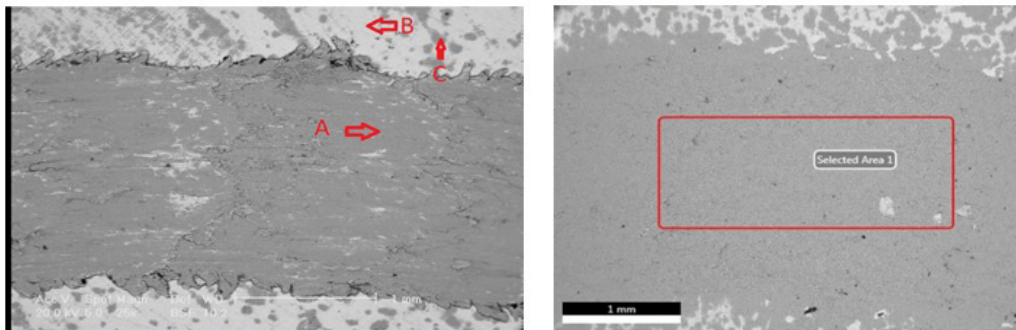


Fig. 10. The wear-surface of the quenched-tempered sample (image above) and the quenched-cryogenic-tempered sample (image below) at 600 °C. Dark and bright areas on the wear surface.

adhesive wear along with the tribochemical wear. Also, as it is clear, the quenched-tempered sample has a deeper wear traces than the quenched-cryogenic-tempered sample. Although there is no sign of the difference due to the residual austenite resulting from tempering at this temperature, the presence of fine and dispersed sediments in the quenched-cryogenic-tempered has increased the re-

sistance to wear and tear and less oxidation in this sample compared to the quenched-tempered sample. The different areas in Fig. 10 can be divided into two dark and bright areas. The EDAX results from the indicated areas are shown in Fig. 11. According to the results of Fig. 11, the dark regions have a high percentage of oxygen, which suggest the oxidizing of these areas. The oxygen content is much

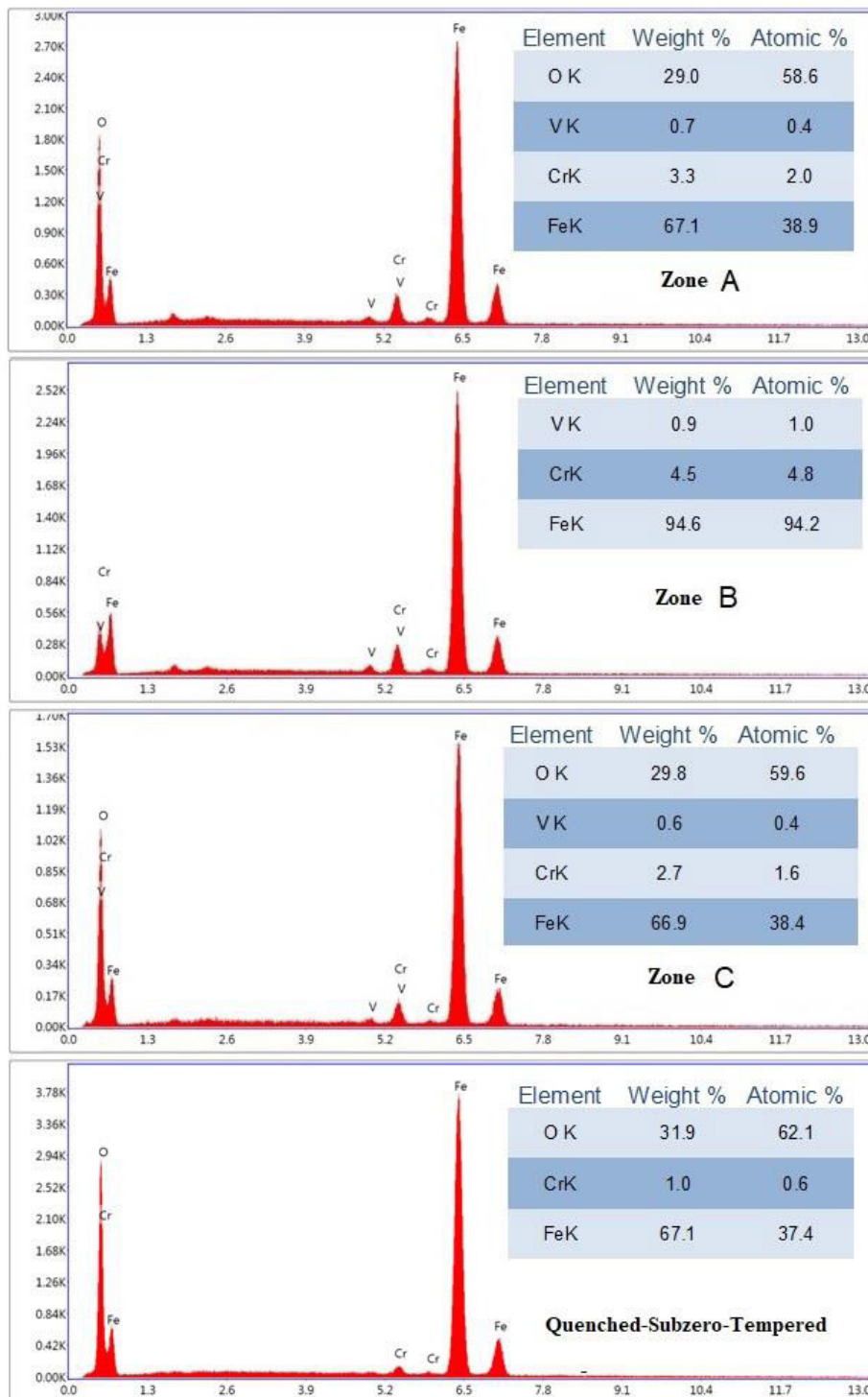


Fig. 11. The EDAX analysis results from the wear surface of the quenched-tempered sample of areas A, B, and C and the quenched-cryogenic-tempered sample at 600 °C; Dark and bright areas on the surface of the abrasion.

higher in the wear area than in the surrounding areas.

4. Conclusion

- In this research, the deep cryogenic treatment was successfully applied, which improved the wear properties by 50% and 44% compared to the quenched-tempered samples respectively at temperatures of 25 °C and 600 °C.
- By comparing the properties of the initial H13 hot-working steel (with perlite and cementite structure) and applying the quenched-tempered operations (with the structure of the martensite and the residual austenite), the hardness was increased 150%, while it showed a maximum increase of 276% by applying the quenched-cryogenic-tempered treatment.
- The highest hardness was obtained in the quenched-cryogenic-tempered treatment that was about 26% higher than the quenched-tempered conditions.
- The application of quenched-cryogenic-tempered treatment to the H13 steel reduced the percentage of residual austenite by 10%. Also, the fine, dispersed, and uniform sediments in this sample were formed more than the quenched-tempered sample.
- Examining the wear surfaces indicated that the dominant mechanism of wear at high temperature was adhesive and tribochemical. Due to the cryogenic operation, the adhesive wear reduced due to the increase and even distribution of carbides. In addition, comparing the wear surface of the quenched-tempered sample with the quenched-cryogenic-tempered sample showed that more surface deformation has occurred at a higher temperature in the former, which is due to the positive effect of carbides and their distribution in the sample with the quenched-cryogenic-tempered conditions.
- The wear traces of the samples at high temperatures indicated the formation of oxides at the abrasion surface. This reveals the presence of tribochemical abrasion that the formation of superficial oxide has been less observed in areas far from the abrasion zone. Moreover, the thickness of this oxide layer at the abrasion surface of the sample with the quenched-tempered thermal treatment is greater than the sample with the quenched-cryogenic-tempered conditions.

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