Investigation on High Temperature Wear Resistance of Cryotreated Hot Work Tool Steel

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ABSTRACT

Deep sub-zero treatment is a complementary operation performed on all types of tool steels, carbonized and high-speed steels to improve wear resistance and hardness. Among these tool steels, H13 is a hot work tool steel that have an extended application in industry as a hot deforming tool. This paper investigates the wear behavior of deep cryogenic treated H13 hot work steel at operating temperature. Two quench-tempered and quench-subzero-tempered samples are compared. The microstructures of the specimens were determined by scanning electron microscopy, and the structures were determined by X-ray diffraction. Vickers hardness used for determining hardness after each treatment. The wear test was carried out at 250°C (mold temperature on forging of copper base alloys). Finally, the wear surface was examined by scanning electron microscope equipped with EDS analyzer. The results show that the highest hardness was in quench-subzero-tempered condition which is about 26% higher than the quench-tempered in oil conditions. This is due to the formation of fine, dispersed and uniform precipitates and higher martensite percentage in quench-subzero-tempered sample compared to quench-tempered sample. Quench-subzero-temper operation reduced the residual austenite percentage by 10% and improved the wear properties by 36% at 250° C. Examination of wear surfaces indicates the presence of oxidized surfaces adhered to the wear surface in the form of abrasive particles. These oxide levels were lower in quench-subzero-tempered sample than quench-tempered sample.

1-Introduction

Sub-zero cryo-treatment on steels includes cooling to low temperatures in order to change the retained austenite phase as the soft phase to martensite as the hard phase. As a permanent treatment that affects the bulk of the materials, it is done at the end of the conventional heat treatment and before the tempering treatment. In the production process, the cryo-treatment has not yet been incorporated into the production cycle as a conventional operation, but in some parts of the aircraft, automobile and electronics industries in the United States, China, and other developed countries it is used to improve the

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wear resistance and dimensional stability of the components [1-3]. However, in many small manufacturing industries, such as cutting tool manufacturers, cryo-treatment is also used as a complementary operation to extend the service life of the parts [4, 5].

Hot work tool steels, which are labeled as H-group steels in the AISI classification system, usually have the ability to withstand softening during prolonged or repeated exposure to high temperatures used for hot work or die casting of metals or other materials. H-type steels are divided into three subgroups according to the alloying method to obtain high hardness in hot state [6]:

Chromated hot work steels contain 0.5 nominal chromium and significant amounts of other elements including silicon, molybdenum and vanadium, tungsten hot work steel and Molybdenum hot work steels. Numerous alloying elements are also added to the tungsten and molybdenum hot work steels and the wear resistance of these steels is fairly better than the chromated steels. Due to the abundance and capability of production at home country and abroad, H10, H11 and H13 hot work tool steels are more common, mostly used to make hot work tools such as blacksmithing molds. In this regard, research on the sub-zero operation of these steels has so far focused on H11 and H13 steels [7–15].

Koneshloo et al. [9] investigated the effect of sub-zero operation on the microstructure, mechanical properties and wear behavior of H13 hot work steel. The operation was performed at -72°C and -196°C. The results indicate that sub-zero heat treatment has many effects on the microstructure and causes the residual austenite to become martensite. By decreasing the sub-zero operating temperature, the amount of martensite increased. The most effect of this treatment is on the wear behavior of the alloy. Proper distribution of martensitic packets, fine and uniform dispersion of carbide particles has caused this phenomenon, so that the deep sub-zero treatment improves the wear behavior of the alloy at ambient temperature.

The machining behavior of cryotreated H13 hot work steel has been investigated in another study [10]. The results indicated that the least amount of tool wear and proper filtration was observed in sub-zero-tempered sample. Research by Priz et al. [11] on sub-zero operation on fracture toughness of hot work tool steels indicates that the best fracture toughness properties were obtained at sub-zero operation at -196°C and then annealing operation. Adam et. al investigated the wear behavior of H13 hot work tool steel at sub-zero operation, the wear behavior of this alloy increased by about 50% at ambient temperature after sub-zero temper operation [12].

In the case of hot work tool steels especially H13, studies have shown an increase in hardness and wear resistance due to sub-zero operation at ambient temperature [9–15]. The great thing about hot work tool steels is that as they are called, they are used at high temperatures. For example, in the casting operation of copper alloys, the mold temperature reaches 250 °C to 350 °C. This is while all the researches and published articles on cryogenic of H13 hot work steel have not addressed this issue and have only investigated the wear behavior of these steels at ambient temperature. The purpose of this study was to investigate the efficiency of sub-zero treatment on H13 hot work tool steel at its operating temperature (250°C) in order to investigate the efficiency of copper base alloys at forging temperature.

2. Materials and Methods

H13 hot work tool steel, widely used as a forming tool in ferrous and non-ferrous alloys, was used as the starting material in this study. The chemical composition of this steel was determined by emission spectroscopy method, the results of which are presented in Table 1.

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

Fe	C	P	S	Si	Mn	Cr	Mo	Ni
retain	0.4	0.025	0.006	1.03	0.38	4.98	1.23	0.2
Al	Co	Cu	Ti	V	W	Sn	As	Nb
0.017	0.019	0.198	0.013	0.92	0.033	0.007	<0.001	----

The samples were austenitized up to 1040°C for preparation of quench-subzero-tempered samples, held at this temperature for 30 min and were quickly quenched in oil. After that, samples were immersed in liquid nitrogen (-196°C) for 24 hours. The samples were then annealed at 560°C for 2 hours. Next they were cooled in the furnace environment. The cycle used to prepare quench-tempered samples involves holding at 1040°C for 30 minutes and then quenching in oil, and then, the samples were then annealed at 560°C for 2 hours and cooled in the furnace. In this research, the microstructure and wear behavior of quench-subzero-tempered samples were compared with quench-tempered samples of H13 steel.

The samples hardness was measured by a Vickers HV30 scale based on ASTM E92 standard. An average of 15 measurements was considered. Microstructural studies were performed using Olympus PGM3 light microscope and LEO 435 VP scanning electron microscope.

X-ray diffraction analyses were performed with a Philips XPERT diffractometer. All experiments used single-wave Cu Kα radiation with a wavelength of 1.554 angstroms. The time of each step was one second; the step size was 0.05 °, and the study area 2θ, 30 to 120 °.

The wear behavior was performed by a pin on disc equipped with a temperature control furnace. The wear behavior and friction coefficient of the samples were studied at 250°C. The criterion for selecting the pin-to-disk tests temperature is based on the assumption that in forging of brass alloys, the die temperature between 200°C and 300°C [16]. In this test, pin was a bearings steel ball with a hardness of 65 Rockwell C (5 mm in diameter and 5 cm in length) and the disks (4 cm in diameter and 7 mm in thickness) were treated samples. First, the roughness of the discs was reduced by using with 1200 emery paper. The wear test was selected at load of 25 kg, wear rate of 0.03 m/s and wear distance of 1000 m. These parameters were selected according to the actual conditions of the brass alloy forging operation. In the brass valve forging operation [16], the mold movement is about 2 cm and takes 0.75 seconds. Therefore, the velocity in real conditions is about 0.03 m/s. Loadability test on quench-tempered specimen was used to select the applied force. Scanning electron microscope equipped with EDS analyzer was used to determine the wear mechanism of wear surfaces and wear products.

3. Results and Discussion

3.1. Microstructure of Hot Work Steel Quench-Tempered in Oil

According to the above, in order to identify and compare the behavior of H13 steel under quench -subzero-tempered and quench-tempered conditions; quench-tempered condition is used by industry, have been first introduced. The structure of the quench-tempered treatment of this alloy is illustrated in Figure 1. The microstructure consists of tempered martensite with alloy carbides with an average size of 1.24±0.8 μm. The presence of residual austenite in these conditions is inevitable, because the quenching operation in oil is at a higher temperature than M_f and it is not possible to form a high percentage of martensite. The presence of residual austenite in tool steels is one of the major factors in reducing the hardness and wears resistance of these well-known steels and reduces their wear test [17-19]. For this reason, it is predicted that the use of quench-subzero-tempered treatment due to removal of residual austenite in these steels will increase the hardness and wear resistance and increase the efficiency of these steels.

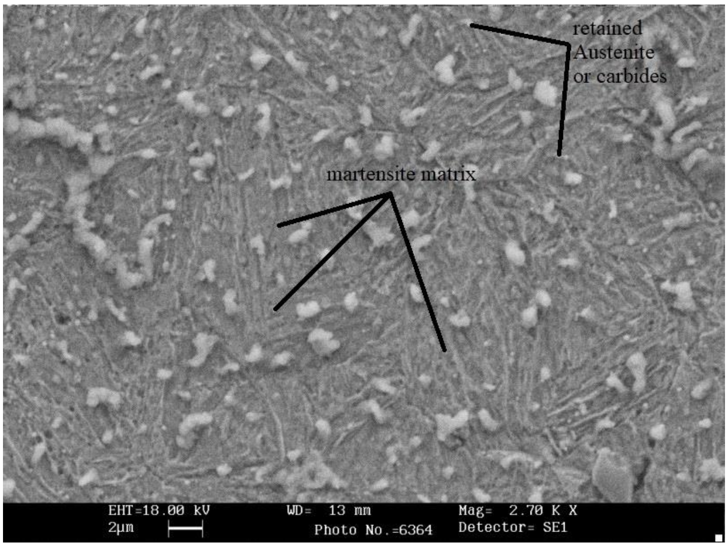


Fig. 1. Microstructure of the Quench-Tempered Sample in Oil.

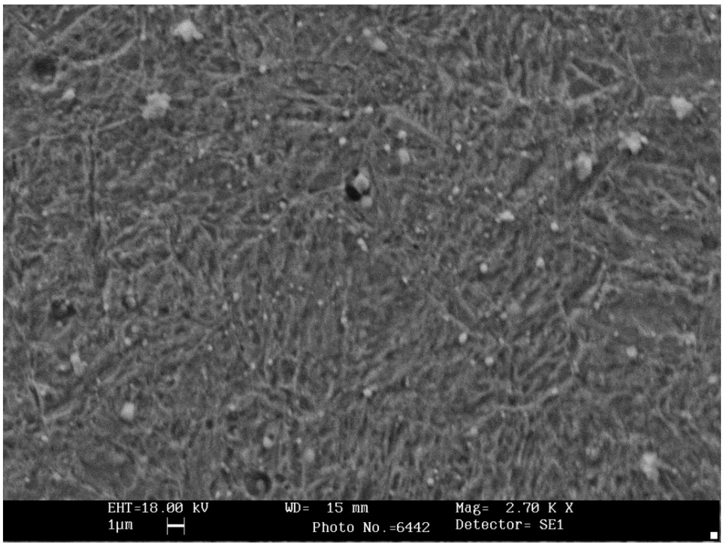


Fig. 2. Microstructure of Quench-subzero-tempered Sample.

3.2. Microstructure of hot work tool steel quench-subzero-tempered sample

Figure 2 shows the microstructure of the quench-subzero-tempered sample. According to this figure, fine precipitates are observed in the structure, which are located next to the tempered martensite. The average size of these precipitates is 0.3 ± 0.05 µm in these conditions. One of the recognizable cases in this form is the existence of precipitate morphology and uniformly tempered martensite phase, which is important in the consistent of mechanical

properties and improvement of alloy behavior against impact loads. As it was mentioned, the absence of residual austenite in quench-subzero-tempered operation conditions is due to the sample being subjected to temperature below M_f which is discussed in the next section.

3.3. Precipitates in Quench-Subzero-Tempered Sample

As discussed above, the existence of uniformly dispersed fine precipitates formed in the Quench-subzero-tempered sample is shown in Fig. 3 at higher magnification. The results of EDS analysis of precipitate A are shown in Figure 4. According to the results of point analysis, precipitates are complex and composed of vanadium, iron, chromium and molybdenum.

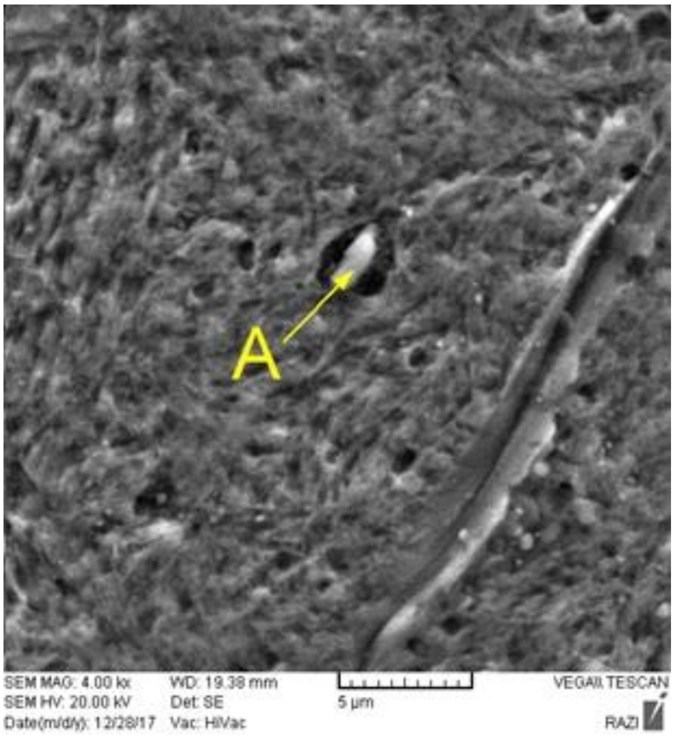
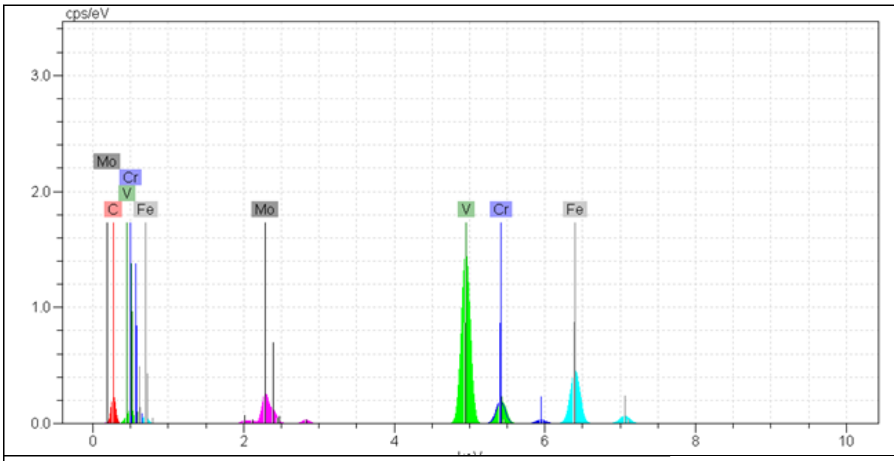


Fig. 3. precipitate observed in quench-subzero-tempered sample.



Element	Series	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]
Carbon	K series	9.36	10.31	34.56
Vanadium	K series	41.25	45.45	35.93
Chromium	K series	6.71	7.39	5.72
Iron	K series	25.05	27.60	19.90
Molybdenum	L series	8.40	9.26	3.89

Fig. 4. EDS analysis of precipitate A in Fig. 3.

3.4. Structural Examination of Quench-Tempered and Quench-subzero-tempered Samples

The X-ray diffraction pattern of quench-tempered and quench-subzero-tempered samples is shown in Fig. 5. As can be seen, the percentage of residual austenite decreased by subzero operation for 24 hours. The percentage of residual austenite was calculated according to Equation 1. In this relation, $I^{hkl,\gamma}$ and $I^{hkl,M}$ are the peak intensities (hkl) of austenite and martensite, and $R^{hkl,\gamma}$ and $R^{hkl,M}$ are the relative intensity factors for the crystallographic plates, respectively.

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

The position of 110 matrensite and 111 austenite is the same, so that for the calculation of retained austenite 220 peaks of austenite and matensite used. By calculating the percentage of residual austenite, the amount of austenite in quench-tempered samples was reduced from 10% to below 5% in quench-sub-zero-tempered samples. This is not detectable by the XRD method. A decrease in the amount of residual austenite by sub-zero operation has also been observed by other researchers [20].

3.5. Hardness of Quench-Tempered and Quench-Sub-zero-Tempered Samples

The hardness value of H13 hot work steel specimens under different heat treatment conditions is shown in Figure 6. The maximum hardness in the subzero-quenched sample is 750 HV, which was reduced to 570 HV by applying tempering operation on it. In all samples, due to the quench-tempered treatment, and transformation of perlite and Cementite to martensite, hardness increased at least 150% compared to as-received sample and up to 276% at sub-zero operation with no annealing. Hardness in quench-subzero-tempered sample conditions is 26% higher than quench-tempered in oil. The reason for this increase can be attributed to the different percentage of austenite remaining in these two conditions. In other words, the increase in hardness in quench-subzero-tempered conditions is due to the lower residual austenite in this sample (Fig. 5). Comparison of the two samples with the final heat treatment conditions including the tempered samples and the quench-subzero-tempered operation indicates that the hardness of the sample with quench-subzero-tempered operation conditions is about 11% higher than the hardness of the quench-tempered sample in oil. The reason for this increase in hardness, according to the results of microstructure analysis of samples (Figures 1 and 2) can be attributed to the higher percentage of fine precipitates and more uniform dispersion in quench-subzero-tempered samples. In summary, the increase in hardness in the quench-subzero-tempered sample compared to the quench-tempered sample was attributed to a decrease in residual austenite content, an increase in the volume fraction of carbides, a more appropriate distribution and a finer grading. The results are consistent with those obtained by other researchers [21, 22].

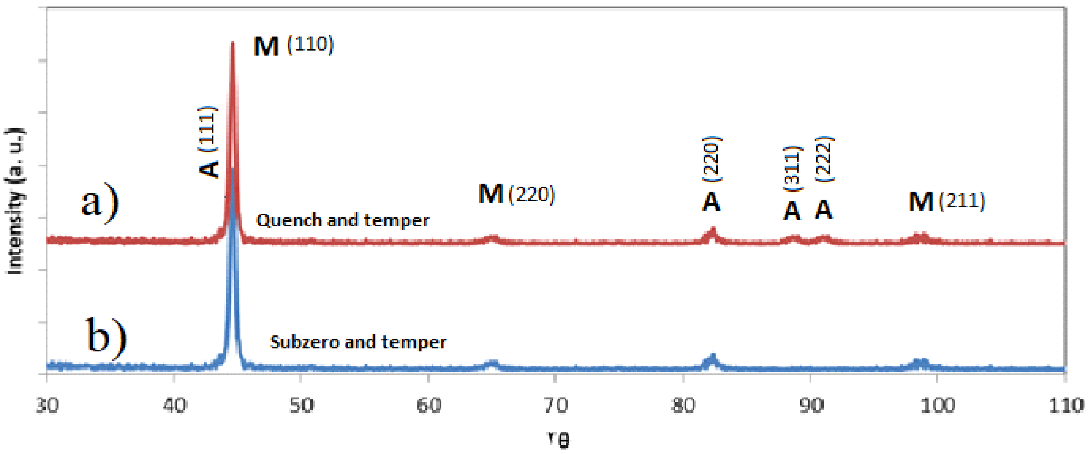


Fig. 5. X-ray diffraction pattern of a) the quench-tempered and b) quench-subzero-tempered samples.

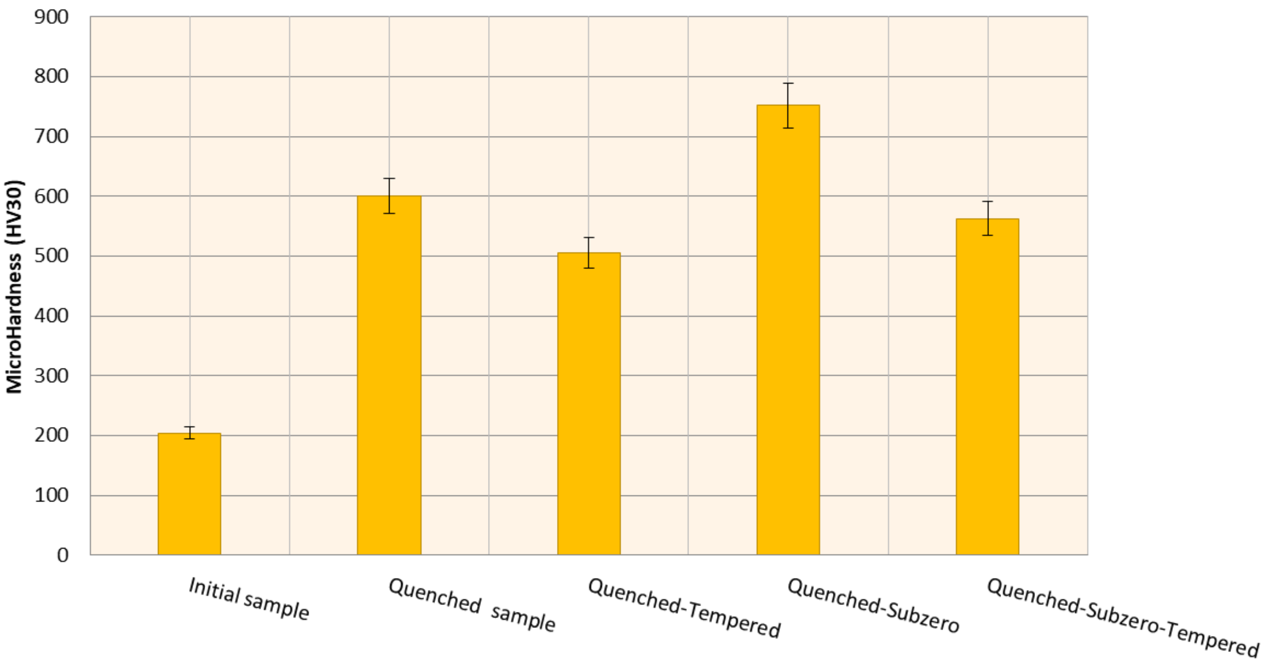


Fig. 6. Microhardness of hot work tool H13 steel samples under different heat treatment conditions.

3.6. Wear Behavior of Quench-Tempered and Quench-Subzero-Tempered Samples

After examining the microstructure and hardness of quench-tempered and quench-subzero-tempered samples, wear test was performed on them at 250°C (die temperature in forging of copper based alloys). The weight loss in the wear test for the quench-tempered sample and the quench-subzero-tempered sample at 250°C is given in Fig. 7. Sub-zero treatment on H13 tool steel increased the wear resistance of the sample by 38%. The reason for this improvement is the removal or reduction of residual austenite (Fig. 6) and a more appropriate distribution, finer grains and increased volume fraction of carbides. Also the increase in wear resistance in deep sub-zero operations, in addition to the removal of residual austenite as a soft phase, is the increase in the percentage of alloy carbides and the more uniform and homogeneous distribution of these sediments. The deposition of fine carbides reduces the percentage of carbon and alloying elements in the matrix phase. Therefore, the toughness of the matrix phase increases. The precipitation of fine-grained carbides and increased background hardness (martensite matrix) reduce wear rates in deep sub-zero operations. In the study on D2 steel, the increase in wear resistance in surface and deep sub-zero operations was 5-11% and 39-68%, respectively, compared to conventional heat treatment. The reason for the increased wear resistance has been reported as the transformation of a part of the residue austenite to martensite in shallow sub-zero operations (at -80 °C) and the complete conversion of residual austenite to martensite and deposition of secondary carbides in deep sub-zero operations (at -196 °C) [3].

The variation of friction coefficient by distance during wear test in the quench-tempered sample and quench-subzero-tempered sample are shown in Fig. 8. The average friction coefficient in the quench-tempered sample and the quench-subzero-tempered sample were 0.55±0.05 and 0.45±0.05, respectively. In general, the friction coefficient in the quench-subzero-tempered sample is slightly lower than the quench-tempered sample. This can be attributed to the higher precipitation of precipices and uniformity of the structure in the quench-subzero-tempered sample, which resulted in lower friction coefficients in these samples. The presence of precipates and martensitic phase make the surface adhesion between the pin and the disk less likely, and the two-layer sliding on each other is easier.

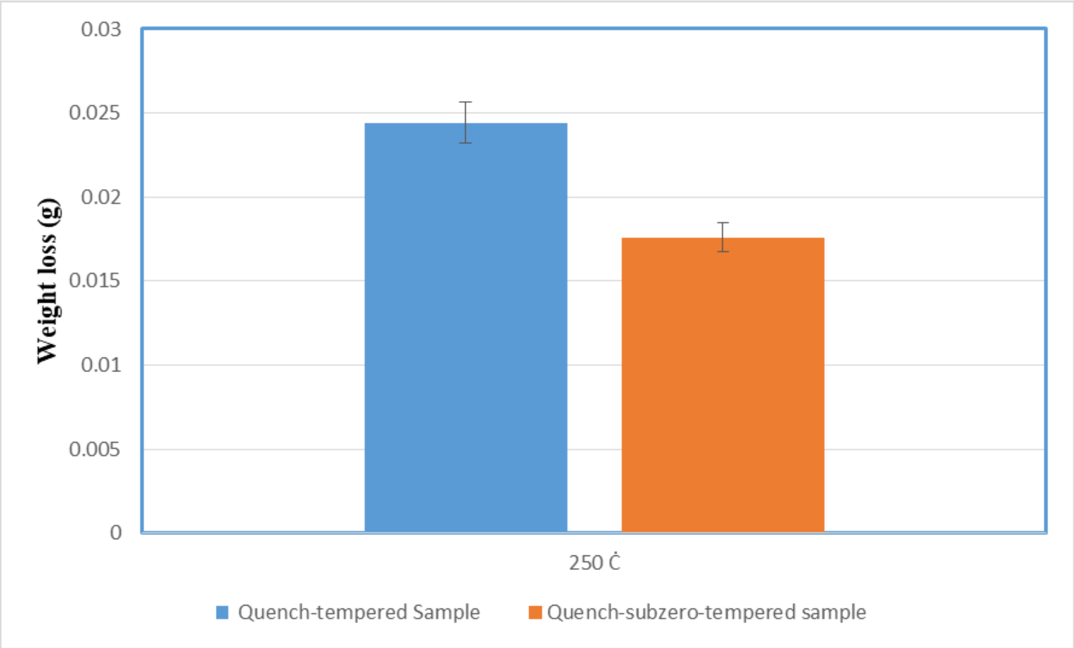


Fig. 7. Weight loss for quench-tempered and quench-sub-zero-tempered samples at 250°C.

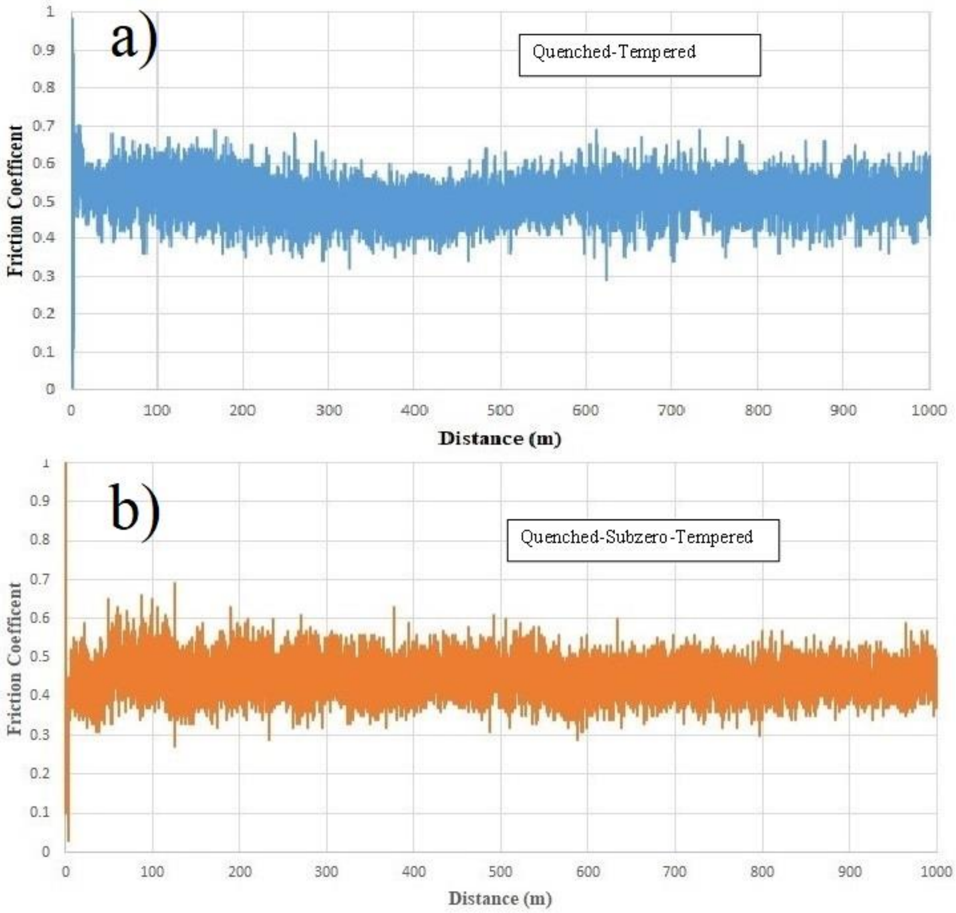


Fig. 8. friction coefficient changes per distance during wear test at 250°C, a) quench-tempered sample, and b) the quench-subzero-tempered sample.

The wear trace of the specimens was studied by scanning electron microscopy to determine the wear mechanism. Figure 9 shows the wear traces of the quench-tempered sample and the quench-subzero-tempered sample at 250°C. According to this figure, it appears that the particles cut off the surface are re-positioned on the surface as a result of being sandwiched between the wear surfaces and adhered to the disk surface. The amount of this adhesion is higher in quench-tempered samples than quench-subzero-tempered samples; this is due to the formation of oxide micron layers and suggests the possibility of a tribo-chemical wear mechanism due to elevated temperatures at the wear surfaces and their oxidation. The results of EDS analysis of the two wear surfaces as shown in Fig. 10 indicate the presence of oxygen on the surface. The amount of this oxygen in the quench-tempered sample is higher than the quench-subzero-tempered sample. According to above information, in the samples subjected to deep sub-zero operation, the adhesive wear of the carbide is reduced by increasing the volume fraction of carbides, their finer morphology and eventually increasing the hardness (due to the increase of martensite and fine carbide sediments).

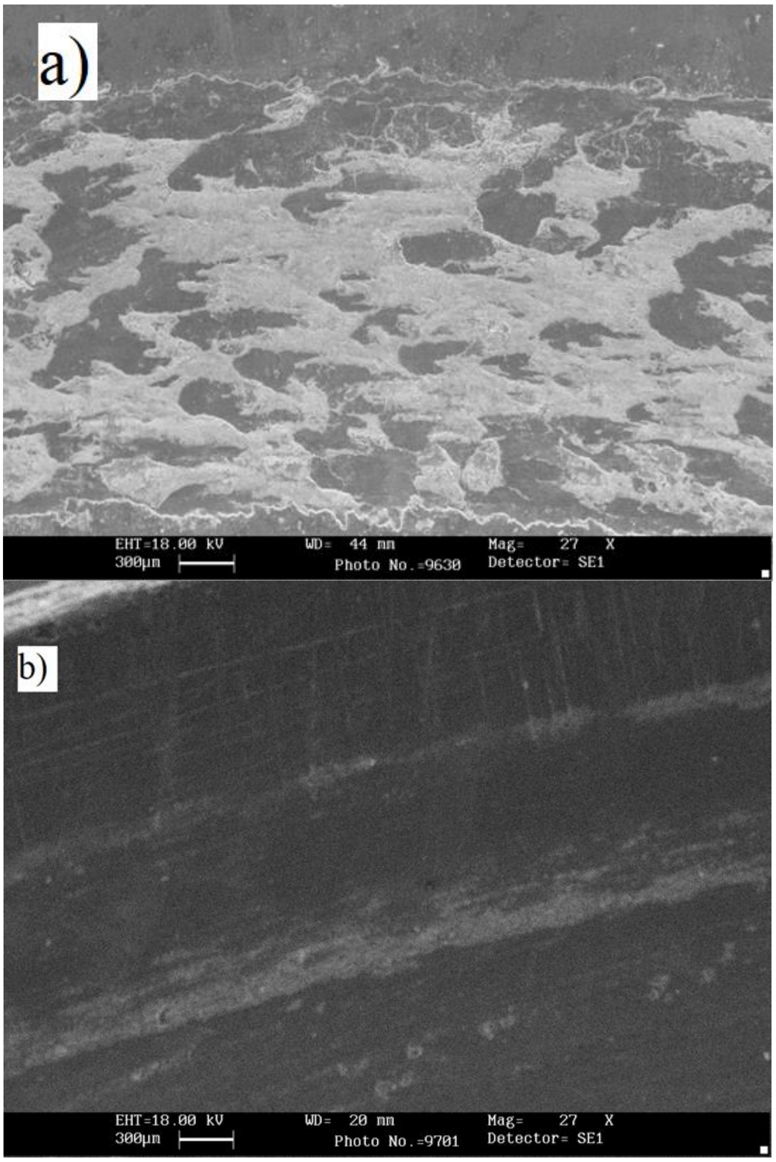


Fig. 9. Wear traces of a) quench-tempered sample, and b) quench-subzero-tempered sample at 250°C.

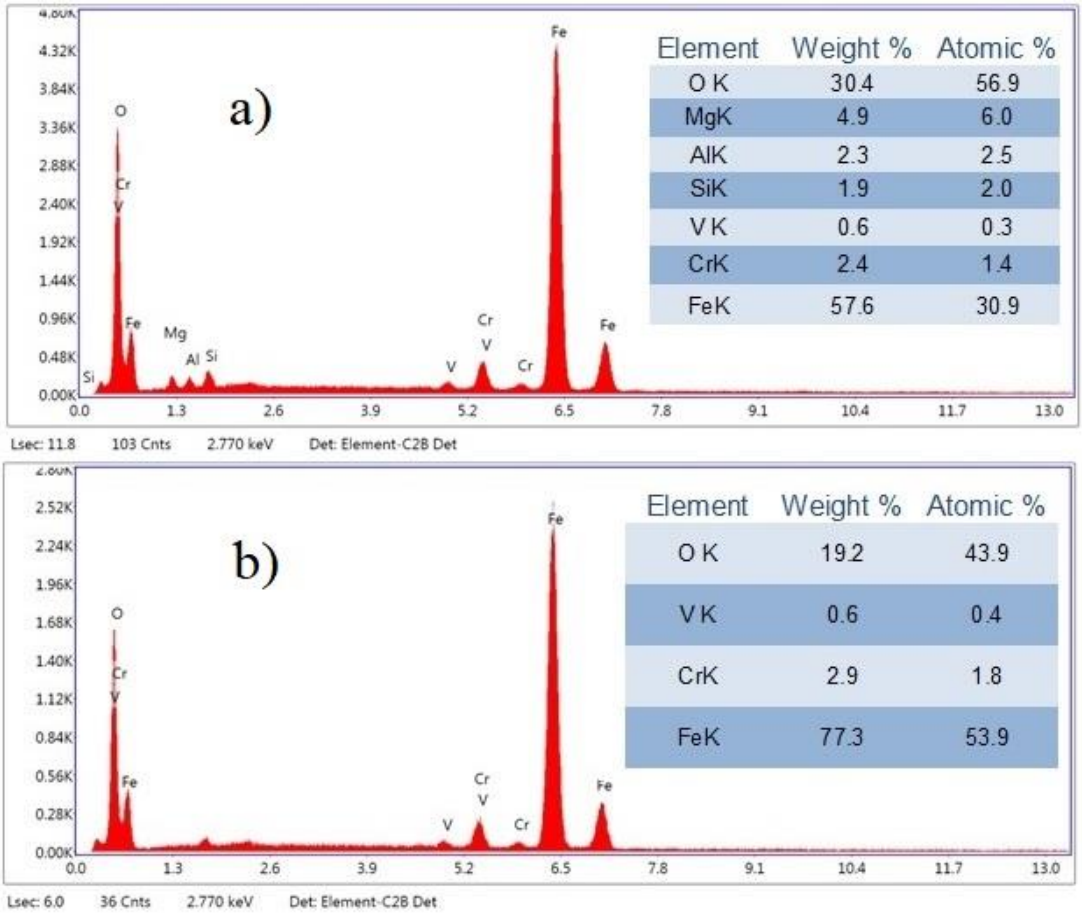


Fig. 10. EDS analysis results of white area a) quench-tempered sample, and b) quench-subzero-tempered sample at 250°C.

4. Conclusion

1- In this study, deep cryogenic treatment was successfully performed, which improved the wear properties by 36% compared to the samples quench-tempered at 250°C.

2. Comparing the properties of the hot work H13 steel (with perlite and cementite structure), the hardness increased by at least 150% by applying quench-tempered treatment (with the residual martensite and austenite structure); and by applying quench-subzero-tempered treatment, it shows an increase of 276 percent.

3. The application of quench-subzero-tempered treatment on H13 steel resulted in more uniformly dispersed and finer pricipates in this sample than the quench-tempered sample.

4. The highest hardness in quench-subzero-tempered sample is about 26% higher than quench-temper one.

5. Examination of wear traces indicates that surface oxide particles adhere to disk surfaces during rubbing. Due to the sub-zero treatment, and because of the increase and uniform distribution of carbides, less wear and oxidation have occurred.

5. Acknowledgments

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