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Investigating the effect of deep cryogenic heat treatment on the wear behavior of 100Cr6 alloy steel

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Abstract

This work has focused on the effect of the deep cryogenic treatment on the wear behavior, microstructural changes, retained austenite percentage and hardness of 100Cr6 (AISI 52100) alloy steel. These analyses were conducted using the scanning electron microscope (SEM), X-ray diffraction (XRD) and EDS to study the microstructure, a pin-on-disk wear testing machine to study the wear behavior, as well as a hardness testing machine to study the hardness changes. It was shown that the deep cryogenic treatment eliminated retained austenite and made a more uniform carbide distribution with a higher percentage. It was also observed that the deep cryogenic treatment improved the wear behavior and hardness of the samples. Moreover, the hardness and wear resistance decreased in longer austenizing durations due to the grain growth and dissolution of carbides in the structure. It was also shown that the predominant wear mechanism was a combination of abrasive and tribo-chemical wear.

Key words: iron alloys, phase transformations, wear

1. Introduction

Since the early decades of the 20th century, cryogenic heat treatment has been used to increase tool life and improve the wear behavior of steels [1]. This behavior was not clearly understood in those days and was regularly used as an empirical conclusion, and only in the later decades of that century did scientists find some explanations to clarify this improvement [1]. Early theories attempted to explain this behavior using retained austenite elimination at low temperatures. It was shown that some austenite always remains after the conventional heat treatment, especially in high-carbon and alloy steels. Retained austenite is a consequence of low martensite finish temperature (M_f) in the high alloy steels. In these steels a high amount of alloying elements increases the essential shear stress that is necessary for martensite transformation, and subsequently the martensite transformation temperature decreases. This higher amount of required cooling decreases the M_f temperature below the room temperature in the high carbon or alloyed steels. In other words, cooling the samples down to room temperature during quenching did not annihilate the retained austenite [2, 3].

Cryogenic treatment consists of cooling the samples to the temperatures lower than the room temperature, holding the samples at that temperature for a period of time and then warming them up to the room temperature [4]. Retained austenite is transformed into martensite during the cryogenic treatment as a slow-rate transformation, as compared with the conventional diffusion less martensite transformation [5, 6]. The cryogenic treatment can be performed in two ways: (a) the shallow cryogenic treatment, which is performed at temperatures between –60 to –120 ℃, and (b) the deep cryogenic treatment, performed at lower temperatures < -125 ℃, and regularly at the liquid nitrogen temperature –190 ℃ [7]. After the shallow cryogenic heat treatment, the retained austenite decreases and after the deep cryogenic heat treatment in most of the steels the retained austenite vanishes completely [8]. In addition to the retained austenite annihilation, the carbide percentage increases, a more

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homogenous carbide distribution is achieved and the wear resistance improves more than the values expected [9–11]. One of the accepted theories to explain this behavior claims that at low temperatures the martensite and austenite endure a high degree of contraction. This contraction together with the different expansion coefficient of martensite and austenite produces some new dislocations in the steel structure. The contracted structures make the highly deformed martensite unit cells in which the carbon atoms cannot be stable in their places. In other words, carbon atoms prefer to jump to the nearby defects containing the newly formed dislocations, old dislocations, twins and other defects, due to a highly contracted martensite structure which forces the carbon atoms to jump. These carbon atoms act as preferential sites for carbide nucleation in the following tempering [11, 14]. These newly formed carbides are eta carbides rather than the regular epsilon ones and are bigger than 20 nm in diameter [15]. These carbides make a more homogenous distribution and increase the carbide percentage. This increase in the carbide percentage, along with the retained austenite elimination, increases the wear resistance of steel for some percentage to some hundred percentages [2, 11, 16–19].

The materials, which have been utilized to study the effect of the deep cryogenic heat treatment, consist of: HSS steels [20, 21], carburized steels [22], tool steels [6, 10, 16, 23–27] and polymers [28]. Despite the great number of studies, a considerable amount of disparity is observed in the results and theories. This study has focused upon the effect of the deep cryogenic heat treatment on the microstructure, wear behavior and hardness of 100Cr6 low alloy steel. This steel is regularly used in the places where the highest wear resistance is necessary, for example in the bearings in the rotating machinery. Due to this application, improving the wear resistance of this steel is important. This study was performed using the scanning electron microscope (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD) and pin-on-disk wear testing machine.

2. Experiments

A bar of 100Cr6 (AISI 52100) alloy steel with the diameter of 20 mm was cut into disks with the height of 5 mm using an electrical discharge machine (EDM). The nominal composition of the selected steel was (wt. %): 1 % C, 0.2 % Si, 0.45 % Mn, 1.4 % Cr, 0.1 % Mo and 96.85 % Fe.

The samples were austenized at 920 °C for 20 and 80 min in a vacuum furnace and then quenched in oil. Two groups of the samples were assumed as the conventionally treated ones and named CHT20 and CHT80 samples, respectively. The other samples were deep cryogenically treated immediately to prohibit the austenite aging [20]. For deep cryogenic heat treatment, samples were cooled down to liquid nitrogen temperature gradually to prohibit severe distortion due to thermal shocks. The samples were held at that temperature for 24 h and then warmed up to room temperature gradually. These samples were named deep cryogenically treated samples, DCT 20 and DCT 80, respectively. All the samples were then tempered at 210 °C for 1 h. For more accuracy in studies the samples surface was polished up to #1000 grinding paper and then polished using 1 μm alumina to reach uniform surfaces with the surface roughness of 0.4 μm.

The samples were analyzed using XRD to evaluate the effect of the deep cryogenic heat treatment on retained austenite percentages. For this evaluation, a Cu Kα radiation was used, and the retained austenite percentage was determined according to the ASTM E975-00 standard. The combination of the phase's percentage should be equal to 100 %. The carbide percentage was evaluated using the SEM micrographs and the austenite percentage was calculated using the following equation:

$$V_\gamma = \left[\frac{(1 - V_\mathrm{C})(I_\gamma/R_\gamma)}{I_\alpha/R_\alpha}\right] + \frac{I_\gamma}{R_\gamma}, \qquad (1)$$

where V_γ and V_C are the retained austenite and carbide percentage, respectively, I_γ and I_α are the integrated intensity per angular diffraction peak (hkl) in the austenite and martensite phases, respectively, and R_γ and I_α are the austenite and martensite constants, respectively.

The value of R can be calculated with respect to the (hkl) plane in combination with the polarization, multiplicity and structure factor of the phases according to the ASTM E975-00 standard. For microstructural studies, the samples surface was etched in Nital 4% (4 % HNO_3 and 96 % ethanol). After etching, the samples surface was analyzed using SEM (SEM Tescan Vega 2) to observe the carbide distribution. The SEM micrographs were then analyzed using an image analyzing software of Clemex Vision (version 3.5.025) to calculate the carbide percentage.

The hardness of the samples was evaluated using a KOOPA UV1 hardness tester by the Vickers method. By this method, the load of 45 kg was applied for 15 s. The hardness tests were carried out for 10 times to reach a trustful average.

Wear tests were performed on the samples using a pin-on-disk wear testing machine. For the wear test, samples surface was examined using 820 HV bearing steel pin with a spherical rounded end and with a curvature radius of 1 mm. Disk weight loss was analyzed in each 100 m sliding distance using a digital scale (Seleron 7500) with 0.0001 mg accuracy. The sample sliding distance was 1000 m, the applied load was 220 N, the sliding velocities were 0.05, 0.1 and 0.15 m s^{-1}, and the tests were performed at room temperature (25 ± 4 °C and 40 ± 12 % humidity). The wear rate was calculated by Eq. (2):

$$W_\mathrm{r} = \frac{\Delta m}{\rho L F_\mathrm{N}} \times 10^3, \qquad (2)$$

where W_r (mm^3 N^{-1} m^{-1}) is the wear rate, Δm (g) is the weight loss, ρ (g cm^{-3}) is the steel density, L (m) is the wear distance, and F_N (N) is the load.

The samples worn out surface was then analyzed using SEM to study the wear mechanism and to observe the severity of wear conjunctions in the samples. For more studies, the samples worn out surfaces were examined using EDS (EDX, Tescan Vega 2) to study the wear mechanism more accurately.

3. Results and discussion

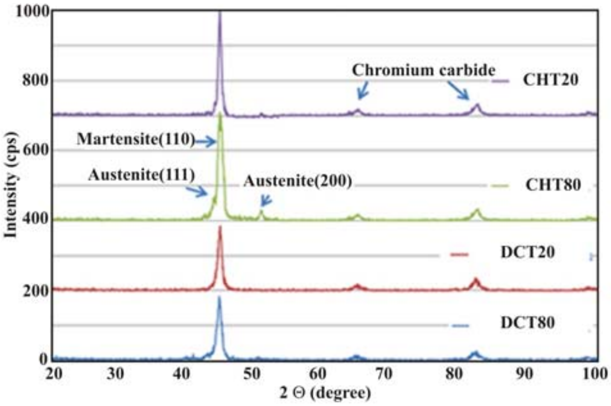


Fig. 1. XRD diagram of the 100Cr6 low alloy steel.

The XRD analysis of the samples showed that the deep cryogenic heat treatment eliminated the retained austenite completely. The retained austenite percentage decreased from 8 and 5 % in the CHT80 and CHT20, respectively, to lower value than the detection limit of the XRD technique (< 1 %) (Fig. 1). In longer

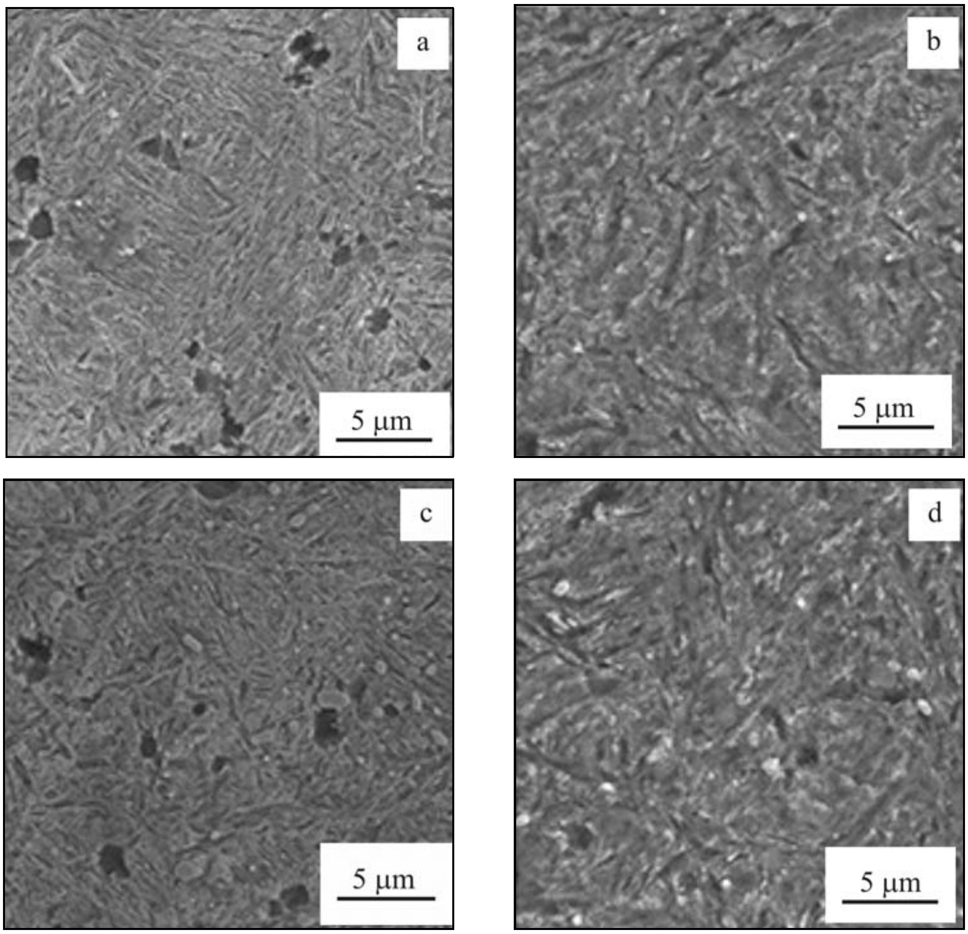


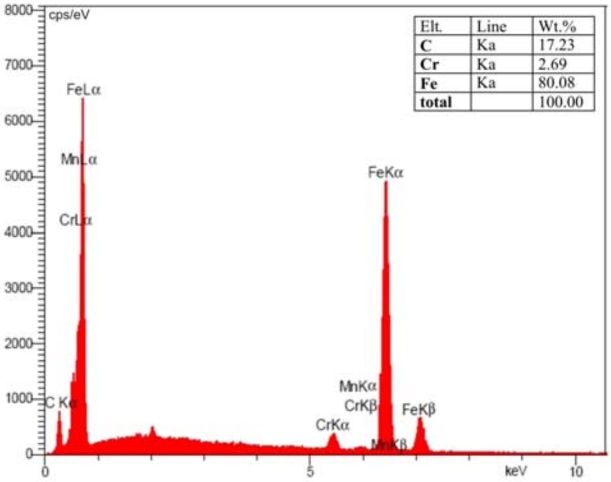
Fig. 2. SEM micrographs of the carbides in the 100Cr6 low alloy steel: CHT80 (a), CHT20 (b), DCT80 (c) and DCT20 (d) (6000×).

Table 1. Carbide percentage of the 100Cr6 samples

Sample	Carbide percentage
CHT20	6
CHT80	4
DCT20	21
DCT80	18

austenizing temperatures the carbides were dissolved and a solid solution of the carbon, chromium and iron atoms was produced. These dissolved atoms reduced the martensite finish and start temperatures (M_f and M_s, respectively) due to increasing the carbon content of the austenite, thereby decreasing the hardenability of steel. In other words, retained austenite is lower in the CHT20, as compared with the CHT80 as a consequence of a reduction in the martensite start and finish temperature at longer austenizing durations.

The SEM micrographs of the samples show that the carbide percentage increases by 14–15 %, in the DCT samples as compared with those of the CHT (Fig. 2). Deep cryogenic heat treatment forces carbon atoms to jump to the nearby defects, thereby increasing the carbide percentage. This increase is a result of the new carbide formation during tempering. The carbon atoms movement inside the structure is due to a highly distorted martensite structure that forces the carbon atoms to jump to the nearby defects [2–6]. Should the samples be austenized for longer durations, more carbides will be dissolved in the structure. The higher amount of dissolved carbon atoms decreases the carbide percentage after quenching due to the effect of the non-solved carbides in the final carbide percentage. After the deep cryogenic heat treatment the carbide percentage increases due to the carbon atoms jumping at low temperatures but the carbides percentage is still lower in the DCT80 samples, as compared with those of DCT20 (Table 1). After the deep cryogenic heat treatment, some newly formed carbides are produced, but the initial carbides are also influential. It was demonstrated that the carbide percentage was higher in the CHT20 samples, as compared with those of CHT80 due to less dissolved carbides at austenizing temperatures. The higher amount of primary carbides in cooperation with the secondary carbides increase the final carbide percentage of the DCT20 samples, as compared with the DCT80 samples. The surface of the samples was also examined using the EDX. Results show that except for the martensite, only chromium carbides existed in the structure (Fig. 3).



Elt.	Line	Wt.%
C	Ka	17.23
Cr	Ka	2.69
Fe	Ka	80.08
total		100.00

Fig. 3. EDX analysis of the carbide in the 100Cr6 samples and its composition.

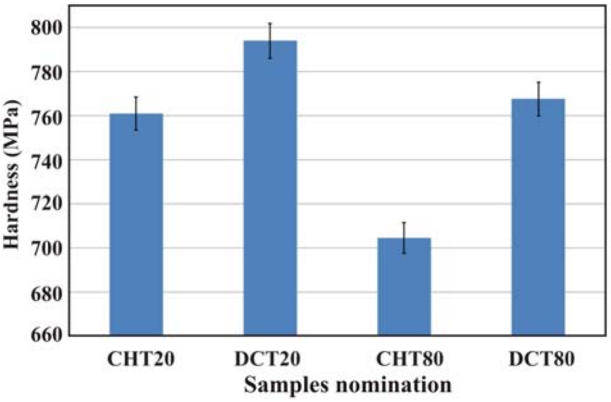


Fig. 4. Vickers hardness of the 100Cr6 low alloy steel samples.

Hardness results show that the deep cryogenic heat treatment increases the hardness from 761 to 794 MPa in the DCT20 and from 704 to 767 MPa in DCT80 samples (Fig. 4). It was shown that at longer austenizing durations the hardness decreased in the DCT and CHT samples. In the conventionally treated samples, by increasing the austenizing durations more carbides are dissolved in the structure. These dissolved carbon atoms decrease the final carbides percentage. That is, by increasing the holding durations, grain growth takes place in the austenite structure. Bigger grains lead to a less distorted martensite structure, which is formed due to the martensitic plane collision at the prior austenite boundaries [31]. By increasing the grain size, the grain boundaries decrease and therefore less distorted boundaries are produced. The less distorted structure, together with lower carbide and more retained austenite percentages, decrease the hardness of the conventionally treated samples at longer austenizing durations. In the deep cryogenically treated samples, the hardness decreases in the DCT80 due to the lower carbide percentage and a less distorted struc-

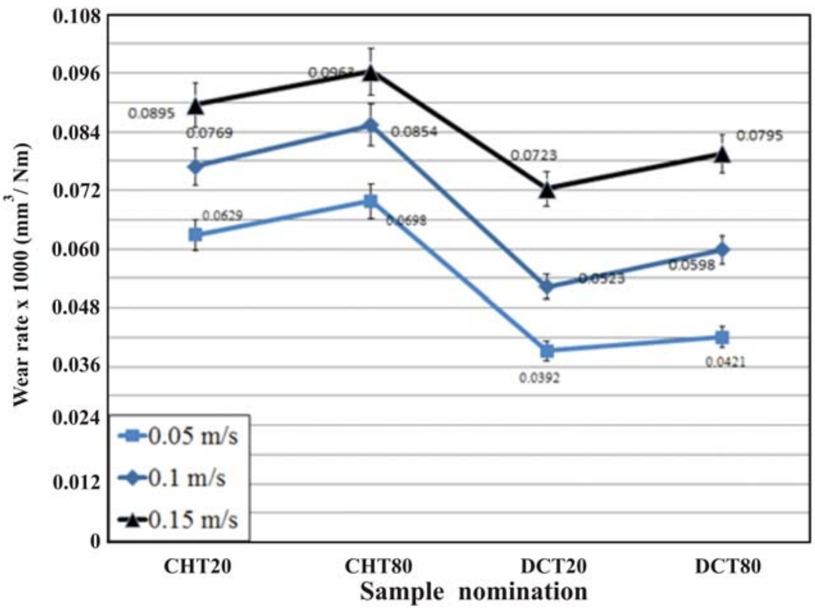


Fig. 5. Wear rate of samples at different sliding speeds of 0.05, 0.1 and 0.15 m s^{-1} under 220 N normal load in pin-on-disk war test.

Table 2. EDX analysis of the worn out surface of the DCT20 sample after 1000 m sliding under the load of 220 N

Element	Series	(norm. wt.%)	(norm. at.%)
Carbon	K-series	4.524717	15.45809
Oxygen	K-series	7.783294	19.96196
Chromium	K-series	2.713465	2.1414
Iron	K-series	84.97852	62.43855
	Sum:	100	100

ture, as compared with the DCT20 samples. These results are in good agreement with the microstructural observations.

The pin-on-disk wear test was performed to investigate the effect of the holding duration on the wear resistance of the samples (Fig. 5). Results show that the deep cryogenic heat treatment increases the wear resistance of the samples at all sliding velocities for 45–70 %. This improvement is a result of the retained austenite annihilation, and an increase in the carbide percentage. At longer austenizing durations, the carbide percentage and the structure distortion decrease. These phenomena lead to a decrease in the wear resistance of the samples austenized for 80 min, as compared with those austenized for 20 min (Fig. 5).

The SEM micrographs of the worn-out surface of the samples show that the CHT samples demonstrate highly damaged surfaces, as compared with the DCT ones (Fig. 6). SEM micrographs also demonstrate that the predominant wear mechanism is abrasive wear (Fig. 6). For more accuracy, the CHT20 samples surface was investigated using EDX (Table 2). The EDX

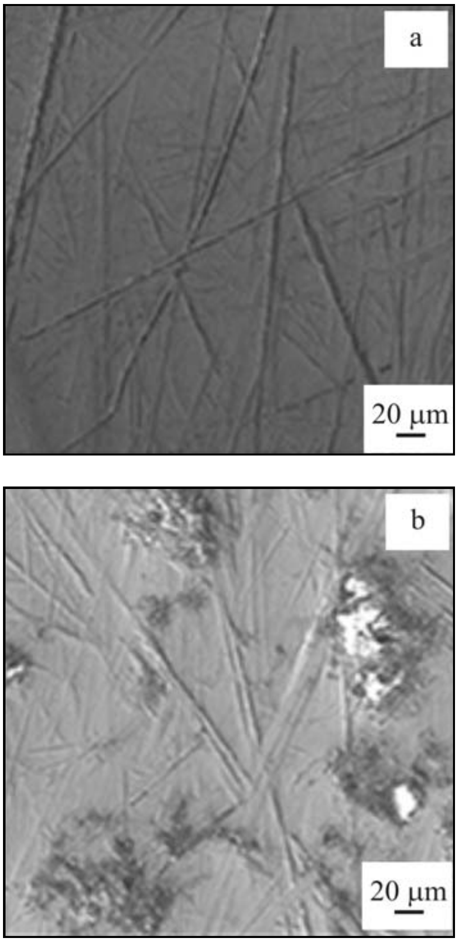
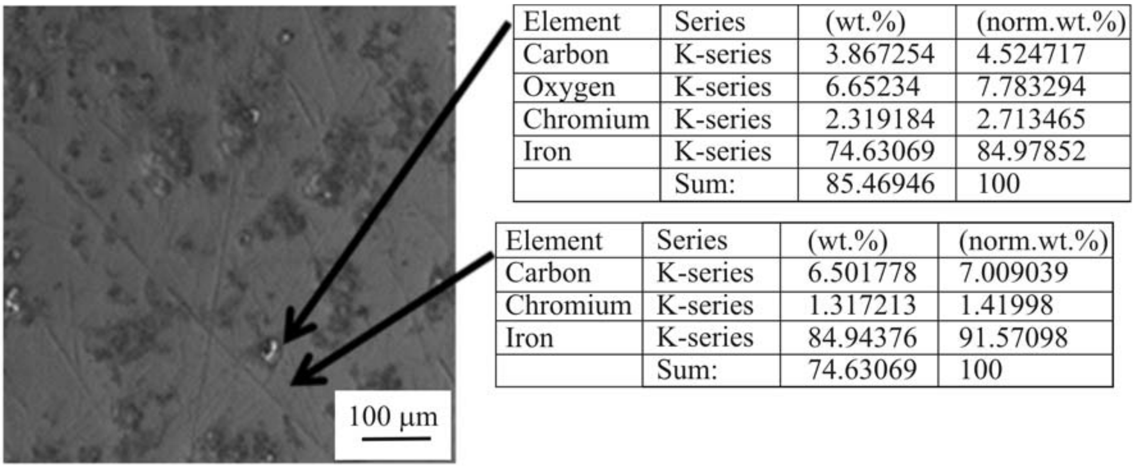


Fig. 6. Worn out surface of DCT20 (a) and CHT20 (b) (800×).



Element	Series	(wt.%)	(norm.wt.%)
Carbon	K-series	3.867254	4.524717
Oxygen	K-series	6.65234	7.783294
Chromium	K-series	2.319184	2.713465
Iron	K-series	74.63069	84.97852
	Sum:	85.46946	100

Element	Series	(wt.%)	(norm.wt.%)
Carbon	K-series	6.501778	7.009039
Chromium	K-series	1.317213	1.41998
Iron	K-series	84.94376	91.57098
	Sum:	74.63069	100

Fig. 7. EDX analysis of the worn-out surface of the CHT20 sample after 1000 m sliding.

analysis was conducted on the different places of the sample, indicating that a high amount of oxygen is observed on the worn-out surface of the samples (Fig. 7). This high amount of oxygen shows that in addition to the abrasive wear, a tribo-chemical reaction is also involved in the wear cycle of the 100Cr6 alloy steel. The tribo-chemical mechanism is a consequence of the highly localized temperature rise in the contact surface of the pin and disk due to metallic surface exposed. The highly localized temperature oxidizes the pin and disk surface at contact areas.

4. Conclusions

The effect of the cryogenic heat treatment of the 100Cr6 alloy steel on the wear behavior, hardness and microstructure was examined using the SEM, EDX, XRD and pin-on-disk wear tests. The obtained results can be summarized as follows:

1. It was shown that the wear resistance and hardness of the samples were improved during the deep cryogenic heat treatment. This improvement is a result of a more uniform carbide distribution, retained austenite elimination and an increase in the carbide percentage.

2. In longer austenizing periods the grain growth occurred and more carbides were dissolved in the austenite structure. These phenomena increased the retained austenite and decreased the hardness and wear resistance of the samples, which were held for longer periods in the furnace.

3. The predominant wear mechanism was abrasive wear but some degree of tribo-chemical wear was also observed. The tribo-chemical wear was a consequence of a localized temperature rise in contact surfaces due to metallic surface exposed that oxidized the contact sites.

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