Evaluation of wear performance of cryogenically treated Vanadis 4 Extra tool steel

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Abstract

In this study, the wear performance of cryogenically treated cold-work tool steel (Vanadis 4 Extra) samples was evaluated in the light of some sliding wear tests. For this purpose, the samples were cryogenically treated for different durations (0, 1, 12, and 24 h) at deep cryogenic temperature (–145 °C). A tempering process was performed at two different temperatures (175 and 525 °C) for 2 h. The wear experiments were carried out in a ball-on-disk tester, by applying two loads of 10 and 20 N and a sliding velocity of 0.3 m s^{-1}. Experimental results showed that samples treated for 24 h and tempered at 525 °C exhibited the best wear performance. This can be explained by the increasing hardness value as a result of the nearly full transformation of retained austenite to martensite, the formation of fine carbide particles and homogeneous distribution of carbides. The hardness measurements and XRD analysis confirmed the wear test results.

K e y   w o r d s: Vanadis 4 Extra, cryogenic treatment, microstructure, wear resistance

1. Introduction

Tool steels are the high-quality steels used to manufacture the tools, die and molds that shape, form and cut different engineering materials such as steels, nonferrous materials and plastics [1]. In many industrial applications, tool steels are subjected to extremely high and variable loads. Therefore, this group of steels must withstand those loads for long times without breaking and without undergoing excessive wear or deformation. Tool steels are broadly divided into six categories: cold work, shock resisting, hot work, high speed, mold and special-purpose tool steels [2, 3]. Among them, cold-work tool steels are the most important category, as they have a wide usage area in the industry because of their high hardness, toughness and wear resistance [4, 5]. For a cold-work tool steel at a certain hardness, wear resistance may vary widely depending on the heat treatment used and wear mechanism in the process [6]. Also, in higher carbon content steels such as cold-work tool steels, the martensite finish line temperature is below 0 °C, which means that at the end of the heat treatment, a low percentage of austenite is retained at room temperature. The retained austenite as a soft phase in steels could reduce the product life in working conditions [7].

Cryogenic treatment has been acknowledged for many decades as an effective method for increasing wear resistance in tool steels [8, 9]. It is a one-time permanent treatment process, and it affects the entire cross section of the material usually done at the end of the conventional heat treatment process but before tempering. Also, it is not a substitute process but rather a supplement to the conventional heat treatment process [10]. According to the previous literature, two metallurgical phenomena are reported as the main reasons for using cryogenic treatment: firstly, the elimination of retained austenite, and secondly, the

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initiating of nucleation sites for subsequent precipita-
tion of a large number of very fine carbide particles. In
many cases, hardness increasing by about one to three
Rockwell points has been claimed; however, some au-
thors have reported a little increase in hardness values.
There is little evidence concerning toughness changes
[11].

    Das et al. [12] examined the influence of cryo-
genic treatment (–196 °C) in between quenching and
tempering on carbide precipitation and the tribolog-
ical behavior of commercial AISI D2 steel. Experi-
mental results showed that the cryogenic treatment
of D2 steel markedly reduces the amount of retained
austenite and makes significant changes in the pre-
cipitation behavior of secondary carbides and the for-
mation of refined secondary carbides, increases their
volume fraction and leads to a more uniform distribu-
tion of the carbides than those in conventionally heat-
treated D2 steel. Akhbarizadeh et al. [13] investigated
wear behavior and hardness via a hardness tester and
pin-on-disk wear testing machine. Results showed that
deep cryogenic heat treatment improves the carbide
percentage, distribution, hardness and wear resistance
of 1.2080 (AISI D3) tool steel, as compared with those
of the conventionally treated. Further improvements
in the wear resistance and hardness were also observed
due to the formation of some newly formed nano-sized
carbides in the structure. Koneshlou et al. [14] studied
the effects of low-temperature treatments on the mi-
crostructure, and mechanical properties of H13 hot-
-work tool steel. Cryogenic treatment at –72 °C and
deep cryogenic treatment at –196 °C were applied, and
it was found that by applying the subzero treatments,
the retained austenite was transformed to martensite.
The deep cryogenic treatment also resulted in precipi-
tation of more uniform and very fine carbide particles.
The microstructural modification resulted in a signifi-
cant improvement in the mechanical properties of the
H13 tool steel. Li et al. [15] examined the effect on the
microstructure and properties (hardness, toughness
and the content of retained austenite) of a newly devel-
oped cold work die steel (Cr8Mo2SiV). Results showed
that the hardness of the DCT (Deep Cryogenic Treat-
ment) specimens was higher (+0.5HRC to +2HRC)
whereas the toughness was lower when compared with
the conventionally treated specimens (quenching and
tempering). After the DCT process, retained austenite
transformed into martensite, although not completely.
Oppenkowski et al. [16] studied the wear resistance
of the powder metallurgically produced and cryogeni-
cally treated cold-work tool steel AISI D2. Further in-
vestigations of this study identified a nearly constant
wear rate for holding times of up to 24 h. The wear
rate reaches a minimum for a longer holding time of
36 h and increases again with further holding.

    The purpose of this study is to investigate the ef-
fects of deep cryogenic treatment (–145 °C) applied

T a b l e 1. Chemical composition of Vanadis 4 tool steel

C	Si	Mn	Cr	V	Mo
1.4	0.4	0.4	4.7	3.7	3.5

at three different holding times (1, 12 and 24 h) to
Vanadis 4 Extra cold-work tool steel, which has supe-
rior wear resistance to conventional tool steel AISI D2
die material [17]. For this purpose, some sliding wear
tests were performed on a ball-on-disk wear testing
device at two loads and two tempering temperatures.

2. Materials and method

    Vanadis 4 cold-work tool steel rods with a diameter
of 20 mm and height of 8 mm were used to prepare the
samples. The chemical composition of the Vanadis 4
steel used is presented in Table 1. Samples were sub-
jected to conventional heat treatment and cryogenic
treatment in separate batches. The conventional heat
treatment (CHT) consisted of hardening and temper-
ing processes, which were conducted according to the
ASM Heat Treater's Guide [18]. DCT was incorpo-
rated intermediately between hardening (D) and tem-
pering (T); the details of each step are illustrated in
Table 2.

    The cryogenic treatment was carried out by grad-
ual cooling (approximate cooling rate of $1.5 °C min^{-1}$)
of the samples to –145 °C, and holding the samples
at this temperature for different durations (0, 1, 12,
and 24 h), followed by gradual heating (approximate
heating rate of $1.5 °C min^{-1}$) to room temperature.
Cryogenically treated samples were tempered at two
temperatures of 175 and 525 °C for 2 h. The samples
subjected to different treatments are referred to hence-
forth with codes as shown in Table 2, where the numer-
als in the codes represent the time of holding in hours
at –145 °C. The contents of the retained austenite and
martensite were measured using XRD, with a Bruker
D8 Advance X-ray diffraction instrument that used a
Cr Kα X-ray source. From the X-ray diffractograms,
the contents of the retained austenite and martensite
were measured using ASTM E975 standard [19]. The
bulk hardness (HRC) was measured using a Rockwell
hardness tester with a load of 1.5 kN. The microstruc-
ture was characterized by a Leo 1430 VP scanning
electron microscope after etching in 4% Nital (96 ml
ethanol and 4 ml HNO_3).

2.1. Wear experiments

    To perform the friction and wear of DCT sam-
ples, a ball-on-disk test device was used. In the wear

T a b l e 2. Different heat treatment cycles for the Vanadis 4 steel samples

Samples	Hardening	Cryogenic treatment	Tempering	Retained austenite percentage (%)	Martensite percentage (%)	Carbide percentage (%)
CHT-LTT	Austenitizing at	None		14.1	74.37	11.53
DCT-1h-LTT	30 min at 1030 °C	1 h at −145 °C		4.01	85.17	10.82
DCT-12h-LTT	and oil quenching	12 h at −145 °C	2 h at 175 °C	3.84	86.32	9.84
DCT-24h-LTT		24 h at −145 °C		3.53	87.20	9.27
CHT-HTT	Austenitizing at	None		2.93	82.98	14.09
DCT-1h-HTT	30 min at 1030 °C	1 h at −145 °C		0.70	86.12	13.18
DCT-12h-HTT	and oil quenching	12 h at −145 °C	2 h at 525 °C	0.41	87.64	11.96
DCT-24h-HTT		24 h at −145 °C		0.25	89.01	10.74

CHT – conventional heat treatment, DCT – deep cryogenic treatment, LTT – low tempering temperature, HTT – high tempering temperature
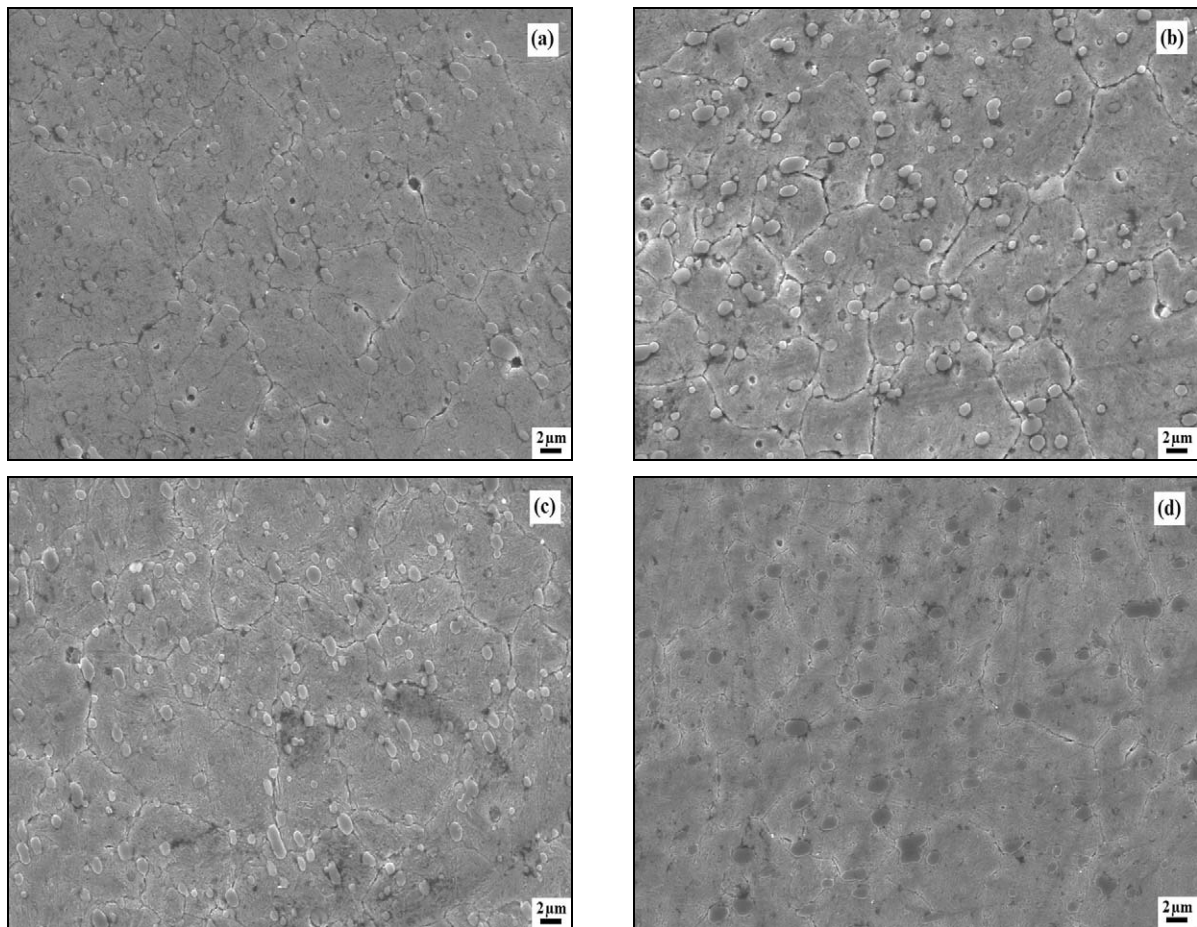
Fig. 1. SEM pictures of CHT and DCT samples tempered for 2 h at 175 °C: a) CHT-LTT, b) DCT-1h-LTT, c) DCT-12h--LTT, d) DCT-24h-LTT.

tests, WC-Co balls of 8 mm in diameter supplied by H.C. Starck Ceramics GmbH were used. Errors caused by the distortion of the surface were eliminated by using a separate abrasion element (WC-Co ball) for each test. The wear experiments were carried out in a ball disk arrangement under dry friction conditions at room temperature with applied loads of 10 N and 20 N with a sliding velocity of 0.3 m s^{-1} at a sliding distance of 600 m. In this way, each sample was tested twice. Before and after each wear test, each sample and abrasion element was cleaned with ethanol. After the test, the wear volumes of the samples were quantified by multiplying cross-sectional areas of wear by the width of the wear track obtained from the device, a Tribotechnic Rugosimeter. Wear rate (mm^3 Nm^{-1}) was calculated by the following formula (1):
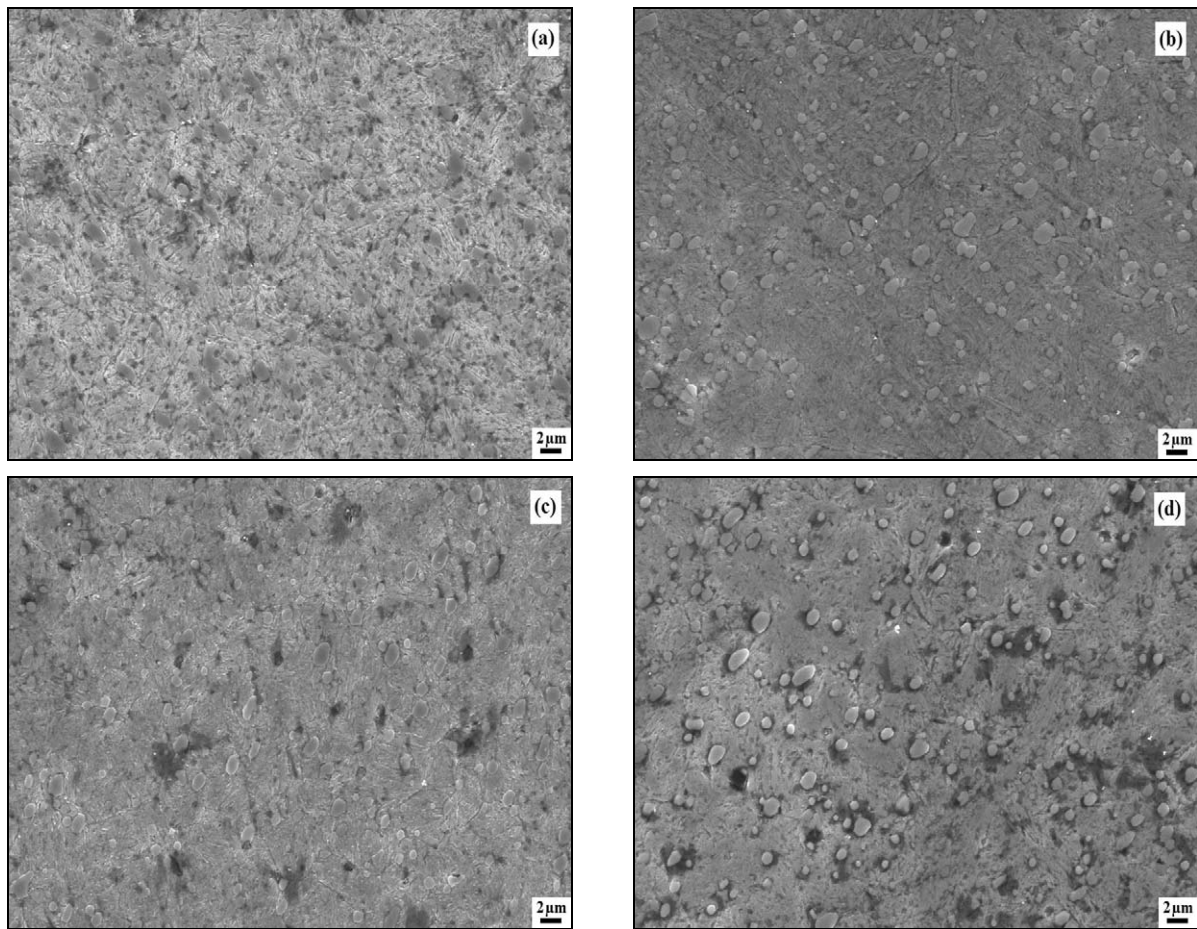
Fig. 2. SEM pictures of CHT and DCT samples tempered for 2 h at 525 °C: a) CHT-HTT, b) DCT-1h-HTT, c) DCT-12h--HTT, d) DCT-24h-HTT.

Wear rate = Worn volume/(Applied load × Sliding distance).   (1)

Friction coefficients depending on the sliding distance were obtained through the friction coefficient program. As a result of the wear tests, graphs of the friction coefficient, surface roughness and wear rate of DCT samples were plotted. Worn surfaces were investigated by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS).

3. Results and discussion

3.1. Microstructure observations

SEM pictures of CHT and DCT samples tempered at two different temperatures are shown in Figs. 1 and 2. These figures show that the SEM image of the undissolved carbide particles (white color) and massive regions around the carbides were probably austenite taken with backscattered electrons (BE).

After cryogenic treatment, the carbides became fine and distributed in a more homogeneous way. In

particular, the finest carbide particles were formed and homogeneously distributed after DCT for 12 h (Figs. 1c, 2c) when compared to CHT and DCT for 1 h and 24 h. There are non-homogeneous carbide particles of different sizes in the microstructure (Figs. 1 and 2a–d). These particles are fragmented and evolve into a more compact and homogeneous structure after cryogenic treatment and tempering. Figures 3a–d show the XRD diffraction of the CHT and the DCT samples.

The peaks corresponding to martensite, retained austenite and carbides are visible. After DCT, the peaks of austenite have a low-intensity value. This indicates that the retained austenite has transformed to martensite during DCT. However, the transformation is not complete [7, 20]. The results in Table 2 lead to the inference that the retained austenite percentage decreases with increasing cryogenic processing time and reaches maximum at DCT-24h samples tempered at 525 °C. The lowest retained austenite volume was obtained in the DCT-24h sample tempered at 525 °C (0.25 %) while the highest retained austenite volume was obtained in the CHT sample tempered at 175 °C (14.1 %). In the DCT samples, the retained
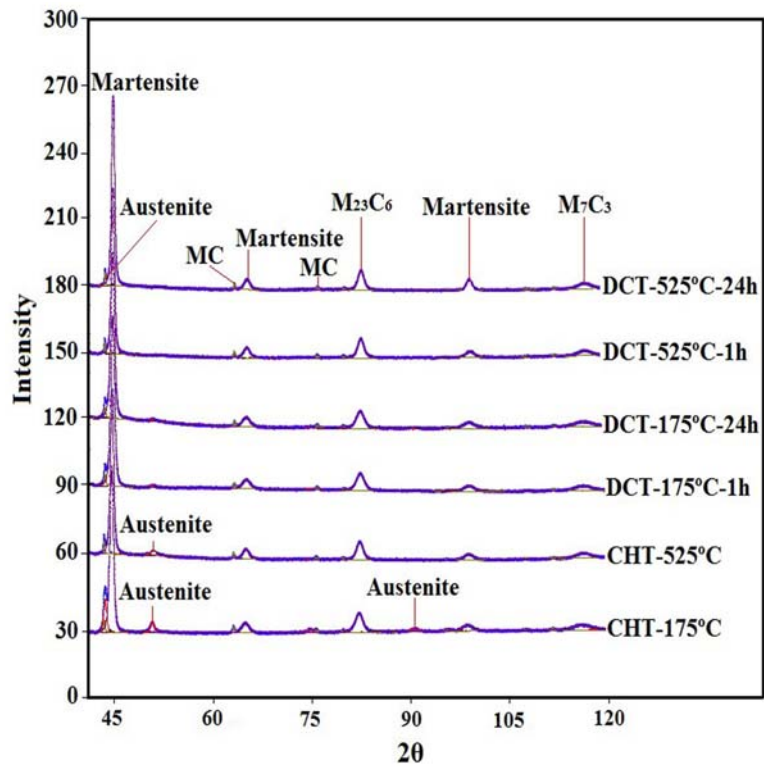
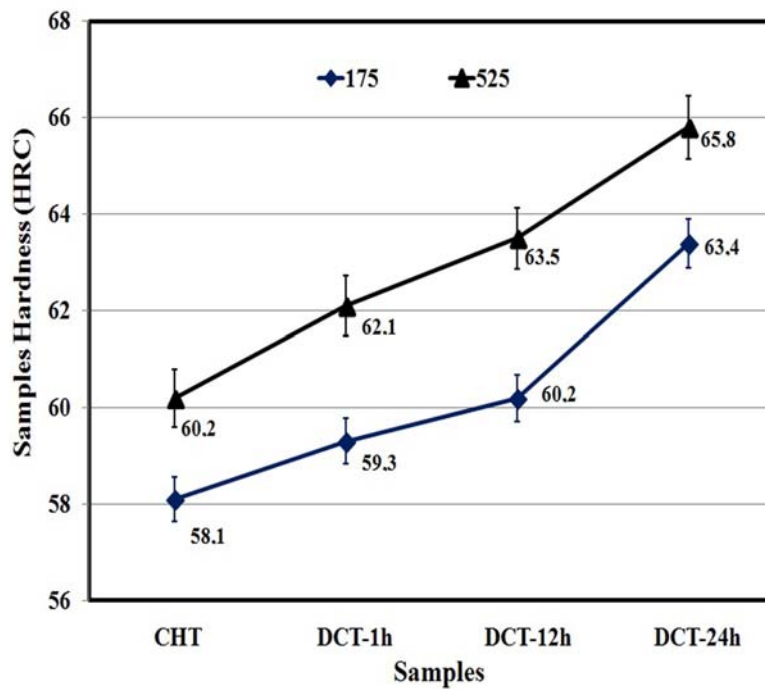Fig. 3. The XRD diffraction of the CHT and the DCT samples.

Fig. 4. The hardness of CHT and DCT samples.

austenite percentage is lower than in the CHT sample. Cryogenic treatment can effectively reduce the retained austenite content, but cryogenic treatment cannot make retained austenite transform to martensite completely [21]. In addition, it was observed from the amounts of retained austenite (Table 2) and hardness values (Fig. 4) that austenite amounts in the samples tempered at 525 ℃ are lower than those in the sam-
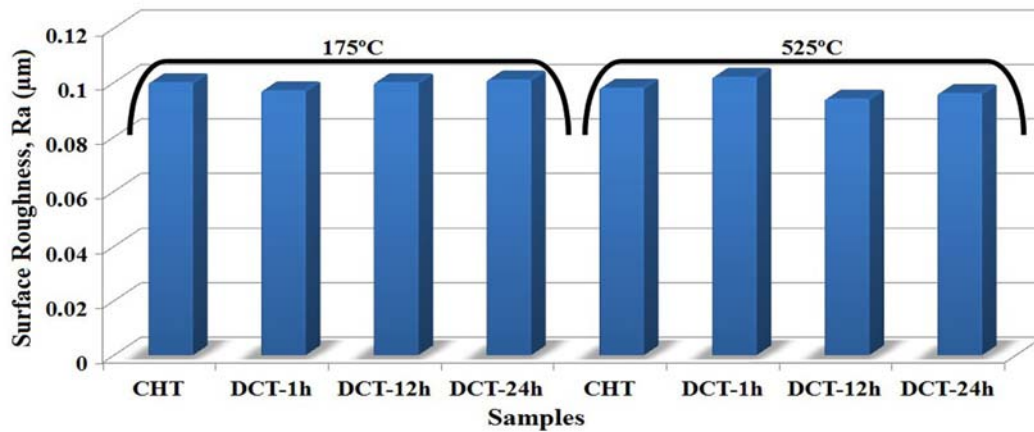
Fig. 5. Surface roughness values of CHT and DCT samples.

ples tempered at 175 °C, whereas the hardness of the samples tempered at 525 °C is higher than that of the samples tempered at 175 °C. This case showed that a tempering temperature of 525 °C was better than a tempering temperature of 175 °C in terms of the maximal transformation of retained austenite to martensite and higher hardness values for Vanadis 4 Extra [22].

3.2. Hardness

The hardness test was carried out using the Rockwell C hardness tester. The results (Fig. 4) show that the cryogenic treatment increased the hardness. The samples that were cryo-treated show a further increase in hardness. The higher hardness of the DCT samples was due to the decrease in retained austenite. Also, in the DCT samples, the retained austenite was nearly eliminated at the tempering temperature of 525 °C, they had a more homogenized chromium carbide distribution, and increased hardness due to the higher chromium carbide percentage. The hardness results show that the DCT increases the hardness of the samples by approximately 6 HRC. The hardness of deep cryogenic-treated samples increases with increasing holding time at cryogenic temperatures, and reaches a maximum at a deep cryogenic processing time of 24 h (Fig. 4).

It can be said that the hardness value was affected by the precipitation process. In the longer holding times, the progress of the carbon segregation increases carbide precipitate sizes, thereby decreasing the number of carbides and their homogenized distribution. This is in agreement with the previous studies reported in the literature [23–26]. In this study, tempering was carried out at temperatures of 175 and 525 °C for 2 h after the cryogenic treatment increasing the hardness of the tool steel and causing secondary carbide precipitation. These are among the most important factors for the improvements in the service life of tool steel. The microstructure and hardness investi-

gations were conducted after the cryogenic treatment confirmed these changes (Fig. 4). Molinari et al. [27] and Pellizzari et al. [28] subjected tool steels to cryogenic treatment. They determined that the increase in tool life following the cryogenic treatment was due to the fact that the cryogenic treatment carried out after a CHT transformed the retained austenite in the structure of the cutting tools to martensite, improving the mechanical properties of the cutting tools, such as the hardness and wear resistance, by providing a more homogeneous distribution of carbides.

3.3. Wear resistance

Wear tests were carried out after all the samples had passed through the 1200 grid grinding stages. Therefore, prior to the wear process, surface roughness values of Vanadis 4 cold-work tool steel at different periods of time were observed to be similar (Fig. 5).

Friction coefficient values at different wear loads of the samples, cryo-treated at different periods of time, can be seen in Fig. 6. Friction coefficient values of the DCT samples increased as the amount of wear load increased. When the friction coefficient of the samples worn under a 20 N load was analyzed, the lowest and the highest friction coefficients were obtained from the DCT-24h-HTT and the CHT samples, respectively. The friction coefficient under a 10 N load ranged from 0.24 to 0.40 while the friction coefficient under a 20 N load was found to vary between 0.29 and 0.58.

CHT and DCT Vanadis 4 samples were subjected to a wear process under 10 and 20 N loads. The results obtained from the wear tests performed on the samples are presented in Fig. 7. It was determined that the wear rates increased as the amount of applied load increased. When the samples worn under each load (10 and 20 N) were examined, the highest wear rate was observed in the CHT-LTT sample, whereas the lowest wear rate was observed in the DCT-24h-HTT sample. The lowest wear rate of the DCT-24h-HTT sample can be attributed to increasing hardness (65.8 HRC) and
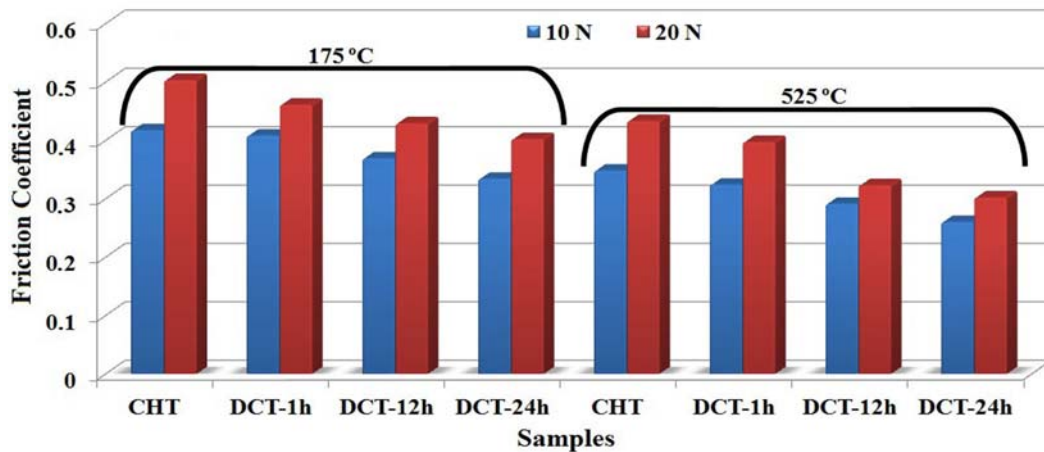
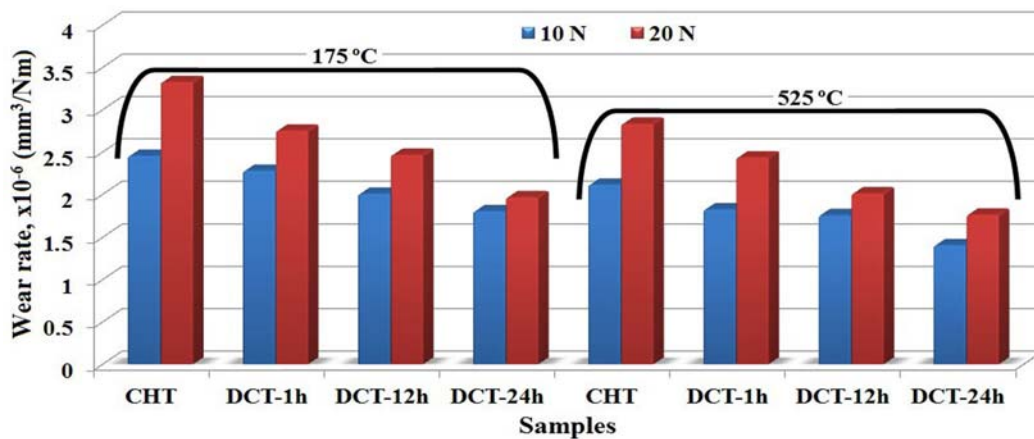Fig. 6. Friction coefficients of CHT and DCT samples.

Fig. 7. Wear rates of CHT and DCT samples.

wear resistance due to the maximal transformation of retained austenite to martensite, the formation of fine carbide particles and homogeneous distribution of carbides (Fig. 2d).

While the wear rate of the CHT-LTT sample was 2.458×10^{-6} mm^3 N^{-1} m^{-1} under a 10 N load, it increased to 3.326×10^{-6} mm^3 N^{-1} m^{-1} as the load increased (36 %). The wear rate of the DCT-24h-HTT sample decreased to 1.402×10^{-6} mm^3 N^{-1} m^{-1} under a 10 N load. Lower wear rates were obtained from the DCT samples when compared to the CHT samples. It was observed that the DCT samples have a higher wear resistance. This can also be related to the presence of retained austenite in the structure and its transformation to martensite under the DCT as stated earlier. The most important effect of tempering on the DCT samples was improving the wear properties of the alloy. The better distribution of martensite laths, along with the more uniform and finer distribution of carbides, increased the wear properties, especially for longer times. Firouzdor et al. [11] investigated the effects of the cryogenic process on tool life during the drilling of the carbon steels at high speeds by M2 HSS drills. It was determined that the cryogenic process and the tempering process applied afterward achieved a 77 and 126 % improvement in drill lives, respectively. Amini et al. [29] investigated the effect of holding time (24–120 h) at a liquid nitrogen temperature during the DCT on the microstructural changes of AISI D3 tool steel. They showed that the hardness, microhardness, microstructure uniformity and carbide percentage reached their optimum values with a 36 h holding duration. Gunes et al. [30] found that similar amounts of carbon and martensite in bearing steel (AISI 51200) increased the hardness values, consequently leading to an increase in wear resistance.

Enhancement of wear resistance by cryogenic treatment is attributed to the elimination of retained austenite, refinement of carbide particles, and their homogeneous distribution. The presence of numerous ultrafine carbides in cryo-treated steels assists in attaining the micro-stress distribution of the material in a way that results in favorable crack growth resis-
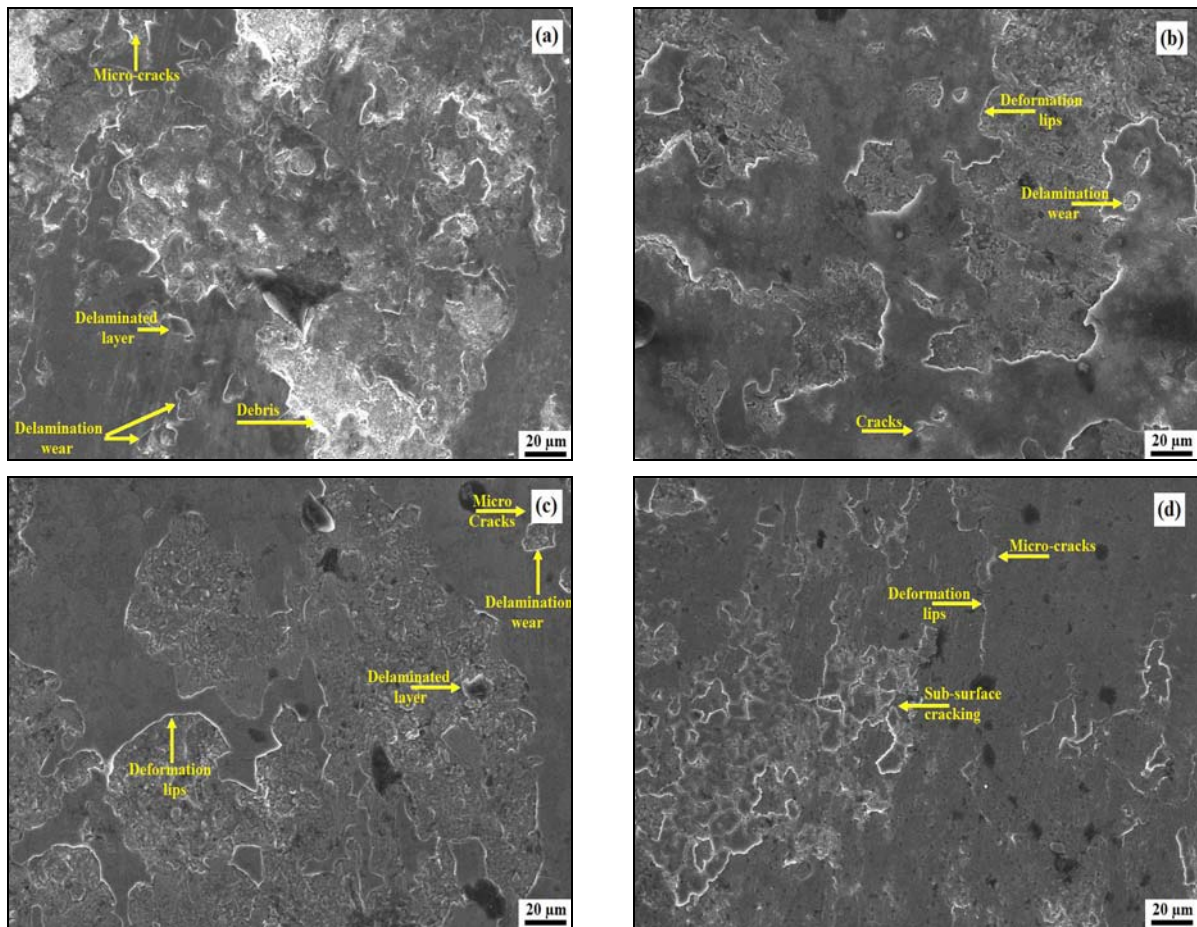
Fig. 8. SEM micrographs of the worn-out surfaces of samples tempered for 2 h at 175 °C: a) CHT-LTT, b) DCT-1h-LTT, c) DCT-12h-LTT, d) DCT-24h-LTT samples.

tance of the material. This, in turn, leads to higher wear resistance [12].

SEM images of typically worn surfaces generated during wear tests of CHT, DCT (1, 12, and 24 h) tempered at 175 °C and 525 °C at a 20 N normal load and a sliding velocity of 0.15 m s^{-1} are shown in Figs. 8 and 9a–f, respectively. The wear mechanisms have been examined by analyzing the morphology of the worn-out surfaces of the CHT and DCT samples. The morphologies present deformation lips, micro-cracks, and delamination characteristics, as marked by arrows. The SEM micrograph of the worn-out surfaces of the samples (Figs. 8 and 9) shows that the predominant wear mechanism is delamination. The worn-out surface of the DCT samples shows a smoother surface than the CHT samples. The DCT samples exhibit fewer ridges; typically the worn surface roughness of the DCT-24h sample is the lowest among all samples (Figs. 8d and 9d). This can be attributed to the formation of fine carbide particles because they lead to less surface damage during wear testing. The wear mechanisms were examined by analyzing the morphology of the worn-out surfaces of the DCT samples and the collected wear debris generated during the stable wear

regime. Nature, size and morphology of the wear debris generated during wear tests of the DCT samples are shown in Figs. 8 and 9b–d. It was observed that micro-cracks formed on the sample surface and degraded as a result of wear (Fig. 9d). In Figs. 8 and 9a–c, delamination wear with the advancement of micro-cracks was observed in the wear tracks of the CHT, DCT-1h, and DCT-12h samples. EDS (Energy Dispersive X-ray Spectrometry) analysis of points A and B in Figs. 8d and 9d are given in Figs. 10a,b. No oxidation was observed on the surfaces of the samples after the wear tests (Fig. 9). The reasons behind this might be the low shear rate and the cleaning of the surfaces of the samples in an ultrasonic bath after the wear tests.

4. Conclusions

The effects of DCT on the wear behavior, hardness, and microstructure of Vanadis 4 cold-work tool steel, which were cryo-treated for different holding times, were studied. The results obtained can be summarized as follows:
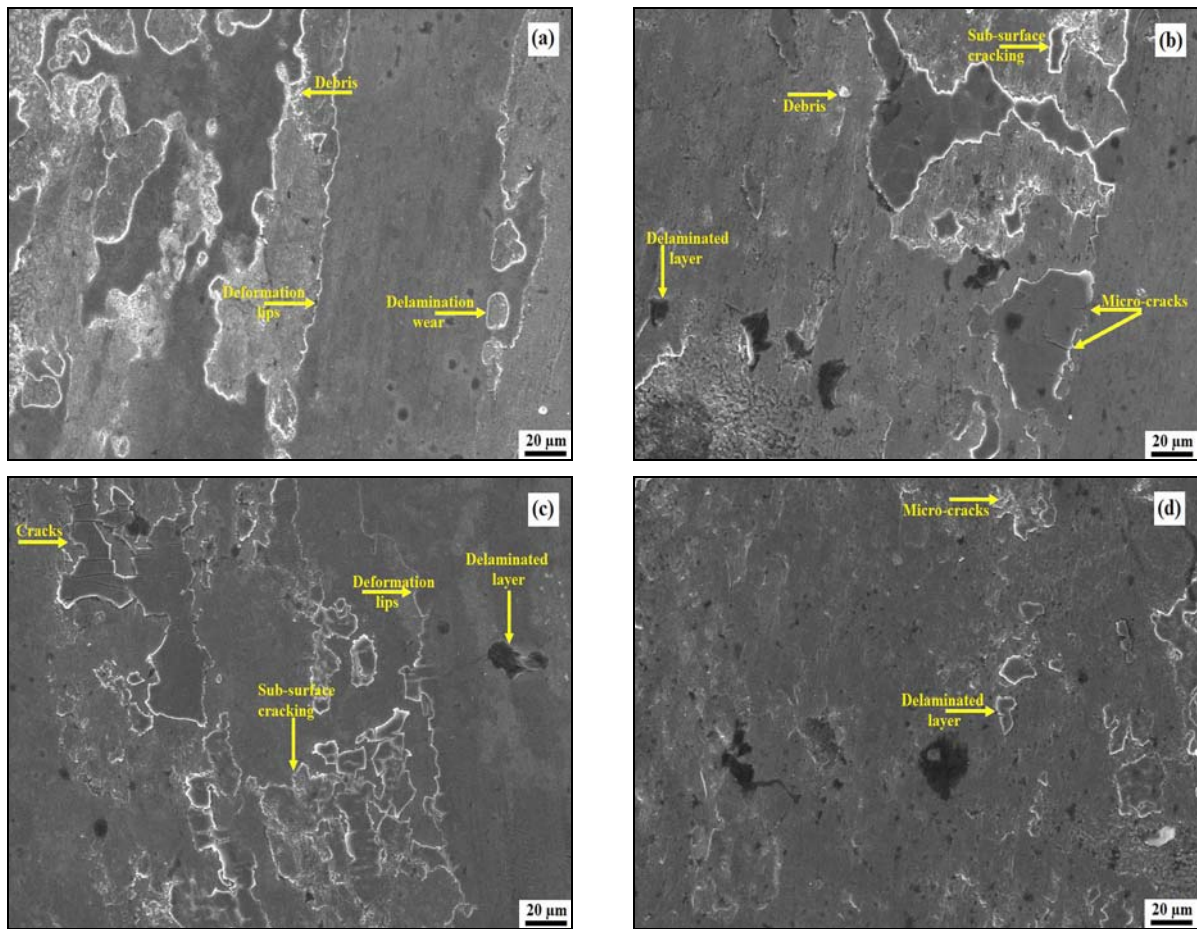
Fig. 9. SEM micrographs of the worn-out surfaces of samples tempered for 2 h at 525 °C: a) CHT-HTT, b) DCT-1h-HTT, c) DCT-12h-HTT, d) DCT-24h-HTT samples.
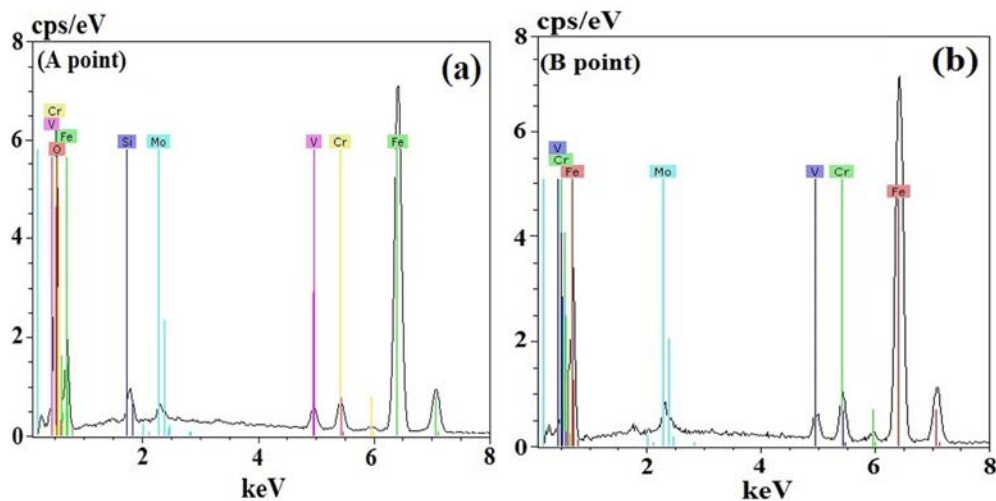

Fig. 10. EDS analysis of the worn-out surfaces of the CHT and DCT samples.

– The DCT created a more homogeneous carbide distribution with a more uniform particle size and some newly formed small-sized carbides. It was shown that the microstructure of the DCT samples was changed by varying the holding duration.

– The cryogenic process increased the redistribution of chromium carbides in the microstructure, the transformation of austenite to martensite and the hardness of the samples; this, in turn, led to an increase in the wear resistance of the Vanadis 4 steel.

– The highest wear rate was observed in the samples worn under loads of 10 and 20 N in CHT samples, while the lowest wear rate was observed in the DCT--24h samples tempered at 525 °C due to the maximum transformation of retained austenite to martensite, the formation of finer carbide particles and the homogeneous distribution of carbides after DCT.

– Delamination wear with the advancement of micro-cracks was observed in the wear tracks of the DCT samples.

– It was observed that a high tempering temperature was more effective than deep cryogenic treatment in the transformation of austenite retained in the microstructure of Vanadis 4 tool steel to martensite.

Acknowledgements

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