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Effect of Deep Cryogenic Treatment on Wear Behavior of Cold Work Tool Steel

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Abstract: Shock resisting cold work tool steel is one of the most applicable steels for several applications such as cutting sheets, chisels, hammers, etc. It has been categorized according to its characteristic properties into different categories as hot and cold work tool steel. This work aims to study the effects of conventional and deep cryogenic treatment (DCT) on shock-resistant cold work tool steel. In this study, three alloys were cast and prepared with different carbides forming elements such as vanadium (V) and niobium (Nb). The samples were quenched in water at 900 °C followed by a tempering treatment at 200 °C for 30 min. After quenching in water, the other samples were subjected to DCT at −196 °C for a 5-h soaking time, followed by tempering at 200 °C for 30 min. To study the wear behavior of the three heats, pin-on-disc tests were used, where the sliding speed was kept constant at a value of 0.5 m/s. The normal applied loads during the wear test were 50 N and 100 N. In order to understand the wear behavior, wear tracks were studied by scanning electron microscopy, coefficient of friction and weight loss were evaluated. The results showed that the lowest average coefficient of friction was achieved by a sample of steel 3 with quenching + DCT at a load of 100 N of load by value of 0.33. A sample of steel 3 at load 50 N achieved the lowest weight loss by using DCT plus tempering. On the other hand, a sample of steel 3 achieved the lowest weight loss at 100 N by using quenching + DCT.

Keywords: cold works; tool steel; coefficient of friction; wear behavior; worn surfaces; deep cryogenic treatment

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1. Introduction

Metal forming (working) tools are necessary when manufacturing metal components for a variety of industries. For instance, the automotive industry accounted for roughly 44% of all metal parts sold globally in 2015. The need for these kinds of instruments increased as automobile production increased in nations like the United States, China, Japan, and Germany [1]. The global market for stamping/punching metals is expanding steadily along with the rise in demand from the automobile industry. Until 2019 [2], a 3% annual growth rate is anticipated for the global market for metal forming.

Tool steels were developed to be resistant to wear at the temperatures needed for forming and cutting operations [3]. When resistance to wear, strength, toughness, and other qualities are chosen for optimum performance, tool steel is employed in a wide range of applications [4]. Cold work, shock resistance, hot work, high speed, mold, and special-purpose tool steels are the six major groups into which they can be separated [5]. In general, several tool steels meet the requirements for a certain application. Thus, the final selection is influenced by the tool life as well as the material and fabrication costs. Adhesion wear is the first logical component to consider when determining tool life [6].

After the common heat treatment known as "retained austenite", only a small proportion of austenite is left in tool steels. Retaining austenite as a soft phase in steels has the potential to shorten product lifespan and can be converted to martensite under certain working circumstances [6]. This new martensite could provide a number of problems for working tools. This unique martensite is quite fragile, in contrast to the tempered martensite utilized in tools. [7]. Additionally, this martensite shortens the lifespan of the product and develops micro-cracks. Moreover, dimensional instability is provided by the maintained austenite-to-martensite transformation [8].

Carbon steel's high hardness and abrasion resistance are critical requirements for cold working [9]. It is possible to increase the resistance of the core and surface layer or apply a coating if significant surface pressures are produced when using the tool [10]. The tool's operating load and strains must be supported by a surface layer resistance that is high enough. By adding alloying elements to steel, this can be achieved. The most common tool steel grades contain alloying elements, such as Cr, Mo, W, and V, that have a strong affinity for carbon [6]. As a result, hard phases can occur in the microstructure of these steels. This microstructure of steel increases its resistance to abrasion. By utilizing alloying elements in the proper amounts, tools' strength and abrasive properties can be improved [11].

The removal of material from solid surfaces, which may result in the failure of industrial components, is referred to as wear [12]. Over the course of many years, numerous examinations of wear modes have been conducted [13,14]. The chemical composition [15], microstructure, load level, and surface qualities of materials all have a significant impact on wear mechanisms and rates. Mild or oxidative wear mechanisms are seen in steels. The production of large, metallic wear debris causes severe wear to begin shortly after sliding begins at low load levels, resulting in a rapid wear rate. After this, the wear mode transitions to a continuous, moderate condition with fine oxidized wear debris [16].

Wear resistance in steels is typically increased by the martensitic phase change [17]. However, a significant fraction of ferrous martensitic components experience unexpected failures, and these failures are typically brought on by wear [17]. In light of this, the volume percentage of the martensite phase not only has a major impact on the surface life of industrial parts, but it also occasionally has the opposite effect [18].

Because of rising die maintenance costs and trash rates, tool steel wear in sheet metal forming continues to be a major concern for the automotive industry [7,16]. Due to the high contact pressures created by sliding contact between the die and the sheet materials, cold-forming tools are subjected to significant tribological stresses that cause significant frictional heat generation, which has an impact on the tool steel's wear characteristics and bulk material [19]. Due to the high temperatures that hot stamping tools are exposed to, the wear of tool steels and the prevalent wear processes have been carefully examined [20].

Understanding the effects of cryogenic processing requires an understanding of how metals are heat-treated. Steel is heat-treated primarily to increase wear resistance through hardening. For extended durability and performance, gears, bearings, and tools, for instance, require exceptional wear resistance [5].

Cryogenic processing (CP) is currently employed in the aerospace and industrial industries, sports and musical instruments, firearms, etc. to enhance the performance of numerous components [21]. A significant amount of research has been done in the last ten years to improve the tribological characteristics, such as wear resistance, of tool/die steels using cryotreatment [22,23]. Increased wear resistance and longer intervals between component replacements for dies, punches, drill bits, end mill cutters, bearings, cams, crankshafts, blocks, pistons, blades, etc., are two benefits of cryogenic treatment of tool steels [24].

The effects of cryogenic treatment on the wear behavior of D6 tool steel were investigated by Akhbarizadeh et al. [25]. Two temperatures were employed for this: –63 °C for shallow cryogenic use and –185 °C for deep cryogenic use.

Cryogenic treatment, which is applied to goods after conventional heat treatment to boost their wear resistance in some materials and generate dimensional stability in others, is now known from recent studies to be a necessary supplemental treatment [21]. Tool steels, combining steel, cast iron, carburized steel, tungsten carbide, plastics, and composites are all subjected to cryogenic treatment [22]. The cryogenic treatment increases the wear resistance and, as a result, the product life for all of the materials indicated. In recent years, cryogenic treatment has been applied as a finishing step [26,27].

It's necessary to understand the factors affecting the wear processes in order to limit the rate of tool wear when hot stamping. The life of the die materials used in hot stamping could be extended by using this knowledge to help with tool material selection and die design [28].

Several researchers [29,30] have indicated the advantage of cryotreatment for the improvement of tool steels' wear resistance. The processes behind how cryotreatment increases wear resistance, however, have not yet been firmly identified. According to several researchers [30], the transition of retained austenite to martensite is the only reason for the improvement in wear resistance.

The large improvement in wear resistance of tool steels by cryotreatment compared to cold treatment, however, cannot be entirely attributed to the minimizing of retained austenite because this phenomenon is a property of both the cold treatment and the cryotreatment [31]. According to a number of researchers [22], the refinement of secondary carbides is the main reason why cryotreatment improves wear resistance. Nevertheless, this opinion lacks the necessary experimental support.

It is now well acknowledged that deep cryogenic treatment increases wear resistance compared to cold treatment [32] and that cold treatment itself increases wear resistance compared to standard heat treatment [33,34]. The questionnaire, however, has not yet received satisfactory responses. What is the predicted difference between deep cryogenic treatment and other treatments in terms of wear resistance? Why, even for the same material, does the reported degree of advancement in wear resistance vary so greatly [35]? Second, how does deep cryogenic therapy outperform cold treatment and shallow cryogenic treatment in terms of wear resistance? The magnitude of the reported variation in their wear behavior cannot be fully explained by the observed difference in the amount of retained austenite after deep cryogenic treatment compared to cold treatment [36].

Any differential in their wear resistance must be due to something other than the differential in retained austenite content because both shallow and deep cryogenic treatments are predicted to almost entirely remove retained austenite [37]. Through a methodical investigation of the microstructure, hardness, and wear behavior of die steel specimens subjected to conventional heat treatment, cold treatment, shallow cryogenic treatment, and deep cryogenic treatment, the main goal of the current study is to provide basic answers to these fundamental questions [38].

In industrial procedures where the ambient temperature is below 200 °C, cold work tools are used. The varieties of cold work tool steels are numerous. These steels have hardness between 51 and 57 HRC, a high compressive strength of more than 2000 MPa, adequate wear resistance as determined by the pin-on-disc test, and adequate un-notched impact toughness as determined by the Charpy impact test performed at room temperature in the transverse direction. Cold work tools are susceptible to failures from intense loading conditions, including plastic deformation, chipping, cracking, and in the worst case, tool breakage.

In the steel industry, cold work tools are widely used in processes like cutting, punching, and shearing of metallic and non-metallic materials, pressing or embossing for the production of coins, cold forming, rolling of thin strip steels, and cold extrusion of steels for the production of gears, tool holders, milling cutters, among other things. Their importance in the automotive industry, however, has recently drawn greater attention.

Since there are no previous studies on the effect of DCT on cold work tool steels; the aim of this research is to study the effect of DCT on wear behavior of cold work tool steel.

2. Experimental Work

2.1. Materials and Heat Treatment

The experimental work performed in this research involved three different compositions of cold work tool steels, as shown in Table 1. The tested samples were melted in an induction furnace under Argon atmosphere with capacity up to 100 kg and then hot-rolled at a temperature of 1100 °C, and a thickness of approximately 5 mm in a single pass from an initial thickness of approximately 10 mm, all samples were machined parallel to the rolling direction with deformation percentage equal to 50%. Conventional heat treatments were done by hardening at 900 °C for half an hour by using muffle furnace with maximum heating temperature 1200 °C, chamber size 200 × 150 × 150 mm^3 (D × W × H), heating element resistance wire and heating rate 15 °C/min. Followed by quenching by using water as a medium for sudden cooling until reaching room temperature, after quenching process treatment, the remaining samples were followed by a tempering treatment at 200 °C for half an hour, and then cooling in the air, as shown in Figure 1, that explain the heat treatment cycle.

Table 1. Chemical composition of the tested samples in weight %.

Sample Name	Chemical Composition (Weight %)										
	C	Si	Mn	Cr	Mo	P	S	Cu	V	Nb	FE
Steel 1	0.370	1.999	1.0	0.30	0.314	0.032	0.008	0.011	...	...	Balance
Steel 2	0.374	2.268	0.996	0.253	0.169	0.026	0.006	0.011	0.251	...	Balance
Steel 3	0.480	2.164	1.10	0.384	0.223	0.024	0.003	0.012	...	0.013	Balance

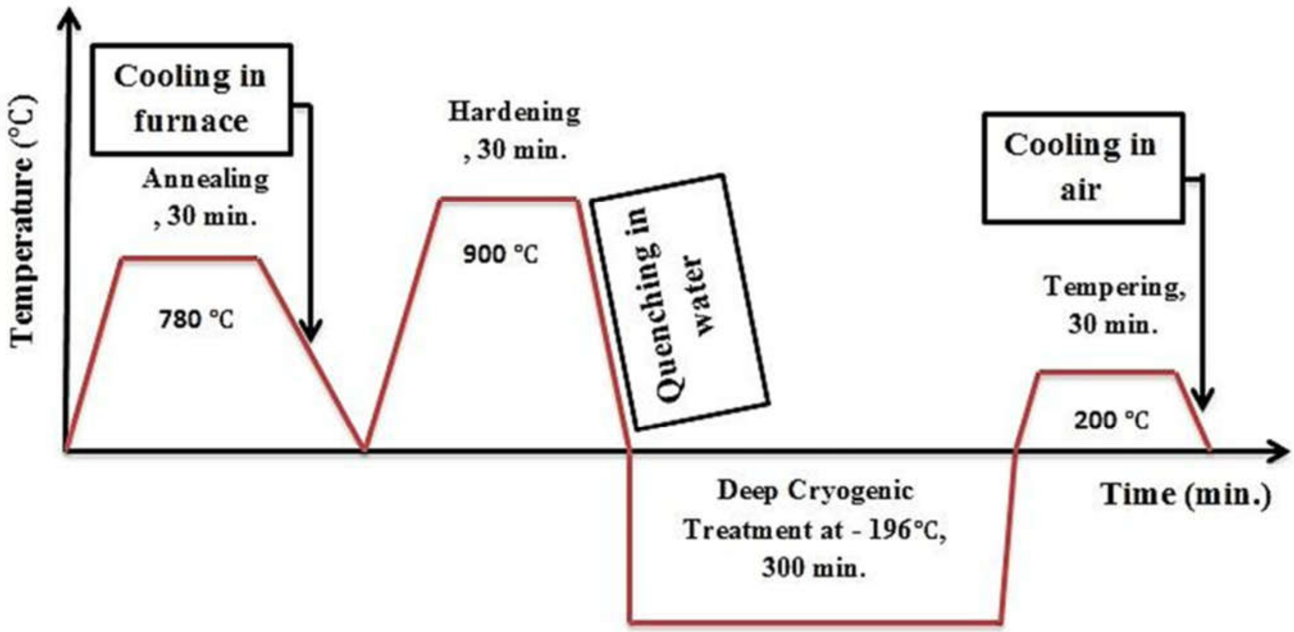

Figure 1. Heat treatment cycle for the tested steels.

For other samples, a heat treatment was carried out in order to investigate the effects of the Deep Cryogenic Treatment (DCT). The treatment cycle was as follows: After hardening in water, the samples were placed in liquefied nitrogen at a temperature of −196 °C for 5 h, and then a tempering treatment was done at 200 °C for half an hour, followed by cooling in the air.

2.2. Microstructure

Microstructure analysis was carried out by image analyzer software with an inverted microscope (Make-CARL ZEISS Germany, Model-Axiovert 40 Mat). On endless emery belt (80) paper, carefully prepared samples were first leveled on the surface. In order to make the surface free of scratches, additional samples (240, 400, 600, 800 and 1000) were treated to separate polishing using emery paper. To achieve a better finish on the polished surface, the final polishing was carried out on a velvet cloth polishing machine with intermittent applications of fine alumina suspensions. For the purpose of identifying the microscopic components of ASTM A681 tool steel, a freshly made etchant called "Nital", consisting of approximately 5 mL of nitric acid and 100 mL of ethyl alcohol (or about 5%), was utilized, After each step, samples were washed with water and ethanol and then dried in air.

A field emission scanning electron microscope (FESEM, Carl Zeiss Sigma AG, Oberkochen, Germany) and laser scanning confocal microscopy (LSCM, VK − ×200, Keyence Ltd., Osaka, Japan) were used for microstructure samples characterization.

2.3. Hardness and Impact Test

Hardness measurements were carried out on specimens having similar dimensions as those used for microscopy. A Zwick/Roell ZHR hardness tester with a diamond indenter and a load of 150 kg was used to assess Rockwell C hardness at room temperature with test area 250 × 150 mm^2. Each reported hardness value is the average of four measurements.

Impact toughness testing with V-notched was produced and performed at room temperature around 25 °C according to ASTM E 23-05 on standard sub-sized 55 × 10 × 5 mm^3 (Length × Width × Thickness). The impact testing was carried out at room temperature with an average of three measurements.

2.4. Wear Test

Pin-on-disc equipment was used to conduct wear testing for various numbers of specimens, using the parameters shown in Table 2 and Figure 2. The pin samples had a diameter of 5 mm and a length of 12.5 mm. The revolving disc's counter face had a wear track diameter of 32 mm, and this was where the pin was secured. Through the use of a dead weight loading method, the pin was forced up against the disc. Precautions were taken to ensure that the load was placed in the correct direction before the commencement of each experiment.

Table 2. Wear test parameters.

No.	Process Parameters	Value
1	Disc Rotation Speed	298 R.P.M
2	Temperature	Ambient
3	Wear Track Radius	16 mm
4	Load	50 N, 100 N
5	Time	300 s
6	Sample Dimensions (Dia. X L)	5 mm diameter with 12.5 mm length
7	Linear Sliding Speed	0.5 m/s

Acetone was used to properly clean every sample before testing. Then, each sample was weighed individually using a digital scale with 0.1 mg precision. The sample was then installed on the tribometer's pin holder and prepared for the wear test. The load and time were set for each experiment to be between 50 and 100 N and 300 s, respectively.

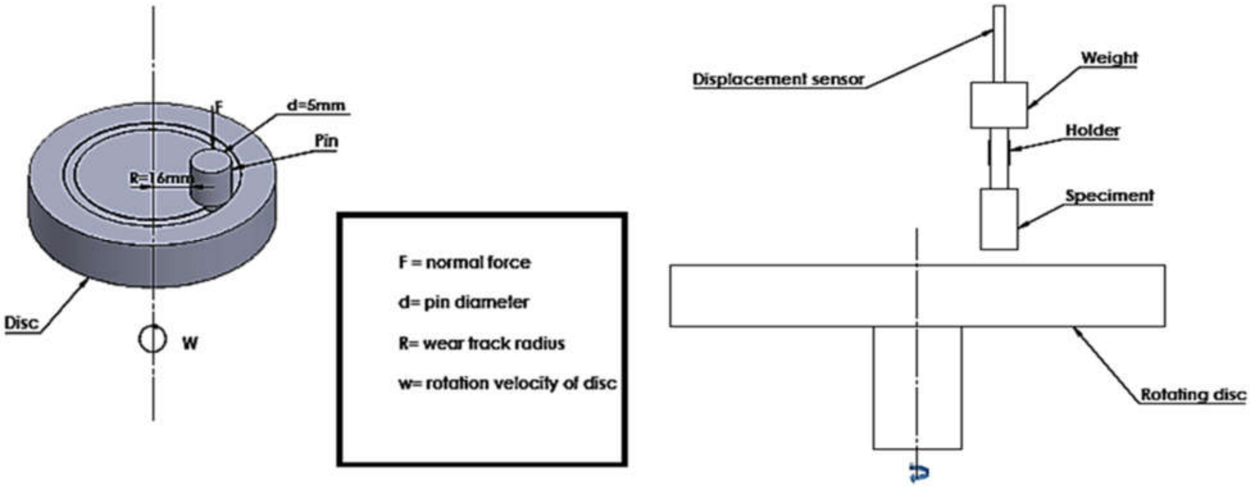

Figure 2. Schematic views of the pin-on-disk apparatus.

The rotating disc (EN31 steel disc) with a worn track diameter of 32 mm was used to hold the pin against the counter face. Through the use of a dead weight loading method, the pin was forced up against the disc. All specimens were put through a wear test with typical loads of 50 and 100 N and a sliding speed of 0.5 m/s. Three sample wear tests were conducted for each load and heat treatment condition.

3. Results and Discussions

3.1. Microstructure Analysis

The samples of shock-resistant cold work tool steel were examined using an optical microscope after they had been heat-treated. Full annealing was used in this research to establish the softest condition required in the cold work shock resisting tool steel. In fact, the annealing process was used to improve machinability and produce a homogeneous microstructure.

Figure 3 depicts a microstructure matrix composed of pearlite and ferrite [7]. Surely, the ferrite phase occurred due to the high percentage of silicon. The presence of the ferrite phase is undoubtedly due to the high percentage of silicon. When the microstructures of samples steel 2 and steel 3 are compared to those of sample steel 1, it is clear that the addition of micro alloying elements (V and Nb) to samples steel 2 and steel 3 results in a finer microstructure than that of sample steel 1.

Following the annealing process, all samples were austenitized at 900 °C for 30 min and quenched in water. The purpose of the hardening process is to obtain a fully martensitic structure to attain the required mechanical properties and to allow the particles of carbides to diffuse into the matrix.

The austenitizing process for shock-resisting cold work tool steel is considered a critical process, and therefore, it was necessary to take precautions to prevent the decarburization phenomenon, which is one of the problems facing the heat treatment of tool steel. The presence of retained austenite during martensitic transformation, on the other hand, is one of the issues that affect the mechanical properties of cold work tool steel.

In this study, the heat treatment processes (traditional treatment) end with a step of tempering specimens at 200 °C for a holding time of 30 min followed by cooling in air. Figure 3 demonstrates the microstructure after the tempering treatment process by using an optical microscope. The main purpose of the tempering process is to obtain a martensitic structure, but this structure is hard and brittle, so tempering treatment is done.

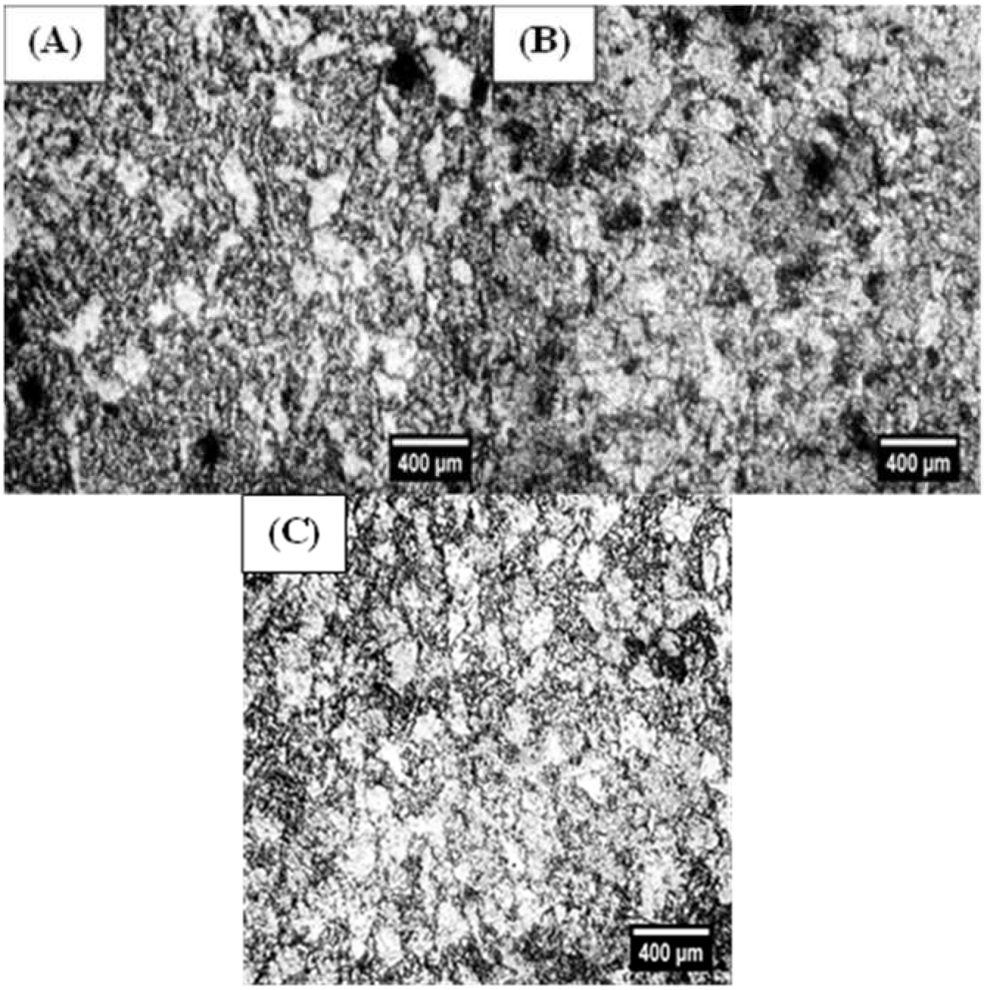

Figure 3. Optical microscope images of the specimens as annealed condition (**A**) steel 1, (**B**) steel 2, and (**C**) steel 3.

In fact, the tempering process is done for two reasons, the first of which is an increase in the heat of the martensitic structure to increase the ductility, and the second is an attempt to reduce the percentage of the retained austenite (R.A). Because the remaining austenite is in an unstable state, it can be converted into martensite using the tempering heat treatment procedure.

Figure 4 shows SEM microstructure of steels 1, 2, and 3 for both as-tempered and DCT + tempering conditions. Tempered martensite was obtained for all steels, but V-added steel (steel 2) shows the finest sub-lath structure compared with steel 1 and steel 3. The DCT + tempering condition results in a lower area fraction of retained austenite and a larger area fraction of carbides compared to tempered conditions for all steels.

Nb-added steel (steel 3) shows the lowest area fraction of retained austenite and the largest area fraction of carbides with homogeneous distribution after DCT + tempering.

The micro-cracks observed in Nb-added steel after DCT + tempering are due to the minimum retained austenite area fraction, which results in an increase in the quenching residual stresses. The large area fraction of carbides may result in the appearance of micro cracks as well. The finer microstructure obtained for the V and Nb addition was reported by Rehan [39]. It is also clear in the samples after DCT the amount of retained austenite was reduced and transformed into martensite, as shown in Figure 3B,D,F.

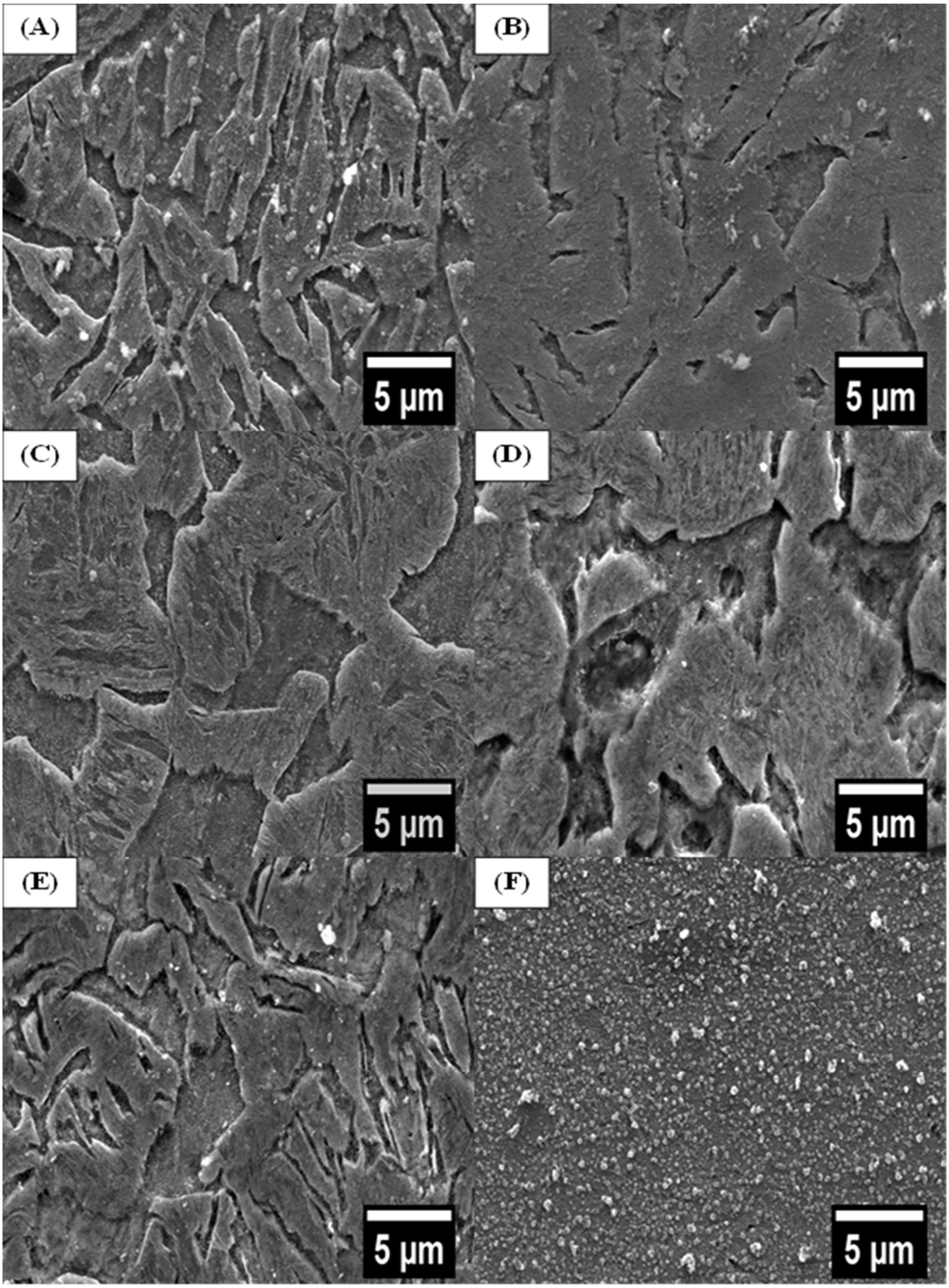

Figure 4. SEM of the specimens as tempered condition (**A**) Steel 1, (**B**) Steel 1 DCT + Tempering (**C**) Steel 2, (**D**) Steel 2 DCT + Tempering, (**E**) Steel 3, (**F**) Steel 3 DCT + Tempering.

Scanner electron microscopy (SEM) and energy dispersive X-rays (EDX) were used to confirm that the addition of the elements V and Nb to the tool steel influenced the formation of carbides. For test steel 2, as outlined in Figure 5A, there are a few carbides, but the essential microstructure is tempered martensite. On the other hand, a heat treatment was utilized utilizing DCT, and it was noticed that the essential microstructure of sample steel 3 is tempered martensite, but the microstructure is finer, and the vanishing of retained austenite was moreover observed, as clarified in Figure 5C. According to EDX analysis, carbides were detected in tests for samples Steel 2 and Steel 3 due to the inclusion of carbide-shaping components, such as vanadium and niobium, as shown in Figure 5B,D.

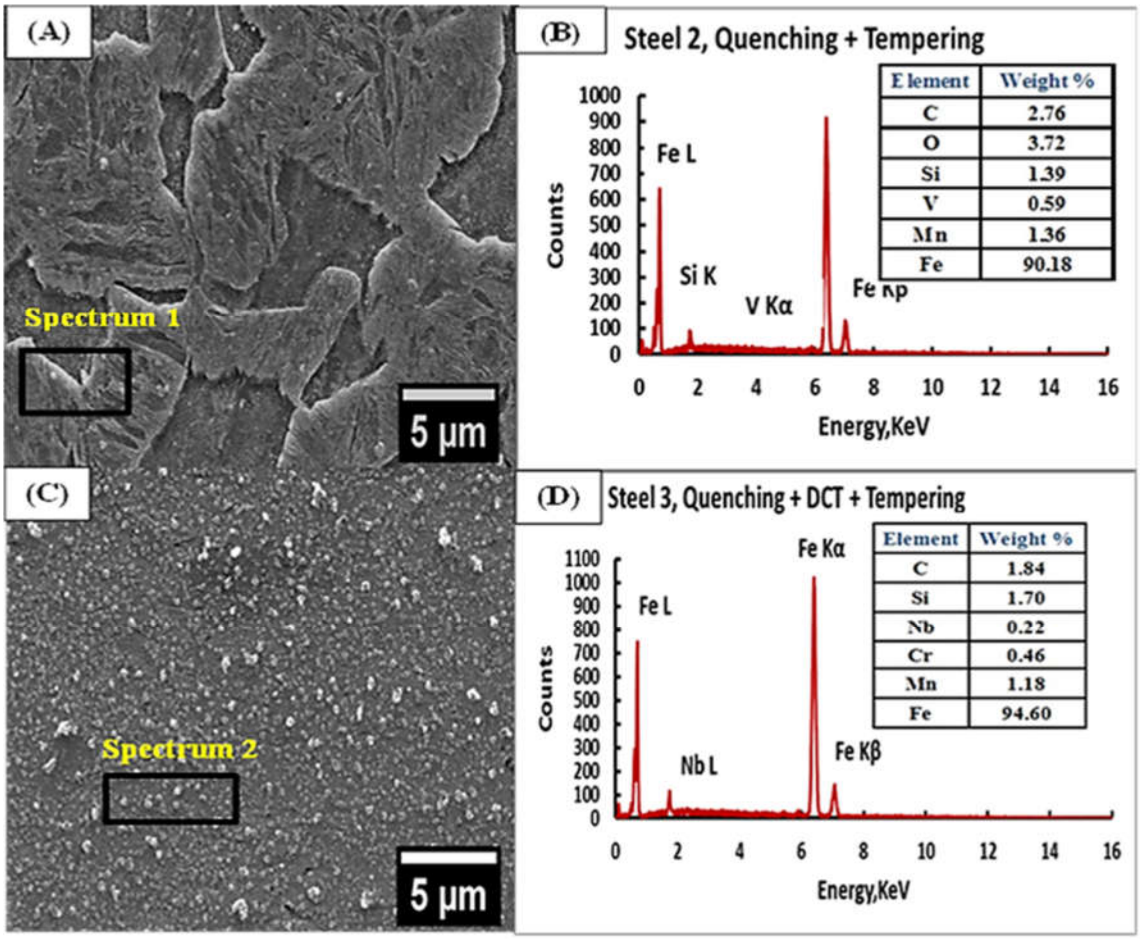

Figure 5. SEM and EDAX, (**A**) Steel 2 tempering, (**B**) EDAX for sample steel 2 tempering, (**C**) Steel 3 tempering + DCT, and (**D**) EDAX for sample steel 3 tempering + DCT.

3.2. HRC Hardness

Figure 6 shows the hardness (HRC) for traditional and deep cryogenic treatment (DCT). As shown in the following figure, the samples have the largest HRC value in the case of quenching treatment, and this is the product of the phase transformation of the steel during the quenching process. The increase of HRC hardness values is related to the transformation of lattice structure from FCC to BCC, or martensite phase. On the other hand, a slight decrease in hardness was seen after the tempering treatment process, which was due to a decrease in residual austenite, as illustrated in Figure 6.

After DCT, as shown by the presence of an increase in hardness, whether after quenching or tempering treatment, this can be explained by the transformation of the retained austenite in the case of conventional heat treatment to martensite as a result of using DCT [40].

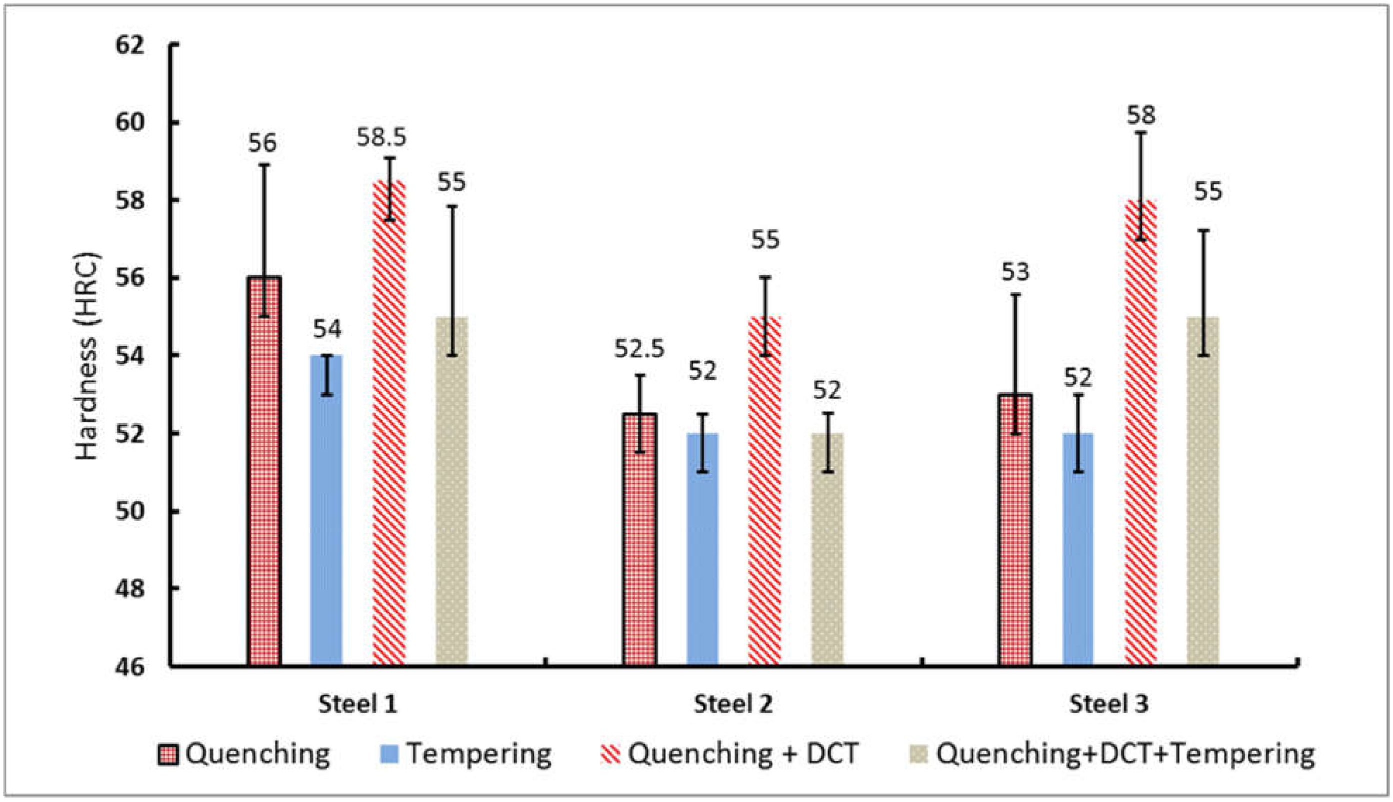

Figure 6. HRC measurements for conventional and DCT treatments.

3.3. Impact Energy

The impact test was carried out because, during the work of the cutting blade, the tool steel is subjected to a dynamic load. The results of the tested samples were plotted as shown in Figure 7. The impact toughness can be considered to give an impression of the amount of retained austenite since there is a direct relationship between the impact toughness value and the amount of retained austenite.

The effect of DCT on impact toughness absorption was carried out, and it was shown that the toughness after cryogenic treatment was 1.95 J/cm^2 whereas that for conventional quenching was 3.95 J/cm^2 for sample steel 1. On the other hand, the results also indicated a decrease in the value of the toughness test after using DCT, as shown in Figure 7, the results of toughness after DCT for sample steel 2, 7.05 and 12.4 J/cm^2 for conventional quenching On the same behavior, the toughness of sample steel 3 after DCT is reduced by about 50% when compared to conventional quenching. This slight reduction in toughness following cryogenic treatment can be attributed to an increase in martensite content once again.

The effect of tempering on the toughness of specimens subjected to cryogenic as well as conventional treatments is illustrated in Figure 7. The sample steel 1 after DCT has a value of toughness of 5.4 J/cm^2 compared to the same sample after traditional tempering treatment, which has 7.55 J/cm^2. Sample steel 2 has toughness value of 8.75 J/cm^2 after DCT lower than the value for traditional tempering treatment that is equal to 17.15 J/cm^2. On the same approach, the results were also similar for the sample steel 3 the toughness result after DCT was 4.3 and 6.3 J/cm^2 for conventional tempering treatment. In general, when comparing the samples after DCT, it was noticed that the impact toughness values had decreased compared to the conventional heat treatment, and this is explained by the fact that the percentage of retained austenite was reduced and transferred into martensite [41].

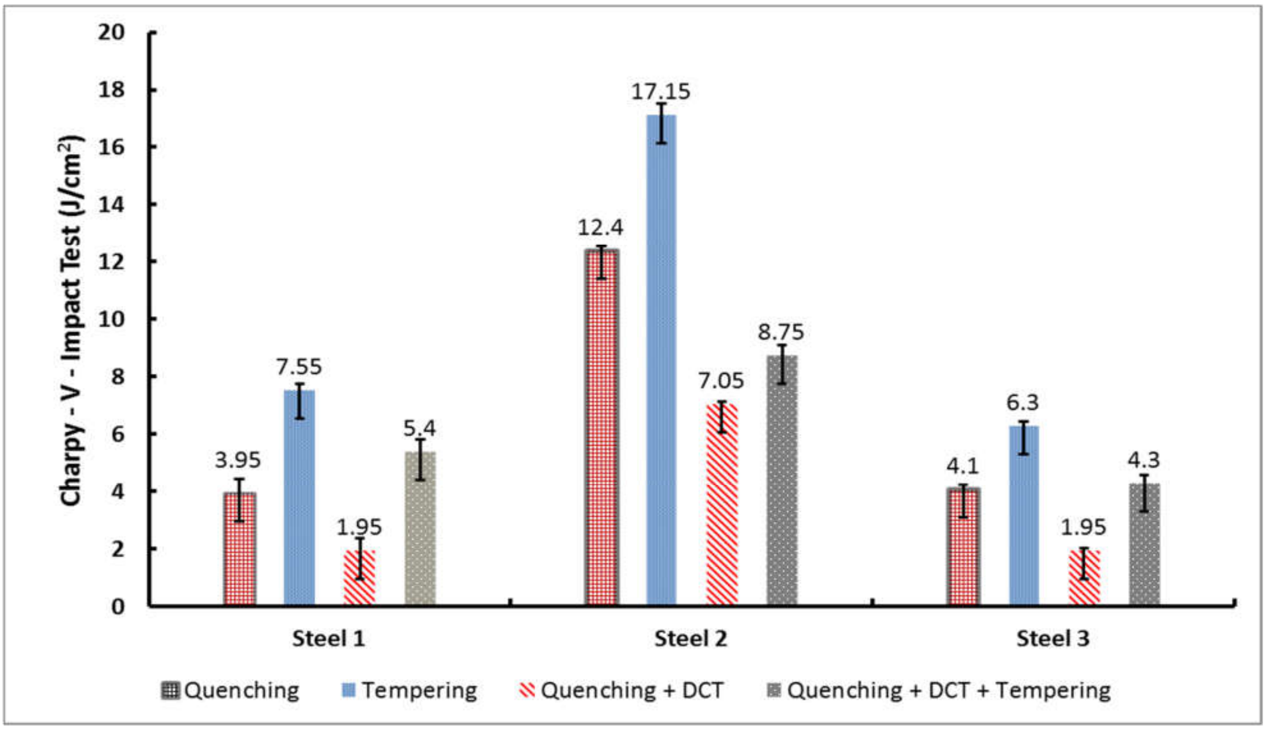

Figure 7. Impact toughness test results for traditional treatment and for DCT.

3.4. Wear Behavior

3.4.1. Friction Coefficient

Figures 8–11 show the development of the coefficient of friction (C.O.F) over time in seconds. Several phases of increasing and decreasing friction can be seen at different loads and heat treatments. When analyzing the C.O.F over time during the test, it was observed that the tested samples of tool steel have different behaviors according to the applied load.

Figures 8 and 9 explain the behavior of friction at loads of 50 N and 100 N with two heat treatment cycles, one of which was conventional heat treatment (hardening followed by quenching in water at room temperature followed by tempering at 200 °C) and the other treatment was (quenching + deep cryogenic treatment in liquid nitrogen at −196 °C for soaking time of 5 h). The behavior of friction is different for all the tested samples.

It can be seen that the conventional treatment sample requires more time to transmit from the run-out zone to the steady state zone than the tested samples of steel 1 before and after DCT exposure because the C.O.F for sample steel 1 after DCT exposure was decreased. Steel 2 has the same trend at both conditions of heat treatment. It approximately takes the same time to transmit into the steady state zone since the C.O.F after DCT was recorded as lower than conventional treatment. Figure 9 shows that at a load of 100 N, all tested samples take approximately the same time to enter the steady state zone.

Steel 3 at hardening conditions has higher fluctuations due to the presence of Nb carbides, which have C.O.F reach 0.85. Moreover, it can be noticed that the samples for conventional hardening treatment take a longer time to transmit from the transition zone to the steady state zone when compared with tested samples exposed to DCT which have the shortest time in the transition zone [40,42,43].

The decreasing COF for tested samples demonstrates the effect of DCT on wear behavior. Figure 9 explains the tested samples at the same conditions of heat treatment, but at load 100 N, by comparing C.O.F at load 100 N and 50 N, it can be noticed that by increasing the load, the C.O.F decreased. Moreover, it is clear that the sample of steel 3 at both loads (50 N and 100 N) has the same fluctuations in the steady state zone due to Nb carbides.

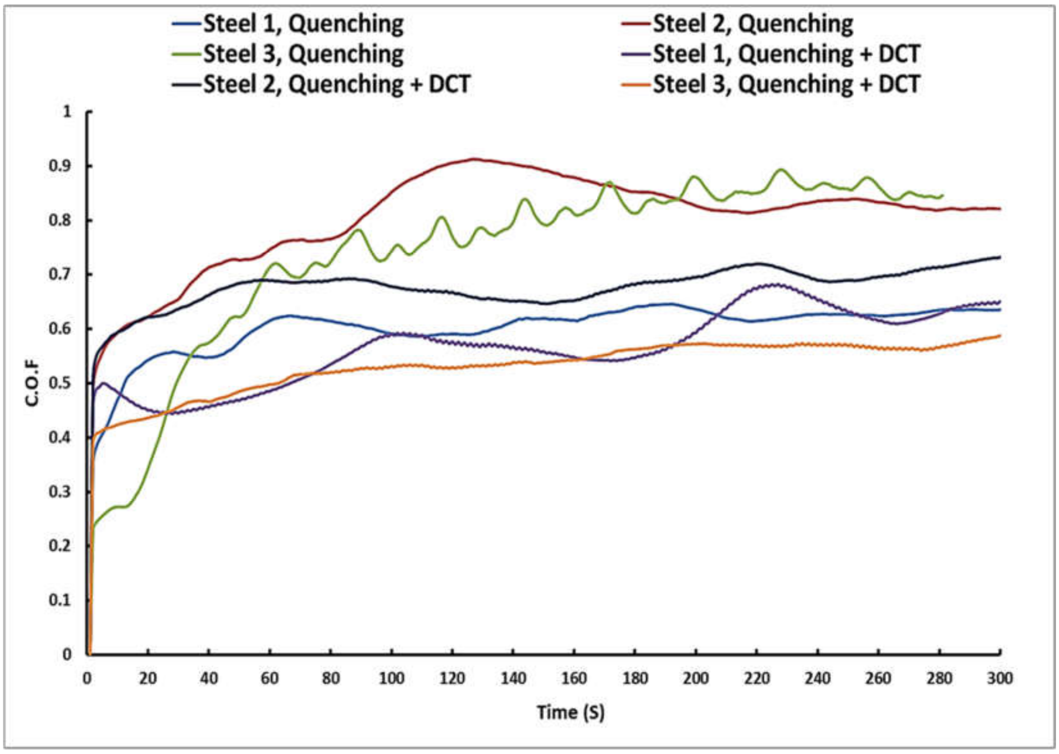

Figure 8. Coefficient of friction at load 50 N for heat-treated samples (quenching and quenching + DCT).

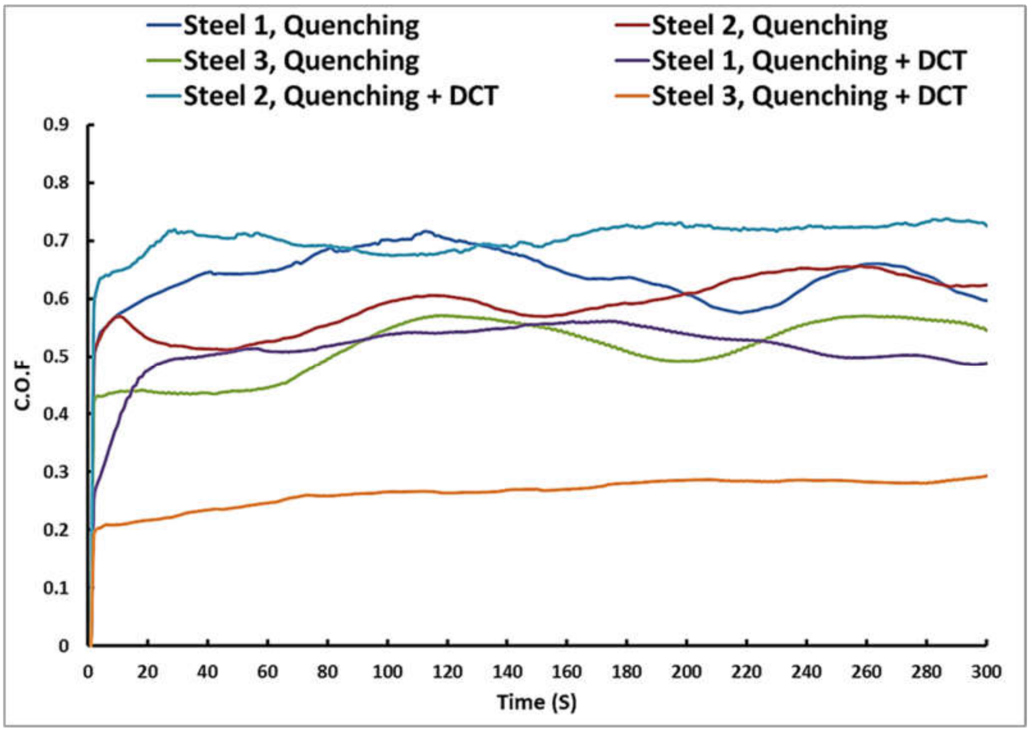

Figure 9. Coefficient of friction at load 100 N for heat-treated samples (quenching and quenching + DCT).

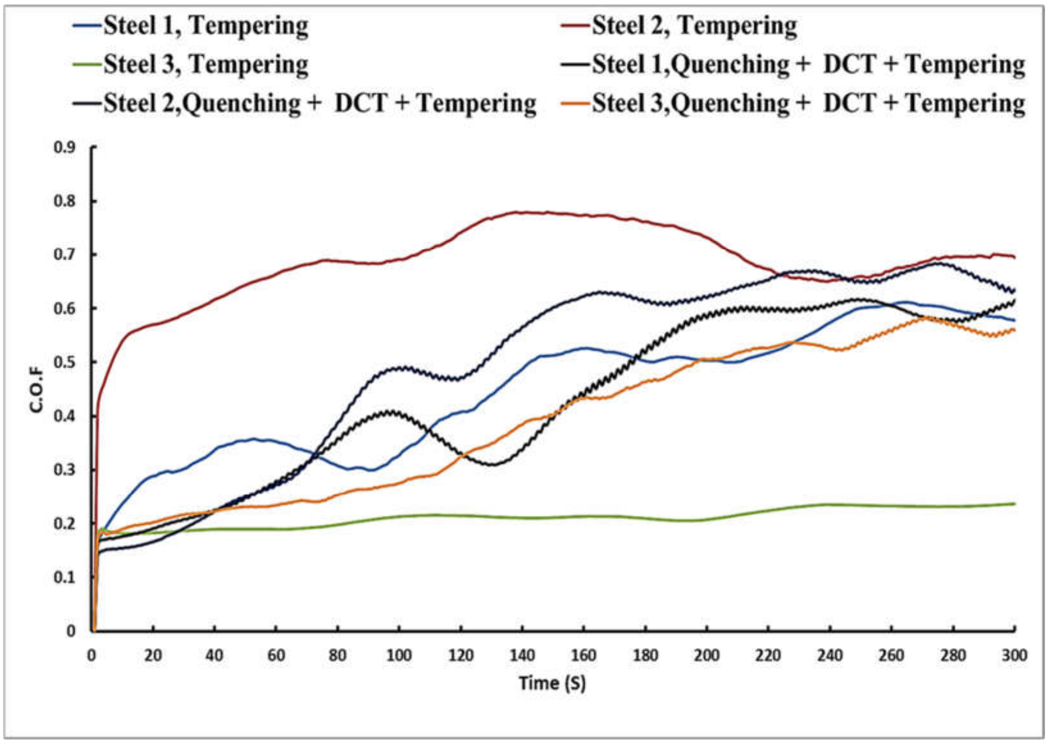

Figure 10. Coefficient of friction at load 50 N for heat-treated samples (tempering and DCT + tempering).

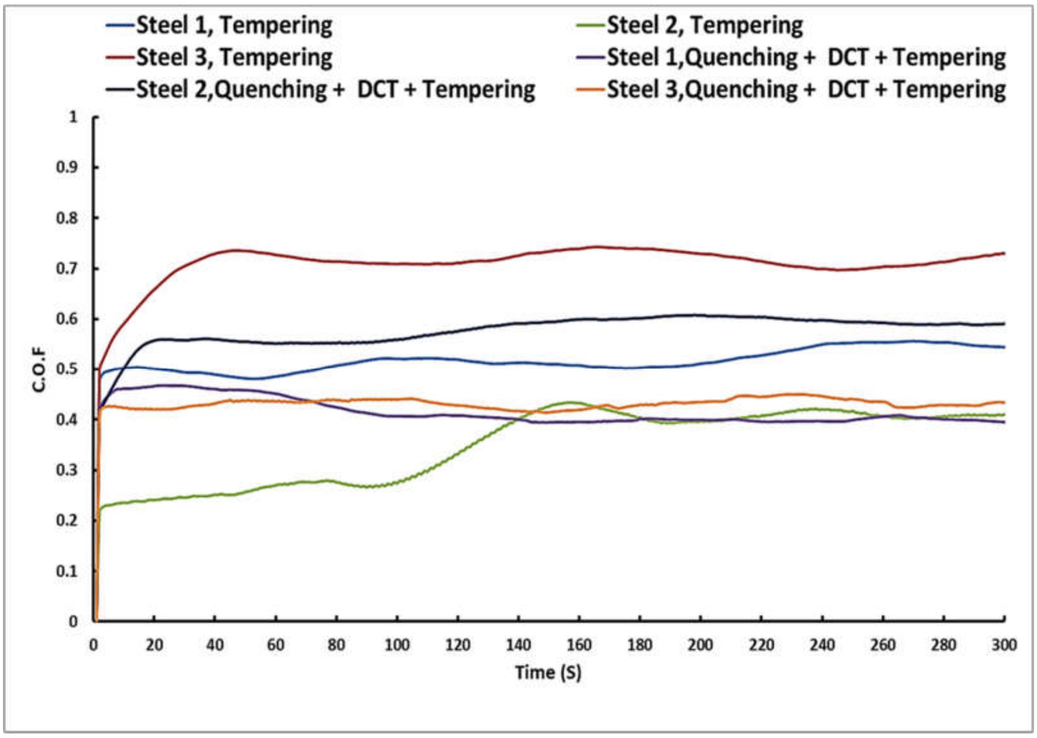

Figure 11. Coefficient of friction for at load 100 N for heat-treated samples (tempering and DCT + tempering).

Figures 10 and 11 demonstrate the behavior of C.O.F at loads of 50 N and 100 N after tempering treatment and DCT + tempering. Steel 2 tempering at 50 N has a higher C.O.F of around 0.78 due to the presence of Vanadium carbides [40].

Figure 10 shows that steel 1 after cryotreatment reaches the steady state zone in a shorter time than conventional treatment; this is due to homogeneity in microstructure.

The COF for cryotreatment with tempering, according to Steel 2, is around 0.54, which is lower than the COF for the same sample during tempering treatment, which was around 0.75.

Figure 11 explains the C.O.F at 100 N of load. As shown in this figure, sample of steel 3 at tempering treatment C.O.F around 0.8 due to the presence of Nb carbides, also in sample of steel 2 the C.O.F have sudden increase from 0.25 to 0.4 after a distance of 130 m, this is due to V carbides. These problems were eliminated by using DCT, as shown in Figure 11, for the same samples where the C.O.F may have been constant without higher fluctuations.

3.4.2. Weight Loss

Figure 12 presents the weight loss of tested tool steel specimens at both loads 50 and 100 N at different heat treatments, and at ambient temperature The same sample has the lowest weight loss after DCT (Figure 12B) at DCT + tempering for the load 50 N steel 3 at quenching treatment (Figure 12A), indicating that this sample has improved after DCT. On the other side, it can be observed that in sample steel 1, which does not contain any carbide elements, the weight loss increased after being subjected to DCT.

At both heat treatment conditions, the weight loss of steel 1 increased as the amount of applied load increased.

As increasing in load, as shown in Figure 12C,D since because of high-pressure localization, surface tempering occurred. The highest weight loss that shows up in samples at 100 N loads can be explained by the martensitic quenched structure transforming to a soft-tempered structure.

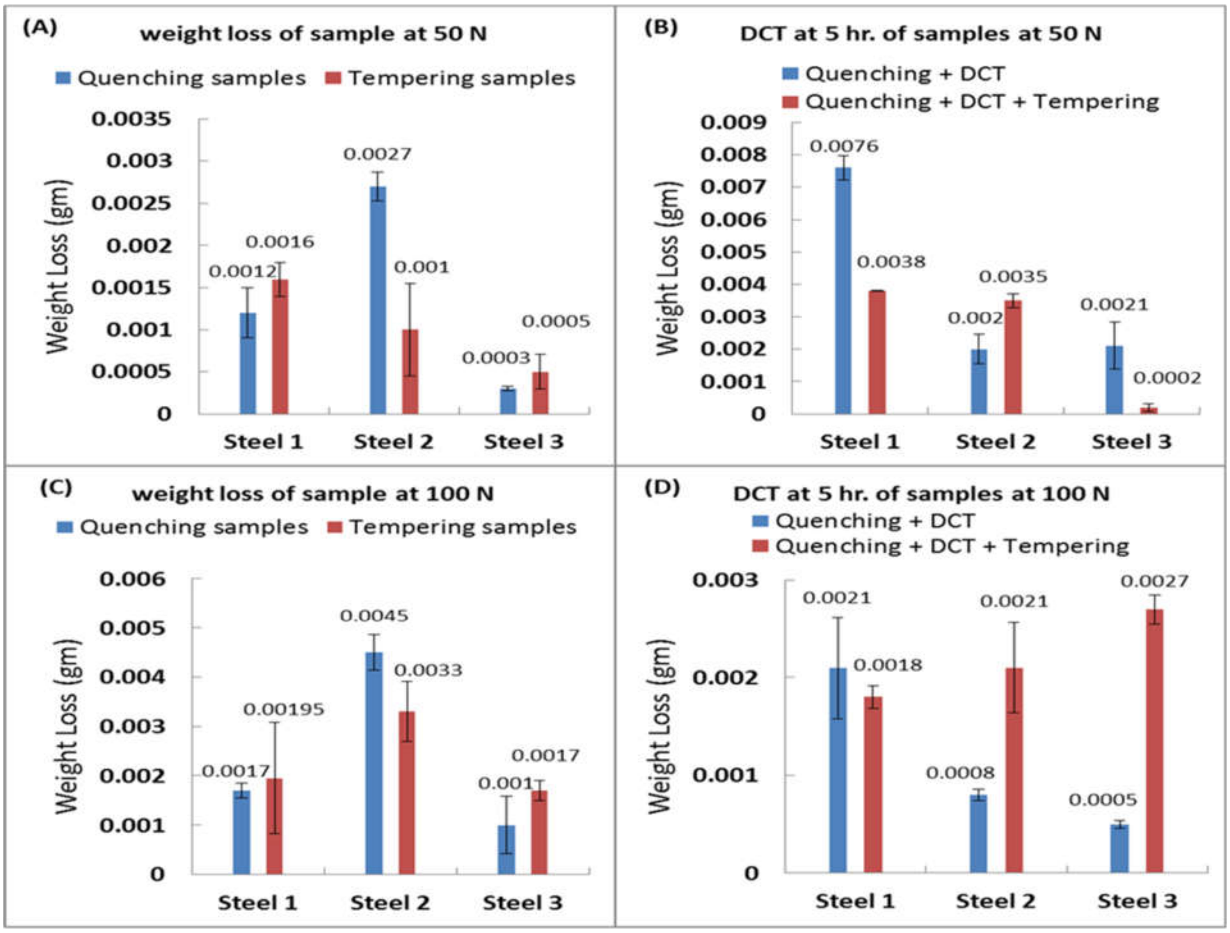

Figure 12. Weight loss for the tested samples at (**A**) quenching + tempering at 50 N, (**B**) DCT at 50 N, (**C**) quenching + tempering at 100 N, and (**D**) DCT at 100 N.

3.4.3. Average Coefficient of Friction

Table 3 shows the average coefficient of friction values at different loads 50 N and 100 N for different heat treatment conditions. The values represented in Table 3 are listed in ascending order.

When a 50 N load was applied, the minimum and maximum values were obtained by steel 3 in tempering treatment conditions and steel 2 quenching, respectively. At 100 N loads, the minimum and maximum average COF values were obtained from steel 3 quenching + DCT and steel 2 tempering condition treatments, respectively.

Additionally, it may be concluded that the average C.O.F. at load 50 N varied between 0.36 and 0.84, while at load 100 N, it was revealed to be between 0.3 and 0.73.

Table 3. Average coefficient of friction for the tested samples.

No.	Steel Sample	Average C.O.F	Standard Deviation	No.	Steel Sample	Average C.O.F	Standard Deviation
1	Steel 3 Quenching + DCT at 100 N	0.33	0.04	13	Steel 2 Quenching + DCT at 50 N	0.62	0.08
2	Steel 3 Tempering at 50 N	0.36	0.12	14	Steel 1 Quenching + DCT at 50 N	0.65	0.09
3	Steel 1 DCT + Tempering at 100 N	0.41	0.03	15	Steel 1 Quenching at 50 N	0.66	0.06
4	Steel 3 DCT + Tempering at 100 N	0.45	0.02	16	Steel 3 Quenching + DCT at 50 N	0.67	0.09
5	Steel 3Quenching at 100 N	0.47	0.08	17	Steel 2 Quenching at 100 N	0.68	0.07
6	Steel 1 Quenching + DCT at 100 N	0.47	0.06	18	Steel 2 Quenching + DCT at 100 N	0.68	0.04
7	Steel 1 Tempering at 50 N	0.54	0.07	19	Steel 1 Quenching at 100 N	0.69	0.07
8	Steel 1 DCT + Tempering at 50 N	0.54	0.09	20	Steel 2 Tempering at 50 N	0.73	0.06
9	Steel 2 DCT + Tempering at 50 N	0.56	0.1	21	Steel 2 Tempering at 100 N	0.73	0.04
10	Steel 2 DCT + Tempering at 100 N	0.57	0.07	22	Steel 3 DCT + Tempering at 50 N	0.74	0.16
11	Steel 1 Tempering at 100 N	0.58	0.03	23	Steel 3 Quenching at 50 N	0.82	0.21
12	Steel 3 Tempering at 100 N	0.58	0.09	24	Steel 2 Quenching at 50 N	0.84	0.06

3.4.4. Worn Surfaces Analysis

Figures 13–15 show the worn surfaces by using a scanning electron microscope at two different loads, 50 and 100 N. It can be believed that the worn debris builds up between both the pin surface and the rotating disc, causing abrasion wear. Additionally, the rotating disc's surface roughness contributes to abrasion wear. Moreover, with higher loads, the wear surface is subjected to greater frictional forces that result in the material's plastic deformation and obvious fracture.

Two types of wear can be distinguished by a closer look at the worn surface under an optical microscope. Depending on the normal load, these two forms of wear might happen one after the other or simultaneously. The first kind of wear is abrasive wear, which is caused by hard micro particles peeling off and leaving deep scratches in the materials. These sharp, free-moving hard micro particles from the discharged substance cause deep scratches in the tested material or fill up the resulting micro craters. These dispersed micro particles harden relative to the substrate material as a result of extreme air oxidation or plastic deformation. The two surfaces that are contacting one another are never perfectly smooth, and the contact only happens at a few contact points rather than the entire surface. The structures on the surface's peaks are plastically distorted as a result of the forces, and the atoms on both surfaces come into contact and create so-called micro couplings. When a surface area forms on one of the friction bodies, all of these micro couplings eventually break at the spots above the material contact.

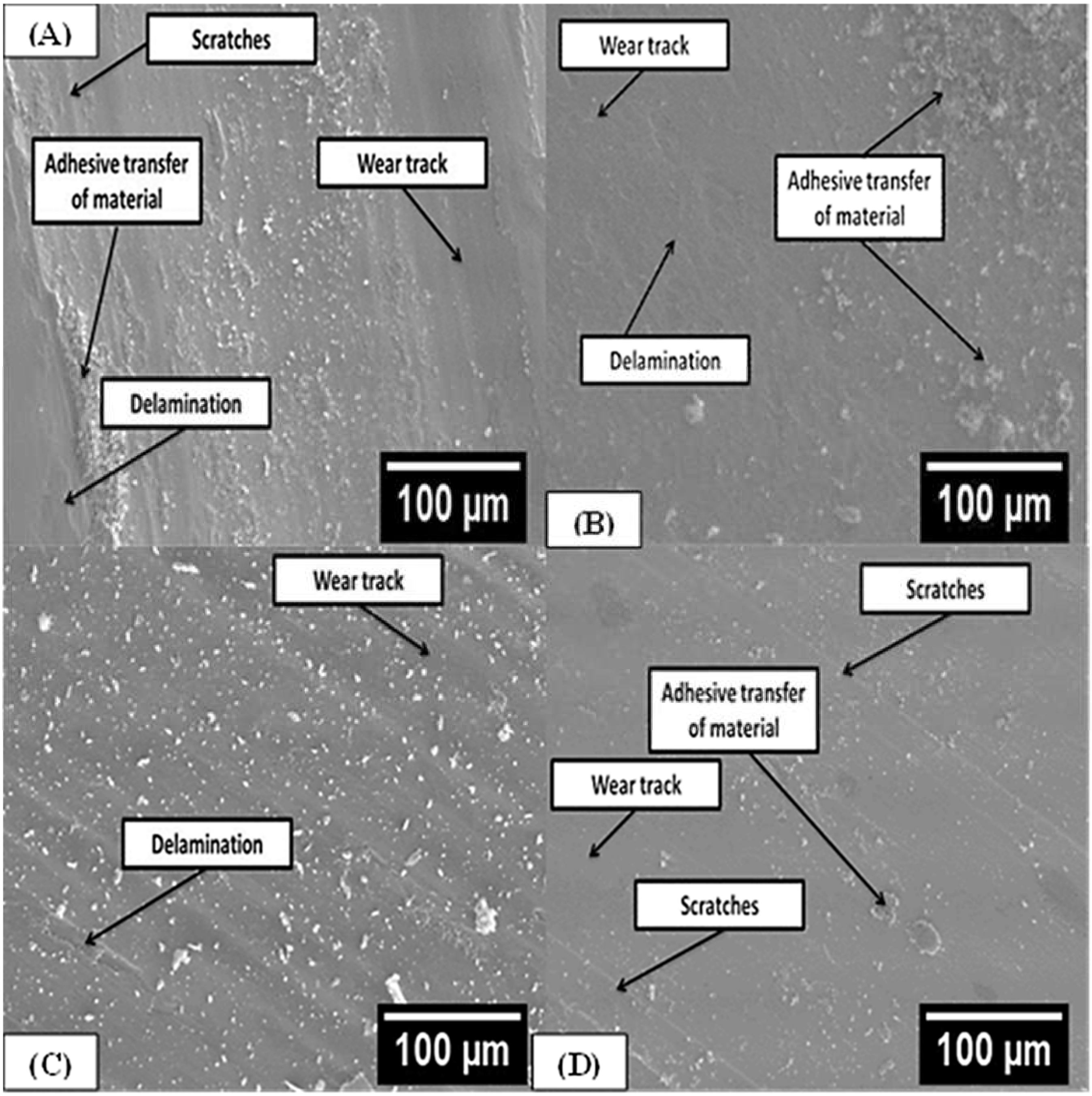

Figure 13. SEM worn surfaces for steel 1 at different heat treatment (**A**) steel 1 at 50 N, tempering treatment, (**B**) steel 1 at 50 N, DCT + tempering treatment, (**C**) steel 1 at 100 N, tempering treatment, and (**D**) steel 1 at 100 N, DCT + tempering treatment.

Two different treatments were applied to the test samples: conventional tempering at 200 °C for 30 min, followed by cooling in air to room temperature, and deep cryogenic treatment at −196 °C for 5 h, followed by the previously mentioned tempering procedure.

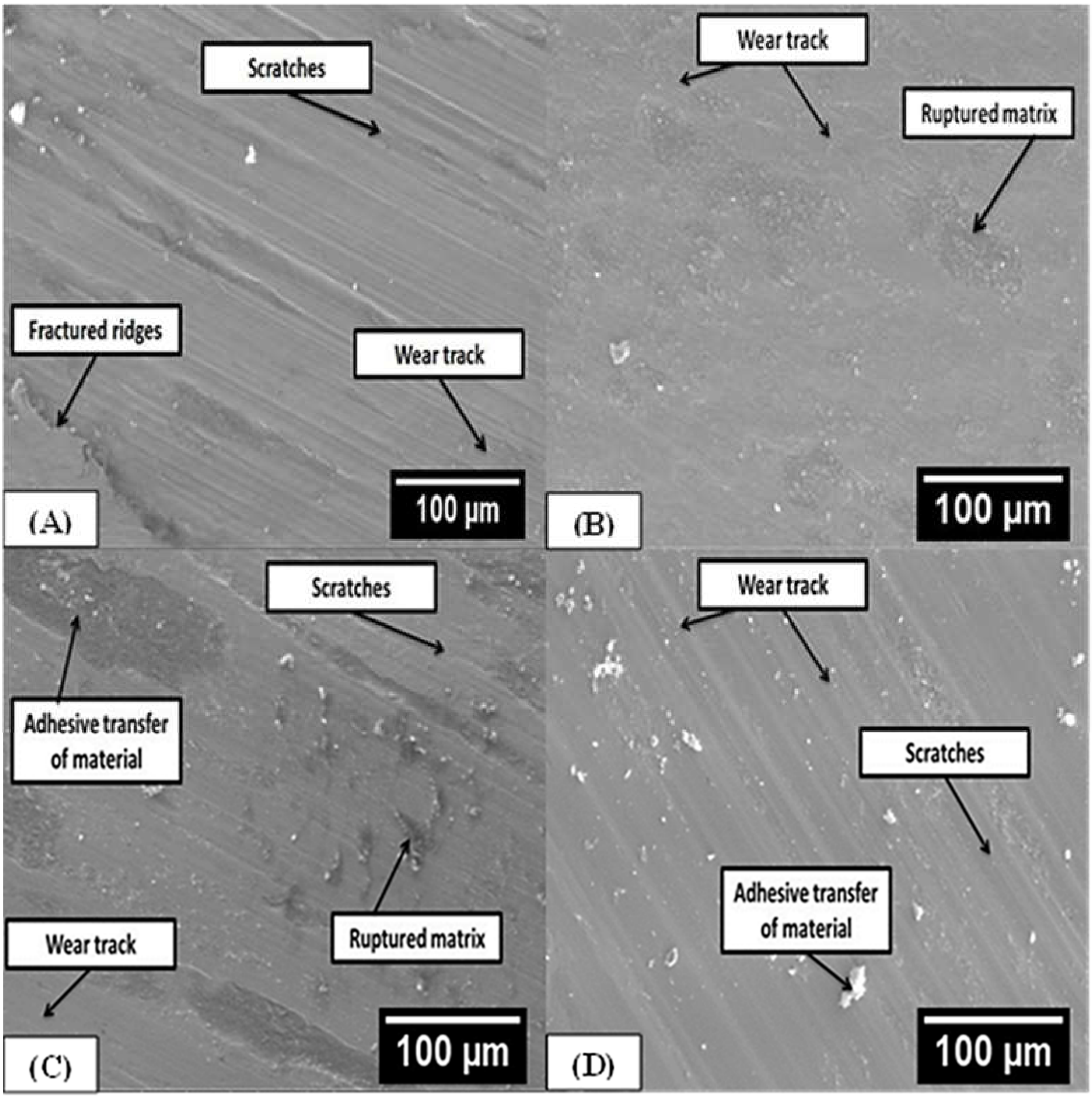

Figure 14. SEM worn surfaces for steel 2 at different heat treatment (**A**) steel 2 at 50 N, tempering treatment, (**B**) steel 2 at 50 N, DCT + tempering treatment, (**C**) steel 2 at 100 N, tempering treatment, and (**D**) steel 2 at 100 N, DCT + tempering treatment.

As shown from these figures, in general, the controlling wear mechanism is adhesive wear for the two applied loads, as presented in Figure 13A. A significant part includes the adhesive patches, which appeared as multi-layered [43]. This adhesive wear has been decreased for the same sample after being subjected to deep cryogenic treatment, as explained in Figure 13B.

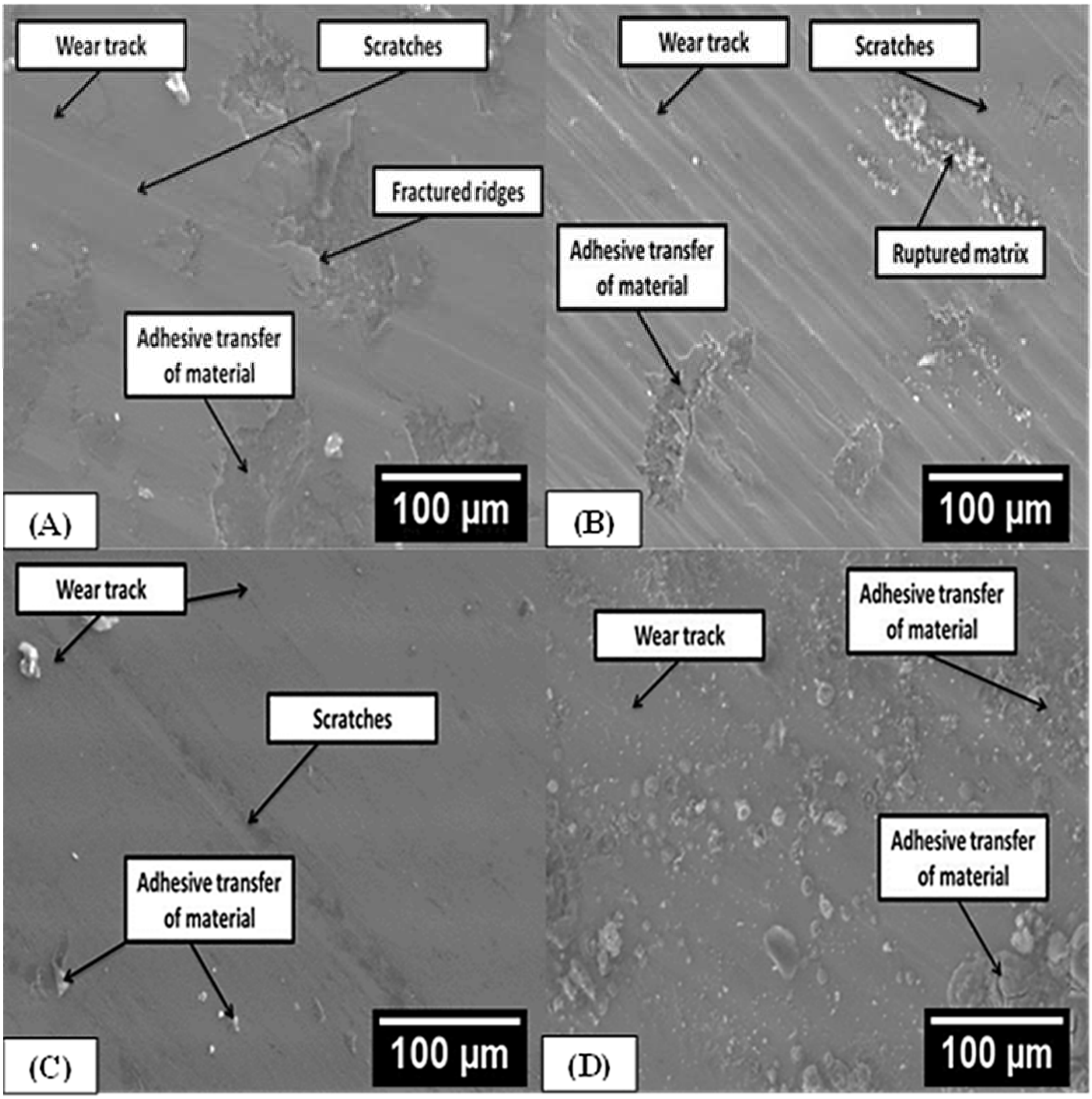

Figure 15. SEM worn surfaces for steel 3 at different heat treatment (**A**) steel 3 at 50 N, tempering treatment, (**B**) steel 3 at 50 N, DCT + tempering treatment, (**C**) steel 3 at 100 N, tempering treatment and (**D**) steel 3 at 100 N, DCT + tempering treatment.

As shown in Figure 13C, for sample steel 1 at load 100 N, the controlling wear mechanism is delaminating and debris, on the other hand, by applying DCT, the mechanism of wear is adhesive transfer of material, and debris were eliminated, as shown in Figure 13D.

Figure 14 depicts the worn-out surfaces of the steel 2 sample, which contains 0.251% vanadium as a carbide-forming element. It can be observed that in Figure 14A, at load 50 N, there are scratches and some fractured ridges, by comparison with Figure 13B, it can be seen that the scratches were reduced due to the exposure of DCT.

By increasing the load to 100 N for the sample of steel 2, as shown in Figure 14C,D. It can be observed that in Figure 14C, the dominant wear mechanism is adhesive wear, and there are scratches and some ruptured matrix. On the other side, as shown in Figure 14D, the wear phenomenon was smoother than the same sample, but exposed to tempering treatment only. In other words, DCT has a significant enhancement on the worn-out surface.

Worn-out surface morphology can be observed in Figure 15. For a sample of steel 3 that contains niobium as a carbide-forming element by 0.013%. Figure 15A shows the tested sample at load 50 N. As shown in this figure, the controlled wear mechanism is adhesive wear. The same behavior of the wear mechanism for the same sample after applying DCT is shown in Figure 15B, with the appearance of some scratches.

The same mechanism of wear can be observed by increasing the load to 100 N, as explained in Figure 15C,D.

4. Conclusions

The effect of conventional and deep cryogenic treatment (DCT) for cold work shock-resisting tool steel was examined to study the wear mechanism, and find out the weight loss and friction coefficient at two different loads 50 N and 100 N. The obtained results can be concluded as the following:

- According to the scanning electron microscope, it can be concluded that the main microstructures of the tested samples were tempered martensite and retained austenite.
- The microstructure of the samples after exposure to deep cryogenic treatment is fine compared to conventional treatment.
- The as-quenched hardness for all steels is more than 54 HRC, and almost no change was observed in the hardness after tempering. However, the impact toughness was reduced after tempering by 30 to 45% due to the transformation of retained austenite to tempered martensite.
- Maximum hardness (HRC) was achieved at quenching + DCT for steel 1, which was recorded at 58.5. In contrast, the minimum hardness was measured for two steel samples (2 and 3) at tempering treatment conditions equal to 52 HRC.
- The friction coefficient at 50 N loads has approximate behavior for all tested samples for both conventional and deep cryogenic treatment, except that samples of steel 3 at hardening treatment conditions have higher fluctuations in the coefficient of friction.
- The coefficient of friction has higher fluctuations for conventional heat treatment in steel 3 samples with niobium (Nb) addition as a carbide forming element at both loads of 50 N and 100 N.
- Steel 3 specimens with DCT + tempering conditions achieved the lowest weight loss at a load of 50 N, with a value of 0.0002 gm. Steel 3 specimens with a hardening + DCT condition, on the other hand, achieved the lowest weight loss at a load of 100 N with a value of 0.0005 gm.
- Analysis of worn-out surfaces indicates that the predominant wear mechanism is adhesive wear for both applied loads, and after applying DCT, the adhesive wear mechanism decreased due to both retained austenite transformation to tempered martensite and fine precipitation of carbides.

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References

1. Toboła, D.; Brostow, W.; Czechowski, K.; Rusek, P. Improvement of wear resistance of some cold working tool steels. *Wear* **2017**, *382–383*, 29–39. [CrossRef]
2. Podgornik, B.; Majdic, F.; Leskovsek, V.; Vizintin, J. Improving tribological properties of tool steels through combination of deep-cryogenic treatment and plasma nitriding. *Wear* **2012**, *288*, 88–93. [CrossRef]
3. Gåård, A.; Krakhmalev, P.; Bergström, J. Wear mechanisms in deep drawing of carbon steel–correlation to laboratory testing. *Tribotest* **2008**, *14*, 1–9. [CrossRef]
4. Bahrami, A.; Anijdan, S.H.M.; Golozar, M.A.; Shamanian, M.; Varahram, N. Effects of conventional heat treatment on wear resistance of AISI H13 tool steel. *Wear* **2005**, *258*, 846–851. [CrossRef]
5. Bourithis, L.; Papadimitriou, G.; Sideris, J. Comparison of wear properties of tool steels AISI D2 and O1 with the same hardness. *Tribol. Int.* **2006**, *39*, 479–489. [CrossRef]
6. Staia, M.H.; Pérez-Delgado, Y.; Sanchez, C.; Castro, A.; Le Bourhis, E.; Puchi-Cabrera, E.S. Hardness properties and high-temperature wear behavior of nitrided AISI D2 tool steel, prior and after PAPVD coating. *Wear* **2009**, *267*, 1452–1461. [CrossRef]
7. Muro, M.; Artola, G.; Gorriño, A.; Angulo, C. Wear and Friction Evaluation of Different Tool Steels for Hot Stamping. *Adv. Mater. Sci. Eng.* **2018**, *2018*, 3296398. [CrossRef]
8. Barrau, O.; Boher, C.; Gras, R.; Rezai-Aria, F. Analysis of the friction and wear behaviour of hot work tool steel for forging. *Wear* **2003**, *255*, 1444–1454. [CrossRef]
9. King, P.C.; Reynoldson, R.W.; Brownrigg, A.; Long, J.M. Pin on disc wear investigation of nitrocarburised H13 tool steel. *Surf. Eng.* **2013**, *21*, 99–106. [CrossRef]
10. Drozd, K.; Walczak, M.; Szala, M.; Gancarczyk, K. Tribological Behavior of AlCrSiN-Coated Tool Steel K340 Versus Popular Tool Steel Grades. *Materials* **2020**, *13*, 4895. [CrossRef]
11. Brezinová, J.; Viňáš, J.; Guzanová, A.; Živčák, J.; Brezina, J.; Sailer, H.; Vojtko, M.; Džupon, M.; Volkov, A.; Kolařík, L.; et al. Selected Properties of Hardfacing Layers Created by PTA Technology. *Metals* **2021**, *11*, 134. [CrossRef]
12. Holmberg, K.; Matthews, A. *Coatings Tribology: Properties, Mechanisms, Techniques and Applications in Surface Engineering*; Elsevier: Amsterdam, The Netherlands, 2009.
13. So, H.; Yu, D.; Chuang, C. Formation and wear mechanism of tribo-oxides and the regime of oxidational wear of steel. *Wear* **2002**, *253*, 1004–1015. [CrossRef]
14. Quinn, T.; Sullivan, J.; Rowson, D. Origins and development of oxidational wear at low ambient temperatures. *Wear* **1984**, *94*, 175–191. [CrossRef]
15. Ueda, M.; Uchino, K.; Kobayashi, A. Effects of carbon content on wear property in pearlitic steels. *Wear* **2002**, *253*, 107–113. [CrossRef]
16. Goto, H.; Amamoto, Y. Effect of varying load on wear resistance of carbon steel under unlubricated conditions. *Wear* **2003**, *254*, 1256–1266. [CrossRef]
17. Lin, Y.; Wang, S.; Chen, T. A study on the wear behavior of hardened medium carbon steel. *J. Mater. Process. Technol.* **2002**, *120*, 126–132. [CrossRef]
18. Krbata, M.; Eckert, M.; Majerik, J.; Barenyi, I. Wear Behaviour of High Strength Tool Steel 90MnCrV8 in Contact with Si3N4. *Metals* **2020**, *10*, 756. [CrossRef]
19. Cora, Ö.N.; Namiki, K.; Koç, M. Wear performance assessment of alternative stamping die materials utilizing a novel test system. *Wear* **2009**, *267*, 1123–1129. [CrossRef]
20. Hardell, J.; Hernandez, S.; Mozgovoy, S.; Pelcastre, L.; Courbon, C.; Prakash, B. Effect of oxide layers and near surface transformations on friction and wear during tool steel and boron steel interaction at high temperatures. *Wear* **2015**, *330*, 223–229. [CrossRef]
21. Jalil, S.; Ghayour, H.; Amini, K.; Gharavi, F. The Effect of Deep Cryogenic Treatment on Microstructure and Wear Behavior of H11 Tool Steel. *Phys. Met. Metallogr.* **2019**, *120*, 888–897. [CrossRef]
22. Kumar, S.; Nagraj, M.; Bongale, A.; Khedkar, N. Deep Cryogenic Treatment of AISI M2 Tool Steel and Optimisation of Its Wear Characteristics Using Taguchi's Approach. *Arab. J. Sci. Eng.* **2018**, *43*, 4917–4929. [CrossRef]
23. Priyadarshini, M.; Behera, A.; Biswas, C.K. Effect of sub-zero temperatures on wear resistance of AISI P20 tool steel. *J. Braz. Soc. Mech. Sci. Eng.* **2020**, *42*, 212. [CrossRef]
24. Collins, D.N. Deep Cryogenic Treatment of Tool Steels: A Review. *Heat Treat. Met.* **1996**, *2*, 8975829.
25. Akhbarizadeh, A.; Shafyei, A.; Golozar, M. Effects of cryogenic treatment on wear behavior of D6 tool steel. *Mater. Des.* **2009**, *30*, 3259–3264. [CrossRef]
26. Amini, K.; Akhbarizadeh, A.; Javadpour, S. Effect of deep cryogenic treatment on the formation of nano-sized carbides and the wear behavior of D2 tool steel. *Int. J. Miner. Metall. Mater.* **2012**, *19*, 795–799. [CrossRef]
27. Deng, L.; Mozgovoy, S.; Hardell, J.; Prakash, B.; Oldenburg, M. Press-hardening thermo-mechanical conditions in the contact between blank and tool. Proceedings of International Conference on Hot Sheet Metal Forming of High-Performance Steel: 09/06/2013-12/06/2013, Luleå, Sweden, 9–12 June 2013; pp. 293–300.
28. Ghiotti, A.; Bruschi, S.; Borsetto, F. Tribological characteristics of high strength steel sheets under hot stamping conditions. *J. Mater. Process. Technol.* **2011**, *211*, 1694–1700. [CrossRef]

29. Das, D.; Dutta, A.K.; Ray, K.K. Influence of varied cryotreatment on the wear behavior of AISI D2 steel. *Wear* **2009**, *266*, 297–309. [CrossRef]
30. Das, D.; Dutta, A.; Toppo, V.; Ray, K. Effect of deep cryogenic treatment on the carbide precipitation and tribological behavior of D2 steel. *Mater. Manuf. Process.* **2007**, *22*, 474–480. [CrossRef]
31. Singh, K.; Khatirkar, R.K.; Sapate, S.G. Microstructure evolution and abrasive wear behavior of D2 steel. *Wear* **2015**, *328–329*, 206–216. [CrossRef]
32. Jovičević-Klug, P.; Sedlaček, M.; Jovičević-Klug, M.; Podgornik, B. Effect of Deep Cryogenic Treatment on Wear and Galling Properties of High-Speed Steels. *Materials* **2021**, *14*, 7561. [CrossRef]
33. Das, D.; Dutta, A.K.; Ray, K.K. Sub-zero treatments of AISI D2 steel: Part I. Microstructure and hardness. *Mater. Sci. Eng. A* **2010**, *527*, 2182–2193. [CrossRef]
34. Das, D.; Dutta, A.; Ray, K. Correlation of microstructure with wear behaviour of deep cryogenically treated AISI D2 steel. *Wear* **2009**, *267*, 1371–1380. [CrossRef]
35. Dhokey, N.; Maske, S.; Ghosh, P. Effect of tempering and cryogenic treatment on wear and mechanical properties of hot work tool steel (H13). *Mater. Today: Proc.* **2021**, *43*, 3006–3013. [CrossRef]
36. Arunram, S.; Nishal, M.; Thirumugham, M.; Raghunath, A. Effect of deep and shallow cryogenic treatment on high speed steel grade M2 drilling tool. *Mater. Today Proc.* **2021**, *46*, 9444–9448. [CrossRef]
37. Padmakumar, M.; Dinakaran, D. A review on cryogenic treatment of tungsten carbide (WC-Co) tool material. *Mater. Manuf. Process.* **2021**, *36*, 637–659. [CrossRef]
38. Singh, G.; Pandey, K. Effect of cryogenic treatment on properties of materials: A review. *Proc. Inst. Mech. Eng. Part E J. Process Mech. Eng.* **2022**, *236*, 1090189. [CrossRef]
39. Rehan, A. *Microstructure and Mechanical Properties of a 5 wt.% Cr Cold Work Tool Steel: Influence of Heat Treatment Procedure*; University West: Ontario, ON, Canada, 2017.
40. García, C.; Romero, A.; Herranz, G.; Blanco, Y.; Martin, F. Effect of vanadium carbide on dry sliding wear behavior of powder metallurgy AISI M2 high speed steel processed by concentrated solar energy. *Mater. Charact.* **2016**, *121*, 175–186. [CrossRef]
41. Günerli, E. Effect of Tempering Temperature on the Mechanical Properties of Hardened 1.2842 Tool Steel. Master's Thesis, Çukurova University Institute of Natural and Applied Sciences, Adana, Turkey, 2010.
42. Chen, W.; Wu, W.; Li, C.; Meng, X. Influence of Deep Cryogenic Treatment and Secondary Tempering on Microstructure and Mechanical Properties of Medium-Carbon Low-Alloy Steels. *J. Mater. Eng. Perform.* **2020**, *29*, 10–22. [CrossRef]
43. Li, H.; Tong, W.; Cui, J.; Zhang, H.; Chen, L.; Zuo, L. The influence of deep cryogenic treatment on the properties of high-vanadium alloy steel. *Mater. Sci. Eng. A* **2016**, *662*, 356–362. [CrossRef]