



Research Article

Investigation of friction performance and surface integrity of cryogenically treated AISI 430 ferritic stainless steel

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ABSTRACT

In this study, the effect of shallow cryogenic treatment on the friction coefficient of AISI 430 ferritic stainless steel was investigated. The friction coefficient experiments were carried out in a ball-on-disc wear tester under 5 N load at 400 rpm. As a result of the tests, the study examined the surface topography of the wear traces, the abrasion profile, microscopic images of the wear traces, and the hardness change of the wear traces. After applying shallow cryogenic treatment, the friction coefficient of the samples was increased by 7.5%. The micro hardness value around the wear traces of the cryogenic (Cryo) samples was 28.4% higher than the value for the commercial samples. The width of the wear trace of the Cryo samples was reduced by 44%. The average roughness value of the wear trace was 33.3% improved in the Cryo sample compared to the commercial sample.

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1. Introduction

Mechanical components used in various fields can be exposed to both mechanical wear and corrosion at the same time [1]. Because of features such as high corrosion and wear resistance as well as their attractive appearance, stainless steels are widely preferred and used in many sectors. Stainless steels generally contain iron, chromium, and nickel and due to the chromium, they exhibit resistance to corrosion [2,3]. Stainless steels, known as corrosion-resistant steels in the aviation industry, appeal to a wide milieu, ranging from the food industry to the chemical industry, and from nuclear applications to the machinery sector [4]. Stainless steel materials can be classified in five different groups according to their chemical and crystal structures: austenitic, ferritic, martensitic, duplex, and precipitation hardening stainless steels. Ferritic stainless steels stand out compared to other stainless steels due to their low cost / high benefit ratio. Because of the high chromium content in ferritic stainless steels, they exhibit very high corrosion resistance, and their iron content renders them magnetic [5]. The most widely known grades of ferritic stainless steels are AISI

430 and AISI 442. In applications where corrosion and wear occur simultaneously, mechanical friction caused by plastic deformation leads to unwanted wear on the material surface [6].

In recent years, cryogenic treatment (-80 °C ~ -196 °C) has been frequently used to improve the wear resistance, toughness, and hardness of the materials [7]. Different gases (nitrogen, helium, oxygen, neon, etc.) are used in cryogenic treatments, which can be classified as deep cryogenic treatment (-140 °C ~ -196 °C) and shallow cryogenic treatment (-80 °C ~ -140 °C) [8]. As a result of studies conducted, the residual austenite in the microstructure of steels is known to transform into martensite under cryogenic treatment. The wear resistance in the steel increases because of this transformation. For example, in his study, Akıncioğlu [3] stated that shallow cryogenic treatment significantly boosted the material wear resistance, hardness, and electrical conductivity of AISI 410 stainless steel. Moreover, cryogenic treatment application is seen in many fields such as space and aviation research, in food storage, and in the transportation sector [9]. Studies performed by applying cryogenic treatment are mentioned below.

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Kızılkaya and Ovalı conducted a series of experiments to determine the mechanical properties of AISI 4140 steel using shallow cryogenic treatment. In this context, they prepared five different samples that were poured and cured, and then cryogenic treatment was applied for 2 h, 4 h, and 6 h after the treatment. First, the notch impact test was performed on the prepared samples, and then abrasive wear tests (pin-on-disk) were applied at three different loads (5 N, 10 N, and 15 N) and at three different sliding distances (95 m, 190 m, and 285 m). At the end of their experimental work, the researchers obtained the lowest wear rate in the sample that was cryogenically treated for 6 h. They achieved the maximum friction coefficient by applying a force of 5 N on the sample that had been cryogenically treated for 6 h [10]. Şirin et al. applied shallow cryogenic treatment to AISI 430 ferritic stainless steel at different holding times and investigated the effect on mechanical properties. Tensile strength tests were applied to four different samples cryogenically treated for 0 h, 6 h, 12 h, and 24 h. They reached the maximum tensile strength (455 N/mm²) with the samples that were treated for 24 h [11]. In his thesis, Yamanoğlu [12] examined the abrasive wear resistance of DIN 1.2379 and DIN 1.2080 cold work tool steels subjected to deep cryogenic treatment at -140 °C. The study included commercial, quenched, quenched + cryogenically treated, and quenched + tempered cold work tool steels. The 12-h deep cryogenic (-140 °C) treatment was the most desirable. According to the test results, he claimed that the wear resistance was dependent on the load and the amount of wear and that the lowest wear loss was found at a 10-N load in the quenched + cryogenically treated samples. Akıncioğlu [3] investigated the effect of shallow cryogenic treatment on the microstructure, hardness, and electrical conductivity of AISI 410 stainless steel. In this context, shallow cryogenic (-80 °C) treatment was applied to AISI 410 stainless steel samples for 24 h, 48 h, 72 h, and 96 h. The researcher claimed that the hardness value of the 96-h sample had increased by 4% and the electrical conductivity by 300% compared to the commercial (untreated) sample. Sert [13] investigated the changes in the microstructure and tribological properties of AISI M2 tool steel subjected to different heat treatment applications. In the experiment, three different samples were used: quenched + tempered, quenched + deep cryogenically treated + tempered, and quenched + tempered + deep cryogenically treated + tempered. It was stated that the carbide grain size and distribution in the microstructure were better in the deep

cryogenically treated samples. According to the tribology test results, it was stated that the wear rate and friction coefficients of the cryogenically treated samples were lower than for the other samples.

A review of the literature yielded a number of studies investigating the mechanical and tribological characteristics of various materials following cryogenic treatment. These studies concluded that the microstructure, hardness, friction coefficient, etc. were improved in shallow or deep cryogenically treated material [14]. However, to date, no study had been carried out to examine the total effects of shallow cryogenic treatment on AISI 430 ferritic stainless steel in terms of the friction coefficient. In addition, the surface quality and topography of the wear traces on this steel had not been adequately studied. Accordingly, this study investigated the friction coefficient, surface quality, and wear trace topography of shallow cryogenically treated AISI 430 ferritic stainless steel.

2. Material and Methods

In the experiments, 40 × 60 × 4-mm samples of AISI 430 ferritic stainless steel (EN 1.4016, Nom. X6Cr17) were used. Tables 1 and 2, respectively, give the chemical composition of the test samples and the mechanical properties of AISI 430.

Pre-experiment samples were prepared as untreated (Commercial) and cryogenically treated for 24 h (Cryo). The shallow cryogenic treatment was applied to the Cryo samples in the cryogenic treatment unit (Figure 1-a) at a 24-h holding time. During the treatment, in order to prevent unwanted micro cracks in the AISI 430 ferritic stainless steel, the samples were gradually brought to -80 °C and at the end of the treatment, gradually returned to room temperature. The Turkyus ball-on-disc tribometer (Figure 1-b) was used to determine the friction coefficient. For the experiments, Ø6-mm DIN 100Cr6 balls were selected and a new ball was used in each experiment.

The tests were performed on the ball-on-disk friction tester according to the ASTM G99-05 standard, at room temperature (23 °C), a constant 20-mm trace diameter, under 5 N load and 6 Hz sampling frequency, and at a speed of 418.9 m/min.

Table 1. Chemical composition of AISI 430 ferritic stainless steel test samples [11]

C	Si	Mn	Cr	Ni	Mo	Al	Balance
0.046	0.25	0.44	17.1	0.15	0.18	0.01	Fe

Table 2. Mechanical properties of AISI 430 ferritic stainless steel at room temperature [15]

Density	Elastic Module	Tensile Strength	Yield Strength	Elongation	Hardness
(g/cm ³)	(GPa)	(MPa)	(MPa)	(%)	(HV)
7.8	200	459	373	22	162

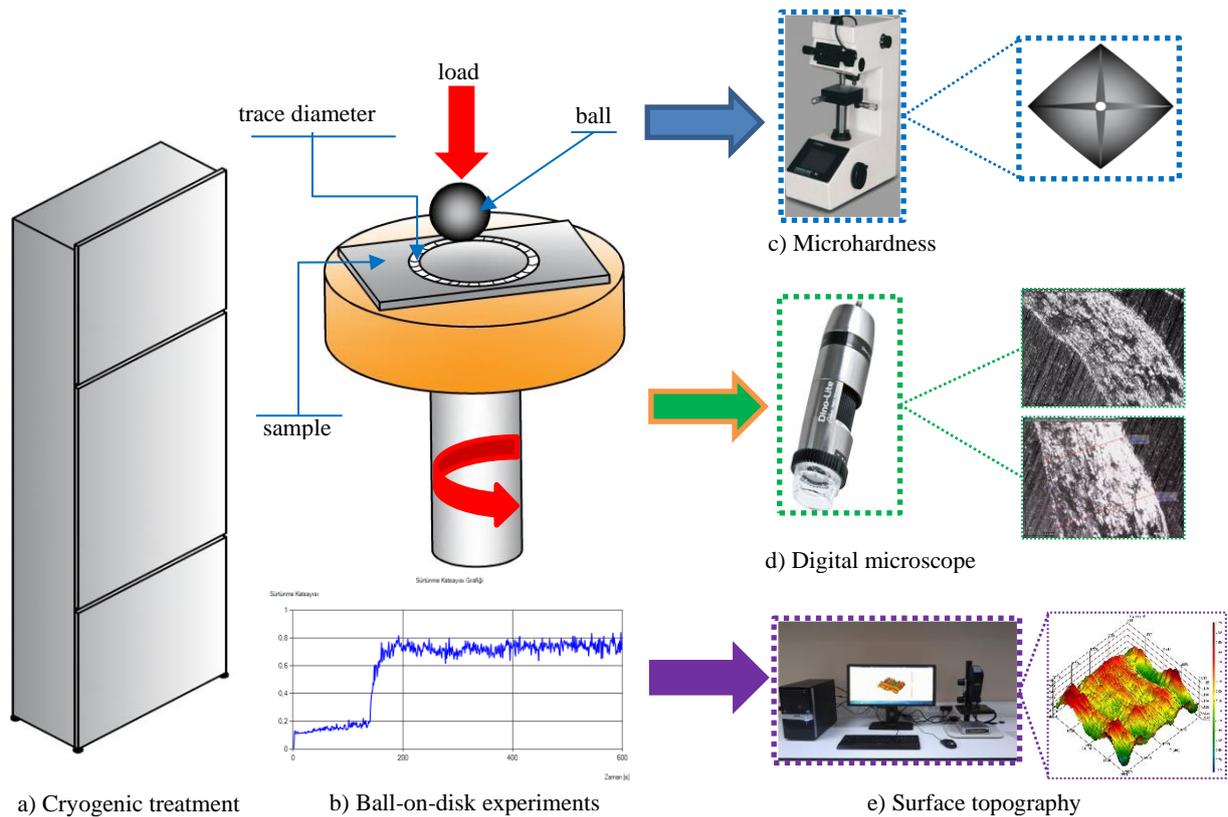


Figure 1. Experimental setup: a) Cryogenic treatment, b) Ball-on-disc experiments, c) Microhardness measurement, d) Wear trace width measurement, e) Surface topography measurement

Before and after the test, the samples were cleaned with an ethyl alcohol solution and dried.

After the friction tests, 10 microhardness measurements were taken at distances starting at $50 \mu\text{m}$ from the wear trace and moving towards the outer edge of the material. Microhardness measurements, at a test load determined as 100 g and a dwell-time of 19 s, were carried out using the Metkon DUROLINE M microhardness tester (Figure 1-c). Before the hardness measurements, the device was calibrated with a measuring block gauge and the average of measurements from three different points was used. The Dino-Lite AM7915MZT digital microscope (Figure 1-d) was used for the analysis of wear traces and determination of wear trace widths. Before carrying out the measurements, accuracy was ensured by calibrating the instrument with a digital microscope calibration gauge. The Phase View surface topography device (Figure 1-e) was used for the three-dimensional examination of the deterioration in the wear traces. The same wear trace sections were examined in all samples. The experimental setup and equipment used in the experiments are given in Figure 1.

3. Results and Discussion

3.1. Evaluation of average friction coefficient

The friction coefficient test was carried out under a constant load of 5 N at a speed of 400 rpm. The results obtained depending on the test period are given in Figure

2. The figure shows that the average friction coefficient varied depending on the time. A higher coefficient of friction was obtained in the Cryo samples compared to the non-treated Commercial samples.

It is thought that the friction coefficients increased because of the increased toughness and hardness of the cryogenically treated samples [15]. Especially after 100 s, the peak trend for the friction coefficient was observed in both test samples. The friction coefficient of the Commercial samples was determined as 0.587 after the ball-on-disc friction coefficient experiments. The Cryo sample friction coefficient increased by 18.25% compared to Commercial samples. Meng et al. applied cryogenic treatment to Fe-12Cr-Mo-V-1,4C tool steel at -50 and -180 °C [16]. It was concluded that the most important factor contributing to the increase in wear resistance was the reduction of residual austenite and the formation of a homogeneous martensitic structure resulting from the cryogenic treatment. Similar findings were obtained in our study. The increase in the friction coefficient in the Cryo samples can be explained by the transformation of residual austenite to martensite.

Plastic deformation of metals occurs when dislocations progress through the crystalline structure. Dislocations are defined as linear defects within the microstructure. The strength, hardness, and mechanical properties of metals are explained by the density of dislocations in the microstructure and the interaction of dislocations with

other defects. Tyshchenko et al. [17] reported that with cryogenic treatment, plastic deformation occurred during martensitic transformation in tool steel. They found that the plastic deformation caused the carbide particles to partially dissolve. This plastic deformation captured stationary carbon atoms by moving dislocations, providing a suitable environment for the nucleation of new η -carbide particles. Deformation hardening occurs as a result of the interaction of dislocations with each other and various obstacles that make the movement of the dislocations difficult [18]. Any factor that makes the movement of dislocations in the microstructure of metals difficult will lead to an increase in the strength of the material. It can be said that the friction coefficient of the Cryo samples was increased due to the deformation hardening caused by the cryogenic treatment.

3.2. Evaluation of the microhardness

In order to determine the change in micro hardness, measurements were taken at distances around the wear traces formed as a result of the wear tests. The changes in micro hardness depending on the measured distances are given in Figure 3. Hardness values were measured higher close to the wear trace at a distance of 50- μm . Moving away from the wear trace, the hardness values decreased,

and the lowest hardness values were reached at a distance of 500 μm , as the farthest point from the wear trace. It can be said that this hardness increase resulted from the wear trace and the material trapped around it due to the wear load.

Hardness values of the Commercial and Cryo samples at a distance of 50 μm from the wear trace were measured as 321.8 HV and 230.1 HV, respectively. The hardness increase around the wear trace of the Cryo sample was 28.4%. The hardness change rate of the Cryo sample between 50 μm and 500 μm (181.4 HV) was 43.6%. Plastic deformation applied to metals in the cold deformation zone leads to deformation hardening. Cold deformation occurs at temperatures lower than half the melting temperature of the metals (usually at room temperature). The deformation process in this temperature range causes strain hardening in the metallic structure. Kalsi et al. [19] investigated the effect of cryogenic treatment and stated that it increased the hardness of many steels. Yang et al. [20] found that the hardness of 13Cr2Mn2 V high chromium white iron samples was increased by deep cryogenic treatment. The increased hardness was attributed to the transformation of residual austenite into martensite and to the increased secondary carbides precipitated in the matrix.

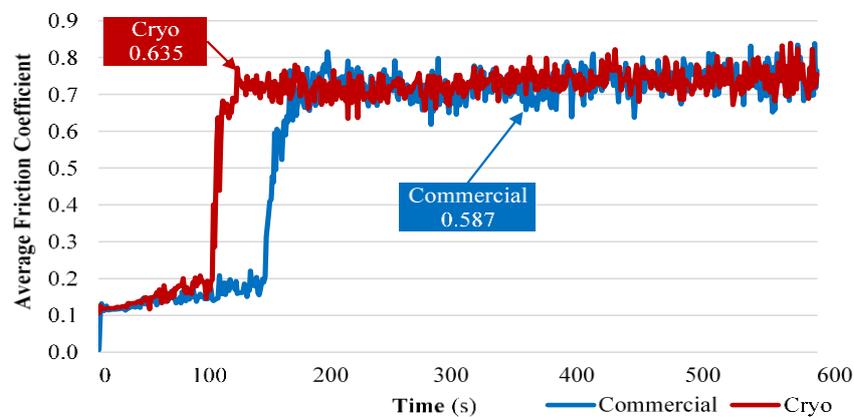


Figure 2. Change in friction coefficient depending on time

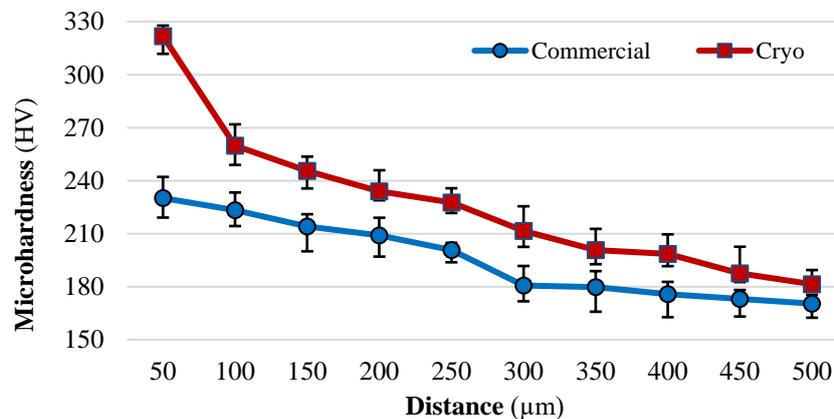


Figure 3. Change in micro hardness depending on measuring distance

Akhbarizadeh et al. [21] attributed the increase in the hardness of D6 steel to the homogeneous carbide distribution and high chromium carbon concentration as a result of deep cryogenic treatment. Benseley et al. [22] found that the hardness of En353 steel increased 3.48% after deep cryogenic treatment. They explained that the increase in hardness was caused by the residual austenite transformed into martensite. They concluded that cryogenic treatment applied to steels generally affects the hardness positively. The increase in hardness was largely explained by the conversion of residual austenite into martensite, and the precipitation of carbides that were homogeneously distributed throughout the microstructure.

In this study, the hardness increase occurring as a result of shallow cryogenic treatments was realized by the more regular microstructure formed in the AISI 430 ferritic stainless steel.

The width of the wear traces of the samples can be seen in Figure 4, which shows that the wear traces in the Cryo samples are narrow. The wear trace width of the Cryo samples decreased by 44% compared to the Commercial samples. This can be attributed to the change in the microstructure and the increase in hardness of the samples after shallow cryogenic treatment. This can be explained by the cold hardening that occurred when rolling pressure was applied to the samples under friction. Akhbarizadeh et al. found that the increase of hardness in D6 steel Cryo samples was caused by the formation of a homogeneous microstructure and the decrease of austenite [21]. With cryogenic treatment at low temperatures, the austenite and martensite lattice structure shrinks. This constriction forces the carbon atoms to dissolve. New carbides are produced at higher temperatures, leading to a more homogeneous distribution of carbides [16, 23].

3.3. Evaluation of worn surface characteristics

Figure 5 shows microscope images of the wear traces obtained after the ball-on-disc wear tests of the AISI 430

ferritic stainless steel. Deflections and pits can be seen in these images. Deflections are more pronounced in the Commercial sample. The traces are slighter and more homogeneous in the Cryo samples. This can be attributed to the higher friction coefficient and hardness values of the shallow cryogenically treated samples. Meng et al. [16] explained that the rate of wear can be controlled by controlling the crack nucleation under the surface, which is related to the strength and toughness of the materials. Residual austenite can prevent crack propagation by changing the growth direction of a progressive crack or by largely absorbing the energy. Cryogenic treatment is known to promote corrosion resistance because of the precipitation of fine η -carbides rather than because the retained austenite was removed.

The surface topography of the wear traces formed after wear tests of the AISI 430 ferritic stainless steel was examined. The surface topography images of the Commercial sample (Figure 6-a) show that the trace profile has a more pitted surface. This situation coincides with the formation of debris observed in the microscopic images of the traces (Figure 5). The low hardness values of the Commercial samples caused more wear. In addition, their softer structure compared to the Cryo samples caused the formation of deflections and pits on the worn surfaces. The trace profile was relatively smoother in the Cryo samples. Shallower wear traces can be seen in the Cryo samples compared to the Commercial samples [21]. The R_a values found for the wear traces of the Commercial and Cryo samples were measured as $0.4012 \mu\text{m}$ and $0.2678 \mu\text{m}$, respectively. The wear trace average roughness value of the Cryo sample was 33.3% improved over the Commercial sample. This result can be attributed to the harder, homogeneous microstructure of the samples because of the shallow cryogenic treatment.

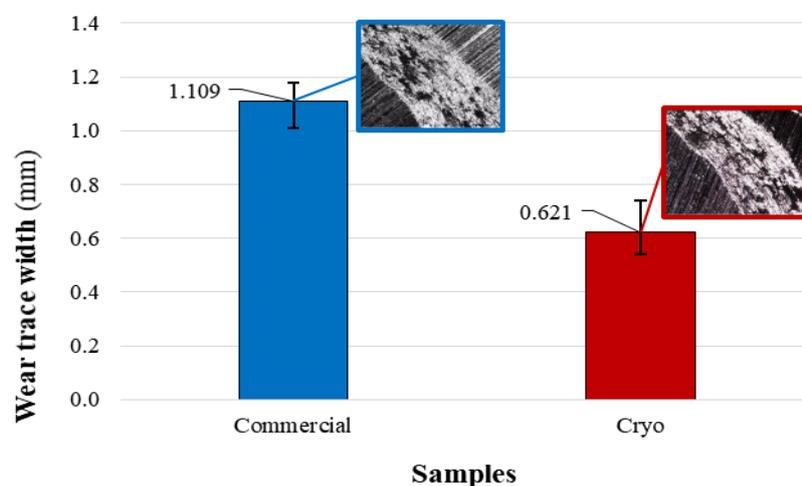


Figure 4. Change in wear trace width after the experiment

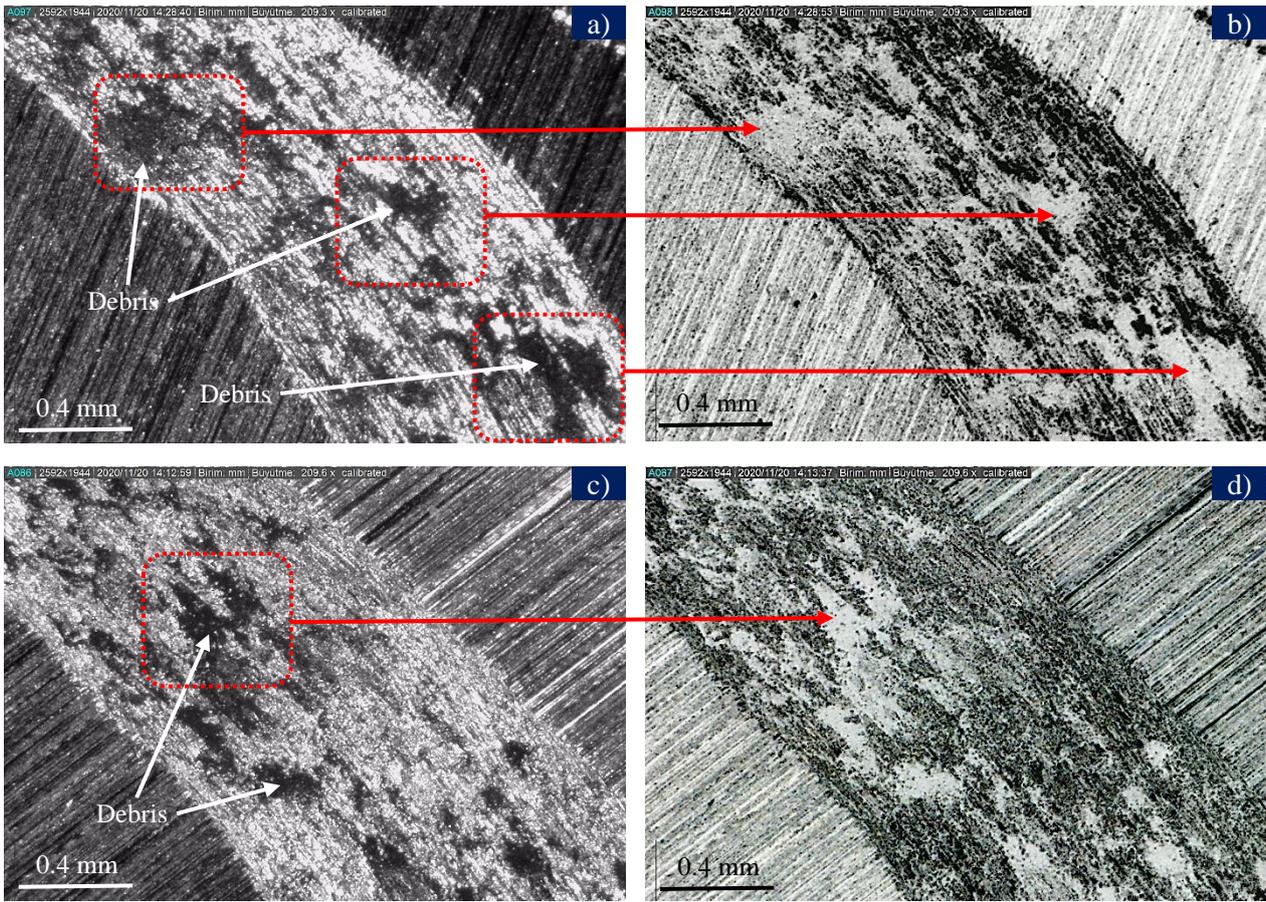


Figure 5. AISI 430 ferritic stainless steel surface photos after wear tests: a) Commercial, b) Commercial (negative photo), c) Cryo, d) Cryo (negative photo)

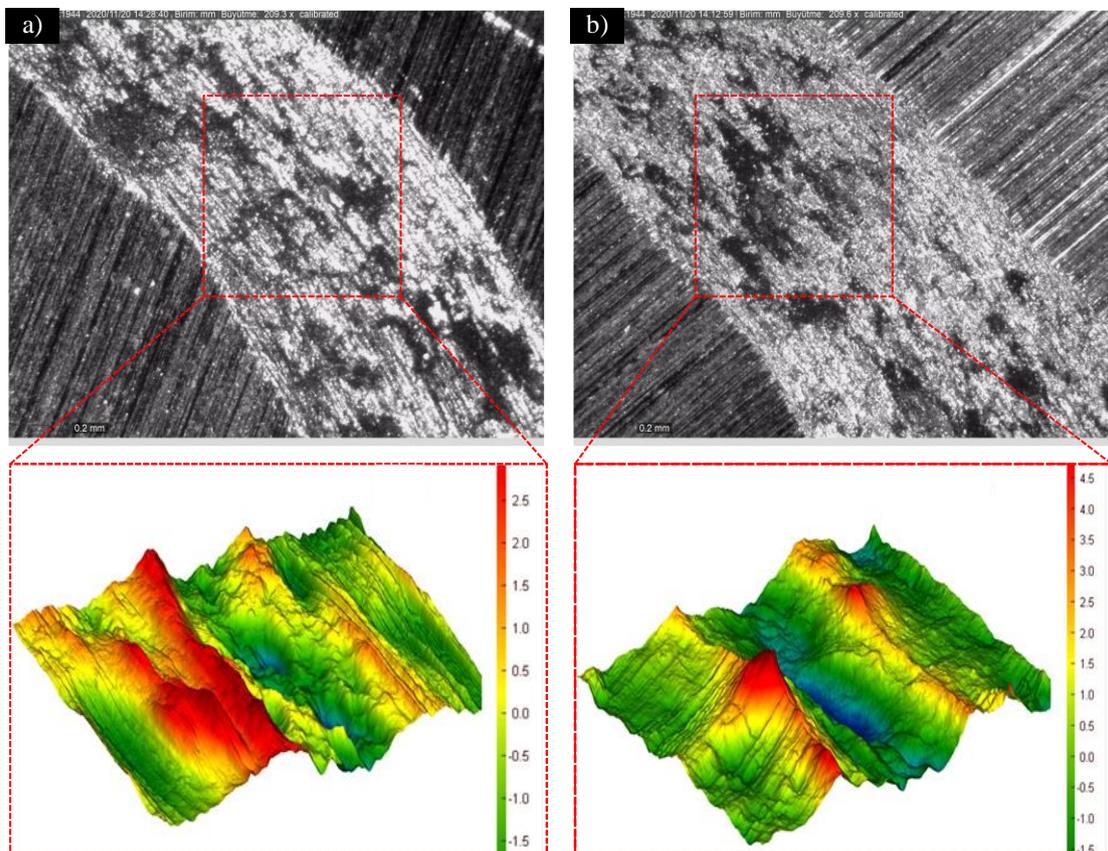


Figure 6. Wear trace topography: a) Commercial sample, b) Cryo sample

4. Conclusions

The study investigated the effects of the shallow cryogenic treatment on the friction coefficient, microhardness, and surface wear of the AISI 430 ferritic stainless steel and the following results were obtained.

- A higher friction coefficient was obtained in the Cryo samples than in the Commercial samples. The friction coefficients were determined as 0.635 and 0.587 in the Cryo and Commercial samples, respectively.
- The *Ra* values of the Commercial and Cryo samples were measured as 0.4012 μm and 0.2678 μm , respectively. The wear trace roughness of the Cryo sample was improved by 33.3% compared to the commercial sample.
- Wear traces were more pronounced in the Commercial samples. The traces were more homogeneous and there were fewer depressions in the Cryo samples.
- The shallow cryogenic treatment reduced the wear trace width on the AISI 403 stainless steel. The wear trace width of the shallow cryogenically treated Cryo samples was reduced by 44% compared to the Commercial samples.
- Hardness values decreased as the distance away from the wear trace increased. The hardness increase around the Cryo sample wear traces was 28.4% higher than for the Commercial samples.
- It was concluded that a positive contribution had been made by the shallow cryogenic treatment in terms of the mechanical properties of the AISI 430 ferritic stainless steel.
- In further studies on AISI 430 ferritic stainless steel, the effect of shallow cryogenic treatment applied at different temperatures and different waiting times can be investigated.

No in-depth studies had yet been conducted on the friction performance of shallow cryogenically treated AISI 430 ferritic stainless steel, as investigated in this study. Additionally the aim of this study was to provide a contribution to the literature in the form of a comprehensive study of the surface integrity of AISI 430 ferritic stainless steel after wear testing.

Declaration

The authors declare that they have no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The authors also declare that this article is original and was prepared in accordance with international publication and research ethics, and that no ethical committee or special permission was required.

Author Contributions

Ş. Şirin: Conceptualization, Investigation, Resources, Methodology, Writing - editing. S. Akıncıoğlu: Writing -

review, Validation, Software, Data curation, Supervision.

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