JOURNAL of
MATERIALS and MECHATRONICS:A

e-ISSN 2717-8811
JournalMM, 2022, 3(2), 151-162
https://doi.org/10.55546/jmm.1113194

Araştırma Makalesi / Research Article

Investigation of The Effects of Shallow Cryogenic Treatment on The Mechanical and Microstructural Properties of 1.2436 Tool Steel

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Geliş/ Received: 06.05.2022; **Kabul / Accepted:** 08.07.2022

ABSTRACT: This study investigates the effects of shallow cryogenic treatment on the microstructure, hardness and wear resistance of 1.2436 steel. For this purpose, quenched (QT) 1.2436 steel samples were subjected to shallow cryogenic treatment at -80 °C for 12 hours (SCT12) and 18 hours (SCT18). Hardness measurement and wear test were carried out on the samples and the samples were examined microstructurally. As a result of the study, it was observed that the cryogenic treatment provided a denser and homogeneous carbide distribution in the microstructure of 1.2436 steel. The amount of carbide in the microstructure increased by 18.80% with shallow cryogenic treatment for 18 hours. As a result of the hardness and wear tests, it was determined that the cryogenic treatment positively affected the hardness and wear resistance of 1.2436 steel. Compared to the quenched sample alone, the sample cryogenically treated for 18 hours exhibited 9.28% higher hardness and 34.37% less wear.

Keywords: 1.2436 Steel, Cryogenic Treatment, Microstructure, Hardness, Wear Resistance.

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Bu makaleye atıf yapmak için /To cite this article

Altan Özbek, N., Özbek, O. (2022). Investigation of The Effects of Shallow Cryogenic Treatment on The Mechanical and Microstructural Properties of 1.2436 Tool Steel. Journal of Materials and Mechatronics: A (JournalMM), 3(2), 151-162.

1.2436 Takım Çeliğinin Mekanik ve Mikroyapısal Özellikleri Üzerine Sığ Kriyojenik İşlemin Etkilerinin Araştırılması

ÖZET: Bu çalışma, sığ kriyojenik işlemin 1.2436 çeliğin mikroyapısı, sertliği ve aşınma direnci üzerindeki etkilerini araştırmaktadır. Bu amaçla, su verilmiş (QT) 1.2436 çelik numunelere -80 °C'de 12 saat (SCT12) ve 18 saat (SCT18) için sığ kriyojenik işlem uygulanmıştır. Numuneler üzerinde sertlik ölçümü ve aşınma testi gerçekleştirilmiş ve numuneler mikroyapısal olarak incelenmiştir. Çalışma sonucunda kriyojenik işlemin 1.2436 çeliğin mikro yapısında daha yoğun ve homojen bir karbür dağılımı sağladığı gözlemlenmiştir. 18 saat süre sığ kriyojenik işlem ile mikroyapıdaki karbür miktarı yaklaşık %18,80 oranda artmıştır. Sertlik ve aşınma testleri sonucunda kriyojenik işlemin 1.2436 çeliğin sertliğini ve aşınma direncini olumlu yönde etkilediği tespit edilmiştir. Yalnızca su verilmiş numune ile karşılaştırıldığında, 18 saat boyunca kriyojenik işlem görmüş numune %9.64 daha yüksek sertlik ve %34.37 daha az aşınma sergilemiştir.

Anahtar Kelimeler: 1.2436 Çeliği, Kriyojenik İşlem, Mikroyapı, Sertlik, Aşınma Direnci.

1. INTRODUCTION

Heat treatment is the controlled heating and cooling process applied to give the steel the desired properties. Heat treatments applied to steel are related to microstructural transformation. The composition of the steel and the change in its structures directly affect the mechanical properties of the steel. Many thermal methods have been developed for microstructural transformation. These methods depend on the type of steel and the expected properties (such as hardness, strength, toughness, and wear resistance) (Callister and Rethwisch, 2011; Rajan et al., 2011; Altan Özbek and Saraç, 2021; Talaş et al., 2020). The word cryogen, which means cold science, comes from the Greek "kryos" meaning cold. Cold science is a simple materials science that significantly changes the properties of materials at low temperatures (Kalia, 2010). Cryogenic treatments are complementary to conventional heat treatment, which has recently been used to improve metals' mechanical and microstructural properties. In steels, a residual austenite phase is formed in the material after traditional heat treatment. This phase is soft and negatively affects the material's mechanical properties. Cryogenic treatment is an effective method for removing the residual austenite phase. The subzero process includes gradually cooling the material to a specified temperature below zero (between -50 and -196 °C), keeping it at this temperature for a specified time, and then gradually reheating it to room temperature. It is known that the cryogenic process provides high hardness and wear resistance in the material, thanks to secondary carbide precipitation and conversion of residual austenite to martensite (Kara et al, 2020; Özbek, 2020; Kara et al., 2021; Zhirafar, 2007; Nas and Altan Özbek, 2020; Kayalı, 2016; Özbek et al., 2014; Özbek, 2021; Özbek et al., 2016)

Yıldız and Altan Özbek (Yıldız and Altan Özbek, 2022) investigated the effects of cryogenic treatment on the mechanical properties of AISI 431 martensitic stainless steel. Their study reported that the cryogenic treatment increased approximately 3.89% and 62% in the hardness and wear resistance of martensitic stainless-steel samples. In the literature, Altan Özbek et al. (Altan Özbek et al., 2018) applied cryogenic treatment on AISI H11 steel at -80 °C for 6 hours. The cryogenic treatment significantly reduced the residual austenite phase and provided new carbide precipitation. However, it has been determined that the cryogenic process provides a slight increase in the hardness of AISI H11 tool steel but also provides a significant increase in wear resistance. Koneshlou et al. (Koneshlou et al, 2011) reported that the cryogenic treatment applied on AISI H13 tool steel at -72

°C for 8 hours decreased the residual austenite phase and increased its mechanical properties (tensile strength, hardness, impact energy and wear resistance). Bensely et al. (Bensely et al., 2009) found that the cryogenic treatment applied on En353 steel at -80 °C for 5 hours significantly reduced residual austenite and increased the fatigue life of the steel by up to 71%.

In their study, Das et al. (Das et al., 2009) aimed to determine the cryogenic treatment holding time that will provide maximum wear resistance to AISI D2 cold work tool steel. Deep cryogenic treatment was applied on the steel samples at different times as 1, 12, 36, 60, 84, 132 hours. As a result of the wear tests, it was reported that the highest wear resistance was obtained with the cryogenic process applied for 36 hours. Altan Özbek et al. (Özbek et al., 2014) investigated the effects of deep cryogenic treatment on tungsten carbide tool at different holding times on hardness and wear resistance. The study applied deep cryogenic procedures in five different periods, 12, 24, 36, 48 and 60 hours. Maximum hardness and wear resistance were obtained after cryogenic treatment applied for 24 hours. Gu et al. (Gu et al., 2014) investigated the effects of deep cryogenic treatment applied at different holding times on the hardness and wear resistance of Ti-6Al-4V alloy. The study applied deep cryogenic procedures in three periods, 3, 48 and 72 hours. It was determined that the maximum hardness and wear resistance were obtained after the cryogenic treatment applied for 72 hours.

1.2436 tool steel is a cold work tool steel with a ledeburitic structure with 12% chromium. This steel is widely used in the powder metallurgy industry to manufacture powder-forming dies. It is used as a die and punches in cutting, drilling and cold forming processes (Mohamad et al., 2018). In the literature, there are mostly studies on deep cryogenic processing applications. There is very little work done on the shallow cryogenic process. At this point, this study, which was carried out by applying the shallow cryogenic treatment on 1.2436 steel, which has a wide usage area, will have an important place in the literature. This study applied shallow cryogenic treatment on 1.2436 steel samples for 12 and 18 hours. The effects of cryogenic treatment on the hardness and wear resistance of 1.2436 steel were investigated. In addition, all samples were also examined microstructurally.

2. MATERIALS AND METHODS

The chemical composition of 1.2436 steel is shown in Table 1. 1.2436 steel samples were subjected to quenching after kept at 920 °C for 25 minutes. After quenching was applied to the steel samples, cryogenic treatment was applied at -80 °C for 12 and 18 hours. Steel samples were subjected to tempering heat treatment at 300 °C for 2 hours after cryogenic treatment. Heat treatments applied to steel samples are given in Figure 1, and details of heat treatment sequences are given in Table 2.

Table 1. Chemical composition of 1.2436 steel

Element	C	Cr	V	Mo	Mn	Si	W
%	2.28	11.58	0.14	0.24	0.4	0.25	0.59

For microstructural analysis, samples were etched using Murakami solution. Microstructure images were taken using a Quanta FEG 250 model scanning electron microscope. Experimental parameters were determined by examining the studies in the literature for micro hardness measurement (Vickers hardness) and wear test, and the parameters were decided after preliminary measurements/experiments. Microhardness measurements were carried out by applying 500 grams of load in 20 seconds. The hardness values of the samples were determined by taking the average of five different measurements. The wear tests were performed on a computer-controlled TRD Wear pin disk device (Figure 2). AISI M2 steel is used as the abrasive disc. The wear tests were carried out under a load of 30 N, with a speed of 0.75 m/s and a sliding distance of 900 m. The load was given in one direction, and no lubricant was used. After the wear tests, the amount of wear on the samples was determined with an electronic balance with a precision of ± 0.0001 g. Before the samples were weighed, no action was taken. The wear rates (Ws) were calculated using Equation 1.

$$W_s = \frac{\Delta m}{Lx\rho xF} \tag{1}$$

With Δm: Mass loss (kg), F: Normal load (N), ρ: Density (kg·m-3) and L: Sliding distance (m).

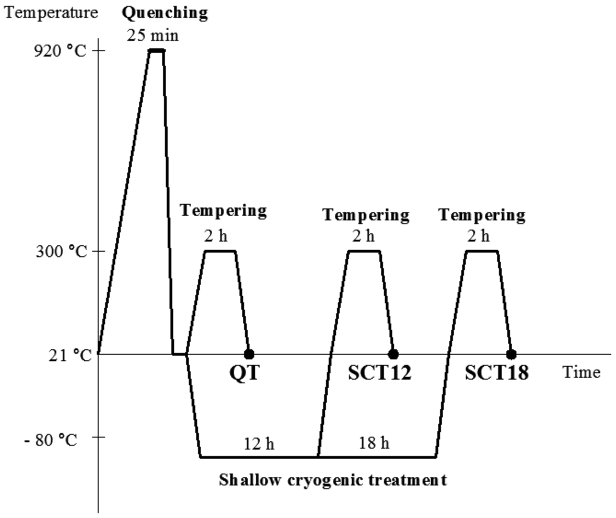


Figure 1. Heat treatments applied on 1.2436 steel.

Table 2. Details of heat treatment sequences.

Sample	Details of heat treatments		
QT	Quenching (at 920 °C for 25 min)	-	Tempering at 300 °C for 120 min
SCT12	Quenching (at 920 °C for 25 min)	Shallow treatment at -80 °C for 12 h	Tempering at 300 °C for 120 min
SCT18	Quenching (at 920 °C for 25 min)	Shallow treatment at -80 °C for 18 h	Tempering at 300 °C for 120 min

Figure 2. TRD wear tester.

3. RESULTS AND DISCUSSION

Figure 3 shows the XRD analysis results of QT and SCT18 steel samples. In Figure 3, it is seen that, in general, phase peaks are at the same 2Θ degrees as a result of both heat treatments. In XRD analysis, it is seen that M_3C, M_7C_3 carbides and Austenite phases can be found in the microstructure of steel samples. Figure 4 shows the carbide distribution of SEM photographs of steel samples using the image processing program. Although there are large and small carbides in all samples, it is seen that large carbides are much more concentrated in certain regions in the microstructure of the QT sample. Compared to the non-cryogenically treated sample, the cryogenically treated samples have a higher carbide content and appear to have a more uniform distribution. As a result of the image processing analysis, the carbide density of the QT, SCT12 and SCT24 samples was found to be 21.64%, 25.46% and 25.71%, respectively. From this, it can be said that the carbide content of SCT12 and SCT18 samples increased by approximately 17.65% and 18.80% compared to the QT sample. In the literature, Kurşuncu et al. (Kursuncu et al., 2018) reported that eta carbide increased by approximately 18% in carbide cutting tools after cryogenic processing using the image processing method. Kurşuncu (Kursuncu, 2020), in another study, determined that the cryogenic process greatly increased the amount of eta carbide in carbide cutting tools.

Figure 5-7 shows the microstructure photographs of 1.2436 steel taken by SEM. The carbide grains (dark colour) in all samples are clearly visible in the microstructure photographs. In the microstructure photographs, the larger carbides are M_7C_3, and the smaller ones are M_3C carbides. EDS analyses confirmed this. M_3C (Point A) is an iron-rich carbide. M_7C_3 (Point B) is a chromium-rich carbide (Vander, 2004). EDS analysis of the smaller carbides in the microstructure photographs confirmed that there are M_3C carbides with more Fe than Cr and some W and Mo. EDS analysis from the larger carbides also confirmed the M_7C_3 carbide with more Cr than Fe.

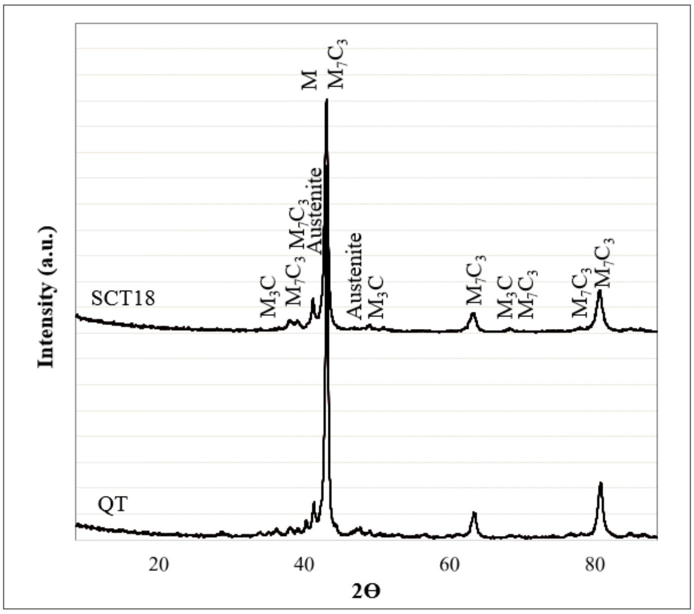


Figure 3. XRD analysis of QT and SCT18 heat-treated 1.2436 steel samples

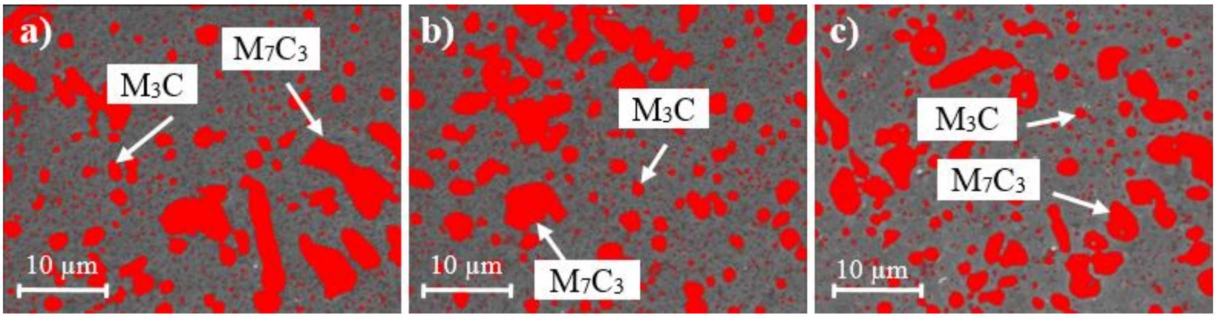
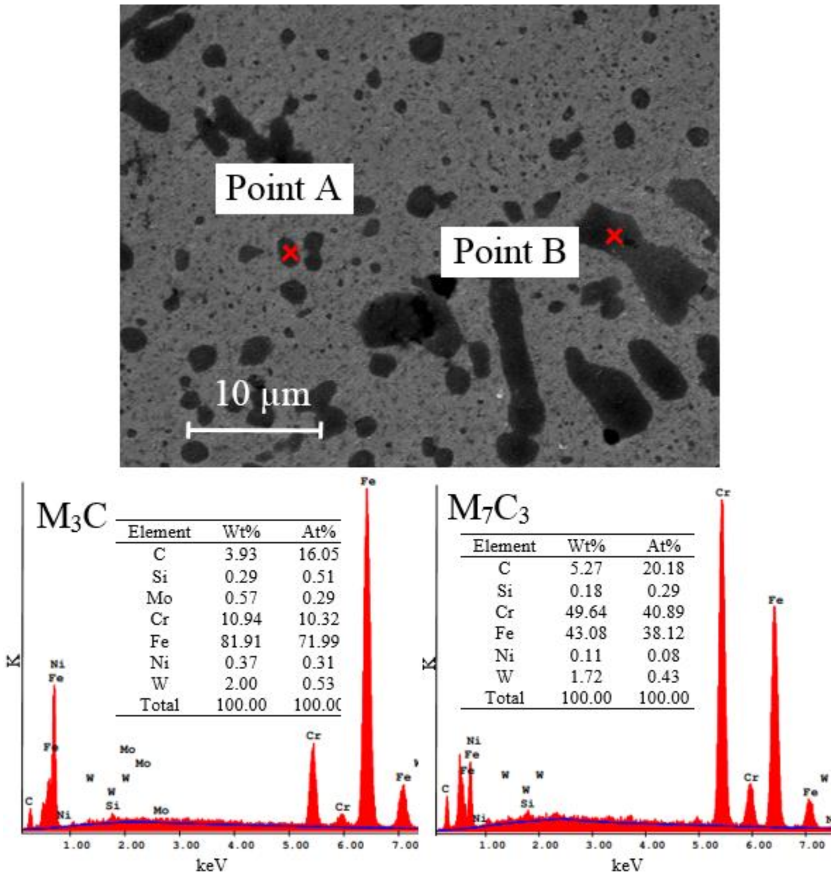


Figure 4. Image processing photographs of different heat-treated 1.2436 steel samples a) QT, b) SCT12, and c) SCT18



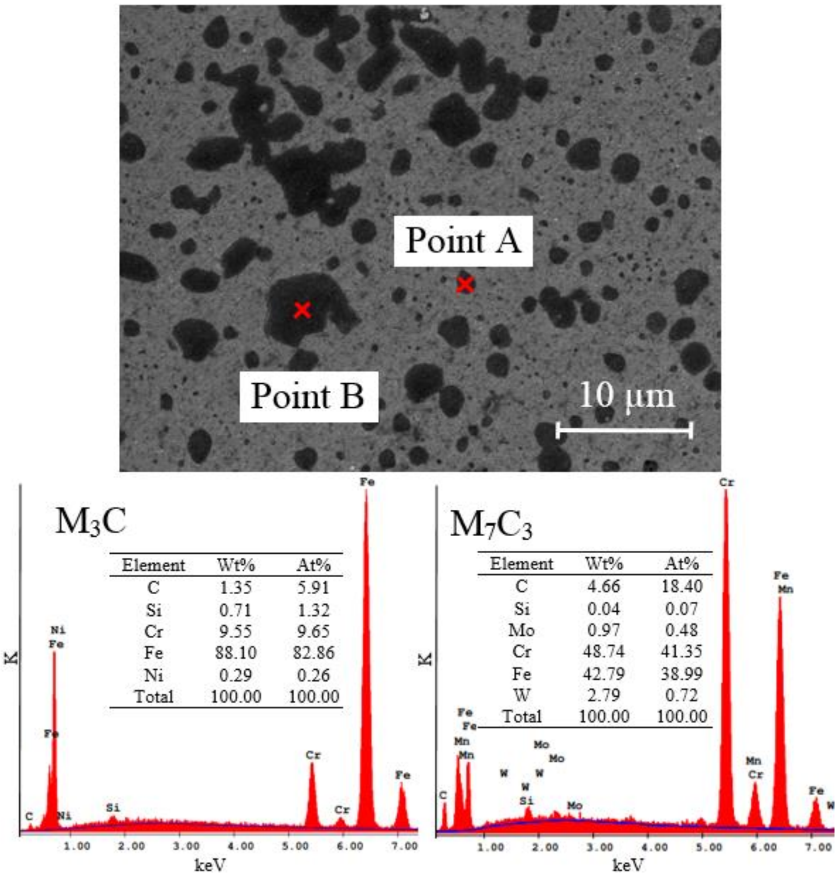
M3C

Element	Wt%	At%
C	3.93	16.05
Si	0.29	0.51
Mo	0.57	0.29
Cr	10.94	10.32
Fe	81.91	71.99
Ni	0.37	0.31
W	2.00	0.53
Total	100.00	100.0

M7C3

Element	Wt%	At%
C	5.27	20.18
Si	0.18	0.29
Cr	49.64	40.89
Fe	43.08	38.12
Ni	0.11	0.08
W	1.72	0.43
Total	100.00	100.00

Figure 5. Microstructure photograph of QT heat-treated 1.2436 steel sample taken by SEM and EDS analysis



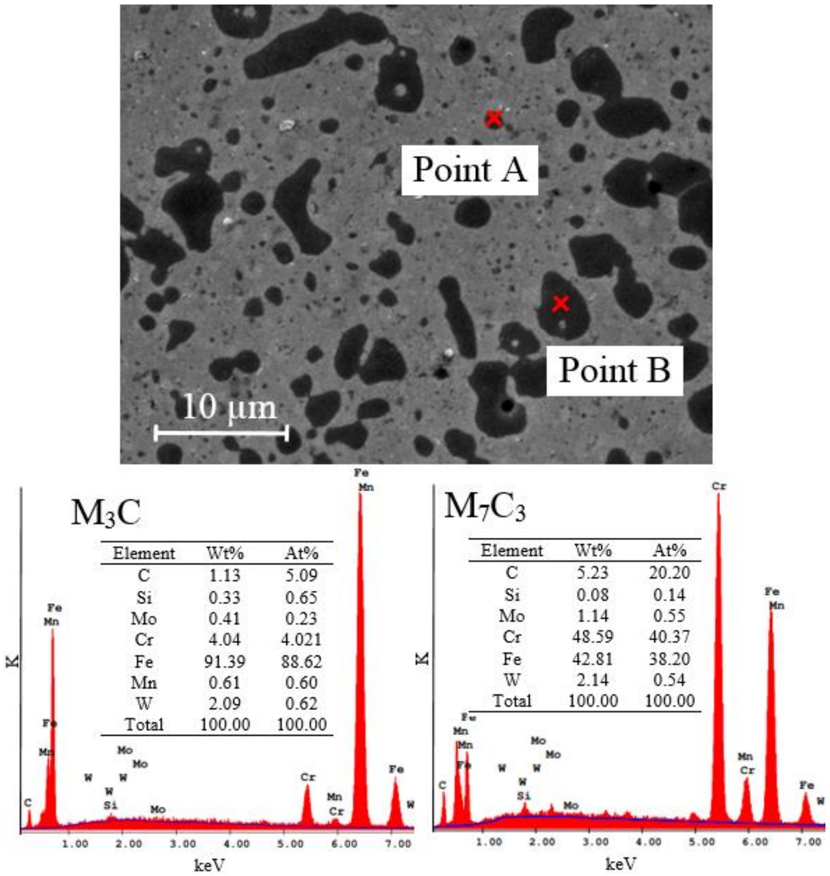
M3C

Element	Wt%	At%
C	1.35	5.91
Si	0.71	1.32
Cr	9.55	9.65
Fe	88.10	82.86
Ni	0.29	0.26
Total	100.00	100.00

M7C3

Element	Wt%	At%
C	4.66	18.40
Si	0.04	0.07
Mo	0.97	0.48
Cr	48.74	41.35
Fe	42.79	38.99
W	2.79	0.72
Total	100.00	100.00

Figure 6. Microstructure photograph of SCT12 heat-treated 1.2436 steel sample taken by SEM and EDS analysis



M3C

Element	Wt%	At%
C	1.13	5.09
Si	0.33	0.65
Mo	0.41	0.23
Cr	4.04	4.021
Fe	91.39	88.62
Mn	0.61	0.60
W	2.09	0.62
Total	100.00	100.00

M7C3

Element	Wt%	At%
C	5.23	20.20
Si	0.08	0.14
Mo	1.14	0.55
Cr	48.59	40.37
Fe	42.81	38.20
W	2.14	0.54
Total	100.00	100.00

Figure 7. Microstructure photograph of SCT18 heat-treated 1.2436 steel sample taken by SEM and EDS analysis

Figure 8 shows the micro hardness of the steel samples. When the graph is examined, the first thing to notice is that subzero treatment increases the hardness of 1.2436 steel. It was observed that higher hardness occurred at a higher subzero treatment holding time. Compared to the QT sample, the hardness of the SCT12 and SCT18 samples is 6.27% and 9.28% higher. This increase in the hardness of 1.2436 steel is thought to be due to the more homogeneous carbide distribution, new carbide precipitation, and conversion of residual austenite to martensite by subzero treatment. Koneshlou et al. (Koneshlou et al., 2011) reported that the shallow cryogenic treatment applied on AISI H13 steel for 8 hours increased the hardness of the steel by approximately 4.08%. Akhbarizadeh et al. (Akhbarizadeh et al, 2009) found in their study that shallow cryogenic treatment increased the hardness of AIDI D6 steel by up to 3.54%. Altan Özbek (Özbek, 2020) found that after 6 hours of shallow cryogenic treatment, the hardness of the tungsten carbide cutting tool increased by approximately 4.4% compared to the untreated cutting tool.

When Figure 9 is examined, it is seen that the cryogenic treatment greatly increases the wear resistance. In the tests carried out under the same conditions, the cryogenically treated samples were less worn than the QT sample. The highest wear resistance was obtained in the SCT18 sample, which was worn 34.37% rate less than the QT sample. Similarly, the SCT12 sample has 25% rate less worn than the QT sample. Cryogenic treatment increased the hardness and, therefore the wear resistance of the steel samples, thanks to the changes in the microstructure. Akhbarizadeh et al. (Akhbarizadeh et al, 2009) reported that shallow cryogenic treatment provides up to 3.54% increase in wear resistance of AIDI D6 steel. Altan Özbek et al. (Altan Özbek et al., 2018) and Koneshlou et al. (Koneshlou et al., 2011) reported that shallow cryogenic treatment increases wear resistance of AISI H11 and AISI H13 steels.

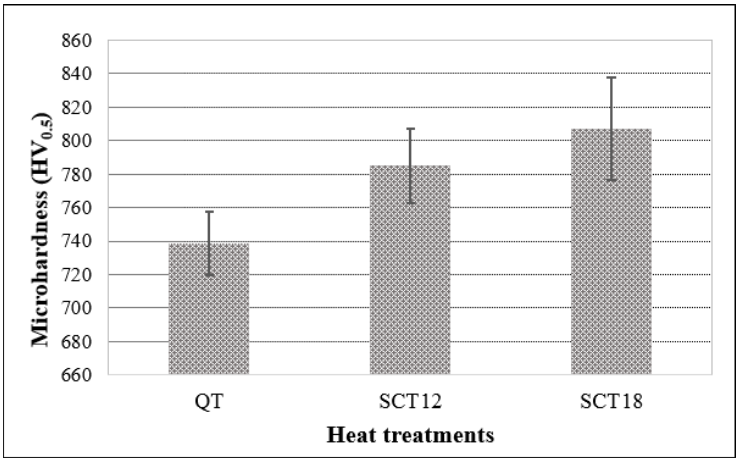


Figure 8. Microhardness graph of different heat-treated 1.2436 steel samples

Figure 10 shows the 1000X magnification SEM images of the worn surfaces as a result of the wear tests. It is observed that plastic deformation occurs with the sliding mechanism due to friction in all three samples. It is observed that oxide layers are formed during wear on the worn surfaces of all samples. It is perceived that the oxide layer acts as a protecting film, which reduces the wear rate of the steel (Prince et al., 2020). Similarly, Kurşuncu (Kurşuncu, 2021) argued in his study that oxidation on the worn surface positively affects wear resistance. Again, it is seen that there are adhesions from the abrasive disc on the worn surfaces of all samples. In addition, micro cavities were formed in the untreated sample, but not in the cryogenically treated samples.

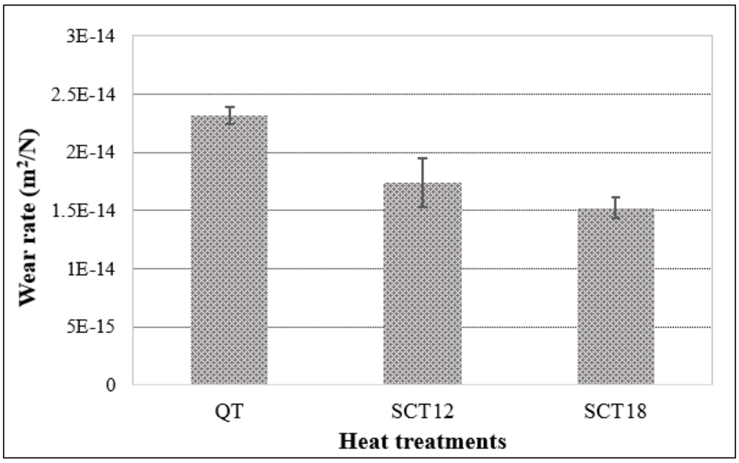
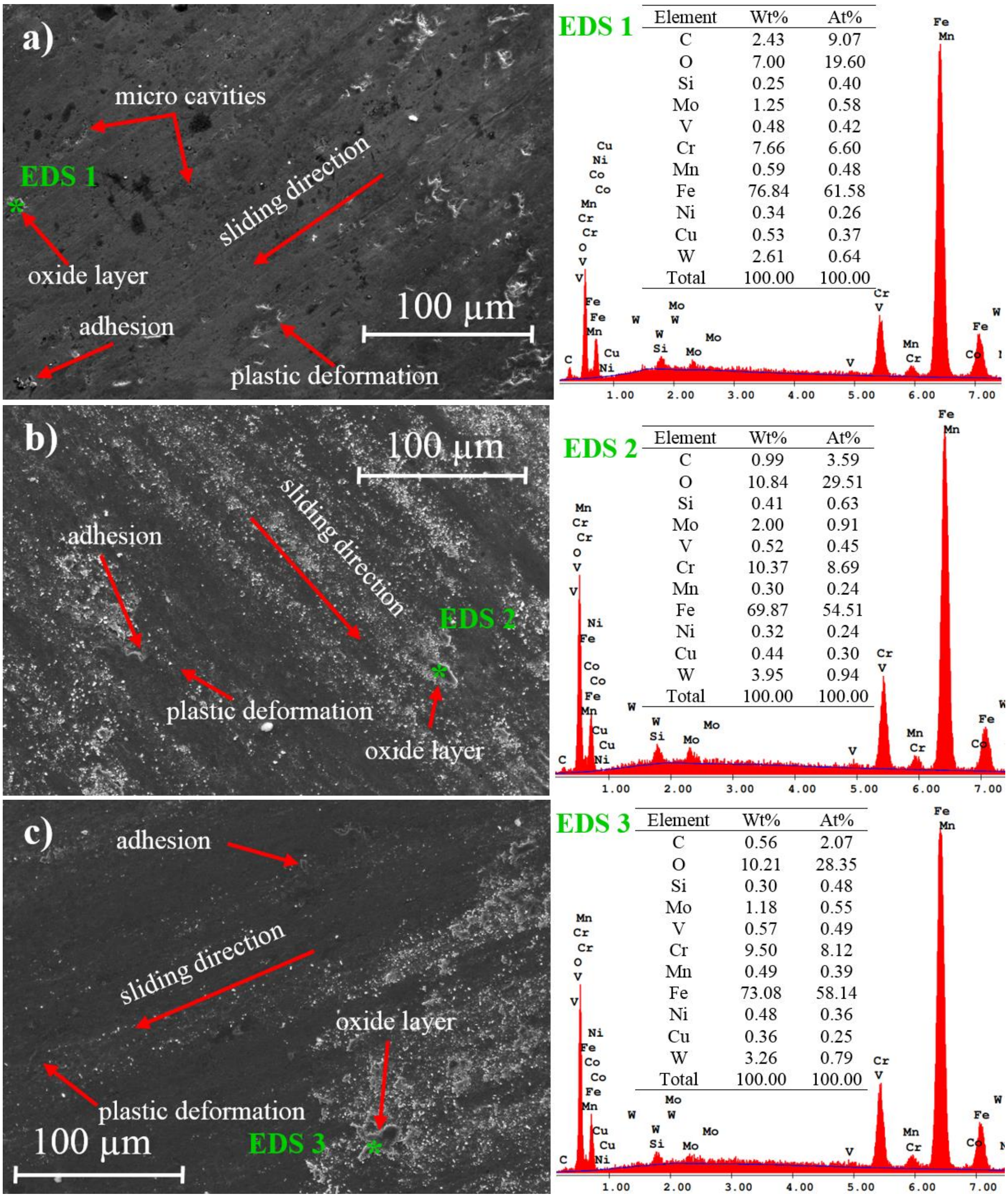


Figure 9. Wear rate graph of different heat-treated 1.2436 steel samples



EDS 1

Element	Wt%	At%
C	2.43	9.07
O	7.00	19.60
Si	0.25	0.40
Mo	1.25	0.58
V	0.48	0.42
Cr	7.66	6.60
Mn	0.59	0.48
Fe	76.84	61.58
Ni	0.34	0.26
Cu	0.53	0.37
W	2.61	0.64
Total	100.00	100.00

EDS 2

Element	Wt%	At%
C	0.99	3.59
O	10.84	29.51
Si	0.41	0.63
Mo	2.00	0.91
V	0.52	0.45
Cr	10.37	8.69
Mn	0.30	0.24
Fe	69.87	54.51
Ni	0.32	0.24
Cu	0.44	0.30
W	3.95	0.94
Total	100.00	100.00

EDS 3

Element	Wt%	At%
C	0.56	2.07
O	10.21	28.35
Si	0.30	0.48
Mo	1.18	0.55
V	0.57	0.49
Cr	9.50	8.12
Mn	0.49	0.39
Fe	73.08	58.14
Ni	0.48	0.36
Cu	0.36	0.25
W	3.26	0.79
Total	100.00	100.00

Figure 10. SEM images of different heat-treated 1.2436 steel samples worn surfaces a) QT, b) SCT12, and c) SCT18

4. CONCLUSION

This study investigated the effects of shallow cryogenic treatment on the microstructural and mechanical properties of 1.2436 tool steel. The results obtained from the study are as follows.

- Shallow cryogenic treatment positively affected the microstructure, hardness, and wear resistance of 1.2436 tool steel.
- Shallow cryogenic treatment provided a denser and more homogeneous carbide distribution in 1.2436 steel. After the shallow cryogenic treatment, the carbide content of the steel sample increased by approximately 18.80%.
- Higher hardness was obtained in the cryogenically treated 1.2436 steel samples. Compared to the QT sample, the hardness of the SCT12 and SCT18 samples is 6.27% and 9.28% higher.
- Cryogenic treatment improved the wear resistance of 1.2436 steel. The SCT12 and SCT18 samples were 25% and 34.37% less worn than the QT sample.
- Shallow cryogenic treatment applied for 18 hours gave better microstructural, hardness and wear resistance results.
- Plastic deformation, oxide layers and adhesions were observed on worn surfaces. Micro-cavities were formed on the worn surface of the QT sample.

5. ACKNOWLEDGEMENTS

This work is supported by Düzce University Scientific Project (2021.22.01.1177).

6. CONFLICT OF INTEREST

Authors approve that to the best of their knowledge, there is not any conflict of interest or common interest with an institution/organization or a person that may affect the review process of the paper.

7. AUTHOR CONTRIBUTION

Nursel ALTAN ÖZBEK contributed determining the concept of the research and research management, Nursel ALTAN ÖZBEK and Onur ÖZBEK contributed design process of the research and research management, data analysis and interpretation of the results, critical analysis of the intellectual content, preparation of the manuscript, and final approval and full responsibility.

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