İmalat Teknolojileri ve Uygulamaları
Cilt: 5, No: 3, 2024 (172-192)
Araştırma Makalesi
e-ISSN: 2717-7475

Manufacturing Technologies and Applications
Vol: 5, Issue: 3, 2024 (172-192)
Research Article
e-ISSN: 2717-7475

Multi-Objective Optimization of Dry Sliding Wear in Cryogenically Treated High-Performance AISI 9310 Steel: An Integrated Approach Using Grey Relational Analysis and Taguchi Method

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ARTICLE INFORMATION

Received: *19.08.2024*
Accepted: *22.10.2024*

Keywords:
AISI 9310 steel
Dry sliding wear
Friction coefficient
Cryogenic treatment
Taguchi method
Grey relational analysis

ABSTRACT

AISI 9310 steel is widely used in the aerospace and defense industries due to its superior mechanical properties and corrosion resistance. This study introduces a novel approach by investigating the effects of both shallow (SCT) and deep (DCT) cryogenic treatments on the wear resistance and surface properties of AISI 9310 steel. An integrated methodology that combines Grey Relational Analysis and the Taguchi method for optimization was applied. Wear performance was evaluated using a ball-on-disc tribometer in dry sliding wear tests, revealing significant improvements. The results show that the hardness of the samples processed with DCT increased by 30%, while their volume loss decreased by 14%. In samples processed with SCT, hardness increased by 12%, with a corresponding 7% reduction in volume loss. Furthermore, the friction coefficient improved by 9% in DCT samples and by 5% in SCT samples. As the load increased, volume loss increased by 16% (from 3400 mm³ to 3950 mm³), while the friction coefficient decreased by 11% (from 0.448 µ to 0.498 µ). ANOVA analyses indicated that cryogenic treatment had the greatest effect on both volume loss and the friction coefficient. Regression analysis revealed an excellent model fit, with R^2 values of 97.63% for volume loss and 99.42% for the friction coefficient. These findings suggest that cryogenic treatments significantly enhance the wear resistance of AISI 9310 steel and improve performance under varying load conditions. Additionally, they highlight the critical role of cryogenic processes in extending the service life of materials used in industrial environments, providing valuable insights for future engineering applications.

Kriyojenik İşlem Görmüş Yüksek Performanslı AISI 9310 Çeliğinin Kuru Kayma Aşınmasının Çok Amaçlı Optimizasyonu: Gri İlişkisel Analiz ve Taguchi Yöntemi Kullanılarak Entegre Bir Yaklaşım

MAKALE BİLGİSİ

Alınma: *19.08.2024*
Kabul: *22.10.2024*

Anahtar Kelimeler:
AISI 9310 çeliği
Kuru kayma aşınma
Sürtünme katsayısı
Kriyojenik işlem
Taguchi methodu
Gri ilişkisel analiz

ÖZET

AISI 9310 çeliği, üstün mekanik özellikleri ve korozyon direnci nedeniyle havacılık ve savunma sanayinde yaygın olarak kullanılmaktadır. Bu çalışma, hem sığ (SCT) hem de derin (DCT) kriyojenik işlemlerin AISI 9310 çeliğinin aşınma direnci ve yüzey özellikleri üzerindeki etkilerini araştırarak, Gri İlişkisel Analiz ve Taguchi yöntemini birleştirerek optimizasyon sağlayan entegre bir metodoloji uygulayarak yeni bir yaklaşım sunmaktadır. Aşınma performansı, kuru kayma aşınma testlerinde ball-on-disk tribometresi kullanılarak değerlendirilmiş ve önemli iyileştirmeler ortaya çıkarılmıştır. Sonuçlar DCT ile işlenmiş numunelerin sertliği %30 artmış ve hacim kaybı %14 azalmıştır. SCT ile işlenmiş numunelerde sertlik %12 artmış ve buna karşılık hacim kaybında %7 azalma olmuştur. Ayrıca, sürtünme katsayısı DCT'de %9 ve SCT numunelerinde %5 iyileşmiştir. Yükün artmasıyla hacim kaybı %16 artış gösterirken (3400 mm³'den 3950 mm³'e), sürtünme katsayısı %11 azalmıştır (0,448 µ'den 0.498 µ'e). ANOVA analizleri, kriyojenik işlemin hacim kaybı ve sürtünme katsayısı üzerinde en büyük etkiye sahip olduğunu göstermiştir. Regresyon analizi, hacim kaybı için R^2 değerlerinin %97.63 ve sürtünme katsayısı için %99.42 olmasıyla model uyumunun mükemmel olduğunu ortaya koymuştur. Bu bulgular, kriyojenik işlemlerin AISI 9310 çeliğinin aşınma direncini önemli ölçüde artırdığını ve değişen yük koşulları altında performansı iyileştirdiğini göstermektedir. Ayrıca, endüstriyel ortamlarda kullanılan malzemelerin kullanım ömrünü uzatmada kriyojenik işlemlerin kritik rolü vurgulanarak, mühendislikte gelecekteki uygulamalara yönelik değerli bilgiler sağlanmaktadır.

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To cite this article: E. Altaş, Multi-Objective Optimization of Dry Sliding Wear in Cryogenically Treated High-Performance AISI 9310 Steel: An Integrated Approach Using Grey Relational Analysis and Taguchi Method, Manufacturing Technologies and Applications, 5(3), 172-192, 2024.https://doi.org/10.52795/mateca. 1535444,

1. INTRODUCTION (GİRİŞ)

Human beings are exposed to tribological effects throughout their lives, whether they are aware of it or not. While wear and friction can sometimes be beneficial, many industrial and engineering applications result in undesirable outcomes. In fact, there are instances where both positive and negative effects of wear and friction are observed within the same studies for different purposes. In many applications involving friction, the primary goal is to minimize material loss. To reduce material and energy losses due to friction and wear, alternative surface treatments and surface enhancement techniques are employed [1].

In addition to the development of new metal alloys, various techniques such as heat treatment, coating, shot peening, and cryogenic processes are commonly used to improve the mechanical properties of materials. These methods aim to reduce costs, lower weight, and extend the lifespan of components. Using these methods, many mechanical properties of materials such as strength, fatigue strength, and wear resistance can be improved.[2,3]. AISI 9310 steel, which is used as the workpiece in this study, is preferred in many areas such as automotive, space and aviation, and defense industries due to its high corrosion resistance, high pressure, and impact resistance [4]. Materials made of AISI 9310 steel, which has a wide range of uses, are required to have a long working and service life, so it is desired to improve the surface properties of these elements. It can be said that unlike traditional heat treatments, the cryogenic treatment affects the entire structure of the material and not only increases the surface hardness but also causes an increase in the uniform hardness in the internal structure of the material [5,6].

It is possible to give various properties to AISI 9310 steel materials by heat treatments. Cryogenic treatment is also known as subzero treatment. With this method, the transformation of residual austenite in the material to martensite, which has been subjected to conventional heat treatment, provides the formation of fine carbide precipitates and homogeneous Fe-C distribution. This process is shallow cryogenic treatment and It is divided into two as deep cryogenic process. The application of cryogenic process between -80 ºC and -140 ºC is called shallow cryogenic process, and the application between -140 ºC and -196ºC is called deep cryogenic process. [7,8]. Cryogenic treatment improves many properties of the applied materials such as hardness, toughness, wear resistance, electrical conductivity [6,9]. Arslan and Özdemir [10] the effects of cryogenic treatment on DIN 9861 punches made of AISI D3 tool steel on the wear behavior of the punch were investigated. The researchers subjected D3 tool steel punches to cryogenic treatment at -145 °C for different periods of time. It was found that the cryogenic treatment increased the wear resistance of D3 tool steel punches, but It was determined that the duration of cryogenic treatment did not have a significant effect on the punch life. It was also observed that the cryogenic treatment increased the hardness value of the samples but the hardness value decreased after the tempering process. Essam et al. [11], investigated the effects of conventional and deep cryogenic treatment (DCT) on shock-resistant cold work tool steel. Three alloys containing vanadium (V) and niobium (Nb) were first hardened in water at 900 °C and then tempered at 200 °C. DCT was applied at -196 °C for 5 hours and then tempered. Wear characteristics were evaluated using pin-on-disc testing at a sliding speed of 0.5 m/s. The results showed that Sample 3, treated with DCT, had the lowest coefficient of friction (0.33) under a 100 N load and exhibited the least weight loss under a 50 N load, as they have determined. Kara et al. [3] investigated the effects of conventional and deep cryogenic treatment (DCT) on the mechanical properties of Sleipner cold work tool steel. The samples underwent shallow cryogenic treatment (SCT) at -80°C and DCT at -180°C. The wear behavior was evaluated using ball-on-disc testing. Cryogenic treatments led to a 6.53% increase in hardness and a more uniform microstructure with secondary carbide precipitations. The conventionally heat treated sample exhibited the highest coefficient of friction (0.63), while the DCT-12 sample achieved the lowest (0.58). Additionally, the DCT-36 specimen demonstrated the lowest wear rate under all conditions. In another study, Gecu subjected AISI H13 hot work tool steels to both shallow and deep cryogenic treatments. The samples underwent either single or double tempering. Cooling to -196°C increased the martensite content and hardness, while tempering altered the martensite morphology and resulted in the precipitation of Cr7C3 and V2C carbides. Both cryogenic treatments and tempering enhanced wear resistance, although the effect of double tempering was less pronounced. Analysis of the worn surfaces indicated that delamination and abrasion were the predominant wear mechanisms during sliding [12]. Nas and Akıncıoğlu investigated the electroerosion machining performance of cryogenically treated nickel-based superalloys. The study revealed that electrical conductivity was highest in samples treated with shallow cryogenic treatment, followed by those treated with deep cryogenic treatment, while untreated samples exhibited the lowest conductivity. It was also stated that cryogenic treatments made a minimal contribution to hardness, and the effect of tempering treatments varied depending on the material's structure [13]. Baldissera et al. [14] conducted deep cryogenic treatment on AISI 302 steel to evaluate its impact on fatigue and corrosion resistance. Their findings indicated that while cryogenic treatment had little effect on corrosion resistance, it significantly improved the fatigue life of the steel. Myeong et al. [15] observed that applying cryogenic treatment to stainless steel enhanced fatigue resistance and led to the formation of a fine martensite structure. Darwin et al. [16] increased the wear resistance of SR34 stainless steel segments containing 18% Cr by subjecting them to cryogenic treatment for 12, 24, and 36 hours. The optimal results were achieved with 36 hours of treatment, revealing that after temperature, the duration of cryogenic treatment was the most influential factor. Höke et al. [17] determined that applying cryogenic cooling to AISI 4140 steel had a positive effect on microhardness. Senthilkumar et al. [18] examined the residual stress in AISI 4140 steel and compared the impacts of shallow cryogenic treatment (-80°C for 5 hours) with deep cryogenic treatment (-196°C for 24 hours). In the results obtained, they observed that the decrease in cryogenic process temperature caused more austenite to martensite.

In recent years, more comprehensive results have been achieved with the help of Taguchi-based GRA in experiments involving multiple factors and multiple characterization of factors [19,20]. In engineering applications, it is quite difficult to determine the best among many alternatives in the optimization of multiple performance criteria. The process of determining the best criterion by evaluating existing alternatives according to multiple criteria is known as the Multi-Criteria Decision Making (MCDM) problem. GRA method is one of these methods. GRA method is used together with Taguchi Method [21, 22]. Natarajan et al. [23] applied the Taguchi Method together with GRA to optimize parameters such as material wear rate, electrode wear rate, and cutting gap in the micro-EDM process. They identified pulse duration, current, and gap voltage as experimental parameters, concluding that pulse duration had the greatest effect on these performance characteristics. Conversely, various statistical programs have been used to optimize wear and other properties in the machining of cryogenically treated AISI D2 steel and other materials [24, 25]. A comprehensive review of the literature shows that cryogenic treatment can significantly enhance the mechanical properties of steel. Improved wear resistance is primarily attributed to the transformation of retained austenite into martensite and the uniform distribution of carbide precipitates, particularly through deep cryogenic treatments, and to a lesser extent, shallow cryogenic treatments. However, while much research has focused on improving the mechanical properties of steel and other materials, there is a notable lack of studies thoroughly investigating the combined effects of GRA and Taguchi optimization on the dry sliding wear of cryogenically treated AISI 9310 steel.

This study presents an innovative approach by combining Grey Relational Analysis (GRA) and the Taguchi method to optimize the dry sliding wear parameters of AISI 9310 steel, addressing a significant gap in the existing research. Although studies in the literature have shown improvements in wear resistance in cryogenically treated materials, it has been determined that sufficient scientific studies have not been conducted on how these processes can be systematically integrated to improve multiple performance metrics, such as volume loss and coefficient of friction. This research aims to fill these gaps by investigating the dry sliding wear performance of cryogenically treated AISI 9310 steel. The optimal parameter levels for improving the friction coefficient and volume loss properties will be determined using GRA in conjunction with the Taguchi method. These enhancements are expected to make significant contributions to industrial applications.

2. EXPERIMENTAL SETUP (DENEYSEL KURULUM)

2.1. Workpiece Material Used In The Study (Çalışmada Kullanılan İş Parçası Malzemesi)

AISI 9310 grade alloy steel is utilized in the automotive, agricultural, defense, and aerospace industries. It is a versatile alloy known for its atmospheric corrosion resistance and reasonable strength. Generally, its wear resistance, toughness, and fatigue strength are superior to those of low-carbon steels. Table 1 presents the chemical composition of the material used.

Table 1. Chemical composition of AISI 9310 low-alloy steel (%Weight) (AISI 9310 düşük alaşımlı çeliğin kimyasal bileşimi (%Ağırlık)

C	Cr	Ni	Mo	Mn	Cu	O	Si	S	Fe
0.11	1.32	3.19	0.12	0.56	0.13	0.001	0.24	0.004	Bal

2.2. Application of Cryogenic Process (Kriyojenik İşlemin Uygulanması)

The cryogenic process for AISI 9310 steel was conducted in a specially designed cooling tank equipped with a fan system, without immersion in liquid nitrogen, and controlled by a computer. Based on a review of the literature, the cryogenic holding temperatures were established as shallow (-80 ºC) and deep (-180 ºC). A schematic diagram of the cryogenic treatment unit is presented in Fig. 1.

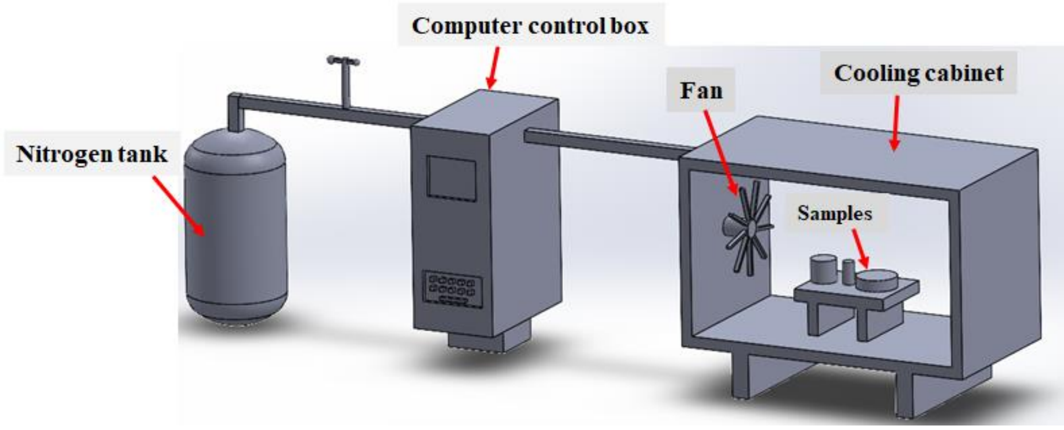

Figure 1. Schematic representation of the cryogenic treatment unit (Kriyojenik işlem ünitesinin şematik gösterimi)

To increase the hardness of AISI 9310 steel and enhance its corrosion and wear resistance, cryogenic treatment was applied. The cryogenic treatment applied to the AISI 9310 steel sample was carried out in a specially designed computer-controlled cryogenic treatment unit (Figure 2). To prevent thermal shocks and microcracks that may occur in the microstructure, cryogenic process tests were carried out with gradual cooling and heating. The samples were cooled to -80 °C and -180 °C for 6 hours. The temperature values of -80 °C and -180 °C The samples were kept at this temperature for 24 hours. After the process, the samples gradually reached room temperature in 6 hours (Fig. 2).

2.3. Hardness Measurements (Sertlik Ölçümleri)

The hardness values of the hardness changes of the cryogenic treatment samples were measured on a micro scale using the Vickers method. The hardness of the prepared samples was determined by using the microhardness measuring device and taking the average of five measurement results. The measurements were carried out under a load of 200 grams for 10 seconds.

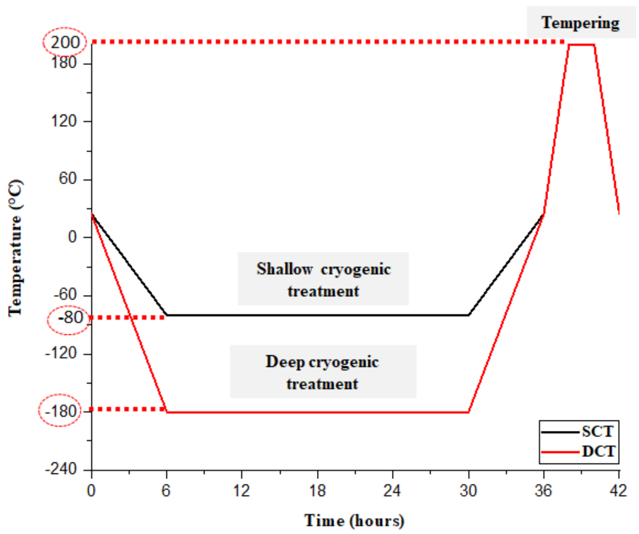

Figure 2. The cryogenic treatment process of AISI 9310 steel (AISI 9310 çeliğinin kriyojenik işlem süreci)

2.4. Tests for dry sliding wear (Kuru kayma aşınması testleri)

In this study, dry sliding wear tests of cryogenically treated AISI 9310 steel were conducted using a reciprocating ball-on-disc tribometer (Turkyus). A 6 mm diameter WC-Co ball (hardness 19 GPa) was used as the abrasive element during these tests. The experimental parameters are summarized in Table 2.

Table 2. Fundamental parameters employed in wear Testing (Aşınma testinde kullanılan temel parametreler)

Parameters	Level 1	Level 2	Level 3
Load (N)	5	10	15
Sliding Speed (m/s)	0.02	0.02	0.02
Test Duration (min)	30	30	30

To ensure reliable results, each experiment was repeated three times and averaged. Friction coefficients were recorded using specialized software, and wear traces were analyzed with a 3D surface profilometer. This equipment provided a 2D profile of the wear depth and width, from which the wear area and volume were calculated. Measurements were taken from multiple points on each trace, and this process was repeated for all sample types. A schematic of the wear testing setup and procedures is shown in Fig. 3.

2.5. Taguchi Experimental Design Approach (Taguchi Deneysel Tasarım Yaklaşımı)

Taguchi's experimental design method assists researchers in systematically organizing the order of changes to processing parameter levels, making the experimental process more efficient. This method employs various experimental designs, including orthogonal arrays, to examine how different factors influence results while minimizing the number of experiments required [21].

In this research, the Taguchi method was used to optimize the processing parameters for dry sliding wear tests conducted on AISI 9310 steel. The Taguchi method is a quality analysis approach typically applied in the optimization of a single output. Initially, the outcomes of the objective function (coefficient of friction and volume loss) were transformed into S/N ratios. Three different performance characteristics are utilized to calculate the S/N ratio: 'nominal best,' 'smallest best,' and 'largest best.' To minimize both volume loss and friction coefficient, the 'smallest best' performance criterion was selected for this study. The S/N ratio for the 'smallest is best' criterion was determined using Equation 1. The Minitab18 software was employed for this analysis. The effects of processing factors were assessed using analysis of variance (ANOVA), and the wear tests on AISI 9310 steel were conducted at a 95% confidence level with a significance threshold of $p < 0.05$ [6,26].

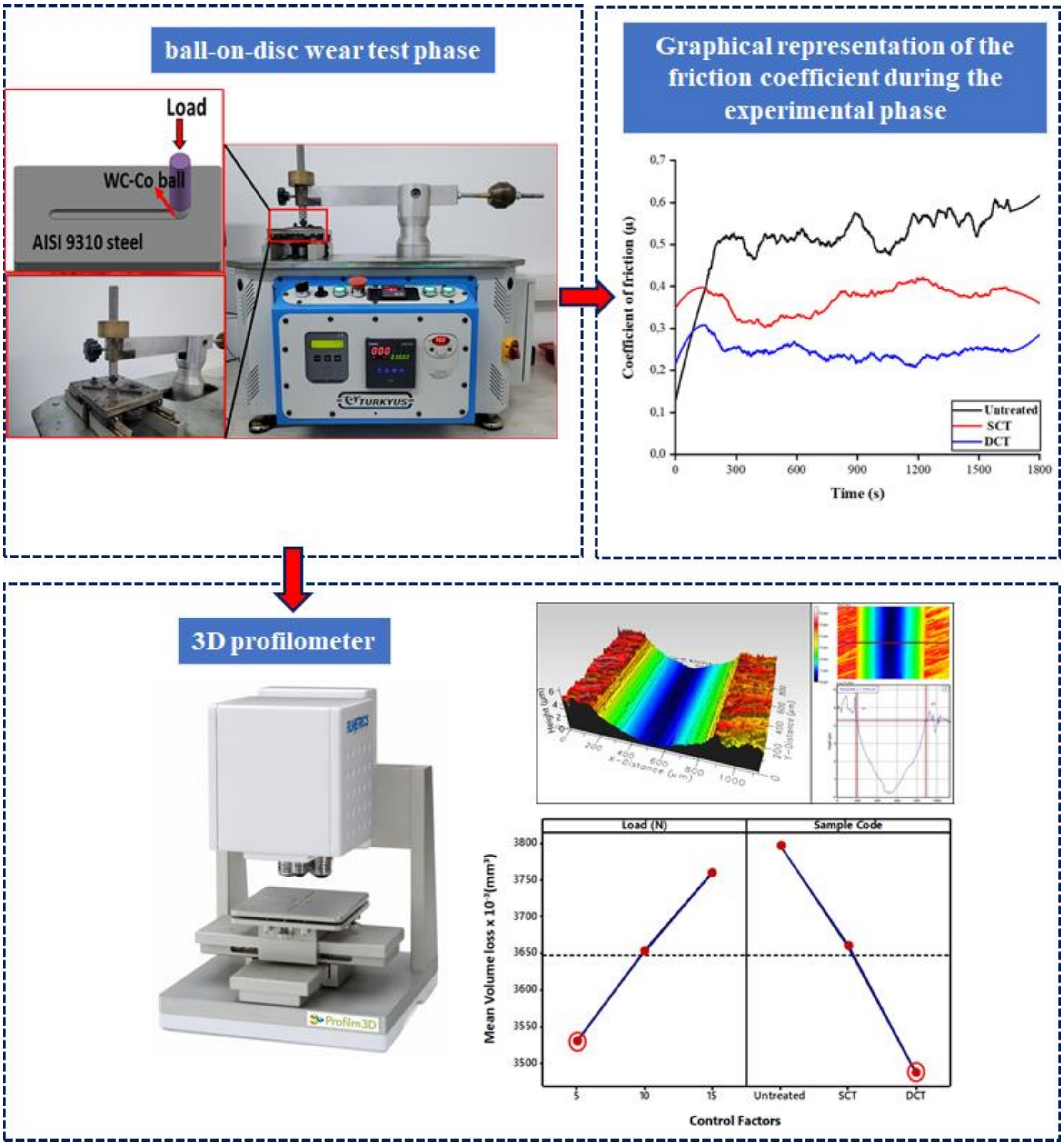

Figure 3. A schematic view of the abrasion tester used in the test phase showing all stages (Test aşamasında kullanılan aşınma test cihazının tüm aşamalarını gösteren şematik görünümü)

$$S/_N = -10\,log\left[\frac{1}{n}\sum_{i=1}^{n} yi^2\right] \qquad (1)$$

This experimental study utilized AISI 9310 steel in three conditions: untreated, shallow cryogenically treated (SCT), and deep cryogenically treated (DCT). Experiments were conducted using three different loads (5, 10, and 15 N), a sliding speed of 0.02 m/s, and a test duration of 30 minutes. The parameters used and their respective levels are presented in Table 3.

Table 3. Factors and levels of the experimental variables (Deneysel değişkenlerin faktörleri ve düzeyleri)

Process parameters	**Level 1**	**Level 2**	**Level 3**
Load (N)	5	10	15
Cryogenic treatment	Untreated	SCT	DCT

Dry sliding wear tests were designed in accordance with the Taguchi L_9 orthogonal arrangement (Table 4).

Table 4. Taguchi L_9 orthogonal arrangement (Taguchi L9 ortogonal düzenlemesi)

Exp. No	Load (N)	Cryogenic Treatment
1	5	Untreated
2	5	SCT
3	5	DCT
4	10	Untreated
5	10	SCT
6	10	DCT
7	15	Untreated
8	15	SCT
9	15	DCT

2.6. Grey Relational Analysis Method (Gri İlişkisel Analiz Yöntemi)

This study aims to identify the optimal machining parameters for dry sliding wear tests through the application of Grey Relational Analysis (GRA). In the study, after the experimental data were normalized between 0 and 1, grey relational coefficients were derived from the normalized results and the grey relational degree was obtained according to these coefficients. The multiple parameters were evaluated according to their grey relational degrees. The 'smallest is best' criterion was calculated according to Equation 2 [27].

$$x_i(k) = \frac{max\, x_i^0(k) - x_i^0(k)}{max\, x_i^0(k) - min\, x_i^0(k)} \tag{2}$$

Here, $x_i(k)$: represents the normalized version of the x_o series, $x_i^0(k)$: represents the kth response value of the ith alternative, $\min x_i^0(k)$: represents the lowest performance value, $max\, x_i^0(k)$: represents the lowest performance value.

In the second phase, the grey correlation coefficient is calculated to represent the relationship between the desired and actual measured data based on the normalized experimental results. Equations 3 and 4 provide the formulas for computing the grey correlation coefficient.

$$\xi_i\,(k) = \frac{\Delta_{min} + \xi \Delta_{max}}{\Delta_{0i}(k) + \xi \Delta_{max}} \tag{3}$$

$$\Delta_{0i}(k) = |x_0(k) - x_i(k)| \tag{4}$$

Here is the absolute value difference between $x_0(k)$ and $x_i(k)$. $x_0(k)$ is the ideal sequence or reference sequence. Δ_{min} is the minimum value of Δ_{0i}, and Δ_{max} is the maximum value of Δ_{0i}. Here, ξ is a separating coefficient between 0 and 1 and is usually taken as 0.5. Equation 5 calculates the Grey Relational Degree after determining the value of the Grey Relational coefficient.

$$\gamma_i = \frac{1}{n}\sum_{k=1}^{n} \xi_i(k) \tag{5}$$

In this contex, γ_i represents the grey relational degree for the i series, while n denotes the number of response characteristics. Ultimately, these calculations are used to identify the optimal processing parameters [21,27].

3. RESULTS AND DISCUSSION (SONUÇLAR VE TARTIŞMA)

3.1. Hardness Measurements (Sertlik Ölçümleri)

Enhanced material hardness is typically associated with improved wear resistance [5]. To assess how cryogenic treatment affects the hardness of AISI 9310 steel, this study performed microhardness measurement tests. The arithmetic average of five different measurements taken for each sample is shown in Fig. 4. Cryogenic treatment led to a substantial enhancement in the material's hardness. Fig. 4 indicates a significant improvement in hardness for samples treated with both shallow and deep cryogenic processes. The samples that underwent cryogenic treatment showed the lowest microhardness value at 410 HV, while the highest microhardness value of 540 HV was observed in the samples treated with deep cryogenic treatment (DCT). Compared to the untreated samples, hardness values increased by 12% in those subjected to shallow cryogenic treatment (SCT) and by 30% in samples treated with deep cryogenic treatment (DCT). Surface hardness after cryogenic treatment is generally directly related to wear resistance. The obtained results strongly align with the findings of Akhbarizadeh et al. [28], who demonstrated the positive effects of cryogenic treatment on the hardness and wear behavior of D6 tool steel. In our study, a 30% increase in hardness and a significant improvement in wear resistance were observed in the samples subjected to deep cryogenic treatment. These findings support the existing literature, which suggests that cryogenic treatments induce fundamental changes in the material structure, leading to enhanced wear resistance. Myeong et al.[15], noted that cryogenic treatment increases martensitic transformation, thereby enhancing the material's hardness and positively impacting wear resistance. In the study by Kara et al.[3], it was demonstrated that cryogenic treatment markedly enhances the hardness of Sleipner steel by decreasing the residual austenite in its microstructure. This reduction in residual austenite, which has relatively low hardness, results in a higher fraction of hard martensite, thereby increasing overall hardness. This supports the positive effects of cryogenic treatment on material hardness, as emphasized in the studies by Senthilkumar et al.[29]. Additionally, Gül et al.[30] applied both deep and shallow cryogenic treatments to Hastelloy C-22 superalloy to improve its wear resistance. Wear tests conducted using the ball-on-flat method with a reciprocating motion showed a 14% increase in hardness for the SCT-treated sample and a 45% increase for the DCT-treated sample compared to the untreated sample.

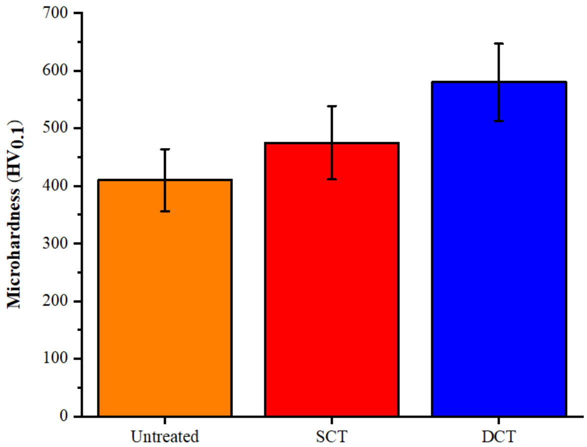

Figure 4. Microhardness measurement values of AISI 9310 steel (AISI 9310 çeliğinin mikrosertlik ölçüm değerleri)

3.2. Dry Sliding Wear Test Results (Kuru Kayma Aşınma Testi Sonuçları)

Table 5 presents the volume loss data from the dry sliding wear tests of AISI 9310 steel, conducted using the Taguchi L_9 orthogonal array, along with the S/N ratios calculated using Equation (1). The arithmetic mean of the volume loss values obtained was calculated to be 3467.778 x 10^{-3} (mm^3). The average S/N ratio for the volume loss values was determined to be-71.23 dB.

Additionally, the influence of each control factor (load and cryogenic treatment) on volume loss was evaluated using the S/N ratio response presented in Table 6. The study determined that the optimal volume loss corresponded to the lowest measured value. To ascertain the most effective S/N ratio for achieving minimal volume loss while enhancing product quality and reducing costs, the 'smallest is best' performance criterion was employed [9,26]. Table 6 summarizes the average S/N ratios calculated for each level of the processing parameters used in the experiments. The highest S/N ratio indicated in the table corresponds to the minimum volume loss.

Table 5. Volume loss and S/N ratios for AISI 9310 steel, obtained from dry-sliding wear tests under various processing conditions

Exp. No.	Load (N)	Cryogenic Treatment	Volume loss x 10^{-3} (mm^3)	Calculated S/N ratio (ni=1-18) (dB)
1	5	Untreated	3640	-71.222
2	5	SCT	3550	-71.005
3	5	DCT	3400	-70.630
4	10	Untreated	3800	-71.596
5	10	SCT	3680	-71.317
6	10	DCT	3480	-70.832
7	15	Untreated	3950	-71.932
8	15	SCT	3750	-71.481
9	15	DCT	3580	-71.078

Table 6. Table of average S/N ratios computed for the volume loss values (Hacim kaybı değerleri için hesaplanan ortalama S/N oranlarının tablosu)

Volume loss x10^3 (mm^3)		
Level	**Load (N)**	**Cryogenic Treatment**
S/N Ratio		
1	**-70.95**	-71.58
2	-71.25	-71.27
3	-71.50	**-70.85**
Delta	0.54	0.74
Rank	2	1
Means		
1	**3530**	3797
2	3653	3660
3	3760	**3487**
Delta	230	310
Rank	2	1

The basic effect graphs, which consider the S/N ratios and average values in accordance with the "smallest is best" performance criterion for the volume loss of processing parameters and levels, are presented in Fig. 5. Based on the graphical analysis of the S/N ratios, the optimal processing parameters for achieving minimal volume loss were identified as a 5 N load combined with DCT cryogenic treatment. Fig. 5 also displays the volume loss measurements of the samples obtained from dry sliding wear tests conducted under varying loads and cryogenic treatment conditions. The smallest volume loss (3400 mm^3) was observed in samples treated with DCT, while the largest volume loss (3950 mm^3) was found in untreated samples. Compared to untreated samples, SCT-treated specimens exhibited a 7% reduction in volume loss, whereas DCT-treated specimens demonstrated a 14% reduction. When examining the results in terms of load, it is evident that volume loss increased with the applied load during the wear tests across all samples [3,5]. This outcome is typically predictable; increasing the load applied to the abrasive tip enhances wear by affecting a larger surface area. In this process, material loss is triggered by the forces generated by different loads, which are generally divided into normal force and shear force. The normal force arises from the load applied by the indenter to the worn part, while the shear force is generated by the relative movement between the worn part and the abrasive tip. The load applied during the wear test allows the indenter to penetrate deeper into the surface of the sample, causing shear forces to remove material from the sample surface as a result of continuous movements [31,32]. Consequently, raising the applied load from 5 N to 10 N and 15 N resulted in increased material wear. Decreases in volume loss were observed with an increase in cryogenic treatment temperature, which can be attributed to the hardness values of the materials [5]. Of the three sample types, the deep cryogenic treated sample exhibited the least amount of wear. Furthermore, an analysis of the volume losses from the cryogenic treatments indicates an improvement in wear resistance due to these treatments. Upon reviewing the volume loss data, it was noted that wear losses increased in samples subjected to cryogenic treatment under lower load conditions compared to untreated specimens. However, this trend was reversed with an increase in load, as the cryogenic treatment improved wear resistance [33,34]. Previous studies have presented significant findings regarding the enhancement of wear resistance in steels through cryogenic treatment. In particular, deep cryogenic treatment (DCT) induces martensitic transformation and optimizes the distribution of fine carbide precipitates, resulting in a marked improvement in the wear resistance of steels [16,28]. These findings are consistent with the 14% reduction in volume loss and the 30% increase in hardness observed in your study on AISI 9310 steel. For instance, the work of Akhbarizadeh and colleagues on D6 steel demonstrated a significant reduction in volume loss due to cryogenic treatment. Similarly, the improvement in wear performance observed in DCT-treated samples in your study aligns with the existing literatüre [28].

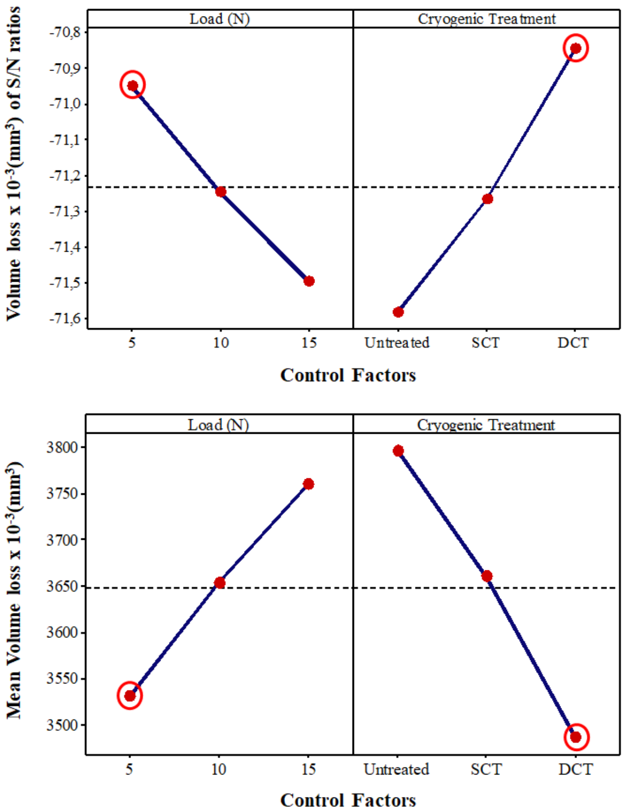

Figure 5. Graphs showing the S/N ratios of the volume loss values and the average of their measured values (Hacim kaybı değerlerinin S/N oranlarını ve ölçülen değerlerin ortalamasını gösteren grafikler)

In Fig. 6, the comparison of 3D surface profile images of wear scars on AISI 9310 steel is presented. The highest wear occurred under a 5 N load in untreated conditions (Fig. 6a), while the lowest wear was observed under a 5 N load in DCT conditions (Fig. 6c). All results are consistent with the hardness findings. Wear depth and width decrease with increasing hardness, which in turn reduces volume loss and enhances wear performance. In all samples, it was clearly observed that plastic deformations occurred on the outer edges of the wear scars. It is hypothesized that wear-induced heating leads to the plastic deformation of the material, causing it to adhere to the edges [5].

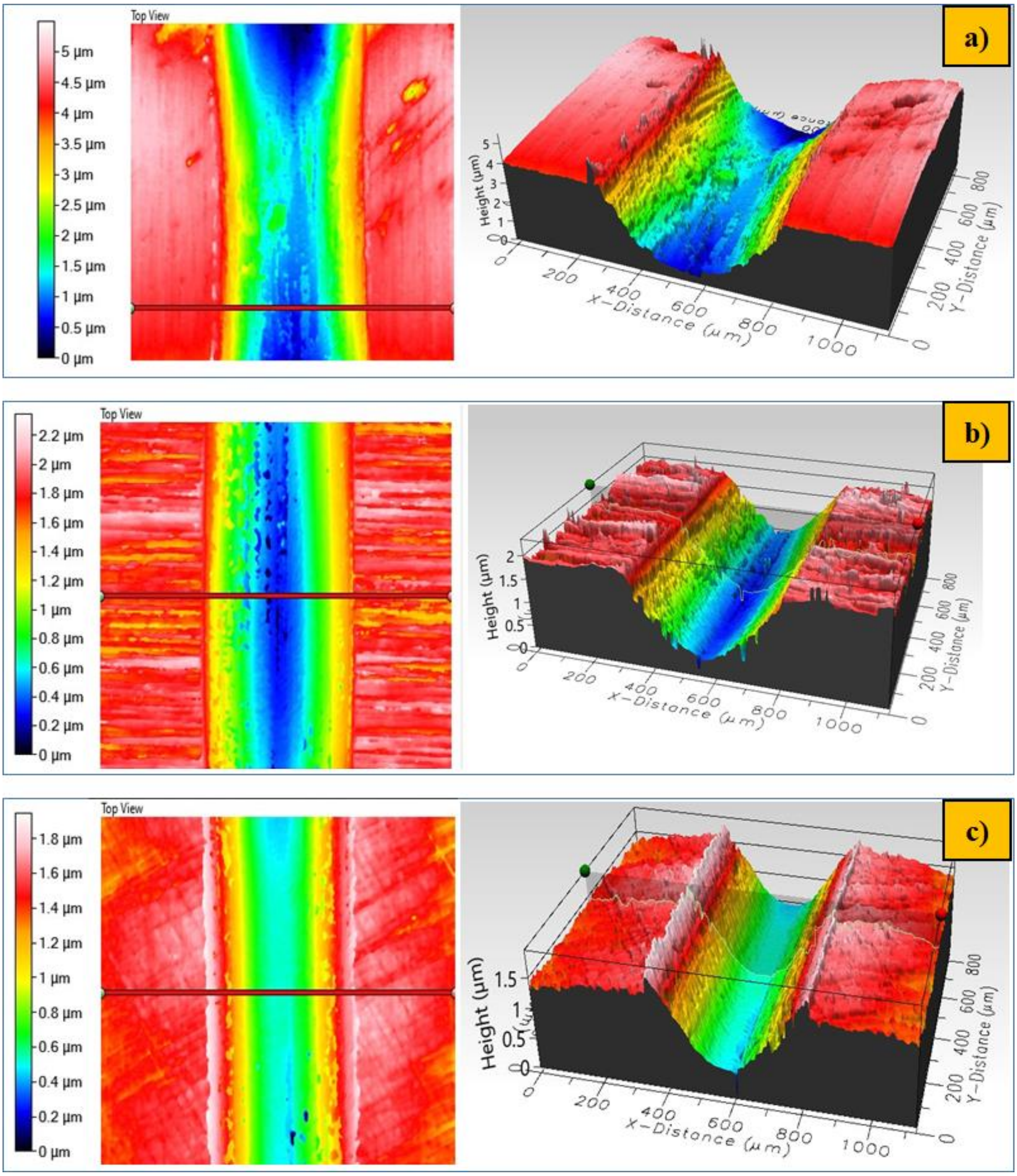

Figure 6. Surface topography images of AISI 9310 steel under 5N load a) untreated, b) SCT and c) DCT (5N yük altında AISI 9310 çeliğinin yüzey topografyası görüntüleri a) işlenmemiş, b) SCT ve c) DCT)

In the experimental analysis, the results of the variance analysis (ANOVA) used to evaluate the effects of processing parameters on volume loss and their statistical examination are presented in Table 7. ANOVA was conducted with a significance threshold of $p < 0,05$ and a confidence level of 95%. The obtained data indicated that the most significant contributions to volume loss, as shown in the ANOVA analysis, were from the cryogenic process at 63,03%, followed by the load at 34,60%. These findings align with the results of Khun et al. [35] on AISI D3 steel, which also reported that cryogenic treatment was the most significant factor affecting material properties. Regression analysis revealed that the model for predicting volume loss is statistically robust, with correlation coefficients of R-sq at 97.63% and adjusted R-sq (adj) at 95.26%, demonstrating a strong alignment between the parameters and their levels.

Table 7. Results of the ANOVA analysis for volume loss values (Hacim kaybı değerlerine ait ANOVA analizinin sonuçları)

	Variance source	Degree of freedom (df)	Sum of squares (SS)	Mean square (MS)	F	P	Contribution (%)
Volume loss x10^{-3} (mm^3)	**Load (N)**	2	79489	39744	29.2	0.004	34.60
	Cryogenic Treatment	2	144822	72411	53.2	0.001	63.03
	Error	4	5444	1361			2.37
	Total	8	229756				100.00

S= 36.8932 R-sq=97.63% R-sq (adj)=95.26%

The regression line and histogram graph used to examine the volume loss values are presented in Fig. 7. Most of the experimental data align well with the regression line. Additionally, the histogram exhibits characteristics of a normal distribution. In the distribution graphs, the data generally clusters around the zero line; however, some outliers were also detected. It is anticipated that these outliers are caused by factors such as vibrations of the wear device and fluctuations in ambient temperature. The low number of outliers indicates that the data generally fit the regression model. Overall, the results suggest a reasonable fit between the input and output parameters determined for volume loss. The concentration of data around a linear line further supports the meaningfulness and validity of the findings [36].

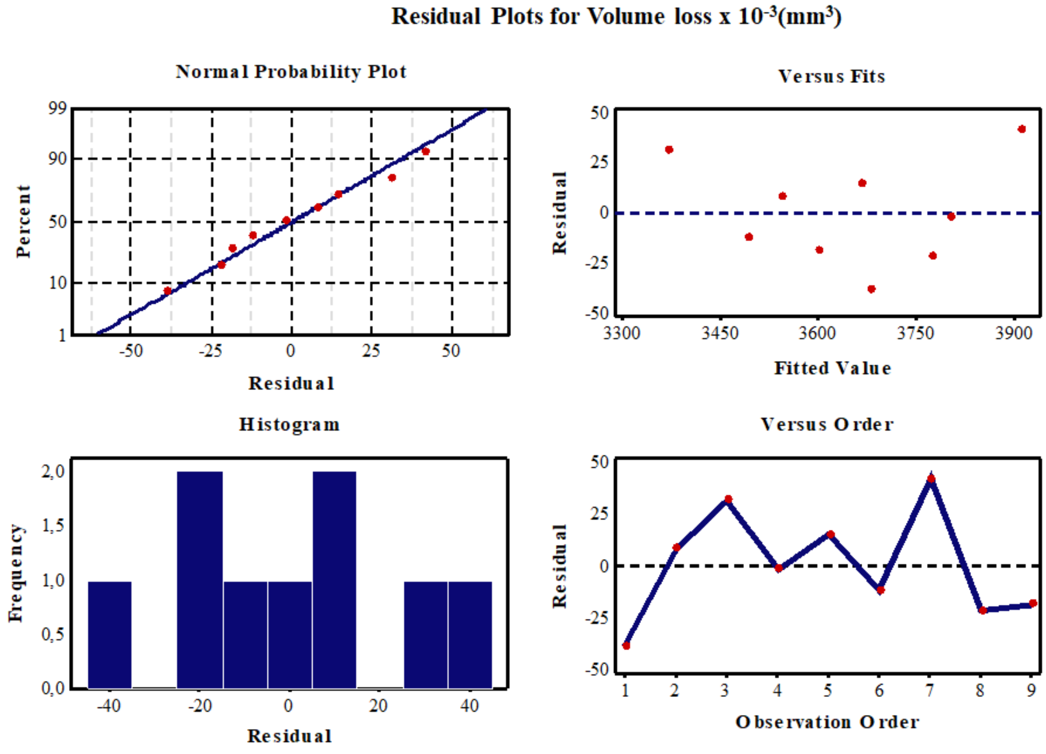

Figure 7. Normal distribution and residual analysis graphs for grand averages of volume loss values (Hacim kaybı değerlerinin genel ortalamaları için normal dağılım ve kalıntı analiz grafikleri)

3.3. Friction Coefficient Results of Assessment (Sürtünme Katsayısı Değerlendirme Sonuçları)

Friction energy is generated between two surfaces in contact due to sliding speed and applied load. The accumulation of this energy in the rough areas of the surface leads to plastic deformations in the material [37]. The friction coefficient data obtained from the dry sliding wear process, conducted according to the Taguchi L_9 orthogonal test design for AISI 9310 steel, are presented in Table 8, along with the S/N ratios calculated using Equation (1). The arithmetic mean of the friction coefficient values was calculated to be 0.474 µ. The average S/N ratio for the friction coefficient values was determined to be 6.475 dB.

Table 8. The friction coefficient and S/N ratios for AISI 9310 steel, obtained from dry-sliding wear tests under various processing conditions (Çeşitli işleme koşulları altında kuru kayma aşınma testlerinden elde edilen AISI 9310 çeliği için sürtünme katsayısı ve S/N oranları)

Exp. No	Load (N)	Cryogenic Treatment	COF (µ)	Calculated S/N ratio (ni=1-18) (dB)
1	5	Untreated	0.498	6.055
2	5	SCT	0.480	6.375
3	5	DCT	0.464	6.670
4	10	Untreated	0.492	6.161
5	10	SCT	0.476	6.448
6	10	DCT	0.458	6.783
7	15	Untreated	0.487	6.249
8	15	SCT	0.470	6.558
9	15	DCT	0.448	6.974

Additionally, the influence of each control factor (load and cryogenic treatment) on the friction coefficient was evaluated using the S/N ratio response presented in Table 9. The study determined that the optimal friction coefficient corresponded to the lowest measured value. To identify the most effective S/N ratio for achieving a minimal friction coefficient while enhancing product quality and reducing costs, the 'smallest is best' performance criterion was employed [9,26]. Table 9 summarizes the average S/N ratios calculated for each level of the processing parameters used in the experiments. The highest S/N ratio indicated in the table corresponds to the minimum friction coefficient.

Table 9. Table of average S/N ratios computed for the friction coefficient values (Sürtünme katsayısı değerleri için hesaplanan ortalama S/N oranları tablosu)

Coefficient of Friction COF (µ)		
Level	**Load (N)**	**Cryogenic Treatment**
S/N Ratio		
1	6.367	6.155
2	6.464	6.460
3	**6.594**	**6.809**
Delta	0.227	0.654
Rank	2	1
Means		
1	0.4807	0.4923
2	0.4753	0.4753
3	**0.4683**	**0.4567**
Delta	0.0123	0.0357
Rank	2	1

The basic effect graphs, which consider the S/N ratios and average values in accordance with the “smallest is best” performance criterion for the friction coefficient of the processing parameters and levels, are presented in Fig. 8. Based on the graphical analysis of the S/N ratios, the optimal processing parameters for achieving the minimum friction coefficient were identified as a 15 N load combined with DCT cryogenic treatment. The highest friction coefficient value was observed in samples subjected to untreated conditions under a 5 N load. The friction coefficient graphs for samples with and without cryogenic treatment under various loads are shown in Fig. 8. The minimum friction coefficient (448 µ) was recorded in samples subjected to deep cryogenic treatment (DCT), while untreated samples exhibited the highest friction coefficient (492 µ). Compared to the untreated sample, a 5% improvement in friction coefficient values was observed in SCT-treated samples, and a 9% improvement was noted in DCT-treated samples. Literature studies indicate that cryogenic treatment significantly reduces the coefficient of friction (COF). One of the primary ways cryogenic treatment achieves this reduction is by increasing surface hardness. Myeong et al. [15] found that cryogenic treatment transforms retained austenite into martensite, thereby enhancing the material's hardness. This increase in hardness directly affects wear behavior by minimizing plastic deformation under load. A harder surface provides greater resistance to deformation, reducing material transfer between contacting surfaces during frictional interactions. This finding reinforces the broader literature and positions cryogenic treatment as a valuable method for extending the lifespan of components subjected to wear and friction.

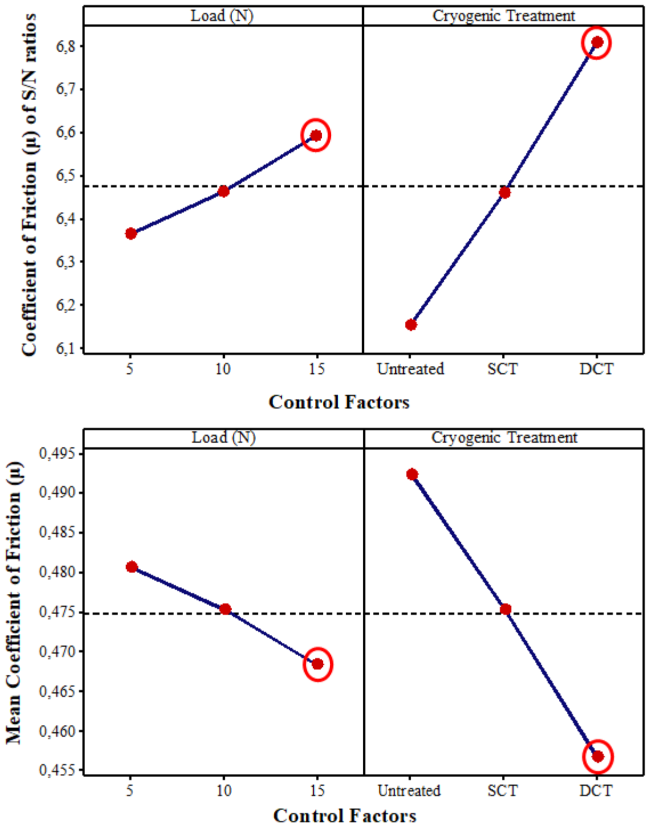

Figure 8. Graphs showing the S/N ratios of the friction coefficient values and the average of their measured values (Sürtünme katsayısı değerlerinin S/N oranlarını ve ölçülen değerlerin ortalamasını gösteren grafikler)

The friction coefficient values obtained from the wear tests performed under dry sliding conditions with a 15 N load and cryogenic treatment applications were measured. The friction coefficient results from these tests are presented in detail in Fig. 9. These findings clearly indicate that cryogenic treatment significantly reduces the COF values [3,5].

In the experimental analysis, the results and statistical investigations of the analysis of variance (ANOVA) used to evaluate the effects of the processing parameters on the friction coefficient are presented in Table 10. The ANOVA was conducted with a significance threshold of $p<0.05$ and a confidence level of 95%. The obtained data showed that the most significant contributions to the friction coefficient were due to cryogenic treatment with 85.75%, followed by load with 10.69%, as shown in the ANOVA analysis. These findings are in line with the results of the study conducted by Khun et al. on AISI D3 steel, who reported that cryogenic treatment was the most important factor affecting the material properties [35]. The regression analysis shows that the friction coefficient estimation model is statistically robust, with R-sq correlation coefficients of 99.42% and adjusted R-sq (adj) correlation coefficients of 97.07%, indicating that the parameters and their levels showed that there is a strong agreement between them.

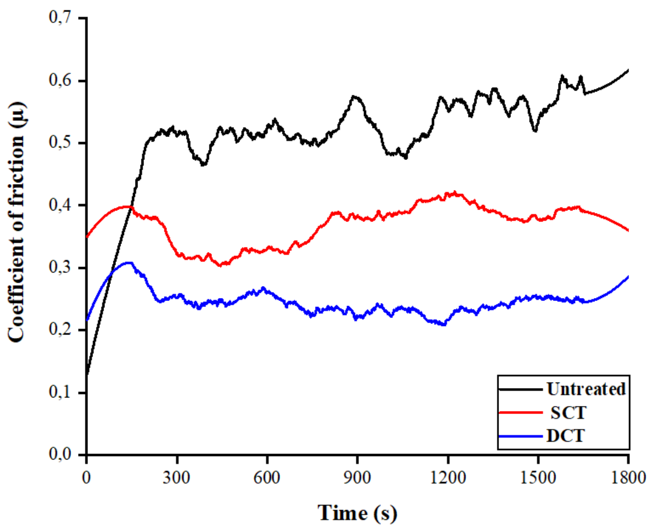

Figure 9. Coefficient of friction (COF) graph of samples under 15N load and various cryogenic treatments (15N yük ve çeşitli kriyojenik işlemler altındaki numunelerin sürtünme katsayısı (COF) grafiği)

Table 10. Results of the ANOVA analysis for friction coefficient values (Sürtünme katsayısı değerlerine ait ANOVA analizi sonuçları)

	Variance source	Degree of freedom (df)	Sum of squars (SS)	Mean square (MS)	F	P	Contribution (%)
Coefficient of Friction COF (µ)	Load (N)	2	0.00023	0.000115	36.89	0.003	10.69
	Cryogenic Treatment	2	0.00191	0.000955	306.89	0.000	88.75
	Error	4	0.000012	0.000003			0.56
	Total	8	0.002152				100.00
S=0,0017638 R-sq=99.42 % R-sq (adj)=97.07%							

The regression line and histogram graph used to examine the friction coefficient values are presented in Fig. 10. Most of the experimental data align well with the regression line, and the histogram exhibits characteristics of a normal distribution. In the distribution graphs, the data generally cluster around the zero line; however, some outliers were also detected. These outliers are likely caused by factors such as vibrations of the wear device and fluctuations in ambient temperature. The low number of outliers indicates that the data generally fit the regression model. Upon examining the results, it can be concluded that a reasonable fit is achieved between the input and output parameters determined for the friction coefficient. The concentration of data around a linear line further supports the meaningfulness and validity of the findings [36].

3.4. Grey Relationship Analysis Evaluation Results (Gri İlişki Analizi Değerlendirme Sonuçları)

In the first stage of the GRA process, the normalization of the data was performed using the "smallest is best" characteristic (Equation 2). Following this stage, the GRA normalization values were calculated, and the results are presented in Table 11 (see Equations 3 and 4). Using the computed normalization values, the Grey Relation Coefficients (GRC) were derived to represent the relationship between the target and actual experimental results (see Equation 5). In engineering designs, the varying importance levels of processing parameters necessitate assigning weight factors to each output parameter, which is crucial for effective GRA. However, many researchers use equal weights when determining the grey relational degrees of multiple responses. This practice can compromise the reliability of the results and may not adequately reflect the importance levels of various parameters.

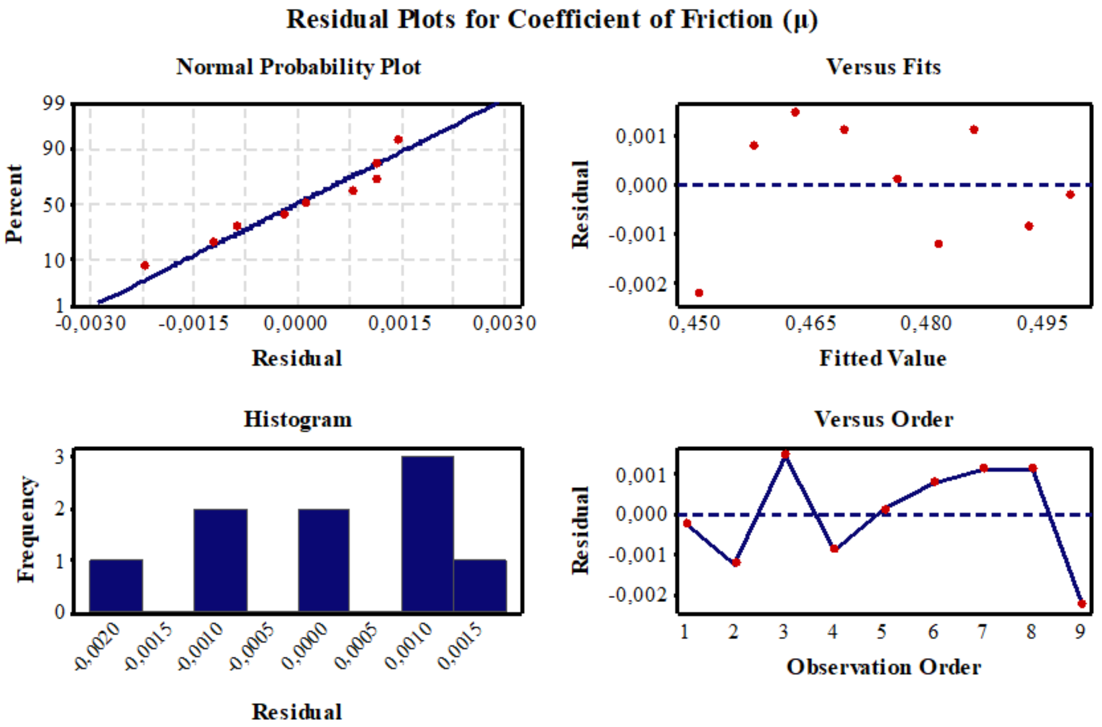

Figure 10. Normal distribution and residual analysis graphs for the grand averages of friction coefficient values (Sürtünme katsayısının genel ortalamaları için normal dağılım ve kalıntı analizi grafikleri)

Table 11 Normalization and coefficient values for volume loss and friction coefficient and grey relational degree values (Hacim kaybı ve sürtünme katsayısı için normalizasyon ve katsayı değerleri ile gri ilişkisel derece değerleri)

Exp. No	Grey Relational Normalized Values		Grey Relational Coefficient values		Grey Relation Grade Values	Order
	Coefficient of Friction COF (µ)	Volume loss (mm^3) x 10^{-3}	Coefficient of Friction COF (µ)	Volume loss (mm^3) x 10^{-3}		
1	0.000	0.564	0.333	0.534	0.434	7
2	0.360	0.727	0.439	0.647	0.543	4
3	**0.680**	**1.000**	**0.610**	**1.000**	**0.805**	**1**
4	0.120	0.273	0.362	0.407	0.385	8
5	0.440	0.491	0.472	0.495	0.483	6
6	0.800	0.855	0.714	0.775	0.745	3
7	0.220	0.000	0.391	0.333	0.362	9
8	0.560	0.364	0.532	0.440	0.486	5
9	1.000	0.673	1.000	0.604	0.802	2

The grey relationship degree reflects the closeness to the reference series. The alternative with the highest grey relationship degree represents the best option in the decision-making process [21]. As a result of the analyses, the highest grey relationship degree was obtained in experiment number 3 (Table 11). This finding indicates that experiment number 3 provided the closest performance to the optimum specifications and was therefore identified as the best alternative (Fig. 11). In the evaluation conducted using the GRA method, the most ideal processing parameters for achieving the lowest volume loss and friction coefficient values were determined to be under a 5 N load with DCT.

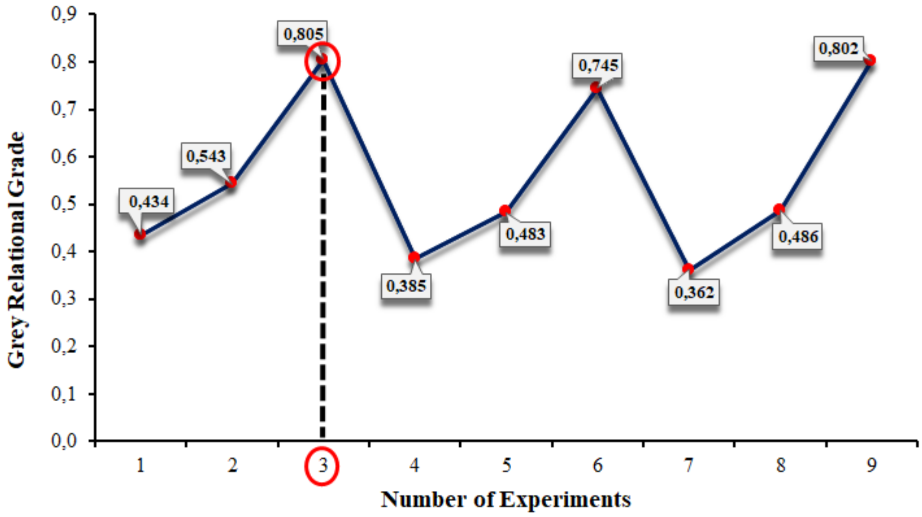

Figure 11. Grey relational grade plot for optimal performance (En iyi performans için gri ilişkisel derece grafiği)

The effect of each processing parameter on the degree of grey relationship was investigated using the mean values of the parameters. The average results of the calculated values for each level of the experimental conditions are presented in Table 12.

Table 12. Grey relational grade response analysis table (Gri ilişkisel derece yanıt analizi tablosu)

	Grey relational grade	
Level	**Load(N)**	**Cryogenic Treatment**
Means		
1	0.5938	0.3934
2	0.5376	0.5040
3	0.5499	0.7838
Delta	0.0562	0.3905
Rank	2	1

The relationship between the grey relational degree of the processing parameters and their levels is presented in Fig. 12, where the basic effect graph was obtained using average values in accordance with the "smallest is best" performance criterion. The graph indicates that the lowest volume loss and friction coefficient were achieved under 5 N load and DCT conditions, based on the grey relational degree values. Since the experiment was conducted using these processing parameters, no additional verification experiment was performed.

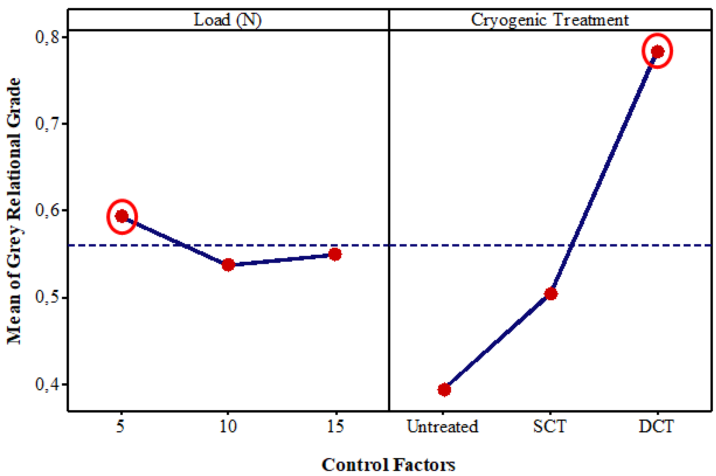

Figure 12. Average response graph for grey relational grade (Gri ilişkisel derece için ortalama yanıt grafiği)

Table 13 presents the results from the variance analysis (ANOVA) conducted to assess the impact of processing parameters on the Grey Relational Grade outcomes for AISI 9310 steel and their statistical significance. ANOVA was performed with a significance threshold of $p < 0,05$ and a confidence level of 95%. The obtained data revealed that the most significant contributing percentages affecting the grey relational grade in the ANOVA analysis were the cryogenic process at 97,10%, followed by the load at 2,09%. Regression analysis indicated that the model for predicting the friction coefficient is statistically robust, with a grey relational grade R-squared value of 99,19% and an adjusted R-squared (adj) value of 95,91%, demonstrating a strong alignment between the parameters and their levels.

Table 13. Results of the ANOVA analysis for grey relational degree values (Gri ilişkisel derece değerlerine ilişkin ANOVA analizi sonuçları)

	Variance source	Degree of freedom (df)	Sum of squares (SS)	Mean square (MS)	F	P	Contribution (%)
Grey relational grade	Load (N)	2	0.005238	0.002619	5.18	0.078	2.09
	Cryogenic Treatment	2	0.242995	0.121498	240.21	0.000	97.10
	Error	4	0.002023	0.000506			0.81
	Total	8	0.250257				100.00
S=0.0699681 R-sq=99.19 % R-sq (adj)=95.91%							

The regression line and histogram graph used to examine the grey relational degree values are presented in Fig. 13. Most of the experimental data align well with the regression line, and the histogram exhibits characteristics of a normal distribution. In the distribution graphs, the data generally cluster around the zero line; however, some outliers were also detected. These outliers are likely caused by factors such as vibrations of the wear device and fluctuations in ambient temperature. The low number of outliers indicates that the data generally fit the regression model. Upon examining the results, it can be concluded that a reasonable fit is achieved between the input and output parameters determined for the grey relational degree. The concentration of the data around a linear line further supports the meaningfulness and validity of the findings [36].

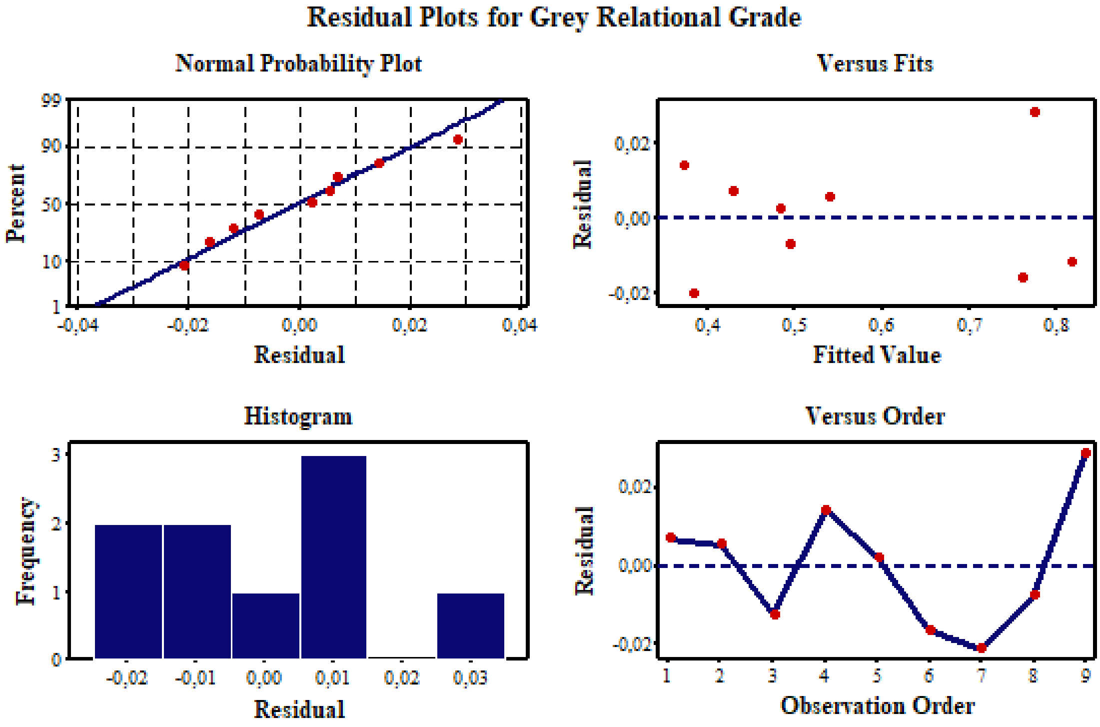

Figure 13. Normal distribution and residuals analysis plots for the overall means of grey relational grade (Gri ilişkisel derece genel ortalamaları için genel ortalamaları için normal dağılım ve kalıntı analizi grafikleri.)

4. CONCLUSIONS (SONUÇLAR)

AISI 9310 steel, known for its high corrosion resistance, performs well in corrosive environments but exhibits lower performance in wear-prone environments due to its mechanical properties. In this research, the friction coefficient and volume loss of cryogenically treated AISI 9310 steel were evaluated using the Grey Relational Analysis (GRA) method in conjunction with the Taguchi approach to determine the optimal parameter levels for dry sliding wear behavior. The results of experimental research are presented below:

- Cryogenic treatment significantly enhanced the material's hardness. Specifically, samples subjected to Shallow Cryogenic Treatment (SCT) exhibited a 12% increase in hardness compared to untreated samples, while those treated with Deep Cryogenic Treatment (DCT) showed a 30% improvement in hardness values.
- Dry sliding wear tests conducted under various load and cryogenic treatment conditions revealed that volume loss increased proportionally with the load, while cryogenic treatment resulted in reduced volume losses. Increasing the temperature during cryogenic treatment was associated with lower volume losses, which correlated with an increase in the material's hardness values.
- Compared to the untreated sample, volume loss values improved by 7% in SCT-treated samples and by 14% in DCT-treated samples.
- The most suitable parameters for minimizing the friction coefficient were identified under a 15 N load with DCT conditions. Cryogenic treatment contributed to the improvement of the friction coefficient by enhancing the surface hardness.
- Compared to the untreated sample, a 5% improvement in the friction coefficient values was observed in the SCT treated samples and a 9% improvement was observed in the DCT treated samples.
- As the load increased, volume loss rose by 16% (from 3400 mm^3 to 3950 mm^3), while the friction coefficient decreased by 11% (from 0.448 μ to 0.498 μ).
- According to the results of the variance analysis (ANOVA), the factor that most significantly affects volume loss is the cryogenic process, with a contribution percentage of 63,03%. Similarly, the factor that most influences the friction coefficient is also the cryogenic process, which has a contribution percentage of 88,75%.
- According to the regression analysis results, the model established for volume loss was statistically robust, R-sq was measured as 97.63% and R-sq (adj) as 95.26%. The model established for the friction coefficient was found to be R-sq as 99.42% and R-sq (adj) as 97.07%.
- In the evaluation conducted using the GRA method, the most ideal processing parameters for achieving the lowest volume loss and friction coefficient values were determined to be under a 5 N load with DCT.
- In the ANOVA analysis, the most significant contribution percentages affecting the gray relational degree were cryogenic treatment at 97.10%, followed by the load at 2.09%.

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