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Derin Kriyojenik İşlemin Kalıntı Gerilme ve Kalıntı Östenit Üzerindeki Etkisinin Araştırılması

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ÖZ

Sıfırın altındaki sıcaklıklarda malzemelerin bekletilmesi olarak bilinen kriyojenik işlem, son yıllarda metal ve metal olmayan malzemelerin özelliklerini iyileştirmek için uygulanan bir yöntemdir. Bu yöntem daha çok kalıp yapımında kullanılan takım çelikleri için uygulanmaktadır. Ülkemizde, birçok özel sektör kuruluşu tarafından öncelikle kalıp malzemeleri olmak üzere birçok üründe kriyojenik işlem kullanımı yaygınlaşmaya devam etmektedir. Savunma sanayiinden otomotiv sanayiine kadar birçok sektörde bu işlemin faydaları kabul görmüştür. Bu çalışmada, geleneksel ısıl işlem uygulanmış (CHT) ve 24 saat derin kriyojenik işlem uygulanmış (DCT-24) Sleipner soğuk iş takım çeliğinin makro sertlik, mikro sertlik ve mikroyapı özellikleri incelenerek, malzemenin mekanik özellikleri ve mikroyapısındaki değişimler tespit edilmiştir. Bununla birlikte, X-Işını Kırınımı (XRD) Yöntemi ile malzemelerdeki kalıntı gerilme ve kalıntı östenit miktarları ölçülerek, numuneler arasındaki fark belirlenmiştir. CHT ve DCT-24 numunelerinin makro sertliği sırasıyla 60,96 HRC ve 61,46 HRC olarak ölçülmüştür. Mikro sertlik değerleri de sırasıyla 734,26 HV ve 761,83 HV olarak ölçülmüştür. Derin kriyojenik işlem makro ve mikro sertliği sırasıyla 0,5 HRC ve 27,57 HV arttırmıştır. Kalıntı östenit miktarı derin kriyojenik işlemden sonra % 36 oranında düşmüştür. Eksenel ve çevresel kalıntı gerilme değerleri de sırasıyla % 48,84 ve % 36,52 oranında düşmüştür. Sonuç olarak derin kriyojenik işlem Sleipner soğuk iş takım çeliğinin sertliği arttırmış, mikroyapıyı homojenleştirmiş, kalıntı östenit ve kalıntı gerilme değerlerini düşürerek olumlu iyileşmeler sağlamıştır.

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Investigation of the Effect of Deep Cryogenic Process on Residual Stress and Residual Austenite

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ABSTRACT

The cryogenic treatment, known as holding materials at sub-zero temperatures, is a method used to improve the properties of metal and non-metallic materials in recent years. This method is mainly applied to tool steels used in mold making. In our country, the use of cryogenic processes continues to be widespread by many private sector organizations, primarily in mold materials. The benefits of this process have been recognized in many sectors, from the defense industry to the automotive industry. In this study, the macro hardness, micro hardness and microstructure properties of Sleipner cold work tool steel, which was applied traditional heat treatment and deep cryogenic process for 24 hours, were examined and the changes in the mechanical properties and microstructure of the material were determined. However, by measuring the residual stress and residual austenite amounts in the materials with the X-Ray Diffraction (XRD) Method, the difference between the samples was determined. The macro hardness of the CHT and DCT-24 samples was measured as 60.96 HRC and 61.46 HRC, respectively. Micro hardness values were

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also measured as 734.26 HV and 761.83 HV, respectively. Deep cryogenic treatment increased macro and microhardness by 0.5 HRC and 27.57 HV, respectively. The amount of residual austenite decreased by 36% after deep cryogenic treatment. Axial and circumferential residual stress values also decreased by 48.84% and 36.52%, respectively. As a result, deep cryogenic treatment increased the hardness of Sleipner cold work tool steel, homogenized the microstructure, reduced residual austenite and residual stress values and provided positive improvements.

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1. INTRODUCTION *(GİRİŞ)*

Tool steels are widely used in the processing and shaping of many tool materials such as metal, plastic and wood that we see around us. Tool steels corresponding to 8% of total steel production are divided into two as hot work and cold work tool steels. Tool steels with carbon content ranging from 0.3% to 2.5% and containing carbide-forming chromium, vanadium, molybdenum and tungsten as well as nickel and manganese as alloying elements are called cold work tool steels. Cold work tool steels have high initial hardness and this hardness value decreases rapidly when the working temperature exceeds 200 °C. For this reason, these steels are used for machining and non-chip forming processes of workpieces operating at temperatures of about 200 °C and below [1, 2]. Sleipner cold work tool steel is widely used in blanking and fine blanking, shearing, forming, coining, cold forging, cold extrusion, thread rolling, drawing and deep drawing, powder pressing molds where high wear resistance, high chipping resistance and high compressive strength are required. It is also very good steel for any type of surface treatment. They have good wear resistance, good chipping resistance, good hardness (> 63 HRC) after high temperature tempering, good dimensional stability in heat treatment, good machinability and grindability, and good surface treatment properties. These steels are widely used in precision cutting, cutting and forming, sintering and cold work applications.

In general, heat treatment is defined as controlled heating and cooling processes applied to steel to impart desired properties. Heat treatments applied to the steel are related to the transformation of its microstructure. The compositions and structures of the products the conversion affect the mechanical properties of the steel directly. Numerous thermal methods have been developed. The use of these methods depends on the type of steel and the expected properties such as hardness, strength, toughness, abrasion resistance [3, 4]. In recent years, cryogenic process, which is a complementary process to heat treatment, has emerged especially to improve the mechanical properties of tool steels. Cryogenic process is known as the process of keeping materials in liquid or gas environment in nitrogen up to -196°C [5-7]. This method is of great importance in terms of sustainable manufacturing since it does not have any negative impact on the environment. It has been subject to a lot of work due to its superior properties such as hardness increase, high wear, fatigue and impact resistance, homogeneous microstructure, low residual austenite ratio, low residual stress, high tensile strength, high dimensional accuracy [5, 6, 8-13]. Demir and Toktaş investigated the effect of deep cryogenic treatment on the microstructure, hardness, residual austenite and residual stresses of AISI D2 steel. Results showed that cryogenic treatment reduced the residual surface stresses in comparison to the conventional heat treatment. There was no significant difference in hardness values and retained austenite contents. However, more dense and homogeneous carbide precipitation was observed mainly for the cryo-treated sample for 24 h [8]. Yan and Li investigated the effect of subzero processing conditions on wear resistance, mechanical behavior and microstructure of W9Mo3Cr4V high speed steel. The samples were cryogenically treated at different holding temperatures (-80 °C, -120 °C, -160 °C and -196 °C). Hardness and wear resistance increased and microstructure improved after cryogenic treatment for all samples. The increase in wear resistance and hardness has been attributed to the conversion of residual austenite to martensite and secondary carbide precipitation by cryogenic treatment [14]. Altan Özbek et al. investigated the effects of cryogenic treatment on the mechanical properties of AISI H11 hot work tool steel. In the study, it was determined that deep cryogenic treatment greatly reduced the residual austenite phase. It has been reported that an increase in the hardness and wear resistance of the material is achieved with the cryogenic treatment [15].

When the studies in the literature are examined, it is seen that the cryogenic process is applied on many materials and positive results are obtained. However, there are no studies investigating the effects of cryogenic treatment on the mechanical properties and residual stress of Sleipner cold work tool steel. In this study, macro hardness, micro hardness and microstructural properties of Sleipner cold work tool steel, which was subjected to deep cryogenic treatment in a 24-hour holding period, were examined and the changes in the mechanical properties and microstructure of the material were investigated. However, the residual stress and residual austenite amounts in the materials were measured by the X-Ray Diffraction (XRD) Method.

2. MATERIAL AND METHOD *(MATERYAL VE METOT)*

In this study, Sleipner cold work tool steel patented by UDDEHOLM Company was used. Uddeholm Sleipner is a high alloyed tool steel with a very specific profile. Chemical components of Sleipner cold work tool steel are given in Table 1.

The application of deep cryogenic process to the test samples was carried out with the MMD Criyo 125 brand cryogenic process device in Duzce University Mechanical Engineering Department. The image of the device in Figure 1, the specifications are given in Table 3.

Test samples will be divided into two groups in the study. The samples in the first group were subjected to conventional heat treatment to 60-62 HRC hardness. The samples in the second group were subjected to deep cryogenic treatment (24 hours at -180 °C) and then tempering process (2 hours at 200 °C) after conventional heat treatment. Classification of test samples and heat treatments to be applied are given in Table 2.

To determine the effects of deep cryogenic process on the hardness of Sleipner steel, macro hardness and micro hardness measurements were carried out. In macro and micro hardness measurements, the measurement result reflects the average of at least 10 hardness measurements. Macro hardness measurements were carried out using Time TH 300 macro hardness device and Rockwell C (HRC) hardness measurement method. Micro hardness measurements were carried out with DUROLINE-M brand micro hardness device by applying a 1000 g load for 30 seconds with a diamond pyramid tip.

Table 1. Chemical components of Sleipner steel (%) *(Sleipner çeliğinin kimyasal bileşimi (%))*

C	Si	Mn	Cr	Mo	V
0.90	0.90	0.50	7.8	2.5	0.50

Figure 1. Cryogenic process device *(Kriyojenik işlem cihazı)*

Table 2. Application of traditional heat treatment and deep cryogenic process *(Geleneksel ısıl işlem ve derin kriyojenik işlem uygulaması)*

Sample Group	Sample Name	Heat Treatment to be Applied
1	CHT	Conventional heat treatment
2	DCT-24	Conventional heat treatment Deep cryogenic process (24 hours) Tempering process

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Table 3. Technical specifications of the cryogenic process device *(Kriyojenik işlem cihazının teknik özellikleri)*

Internal dimensions	60cm L x 50cm W x 50cm D
Inner Chamber	304 stainless steels
Insulation	15cm multilayer insulation
Capacity	<100kg
Control	Programmable PLC control with touch screen
Temperature sensor	PT100
LN2 source	Can be used with medium or high pressure liquid nitrogen containers
LN2 Transfer Connection	180cm flexible, insulated cryogenic liquid transfer hose
LN2 Dosing	Solenoid valve
Process temperature	< -190 °C
Temperature distribution	± 2 °C
Cooling-Heating Speed	≥ 10 °C / min (depending on the material loading capacity)

Samples of 10 mm diameter and height were prepared to be used in microstructural studies. These samples were sanded with 120, 240, 600, 800 and 1200 grit SiC abrasives, respectively, after conventional heat treatment and deep cryogenic treatment. It was then etched with Nital solution and examined under an optical microscope.

Residual stresses in both circumferential and axial directions formed after heat treatment and deep cryogenic treatment in Sleipner cold work tool steel samples were measured by X-Ray diffraction technique using the $\sin^2$Psi method. Residual austenite and residual stress measurements were performed on the SEIFERT Analytical X-ray MZ VI brand X-ray diffraction device.

3. RESULTS AND DISCUSSION (*SONUÇLAR VE TARTIŞMA*)

There are four main metallurgical cases that are claimed to explain the changes in the properties of materials that are cryogenically treated. These; transformation of residual austenite to martensite, the formation of eta carbides, the precipitation of fine carbides and a homogeneous microstructure [16, 17]. One of these situations, the transformation of residual austenite to martensite plays an important role on the mechanical properties of materials. The combination of soft residual austenite and hard martensite in the structure of steel is absolutely undesirable. Because the residual austenite in the structure negatively affects all the mechanical properties of the steel, especially the hardness. In addition, the presence of residual austenite decreases residual stresses, thus reducing fatigue resistance [18]. Therefore, the transformation of residual austenite to martensite is required. This transformation can be accomplished by the cryogenic process in which almost all of the residual austenite is converted to martensite or by tempering process where martensite transformation is achieved at a lower rate [5, 15]. Figure 2 shows the change in the residual austenite volume ratios of the CHT and DCT-24 samples.

As seen in Figure 2, the residual austenite volume ratio of the conventional heat treated DCT sample was measured as 2.5%. The residual austenite volume ratio of DCT-24 sample, which was applied deep cryogenic treatment for 24 hours, was found to be 1.6%. After deep cryogenic processing, the residual austenite volume ratio decreased by 36%. This result once again demonstrated that the deep cryogenic process significantly improved the residual austenite content. In studies in the literature, it has been repeated many times that the deep cryogenic process reduces the residual austenite volume ratio [19-21]. In the study conducted by Das et al. in 2007, it was stated that the residual austenite volume ratio of AISI D2 cold work tool steel decreased from 9.8% to below 2% after deep cryogenic processing. This positive improvement was attributed to the Mf (Martensite finish) temperature of AISI D2 steel below room temperature [22]. In another study, it was emphasized that 30-40% decreases were detected in the residual austenite volume ratio after the cryogenic process applied to tool steel. This result was associated with the increase in the residual austenite content in the microstructure after quenching and the decrease in the Ms (Martensite starting) temperature [23].

Residual stresses are defined as the stresses remaining in the part because various manufacturing methods, heat treatment processes or application of non-homogeneous deformation to the materials. Since it remains in the material after the production of the part, the loads and forces to be applied externally during operation affect the workpiece together with these stresses [5]. Therefore, residual or residual stresses directly affect the working life of the produced parts [24]. Residual stresses are classified into two types as compression and tension. Compressive stress delays crack initiation and crack growth. This delay makes residual compressive stresses very important, especially on fatigue life and corrosion resistance. On the contrary, the tensile residual stress decreases the mechanical performance of the materials, so it is not a very desirable situation for materials [5, 25]. In Figure 3, the residual stress values in axial and circumferential directions of the CHT and DCT-24 samples were given.

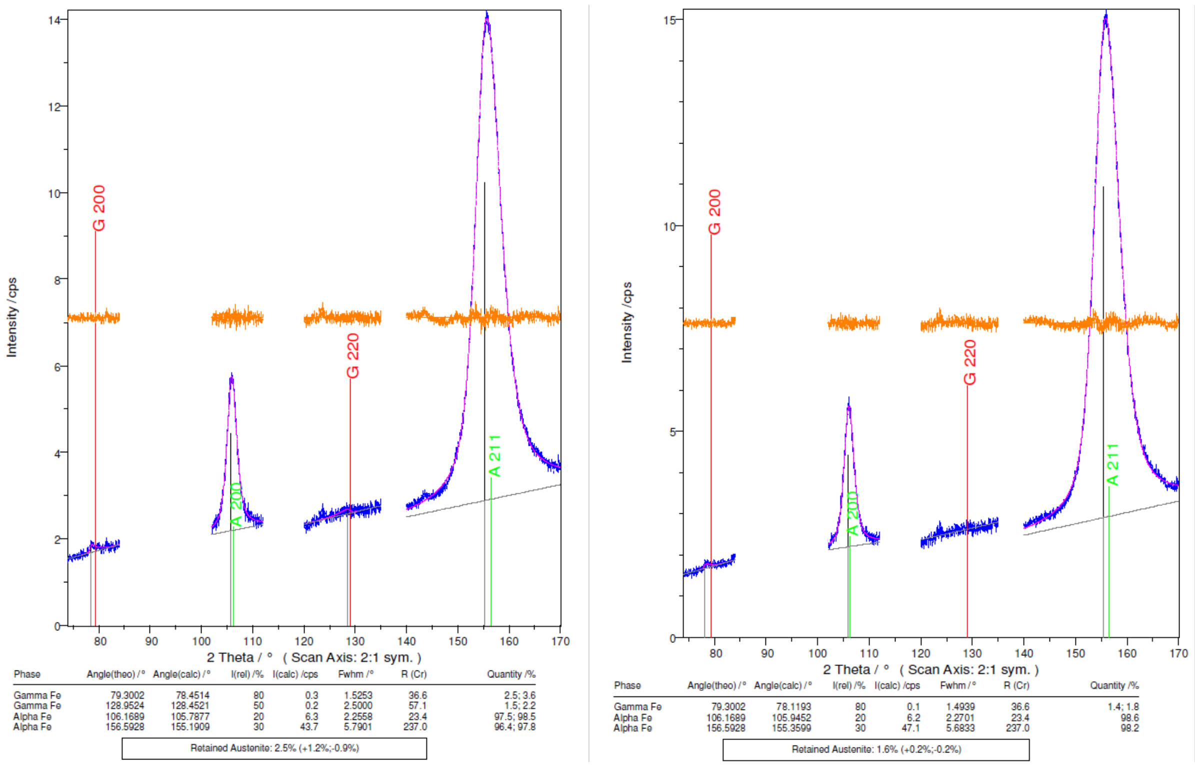

Figure 2. Residual austenite volume ratio changes of samples (a) CHT and (b) DCT-24 *(Numunelerin kalıntı östenit hacim oranlarının değişimi (a) CHT ve (b) DCT-24)*

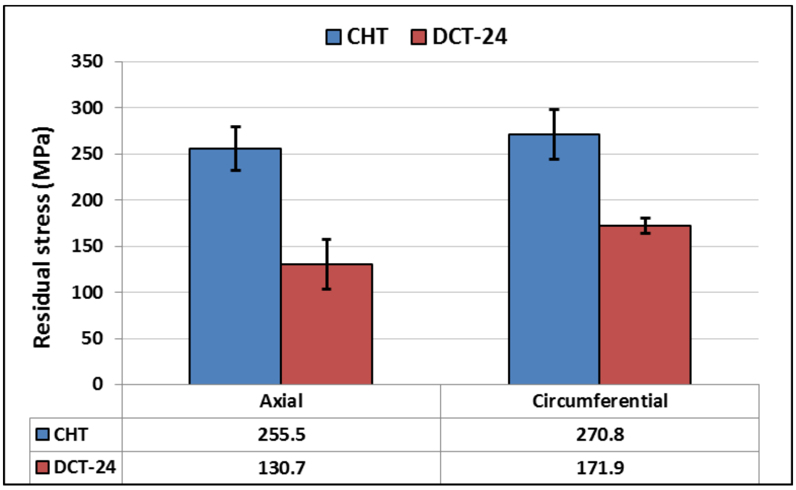

Figure 3. Residual stress changes of CHT and DCT-24 samples *(CHT ve DCT-24 numunlerinin kalıntı gerilmelerindeki değişim)*

While the axial residual stress value of the CHT sample was 255.5 MPa, the axial residual stress value of the DCT-24 sample was measured as 130.7 MPa. Deep cryogenic process reduced the axial residual stress value to 124.8 MPa, resulting in an improvement of 48.84%. It was found to be 270.8

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MPa and 171.9 MPa for the CHT and DCT-24 samples, respectively. Deep cryogenic process has been positively effective in environmental residual stress values as well as in axial stresses. After the deep cryogenic process, environmental residual stress values decreased by 98.9 MPa, an improvement of 36.52% was obtained.

Microstructure images were taken under metal microscope in order to determine the changes in microstructure caused by deep cryogenic process applied to Sleipner cold work tool steel with 24 hour holding time compared to conventional heat treated tool steel. The purpose of this analysis is to explain the improvements in mechanical properties after deep cryogenic processing. Figure 4 shows the microstructure images of the CHT and DCT-24 samples.

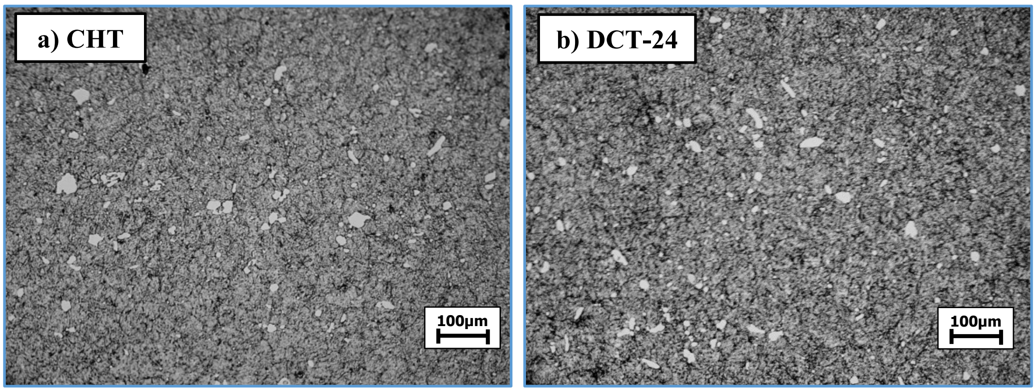

Figure 4. Microstructural images of (a) CHT, (b) DCT-24 samples
(Mikroyapı fotoğrafları (a) CHT, (b) DCT-24 numuneleri)

In Figure 4, while the CHT sample showed an inhomogeneous carbide distribution, the DCT-24 sample showed a more homogeneous carbide distribution than the CHT sample. However, after 24 hours of cryogenic treatment and subsequent tempering, it is seen that the carbide sizes in the microstructure of the samples decrease and a more uniform carbide distribution is realized.

Figure 5 shows the macro hardness values of the CHT and DCT-24 samples. The average macro hardness of the CHT and DCT-24 samples was measured as 60.96 HRC and 61.46 HRC, respectively. It was observed that the macro hardness value increased by 0.5 HRC after 24 hours of deep cryogenic treatment applied to Sleipner cold work tool steel. This increase in macro hardness was attributed to the formation of a more brittle structure as a result of the transformation of the austenite phase, which has a soft structure in the internal structure of Sleipner cold work tool steel, into the martensite phase with a hard structure, together with the deep cryogenic process [5, 26].

Figure 6 shows the micro hardness values of conventional heat treated CHT and 24 hours deep cryogenic DCT-24 samples. The average micro hardness of the CHT and DCT-24 samples were measured as 734.26 HV and 761.83 HV, respectively. It was observed that the macro hardness value increased by 27.57 HV after 24 hours of deep cryogenic treatment applied to Sleipner cold work tool steel. As with macro hardness, this improvement in micro hardness has been associated with the formation of a more brittle structure as a result of the transformation of the austenite phase, which has a soft structure in the internal structure of Sleipner cold work tool steel, into the martensite phase with a hard structure with deep cryogenic process [27].

If a general evaluation is made for all output parameters, it is seen that the cryogenic process increases the macro and micro hardness, decreases the residual austenite volume ratio, makes the microstructure more homogeneous and reduces the residual stresses on the surface of the material. These improvements obtained in the literature studies have been renewed once again with this study. In addition, it has been shown that the mechanical and metallurgical properties of Sleipner cold work tool steel have improved after deep cryogenic processing, as is the case with many tool steels.

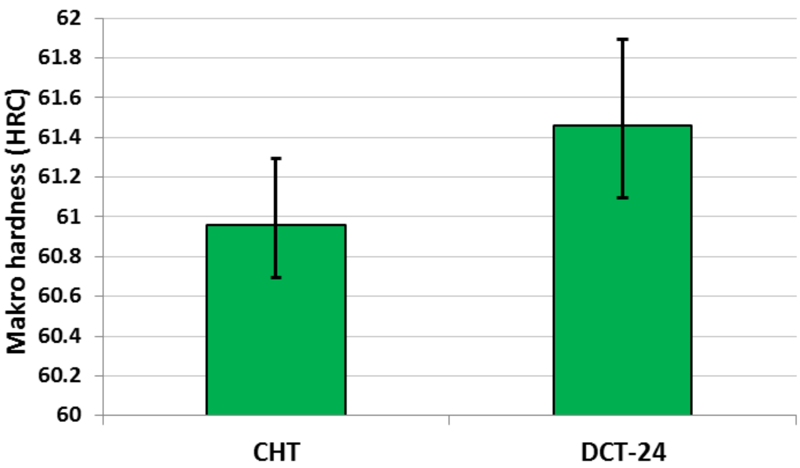

Figure 5. Macro hardness changes of CHT and DCT-24 samples *(CHT ve DCT-24 numunelerin makro sertliklerindeki değişim)*

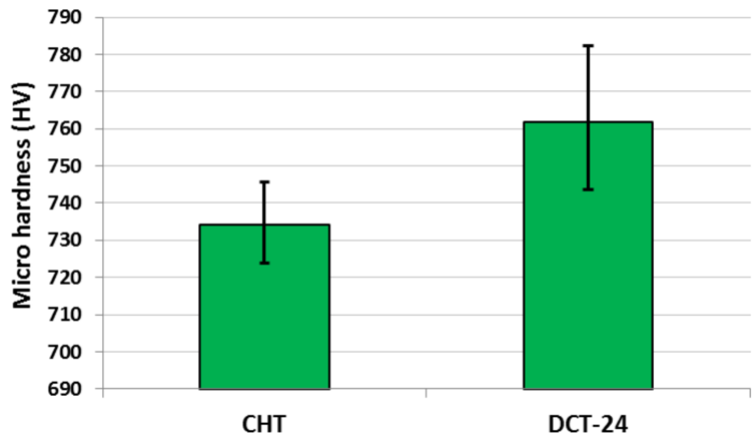

Figure 6. Micro hardness changes of CHT and DCT-24 samples *(CHT ve DCT-24 numunelerin mikro sertliklerindeki değişim)*

4. CONCLUSIONS (*SONUÇLAR*)

In this study, the effects of deep cryogenic process (-180 °C) applied for 24 hours on the microstructure, residual austenite volume ratio, residual stress values and mechanical properties of Sleipner cold work tool steel were investigated. At the end of this study, the following results were obtained.

- While the residual austenite volume ratio of the CHT sample was 2.5%, this ratio dropped to 1.6% in the DCT-24 sample after deep cryogenic processing. After deep cryogenic processing, the residual austenite volume ratio decreased in accordance with the studies in the literature.
- Residual stress values also decreased negatively after deep cryogenic processing.
- After conventional heat treatment, the hardness of 60.96 HRC increased to 61.46 HRC with 24 hours of deep cryogenic process and subsequent tempering.
- While the macro hardness of the CHT sample was 734.26 HV, the hardness of the DCT-24 sample was determined as 761.83 HV. Deep cryogenic processing resulted in an increase in microhardness of about 30 HV.
- Deep cryogenic process according to microstructure analysis made the inner structure of Sleipner cold work tool steel more homogeneous.

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As a result, after Sleipner cold work tool steel was subjected to a 24-hour deep cryogenic process and subsequent tempering, it gained improvements in its mechanical and metallurgical properties.

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CONFLICT OF INTEREST STATEMENT ***(ÇIKAR ÇATIŞMASI BİLDİRİMİ)***

No potential conflict of interest was reported by the authors.

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