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Investigation of the Effect of Tempering and Cryogenic Treatment on Mechanical Properties of Boron Steels

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Article Info:

Graphical/Tabular Abstract (Grafik Özet)

In this study, boron steels are shaped by forging process and the differences created by cryogenic and tempering processes in the structure are mentioned))

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Keywords

Boron steels, Cryogenic treatment, Tempering

Makale Bilgisi

Araştırma makalesi Başvuru:27/03/2023 Düzeltme: 06/04/2023 Kabul: 13/04/2023

Anahtar Kelimeler

Borlu çelikler Kriyojenik işlem Temperleme



Figure A: Forming and heat treatment processes /Şekil A: Dövme ve ısıl işlem süreçleri

Highlights (Önemli noktalar)

- > Heat treatment of boron steels
- > Shallow cryogenic temperature
- Improvement of mechanical properties

Aim (Amaç): The aim of this study is to shape boron steels by forging method and to investigate the effect of cryogenic and tempering processes./ Bu çalışmanın amacı, borlu çelikleri dövme yöntemi ile şekil vermek ve kriyojenik ve temperleme işlemlerinin etkisini araştırmaktır.

Orginality (Özgünlük): In this study, a new heat treatment route was created for boron steel produced by forging method by using temper and cryogenic treatment together. / Bu çalışmada, temperleme ve kriyojenik işlemin bir arada kullanılarak dövme yöntemiyle üretilen bor çeliği için yeni bir ısıl işlem rotası oluşturulmuştur.

Results (Bulgular): With this article, it was found to be mechanically better when tempering and cryogenic treatment were used together. Wear resistance is improved by tempering and cryogenic treatment./ Bu makale ile temperleme ve kriyojenik işlemin birlikte kullanılmasının mekanik olarak daha iyi olduğu görülmüştür. Aşınma direnci temperleme ve kriyojenik işlemle iyileştirilir.

Conclusion (Sonuç): It was determined that the abrasion resistance of the tempered and cryogenically treated 33B+TK samples increased by approximately 55% compared to the raw material./ Temperlenmiş ve kriyojenik işlem görmüş 33B+TK numunelerinin aşınma direncinin ham maddeye göre yaklaşık %55 arttığı belirlenmiştir.



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Abstract

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Boron steels, Cryogenic treatment, Tempering Boron steels are a group of steels that stand out with their high wear resistance and hardenability. In this study, 33MnCrB5-2 boron steel was shaped by applying hot forging process. After the hot forging process, the microstructure examinations and mechanical tests of the materials were carried out. A group of materials was shallow cryogenically treated at -80 °C for 2 hours. Then, a different group of materials was austenitized at 890 °C and quenched, and then tempered at 400 °C for 90 minutes. In the last group of materials, after tempering heat treatment, cryogenic treatment was applied at -80 °C for 2 hours. Hardness and abrasion tests were carried out on the samples that were subjected to cryogenic treatment and tempering heat treatment. Microstructure analyzes were examined with scanning electron microscope (SEM) and optical microscope. Element distributions from different regions in the microstructure were analyzed with energy-dispersive X-ray spectrometry (EDS). The crystallite size of the materials was calculated by X-ray diffraction. The results showed that the wear resistance of cryogenic treat samples after tempering improved by 55% compared to the raw sample. The hardness value was measured as 613 HV1 by cryogenic treatment after tempering.

Borlu Çeliklere Uygulanan Temperleme ve Kriyojenik İşlemin Mekanik Özelliklere Etkisinin İncelenmesi

Makale Bilgisi

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Öz

Borlu çelikler, aşınma direnci yüksek ve sertleşebilme kabiliyeti ile ön plana çıkmış bir çelik grubudur. Bu çalışma da 33MnCrB5-2 borlu çeliğe sıcak dövme işlemi uygulanarak şekillendirilmiştir. Sıcak dövme işlemi sonrası malzemelerin mikroyapı incelemeleri ve mekanik testleri yapılmıştır. Bir grup malzemeye -80 °C de 2 saat kriyojenik işlem uygulanmıştır. Ardından farklı bir grup malzeme 890 °C östenitlenip su verilmiş ardından 400 °C'de 90 dakika temperleme işlemi uygulanmıştır. Son grup malzeme de temperleme ısıl işlemi sonrası -80 °C de 2 saat kriyojenik işlem uygulanmıştır. Kriyojenik işlem ve temperleme ısıl işlemi uygulanan numunelerin mekanik olarak sertlik ve aşınma testi yapılmıştır. Mikroyapı analizleri tarama elektron mikroskobu (SEM) ve optik mikroskop yardımı ile incelenmiştir. Enerji-dağıtıcı X-ışını spektrometresi (EDS) ile ısıl işlemler sonrası mikroyapıdaki farklı bölgelerden element dağılımları analiz edilmiştir. X-ışını difraksiyonu ile malzemelerin tane boyutları hesaplanmıştır. Sonuçlar temperleme sonrası kriyojenik işlem uygulanan numunelerin sertlik değerinin ve aşınma dayanımının diğer numunelere kıyasla daha yüksek değerler verdiğini göstermiştir.

1. INTRODUCTION (GİRİŞ)

Boron micro alloy steels offer better hardenability at a lower cost. Trace amounts of boron allow the production of high-strength parts with a uniform martensite microstructure [1]. It prevents the formation of micro-structures such as boron, ferritin, and perlite, which are separated at the austenite grain boundaries. It has been reported that the positive effect of boron on hardenability is due to the separation of boron atoms at the austenite grain boundaries. [2-6]. The formation of these precipitates consumes boron atoms that dissociate at the austenite grain boundaries and the boron addition loses its beneficial effect on hardenability [7,8]. Boron steels form a group of special steels and although they offer good hardenability, the boron content should be limited as contents higher than 30 ppm impair toughness [9-11]. The most common applications of boron steels require a tempered martensitic microstructure because of the better properties tempered martensite provides [12,13]. Therefore, annealed boron steel is suitable for applications in manufacturing automotive structural parts and allows the production of lighter components in vehicles that contribute to weight reduction [14]. Currently, the most important requirement in the automotive industry is to design vehicles with reduced weight and high safety to meet the needs for a wide range of products. The critical goals for a vehicle designer are cost savings and reduction of gas emissions. For this purpose, the cross-sectional areas of the vehicle parts have been reduced and thinner parts made of high-strength steel have been used. Among these steels, the most popular are dual-phase steels, transformation TRIP steels, boron steels, martensitic steels and more recently developed (TWIP) steels [15-17]. Boron manganese hot rolled alloy steels, also called boron steels or MnB steels, are popular due to their excellent mechanical properties [18]. During the cryogenic treatment, the retained austenite phase transforms into a hard martensite phase resulting in an improvement in both wear resistance and dimensional accuracy [19]. It has been stated in different studies that the cryogenic process can be performed in two ways. It is divided into shallow and deep cryogenic treatment. Shallow cryoprocessing is performed between -100 and -40°C, while deep cryo-processing is performed below -100°C. It is thus done to achieve the common goal of an improvement in wear resistance by reducing retained austenite and strengthening atomic bonds. It has also been reported that tempering after cryogenic treatment can provide greater wear resistance due to the formation of fine carbides on the tempered martensite matrix [20,21].

As summarized above, numerous studies have been conducted on the tempering conditions of 33MnCrB5-2 boron steel. However, the researchers did not perform experimental studies combining the cryogenic treatment and tempering parameters. Considering the microstructural evolution according to these heat treatments, they did not reveal the wear behavior of these steels. For the first time, the effects of shallow cryogenic, tempering, tempering + shallow cryogenically treated 33MnCrB5-2 boron steel were investigated in detail.

2.MATERIALS AND METHODS (MATERYAL VE METOD)

The chemical composition of the commercially 33MnCrB5-2 material available used in experimental studies is given in Table 1. This material is shaped by forging, which is a plastic forming process. The samples were first cut with a diameter of 55mm and a length of 250mm and used in a 60mm diameter coil in a 600 kW induction device, and heating with inductance was applied. After the heating process, the forging process was carried out at 1150 °C with the help of a 6300 ton air hammer. The forging process temperature was controlled with a laser thermometer. After the thickness of the materials was reduced to 12 mm, the materials were left to cool. This material is coded as 33B. In order to remove scales on the surfaces of the forged steels, 30 minutes of sandblasting was applied with steel granules, which are S390 material. In order to see the invisible cracks and damages on the surface of the materials during the forging process, the inspection process was carried out with a TMM brand MP 800-2AC model crack control device. Forged materials are cut by wire erosion method according to ASTM G-99 standard. After the cutting process, a group of materials was cryogenically treated at -80 °C for 2 hours. These materials are coded as 33B+K. Other group materials were austenitized at 890 °C and quenched after forging, and then tempered at 400 °C for 90 minutes. These materials are coded as 33B+T. After tempering, some materials were cryogenically treated at -80 °C for 2 hours and coded as 33B+TK.

Table 1. Chemical composition of materials (Malzemelerin kimyasal bileşimi)

Chemical Composition (%)								
С	Mn	Si	Cr	Al	Р	S	В	Fe
0.32	1.45	0.19	0.47	0.032	0.007	0.004	0.0033	Balance

Microstructures of the samples, which were subjected to classical metallographic processing for microstructure analysis, were examined with JEOL JSM-6060LV Scanning Electron Microscope (SEM) and Leica DMI5000 model optical microscope. Hardness tests were measured with Qness brand macro hardness tester. The diffraction planes of the samples and the distribution of carbides were determined with the Bruker brand XRD device. At the same time, crystallite size calculations were made with XRD analysis. UTS brand abrasion tester was used to determine weight losses at 1000 m distance under 25N load.

3.RESULTS (BULGULAR)

In Figure 1, optical microstructure images of the samples are given. It belongs to the 33MnCrB5-2 material after forging, which we encoded as 33B in Figure 1 (a). This structure consists of ferrite and perlite. Figure 1 (b) shows the 33B+K specimen, which was cryogenically treated at -80 °C after forging. Its microstructure consists of ferrite and perlite. However, with the effect of the cryogenic

process, it is seen that the ferrite networks at the grain boundaries become coarser and cause an increase in grain size. Here, the ferrite and pearlite bands are probably due to the segregation of the alloy during initial solidification, when alloying elements are ejected into interdendritic regions and then become prominent after hot forging [22]. Preeutectoid ferrite appears as a fine, continuous network in previous austenite grains, and the volume fraction of ferrite increases with increasing cooling rate. These effects are generally related to the effect of the cooling rate on the ferrites coalescence and growth rates [23]. The increase in cooling rates lowers the transformation temperature, and the lower temperature perlite form results in finer pearlite grains. In Figure 1 (c), the tempered sample, which we coded as 33B+T, can be seen. The dominant microstructure here is tempered martensite. The microstructure obtained when the samples are continuously cooled in water is martensite, which is not a desirable phase because of its detrimental effect on toughness [24]. For this reason, tempering heat treatment was applied to these materials after quenching.



Figure 1. Optical microscope images of the samples (a) 33B (b) 33B+K (c) 33+T (d) 33B+TK (*Numunelerin optic mikroskop görüntüleri*)

Figure 2 shows the SEM microstructures of the samples. Compared to an optical microscope, the phases are seen more distinctly as it can be viewed at large magnifications. Samples 33B and 33B+K are shown in Figure 2 (a-b). It is seen that the cryogenic process opens the gap between the perlite lamellae. In other words, it caused the formation of a coarser perlite structure. Also, the grain size increased. It was seen in Figure 4 that there was a decrease in hardness in the samples that underwent cryogenic treatment after forging. This supports why the hardness decreases with the hall petch effect [25]. It has been observed that the pre-eutectoid ferrite networks expand by cryogenic treatment and there are separations in the ferrite

networks in some regions. Figure 2 (c-d) 33B+T and 33B+TK samples are shown. It has been observed that carbides are frequently formed in a spherical form (yellow areas) by cryogenic treatment after tempering. As it is known, the structure tends to decrease its free energy. Since the process is done below 0 °C with the cryogenic process, the entropy decreases with the disorder. Since the disorder is reduced, it is thought that carbides are formed in a spherical form in order to provide the internal balance of the structure and decrease the free energy thermodynamically [26].



Figure 2. SEM microstructure images of the samples (a) 33B (b) 33B+K (c) 33+T (d) 33B+TK (*Numunelerin SEM mikroyapı görüntüleri*)

Figure 3 shows the EDS analyzes taken over the SEM microstructures. It was observed that the Cr element was higher at the 1st point in the microstructure of the 33Mn+K sample in Figure 3 (a). Cr is in the group of ferrite-forming elements. Since these regions are known as ferrite networks, the Cr element is therefore thought to be in higher

proportion. Figure 3 (b) shows the spherical carbides formed in the 33Mn+TK sample. In the EDS analyzes in these regions, 15% B and 3% C elements were detected. Clustering of Boron element by cryogenic treatment is seen both in EDS analysis and microstructures.



Figure 3. EDS analysis of cryogenic treated samples (a) 33B+K (b)33B+TK (Kriyojenik işlemli numunelerin EDS analizleri)

The macro hardness values taken from each sample according to HV1 are shown in Figure 4. The average values of the hardness values taken from 10 different points are given in the graph. The lowest hardness value was observed in the sample that underwent cryogenic treatment after forging. This is thought to be due to the increase in grain size as seen from the microstructures. The highest hardness was obtained in 33B+TK specimen as 613 HV1. At the same time, it is thought that the spherical carbides in the 33B+TK sample may increase the hardness. The hardness values of the forged and tempered samples are similar to the literature [27,29,30].



Figure 4. Samples HV1 hardness values (*Numunelerin HV1 sertlik değerleri*) Table 2 shows the weight losses in the material after the wear test. The least weight loss was measured in the 33B+TK sample. Tempering and cryogenic treatment (33B+TK) were found to improve wear resistance by 55% compared to sample 33B. This is thought to be due to the cryogenic process, which reduces the amount of retained austenite and strengthens the interatomic bonds.

Samples	Weight Loss (mg) 1000 meters
33B	0.0028
33B+K	0.0031
33B+T	0.0022
33B+TK	0.0013

 Table 2. Weight losses in samples after wear test/ (Aşınma testi sonrası numunelerin ağırlık kayıpları)

XRD graphics of the samples with X-ray analysis are shown in Figure 5. It is seen that all samples are in the (110) plane of the main peak. The crystallite size of all samples was calculated with the FWHM value obtained by XRD analysis. These calculations were made using the Williamson–Hall equation (eq.1) [28].

 β *cos θ =k* λ /D+4 ϵ sin θ (1) where; β = FWHM k= 0.94 λ =1,54 Å (Cu-K α) D = refers to the crystallite size As a result of the calculations, the crystallite size of the 33B sample was found to be 100.43 Å and the 33B+K sample was 170.12 Å. Microstructural analyzes also support this situation. These calculations proved that the cryogenic treatment caused an increase in grain size. At the same time, it was seen in Figure 4 that the hardness value was the lowest in the 33B+K sample when the cryogenic treatment was applied alone. This situation directly relates to the Hall Petch effect [25].



Figure 5. XRD pattern of samples(*Numunlerin XRD desenleri*)

4.CONCLUSIONS (SONUÇLAR)

In this study, after the 33MnCrB5-2 material in the boron steel group was shaped by forging method, some of the samples were cryogenically treated and the other part was cryogenically treated after quenching and tempering, and how it affected the microstructure and mechanical properties were investigated. The following results were obtained from the experimental studies:

- The hardness value was measured as 293 HV1 in the sample that underwent cryogenic treatment after forging at least.
- It has been determined that the abrasion resistance of 33B+TK samples, which have been tempered and cryogenically treated, has increased by approximately 55% compared to the raw material.
- By XRD analysis, the largest crystal size was found to be 170.12 Å in the 33B+K sample.

• It was observed that the precipitate forms changed in the microstructure of the 33B+TK sample that was tempered and cryogenically treated.

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DECLARATION OF ETHICAL STANDARDS (ETİK STANDARTLARIN BEYANI)

The author of this article declares that the materials and methods they use in their work do not require ethical committee approval and/or legal-specific permission. Bu makalenin yazarı çalışmalarında kullandıkları materyal ve yöntemlerin etik kurul izni ve/veya yasal-özel bir izin gerektirmediğini beyan ederler.

AUTHORS' CONTRIBUTIONS (YAZARLARIN KATKILARI)

Gözde ALTUNTAŞ: She completed the microstructure characterization, XRD analysis, wear test, hardness measurements and article writing.

Mikro yapı karakterizasyonu, XRD analizi, aşınma testi, sertlik ölçümleri ve makale yazımını tamamladı.

Ömer Faruk KAPLAN: Made the material supply, forging process and heat treatment processes

Malzeme temini, dövme işlemi ve ısıl işlem işlemlerini yaptı.

Bülent BOSTAN: He followed all the experimental processes. He made comments after each analysis.

Tüm deneysel süreçleri takip etti. Her analizden sonra yorum yaptı.

CONFLICT OF INTEREST (ÇIKAR ÇATIŞMASI)

There is no conflict of interest in this study.

Bu çalışmada herhangi bir çıkar çatışması yoktur.

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