VANOS
JOURNAL OF MECHANICAL ENGINEERING EDUCATION
http://jurnal.untirta.ac.id/index.php/vanos
ISSN 2528-2611, e-ISSN 2528-2700
Volume 8, Number 2, November 2023, Pages 136-145

The Effect of Cryogenic Treatment Temperature on the Mechanical Properties and Microstructure of 440C Martensitic Stainless Steel

Indah Uswatun Hasanah[1], A. Ali Alhamidi[2], Rachmat Suryana[3]

[1,3]Metallurgical Engineering, Faculty of Engineering, University of Sultan Ageng Tirtayasa, Indonesia
Jenderal Sudirman Km 3, Kotabumi, Kec. Purwakarta, Kota Cilegon, Banten, Indonesia
[2]Mechanical Engineering, Faculty of Engineering, University of Sultan Ageng Tirtayasa, Indonesia
Jenderal Sudirman Km 3, Kotabumi, Kec. Purwakarta, Kota Cilegon, Banten, Indonesia

Corresponding author: indah.uswatun.h@untirta.ac.id

Received: 25 July 2023. Accepted: 10 October 2023. Published: 30 November 2023

ABSTRACT

The use of stainless steel worldwide is increasing due to its favorable mechanical properties, such as high hardness, corrosion resistance, and wear. One of the martensitic stainless steels is SS 440C. One of the main effects of increasing the significant carbon content is lowering the final martensitic temperature (MF) of the steel. If this temperature is below room temperature, the quenching process leaves austenite in the microstructure. This is commonly known as retained austenite (RA). In general, minimizing the number of RAs is recommended, as they can cause excessive wear. Therefore, the aim this research is for reduce the retained austenite content in SS 440C steel with a cryogenic treatment process. The cryogenic treatment was carried out for 50 minutes at temperatures of -80, -110, and -140°C and compared with non-cryogenic treatment to determine the residual austenite, hardness, and wear resistance. The highest hardness and wear resistance values were obtained from cryogenic treatment at -140°C at 58 HRC and 2.2 x 10-3 mm^3/m. The metallographic results produce a martensite microstructure, residual austenite, and carbide phases. XRD analysis on cryogenic treatment samples at -140°C yielded structures of iron bcc, iron fcc, M7C3, and M23C6 compounds.

Keywords: Retained Austenite, Cryogenic Treatment, Stainless Steel Martensitic 440C

INTRODUCTION

The use of stainless steel in the world is increasing due to its advantageous mechanical properties, such as corrosion resistance, low maintenance cost, and high strength [1][2][3][4]. There are industries that use stainless steel for the same reason, as it is a well-known fact that stainless steel does not require additional treatments such as surface treatment, painting, coating, and others to enhance its corrosion resistance[5][6]. Due to its great mechanical strength and strong resistance to corrosive environments, such as high humidity and acidic conditions, stainless steel SS 440C is a high-carbon martensitic stainless steel. SS 440C is used for manufacturing bearing elements in turbopump machines for aircraft engines [7][8][9]. One of the main effects of alloying elements in SS, such as chromium, is the reduction of the martensitic transformation temperature in the steel [3][10][11][12][13]. The phenomenon known as retained austenite (RA) occurs when the temperature falls below room temperature, indicating an incomplete quenching process and the retention of austenite in the microstructure [14][15][16]. Generally, it is recommended to minimize the amount of retained austenite, as it can lead to excessive wear and dimensional changes during component service [12][17][18]. Therefore, this type of steel requires cryogenic treatment to reduce the retained austenite content, which can enhance its wear resistance and other mechanical properties, such as toughness and hardness [19][20][21].

Idayan et al. (2014) focused their research on the influence of deep cryogenic treatment (-198°C) on the mechanical properties of AISI 440C ball bearing steel. The hardness of the material increased by 7%, reaching 61 HRC during the deep cryogenic process, and by 4%, reaching 57 HRC during the shallow cryogenic process, compared to conventional heat treatment [22][23]. Furthermore, Prieto et al. (2020) focused their research on the Effect of cryogenic treatment on the RCF Performance of AISI 440C martensitic stainless steel. In their study, they divided the research into two methodologies. First, the steel underwent oil quenching and tempering [24]. The next methodology involves performing cryogenic treatment (-196°C) and shallow cryogenic treatment (-80°C) for 5 hours, followed by tempering. The results showed that although cryogenic treatment was conducted, it did not significantly improve the RCF (Rolling Contact Fatigue) resistance of the material. It only reduced the amount of carbide, which in turn decreased the interfacial area between carbide and the martensitic matrix [25].

Therefore, the aim this research is for reduce the retained austenite content in SS 440C steel with a cryogenic treatment process. In this research, the cryogenic treatment process will be conducted on the SS 400C substrate, varying the temperature at -80°C, -110°C, and -140°C. Furthermore, this research will compare the cryogenic treatment process with the non-cryogenic treatment using N2 gas quenching. The independent variables to be analyzed are the mechanical properties, wear resistance, and microstructure of the samples.

RESEARCH METHOD

The SS 440C steel was prepared in two different dimensions, tailored to the requirements of characterization and testing. The cutting process was carried out to obtain sample sizes with dimensions of 20 x 40 x 5 mm for wear testing and dimensions of 10 x 10 x 5 mm for X-ray Diffraction, hardness testing, and metallographic characterization.

The treatment process was conducted using two methods: non-cryogenic treatment and cryogenic treatment. The prepared samples were placed in the vacuum furnace VHT 30 NVF-30P for pre-heating at temperatures of 650°C and 850°C for 1 hour and then heated to reach the austenitizing temperature of 1040°C for 50 minutes. Next, the samples underwent the quenching process using N2 gas at a pressure of 3 bar. Meanwhile, the cryogenic treatment method began by inserting the samples into the subzero treatment machine UP 35A. This process involved using liquid N2 as a cooling medium for 50 minutes at varying temperatures of -80°C, -110°C, and -140°C. Subsequently, the samples underwent tempering at a temperature of 520°C for 6 hours inside a vacuum furnace VHT 250 TITAN H2.

Testing and characterization were conducted to determine the changes in mechanical properties of the samples. Hardness testing was performed using the Nobel MHVS-1000AT hardness testing instrument in accordance with the ASTM E-384 Standard Test Method for Micro indentation Hardness of Materials. The wear resistance testing was performed using a wear testing instrument, following the ASTM G99 Standard Test Method for Wear Testing with a Pin-on-Disk Apparatus, using the Ogoshi method. Meanwhile, the metallographic process was conducted using Kalling's reagent no.1 and observed under an Olympus BX41M-LED optical microscope at magnifications of 200x and 500x.

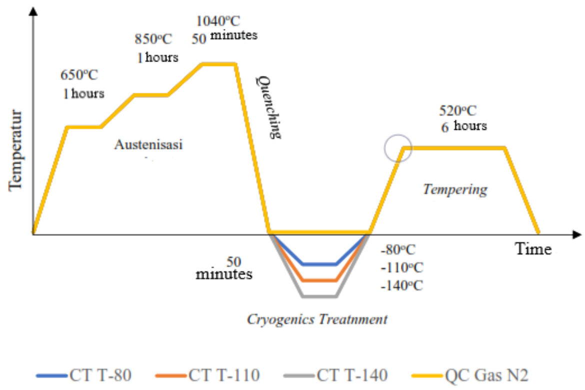

Figure 1. Temperature and time charts

RESULT AND DISCUSSION

The hardness testing standard referred to in this study follows ASTM E-384 Standard Test Method for Micro indentation Hardness of Materials. The testing was conducted using the Vickers method with a 1 Kgf load. Each test specimen underwent 3 repetitions of testing at the center of the specimen, with a spacing of 0.3 mm between test points. The obtained results were then converted into HRC units. The hardness test results can be seen in Figure 2.

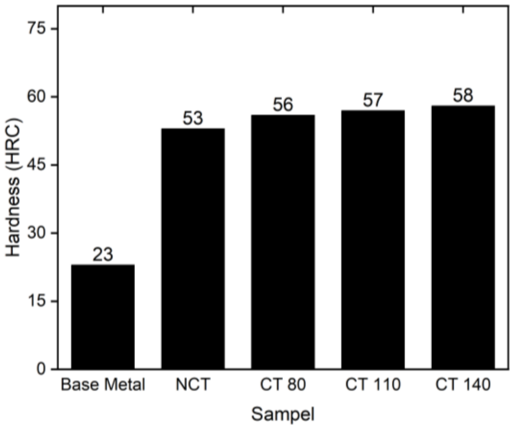

Figure 2. Hardness value of cryogenic treatment

Figure 2 shows that CT 140°C yields the highest hardness value of 58 HRC. This indicates that decreasing the cryogenic treatment temperature will increase the hardness value of 440C martensitic stainless steel. This is due to the phenomenon of forming a body-centered tetragonal (BCT) structure, the reduction of retained austenite, and the formation of carbides. The formation of the BCT structure occurs when rapid cooling leads to the formation of the martensite phase. The martensite phase forms during rapid cooling from the austenite phase, where the diffusion process does not occur. As a result, carbon does not have enough time to diffuse out and becomes trapped within the crystal structure, forming a tetragonal structure with small interatomic voids. This leads to an increase in hardness value. Furthermore, the lower the cryogenic treatment temperature, the greater the amount of austenite transforming into martensite. Therefore, cryogenic treatment can facilitate the transformation of retained austenite into martensite [26]. The increase in hardness caused by the cryogenic treatment process can be explained by the transformation of austenite into martensite. This can occur because the martensite finish temperature of the stainless steel is -53.21°C, whereas NCT (Non-Cryogenic Treatment) only cools down to room temperature, so the austenite does not fully transform into martensite [24]. Additionally, a larger number of carbides are formed during cryogenic treatment compared to NCT, resulting in higher hardness values for cryogenic treatment than for NCT.

The wear resistance testing in this research was conducted using the Ogoshi method, which refers to the ASTM G99 Test Method for Wear Testing with a Pin-on-Disk Apparatus. The wear test involved a loading of 3.16 kg, a ring thickness of 3 mm, a ring diameter of 30 mm, a sliding speed of 1.97 m/s, and a sliding distance of 100 m.

Figure 3 presents the wear resistance values obtained from cryogenic treatment on 440C martensitic stainless steel at various temperatures, which show a decrease. Among the samples, the CT 140°C condition yields the

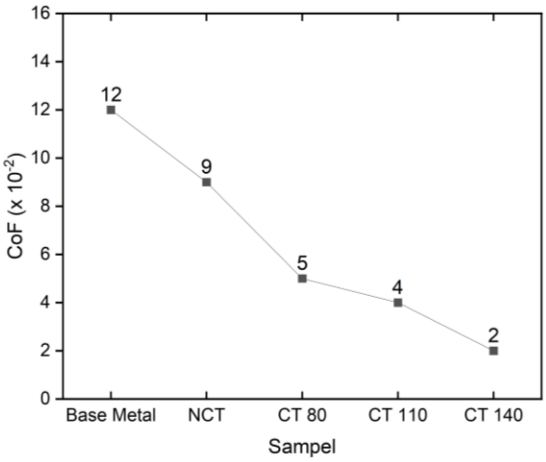

Figure 3. Coefficient of friction

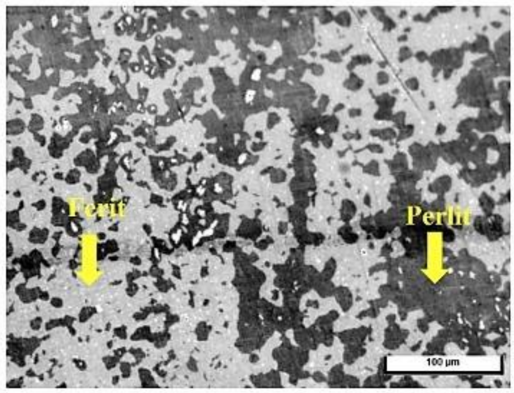

Figure 4. Microstructure of the Base Metal of 440C Martensitic Stainless Steel.

Lowest CoF value, which is 2 x 10-2. The reduction in wear resistance is attributed to the transformation of retained austenite into a harder martensitic structure as the temperature decreases. The main factor contributing to the increased wear resistance through cryogenic treatment is the elimination of retained austenite and the formation of a martensitic structure. Thus, as the cryogenic treatment temperature decreases, the wear resistance value will increase [27] [28]. The comparison of the CoF (Coefficient of Friction) values between cryogenic treatment and NCT (Non-Cryogenic Treatment) shows an increase of 69% and 46% respectively when applied to the base metal. The factors influencing these two conditions are the retained austenite effect and carbide formation [29].

In Figure 4, the microstructure of the as received 440C martensitic stainless steel base metal is presented, showing the presence of ferrite, and pearlite structures. The dark areas indicate the pearlite phase, while the light areas represent the ferrite phase. It can be observed that the perlite phase dominates with a percentage of 51.75%, providing toughness to the 440C martensitic stainless steel. Meanwhile, the perlite phase in the 440C martensitic stainless steel contributes to its strength[8].

Figure 5 shows the microstructure of quenching 440C martensitic stainless steel with H_2 gas, which exhibits a microstructure comprising both martensite and retained austenite. The comparison of retained austenite between the sample treated with cryogenic treatment at -140°C and quenching gas H_2 shows a difference of 22.09%. This can occur because the martensitic stainless steel 440C has a carbon content of 1.05% and a chromium content of 16.7%, causing the final tempering temperature to be below room temperature. As a result, austenite has not fully transformed into martensite. This will result in the formation of high retained austenite content, leading to low wear resistance and hardness [30].

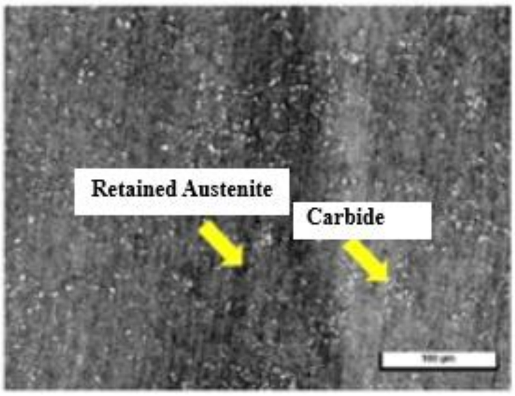

Figure 5. Microstructure of 440C martensitic stainless steel with H_2 gas quenching

Figure illustrates the microstructure of 440C stainless steel subjected to cryogenic treatment. From Figure 5, it is observed that CT 80°C, CT 110°C, and CT 140°Chave retained austenite contents of 10.54%, 8.93%, and 5.09%, respectively. Meanwhile, the percentage of carbide in CT 80°C, CT 110°C, and CT 140°C is 4.06%, 5.66%, and 10.22%, respectively. The lower the cryogenic treatment temperature, the reduction of retained austenite and the increase in carbide formation occur. The smallest amount of retained austenite is observed in the sample treated at -140°C, while the largest amount is found in the sample treated at -80°C.

The X-ray Diffraction analysis conducted involves comparing the as-received and as-treatment 440C stainless steel samples. The stainless-steel AS-treatment is a 440C stainless steel that has undergone cryogenic treatment at a temperature of -140°C for 50 minutes. In Figure 6, it is shown that in the as-received and as-treated samples, iron-α, iron-γ, M7C3 carbide, and M23C6 carbide are obtained. The formation of carbides strengthens the hardness data in Figure 1, indicating that the formation of carbides is responsible for the increase in hardness values. This is supported by previous research suggesting that retained austenite transformation within the martensite and the formation of M7C3 and M23C carbides are considered key factors in enhancing mechanical properties [28].

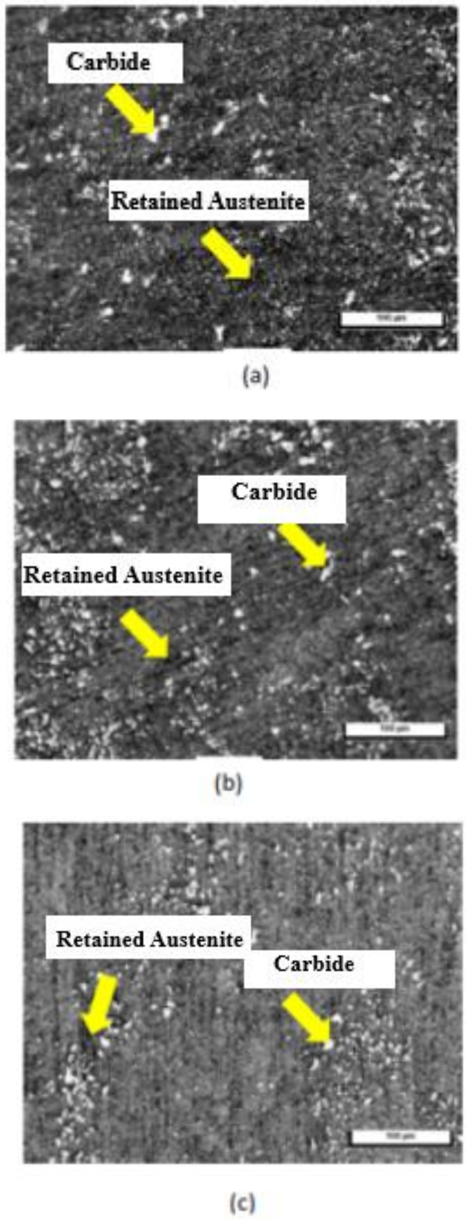

Figure 6. The microstructure of cryogenic treatment temperatures (a) -80°C, (b) -110°C, (c)-140°C

In each XRD peak in Figure 7 with the same phase, a shift in the 2θ value is observed. The shift towards higher 2θ values is caused by the main phase of the martensitic matrix shifting after undergoing cryogenic treatment. The substitution of Fe atoms causes a slight outward shift of carbon atoms due to lattice deformation. This phenomenon leads to a change in the martensitic structure, resulting in a lower carbon content within the matrix.

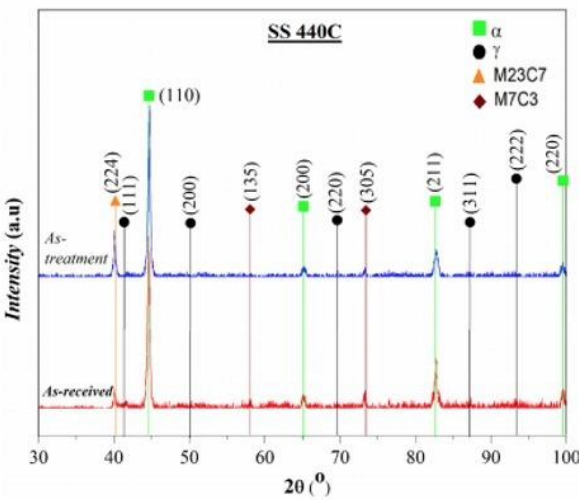

Figure 7. XRD characterization

CONCLUSION

Based on the research on the effect of cryogenic treatment at various temperatures on martensitic stainless steel 440C, it can be concluded from the microstructure that cryogenic treatment samples exhibit a lower residual austenite, resulting in improved mechanical properties. This is evidenced by the highest hardness and wear resistance values obtained from the cryogenic treatment sample at a temperature of -140°C, which are 58 HRC and 2.2 x 10-3 mm3/m, respectively. Furthermore, XRD characterization strengthens the previous data that the carbide produced from this process forms M7C3 and M23C6 carbide compounds.

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