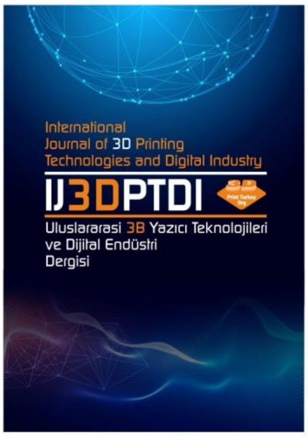

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EFFECT OF CRYOGENIC TREATMENT AND TEMPERING TEMPERATURE ON MECHANICAL AND MICROSTRUCTURAL PROPERTIES OF AISI 431 STEEL

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ABSTRACT

This study investigated the effects of cryogenic treatment and tempering temperature applied after cryogenic treatment on the mechanical and microstructural properties of AISI 431 martensitic stainless steel. After conventional heat treatment (CHT), the steel samples were cryogenically treated at -180 °C for 6 hours and then tempered at 200 °C (CT200) and 300 °C (CT300) for 2 hours. After these processes, hardness measurement, tensile test, and abrasion test were carried out to determine the mechanical properties of the steel samples. In addition, microstructure photographs were taken to determine the microstructural properties. As a result of the study, it was observed that the cryogenic treatment applied after the conventional heat treatment was effective on the mechanical properties of AISI 431 martensitic stainless steel. Cryogenic treatment and tempering temperature showed the greatest effect on wear resistance. CT200 and CT300 samples were 62% and 56% less worn than the CHT sample. Compared to the CHT sample, the yield strength of CT200 and CT300 samples increased by 6.95% and 7.03%, while the hardness increased by 3.89% and 3.52%.

Keywords: AISI 431 Martensitic Stainless Steel, Cryogenic Treatment, Tempering, Mechanical Properties, Microstructure.

1. INTRODUCTION

Stainless steels, which are among the indispensable materials of our modern industry, are iron-based alloys containing a maximum of 1.2% C and at least 10.5% Cr. These steels are the most important material for machinery and construction elements working in a corrosive environment. In addition, thanks to their mechanical properties, they have a wide range of use in aircraft, chemistry, petro-chemistry, food, pharmaceutical industry, nuclear power plants, tool and stainless goods industries [1]–[4]. The most important properties of martensitic stainless steels are high hardenability, good mechanical properties, and corrosion resistance.

Cryogenic treatment is a complementary process to conventional heat treatment, which has been used in recent years to improve the properties of metals. In the cryogenic process, materials are cooled to very low temperatures (-196 ºC) to obtain the desired mechanical and microstructural properties. In the cryogenic process, the material is kept at the specified temperature for a specified holding time and then gradually heated to room temperature. Thus, the transformation of residual austenite to martensite and the formation of secondary carbide precipitates in the nucleation zones provide high wear resistance in the material [5]–[10]. It is also known that the cryogenic process has positive effects on other mechanical properties (hardness, tensile strength, corrosion resistance, impact energy, etc.) depending on the material type [11]–[16].

Wang et al. [17] found that cryogenic treatment significantly increased the hardness and wear resistance of 16Cr1Mo1Cu cast iron. Altan Özbek et al. [18] reported an increase in hardness and wear resistance after cryogenic treatment applied on AISI H11 steel. Similarly, Harish et al. [19] and Benselly et al. [20] determined that the hardness and wear resistance of the steel increased after cryogenic treat”ment applied on En 31 bearing steel and En 353 cementation steel. In addition, these authors reported that tempering after cryogenic treatment increases secondary carbide precipitation and reduces residual stresses, so tempering must be applied after cryogenic treatment. The tempering heat treatment is carried out by heating the material below the austenitizing temperature. At the end of this heat treatment, the hardness of the steel, which has a tight structure, decreases, and its toughness increases due to the decrease in the tension level. The tempering temperature varies depending on the type of steel [21]–[23].

Dhokey et al. [24] investigated the effects of the multiple tempering processes on the wear resistance of the steel by applying single, double, and triple tempering processes at 150 °C after cryogenic treatment on D3 tool steel and reported that the single tempering process provides more wear resistance increase. Darwin et al. [25] determined that the optimum tempering temperature was 250 °C between the temperatures of 200, 250, and 300 °C in their optimization study to maximize the wear resistance of a commercial piston ring made of martensitic stainless steel containing 18% Cr. Zhirafar et al. [26] determined that the highest hardness was obtained in the tempered sample at 200 °C when tempering was applied to AISI 4340 steel at 200, 300, and 455 °C for 2 hours after cryogenic treatment. The hardness of the steel samples decreased with increasing tempering heat treatment temperature.

In the present study, after conventional hardening of AISI 431 martensitic stainless steel, cryogenic treatment was applied at –180 °C for 6 hours, and then tempering heat treatment was applied at 200 °C and 300 °C for 2 hours. The effects of cryogenic treatment and tempering temperature on the hardness, tensile strength, yield strength, abrasion resistance, and microstructural properties of steel samples were investigated.

2. MATERIAL AND METHODS

The chemical composition of AISI 431 martensitic stainless steel used in the study is given in Table 1. Heat treatments applied on AISI 431 martensitic stainless steel are given in Fig. 1. Steel samples were first subjected to austenitizing, quenching, and tempering (CHT) processes. After these processes, cryogenic treatment was applied to a group of samples at -180 °C, and then tempering heat treatment was applied at 200 °C and 300 °C.

Table 1. Chemical components of AISI 431 martensitic stainless steel (%).

C	Cr	Mn	Ni	Si	Mo	Cu	P	S	Fe
0.188	15.597	0.892	1.53	0.42	0.148	0.113	0.027	0.006	Bal.

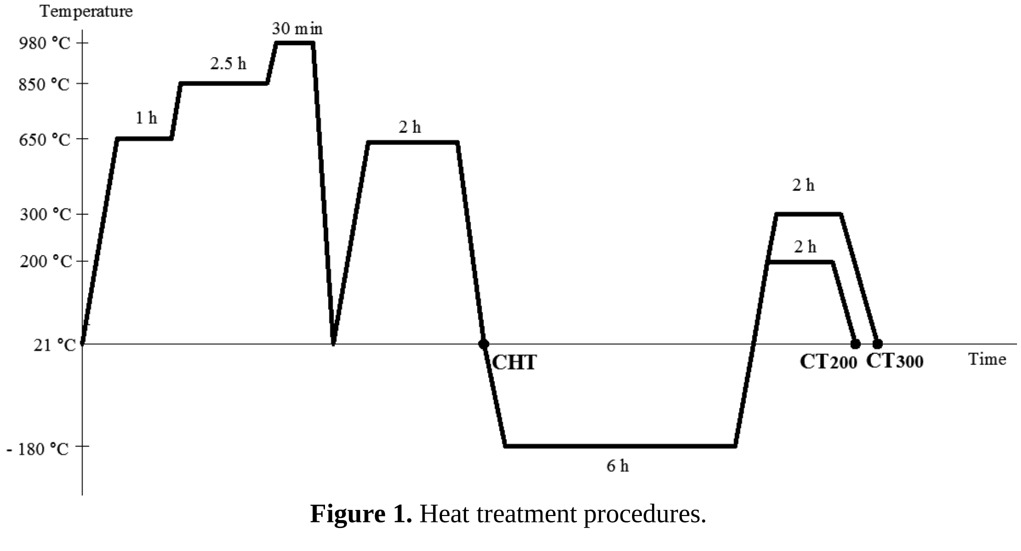

Figure 1. Heat treatment procedures.

Microhardness measurements were made using Microbul-1000 D type microhardness tester. Test parameters are given in Table 2. 5 hardness measurements were made from each sample and the microhardness of the samples was determined by taking the arithmetic average of the measured values.Tensile tests were carried out using an UTM200 model tensile tester (Fig. 2). For the tensile tests, the steel samples were brought to the dimensions in Fig. 2.

Wear tests were performed on a computer-controlled TRD Wear pin-on-disc device (Fig. 3). The load was given in one direction and no lubricant was used. Wear test parameters are given in Table 2. After the wear tests, the amount of wear on the samples was determined via electronic scales with an accuracy of ± 0.0001 g.

Table 2. Test parameters.

Test	Load	Application time	-
Microhardness	300 gr	30 s	-
Test	**Load**	**Speed**	**Sliding distance**
Wear	40 N	0.55 m/s	500 m

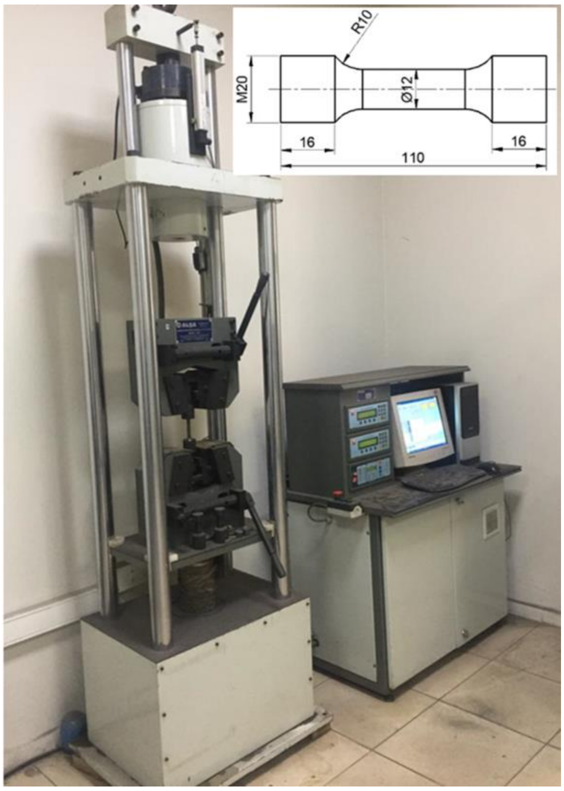

Figure 2. Tensile test device and test sample.

Figure 3. TRD Wear abrasion tester.

3. EXPERIMENTAL RESULTS AND DISCUSSION

Microstructure photographs show that a lath-type martensite structure with residual austenite was formed for all samples (Fig. 4). However, the martensitic laths in the cryogenically treated samples are smaller than the CHT sample. In addition, carbide particles are clearly visible in optical and SEM photographs. The EDS analysis confirmed the matrix structure of the steel samples and the carbide particles. EDS 1 analysis matrix and EDS 2 analysis show carbides (Fig. 5). Jovičević-Klug et al. [27], in their study, determined that after the cryogenic treatment applied by keeping it in liquid nitrogen for 24 hours, there was a decrease in the amount of residual austenite and an increase in the amount of carbide of AISI 431 steel. They also confirmed by XRD, SEM-EBSD, and TEM analyses that carbides are generally in two main types, M_3C_2 (enriched with Cr and Fe) and $M_{23}C_6$ (enriched with Cr and Fe). However, the authors reported that in addition to the carbide groups (M_3C_2 (Pnma) and $M_{23}C_6$ (Fmm)) confirmed by TEM results, M2C (Pnnm) and M_7C_3 (Pnma) could be found, all enriched with Cr and Fe.

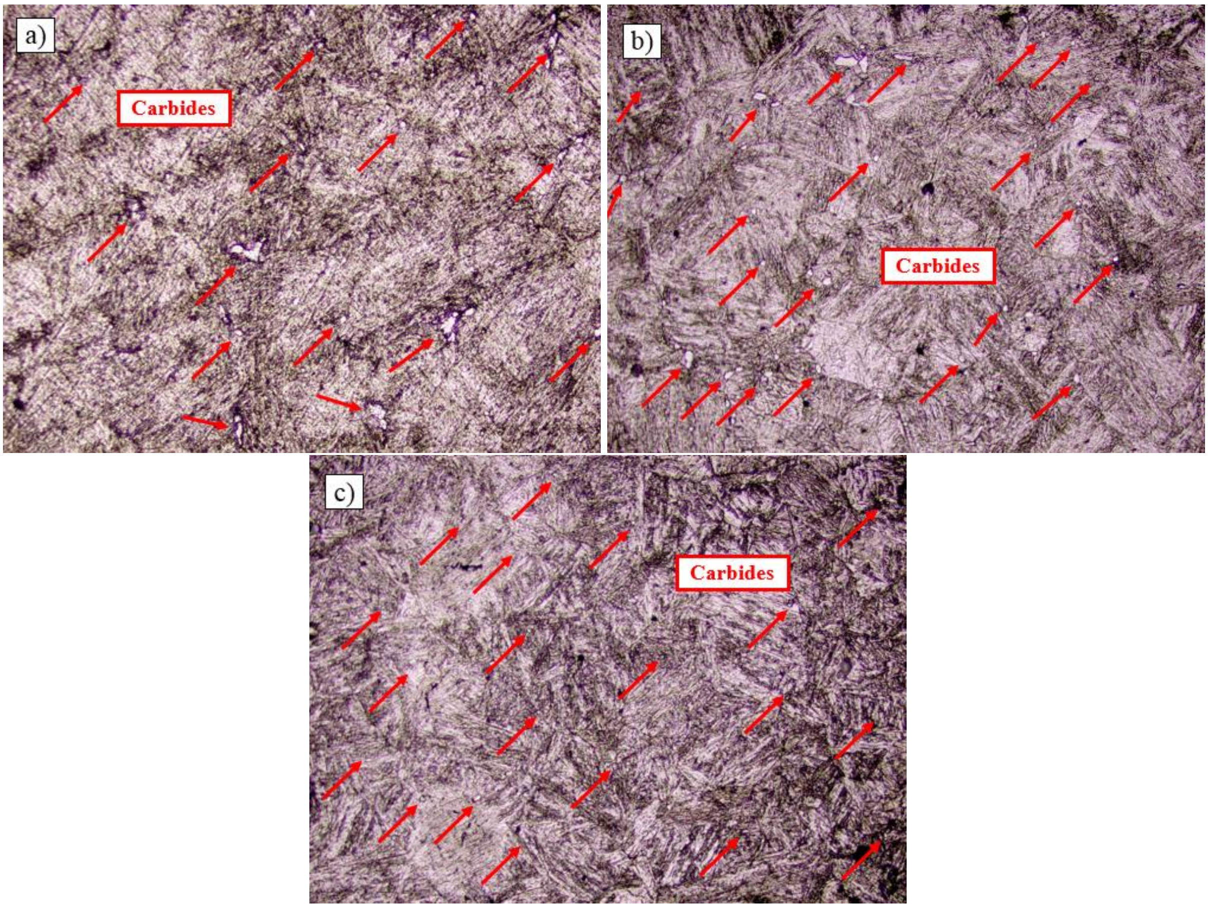

Figure 4. Photographs of the microstructure of AISI 431 martensitic stainless steel taken with an optical microscope a) CHT, b) CT200, c) CT300.

The graph showing the hardness measurement results of AISI 431 steel is given in Figure 6. Regardless of the tempering temperature, the hardness of both cryogenically treated samples is higher than the conventional heat-treated sample. The cryogenic treatment enables the transformation of residual austenite in the microstructure to martensite, the formation of carbide precipitates, and a more homogeneous carbide distribution [18], [28]–[30]. Thus, it provides an increase in the hardness and wear resistance of the material.

While the hardness of AISI 431 steel was 428,577 HV in the conventional heat-treated sample, it reached 445,285 HV after the cryogenic treatment at 200 °C. When tempering was treated at 300 °C after cryogenic treatment, the hardness value of the steel reached 443.655 HV. Thus, it can be said that the hardness increase of 3.89% was achieved in the sample that was tempered at 200 °C after the cryogenic treatment, compared to the sample that had been tempered at 300 °C after the cryogenic treatment, and 3.52% in the sample that was tempered at 300 °C after the cryogenic treatment. In their study on 18NiCrMo5 steel, Baldissera and Delprete [15] reported that hardness increases from 0.6% to 2.4% depending on the cryogenic treatment condition. In another study, Özbek et al. [18] observed an increase in hardness on AISI H11 steel after cryogenic treatment. In another study, Wang et al. [17] determined that cryogenic treatment significantly increased the hardness of 16Cr1Mo1Cu cast iron.

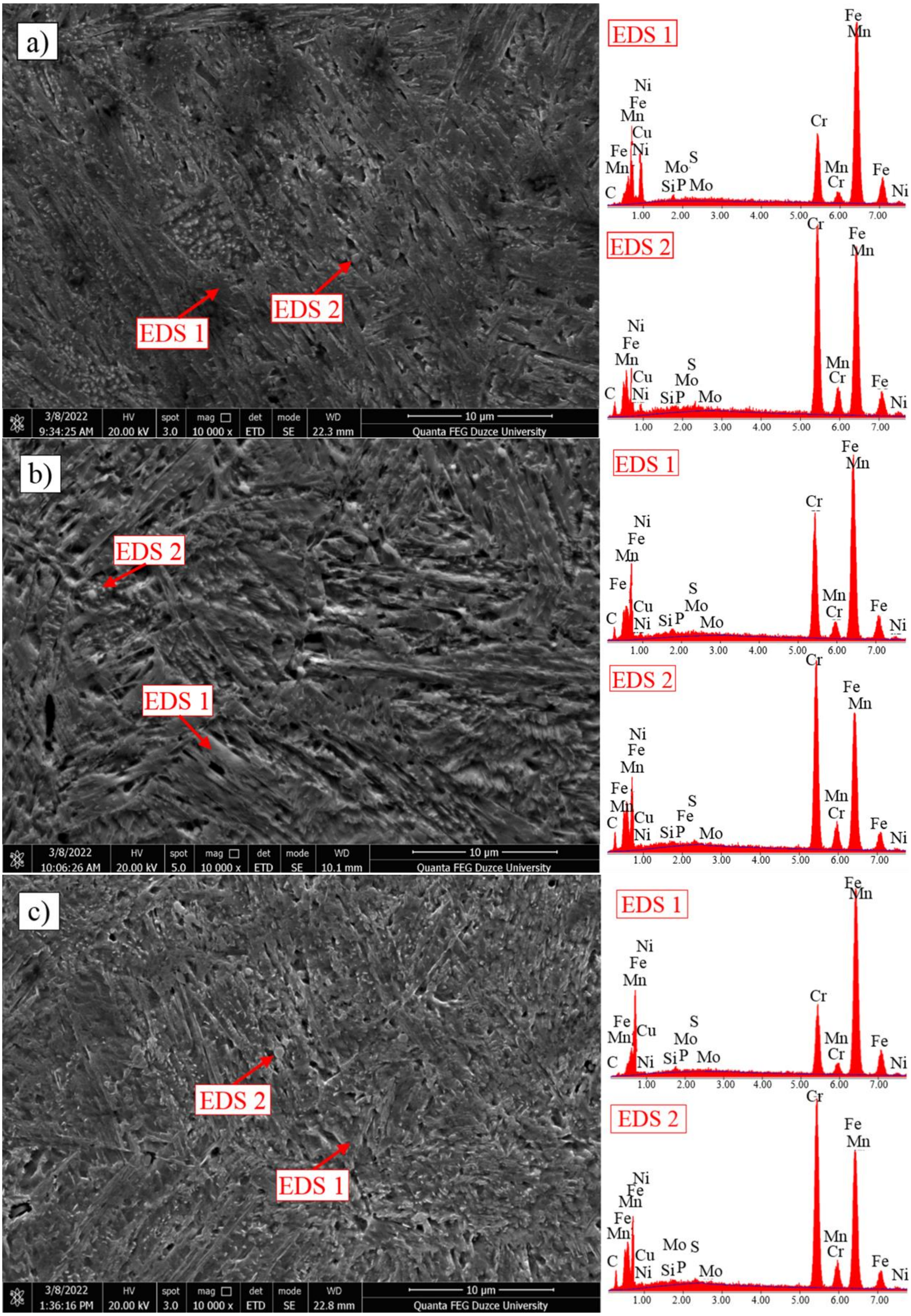

Figure 5. SEM photographs of the microstructure of AISI 431 martensitic stainless steel a) CHT, b) CT200, c) CT300.

After the cryogenic treatment, it was observed that a slightly lower hardness was obtained in the sample tempered at 300 °C compared to the sample tempered at 200 °C. In the literature, Zhirafar et al. [26] reported that the tempering heat treatment applied on AISI 4340 steel after cryogenic treatment achieved significantly lower hardness at higher tempering temperatures. Altan Özbek and Saraç reported in their studies that a decrease in the hardness of the samples was observed with the increase of the tempering temperature in the tempering process applied after the conventional heat treatment on AISI 4140, AISI 1020 and AISI 1040 steels [23], [31].

Figure 7 shows the graphs showing the samples' tensile strength and yield strength changes. The lowest tensile strength and yield strength were measured in the CHT sample. A slight increase was observed in the steel samples' tensile strength and yield strength values after the cryogenic treatment. While the tensile strength of the CHT sample was 1383 MPa, the tensile strength of the CT200 sample was 1403 MPa with an increase of 1.44%. The tensile strength of the CT300 sample was measured as 1399.33 MPa with an increase of 1.18% compared to the CHT sample. While the yield strength of the CHT sample was 1187 MPa, the yield strength of the CT200 sample was 1269.5 MPa with an increase of 6.95%, and the yield strength of the CT300 sample was 1270.5 MPa with an increase of 7.03%. The tensile strength and yield strength values of CT200 and CT300 samples are very close. In general, it is seen that the tensile strength and yield strength graphs are in parallel with the microhardness graph.

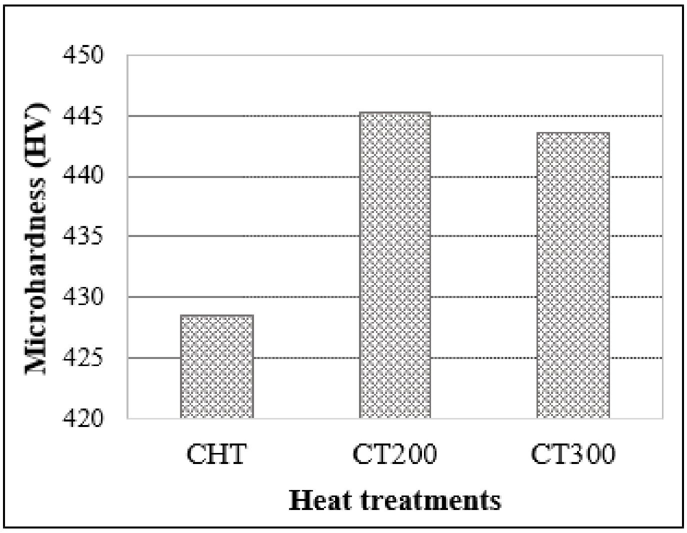

Figure 6. Microhardness change graph.

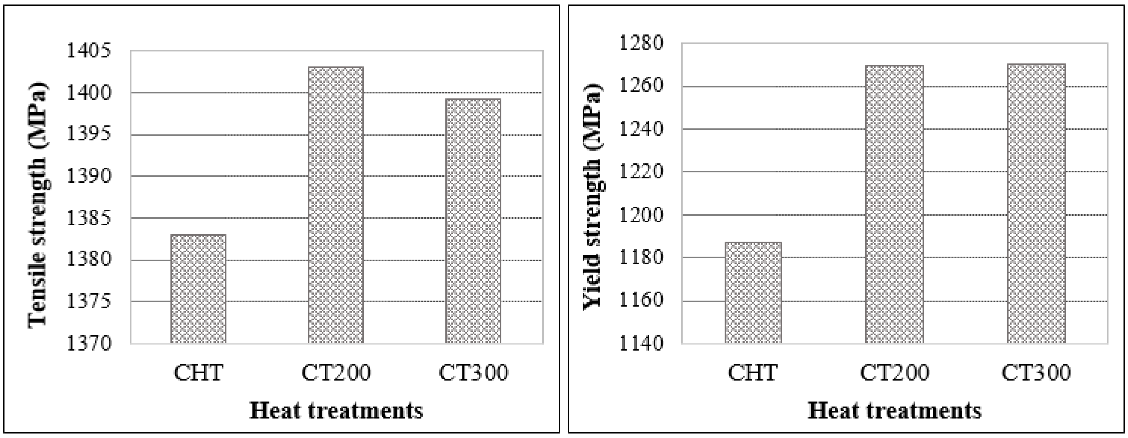

Figure 7. Tensile strength and yield strength change graph.

Contrary to the detected effects of cryogenic treatment on tensile strength and yield strength in this study, Bensely et al. [29] reported a marginal decrease in tensile strength after cryogenic treatment for 815M17 steel compared to conventional heat treatment. Contrary to this study, in another study, Sonia et al. [14] reported that cryogenic treatment increased the tensile strength and yield strength of Al6082 aluminum alloy. Similarly, Baldissera and Delprete [15] reported that the tensile strength of 18NiCrMo5 steel increased up to 11% with cryogenic treatment.

The graph showing the wear rates resulting from the wear test is given in Figure 8. Among the steel samples, the most worn sample was CHT. There was much less wear in the cryogenically treated samples compared to the CHT sample. Cryogenic treatment converts residual austenite to martensite, provides carbide precipitation and provides a more homogeneous carbide distribution, thus increasing the hardness of the steel and thus the wear resistance.

The least worn specimen was the CT200. The wear rate of CT200 and CT300 samples is 62% and 56% less compared to the CHT sample. From this, it can be said that the cryogenic treatment has a significant effect on the wear resistance of AISI 431 martensitic stainless steel. The wear rate of steel tempered at 300 °C after cryogenic treatment is 15.80% higher than that of steel tempered at 200 °C.

Similarly, there are studies in the literature reporting that cryogenic treatment provides an increase in wear resistance on 16Cr1Mo1Cu cast iron [17], AISI H11 hot work tool steel [18], En 31 bearing steel [19], En 353 cementation steel [20]. It is seen that the results obtained in this study are compatible with the literature. However, Darwin et al. [25] claimed that the effect of tempering temperature on the wear resistance of martensitic stainless steel is very small regarding the tempering process applied after cryogenic treatment. However, in this study, it was observed that the tempering temperature greatly affects the wear resistance.

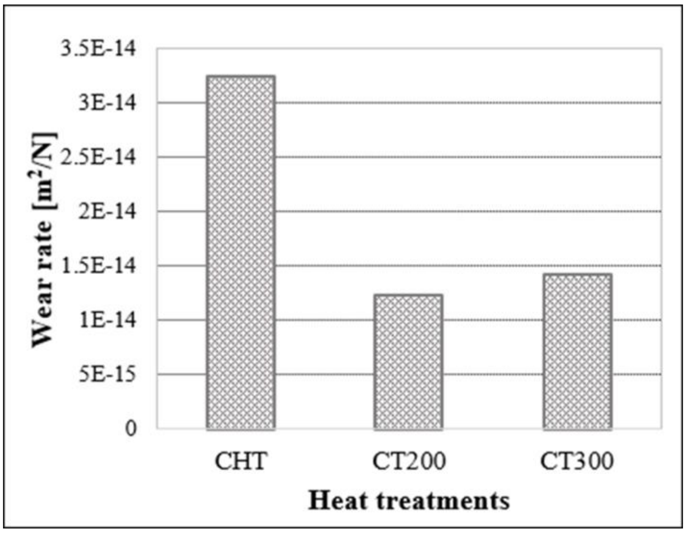

Figure 8. Graph of change in wear rate of samples.

4. CONCLUSIONS

This study investigated the effects of cryogenic treatment and cryogenic treatment tempering temperature on the microstructure and mechanical properties of AISI 431 martensitic stainless steel. The results obtained in the study are as follows:

- The cryogenic treatment and tempering temperature caused a change in the microstructure of the steel.
- Cryogenic treatment applied after conventional heat treatment resulted in an increase of 3.89% and 3.52% in the hardness of AISI 431 martensitic stainless steel.
- Cryogenic treatment did not significantly affect the tensile strength of AISI 431 martensitic stainless steel but produced a small increase (1.44% and 1.18%). It has increased the yield strength up to 7.03%.

- Cryogenic treatment showed the greatest effect on wear resistance. Cryogenically treated specimens worn up to 62% less.
    - After cryogenic treatment, there was no significant difference in the hardness, tensile strength and yield strength of the samples after tempering at 200 °C and 300 °C. However, it is seen that the tempering temperature has a significant effect on the wear resistance.

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