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EFFECT OF HEAT TREATMENT ON MECHANICAL PROPERTIES OF TYPE-304 AUSTENITIC STAINLESS STEEL

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Abstract. In this study, the effects of cryogenic treatment and heat treatment on the microstructural evolution and mechanical properties of type-304 austenitic stainless steel are investigated. Cryogenic treatment is conducted in a liquid nitrogen environment at −196°C, both with and without a quenching process. The experimental results indicate that traditional heat treatment processes do not lead to improvements in hardness or phase transformation. Although a modest increase in the ultimate tensile strength is observed with prolonged immersion in liquid nitrogen, deep cryogenic treatment does not produce a significant enhancement in the tensile properties of the examined material. Among the cases examined, water-quenched heat treatment without cryogenic treatment demonstrates a considerable increase in ductility. Additionally, significant differences in microstructure and hardness are observed at different locations of the tensile specimen due to martensitic transformation from the austenitic phase during plastic deformation. Consequently, improvement in the mechanical properties of the steel is not a result of the heat treatment process; rather, the heat treatment affects the stability of the austenitic phase, thereby influencing the potential for martensitic transformation during plastic deformation.

Keywords: austenitic stainless steel, cryogenic, heat treatment, mechanical properties, microstructure.

1. INTRODUCTION

Among stainless steels, type-304 austenitic stainless steel is widely used in various industries due to its excellent corrosion resistance, since its chemical composition contains a considerable amount of chromium and nickel [1, 2]. In addition, it has been proven that the austenite phase in this steel can transform into the martensitic phase by the mechanism of strain-induced martensitic transformation during plastic deformation [3–5]. Thus, the mechanical properties of the steel, such as strength and ductility, are improved due to the phase transformation phenomenon during plastic deformation. However, the possibility of strain-induced martensitic transformation during plastic deformation of this steel strongly depends on the stability of the austenitic phase as well as the stacking fault energy [6–8]. Meanwhile, Pham et al. [9] showed that the heat treatment process plays a vital role in affecting the stability of the austenitic phase. On the other hand, literature surveys [10–12] indicated that deep cryogenic treatment (DCT), which involves cooling material to extremely low temperatures, can be considered a potential process to refine the microstructure and improve the mechanical properties of the material. According to Lee [13], the DCT environment can modify the stacking fault energy during deformation of austenite in austenitic steels. In order to enhance the performance of type-304 stainless steel in various engineering applications, it is necessary to understand the influence of heat treatment processes such as the quenching process and DCT on the possibility of martensitic transformation due to plastic deformation.

Recently, several investigations on cryogenic treatment of austenitic stainless steel have revealed significant advancements in understanding its effects on microstructure and mechanical properties. Shi et al. [14] examined the modification of microstructure and mechanical properties of AISI304 stainless steel affected by cryogenic rolling and annealing processes. In the research work of Zheng et al. [15], it is illustrated that the yield strength of AISI304 steel is improved by cryogenic rolling and cyclic annealing compared to the solution-treated state, while the enhancement of the tensile strength cannot be observed. The investigation of Lu et al. [16] focused on how warm forming influences the cryogenic tensile and impact properties of austenitic stainless steel in a liquid nitrogen environment. Jovičević-Klug et al. [17] investigated the influence of DCT on microstructure and surface properties of austenitic stainless steel AISI 304L and presented that DCT causes a modification in the microstructure of the material through the promoted precipitation of Cr_7C_3 carbides, induced twinning, and α'-martensite formation. Wang et al. [18] reported that the secondary hardening phenomenon can be observed at cryogenic temperature for 316L stainless steel fabricated by selective laser melting because of the strain-induced martensite transformation at low temperature. The mechanical properties of 316LN stainless steel with cryogenic pre-strain were studied at different cryogenic temperatures by Wu et al. [19]. More recently, Wei et al. [20] examined the effect of cryogenic pre-deformation on the microstructure and strain hardening behavior of 316L stainless steel. According to this research work, the cryogenically-rolled 316L stainless steel possesses higher strength and ductility in comparison with the conventional cold-rolled counterparts due to α'-martensitic transformation. Although a large number of studies on the microstructural characteristics of austenitic steels under cold deformation have been conducted, there are insufficient data on the effect of cryogenic or heat treatment on the microstructure evolution as well as the mechanical properties of type-304 austenitic stainless steel.

While recent studies have examined the effects of conventional heat treatments such as annealing or tempering on the microstructure and mechanical properties of AISI304 stainless steel, the application of cryogenic treatment combined with heat treatment remains limited in the literature. Moreover, its influence on phase transformation during plastic deformation, which leads to improvements in the mechanical behavior of the steel, remains insufficiently understood. It is expected that the stability of the austenite phase in the steel may be altered by cryogenic treatment at ultra-low temperatures, thereby affecting martensitic transformation as well as the strength and ductility of the material. Therefore, an investigation into the influence of cryogenic and heat treatments on the microstructure and mechanical properties of AISI304 stainless steel is indispensable.

The goal of this study is to investigate how cryogenic treatment and heat treatment affect the microstructure evolution and mechanical properties of type-304 austenitic stainless steel. The specimens are cryogenically treated in the liquid nitrogen environment at −196°C with and without a quenching process for different holding times. The microstructure of the material is observed at high magnification using a metallurgical microscope. The mechanical properties are evaluated by applying a hardness tester and a conventional tensile testing machine. Then, based on the results obtained from the microstructure and mechanical properties, the effects of cryogenic and heat treatment on the strain-induced martensitic transformation in type-304 austenitic stainless steel can be clearly explained.

2. METHODOLOGY

The chemical composition of the investigated steel as delivered is analyzed by an emission spectrometer, and the analysis results are given in Table 1. This steel contains a high amount of chromium, leading to excellent corrosion resistance. The high percentage of nickel in the steel might stabilize the austenitic structure at room temperature, enhance corrosion resistance, and improve the toughness of the steel [21]. The specimens were cut into a cylindrical shape with a diameter of 20 mm and a height of 15 mm for microstructure examination as well as hardness measurement. For microscopic observation by a metallurgical microscope, the specimens were ground on silicon carbide sandpaper with different grades of P120, P360, P500, P800, P1000, P1200, P1500, and P2000, then polished using Cr_2O_3 powder and finally etched in a solution of HCl and HNO_3 with a ratio of 3:1, respectively.

Table 1. Chemical compositions of SUS304

Chemical element	C	Si	Mn	Cr	Ni	Co	N	V
Wt. (%)	0.053	0.31	1.17	18.8	8.03	0.22	0.113	0.103
Chemical element	P	S	Mo	Al	Cu	Ti	W	Fe
Wt. (%)	0.023	0.004	0.007	0.001	0.02	0.001	0.007	Blance

On the other hand, in order to evaluate the tensile properties of the investigated steel, the specimens have dimensions following ASTM-E28/E8M-09 standards as shown in Fig. 1. The specimens were manufactured using the electro-discharge machining method to ensure dimensional accuracy and to avoid thermal effects on the microstructure and mechanical properties of the steel during machining.

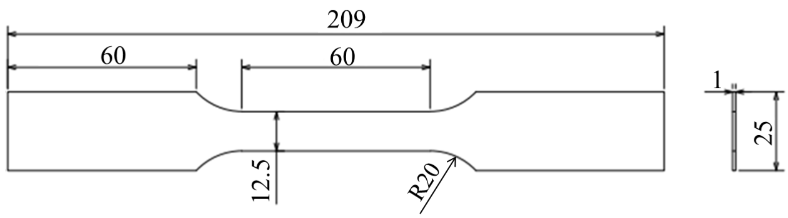


Fig. 1. Specimen for tensile test

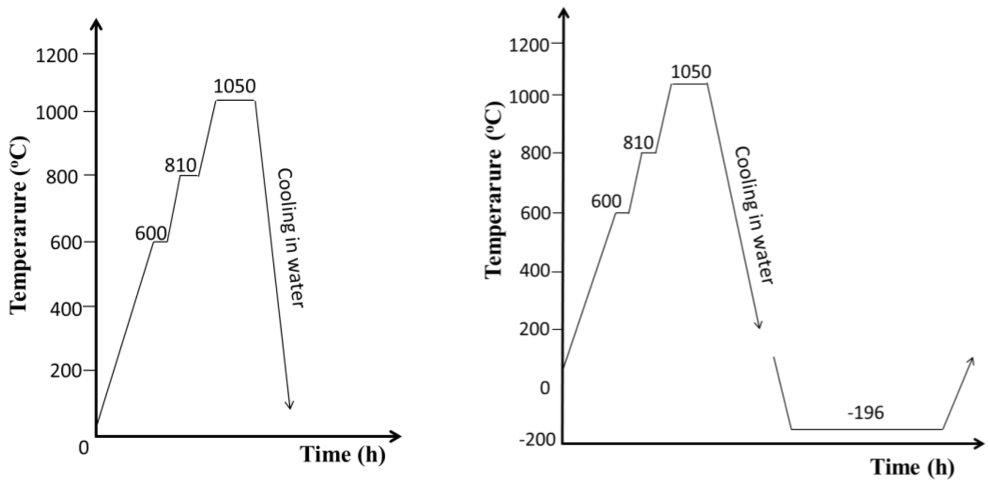
According to Jain et al. [22], the suitable annealing temperature for AISI 304 stainless steel is 1050°C, which promotes a microstructure of uniform austenite, thereby facilitating the austenite-to-martensite phase transformation during plastic deformation. Similarly, in previous studies [3, 23], the heat treatment condition with a heating temperature of 1050°C, a holding time of 30 minutes, and a cooling medium of water was often applied for type-304 austenitic stainless steel. This heat treatment condition is also applied to the same material in the present study. The experimental parameters of the quenching and DCT processes are presented in Table 2.

The heat treatment process is carried out in the electrical resistance furnace device Nabertherm model LT5/14/B410. The cryogenic experiment is performed in liquid nitrogen environment at −196°C for 2 hours, 5 hours, and 20 hours. The detailed parameters of the quenching process without and with DCT are indicated in Fig. 2.

The tensile test is performed by a WA-1000C conventional tensile testing machine at a displacement rate of 2 mm/min at room temperature. A hardness tester Rockwell Mitutoyo model HR-430MR is applied for hardness measurement. The microscopic examination is carried out by a metallurgical microscope Olympus GX41. The phase composition distributed in the specimen after tensile testing is analyzed using X'Pert Pro equipment.

Table 2. Testing conditions for quenching and DCT processes

Heat treatment process	Specimen symbol	Quenching		Deep cryogenic treatment	
		Temperature	Holding time	Temperature	Holding time
None	As delivered	-	-	-	-
Quenching	QT	1050°C	30 min	-	-
Quenching + DCT	QT-DCT-2h	1050°C	30 min	−196°C	2 h
Quenching + DCT	QT-DCT-5h	1050°C	30 min	−196°C	5 h
Quenching + DCT	QT-DCT-20h	1050°C	30 min	−196°C	20 h
Deep cryogenic treatment	DCT-2h	-	-	−196°C	2 h
Deep cryogenic treatment	DCT-5h	-	-	−196°C	5 h
Deep cryogenic treatment	DCT-20h	-	-	−196°C	20 h

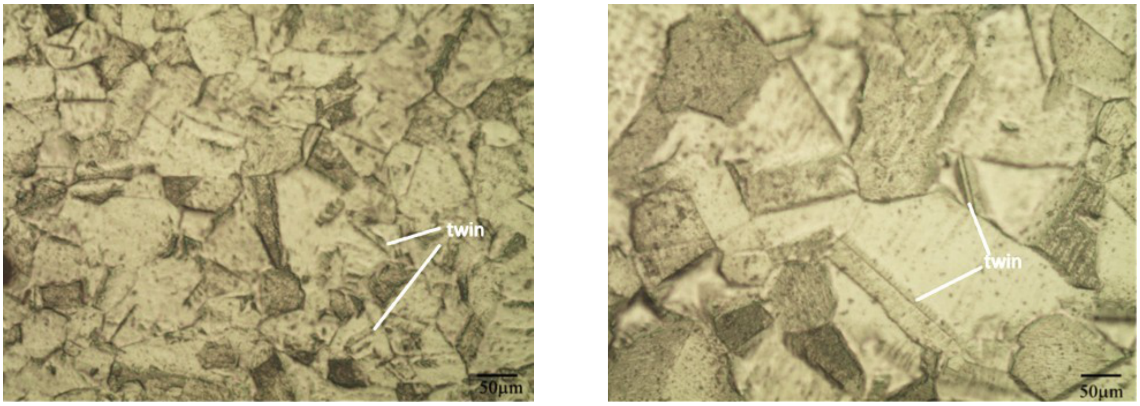


(a) Without DCT (b) With DCT

Fig. 2. Quenching process (a) without and (b) with DCT

3. RESULTS AND DISCUSSION

The microstructure of type-304 austenitic stainless steel as delivered and after heat treatment following Fig. 2(a) process is shown in Fig. 3. Obviously, the heat treatment process does not result in the formation of a new phase in the steel although it is quenched with a rapid cooling rate. Thus, a microstructure with almost fully austenitic phase as well as some deformation twins can be observed in the case with and without heat treatment. However, the morphology and grain size of the austenite phase after heat treatment differ from those in the as-delivered case. The austenite phase after heat treatment appears more homogeneous and exhibits a larger grain size compared to its state before heat treatment. The results regarding the microstructure of this steel after heat treatment are quite similar to those observed in previous studies [3, 24].



(a) As delivered (b) After quenching

Fig. 3. Microstructure of the material (a) as delivered and (b) after quenching

Fig. 4 shows the hardness value of the material in different experimental conditions. The difference in hardness among various heat treatment methods cannot be seen obviously. The obtained value of hardness is roughly 55–57 HRA for each testing condition and slightly higher than that of the as-delivered material. Therefore, it can be said that a modification of the surface hardness of type-304 austenitic stainless steel cannot be achieved by DCT or heat treatment processes.

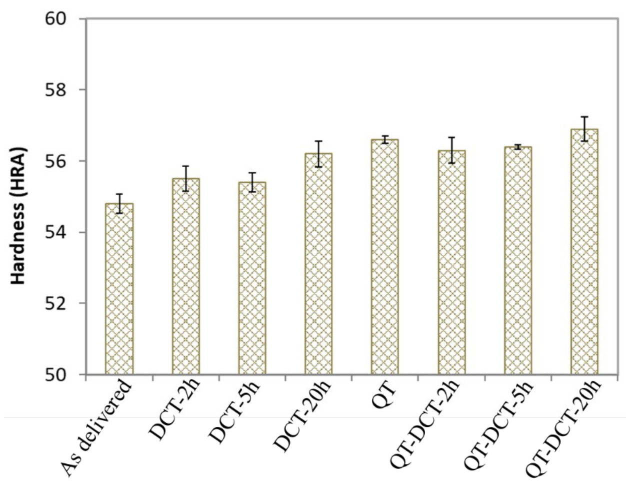
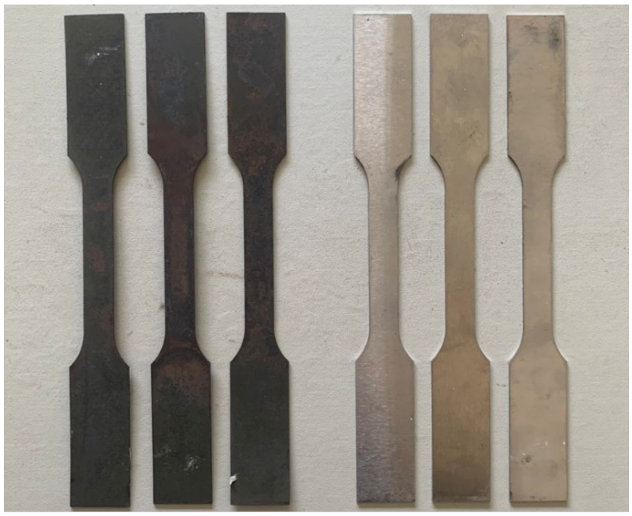


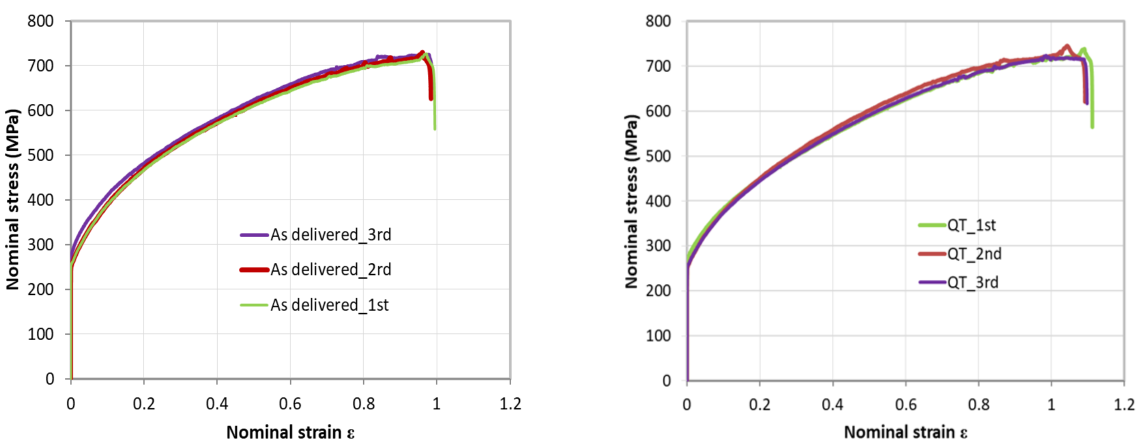
Fig. 4. Hardness after heat treatment process



After heat treatment   Before heat treatment

Fig. 5. Photograph of the specimens before and after heat treatment

Fig. 5 presents photographs of the specimens before and after heat treatment. It can be observed that the shape and dimensions of the specimens after heat treatment are nearly unchanged compared to their initial conditions. It can therefore be inferred that heat treatment does not significantly affect the size and shape of the specimens. Consequently, the influence of dimensional changes induced by heat treatment on the tensile test results can be considered negligible.

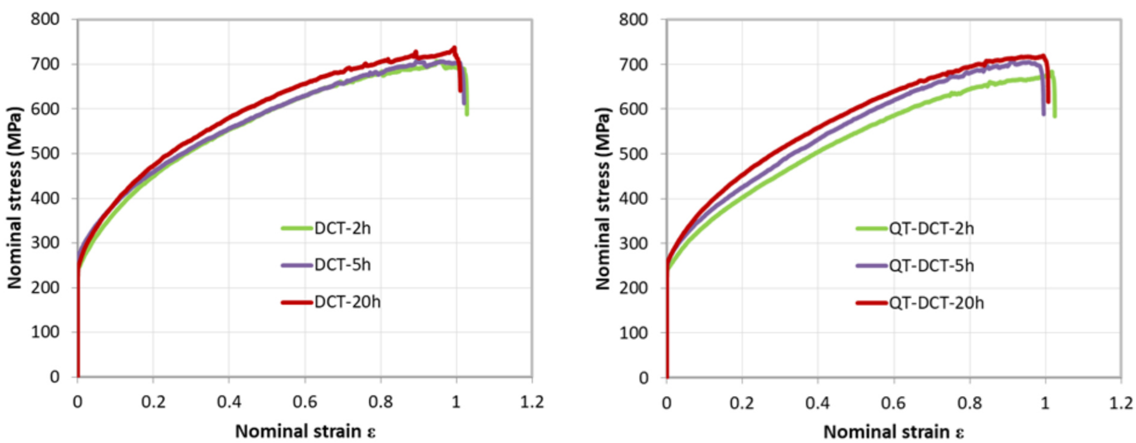


(a) As-delivered specimen   (b) After quenching

Fig. 6. Nominal stress–nominal strain curves (a) before and (b) after the quenching process

Nominal stress–nominal strain curves before and after quenching obtained from tensile tests are indicated in Fig. 6. The tensile test was repeated at least three times to confirm the reproducibility of the experimental results for each heat treatment condition. From Fig. 6, the repeatable results can be achieved for two cases without and with the quenching process. In addition, the obtained results of the stress-strain curve for the investigated steel are totally correspond to those in the research work of Meichuan et al. [25] for the same material. As a result, the validity of the experimental results obtained from the tensile test can be confirmed.

Fig. 7 shows the influence of the holding time of cryogenic treatment on the tensile properties in the case (a) without and (b) with the quenching process. As observed in this figure, the ultimate strength of the material improves slightly with an increase in the duration of liquid nitrogen exposure. For the QT-DCT specimens subjected to cryogenic treatment in combination with the heat treatment following the process in Fig. 2(b), the enhancement is more clearly indicated compared to the DCT specimens. However, the effect of the holding time of cryogenic treatment on the ductility of the investigated material is not clearly observed.



(a) Without quenching (b) With quenching

Fig. 7. Effect of the holding time of cryogenic treatment on the tensile properties (a) without and (b) with quenching

Fig. 8 shows the effect of DCT and quenching process on the tensile properties of type-304 austenitic stainless steel. As discussed above, the specimens immersed in liquid nitrogen for 20 hours might yield the best results among the three cases investigated regarding nitrogen immersion time. Therefore, the results from the DCT-20h and QT-DCT-20h specimens are used for further discussion in Fig. 8. The obtained stress-strain curves indicate that the heat treatment and cryogenic treatment have a negligible influence on the strength of the material. In contrast, the QT heat treatment demonstrates considerably better ductility compared to the other examined cases.

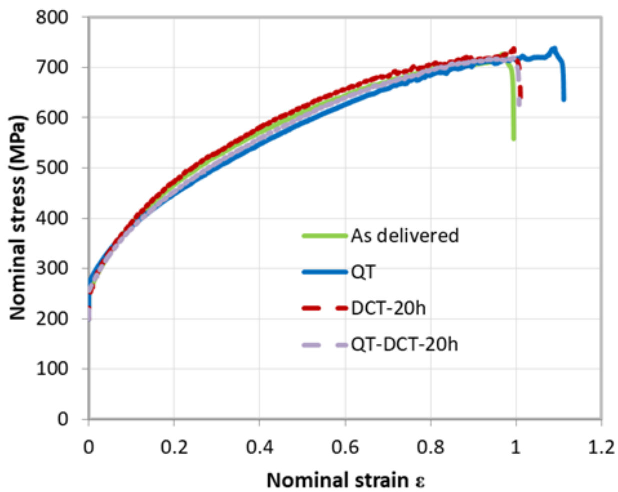


Fig. 8. Effect of DCT and quenching processes on the tensile properties

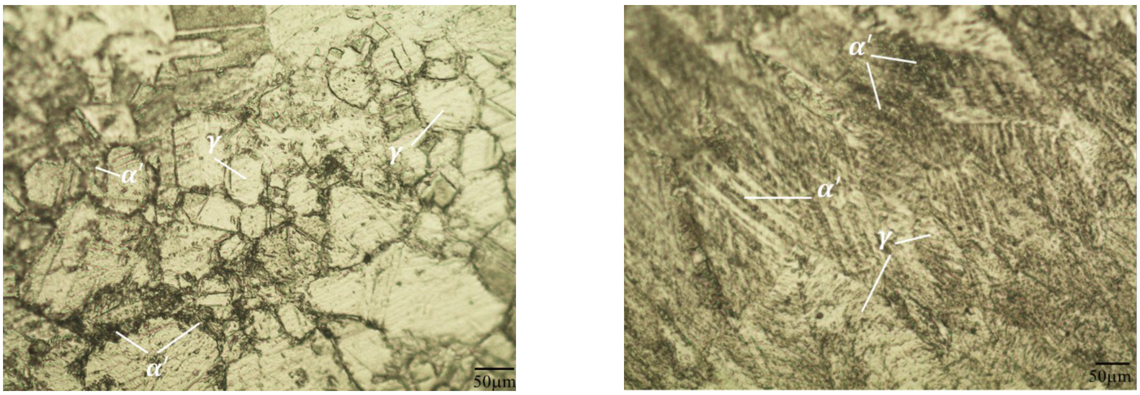
From the stress-strain curves obtained from tensile tests before and after heat treatment, the average values of three tests of yield strength, ultimate strength, and maximum nominal strain are presented in Table 3. From the data in this table, it can be observed that the QT specimen shows an improvement in mechanical properties compared to the as-delivered specimen in yield strength, ultimate tensile strength, and ductility by 12.16%, 1.59%, and 12.12%, respectively.

Table 3. Average values of three tests of yield strength, ultimate strength, and maximum nominal strain before and after heat treatment

	Yield strength (MPa)	Ultimate strength (MPa)	Maximum nominal strain (%)
As delivered	245.89	726.72	99
QT	275.79	738.27	111
DCT-20h	255.07	733.83	101
QT-DCT-20h	248.85	716.06	99

Fig. 9 shows the positions for measurement of hardness and microstructure observation of the material after tensile test. Position 1 is considered as a representative of the material in the clamping region. Position 2 is located around the region of fracture, where the material experiences significant plastic deformation.

Fig. 9. Positions for measurement of hardness and microstructure observation



(a) Position 1 (b) Position 2

Fig. 10. Microstructure of the material at different positions after quenching process (QT specimens)

Microstructure of the material at different positions after the quenching process is presented in Fig. 10. After tensile testing, the microstructure of the material at the clamping location (position 1) exhibits more twins compared to it before testing as shown in Fig. 3(b). The presence of the α'-martensite can be seen in Fig. 10(a). On the other hand, the material at position 2, where significant plastic deformation occurs, has a considerable modification in the microstructure with a large amount of α'-martensitic phase, as observed in Fig. 10(b). Therefore, it can be considered that the phase transformation from austenite into martensitic phase has occurred due to the mechanism of strain-induced martensitic transformation during plastic deformation of type-304 austenitic stainless steel.

Furthermore, X-ray diffraction results were taken at different positions of the specimen after tensile test in order to obtain additional evidence of phase transformation induced by plastic deformation mechanisms occurring in the investigated steel. The X-ray results for the quenched specimen after tensile deformation are presented in Fig. 11.

Obviously, the X-ray results indicate the presence of the martensite phase in the specimen after tensile testing at both examined positions. At position 1, the intensity peaks of both the austenite and martensite phases are pronounced, demonstrating the coexistence of these two phases as observed in the microstructural observation in Fig. 10(a). Meanwhile, at position 2, the intensity peaks of the austenite phase are relatively low, indicating that at this position, with a high amount of plastic deformation, most of the austenite phase has transformed into martensite due to the plastic deformation mechanism, leaving only a small amount of residual austenite. This exhibits a strong correlation between the X-ray results and the microstructural observations shown in Fig. 10.

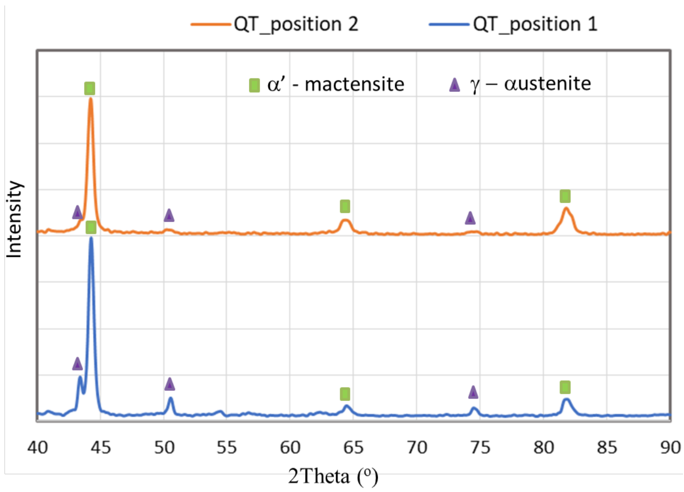
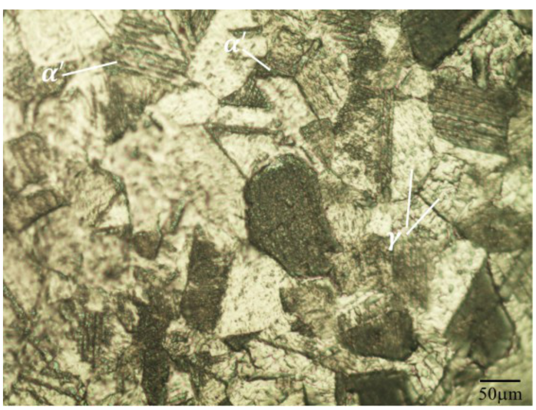
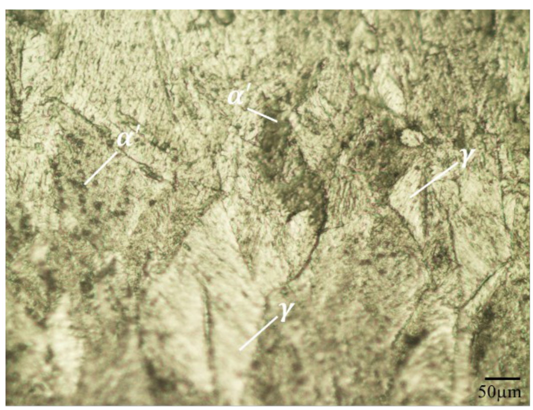


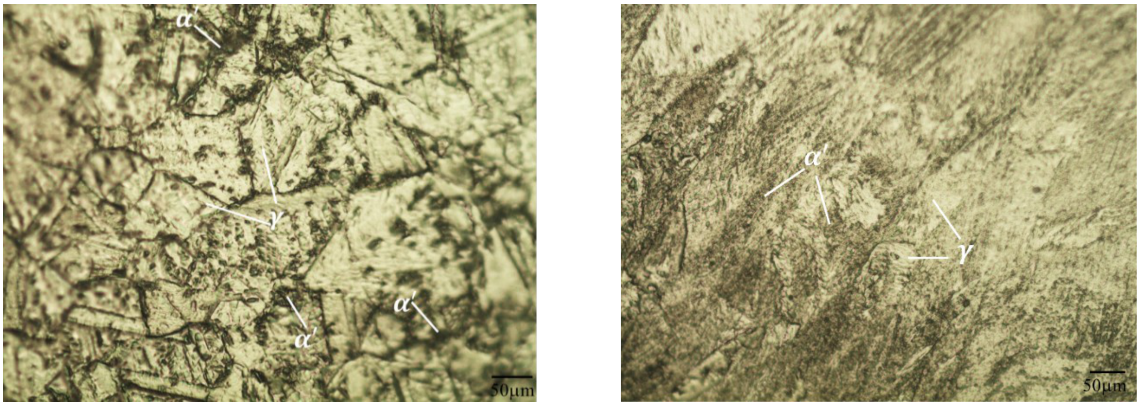
Fig. 11. XRD results of the material at different positions after quenching process (QT specimens)



(a) DCT-20h specimen - Position 1



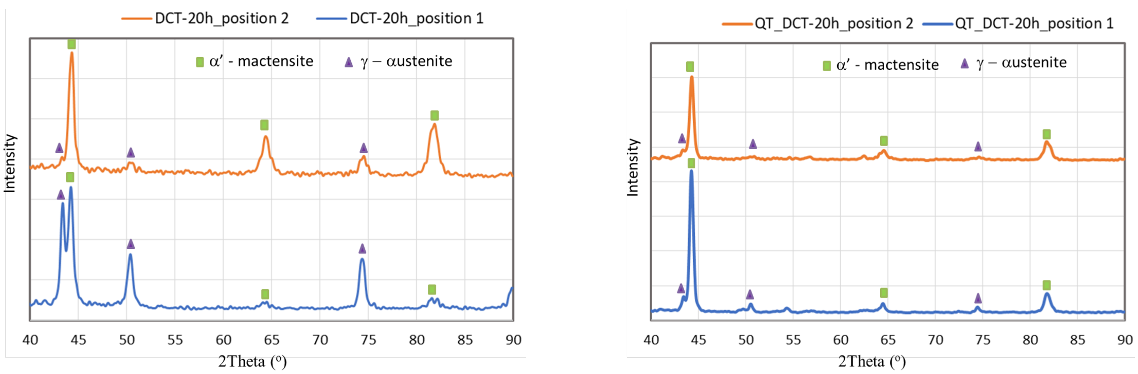
(b) DCT-20h specimen - Position 2



(c) QT-DCT-20h specimen - Position 1 (d) QT-DCT-20h specimen - Position 2

Fig. 12. Microstructure of the material treated in cryogenic environment at different positions before and after quenching process

Fig. 12 illustrates the microstructure of the material treated in cryogenic environment during 20 hours at different positions before and after quenching process. A similar phenomenon as indicated in Fig. 10 can be obtained for the specimen immersed in liquid nitrogen for 20 hours. The microstructure at position 1 after tensile testing is not significantly different from that observed before testing. Meanwhile, the large plastic deformation at position 2 has generated a considerable amount of martensitic phase, as seen in Fig. 12(b) and (d).



(a) (b)

Fig. 13. XRD results of the material treated in cryogenic environment at different positions before and after quenching process

Fig. 13 shows the XRD results of specimen after tensile test at different positions for the material treated in cryogenic environment before and after quenching process. The presence of both austenite and martensite phases was observed at two positions of the DCT specimen with and without quenching process. Fig. 13(b) indicates the indistinct intensity peaks of austenite at position 2, indicating a significant amount of martensite phase in the region of high plastic deformation for DCT specimen after quenching process. This phenomenon is quite similar to the results of the specimen with only quenching process as shown in Fig. 11. At the same time, the DCT specimen without heat treatment exhibits clear austenite peaks at position 2 in Fig. 13(b). These observations suggest that the quenched steel might induce a microstructure that is more favorable for martensite transformation because of the deformation-induced phase transformation mechanism.

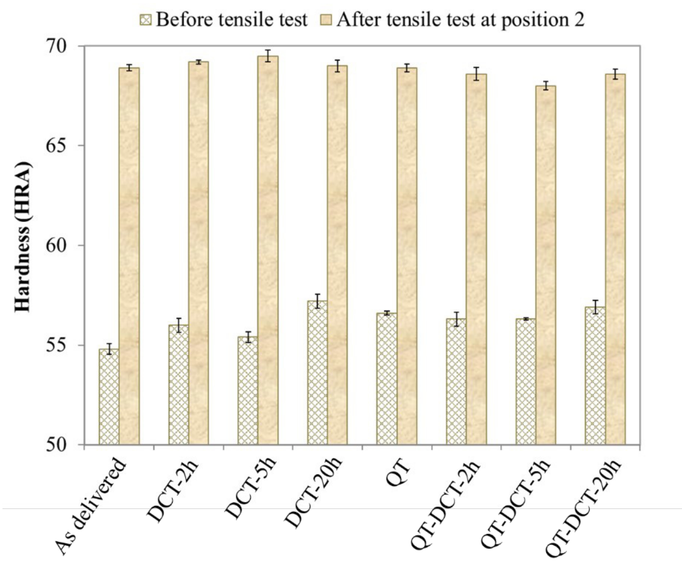


Fig. 14. Hardness of the material before and after tensile test at position 2

Fig. 14 indicates the value of surface hardness of the material in different heat treatment processes at position 2. From this figure, all investigated cases present a significant increase in the hardness when measured at position 2 compared to the results obtained before tensile test by approximately 15 HRA. However, the different heat treatment conditions show similar results that an improvement of the surface hardness of the heated-treatment material cannot be achieved compared to the as-delivered material. Thus, the enhancement in hardness of type-304 stainless steel is not attributed to the heat treatment process. As observed in the microstructural images in Figs. 10 and 12 as well as XRD results in Fig. 11 and 13, it is indicated that a large amount of martensitic phase is formed during the tensile test by the phase transformation mechanism induced by plastic deformation around position 2. The presence of a substantial amount of martensitic phase might play a vital role in increasing the hardness of the steel at position 2. Furthermore, the material around this region undergoes significant plastic deformation level, which may result in strain hardening of the material. Consequently, the hardness of the material at position 2 after tensile test is higher compared to that before tensile test.

4. CONCLUSIONS

This study investigated the influence of cryogenic treatment and heat treatment on the microstructural evolution and mechanical properties of type-304 austenitic stainless steel. The specimens are cryogenically treated in liquid nitrogen environment at -196^{o}C in cases with and without quenching process for different holding times. The experimental results indicate that the modifications in hardness and phase transformation cannot be achieved through the traditional heat treatment processes. In the cases tested in cryogenic environment, a longer immersion time in liquid nitrogen leads to a slight increase in the ultimate strength of the material. However, cryogenic treatment does not result in a significant enhancement of the tensile mechanical properties of the investigated material. Among the studied cases, the water-quenched heat treatment without cryogenic treatment shows a considerable increase in ductility compared to the material in its as-delivered condition or that which has undergone cryogenic treatment. Furthermore, there are significant differences in microstructure and hardness at different positions of the tensile specimen. A considerable amount of martensitic phase formed in the region of large plastic deformation in the tensile specimen due to the phase transformation mechanism induced during plastic deformation. The case with the quenching process indicated a greater amount of martensitic phase transformed during plastic deformation compared to the case without quenching process. Accordingly, the quenched specimen exhibited an improvement in mechanical properties compared to the as-delivered specimen in yield strength, ultimate tensile strength, and ductility by 12.16%, 1.59%, and 12.12%, respectively. Thus, although heat treatment hardly altered the microstructure and hardness of the investigated steel, this process can reasonably promote phase transformation during subsequent plastic deformation, leading to changes in the mechanical properties of the material.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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