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Influence of Heat Treatment on Strain-Induced Martensitic Transformation for Type-316 Stainless Steel

Thong Chung Nguyen[1], Hang Thi Pham[2]*, Nga Thi Hang Nguyen[2]

[1] *Faculty of Engineering,*
Vietnam National University of Agriculture, Trau Quy, Gia Lam, Hanoi, VIETNAM
[2] *Faculty of Mechanical Engineering*
Thuyloi University, 175 Tay Son, Dong Da, Hanoi, VIETNAM

*Corresponding Author: pthang@tlu.edu.vn
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Abstract

Type 316 stainless steel, renowned for its excellent corrosion resistance and formability, undergoes strain-induced martensitic transformation during plastic deformation, which significantly alters its mechanical properties. Understanding the role of prior thermal treatments in modulating this transformation is crucial for optimizing performance in structural applications. In this study, the influence of prior thermal treatment on the strain-induced martensitic transformation in SUS316 stainless steel is investigated. The steel is subjected to various heat treatment processes, including water quenching and deep cryogenic treatment at -196 °C for duration of 6, 10 and 24 hours. Then, the effect of heat treatment and deep cryogenic treatment on the microstructure and the mechanical properties of the material is examined, taking into account phase transformations during plastic deformation. The obtained results show that the grain size of the austenite changes considerably after heat treatment. The cases without heat treatment indicate lower strength and ductility compared to the cases with heat treatment. Among investigated cases of thermal treatment, the specimen with heat-treated and cryogenic treatment during 24 hours exhibits the best strength and ductility. This result indicates that the combination of heat treatment and prolonged cryogenic treatment using liquid nitrogen produced the most effective improvement for this steel. This can be explained by the formation of a larger amount of martensite phase during tensile testing for specimen after combined heat and cryogenic treatment.

1. Introduction

Among stainless steels, type - 316 stainless steel is widely applied in various industries due to their combination of excellent corrosion resistance and fairly good mechanical properties [1-3]. Numerous studies have proven that the metastable austenite phase in austenitic stainless steel might transform into martensite during plastic deformation due to strain-induced martensitic transformation mechanism, thereby affecting the mechanical properties of this steel [4-6]. According to Huang et al. [7], the parent phase in austenitic stainless steel can transform into either the α′ or ε - martensite phase during plastic deformation depending on the chemical composition of the steel, plastic deformation parameters, and stacking faults. Additionally, although the austenitic stainless steel cannot be hardened through a conventional heat treatment [8], Lieth et al. [9] indicated that the kinetics of strain-induced martensitic transformation are not only governed by deformation parameters, but are also strongly influenced by the prior heat treatment conditions.

Several studies have been conducted experimental works on phase transformation induced by plastic deformation of type - 316 stainless steel, taking into account the effect of prior heat treatment. Solomon et al. [10] demonstrated that strain-induced phase transformation strongly depends on the austenite grain size, which is primarily determined by the prior heat treatment process. According to this study, the stability of austenite decreases as grain size increases; thus, the martensitic transformation during plastic deformation becomes more favorable. Lieth et al. [9] investigated the effect of heat treatment and cold working on the corrosion behavior, surface hardness, and microstructure of the steel. Microstructural evolution and mechanical properties were studied as affected by cold deformation and annealing parameters in the research work of Wu et al. [11]. Moreover, Solomon et al. [12] further emphasized that strain-induced martensitic transformation markedly affects corrosion resistance of AISI316 stainless steel. Lv et al. [13] examined the mechanical properties and fracture toughness of the steel at different temperature during tensile test. Singh et al. [14] conducted the research work to explore the relationship between microstructural evolution and mechanical characteristics of type - 316 stainless steel during constrained groove press. Then, Kweon et al. [15] obtained stress-strain curve of AISI316 stainless steel at different temperatures and loading conditions. Although previous studies have investigated the influence of heat treatment on the mechanical properties of 316 stainless steel, taking into account strain-induced phase transformation, these efforts have primarily focused on conventional processes such as solution annealing. The effect of a combination of heat treatment and deep cryogenic processing has not yet been explored in these studies. Furthermore, the microstructure of the material after plastic deformation has not been thoroughly examined to observe the emergence of martensite phase formed due to strain-induced phase transformation, thereby leaving the underlying mechanisms by which heat treatment affects the microstructure and mechanical properties of type - 316 stainless steel unresolved.

On the other hand, deep cryogenic treatment (DCT) has been recently considered an extension of traditional heat treatment method to improve the mechanical properties of material [16]. According to the research work of He et al. [17], the grain size of the austenite phase might be affected by DCT, thereby influencing the martensitic transformation during plastic deformation. Zeng et al. [18] concluded that DCT can induce results of higher strength and ductility for 316L stainless steels fabricated by selective laser melting. Wang et al. [19] indicated that the excellent ultimate strength of 316L stainless steel synthesized by selective laser melting is attributed to the martensite transformation during cryogenic deformation owing to its low stacking fault energy at low temperature. Although recent research works have introduced DCT as a promising supplementary method to conventional heat treatments in order to improve mechanical performance of austenitic stainless steel, the mechanisms through which DCT affects the grain size, austenite stability, and subsequent martensitic transformation during plastic deformation are not yet fully understood. This research gap underscores the need for systematic investigations into the microstructural evolution and mechanical behavior of austenitic stainless steels subjected to combined heat treatment and DCT.

This research aims to investigate the influence of prior thermal treatment on the strain-induced martensitic transformation in type - 316 stainless steel. The steel is subjected to various heat treatment processes, including water quenching and DCT at different duration. Then, the effect of heat treatment and DCT on the microstructure and the mechanical properties of the material is investigated, taking into account phase transformations during plastic deformation. The formation of the martensite phase during plastic deformation, along with the combined effects of prior thermal treatment, is discussed based on microstructural characterization and X-ray diffraction (XRD) analysis. Based on the obtained experimental results, the mechanism for the improvement of mechanical properties can be explained for type - 316 austenitic stainless steel.

2. Methodology

2.1 Material

The steel used in this study was compositionally analyzed using a spectroscopic analyzer. The chemical composition results are presented in Table 1. From this table, the chemical composition of the steel contains a high amount of nickel, chromium and a significant amount of molybdenum. Therefore, the composition of the investigated material corresponds to that of grade SUS316 according to the JIS standard.

Table 1 *Chemical composition of the investigated material*

Element	C	Si	Mn	Cr	Ni	P	S	Mo	Cu	Fe
Wt. (%)	0.061	0.58	1.08	16.59	10.42	0.023	0.0085	2.07	0.25	Blan.

2.2 Experimental set-up

The standard tensile specimen used in this study has dimensions illustrated in Figure 1. The specimens are wire-cut from a plate with a thickness of 0.5 mm made of SUS316 stainless steel.

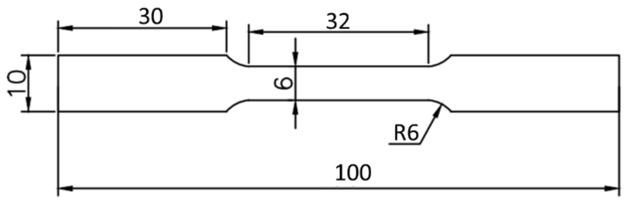


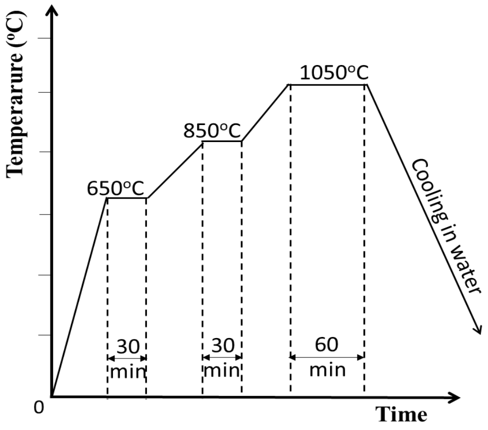
Fig. 1 *Tensile specimen*

The thermal treatment procedures are carried out in two main stages: conventional water quenching and DCT. In the water quenching process, firstly, the specimens are heated in stages up to a peak temperature of 1050 °C in the electrical resistance furnace device Nabertherm model LT5/14/B410. Upon reaching the target temperature, they are held isothermally for 30 minutes to ensure thermal homogenization. Then, the specimens are immediately removed from the furnace and rapidly quenched in water. Following the quenching step, selected specimens undergo DCT. Previous studies [20-21] have typically applied DCT to certain types of steel for durations ranging from 2 to 24 hours. Therefore, in this study, three representative durations of 6, 10 and 24 hours for the cryogenic treatment were selected to investigate the effect of cryogenic exposure duration on microstructural and mechanical properties. The cryogenic process is conducted using a liquid nitrogen cooling system at -196 °C. In addition, some specimens in the as-delivered condition were subjected to DCT. The thermal treatment conditions are detailed in Table 2. Temperature – time schematics of quenching process and quenching process with DCT are presented in Figure 2.

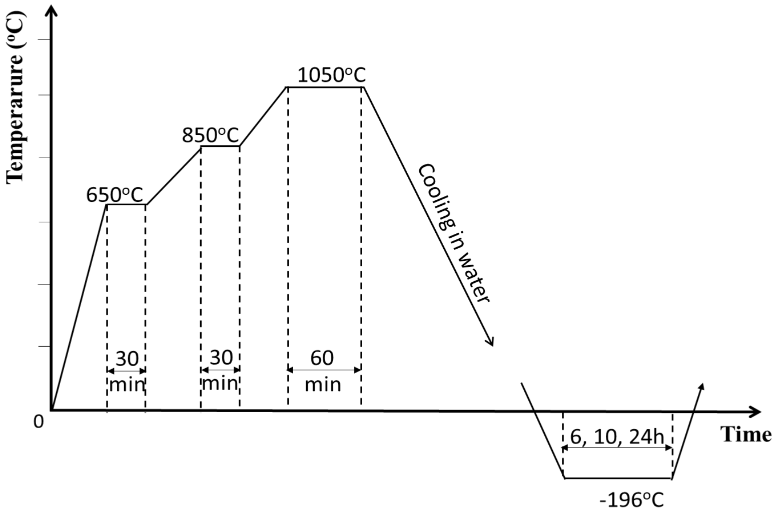
Table 2 *Thermal treatment conditions*

Specimen symbol	Heat treatment	DCT	Explanation
As-delivered	No	No	As delivered
HT	Yes	No	Quenching in water
HT_Cryo_6h	Yes	Yes	Quenching, then cryogenic treatment in 6 hours
HT_Cryo_10h	Yes	Yes	Quenching, then cryogenic treatment in 10 hours
HT_Cryo_24h	Yes	Yes	Quenching, then cryogenic treatment in 24 hours
Cryo_6h	No	Yes	Cryogenic treatment in 6 hours for as-delivered specimen
Cryo_10h	No	Yes	Cryogenic treatment in 10 hours for as-delivered specimen
Cryo_24h	No	Yes	Cryogenic treatment in 24 hours for as-delivered specimen

Hardness measurements of the steel are performed using hardness tester Rockwell Mitutoyo model HR-430MR. The tensile test is carried out by conventional tensile testing machine WA-1000C. The microstructure is observed using an optical metallurgical microscope Olympus GX41 after the specimens are etched using a mixture of solution HCl and HNO_3 in a 3:1 ratio, respectively. The phase composition of the material after tensile test is analyzed using X-ray diffraction (XRD, X'Pert Pro) equipment. The flowchart of specimen conditions in experiment is shown in Figure 3. The equipment used in experiment is presented in Table 3.



(a) Temperature – time schematic illustration of quenching process



(b) Temperature – time schematic illustration of quenching process with DCT

Fig. 2 *Temperature – time schematic illustration of quenching process and quenching process with DCT*

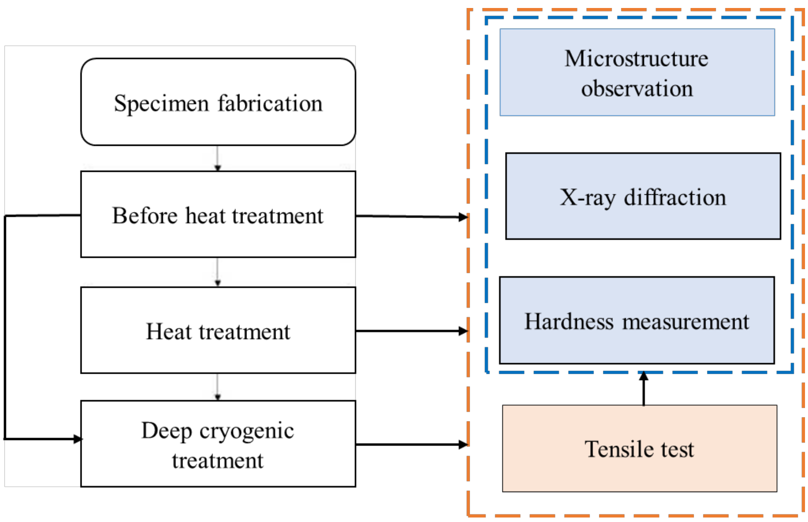


Fig. 3 *A flowchart of specimen conditions*

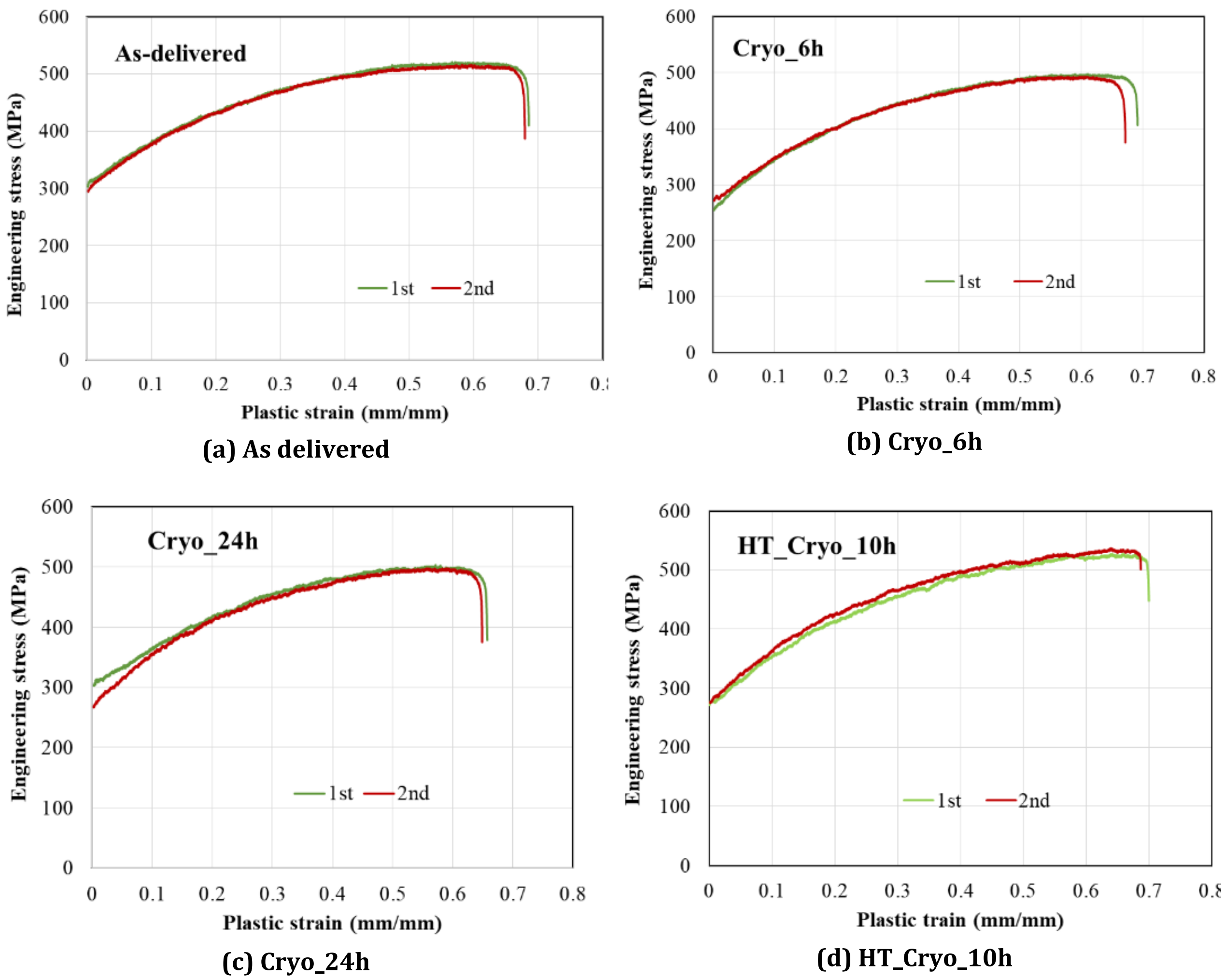
Table 3 *Equipment used in experiment*

Equipment	Model	Usage
Electrical resistance furnace	LT5/14/B410	Heat treatment
Liquid nitrogen container	YDS-3	Quenching in water
Rockwell hardness tester	HR-430MR	Hardness measurement
Tensile testing machine	WA-1000C	Tensile test
Optical metallurgical microscope	Olympus GX41	Microstructure analysis
X-ray diffraction equipment	XRD, X'Pert Pro	X-ray diffraction

3. Results and Discussion

3.1 Effect of Heat Treatment on Tensile Properties

Figure 4 shows the engineering stress - plastic strain relationship obtained from tensile tests conducted at a strain rate of 0.001 s^{-1} for the different cases with and without heat-treatment. To ensure the reliability of the experimental data, the engineering stress - plastic strain curve is repeated at least twice for every testing condition. From this figure, a good consistency and overlap among the repeated tests can be achieved, indicating reliable and reproducible results. Moreover, the results presented in Figure 4 (a) for the as-delivered specimen closely corresponds to the tensile strength and ductility reported by Kweon et al. [15] for SUS316 austenitic stainless steel tested at room temperature and quasi-static condition. This correlation further confirms the reliability and validity of the tensile test results.



(a) As delivered **(b) Cryo_6h**

(c) Cryo_24h **(d) HT_Cryo_10h**

Fig. 4 *Validity of the experimental results of tensile test*

Figure 5 illustrates the effect of DCT on the engineering stress – plastic strain curve of the non-heat-treated specimens. The non-heat-treated specimen without cryogenic treatment exhibits higher stress values. The yield strength and ultimate tensile strength of the non-heat-treated steel are roughly 300MPa and 520MPa, respectively. Meanwhile, the yield strength and ultimate tensile strength of the non-heat-treated steel with cryogenic treatment are about 275MPa and 500MPa, respectively. As a result, the strength of type - 316 stainless steel slightly decreases after DCT for cases without quenched treatment. Furthermore, as the cryogenic treatment duration increased, the strength remains relatively constant, while the ductility of the material exhibited a slight reduction.

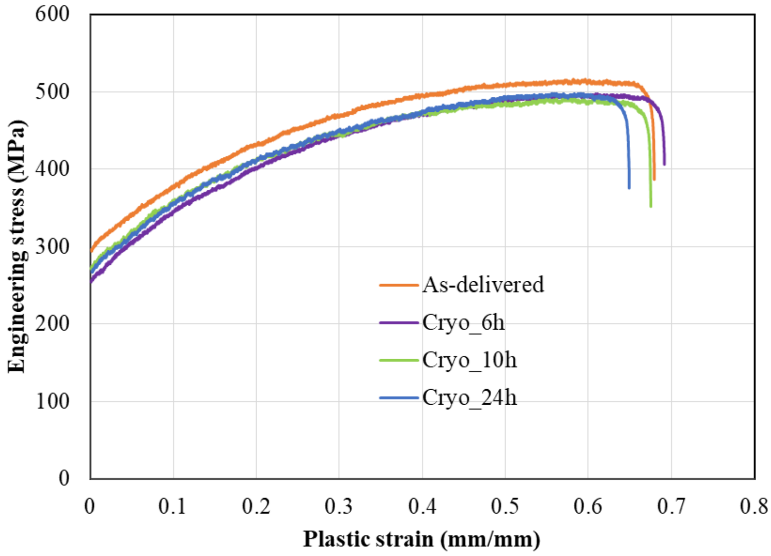


Fig. 5 *Engineering stress - plastic strain curve of different cases in DCT without quenched treatment*

Figure 6 presents engineering stress - plastic strain curve of different cases in DCT with quenched treatment. A slight reduction in stress values and maximum plastic strain is observed when the specimen is heat-treated and subjected to DCT in 6 and 10 hours, compared to the specimen without cryogenic treatment. Meanwhile, when the cryogenic treatment duration increases to 24 hours, the mechanical properties of the steel, including both strength and ductility are improved compared to the untreated cryogenic specimen. Thus, it can be said that the duration of DCT has a considerable effect on the strength and ductility of prior heat-treated steel.

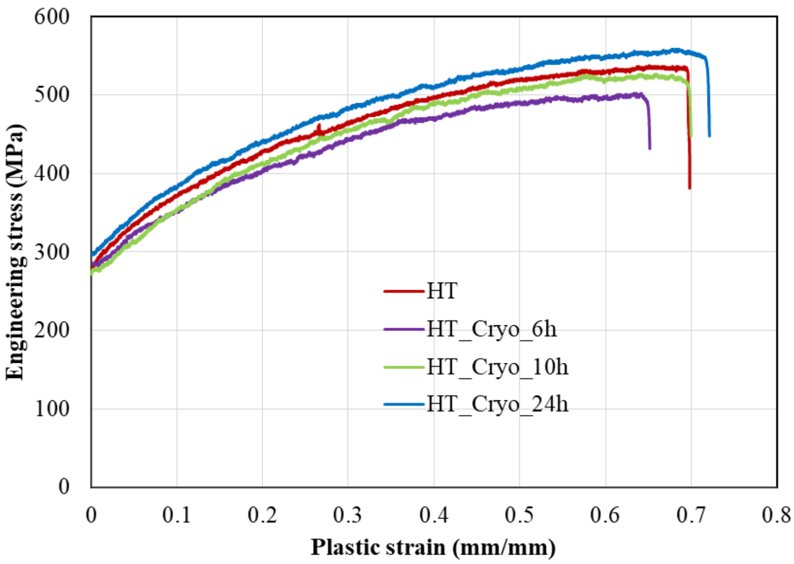


Fig. 6 *Engineering stress - plastic strain curve of different cases in DCT with quenched treatment*

Figure 7 shows engineering stress - plastic strain curve of different thermal treatment cases obtained from tensile test. From this figure, the cases without heat treatment indicate lower strength and ductility compared to the cases with heat treatment. All quenched specimens demonstrate higher yield strength and ultimate strength than the untreated ones. After quenching, the ultimate strength of SUS316 steel increases by approximately 50 MPa, while a slight improvement is observed in ductility. Notably, among investigated cases of thermal treatment, the specimen with heat-treated and cryogenic treatment during 24 hours exhibits the best strength and ductility. This result indicates that the combination of heat treatment and prolonged cryogenic treatment using liquid nitrogen produced the most effective improvement for this steel. Thus, the mechanical properties of the steel can be enhanced by an appropriate heat treatment process. The simultaneous improvement in both strength and ductility of type - 316 austenitic stainless steel is a notable advantage compared to other conventional steels. Therefore, it is expected that this material might exhibit excellent fracture toughness when it is subjected to an appropriate heat treatment process.

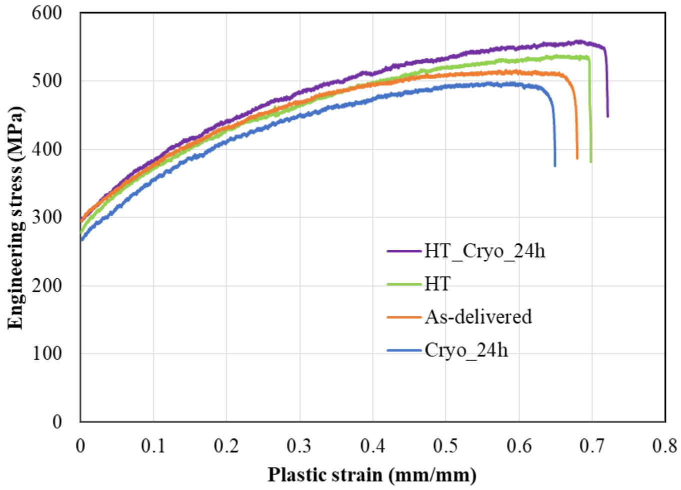
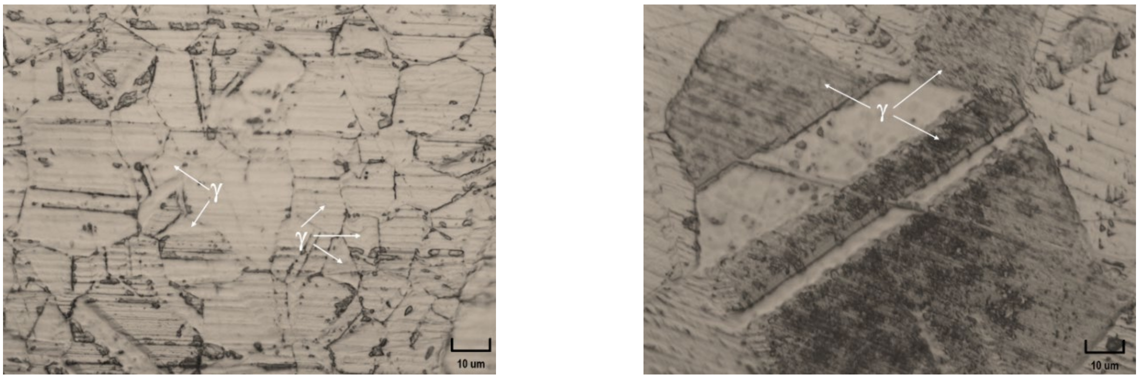


Fig. 7 *Engineering stress - plastic strain curve of different thermal treatment cases*

3.2 Microstructure of Material Before and After Tensile Test

3.2.1 Microstructure Before Tensile Test

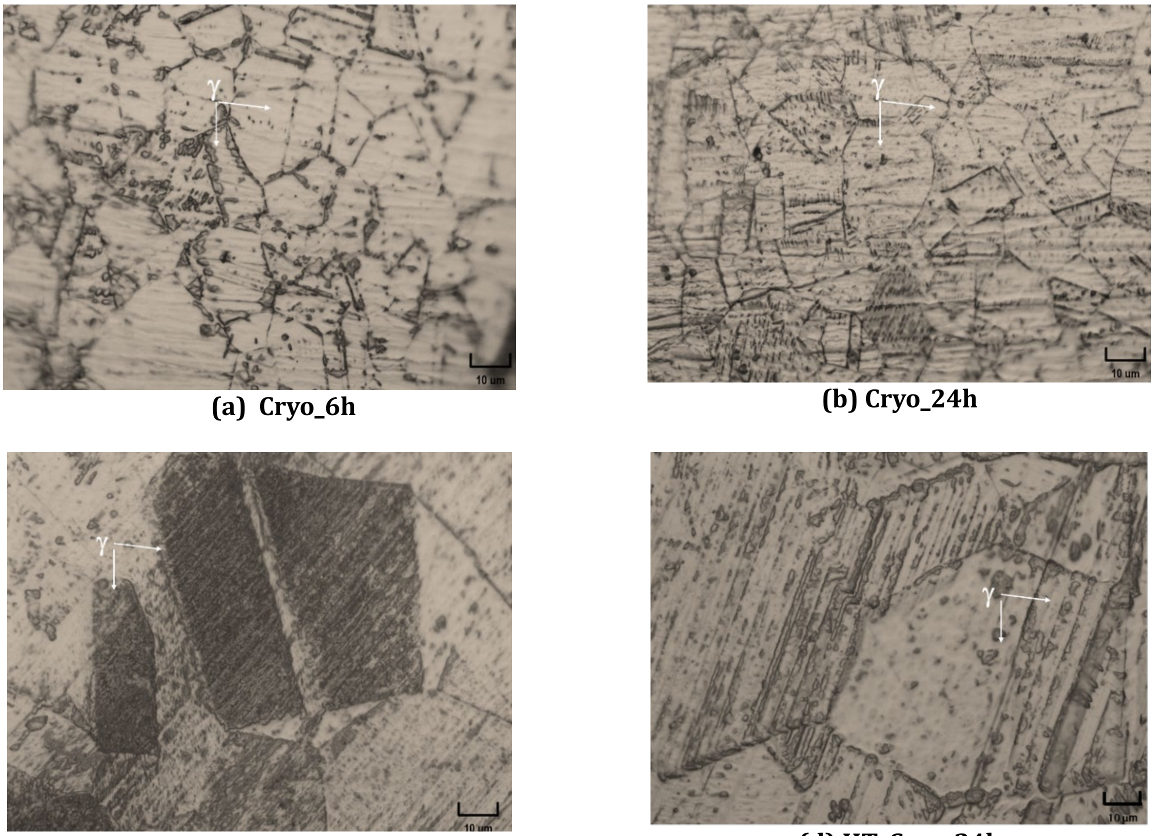
The microstructures of the as-delivered and water-quenched specimens are shown in Figure 8. It can be observed that the as-delivered steel primarily consists of the austenite phase. After water quenching, the microstructure of the steel does not exhibit any new phases other than the parent phase. However, the grain size of austenite phase after heat treatment is significantly larger than that in the as-delivered condition. Thus, it can be assumed that although phase transformation cannot be occurred during rapid quenching, the grain size of the parent phase changed significantly. According to a previous study by Solomon et al. [10], the phase transformation during plastic deformation is strongly influenced by the austenite grain size. Therefore, it is predicted that the mechanical properties of the material after heat treatment will be affected from those before heat treatment due to the martensitic transformation induced during plastic deformation.



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

Fig. 8 *Microstructure of the steel (a) As-delivered; and (b) After quenching condition*

Figure 9 shows the microstructures of the steel in different DCT conditions. The effect of cryogenic treatment duration on the microstructure of the steel cannot not clearly observed. For the non-heat-treated specimens subjected to cryogenic treatment, the microstructure remains predominantly austenitic, without new phase transformation compared to the as-delivered condition as shown in Figure 8 (a). Similarly, the microstructure of the heat-treated steel subjected to DCT remained homogeneous austenite, with a larger grain size compared to the non-quenched specimen. Thus, the martensite transformation might not occur during DCT for both the heat-treated and non-heat-treated specimens due to the stability of the austenite phase in type - 316 stainless steel.



(a) Cryo_6h **(b) Cryo_24h**

(c) HT_Cryo_10h **(d) HT_Cryo_24h**

Fig. 9 *Microstructure of the material after DCT with and without heat treatment*

3.2.2 Microstructure After Tensile Test

The microstructure observation and XRD analysis of the specimens after tensile test are performed at the region marked in Figure 10. This region locates near the fracture zone and experiences a significant plastic deformation level.

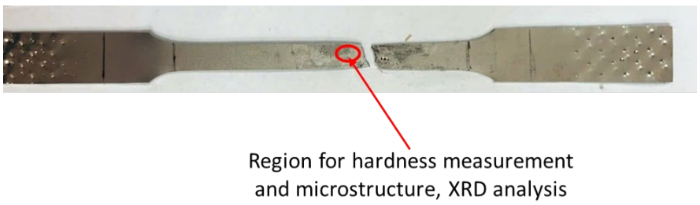
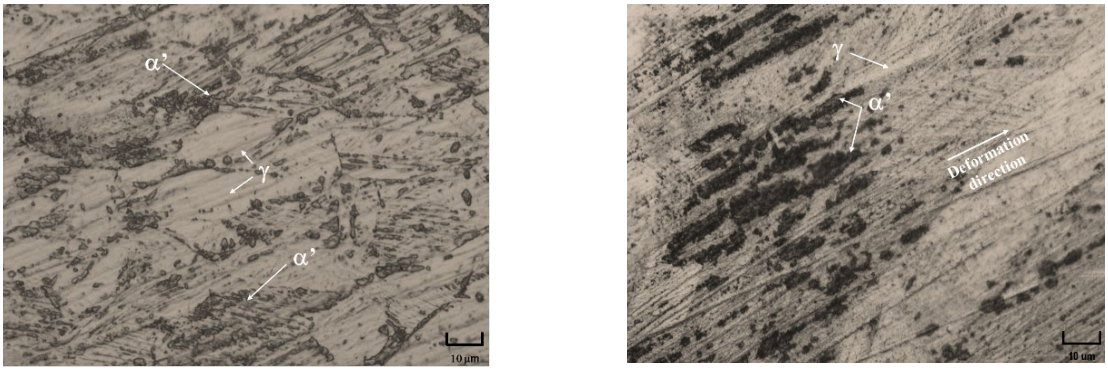


Fig. 10 *Region for microstructure observation and XRD analysis after tensile test*

Figure 11 shows the microstructures of SUS316 stainless steel after tensile test for both the as-delivered and the heat-treated specimen at the region marked in Figure 10. It is clearly seen that the microstructure of the steel after plastic deformation, as shown in Figure 11, differs significantly from that of the undeformed specimen presented in Figure 9. The formation of martensite phase can be observed in the steel after tensile test in the large plastic deformation regions of the as-delivered as well as quenched material. The α'- martensite phase might be formed along the slip planes aligned with the deformation direction of the specimen. In the heat-treated specimen, martensite formation is more clearly indicated, with a significant amount of martensite generated due to the plastic deformation process.



(a) Without heat treatment **(b) With heat treatment**

Fig. 11 *Microstructure of the steel after tensile test*

Figure 12 presents the XRD results of the specimen before and after tensile test. The XRD results of deformed specimens are taken at the region marked in Figure 10. The specimen before tensile test exhibited only austenite phase. This result is entirely consistent with the microstructural observations shown in Figure 8. On the other hand, all investigated cases after tensile test indicate that the phase composition of the steel in the regions with high level of plastic deformation consisted of austenite and α'-martensite phase. In detail, the steel without heat treatment also contained both austenite and α'-martensite phases, but the 2-theta peaks corresponding to the martensite phase were not clearly observed. The X-ray diffraction results are consistent with the microstructure observations shown in Figure 11. Based on these results, it can be concluded that the phase transformation can occur during the plastic deformation of type - 316 stainless steel. The amount of α'-martensite formed due to the strain-induced martensitic transformation mechanism in the heat-treated specimens might be significantly greater than that in the non-heat-treated specimens. This phenomenon can be explained by the difference in grain size of the parent phase after heat treatment, as above-discussed. As a result, the strain-induced martensitic transformation in SUS316 steel is strongly influenced by the heat treatment process, which in turn affects the tensile properties of the steel, as shown in Figure 7. The formation of a greater amount of martensite phase after tensile testing in heat-treated specimens, particularly those subjected to combined heat treatment and deep cryogenic processing, considerably improved the tensile properties of the investigated material. As illustrated in Figure 7, both the strength and ductility of the material post-heat treatment exceeded those of the untreated samples. Consequently, it is evident that although the heat treatment does not induce new phase transformations in 316 stainless steel, this process plays a crucial role in enhancing the material's strength and ductility by altering the austenite phase morphology, which in turn influences the martensitic phase transformation during tensile testing. Notably, the formation of α'-martensite due to plastic deformation contributed to an improvement not only in strength but also in ductility.

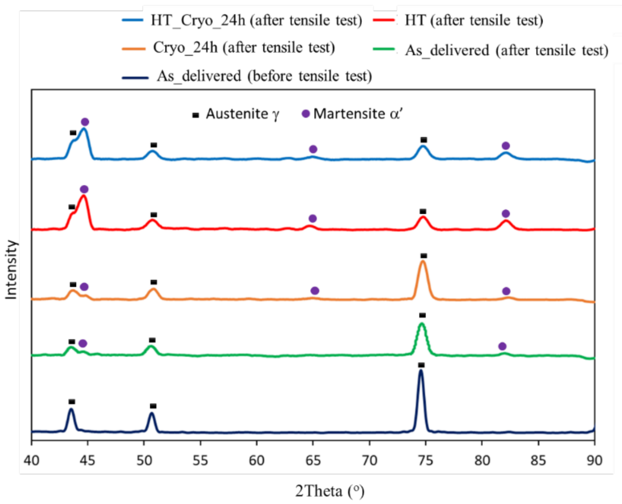


Fig. 12 *XRD results of the specimens before and after tensile test*

3.3 Hardness of Material Before and After Tensile Test

Figure 13 presents the hardness values of the material before tensile test under different thermal treatment processes. These values represent the average of at least three measurements taken at different locations on each specimen. An insignificant difference in hardness is observed among the cases of various heat treatment conditions. The hardness of the steel remained approximately 48-53 HRA. Thus, it can be considered that the heat treatment process cannot result in an improvement in the hardness of the steel, even though the material is subjected to rapid water quenching or DCT. This outcome can be explained by the absence of phase transformation during the heat treatment, as discussed in Figures 8 and 9.

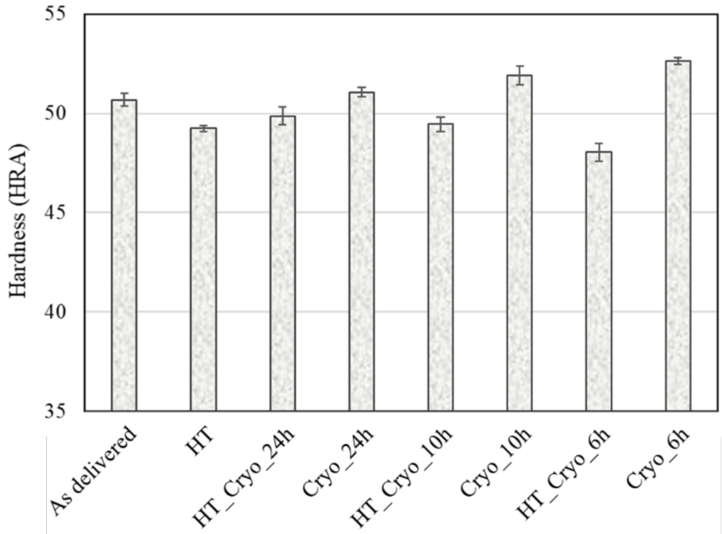


Fig. 13 *Hardness of the steel before tensile test*

Figure 14 shows the hardness results after tensile test measured at the region of the tensile specimens, as indicated in Figure 10. Overall, the heat-treated cases exhibit lower hardness values compared to the non-heat-treated cases after tensile test. Although a significant amount of α'-martensite formed during tensile test, the hardness measured at the region of high level of plastic deformation for the cases with heat treatment remains lower than that of the non-heat-treated steel. This can be explained that the α'-martensite phase is a ductile phase that might improve the strength of the steel but the hardness cannot be increased. On the other hand, the hardness values measured in this region for all investigated cases are higher than those of the steel before tensile test, as shown in Figure 13. The increase in hardness can be attributed to strain hardening of the material caused by plastic deformation during the tensile test.

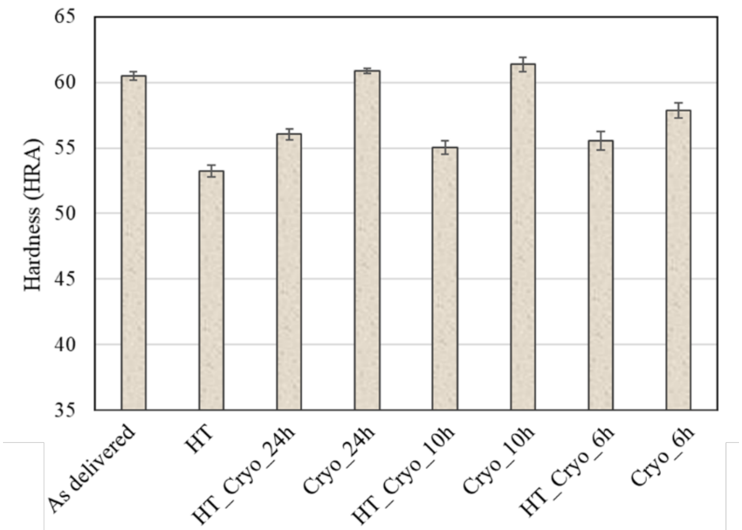


Fig. 14 *Hardness of the steel after tensile test*

4. Conclusions

This study investigates the effects of heat treatment and DCT on the strain-induced phase transformation and mechanical properties of 316 stainless steel. The obtained results show that phase transformation does not occur during heat treatment and deep cryogenic treatment. The microstructure of the steel before and after heat treatment consists predominantly of austenite phase. However, the grain size of the austenite changes considerably after heat treatment. The cases without heat treatment indicate lower strength and ductility compared to the cases with heat treatment. Among investigated cases of thermal treatment, the specimen with heat-treated and cryogenic treatment during 24 hours exhibits the best strength and ductility. This result indicates that the combination of heat treatment and prolonged cryogenic treatment using liquid nitrogen produced the most effective improvement for this steel. This improvement is attributed to the formation of a larger amount of martensite phase during tensile testing because of strain-induced martensitic transformation mechanism. Thus, heat treatment has a considerable influence on strain-induced martensitic transformation due to changes in the grain size of the parent phase. Despite the presence of α'-martensite, the hardness measured at regions of large plastic deformation in the heat-treated steel is lower than that of the non-heat-treated steel since α'-martensite phase formed during plastic deformation is a ductile phase.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Hang Thi Pham, Thong Chung Nguyen, Nga Thi Hang Nguyen;* ***data collection:*** *Hang Thi Pham, Thong Chung Nguyen;* ***analysis and interpretation of results:*** *Hang Thi Pham, Nga Thi Hang Nguyen;* ***draft manuscript preparation:*** *Thong Chung Nguyen, Hang Thi Pham, Nga Thi Hang Nguyen. All authors reviewed the results and approved the final version of the manuscript.*

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