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HEAT TREATMENT TECHNOLOGY FOR STAINLESS STEEL 316 (L)

Stainless steel 316 (also known in the literature as 1.4401) or 316L (also known as 1.4404) is alloyed steel with good corrosion resistance; so, it is widely used in the modern steel tools. However, its application is still limited due to some properties needed to be improved. This improvement, first of all, relates to the mechanical properties. The hardness value of stainless steel depends on its phase and constituent (alloying) elements. The increase of the hardness of stainless steel can be done by means of the engineering of microstructure and phases formed through the heat treatment. There are several variations in treatments such as annealing, normalizing, tempering, hardening, and cycling. Cyclic heat treatment is a heat treatment that is expected to increase the hardness value of stainless steel by maintaining the toughness of the steel. Cyclic heat treatment can be used as an efficient method in the future.

Keywords: mechanical properties, stainless steel 316, cyclic heat treatment, microstructure, toughness.

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1. Introduction

The development of state-of-the-art technology causes the use of stainless steel to be increasingly rapid because it has unique properties that are resistant to corrosion. Stainless steel is also the primary material used in the world nuclear power plants, especially in stainless steel that contains nickel alloys with austenitic stainless steel of 304 and 316 types [1]. Research of stainless steel continues to be conducted; so, data are found as a guideline in subsequent research in technology development [2]. Generally, stainless steel is a type of steel still associated with ferrous compound and the detail composition is in Table 1. It is explicitly designed to overcome corrosion in machinery that is often used widely in industrial, construction, and household appliances due to its excellent mechanical properties and corrosion resistance. Contemporary science still needs to improve the quality of stainless steel to get good mechanical properties [3]. The primary purpose of developing austenitic stainless steel 316 (1.4401) is corrosive resistant and applies in the marine industry, chemical industry, food processing units, and refineries. To improve corrosion resistance and the mechanical properties of stainless steel, molybdenum elements are generally added to the stainless steel 316 [3–5]. The stainless steel 316 has chemical composition: 16.47% Cr, 10.1% Ni, 1.97% Mo, 0.03–0.1% C, 0.53% Si, 1.42% Mn, 0.244% Co, 0.146% Cu, 0.03% W, 0.01% Nb, 0.03% P, 0.005% S and Fe (in mas.%) and has good mechanical and corrosion properties. However, its low hardness, wear resistance, and application are severely restricted. The microstructure of austenitic steel is stable at low temperatures due to the presence of different alloying elements [6]. This steel is also used in extreme environmental conditions at high and low temperatures. Some researchers have tried to minimize the wear and tear of 316L rust either by using advanced wear-resistant materials or surface treatment techniques. The wear and tear properties play an important role in determining the life cycle of each element of the machine. Failures in the machining process often occur due to wear and tear. Metal wear generally occurs caused by plastic distortion on surfaces and materials near the surface or by the release of particles. Wear can be classified into different categories, but the most common types

Table 1. Chemical composition of stainless steel 316 and 316L [8, 9]

Grade	Chemical composition (wt.%)						
	C	Mn	P	S	Ni	Cr	Mo
316	0.08	2.00	0.045	0.03	10–14	16–18	2–3
316L	0.03	2.00	0.045	0.03	10–14	16–18	2–3

are abrasive, erosion, metal-to-metal, fretting, corrosion, *etc.* This type of low carbon austenitic steel is formulated to limit chromium carbide formation, resulting in the depletion of chromium from the matrix (austenite), resulting in loss of corrosion-resistant properties. In improving the wear resistance and hardness of the material and without changing other desirable properties, hardening of the stainless-steel surface is the best and most economical method. Heat treatment is the optimal way to obtain optimal mechanical properties such as annealing, normalizing, tempering, hardening, and others [7]. This article briefly reviews some of the methods available to improve the mechanical and chemical properties of austenitic stainless steel.

2. Mechanical Properties and Improving Methods

Stainless steel is steel that can be applied to a variety of uses. This is because stainless steel has good mechanical properties and can resist corrosion. This relates to the process used in processing stainless steel and the number of alloy elements that can affect grain growth [10]. There are several grades in stainless steel, such as austenitic, martensitic, ferritic, and precipitation carbon [8]. Grades in stainless steels have different mechanical properties and can be obtained by quenching, tempering, normalizing, or annealing [11]. One popular type is austenitic stainless steel 316L (1.4404) because this steel has excellent corrosion resistance and good welding capabilities, so it is widely applied to industry especially in welding. Heat treatment is a process used to change the structure of metals by heating test specimens at the furnace, at a specific temperature, and at a particular time. The result of heat treatment is also greatly influenced by the cooling process, which includes the rate of cooling, resistance time, and fluid used in the cooling process. This heat treatment process dramatically affects the structure of metals and the mechanical properties of the materials formed. Heat treatment also consists of several types, including quenching, annealing, normalizing, and tempering [11]. Heat treatment in stainless steel is generally not the same as carbon steel because it has different constituent element content. The most used treatment in stainless steel is quenching and annealing methods. Usually, the heat treatment method is accompanied by a combination of parameters such as resistance time and heating that is done cyclically. Stainless steel in austenitic phase (FeCr) must be heated above 1035 °C This is intended so that carbide deposition does not occur during the austenite phase during the cooling process, and then continued by quenching with water media. Other heat treatments to reduce surface voltage with annealing techniques at temperatures of 345 to 455 °C aim to improve the mechanical elasticity properties of stainless steel or also known stabilization treatment meth-

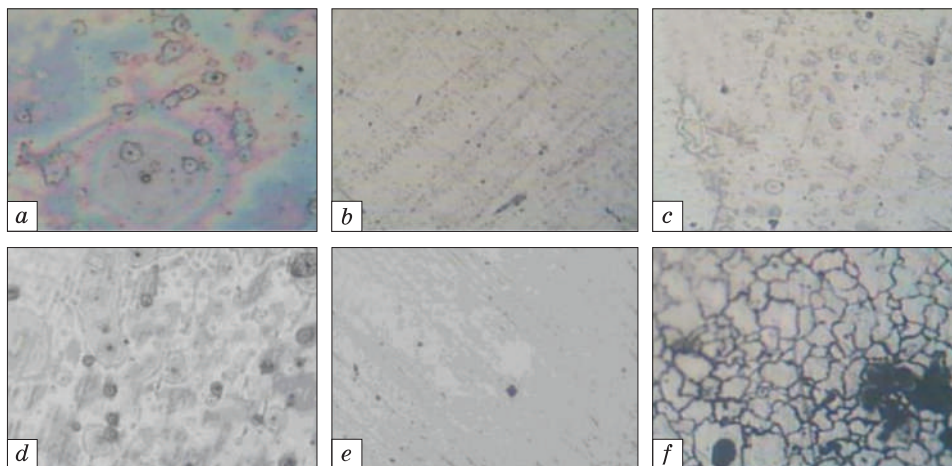


Fig. 1. The photomicrographs of the normalized specimens of 316L stainless steel: *a* — sample without treatment; *b* — heated to 750 °C held for 8 hours; *c* — heated to 800 °C held for 8 hours; *d* — heated to 850 °C held for 8 hours; *e* — heated to 900 °C held for 8 hours; *f* — heated to 950 °C held for 8 hours [12]

ods are carried out at temperatures of 870 to 900 °C to form titanium and niobium that are stable in steel grade 321 or 347 and prevent the occurrence of deposition (FeCr). Moderate heating between 485 and 815 °C is not recommended in austenite as it can make stainless steel vulnerable to rusting and bitterness [8]. Martensite in stainless steel is obtained by heat treatment at 760 to 815 °C by slow cooling method or by quenching process with oil media at a heating temperature of 1000 °C [8].

Normalizing was carried out using temperature variations as follows: 750, 800, 850, 900, and 950 °C, and with resistance time for 30 minutes, 1 hour, 2 hours, and 8 hours; obtained microstructures are in Fig. 1 [12]. The data obtained can be known based on the morphological structure of 316L stainless steel; when heated at a temperature of 750–850 °C and a resistance time of 30 min–2 h, steel undergoes sensitization, which decreases the corrosion-resistant properties of stainless steel caused by deposition of carbide containing much chromium (Cr) elements causing interstitial carbon to diffuse rapidly to the grain limit. However, slower diffusion of chromium results in depletion zones that result in steel being susceptible to intergranular corrosion. At a temperature of 900 °C, sensitization is observed at a resistance time of 1–2 hours before the normalization process. Increased resistance time leads to desensitization process (return of corrosion-resistant properties in steel after prolonged heat treatment). As can be observed in morphological temperature treatment at 750–900 °C, desensitization occurs at a time of resistance of 8 hours and in heat treatment at a temperature

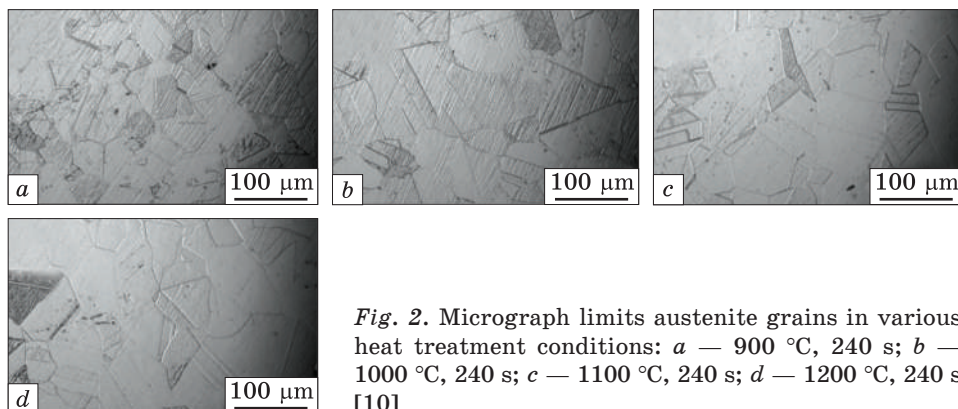


Fig. 2. Micrograph limits austenite grains in various heat treatment conditions: *a* — 900 °C, 240 s; *b* — 1000 °C, 240 s; *c* — 1100 °C, 240 s; *d* — 1200 °C, 240 s [10]

of 950 °C, sensitization can be observed at a time of 30 minutes, resistance and desensitization take place at a resistance time of 1–2 hours. At a resistance time of 8 hours, there has been complete desensitization in 316L stainless steel [12]. At heat treatment at temperature of 950 °C, the slow diffusion of Cr can be accelerated with thermal assistance; so, the initial sensitization is cancelled. According to Table 2, the hardness of normalized 316L stainless steel decreases with the addition of resistance time and normalization temperature [12].

The experiment was performed on a 316L austenitic stainless-steel specimen with the initial stage of this experiment was to conduct chemical composition testing using a Bruker Q4 Tasman machine [10]. Then, the heat treatment process is carried out at 900, 1000, 1100, and 1200 °C, and the resistance time of 30, 60, 120, and 240 s, which is continued at cooling at room temperature [10]. Based on this study, the results that the size of stainless-steel grain 316L increases along with the increasing of temperature and holding time during the heat treatment process can be observed in Fig. 2. Graphs of the relationship between grain size and holding time is increasing at 1200 °C, which can be seen in Fig. 3 [10]. Different constituents control the development of austenite grains in microalloyed steel; in this case, it involves temperature and austenitization time factors, heat work history, chemical composition, initial grain size, and heating range at austenitization temperature [10].

Experiments were conducted on the samples of austenitic stainless steel (ASS) types 304 and 316, which are aimed to determine the effect

Table 2. Brinell hardness test sample [12]

S/N	Samples	Hardness, <i>HB</i>	S/N	Samples	Hardness, <i>HB</i>
1	Untreated sample	187	4	850 °C held for 8 h	143
2	750 °C held for 8 h	166	5	900 °C held for 8 h	110
3	800 °C held for 8 h	161	6	950 °C held for 8 h	–

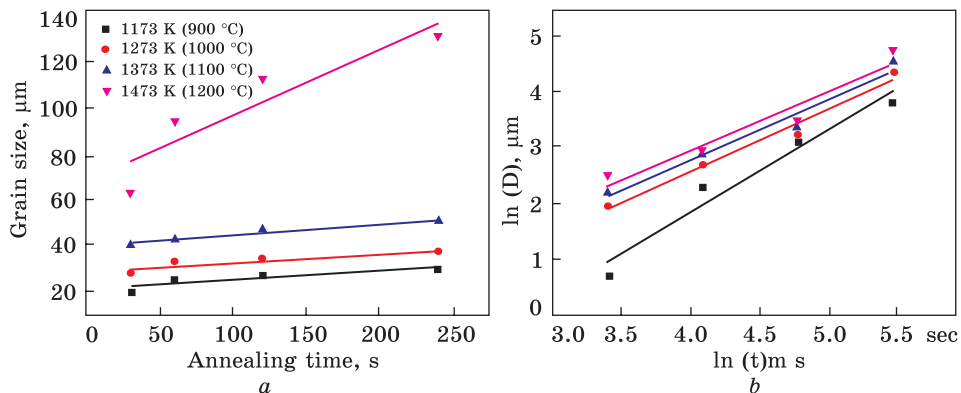


Fig. 3. Grain size vs. annealing time (a) and isothermal grain growth (b) at 900–1200 °C for the 316L austenitic stainless steel [10]

of temper temperature and holding time in oxalic acid medium against corrosion resistance properties in stainless steel 304 and 316 by using the weight-loss calculation method [13]. Stainless steel is a type of steel with high corrosion resistance, but several factors affect the rate of corrosion and resistance properties in stainless steel namely natural properties, chemical alloys, heat treatment, and factor from environment. According to Fig. 4, the corrosion rate in stainless steel types 304 and 316 increases with increasing heat-treatment temperature and prolonged tempering time, caused by carbide precipitation at the grain limit of both metals [13]. Type 304 stainless steel is more rationed against corrosion than type 316 stainless steel due to the content of molybdenum elements found in the 316-type steel [13]. The microstructure of temper conditions consists of uneven carbide phases and retained austenite, which can cause galvanic effects when soaked in oxalic acid media.

Experiments were conducted on a type of 316L stainless steel aimed at improving corrosion resistance and mechanical properties. The heat treatment process is carried out at temperatures (450–500 °C) to avoid the formation of chromium carbide at high temperatures (≈ 950 °C), which can drain chromium from the microstructure of steel and can significantly reduce corrosion resistance. This research method relies on parameter kinetics, which refers to substances such as Cr and Ni diffusing more slowly than interstitial solutes such as carbon [4]. Based on the data, it can be known that the treatment of interstitial hardening (IH) significantly increases the hardness, tensile strength, and corrosion of cracks, fatigue, pull, and pain resistance of treated 316L-type stainless steel compared to untreated alloys. The importance of this achievement cannot be overstated, as it is rare to find material modification technologies that provide simultaneous benefits in the properties of me-

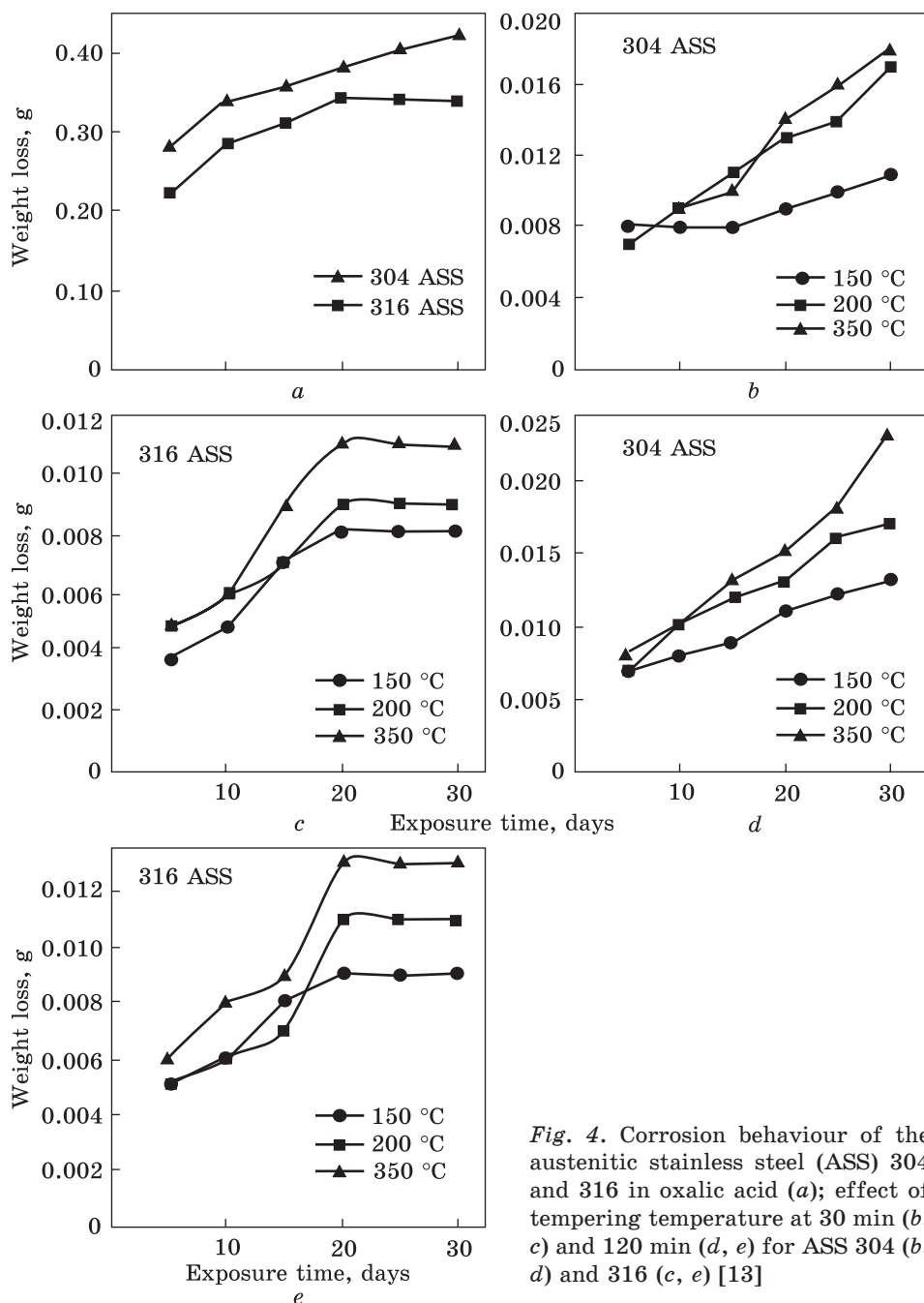


Fig. 4. Corrosion behaviour of the austenitic stainless steel (ASS) 304 and 316 in oxalic acid (a); effect of tempering temperature at 30 min (b, c) and 120 min (d, e) for ASS 304 (b, d) and 316 (c, e) [13]

chanical performance and corrosion. Specifically, the study showed that 316L-type stainless steel treated with IH provided increased hardness (by a factor of 3.2) potential initiation of fissure corrosion (600 mV). It

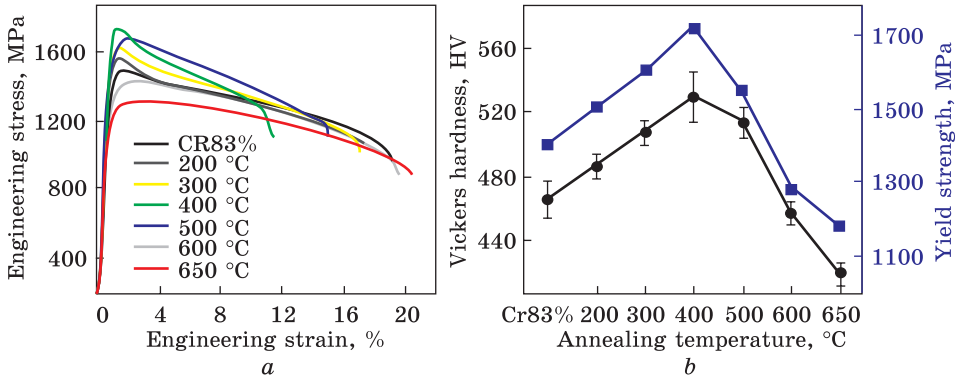


Fig. 5. The stress–strain curves for pre- and post-annealed samples of coarse-grained 316L stainless steels cold rolled with a thickness reduction of $\approx 83\%$ (CR 83%) (a). The annealing temperature dependent hardness and yield strength for CR 83% and post-annealing samples (b) [14]

has also been shown that martensite in coldly worked fastener threads is susceptible to corrosion attacks if treated under standard IH treatment conditions [4]. Experiments conducted on 316L stainless steel specimens with a commercial 316L stainless steel chemical composition used in the study: 16.47% Cr, 10.1% Ni, 1.97% Mo, 0.03% C, 0.53% Si, 1.42% Mn, 0.244% Co, 0.146% Cu, 0.03% W, 0.01% Nb, 0.03% P, 0.005% S and Fe (in % balance mass) [14]. The initial thickness of the 316L stainless steel sheet of rough-grained steel obtained is of 10 mm. Cold rolling is done at room temperature. Thickness was reduced to 1.7 mm with a total thickness reduction of 83%. The 316L stainless steel cold microstructure can be considered a heterogeneous nanostructure, *i.e.*, flattened rough grains sandwiched between a mixture of nanograins and nanotwins. Nanostructured samples were annealed at 200, 300, 400, 500, 600, and 650 for 5 hours. Based on the data obtained in the image, it can be concluded that the strong hardening effect caused by annealing was found in a sample of CR 83% 316L stainless steel for the first time. With annealing at 400 °C for 5 hours, the yield strength and *HV* increased to 1720 MPa and 530 *HV* compared to 1400 MPa and 466 *HV*, respectively, in the CR 83 pair [14].

The investigation showed that the yield strength and hardness from the 316L stainless steel sample have a linear relationship in the range of 7.5% and 47.9%, respectively, suggesting that was the dominant mechanism for the hardening effect observed after annealing. Based on this research, annealing parameters have a significant role in determining the hardness of stainless steel 316L can be observed in Fig. 5, b. The optimum hardness level is at 400 and then turns down as it passes at 400 °C.

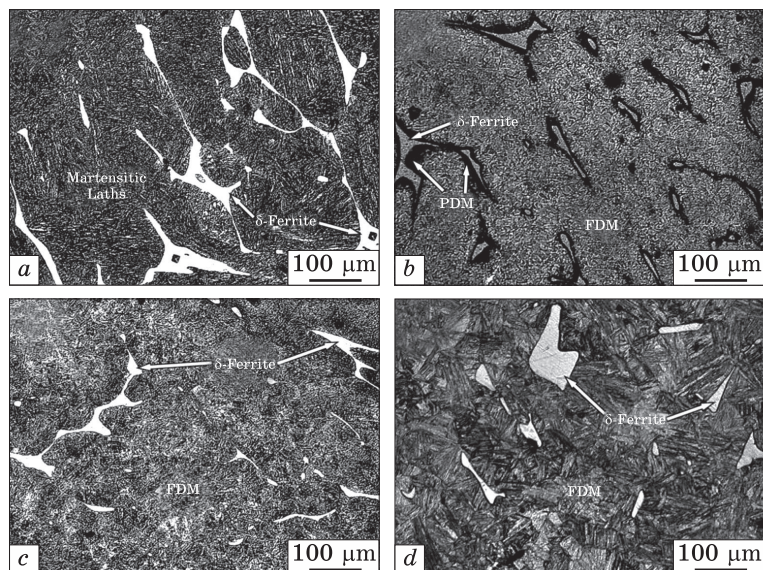


Fig. 6. SEM images of martensite and ferritic features observed for the: *a* – 13-4 ASR; *b* – TCHT-850; *c* – TCHT-950; *d* – specimens of TCHT-1050 [5]

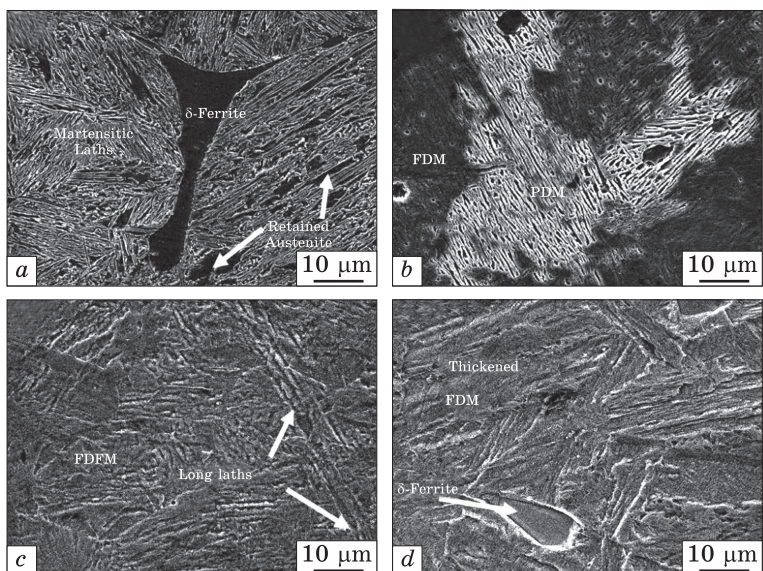


Fig. 7. SEM micrographs of the as-received and TCHT processed samples. Here, the dissolution of martensite and the refinement of martensitic blocks along with the temperature of thermal cycling are observed in 13-4 ASR (*a*), TCHT-850 (*b*), TCHT-950 (*c*), and TCHT-1050 (*d*) [5]

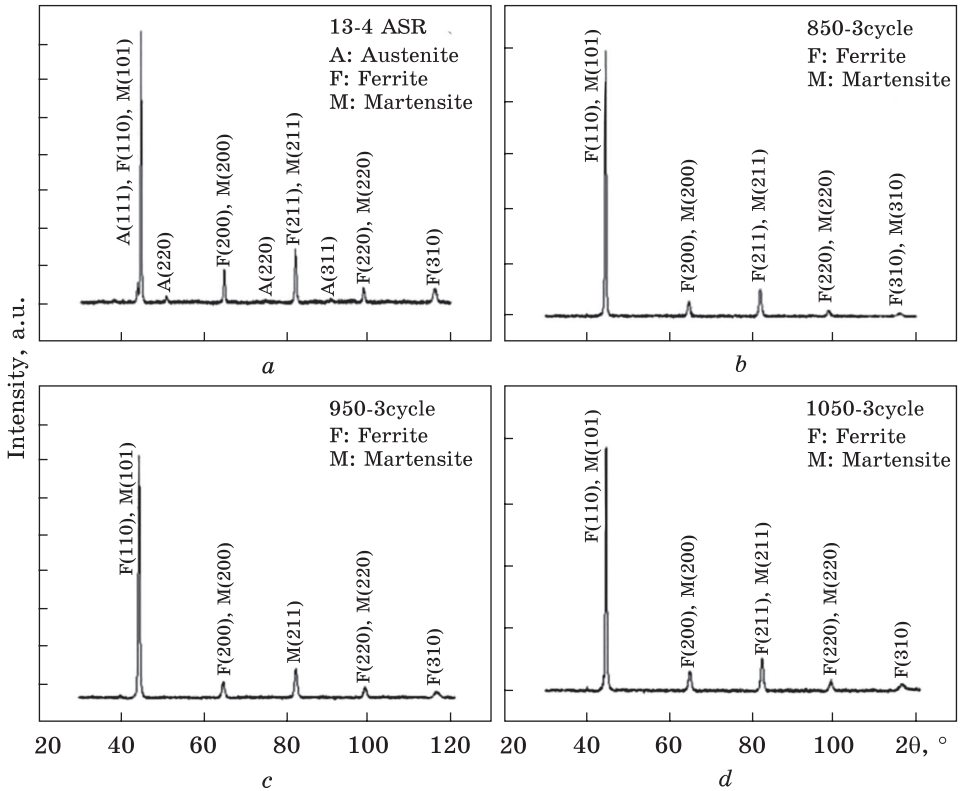


Fig. 8. The XRD analysis of the as-received samples and after thermal cycling heat treatment: *a* – 13-4 ASR, *b* – TCHT-850, *c* – TCHT-950, *d* – TCHT-1050 [5]

Based on the data obtained, the specimen morphological structure can be observed with the results of specimen characterization and mechanical properties of stainless-steel martensitic specimens [15]. Generally, modification of chemical composition is one way to increase the oxidation resistance of high-temperature martensitic stainless steel. Chromium levels of less than 18% have resistance to temperatures below 816 °C. Chromium levels between 18–20% are used at 982 °C. Chromium levels up to 25% have sufficient oxidation or scaling resistance [15]. Based on data from optical and scanning electron microscope (SEM) micrographs from specimens shown by Figs. 6 and 7, as can be seen, there are martensite blades, elongated δ -ferrite colonies, and fractions of small δ -ferrites, for the martensite lath phase can be seen the presence of austenite held between the blades in small quantities in Fig. 7, *a*. The microstructures in thermal cycling heat treatment (TCHT)-850 are shown in Figs. 6, *b*, and 7, *b*. There are three regions: fully dissolved martensite (FDM), partially dissolved martensite (PDM), and a few fragmented ferrites. In SEM testing on Fig. 7, *b*, PDM can be clearly ob-

served. Observable FDM contributes up to 85% in microstructure to the TCHT-850 treatment. The micrograph structure for TCHT-950 is shown in Fig. 6, *c*, indicating the presence of two regions in the microstructure, namely fully dissolved fine martensite (FDFM), and the slightly fragmented ferrite. Figure 7, *c* shows SEM images on TCHT-950 observed there are several blocks containing long martensite at along with partnerships and packages that have fragmented martensite slats. At this stage, there is also no PDM phase fraction in the TCHT-950. The fraction of martensite content in microstructures in the form of FDFM rose to 94, and delta-ferrite dropped to 6.3. The evolution of microstructures in TCHT-1050 as shown in Figs. 6, *d* and 7, *d*. Figure 7, *d* shows two regions of the microstructure: FDM and fragmented ferrite [5]. The x-ray diffraction (XRD) patterns for specimens conducted at raw are shown in Fig. 8, which is indicating the existence of δ -ferrite phase (b.c.c.), martensite phase (b.c.t.) and austenite phase (f.c.c.) restrained. The intensity of austenite is low at the peak; so, the austenite phase in 13%Cr-4%Ni (13-4) as-received (ASR) steel is negligible. TCHT-850 specimens were observed not to contain the austenitic phase (f.c.c.), however, included martensite (b.c.t.) and ferrites (b.c.c.). In samples of TCHT-950, the ferrite phase was reduced, the ferric peaks (211) of TCHT-950, and (310) of TCHT-950 and TCHT-1050 disappeared [5]. Note that the effect of structural defects [16–21] (*viz.*, vacancies) in the b.c.t. iron nitride annealed and rapidly quenched may be crucial for manipulation some physical properties [22–24].

The results of mechanical properties can be seen in the graph above. This study aims to obtain an excellent mechanical structure and properties of stainless steel 13-4 ASR with the increased strength of the material. Based on research, it can be concluded that the steel depends on the amount of austenite and delta-ferrite retained, block size, and blade size but in the reverse mode of hardness and ultimate tensile strength (UTS). Based on the data obtained, the highest microhardness (437 *HV*) was obtained in the martensitic phase of the TCHT-1050 specimen for the highest bulk hardness in TCHT-950 (413 *HV*), while the highest UTS value at 1317 MPa and 25% was achieved at TCHT-950, so, the TCHT-950 specimen has obtained the best combination of UTS, hardness, and tactility. TCHT cyclic treatment can be the best solution for improving mechanical properties (UTS, hardness, and tenacity) compared to other methods.

Based on the topic of this literature review, several heat treatment methods can be found that can affect microstructures and mechanical properties in stainless steel 316. The main goal is to find a suitable way which it can produce good mechanical properties. In general, the application of stainless steel in the industry is widely found in the main engine components such as beaters on hammer and disk mill that are

likely to occur collisions or friction directly with working materials. Stainless steel was chosen because it has a high resistance to corrosive properties, reduce engine maintenance costs, and replace components damaged due to corrosive. But in its application, stainless steel can fail mechanically and wear out due to the low hardness and toughness, which can make the lifetime of components becomes shorter [25, 26]. Such mechanical failures can be overcome by increasing the mechanical hardness and toughness of stainless steel without reducing resistance to corrosion. Some of the above methods of hardening with cyclic heat treatment become the most compatible method in answering the above problems so that it can produce stainless steel properties that have optimum hardness without reducing the toughness of steel.

3. Conclusion

Stainless steel is one of the popular types of steel and is widely applied in various equipment and tools [27–30]. This is because stainless steel has mechanical properties and the ability to withstand excellent corrosion. The automated nature of the material is needed to create a good product and work according to its function. The mechanical properties of materials are significantly increased by microstructures and their constituent elements. To get mechanical properties needed, it is necessary to adjust elements and microstructures in stainless steel. Those can be controlled if the steel is given treatment, such as heat treatment. Heat treatment with temperature and resistance time parameters can change the morphology and microstructure of stainless steel, which will then influence changes in mechanical properties. This research can determine the influence of temperature parameters and variations in resistance time so that the mechanical properties of superior stainless-steel material are obtained. The optimal heat treatment process is the cyclic heat treatment process on the mechanical properties of austenitic stainless steel 316(L).

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ТЕХНОЛОГІЯ ТЕРМІЧНОГО ОБРОБЛЕННЯ НЕІРЖАВІЙНОЇ КРИЦІ 316(L)

Неіржавійна криця типу 316 (відома в літературі також як 1.4401) або 316L (відома також як 1.4404) — це легована криця з хорошою корозійною стійкістю; тому вона широко використовується в сучасних сталевих інструментах. Але її застосування все ще обмежене через деякі властивості, що потребують поліпшення. Це поліпшення, перш за все, стосується механічних властивостей. Твердість неіржавійної криці залежить від її фази та складових (легувальних) елементів. Підвищення твердості неіржавійної криці можна здійснити, змінюючи її мікроструктуру та фази термічним обробленням. Є кілька різновидів оброблення, таких як відпал, нормалізація, відпуск, зміцнення та циклування. Циклічне термічне оброблення є термічним обробленням, що, як очікується, підвищить твердість неіржавійної криці за рахунок збереження ударної в'язкості криці. Циклічне термічне оброблення можна використовувати як ефективний метод у майбутньому.

Ключові слова: механічні властивості, неіржавійна криця 316, циклічне термооброблення, мікроструктура, ударна в'язкість.