The Effect of Heat Treatment on The Mechanical Properties and Microstructure of Martensitic Stainless Steel AISI 410

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Abstract

Engineering materials, mostly steel, are heat treated under controlled sequence of heating and cooling to alter their mechanical properties to suit a particular design purpose, Martensitic stainless steel are widely using for their good mechanical properties and moderate corrosion resistance. The steel samples were heat treated in an electric furnace at different austenitizing temperature (at 860˚C in annealing, 920˚C in normalizing and 972˚C in hardening) and holding times (24 min, 1 h and 30 min); and then the heated samples were directly cooled down from the austenitizing to room temperatures using three different cooling medium, which were; oil quenching "hardening treatment", air cooling "normalizing treatment" and cooling in shutdown furnace "annealing treatment". Further, oil quenched samples were subjected to a tempering treatment at 510˚C for 1 hour. The mechanical properties of AISI 410 martensitic stainless steel in the as-received condition, as well as, at the various heat treatment conditions were evaluated by conducting tensile and hardness testes. The microstructure of the treated and untreated samples was examined using optical microscope. Results showed that the mechanical properties of AISI 410 martensitic stainless steel can be changed and improved by various heat treatments for a particular application. It was also found that the annealed samples with mainly ferrite structure gave the lowest tensile strength (782.31 N/mm^2) and hardness value (10 HRC) and highest Percentage elongation value (45%), while hardened sample which comprise martensite gave the highest tensile strength (1595.62 N/mm^2) and hardness (39 HRC) value and lowest Percentage elongation 14.71%, also tempered specimen gave the highest hardness. The value of the yield strength of (1055 N/mm2) was observed to be higher for the tempered specimen possibly as a result of the grain re-arrangement, followed by the hardened, normalized and annealed specimens. The value of the ultimate tensile strength was also observed to be in the order; hardened> tempered>normalized>annealed. The investigation results showed significant variation in the microstructure and mechanical properties of AISI 410 martensitic stainless steel with the cooling rate employed upon heat treatment.

Keywords: *Heat treatment; Mechanical properties; Microstructure; AISI 410.*

1. Introduction

Stainless steel was first invented in the beginning of the 20th century [1] where the use of conventional carbon steels was restricted to their strong susceptibility to corrosion and oxidation [2]. In August 1913, Harry Brearley in Sheffield (UK) melted stainless steel for the first time which was microstructurally martensitic with a carbon content of 0.24% and chromium content of 12.8% [3]. However, Strauss and Maurer in Germany produced the first austenitic grade during the same year by adding an austenite stabilizing element, nickel. Moreover, in the United States, Dansitzen was investigating the invention of Brearley with a lower carbon content which led to the discovery of ferritic stainless steel [4]. Generally, the austenitic, martensitic and ferritic stainless steel grades had been discovered prior to World War I [5]. Nowadays, considering the global warming, environmental pollution and life cycle costs, the need for durable materials is continuously increasing stainless steel production. Carbon and alloy steels are the most widely produced metallic materials in the world, and approximately 2% by weight of all produced steels are stainless steels [1]. Austenitic stainless steels due to their superior corrosion resistance, excellent mechanical properties and good weld ability are consistently used in oil and gas production and power generation industries [4, 6, and 7]. The chemical composition, the proportion of constituents and exposure temperature range determine the response of austenitic stainless steels to thermal treatments at room temperature [8]. The austenite phase is a supersaturated solid solution which cannot accommodate more than 0.006wt% carbon in an equilibrium state. In industry, stainless steel is often applied in making gas turbine, high temperature steam boilers, heat-treating furnaces, aircraft, missiles, and nuclear power generating units. It is also used in hospitals, kitchens, abattoirs and other food processing plants because it is easy to clean which fulfills strict hygiene conditions. Due to wide application of stainless steel, it is important to improve the mechanical properties of stainless steel in order to enhance properties of it. This is because heat treatment can improve durability and reliability of stainless steel. It will make a step forward in bringing more benefits to user in various application fields [9 and 10]. Previous studies have investigated the influences of the corrosion resistance on sensitized stainless steels after solution treatment on stainless steel, but limited literature was observed on investigation of heat treatment process on Microstructures and Mechanical Properties of AISI 410 martensitic stainless steel. The objective of the present study is to investigate the effect of heat treatment (annealing, normalizing, hardening and tempering.) on the mechanical properties of AISI 410 martensitic stainless steel.

2. Methodology, Materials, Equipment and Tools

2.1 *Methodology*

First, stainless steel specimens are machined into standard size to ASTM E8 standard will be heat treated in furnace for specific time. After that, stainless steel will be quenched by varying the quench medium and time duration of agitation to identify the effect of these variables to the properties of heat treated stainless steel. There are five main processes in this experiment, which are heat treatment, tensile test, and hardness test and microstructure observation to obtain result for this project. Rockwell Hardness Test Machine will be used to define the hardness value of the test specimens. Optical Microscope will be used to observe the changes in the structure of stainless steel.

2.2 *Materials*

The specimen used is a martensitic stainless steel AISI 410, For different tests AISI 410 was cut to fifteen specimens using power hacksaw (with 10 mm diameter and 400 mm long), which was obtained from the industrial complex-Esbeia. Chemical composition for the raw material is shown in Table 1 and Mechanical properties of this material are shown in Table 2.

Table 1 Chemical composition of steels

Material	Constituent percentage in weight					
	Carbon C	Manganese Mn	Phosphorus P	Sulfur S	Silicon Si	Chromium Cr
AISI 410	0.15	0.9	0.040	0.035	0.7	14

Table 2 Mechanical properties of raw material [11]

Material	Ultimate Tensile Strength (MPa). Min	Yield strength (MPa). Min	Hardness Rockwell (Max)
AISI 410	450	205	96 HRC

2.3 *Objectives of the Study*

The objectives of the present work were to investigate the influence of various heat treatments, hardening, tempering, normalizing and annealing, on the mechanical properties and microstructures of the martensitic stainless steel (AISI 410).

2.4 *Equipment and Tools*

The following equipment and tools have been used in this study:

- ✓ Measuring instruments.
- ✓ Tensile test machine (ZWICK 1000).
- ✓ Rockwell hardness machine (BULUT-BMS 201- R).
- ✓ electric furnace, optical microscope;
- ✓ Mounting machine,
- ✓ Polishing machine.

3. Experimental

3.1 *Specimen preparation*

Each specimen was prepared for hardness and tensile tests. The samples were machined into standard specifications for tensile testing, following standard test procedures in accordance with the ASTM E8 standards. An optical metallurgical microscope was used for taken the micrograph of the samples.

3.2 *Heat treatment*

Samples were subjected to different heat treatment: annealing, normalizing, hardening, and tempering in accordance to ASM International Standards [12]. The first procedure for heat treated involves austenitizing process by heating the sample at 860°C for 24 min, follow heating again at 920°C for 1 hr. heating at 972°C for 24 min quenched the sample produced martensitic structure. Higher austenitizing temperature was sufficient to transform the steel into austenite and to form martensite phase upon quenching [13]. The second procedure involves tempering process. To exam the tempered martensite and ferrite phase the tempering temperature was chosen varied between (400-700) °C for 1 hr. [14]. Heat treatment processes of all the specimens are identified as show in Table 3.

Table 3: Heat treatment processes of all the specimens

Condition	Annealed	Normalized	Hardened	Tempered
Temperature ^{0}C,	860	920	972	510
Holding time,	24 min	1 hour	30 min	1 hour
Cooling medium	Furnace	Air	Oil	Air

3.2.1 *Annealing process*

The specimens were heated to a temperature of 860^{0}C. At 860^{0}C the specimens were held for 24 min in furnace, then the furnace was switched off and samples left inside it. The specimens were taken out of the furnace after 24 h, when the furnace temperature had already reached the room temperature.

3.2.2 *Normalizing process*

Each samples of the MSS 410 to be normalized were placed in the furnace and heated to temperature of 920°C. The samples were retained at this temperature for the period of one hour for full transformation to austenite. They were later removed from the furnace and left in air for cooling.

3.2.3 *Hardening process*

The specimens to be hardened were placed inside the furnace as shown in Figure 1 and heated to a temperature of 972°C. At this temperature, there is transformation of the steel to austenite. The samples were retained at this temperature for a period of 30 min (because of its mass) during which the transformation must have been completed, after which they were later removed from the furnace and dropped inside container of oil for rapid cooling to room temperature .

3.2.4 *Tempering process*

In the hardened carbon steel specimens, the as-quenched martensite is not only very hard but also brittle. The brittleness is caused by a predominance of martensite. This brittleness is therefore removed by tempering [12]. The process of tempering involves heating the hardened steel specimen to 510ºC at 1 hr. At this temperature, the prevalent martensite is an unstable structure and the carbon atoms diffuse from martensite to form a carbide precipitate and the concurrent formation of ferrite and cementite. This process allows microstructure modifications to reduce the hardness to the desire level while increasing the ductility.

Fig. 1, electric furnace

3.3 *Microstructure examination:*

Microstructure examination of the treated and untreated samples was carried out. Each sample was carefully grounded progressively on emery paper in decreasing coarseness. The grinding surface of the samples were polished using Al_20_3 carried on a micro clothe. The crystalline structure of the specimens was made visible by etching using solution containing 2% Picric acids and 98% methylated spirit on the polished surfaces. Microscopic examination of the etched surface of various specimens was undertaken using an optical microscope.

3.4 *Mechanical testing*

Heat-treated samples (different heat treatment sequences) were tested for various mechanical properties. For hardness testing, oxide layers etc. formed during heat treatment were removed by stage-wise grinding. Average HRC readings were determined by taking three hardness readings at different positions on the samples, using a Rockwell hardness tester as shown in Figure 2. For tensile properties, All the tensile tests in this study were done with a tensile test machine which called “ZWICK 1000”, as shown in Figure 3, Load-elongation data were recorded and converted into stress-strain graphs. Ultimate (tensile) strength, and ductility (% elongation) were determined from these graphs, reported values being average of three readings. All testing was done in accordance with ASTM standard test procedures.

Fig.2 Hardness testing machine

Fig. 3 Tensile test machine

4. Results and discussion

As described above, samples were subjected to four types of heat treatment sequences: annealing, normalizing, hardening and tempering, Variation of mechanical properties of martensitic stainless steel AISI 410 after these heat treatments is presented below in a graphic format. All mechanical testing was performed at room temperature.

4.1 *Effect of heat treatment on microstructure*

The microstructure of as-received sample (Figure 4) showed a combination of ferrite (white) and pearlite (black), Heat treatment of the as-received martensitic stainless steel AISI 410 at 860˚C for 24 min, 920 ˚C for1 hour and at 972 ˚C for 30 min resulted in eutectoid transformation of the ferrite and pearlite phases into austenite. The microstructure of the annealed sample is shown in Figure 5. As it can be seen in Figure 5, the ferrite grains had undergone complete recrystallization and these constituted the major portion of the microstructure the annealed martensitic stainless steel AISI 410 with stress free matrix. Annealing the specimen, results in the transformation of austenite grains into ferrite and pearlite. The microstructure observed, shows a coarse grain of ferrite and a coarse structure of pearlite as a result of the furnace cooling which imparts ductility on the material. The value for the annealed sample is minimal for hardness and maximum for toughness. This is due to the softening effects of the ferrite matrix which arise from liberation of trapped carbon atoms in the saturated ferrite during annealing. So subjecting the steel to annealing heat treatment at 860ºC affected the spatial distribution of ferrite at the grain boundaries, at 860ºC the deformed structure was fully homogenised and during the slow cooling from austenizing range to room temperature the final microstructure consisted of ferrite grains in which the pearlite was more uniformly distributed. Figure 6 shows the microstructure of the normalized AISI 410 martensitic stainless steel. The normalized sample showed that the shape and size of the original austenite grains were influenced to a remarkable extent. The sample revealed a pearlitic matrix in which shorter graphite flakes than in annealed sample existed. It was observed that there were many short graphite flakes surrounded with patches of uniformly distributed pearlite grains as seen in Figure 6.

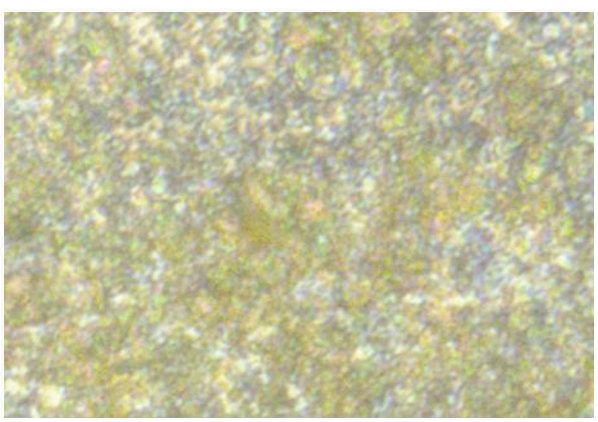


Fig. 4 Microstructure of untreated of the AISI 410

Figure 7 shows the massive martensite structure of hardened sample, when martensitic stainless steel is rapidly quenched from its austenite temperature to room temperature, the austenite will decompose into a mixture of some martensite and fewer pearlite as a result of this microstructure which is hard, hence, there was increase in tensile strength, hardeness and reduction in ductility of the material [15].

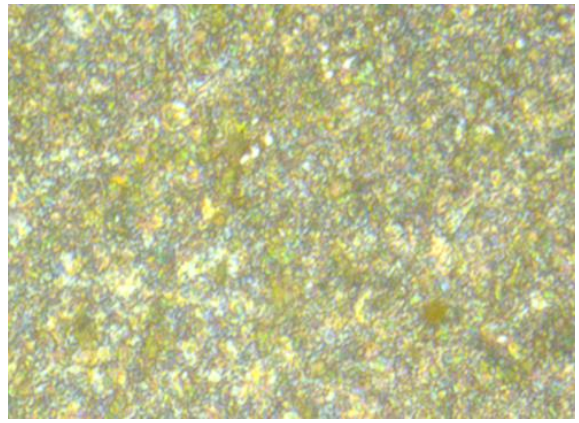


Fig. 5 Optical micrographs of the AISI 410 microstructure developed by austenitizaion treatment at 860°C for 24 min followed by furnace cooling to room temperature "annealing". Picric etch.

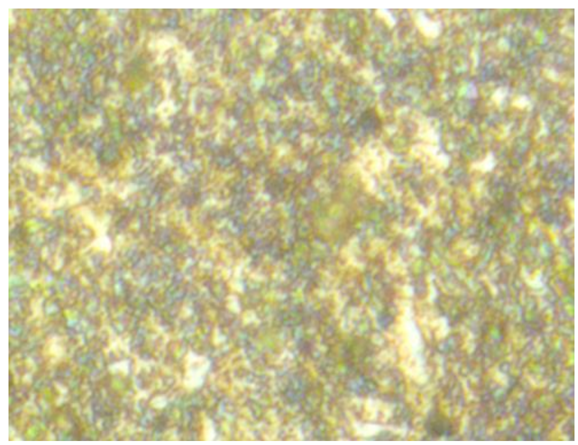


Fig. 6: Optical micrographs of the AISI 410 microstructure developed by austenitizaion treatment at 920°C for 1 hour followed by air cooling to room temperature "normalizing". Picric etch

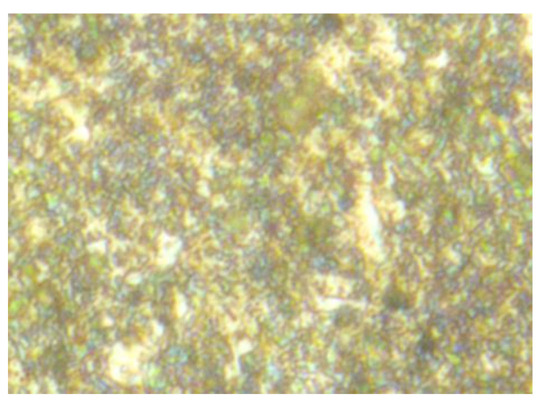


Fig. 7 Optical micrographs of the AISI 410 microstructure developed by austenitizaion treatment at 972°C for 30 min followed by oil quenching to room temperature. Picric etch

When quenching and tempering is done, the microstructure consists of a martensite matrix with graphite nodules. After tempering at higher temperatures, the matrix phase changes to tempered martensite, thus relieving the internal stresses and increasing the strength and ductility with hardness. Under quenched condition when specimens are heated at a temperature 972℃, held for 30 min at that temperature than quenched rapidly in water at that time phase When quenching and tempering is done, the microstructure consists of a martensite matrix with graphite nodules. After tempering at higher temperatures, the matrix phase changes to tempered martensite, thus relieving the internal stresses and increasing the strength and ductility with hardness. Under quenched condition when specimens are heated at a temperature 972℃, held for 30 min at that temperature than quenched rapidly in water at that time phase transformation of steel takes place, where the lattice structure of steel changes immediately from a face centered cubic ($\gamma-phase$) to body centered tetragonal martensite [16]. Furthermore, large amount of distortion occured during the formation of platelets of martensite, which in turn leads to rapid increase in its hardness [17]. The microstructure of hardened and tempered at 450ºC is show in Figure 8. A highly recrystalised ferrite grains (white dotted areas) with some secondary graphite site was observed. This micrograph revealed that the microstructure of tempered specimen consisted cementite precipitate and ferrite matrix, phase transformation takes place from retained austenite to bainite. Also cementite precipitate and ferrite matrix are found [18]. When the specimens are tempered at temperature 510℃ the phase transformation takes place from an unstable martensite to a mixture of ferrite and cementite. This showed that tempering temperature improved the degree of tempering of the martensite, softening the matrix and decreased its resistance of plastic deformation.

4.2 *Effect of heat treatment on mechanical properties*

The effect of heat treatment (annealing, normalizing, hardening, and tempering) on the mechanical properties (ultimate tensile strength, hardness, yield Strength, percentage elongation, and failure Stress) of the treated and untreated samples is shown in Table 3. The tensile strength of the as treated specimen was 858.17 N/mm2 and hardness value of 16 HRC, Percentage elongation 28.93%, yield strength 610 N/mm2 and failure stress of 581.44 N/mm2 were obtained. Comparing the mechanical properties of annealed sample with the as treated sample, annealed sample showed lower tensile strength (782.31 N/mm^2), yield strength (430 N/mm^2), failure stress (558.28 N/mm^2) and hardness (10 HRC) and increase in percentage elongation (45%).The decrease in tensile strength and hardness can be associated with the formation of soft ferrite matrix in the microstructure of the annealed sample by cooling, this can be associated with the very slow cooling rate employed during this annealing treatment and that caused a significant increase in the inter-lamellar spacing of pearlite phase (Figure 6) Since the pearlite inter-lamellar spacing is known to be an important factor for controlling ductility and strain hardening, this identical Referenced [19].

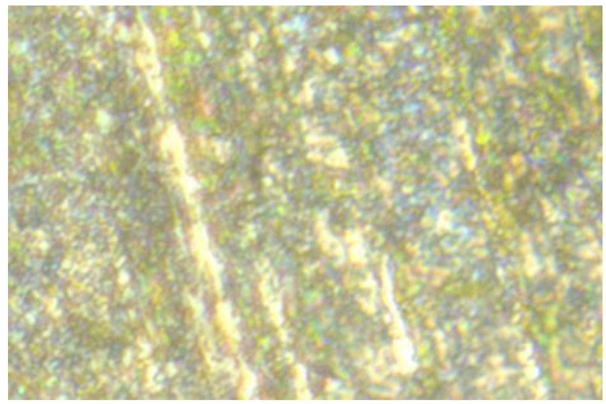
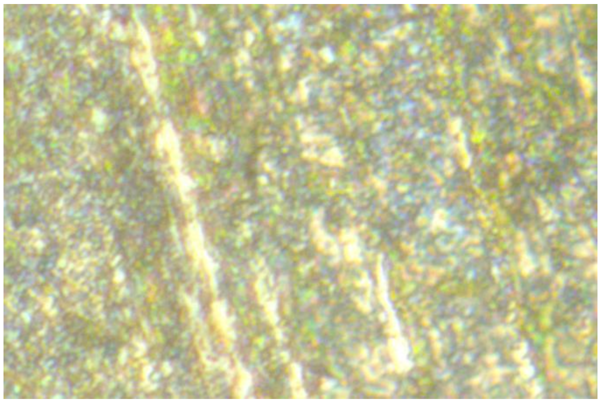


Fig. 8 Optical micrographs of the AISI 410 microstructure developed by austenitizaion treatment at 972°C for 30 min followed by oil quenching to room temperature then tempered for 1 hour at 510°C and air cooled. Picric etch

Table 3: Mechanical properties of treated and untreated samples

Heat treatment	Average of Mechanical properties				
	Tensile Strength, (N/mm^2)	Yield Strength (N/mm^2)	Failure Stress (N/mm^2)	Percentage elongation %	Hardness Rockwell C (HRC)
As received	858.17	610	581.44	28.93%	16
Annealed	782.31	430	558.28	45%	10
Normalized	902.93	465	712.87	25%	20
Hardened	1595.62	1050	1446.24	14.71%	39
Tempered	1185.18	1055	900.69	19.13%	39



The mechanical properties of the normalized sample were found to be 902.93 N/mm^2, 465 N/mm^2, 20 HRC, 712.87 N/mm^2 and 25 % for tensile strength, yield strength, hardness, failure stress and percentage elongation, respectively. The increase in tensile strength and hardness as compared to annealed and as treated sample was due to proper austenising temperature at 920oC and higher cooling rate, which resulted in decrease in percentage elongation, which was lower than those obtained for as treated and annealed samples due to pearlitic matrix structure obtained during normalization of AISI 410 martensitic stainless steel.The mechanical properties of the hardened sample revealed that it had the highest value of tensile strength 1595.62 N/mm^2, failure stress 1446.24 N/mm^2 and highest hardness (39 HRC) were obtained. The specimen was austenised at 972^{0}C for 30 minutes and then oil quenched. This treatment increased the tensile strength and hardness but there was massive reduction in percentage elongation in area 14.71%. The mechanical properties of tempered sample showed that the tensile strength, yield strength, hardness, failure stress and percentage elongation were 1185.18 N/mm^2, 1055 N/mm^2, 39 HRC, 900.69 N/mm^2 and 19.13%, respectively. Comparing the mechanical properties of tempered sample with hardened sample, it was found that there was decrease in tensile strength and failure stress at tempering temperature 510^{o}C while the percentage elongation, and yield strength increased which can be associated to the graphitisation of the precipitated carbides that resulted in the formation of ferrite at tempering temperature of 510^{o}C. This showed that tempering temperature improved the degree of tempering of the martensite, softening the matrix and decreased its resistance of plastic deformation. However, the test results showed that annealing treatment gave an elongation superior to any other heat treatment studied. The variability in ultimate tensile strength, percentage elongation, failure stress, hardness and yield strength of treated and untreated AISI 410 martensitic stainless steel are shown in Figures 9 to 13, respectively.

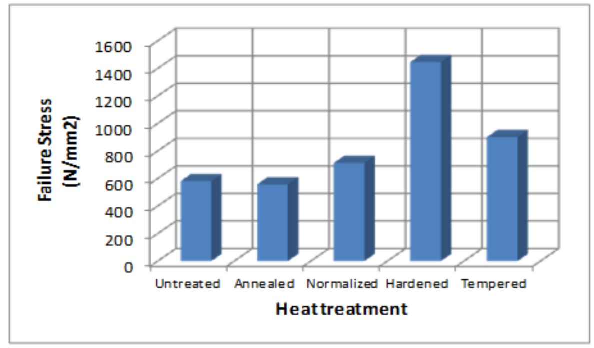


Fig. 9 Tensile strength of treated and untreated samples of AISI 410 martensitic stainless steel.

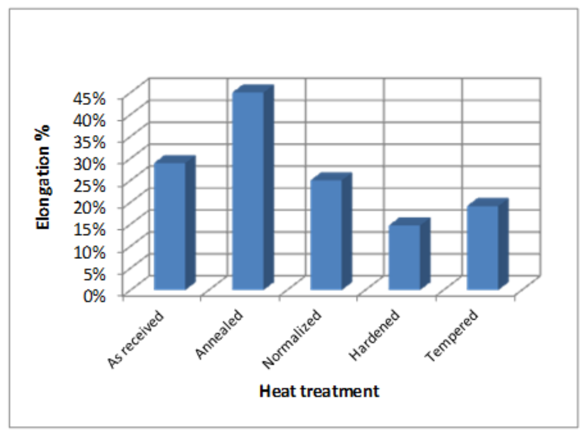


Fig. 10 Percentage elongation of treated and untreated samples of AISI 410 martensitic stainless steel.

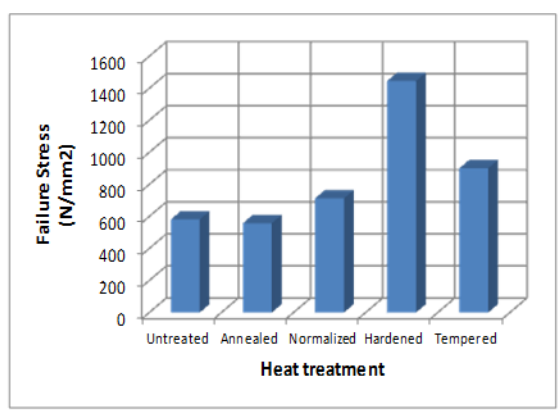


Fig. 11 Failure stress of treated and untreated samples of AISI 410 martensitic stainless steel.

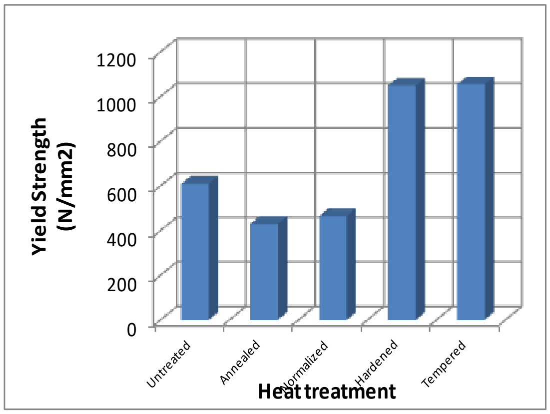


Fig. 12 Hardness of treated and untreated samples of AISI 410 martensitic stainless steel.

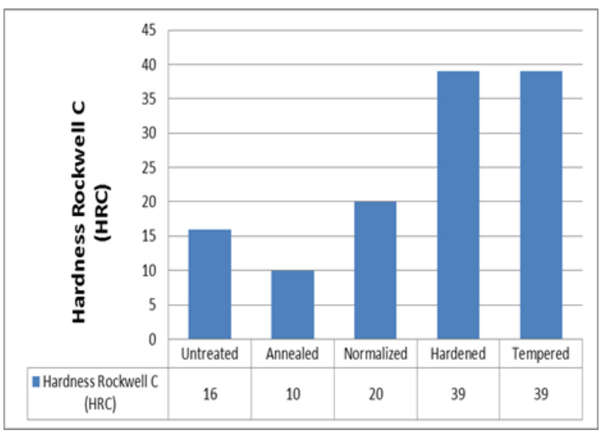


Fig. 13 Yield strength of treated and untreated samples of AISI 410 martensitic stainless steel.

5. Conclusions

The following conclusion has been drawn from the experimental result and discussion already made.

- Hardness test showed highest value at 510°C tempering temperature. It is thought to have occurred due to secondary hardening phenomenon. And also higher hardness value has been obtained when quenched in cold oil, in austenitizing temperature at 972°C.

- The fast cooling rate employed during oil quenching from the of austenitizing temperature results in the transformation of austenite into martensite, which in turn, significantly increases the alloy hardness at the expense of severe reduction in the alloy tensile strength, failure Stress and percentage of elongation.

- Heating the AISI 410 samples at austenitizing temperature (860°C for 24 minutes, 920^{0}C for 1 hour and at 972^{0}C for 30 minutes) results in the transformation of the ferrite-pearlite microstructure into austenite during austenitizing treatment.

- During normalizing treatment, the cooling rate from the austenitizing temperature is slow enough to allow re-formation of the ferrite and pearlite phases, which enhances the percentage of elongation of the AISI 410 samples. The nearequilibrium conditions employed during normalizing treatment, however, raise the alloy tensile strength, failure stress and yield strength.

- Upon annealing treatment, the very slow cooling rate from the austenite phase field allows a slow decomposition of the austenite into ferrite and perlite phases. Compared with the normalized condition, the ferritic-perlitic structure of the annealed AISI 410 is coarser and the spacing between the pearlite lamellae is larger. Consequently, the annealed samples possess a higher percentage of elongation, but with lower hardness, tensile strength, failure Stress and yield strength relative to the normalized condition.

- The hardness and failure Stress decrease, whereas, the percentage of elongation increases with increasing the cooling rate of the AISI 410 from the austenitizing temperature.

- Tempering following hardening treatment does not induce a significant change in the lath-shaped martensitic structure of the as-quenched AISI 410 MSS specimen. This treatment, however, results in a significant improvement in the mechanical properties of hardened AISI 410 MSS specimen, such as yield strength percentage of elongation.

- The mechanical properties of stainless steel (AISI 410) can be altered through various heat treatments. The results obtained confirmed that improvement in mechanical properties that can be obtained by subjecting stainless steel (AISI 410) to different heat treatments investigated in this paper.

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