Effect of Austenitizing Heat treatment on the Hardness and Microstructure of Martensitic Stainless Steel AISI410

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الملخص:-

يعد الفولاذ المقاوم للصدأ المارتينسيتي من المواد الهندسية الواعدة التي تظهر الجودة في مقاومة التآكل وخواصها الميكانيكية. ومع ذلك، تتأثر خواصها الميكانيكية بشكل كبير بالمعالجة الحرارية الأوستنيتية الناجمة عن التحول المارتنسيتي. لذا، خلال هذا البحث، قمنا بدراسة تأثير الأوستنيتي على الصلابة والبنية المجهرية للفولاذ المقاوم للصدأ المارتنسيتي (410) بهدف دراسة نتائج المعالجة الحرارية التي ستضمن بنية مارتنسيتية مع الحد الأدنى من الأوستينيت المحتفظ به، والكربيدات المشتتة بالتساوي وصلابة بين 36.5 و 42 HRC بعد التبريد بالغمر في الزيت والمعادلة. الفولاذ الذي تم فحصه خلال هذه الدراسة يتوافق في تركيبته مع الفولاذ المقاوم للصدأ مارتنسيتي متوسط الكربون AISI 410، باستثناء إضافة 0.45% من الموليبدينوم إلى السبائك. تمت معالجة عينات الفولاذ حرارياً في فرن كهربائي عند درجات حرارة مختلفة للأوستينيت (عند 860 درجة مئوية في التلدين، 920 درجة مئوية في التطبيع و 972 درجة مئوية في التصليب) وأوقات الاحتفاظ (24 دقيقة، 1 ساعة و 24 دقيقة)؛ ومن ثم تم تبريد العينات الساخنة مباشرة من الأوستنيتي إلى درجة حرارة الغرفة باستخدام وسط التبريد، والذي كان عبارة عن تبريد بالغمر في الزيت "معالجة التصلب"، وتبريد الهواء "معالجة التطبيع" والتبريد في فرن الإغلاق "معالجة التلدين".

علاوة على ذلك، تم تعريض العينات المغمورة بالزيت إلى معالجة تصلب عند درجة حرارة 510 مئوية لمدة ساعة واحدة. تم فحص البنية المجهرية للعينات المعالجة وغير المعالجة باستخدام المجهر الضوئي. أظهرت النتائج أن صلابة الفولاذ المقاوم للصدأ المارتنسيتي AISI 410 يمكن تغييرها وتحسينها عن طريق المعالجات الحرارية المختلفة بتطبيق خاص. كما وجد أن قيمة الصلابة (HRC 36.5)، في حين أن العينة المتصلبة التي تحتوي على المارتينسيت أعطت أعلى صلابة (HRC 42)، وكذلك العينة المقساة أعطت أعلى صلابة.

وقد أظهرت نتائج الدراسة تباينًا كبيرًا في البنية المجهرية والخواص الميكانيكية للفولاذ المقاوم للصدأ المارتنسيتي AISI 410 مع معدل التبريد المستخدم عند المعالجة الحرارية.

Abstract

Martensitic stainless steels are promising engineering materials demonstrating good corrosion resistance and mechanical properties. Their mechanical properties are however significantly affected by the austenitizing heat treatment induced martensitic transformation. So during this examination we study the effect of austenitizing on the hardness and microstructure of martensitic stainless steels 410 was examined with the objective of supplying heat-treatment guidelines to that will ensure a martensitic structure with minimal retained austenite, evenly dispersed carbides and a hardness of between 36.5 and 42 HRC after quenching in oil and tempering. The steels examined during the course of this examination conform in composition to medium-carbon AISI 410 martensitic stainless steel, except for the addition of 0.45% molybdenum to the alloys. 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 24 min); and then the heated samples were directly cooled down from the austenitizing to room temperatures using cooling medium, which was 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 microstructure of the treated and untreated samples was examined using optical microscope. Results showed that the hardness 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 hardness value (36.5 HRC), while hardened sample which comprise martensite gave the highest hardness (42 HRC), also tempered specimen gave the highest hardness.

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; Hardness; Microstructure; AISI 410.

1. Introduction:

Martensitic stainless steels were developed to satisfy a need in industry for corrosion resistant alloys that respond to hardening through heat treatment. These steels are alloyed with at least 10.5% chromium and up to 0.6% carbon, and are designed to be fully austenitic at elevated temperatures. The austenite can be hardened by quenching or cooling to room temperature from the austenitising temperature, which enables transformation to martensite [1]. Due to their high alloying element content, martensitic stainless steels demonstrate excellent hardenability and are normally considered to be air hardening [2]. Martensitic stainless steels containing low carbon content are commonly used in quenched and tempered condition. The heat treatment consists of annealing to obtain austenite and dissolve the carbides, followed by cooling to transform the austenite into martensite and subsequent tempering of martensitic microstructure[3]. Depending on the composition and processing history, the microstructure of martensitic stainless steel consists of martensite, undissolved carbides as well as re-precipitated carbides, retained austenite[4].

It is well-known that properties obtained in these steels are strongly influenced by such treatments. The amount of carbides in the as-quenched microstructure exerts an influence on the properties of these materials such as hardness, strength, toughness, corrosion and wear [5]. In the present study, a grade of stainless steel (SS, 410) was investigated in order to determine the effects of various austenitizing temperatures on microstructure and hardness [6].

2. Material and methods:

2.1. Material:

The stainless steel used in this work was a hot-rolled AISI 420 martensitic stainless steel and its chemical composition is shown in Table 1.

Table 1: Chemical composition of the investigated martensitic [higher vocational center of casting].

Grade	Fe	C	Mn	Si	P	S	Cr	Mo	Ni	N
410	85.7	0.120	0.800	0.443	0.0230	0.0109	12.2	0.450	0.450	0.0577
Grade	**Al**	**Co**	**Cu**	**Nb**	**Ti**	**V**	**W**	**Pb**	**Sn**	**B**
410	0.0243	0.0199	0.111	0.0539	0.0391	0.0575	0.0158	0.0050	0.0102	0001

As it is mentioned before, AISI410 was supplied as rods with dimensions of 250 mm length, Diameter of 50 mm.

2.2. Heat Treatment:

Samples were subjected to different heat treatment: annealing austenitizing/ and tempering accordance to ASM International Standards [7]. 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 [8]. 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. Heat treatment processes of all the specimens are identified as show in Table 2.

Table 2: the heat treatment processes of all 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

2.2.1 Annealing:

Annealing of AISI410 martensitic stainless steel softens the steel by producing a fully ferritic matrix with the majority of the carbon precipitated as coarse, globular carbide particles. Since the precipitation of chromium carbide is diffusion-controlled, the annealing conditions of AISI410 have to be optimized to ensure sufficient softening within a reasonable annealing time. Prior austenitizing heat treatment, AISI410 examined in this study was annealed at temperature of 850°C for 3 hours, then furnace cooled to room temperature.

2.2.2 Austenitizing:

The austenitization heat treatment process were carried out by using muffle furnace which consists of adjustable temperature controls. AISI410 samples were positioned horizontally in the furnace. It is necessary to be mentioned here that AISI410 samples were put in the furnace before switching on the temperatures controller to ensure homogeneous matrix of microstructures during austenitizing process.

2.2.3 Quenching.

The quenching process was carried out by using quenching oil. Temperature of the quenching oil is maintained at 35ºC. The quench rate is so critical for martensitic AISI410 grades. Oil cooling is preferred in order to avoid carbide precipitation[2].Figure 1 illustrate the cooling cycle annealing process.

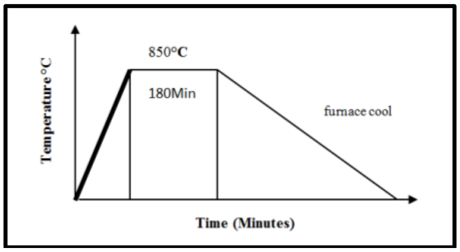

Fig. 1: Heat Cycle of annealing process.

The subdivision temperatures were illustrated in table 3.

Table 3: Subdivision of austenitizing temperatures range.

Number of Sample	S1	S2	S3	S4	S5	S6	S7
Temperature °C	925	940	955	970	985	1000	1010

AISI410 steels samples were subdivided into seven samples according to the temperatures range of austenitization that just reach up to 85°C. However, this range is too small comparing to the other austenitization heat treatment temperatures of martensitic stainless steels such as AISI420 grade thus, it is necessary to be mentioned that AISI410 grade was technically precise procedures needed to get optimal results. AISI410 grade samples are presented in this investigation containing 0.120 wt% carbon, which means fully homogenous austenite could be occurs. It is doable due to the thickness and chemical composition of specimens were examined.

The optimum soaking time and austenitizing treatments were carried out in the austenitization region starting of 925°C up to 1010°C as recommended[15]. The heating rate was 10°C/min.

2.2.4 Tempering:

In the present study, the tempering furnace temperature was set in a way that stress relieve can occur. Tempering temperature was performed for all samples at the same time and the same period inside the device. Tempering temperature was selected as an average of the recommended range for AISI410 grade, which was (150-370) °C. Samples were positioned together in the furnace such as austenitizing position. Then furnace switched on up to 260°C (Tempering temperature) for one hour maintained and then temperature controller was switched off until tempering process was finished. Must be remembered that tempering temperature is determined by the required mechanical properties. In general, in high carbon content of stainless steel martensitic grades, the 420°C to 600°C temperature range must be avoided, because within this range brittleness may be induced apart from loss in corrosion resistance [9].

2.2.5 Metallography:

An ASTM standard metallographic procedure was conducted. AISI410 samples preparation included, placing a small piece of steel specimen in longitudinal direction in a mounting, Once the mounted sample is consolidated, the mounted sample is ground and polished using standard metallographic procedure.

Using grinding and polishing machines. The finished samples have smoothness surfaces as shown in figure (2).

Fig. 2: The sample of Martensitic stainless steel AISI 410.

3.Results and Discussions:

It has been mentioned in previous chapters that the range of austenitizing temperatures of Martensitic stainless steel AISI410 just reach up to 85°C. This range is too small comparing to the other types of the same Martensitic grade such as AISI 410, Which is has a large values and characteristics of change occurring in hardness and microstructures due to higher austenitizing temperatures (920-1010) °C in some studies which means that the range will reach up to 300°C. This large range can accommodate a lot of change, which means a lot of results and data will be achieved in order to understand the characteristics of material. Thus, in the present study, change in microstructures specifically will be obviously mentionable.

3.1 Results:

Experiments conducted for this work includes characterizing the AISI410 steels samples in the as-annealed condition, carrying out austenitizing heat treatment process, and hardness tests, other microstructural characteristics and inclusions, based on the experimental procedures described in chapter four. The experimental results are presented and analyzed in this section.

3.2. Hardness evaluation:

The samples were held at various austenitising temperatures for 24 minutes as recommend (one hour for 25 mm thickness as shown in table (4), followed by oil quenching (rapid cooling). The soaking time was calculated according to the Lower thickness of specimens. However, the effect of austenitizing temperatures on the hardness of the AISI410 steel specimens was explained and detailed as plotting data which are shown in figure (3).

The hardness of the samples was in the range of 36.5 to 42 HRC. Peak hardness of 42 HRC was observed at 985°C which decreased thereafter a little bit with a minimum of 36.5 HRC at 1010°C. Therefore, 985°C was chosen as optimum austenitizing temperature due to the highest hardness was obtained. The results of the determinations of hardness values are summarized in table (4). The results were reported as the average of three tests for each sample as shown in figure (4).

Table 4: Austenitizing and tempering temperatures are performed with the hardness values which are obtained for AISI410.

Sample Number	Austenitizing Temperature ^{0}C	Soaking Time in Min	Quenching Media	Tempering Temperature ^{0}C	Hardness value on the treatment surface		Average Of Hardness
					Edge	Middle	
S1	925	24	Oil	260	39	38	38.5
S2	940	24	Oil	260	37	37	37
S3	955	24	Oil	260	39	38	38.5
S4	570	24	Oil	260	42	39	40.5
S5	985	24	Oil	260	43	41	42
S6	1000	24	Oil	260	42	32	37
S7	1010	24	Oil	260	37	36	36.5

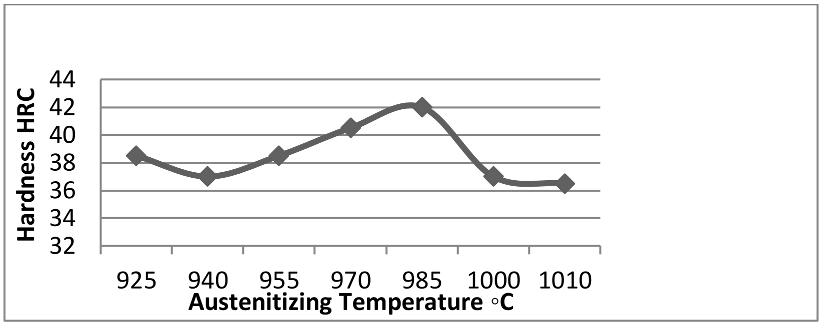

Fig. 3: The influence of varying austenitizing temperatures on the hardness of martensitic AISI410 Type.

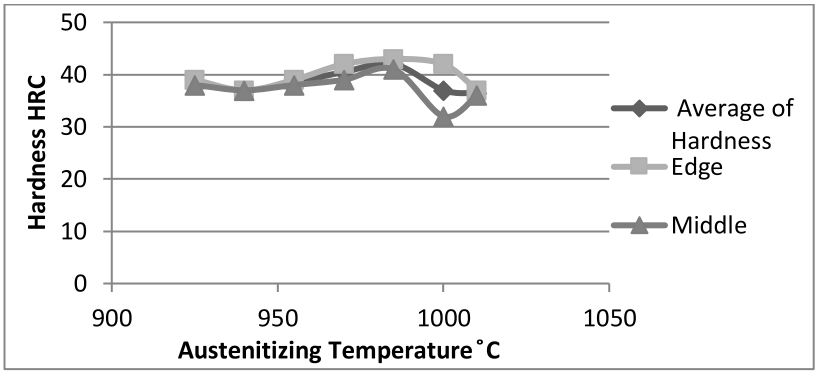

Fig. 4: The influence of varying austeniting temperatures on the hardness at different regions of martensitic AISI410 samples,

3.3. Metallographic evaluation:

3.3.1 Phase characterization:

Once the steels have been austenitized, quenching or cooling to below the martensite transformation range results in the formation of martensite. The Ms temperature of AISI410 martensitic stainless steel is reported to be in 350°C, whereas the Mf temperature is estimated to be approximately 90°C lower than the Ms temperature.
Almost all alloying elements in solid solution reduce the Ms and Mf temperatures, with carbon having the greatest effect. If the Mf temperature is depressed to below room temperature or even 0°C, the more highly alloyed martensitic steels may contain retained austenite after quenching due to the sub-zero temperatures required to transform all the austenite to martensite.
The carbide phases present in the AISI410 steel samples were identified by optical microscopy for the annealed steels. Figure (5) shows the optical micrographs of AISI410 steel in the annealed condition, carbides and ferrite were identified as well. In the hardened state, martensite, retained austenite and carbides were identified as shown in figures (6) to (8).
Microstructure of low-carbon martensitic steel is needle-like microstructure, and may contain large amounts of retained austenite after quenching, depending on nickel and percentage of carbon contents[5]. In this investigation, AISI410 consists nickel content of 0.450 wt%, which is playing a role in the retained austenite formation.

3.3.2 Microstructure evaluation:

The following optical micrographs, Figures (5) and (6) were taken when the annealing process was carried out. The optical micrographs (5) up to (10) were taken when the annealing and austenitizing heat treatment and tempering process were individually carried out at 260°C.
The polished and etched samples were examined under the optical microscope at 200x magnification. Figure (5) is an another micrograph of the sample which is selected randomly from the same rod, showing the same defects in the annealed condition.
As indicated in the micrograph below, the microstructure is composed of carbides in the ferrite matrix.
To reveal the carbide particles at suitable magnification, the annealed steels were
investigated by optical microscope. Figures (5) and (6) are apparent that the AISI410 steels exhibit virtually identical initial annealed microstructures where fine, well spheroidized secondary carbide particles are uniformly distributed in the ferrite matrix. Visible non-metallic inclusion and defects were observed optically in the steels, but without mentionable effect on the alloy at all. Figure (7) shows an OM image for the treated AISI410 steel sample which is austenitized at 925°C for the constant period of holding time (24 min). Image appears to have martensite and more undissolved carbides in a unit area which indicates less carbide dissolution in comparison to the next images which are illustrate the microstructures influenced by the higher varying austenitizing temperatures.
While the image in figure (8) has higher austenitizing temperature and appears to have martensite and less un-dissolved carbide. Martensite was obtained and obviously visible on the all images.

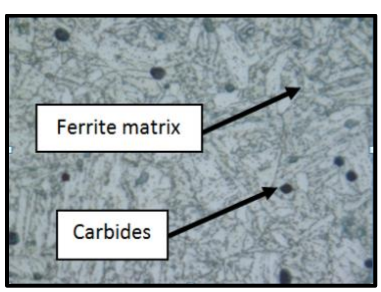

Fig. 5: Optical photomicrograph of AISI410 in the annealed form.

The microstructure consists of carbides in a ferrite matrix. (Small Black dots are inclusions remains in as-supplied steels), magnification of 200X. Hardness was 10HRC.

In this steel may contains little unwanted amounts of retained austenite after quenching, as shown in figures (7), (8).

The presence of significant amounts of retained austenite reduces the as-quenched hardness of the alloy, and may promote embrittlement[10]. Multiple tempering steps can be used in martensitic stainless steels to temper the fresh martensite or cryogenic (sub- zero) tempering can be used to reduce the retained austenite content prior to tempering.

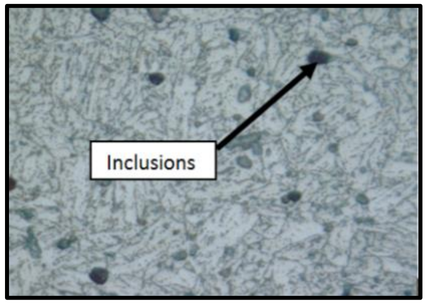

Fig. 6: Optical photomicrograph of AISI410 sample in the annealed form showing the defects. Magnification of 200X

It has been known from the experimental results of the studies that are concerning the austenitizing heat treatment for martensitic stainless steels grades, which are clearly indicate that with increase in austenitization temperature, the volume fraction of retained austenite gradually increases, and the volume fraction of undissolved carbides progressively decreases and the grain size increases.

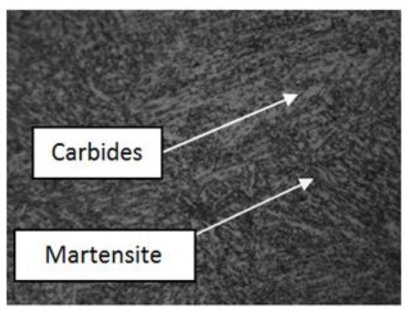

Fig. 7: Photomicrograph of AISI410 austenitized at 925°C, quenched and tempered at 260°C shows Martensite with fine-carbides precipitates. The hardness is 38.5HRC.

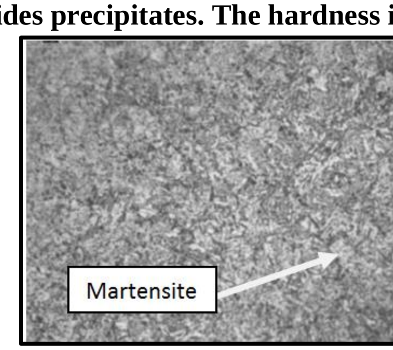

Fig. 8: Photomicrograph of AISI410 austenitized at 955°C, shows fine martensitic matrix with less undissolved carbides. The hardness is 38.5HRC. Magnification of 200X.

The hardness increases with higher Austenitising temperatures between 940°C and 985°C. At austenitising temperatures higher than approximately 1000°C, the hardness decreases as the high concentration of carbide-forming elements and carbon in solid solution in the austenite due to carbide dissolution reduces the martensite transformation range.

The peak hardness value obtained for the samples austenitised at temperature of 985°C can be attributed to that the initial austenitized temperature at 985°C for 24 minutes, is too low to dissolve significant amounts of $M_{23}C_6$. The reported temperature range for $M_{23}C_6$ chromium carbides to dissolve is between 950-1050°C. As only a small percentage of carbides goes into solution during heat treatment, very little retained austenite is expected after quenching.

The increase in the carbon content of the martensite phase due to the partial dissolution of carbides makes the martensite is therefore harder due to its higher carbon content and hence high hardness value was obtained.

The carbide densities in the examined samples decrease with an increase in austenitizing temperature. At austenitising temperatures above 985°C, complete carbide dissolution takes place and the increased retained austenite had led to decrease in hardness values.

Retained austenite content Increases with increase in the austenitization temperature is due to higher carbon content in solution, as carbon is an austenite stabilizer. From this concept, although AISI410 examined in this study has low carbon content 0.120 wt% the martensite was obtained with retained austenite.

Figure (9) shows the image of austenitized sample at temperature of 985°C, quenched and tempered. Microstructure consists of predominantly retained austenite and some undissolved carbides in a fine martensitic matrix. This performed temperature considering as optimal temperature which gives a peak value of hardness. Must be mentioned that The hardness of austenitized and tempered samples depends on grain size, dissolution of carbides during austenitizing treatment and carbide precipitation after tempering. Lower austenitizing temperatures resulted low dissolution of carbides with fine grain size. Finer grain size could be responsible for an increased rate of precipitation owing to a larger grain boundary area available for nucleation and the martensitic matrix will be soft.

With increase in austenitizing temperature, the grain size increased to maximum at 985°C, which resulted low precipitation of carbides and hence the marginal decrease in hardness is observed in samples austenitized at temperatures above 985°C.

In the martensitic grades, the austenitizing temperature playing a greater role on the carbide volume fraction, and the volume fraction of carbide particles decreases with increases in the austenitizing temperature. This result confirms that the carbide particles dissolve with increasing austenitizing temperatures.

From the figures (8), (9), it is evident that the martensitic microstructure experiences coarsening at the small range of austenitizing temperatures (from 955°C to 985°C), the degree of coarsening increases with an increase in the austenitization temperature, and will be noticed optically above 985°C. The extent of coarsening of martensitic microstructure is observed high in this range compared to the other intervals.

Retained austenite content was obviously observed irrespective of austenitizing temperature.

Fig. 9: Photomicrograph of AISI410 austenitized at 985°C.
The structure consists predominantly of retained austenite in a martensite matrix,
magnification of 200X. The hardness is 42HRC.

Martensitic stainless steel AISI410 which is examined in this study contains Molybdenum of 0.450wt%. Thus, the volume fraction of carbides in the Molybdenum containing AISI410 steel consistently higher than that of the steel with no Molybdenum addition. Molybdenum is reported to combine with carbon to form Mo_2C or to form part of the Chromium-rich $M_{23}C_6$ carbide that is commonly observed in martensitic stainless steels. The presence of Molybdenum is expected to retard the coarsening and dissolution of $M_{23}C_6$. Higher austenitizing temperatures are therefore required for complete dissolution of carbides in the presence of Molybdenum. Molybdenum is expected to increase hardenability, raise the temper resistance and improve the high temperature strength of the alloy. Figure (10) shows the microstructure of the sample austenitized at temperature of 1010°C followed by tempering. As increase in retained austenite content is evident with an increase in austenitizing temperature. At this temperature or higher the microstructures contained no visible carbide particle.

The conclusion was expected that all carbides had dissolved at the higher heat treatment temperatures, reducing the martensite transformation range and favoring retained austenite. In its as-quenched martensitic condition the steel is hard and brittle and may contain pockets of retained austenite. Quenching is therefore followed by tempering to reduce brittleness, increase ductility and toughness and reduce residual stresses. During tempering the steel is kept at a temperature below the austenite transformation range, and cooled at a prescribed rate. Tempering allows controlled precipitation of fine carbide particle, transformation of retained austenite, and recovery and recrystallization of the highly distorted martensite matrix.

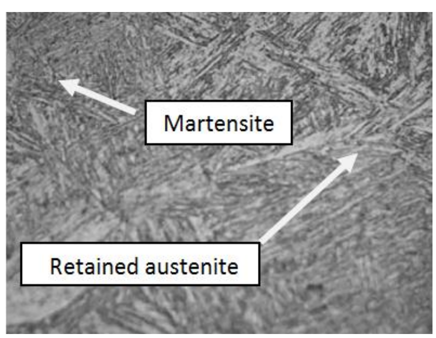

Fig. 10: Micrograph of martensitic AISI410 austenitized at 1010°C,
consists of retained austenite and martensite matrix with no visible carbides.
The hardness is 36.5HRC. Magnification of 200X.

Although that martensitic steels have a high temper resistance, AISI410 stainless steel is usually tempered at low range of temperatures (150-370) °C comparison to the other grades of martensitic stainless steels such as AISI420 which are usually tempered at temperatures higher than 550°C. Thus, as mentioned previously in chapter two that the tempering temperature is determined by the required mechanical properties, but at the same time, in AISI410, the 420 to 600°C temperature range must be avoided, because within this range brittleness may be induced apart from loss in corrosion resistance.

5. Conclusions:

On the bases of the performed investigations it was shown that:

- The increase in austenitizing temperature effect greatly on the hardness (as expected). Increase in austenitizing temperature in AISI410 has increase in the hardness of the alloy. (If hardness never increased when cooling rapidly is carried out in quenched media, that means there is a wrong procedure was done).
- Optimum hardness property in AISI410 can be obtained at temperature of 985°C.
- Austenitizing heat treatment effecting strongly on the microstructures of martensitic AISI410. Martensite phase can be obtained in martensitic AISI 410 when homogeneous austenitic matrix is obtained due to high austenitizing temperatures are performed and rapidly cooling was carried out. That means no matter carbon content it was in martensitic stainless steel AISI410. Martensite phase depending on other factors beside the carbon content, such as the perfect performance of the technical procedures for heat treatment process.
- AISI410 steels which do have low carbon content such as investigated grade in this study, Increased austenite content with increase in the austenitization temperature. Thus, in order to minimize the formation of retained austenite in the as-quenched, as-tempered steel, the selected austenitization temperature should be as low as possible. which means that retained austenite could be formed in martensitic AISI410 steels irrespective of how low carbon content it was. Retained austenite could be formed in AISI410 due to the existence of austenite formers such as Nickel which is reach up to 0.450 wt% in martensitic steels are studied in this research.

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