

Effect of Retained Austenite on the Micro-structure and Mechanical Properties of AI-SI4340 High Strength Low Alloy Steel (HSLA steel) Using Magnetic Saturation Measurement and X-Ray Diffraction methods

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Abstract- Retained Austenite (RA) has great deal with the mechanical properties of high strength low alloy steel. Therefore, in this paper, Retained Austenite volume fractions have been evaluated in AISI4340 alloy steel using two well-known methods, X-Ray diffraction (XRD) and magnetic measurement methods. The specimens were heat treated using different heating temperature and different cooling rate (different quenching media). A comparison between the results of two methods proved that these results were approximately Identical .The results show that Retained Austenite formation increase as heating (Austenizing) temperature increase for the same quenching media ,as well as ,it increases by increasing cooling rate . The maximum amount of Retained Austenite found as (27.2 Wt %) which recognized when the specimens heated up to 1000°C then quenched in Water while the minimum amount of Retained Austenite found as (7.06 wt%) when the specimens heated up to (800 °C) then quenched in Sand. Hardness tests using Vickers and Rockwell methods were used and the results show that hardness values decreased with increasing heating temperatures and the maximum Vickers micro-hardness and Rockwell hardness numbers were equal to (121.8HRB) and (516.35 HV) which were detected when heating up of the specimens were up to 800 °C then quenched in water. Tensile tests show that increasing cooling rate lead to increasing in Strength due to increasing of hardness which in turn, leads to increase in yielding points and ultimate strengths. Retained austenite effects on microstructure were investigated using scanning electron microscopy (SEM) and optical microscopy and the results show that at low cooling rate the microstructure consist of bainite and/or martensite phase with small amount of retained austenite, while, increasing heating temperature and cooling rate results in microstructure consist of martensite and retained austenite phases.

Index Term: Martensite, Mechanical properties, Magnetic measurement, Retained austenite, X-Ray diffraction.

I. INTRODUCTION

When the hot steel is quenched in cooling media, the soft Austenite transform into hard and brittle martensite. However, the conversion from Austenite to martensite is usually not 100% effective because the steel must reach a temperature much lower than room temperature to finish the transformation. The retained austenite can change the mechanical properties of materials.

Several techniques were developed to calculate the amount of retained austenite in Heat-treated steel, including X-ray diffraction (XRD), neutron diffraction, and optical microscopy combined with image analysis, scanning electron microscopy (SEM), Mössbauer spectroscopy, Dilatometry, and magnetization measurements. Among them, the XRD method is the most commonly used as it is a suitable technique and XRD services are widely available. Other methods are frequently used to overcome the disadvantages

of the XRD method. However, these methods also suffer from their own disadvantages with respect to evaluation of the retained austenite volume fraction in steels with a multi-phase microstructure. For example, the Mössbauer spectroscopy measurements need a very thin foil (20–50 μm), which leads to a dissimilar internal stress condition, in contrast to bulk specimens. This may affect the results, since the retained Austenite known for its stress-sensitive. Dilatometry measurements make it likely to detect the fraction of transformed phase in-situ from the length change, but the measurement precision is not high since the information of lattice parameters is limited and transformation plasticity may also influence the net dilatation. Magnetization measurements have intrinsic advantages as they are accurate and probe the bulk of the materials.

Determined volume fraction of RA in transformation induce plasticity (TRIP) steel using XRD method and magnetization measurement method was done by L. Zhao, et al. [3], they compared the results obtained from the magnetization and XRD methods , finally they found that the magnetization measurement method lead to reliable results and more sensitivity to detect the RA than XRD method . N.H. Van Dijk, et al. [4] Study the thermal stability of the RA phase in (TRIP) steel during cooling from room temperature to -173°C, using XRD method. The first conclusion was that the amount of RA transformed during cooling depending mainly on holding time and the chemical composition of the steel, while the second conclusion was that Austenite grain with lower carbon concentration produce lower stability through cooling. Felipe Lucas Sicupira, et al. [5] quantified the amount of retained austenite in super martensitic stainless steel (SMSS) used XRD and saturation magnetization methods to study the influence of secondary tempering temperature on phase transformation, they tempered the steel specimens for one hour within a temperature range of (600°C to 800°C), finally their results showed that the amount of retained austenite decrease with increasing of secondary tempering temperature in both methods. Ryu H.B., et al.[6] Studied the influence of thermo mechanical processes on the retained austenite content in a Si – Mn transformation induced plasticity (TRIP) steel by evaluating the effect of hot deformation on the microstructure of TRIP steel. They found when a hot deformation was absence, so the increasing in cooling rate from 10°C/s to 35°C/s led to raising Retained Austenite content from 18 % to 24 %, while increasing the amount of hot deformation led to decreasing the amount of Retained Austenite.

This paper Present experimental work to study the effects of Retained austenite on microstructure and mechani-

cal properties of specimens made from AISI4340 at different Austenizing temperature and different Quenching media.

II. EXPERIMENTAL WORK

It is difficult to found a precise association between the retained austenite and factors affecting on it, the effect of retained austenite and mechanical properties. To overcome such problems, several experimental works should be made. The required material (AISI4340) was purchased from (ONLINE METAL.COM™) company which supplied also the chemical composition tests paper and also a chemical composition test was done according to ASTM A751 standard [7], in order to verify their results, the chemical composition of the specimens was as shown in Table (1).

Table (1) chemical composition of AISI4340 specimens

Element	Measured Values Wt%	Standard Values Wt % [33]
C	0.42	0.37 – 0.43
Cr	0.79	0.70 – 0.90
Mn	0.78	0.7
Ni	1.61	1.6 – 2
Mo	0.26	0.2 – 0.3
Si	0.25	0.2 – 0.25
S	0.020	≤ 0.035
P	0.03	≤ 0.04
Fe	Balance	Balance

For AISI4340 alloy steel the austenite stabilized element are 1.61 % nickel and 0.78 % manganese and the ferrite stabilized element are 0.79 % chromium, 0.26 % molybdenum and 0.25 % silicon. The microstructure of such steel will be martensite and/or bainite with high content of retained austenite. The strength and hardness values for this steel will be either reduce or stilled without change with increasing heating temperature and decreasing critical rate of cooling due to the presence of retained austenite.

A. SPECIMENS MACHINING AND PREPARATION

One hundred twenty specimens were prepared according to ASTM A370 [8] and ASTM E975 [9] standards. These specimens were machined in a workshop with the following procedure:

- 1) The specimens were brought as long bars.
- 2) The bars were cut into the required dimensions which were exactly as (200 mm) long with (12 mm) diameter for tensile specimens as shown in Figure(1) and (5mm) long with (25 mm) diameters for electron microscopic, X-ray diffraction, magnetization measurement and hardness specimens, Figure (2)

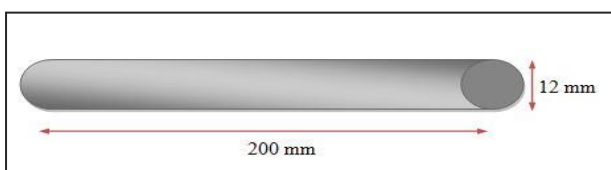


Fig. (1) Tensile specimen before machining

- 3) The tensile specimens were made at local workshop to suitable dimension using turning machine as shown in Figure (3)
- 4) Heat treatment processes then used at different temperatures and different quenching Media were used, after that, fine grinding machine used to grind the surfaces of the specimens that shown in figure (2).

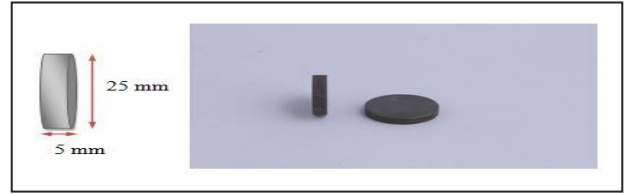


Fig. (2) Specimen for XRD and Magnetic tests



Fig. (3) Tensile Specimen after Machining.

B. HEAT TREATMENT, QUENCHING AND TEMPERING PROCESSES

Heat treatment process used at different temperatures and different cooling rates in order to achieve retained austenite presence in specimens microstructure. Quenching means the process in which a rapid cooling of metal parts from the austenitizing or solution treating temperature. The first step in the heat treatment process of low alloy steels is the heating of the specimens to a temperature ranging between (800 °C – 1000 °C) by step of (50 °C) using electrical furnace (THERMO type) with temperature range from (0 °C to 1100 °C) with digital temperature measuring and controlling. This range of heating temperatures was selected to showing the effect of heating temperature on the generated microstructure and mechanical properties. It was started with 800 °C which represented the beginning of transformation to austenite, then increasing by step 50 °C to reached 1000 °C. The second step is holding the specimens in certain temperature for one hour in order to ensure that all of the phases transform completely to austenite . Then, the specimens cooled to room temperature. The third step in this process is the quenching the specimens in different quenching media (Oil, Water, sand and air) to obtain difference cooling rate leads to different mechanical properties. The results of the above processes are very hard martensite and retained austenite with high concentrated internal stresses.

The final step is tempering which refers to the process during which a previously hardened or normalized steel is typically heated to a temperature below the lower critical temperature and cooled at a suitable rate. The tempering processes were done with the same furnace by heating the specimens to temperature of 300°C for one hour to remove residual stresses .All heat treatment processes were done according to ASM heat treatment hand book procedures [10].

C. RETAINED AUSTENITE CALCULATIONS

When the crystalline material is irradiated by X – rays, a distinctive diffraction pattern is produced, this pattern is determined by the crystalline structure of the phases presented in a certain substance [11]. After XRD tests done, the results would be observed as peaks with varying height due to X – ray energy diffracted at discrete 2θ angle from different (hkl) planes of atoms in lattice of the crystalline structure of the various phases [8]. Comparing of the integrated X – ray diffraction intensity of ferrite and/or martensite (BCC phase) and austenite phase with the theoretical intensities [9] to determine retained austenite volume fraction in steel.

For hardened and tempered steels, Chromium, Copper, Molybdenum, and Cobalt radiation are used to show the characteristic X – ray diffraction pattern reflections specification (diffractions peaks) which are frequently present as martensite (α) and austenite (γ) phases [9], [11]. In the present work copper radiation with monochromator is used.

Volume fraction of retained austenite (V_γ) calculated by using eq. (1) [9]:

$$V_\gamma = \left[\left(\frac{I_\gamma}{R_\gamma} \right) / \left\{ \left(\frac{I_\alpha}{R_\alpha} \right) + \left(\frac{I_\gamma}{R_\gamma} \right) \right\} \right] \quad (1)$$

Where:

I_γ : is the integrated intensity in the γ – phase.

I_α : is the integrated intensity in the α – phase.

R_α and R_γ : is the theoretical integrated intensity of α and γ phases respectively.

The volume fraction of carbides (V_C) should be determined by chemical extraction metallographic method [9]. In this paper the phase composition is considered to be consisted of martensite and austenite only.

D. MAGNETIC MEASUREMENT METHOD

Magnetic measurement is one of the widely methods that used to measure the amount of retained austenite in steel, this method depended on the magnetic properties of different phases of steel. In this method the variation in saturation magnetization of specimens or magnetic flux density that induced in specimens with and without austenite is directly transmitted to the volume fraction of non-magnetic retained austenite, This is because of the fact that ferrite, in addition to martensite and cementite, is ferromagnetic at temperature below the Cure temperature T_c (for pure ferrite $T_c=770^\circ\text{C}$ and for cementite, is $T_c=210^\circ\text{C}$) while the austenite is paramagnetic. The saturation magnetization depends on three main factors which are [2]:

- The chemical composition of the metal.
- Character of the phases present in the material.
- Permeability of materials.
-

The existence of alloying elements such as Cr, Ni, and Mo contributes to decrease the magnetic moment of iron and, consequently, the value of saturation magnetization. In this paper, a magneto charger device was made according to Lucas magneto method which used to magnetize the specimens [12]. The device consist of cores of electromagnet which are made of soft steel bar which are tightly attached to a steel square base, the cores are wounded with wires of

(2000 turns). All contacting surfaces were absolutely flat and square in order to achieve a perfect contact between the entire surfaces. Before the wire is wound on them, the cores must be insulated. A coil may be formed by placing fiber or cardboard washers around each end of cores and then wrapping cores themselves with several layers of electrician's taps. Two batteries were used to supplying the charger by 12 volts direct current. This device shown in figure (4). The magnetization process carried out by putting the specimen of type (B) over poles to complete the circuit. As the current is switched on, the coils on each limb produce a magnetic field. The intensity of field depended on the number of turns of wire on the core. This magnetic field induced a magnetic flux density in the specimen and made it magnetized. The amount of magnetization that induced in specimen was inversely proportional to the amount of retained austenite in it. The inducing magnetic flux was measured by Tesla meter devise by the following procedure:

1-Device probe applied to the specimen at different positions and the peak value was detected near the edge of the specimen.

2-The readings were taken five times to ensure small error percentage in the final reading, the average of these readings was calculated and considered as the final reading.

3-The volume fraction percentage of retained austenite, f_γ , can be determined from the magnetic flux density (B) values, according to eq. (2) [13]:

$$f_\gamma = \frac{B_\alpha - B}{B_\alpha} * 100\% \quad (2)$$

Where:

B_α : Magnetic flux value of specimen without retained austenite.

B: Magnetic flux value of specimen with retained austenite contains.



Fig. (4) Magneto charger device.

E. MECHANICAL PROPERTIES TESTS

Tensile test consider as the essential and most important test to show many of metals properties such ductility, yielding strength, ultimate strength and energy absorb for fracture, it measures the resistance of material to static or slowly applied force [14]. The experimental procedure and the dimensions of the specimens were made according to the ASTM A370 [8] specifications with the following standard specimen dimension as shown in Figure(5).

Gauge Length = 60 mm

Total Length = 200 mm

Specimen Diameter = 10 mm

Tensile test machine was used to carry out these testes, Three specimens for four quenching media at different temperatures were tested (total numbers were 60 specimens). The ultimate tensile strengths and elongation percentage

were evaluated for each specimen. The mean value for each specimen's case was calculated and any unusual values were neglected.

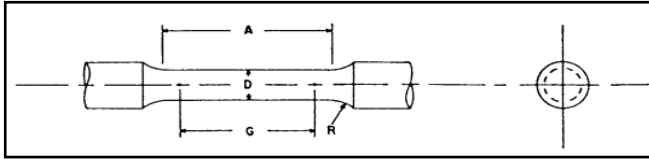


Figure (5) Schematic Diagram for Tensile Specimen.

Hardness tests were made in order to show the effect of RA presence on AISI4340. In this paper, two types of hardness tests were done:

- 1- Rockwell hardness test done according to ASTM E18[15], by using hardness device (Brooks universal hardness machine, Turkey made) to calculate value of HRB number by determining the depth of penetration of hardness 1/16" diameter ball under certain predetermined conditions. The minor load of 100 Kg was applied.
- 1- Vickers hardness test done according to ASTM E92 [16], by using a digital micro indentation tester to calculate value of HV number of the specimens after grinding, polishing and etching processes.
- 2- The mean value of five readings for each specimen and each test was calculated.

F. METALLOGRAPHIC TESTS

This test was carried out according to ASTM E3 [34] using optical microscopic (Olympus type) to show the microstructure contained and recognize the austenite phase that present in the hardened and tempered AISI4340 steel.

Before microscopic test, specimens were prepared using grinding and polishing using grinding and polishing machine (UNIPOL-820). Followed by etching process according to ASTM E407 [18] using etching liquid consists of:

- 1 g picric acid
- 100 mm HCL
- 100 mm ethanol

Immersing the specimen in this liquid to 2.5 min., and then immersing in distilled water. Finely examination by microscopic with magnification force of x500.

Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) analysis done with the following procedure, the specimens were first cleaned by using ultrasonic cleaner and jewelry solution then loaded on specimen stub using double side carbon tape and sprinkle powder on it, all stubs then tightened on specimens holder, blowing process then used to remove non-adherent particles. Prepared specimens

were loaded on FE-SEM. from (Zeiss Supra 55 VP) via air lock door which depends on low voltage to exceed the coating technique and avoid charging. Secondary electron detector (SE2) is used for high resolution and to sharp the image with fix magnification. A well calibrated (Quantax EDS XFlash) from Bruker™ used to qualitative and quantitative determination of the elements with mapping for elements distribution due to their concentrations in the alloy.

III. RESULTS

Retained austenite results using XRD methods are summarized in Table (2) while magnetic measurement method results show in Table(3). Noting that all AISI4340 used specimens were tempered up to 300 °C before heat treatment.

It can be recognized that Retained Austenite formation increase as heating (Austenizing) temperature increase for the same quenching medium. This is because that as heating temperature increase some (or all) of the carbides during heating dissolve and carbon content increases and becomes part of the crystal structure and the result is large content of RA fractions[12].

It also can be noticed that RA fractions increase by increasing cooling rate, therefore, the maximum RA fractions results achieved when water used as quenching medium while the minimum ones result from sand quenching. This is because that as cooling rate is increased (rapid quenching), there will be insufficient time for RA to transform into martensite and RA fractions would be increasing. By comparing these results with the results of other researchers [19, 20, 21], it can be observed that for the same tempering temperature (300 °C), the results are approximately the same.

Table (2) XRD results

Heating temperature	Quenching medium	RA fraction calculated value wt%
800 °C	Sand	7.06
	Air	9.17
	Oil	11.47
	Water	14.95
900 °C	Sand	8.50
	Air	10.95
	Oil	14.30
	Water	16.40
1000 °C	Sand	12.08
	Air	14.2
	Oil	25.5
	Water	27.2

Table (3) Magnetic measurement results

Heating temperature	Quenching medium	RA fraction calculated value wt%
800 °C	Sand	6.55
	Air	14.20
	Oil	11.74
	Water	16.94
900 °C	Sand	6.01
	Air	8.19
	Oil	15.8
	Water	16.94
1000 °C	Sand	14.21
	Air	16.94
	Oil	20.76
	Water	24.59

Figure (6) shows the relation between results of XRD method and magnetic measurement methods, it can be clearly recognized that their results are approximately identical. The standard error between the results of the two methods was; $E_r = \pm 0.82\%$, and the coefficient of determination (R^2) equals to (0.96).

Figure (7) shows the results of RA fraction achieved by magnetic measurement method is directly proportional to magnetic field densities, therefore, equation (3) has been developed to relate RA fraction with Magnetic field density (B) and this equation can be used directly to evaluate RA

fraction of AISI 4340 low alloy steel using reading of Tesla meter device only.

$$RA = 99.979 - 16.39(B) \tag{3}$$

Where;

B: Magnetic field density in (mT) [reading of Tesla meter].

RA: Retained Austenite fraction in (wt %).

Figure (8) shows the effect of volume fraction of retained austenite on the tensile strength, it shows that the increasing of retained austenite fraction is leading to increase in tensile strength until it reached approximately 14% then strength commence to decrease and the reason of that is the formation of martensite which is known for its hardness while, when austenite is produced there will be decreasing in strength and increasing in ductility because austenite is soft and ductile[12].

It can be notice from figure (9) that increasing in heating temperature is leading to decrease in the value of tensile strength because increasing in heating temperature leads to increase in austenizing grains growth and this effects on shape of produced martensite phase which in turn effects on tensile strength[22]. Quenching of fine austenizing grains leads to produce fine martensite (lath shape) structure and this is increase grains boundaries which in turn gives increasing in tensile strength, while quenching of coarse austenizing grains produce coarse martensite (plate shape) with less grains boundaries and larger areas of martensite plates giving decrement in tensile strength[22].

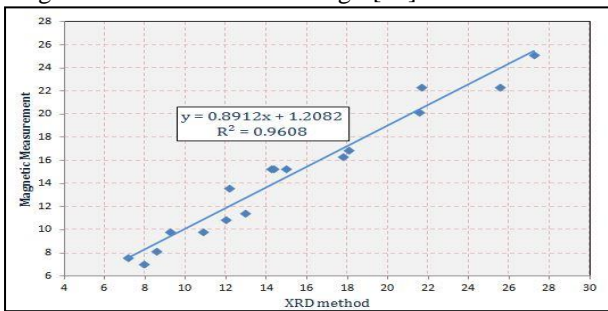


Fig. (6) XRD Vs Magnetic measurement results.

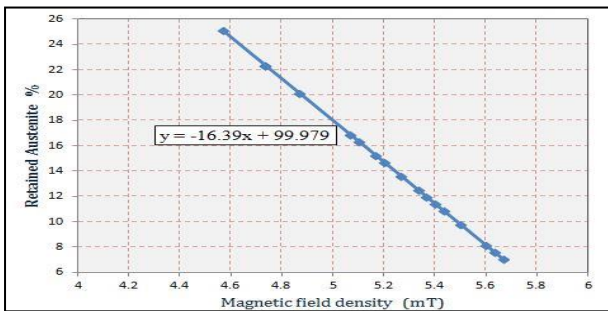


Fig. (7) The relation between magnetic field densities and Retained Austenite calculated

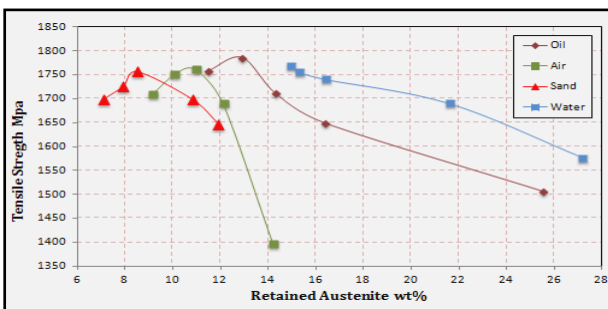


Fig. (8) Tensile strength Vs Retained austenite fractions.

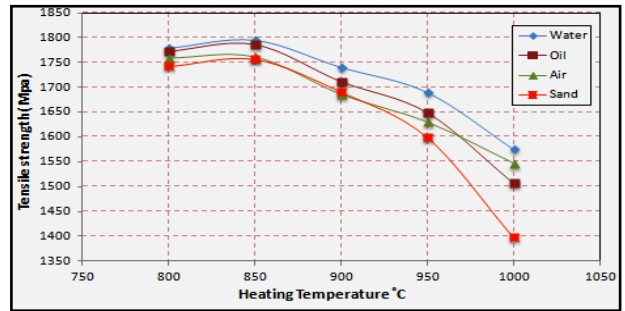


Fig. (9) Tensile strength Vs Heating temperatures.

Figures (10, 11) show effects of retained austenite fractions on hardness and micro-hardness which are determined by Rockwell hardness (HRB) and Vickers hardness (VH) respectively. The results show that the increasing in retained austenite fraction is leading to decrease in hardness because the presence of soft and ductile austenite phase. It also can be recognized the effect of cooling rate on hardness, since increasing of cooling rate at the same heating temperature leads to increase in hardness because high cooling rate (Rapid Quenching) increases possibility of martensite formation[10]. By comparing these results with the results of other researchers [19], [20], [21], it can be identify that both results are the same.

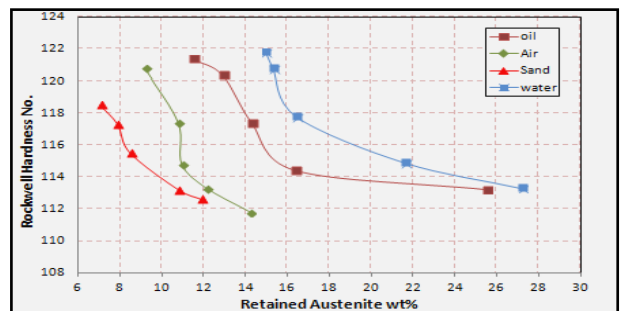


Fig. (10) Rockwell Hardness Vs Retained austenite fractions.

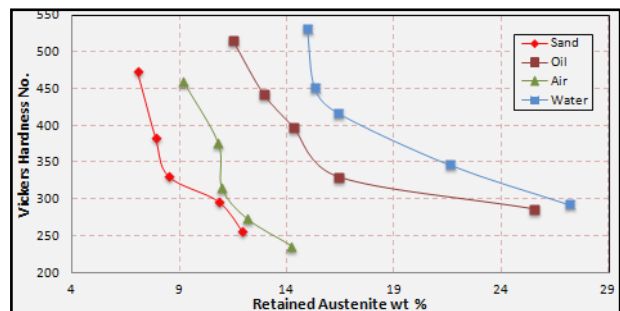


Fig. (11) Vickers Micro-hardness Vs Retained austenite fractions.

Figure (12) to Figure (15) show the effect of cooling rate on the final microstructure. At low cooling rate the microstructure consist of bainite phase (gray and black area) with small amount of retained austenite (black area) with small areas of ferrite (gray area) and martensite phases (lath shape), while , increasing cooling rate results in microstructure consist of martensite (needle shape) and retained austenite phases (black area). Figure (16) shows microstructure of annealed specimen, which consist of pearlite phase only which is has grain size finer than grain size of bainite phase , therefore , it was used in magnetic measurement method as a reference (i.e. specimen without martensite phase) in evaluation of Retained Austenite volume fractions of the heat treated specimens. Its magnetic flux density found equal to (6.1 mT).

Figure (17) to Figure (20) show the microstructure achieved by SEM, it can be recognize needle shape of martensite and retained austenite phase (dark small areas), and the effect of cooling rate and heating temperature on the shape of martensite. At low cooling rate, martensite appeared as a fine needle while, at rapid cooling rate and increasing of heating temperature, the needle become thicker. Figure (21, 22) shows the mapping image technique using SEM. In this technique, concentration of carbon atoms through microstructure (Green nodes) was observed. it can be seen that because austenite phase has high percentage of carbon atoms than other phases, there were plenty of green nodes which in turn illustrate the areas of retained austenite concentrations. By comparing the microstructure of at 800 °C and 1000 °C with results of other researcher [19], [20], [21], the results were identical for the same tempering temperature.

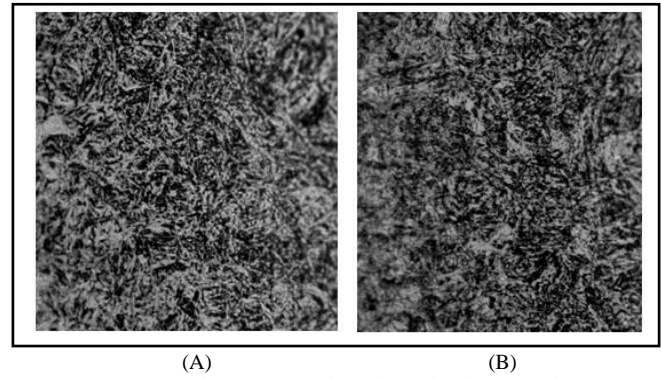


Fig. (15) Microstructure of specimens heating to 800°C, (A) water quenched while (B) oil quenched.

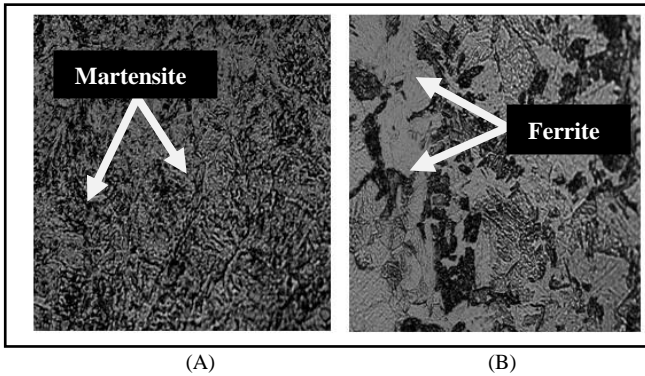


Fig. (12) Microstructure of specimens heating to 1000 °C, (A) air quenched while (B) sand quenched

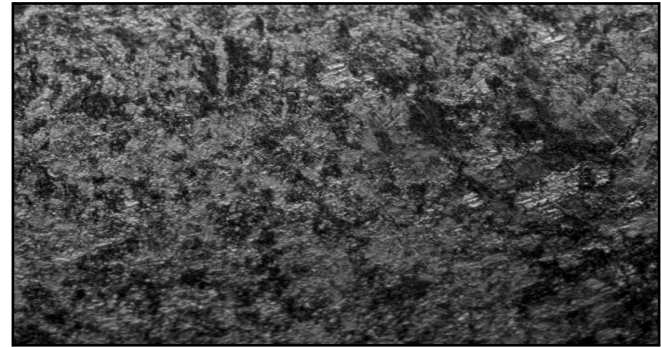


Fig. (16) Microstructure of annealed specimen

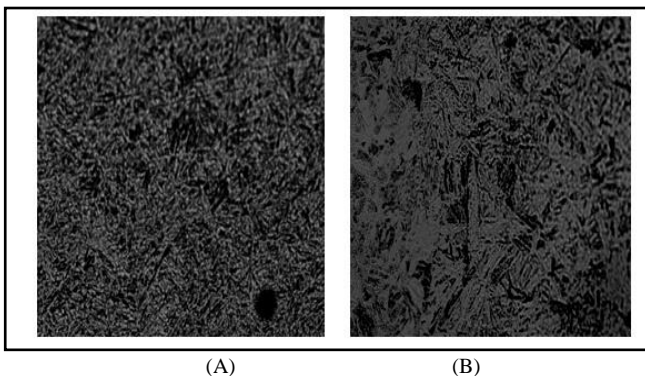


Fig. (13) Microstructure of specimens heating to 1000 °C, (A) water quenched while (B) oil quenched.

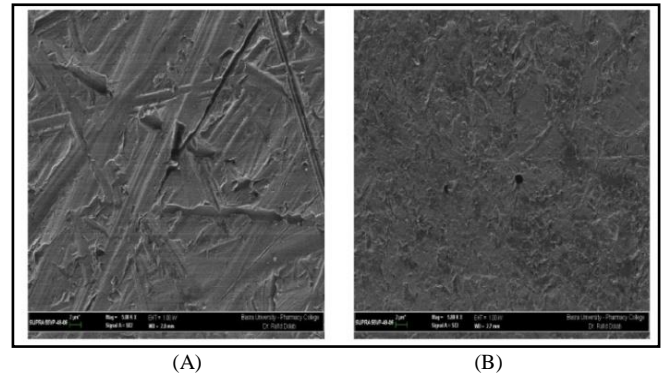


Fig. (17) SEM of specimen heated to 800°C, (A) air quenched while (B) sand quenched

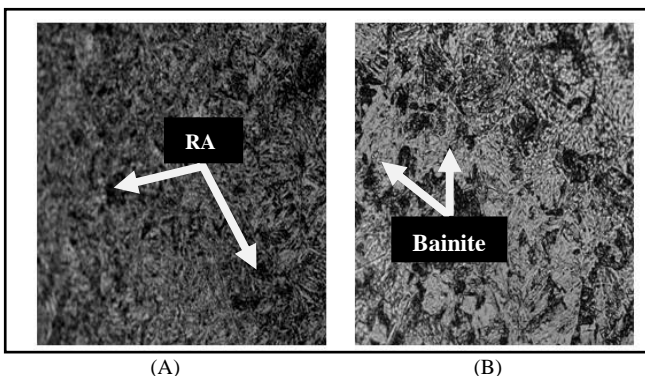


Fig. (14) Microstructure of specimens heating to 800°C, (A) air quenched while (B) sand

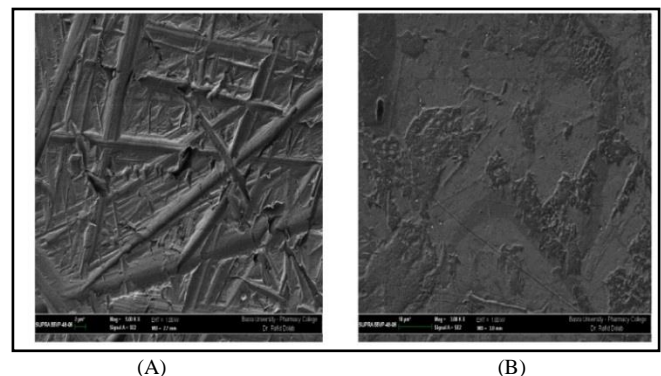


Fig. (18) SEM of specimen heated to 800°C, (A) water quenched while (B) oil quenched.

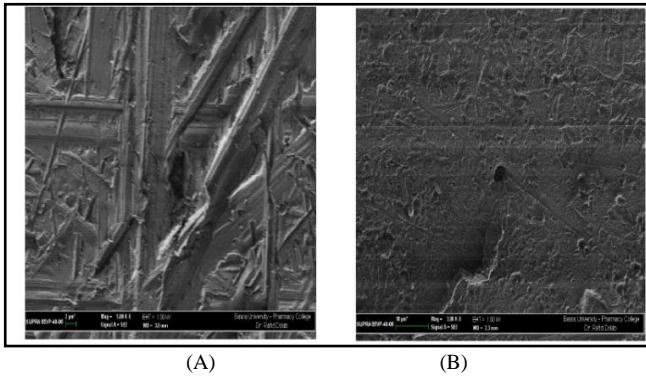


Fig. (19) SEM of specimen heated to 1000°C, (A) air quenched while (B) sand quenched.

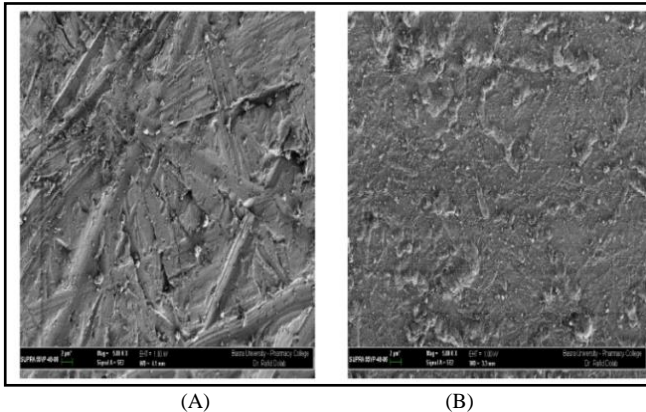


Fig. (20) SEM of specimen heated to 1000°C, (A) water quenched while (B) oil quenched.

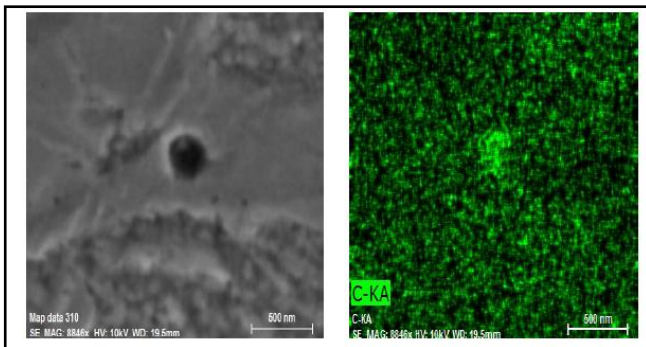


Fig.(21) SEM mapping of specimen heated to 800°C /Quenched in air.

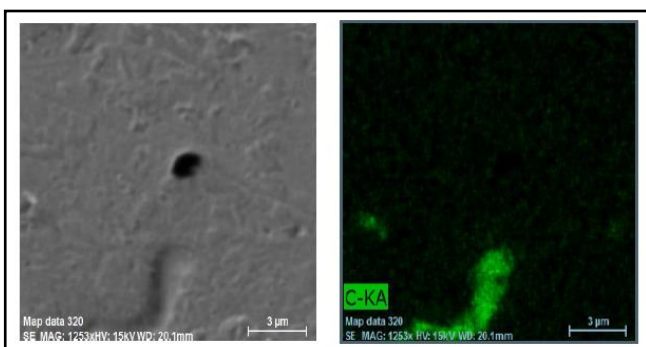


Fig. (22) SEM mapping of specimen heated to 1000°C /Quenched in sand.

IV. CONCLUSION

The conclusions of this study can be summarized into the following points:

- 1) Retained austenite presence in low alloy steel effects on its mechanical properties and microstructure.
- 2) Magnetic measurement method for retained austenite evaluation gives precise and durable results;

therefore, it can be used as alternate for expensive XRD methods since it is consider economically method when compared with XRD methods.

- 3) Retained austenite fraction can be calculated directly from magnetic field density using the following developed equation (3).
- 4) Increasing heating (austenizing) temperature leads to decrease in tensile strength while increasing of Retained Austenite fractions in the alloy lead to increase its strength until reached to proximately 14% after that tensile strength and hardness decrease with increasing Retained Austenite.
- 5) Increasing cooling rate leads to increase in hardness, strength, yielding point and ultimate strength.
- 6) From the results that is acquired, the optimal heating temperature to obtained higher Tensile Strength and Hardness values are ranged between 800 °C and 850 °C
- 7) The microstructure of only annealed specimen was consist of Bainite while quenching specimen in air and sand after heating to a certain temperature produce Bainite with small particles of Retained Austenite and Martensite, and when quenching process done in water and oil, the structure found to be of lath Martensite and Retained Austenite.

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VI. REFERENCES

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VII.BIOGRAPHIES

Murtadha Abbas Jabbar has expertise in mechanical, materials, production and metallurgical engineering. He awarded his B.Sc., M. Sc., and Ph. D. degrees in Mechanical Engineering from university of Basra. He working as undergraduate lecturer and he worked as supervisor, materials inspector, consulting engineering, and designer for several projects such as cathodic protection projects, installation of bridges, and the projects that including different type of welding process.