Effect of Heat Treatment on Microstructure, Magnetic and Mechanical Properties of HSLA-100 Steel

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Abstract

In this study, the effects of various heat treatments on microstructure, mechanical and magnetic properties of HSLA-100 steel were evaluated. The heat treatments consisted of austenitizing at 900 °C for 60 minutes, then quenching by different cooling rates via furnace, air, oil and water; and quenched specimens were aged at 600 °C for one hour. Field emission scanning electron microscopes were used for the characterization of the microstructure. A vibrating sample magnetometer, an inductance meter and a susceptometer were used for the characterization of the magnetic properties. Mechanical properties of the specimens were also studied using hardness, tensile strength and impact toughness methods. The fracture surfaces of charpy specimens were examined using scanning electron microscope. The results showed that martensite phase was spread in the all of the cooling rate. The results of magnetic coercivity force indicated that coarse Cu precipitates and martensite-austenite (MA) constituents hinder the domain wall motion and behavior in effective magnetic susceptibility. Considerable improvement in the magnetic properties achieved by the aging process at 600 °C without much deterioration in mechanical strength.

Keywords: Aging; Coercivity force; Heat treatment; HSLA-100 steel; Mechanical properties.

1. Introduction

High-strength low alloy steels are a class of materials that have been developed to simple low carbon steels; and HSLA-100 steel is one of the most prominent of this category. Carbon is less than 0.06 wt. % in chemical composition of this steel. Due to the presence of very low carbon, the steel has a high resistance to corrosion, formability and weldability [1-5]. The Austenite grain size is small (approximately 10 μm), and this is one of the main reasons for the high yield strength of the steel. Carbides (or nitrides) and thermo-mechanical process cause the austenite grain size to be reduced. In addition, the yield strength was increased due to the formation of rich copper precipitation during ageing heat treatment [6, 7]. Type of interface and size of copper precipitates depend heavily on aging temperature. Thus, the size and type of the interface of copper precipitates are effective in the mechanical and magnetic properties [8-10].

Cooper-bearing steels such as HSLA-100 steel, due to its high atmospheric corrosion resistance, is widely used in naval applications. First, high yield (HY) steel was used to make the body of submarines in the United States Navy. The essential problem of HY steels was in welding operations. In order to prevent the creation of cracks due to the presence of high carbon equivalent in HY steels, it needs to be preheated; and the preheat

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process increased the cost of building ships and submarines. For this purpose, by decreasing the carbon in HSLA-80 and HSLA-100 steels, weldability increased and the cost of production of the submarines reduced. In 1984, HSLA-80 and then in 1990, HSLA-100 was produced [5]. Dhua et al. investigated the influence of cooling rate on the microstructure and mechanical properties of HSLA-100 steel [11]. They concluded that by increasing the cooling rate, yield strength, ultimate strength and impact toughness were increased. Das et al. inspected the effect of ageing temperature on mechanical and magnetic properties of HSLA-100 steel [10]. They showed an increase in hardness and a decrease in coercivity force with increasing the ageing temperature below 600 °C. Panda and coworkers found an increase in the hardness and a reduction in the coercivity by forming nano-size and coherent copper precipitates in the ageing process at 550 °C [12]. Tarafder and et al. studied magnetic properties of HSLA-100 steel by power decay exponents of Barkhausen emission signal [13]. They reduced the obstacles on the movement of the magnetic domain walls by increasing ageing temperature of 350 to 600 °C. The copper precipitates coarsened at higher ageing temperatures; thus, magnetic domain walls were pinned and coercivity force was increased.

In previous studies, researchers have conducted many studies to achieve proper ageing temperature for the improvement of mechanical and magnetic properties. A few of the studies were carried out on the effect of cooling rate on the microstructure, magnetic and mechanical properties. Evaluation of cooling rate is important because it is effective in the final microstructure of the later ageing process [11]. Moreover, the microstructure and mechanical properties of the steel non-destructive examination were tested by evaluating the magnetic properties [9]. In this study, the effect of heat treatments such as full annealing, normalizing, quenching (in water and oil) and ageing process on the magnetic and mechanical properties of HSLA-100 steel were evaluated.

2. Materials and Methods

HSLA-100 steel was produced in Esfarayen Industrial Complex (E.I.Co), Iran. The heat treatment of this product was carried out in two stages: austenitizing at 900 °C for 60 minutes and quenching in water immediately, and ageing at 650 °C for 60 minutes. Primary specimens are supplied as forms of plates with dimensions of 6000×3000×18 mm^3. Table 1 shows its chemical composition determined by spark emission spectroscopy (Spectrum Max), and it is compared with the NAVSEA T9074 standard [14]. The amount of nickel and phosphorus elements in the chemical composition of the steel sample is negligible in comparison to the standard. Specimen dimensions of heat treatment as 200×250×18 mm^3 were prepared.

Six of heat treatment cycles consist of full annealing, normalizing, quenching in the water and oil and ageing of samples were considered (Table 2). The austenitizing process was done at 900 °C for 60 minutes. After the

Table 1. Chemical Composition of the HSLA-100 steel (Weight Percent) compared to the NAVSEA T9074 standard [14].

	C	Mn	P	S	Si	Cu	Ni	Cr	Mo	V	Nb
Steel	0.04	0.88	0.005	0.005	0.03	1.55	3.91	0.56	0.64	0.005	0.03
Standard NAVSEA	0.04 - 0.06	0.75 - 1.15	0.02 Max	0.004 Max	0.04 Max	1.15 - 1.75	3.35 - 3.65	0.45 - 0.65	0.55 - 0.65	0.03 Max	0.02 - 0.06

Table 2. Heat treatment schedules of HSLA-100 steel plates.

symbol	characteristics and process	heat treatment conditions				
		austenitising			aging	
		temperature (°C)	time (min)	environmental cooling	temperature (°C)	time (min)
AR	As-Received	-	-	-	-	-
FA	Full Annealed	900	60	furnace	-	-
N	Normalized	900	60	air	-	-
OQ	Oil Quenched	900	60	oil at room temperature	-	-
WQ	Water Quenched	900	60	water at room temperature	-	-
OQ-A	Oil Quenched+Aged	900	60	oil at room temperature	600	60
WQ-A	Water Quenched+Aged	900	60	water at room temperature	600	60

austenitizing process, samples were cooled in a furnace, air, oil and water, with nominal cooling rate of 0.5, 1, 50 and 150 °C/s, respectively [11]. Then, the water and oil-quenched specimens were aged at 600 °C for 60 minutes.

To study the microstructure by scanning electron microscopy, steel was etched in 2% Nital (2cc HNO_3 + 98cc CH_3COOH). The microstructures of the samples were analyzed by field emission scanning electron microscope (FESEM) model MIRA LMU-TESCAN. Magnetic hysteresis curve samples were studied at room temperature using a vibrating sample magnetometer (VSM) model 7400- Kavirmagnet, and an LCR meter was used to measure inductance (model HM 8118-Hamg). To measure inductance, toroid-shaped samples were prepared with an outer diameter (d_2) of 14.00 mm, internal diameter (d_1) of 7.00 mm and height (h) of 7.00 mm. Inductance was measured at a frequency of 1kHz at room temperature. The relation between initial relative permeability and inductance is shown in Eq. (1) [15, 16]:

$$\mu_{ir} = \frac{2\pi L}{N^2 h \mu_0 \log\frac{d_2}{d_1}} \qquad \text{Eq. (1)}$$

, where μ_{ir} is the initial relative permeability, L is the inductance (μH), N is the number of turns, h is the height of the core (cm), $\mu_0=4\pi \times 10^{-7}$ (H/m) is the permeability of free space, d_2 is the outer diameter (cm), and d1 is the inner diameter (cm) of toroid [16]. To evaluate the real part, the magnetic susceptibility was measured by a.c. susceptometer (model 7000-Lake Shore). Susceptibility was measured at frequency of 1 kHz and a.c. magnetic fields of 200, 300, 400, 500, 600, 700 and 800 A/m at room temperature. Hardness tester model M4U-250-EmcoTest was used with a load of 30 kgf. Tensile test was carried out at room temperature and with a crosshead speed of 4 mm/min according to ASTM E8 by tensile tester model STM400-Sntam. To carry out impact tests, specimens were prepared (In accordance with ASTM E23 specifications) at longitudinal direction rolling and V notch parallel to the normal direction (ND). The CVN impact tests were conducted at -84 °C by an impact tester model SIT-300-Santam. The fracture surface of Charpy specimens was observed by a model EVO-ZEISS scanning electron microscope (SEM).

3. Results and Discussion

3. 1. Microstructure

The FESEM micrograph of AR steel is shown in Fig. 1. The lath martensite (LM) and acicular ferrite (AF) were formed owing to very low carbon content in chemical composition of the steel [10]. Moreover, AR steel was aged at a temperature of 650 °C after quenching in water, and finally the microstructure includes tempered martensite. The FESEM micrograph of the fully annealed, normalized, quenched in oil and water specimens are shown in Fig. 2(a) to (d), respectively. The cooling rate less than 10 °C/s caused the formation of granular bainite (GB) based on the continuous cooling transformation diagram of the steel [17, 18]. In addition, as-martensite-austenite constituents (MA) are formed in the cooling rate of less than 10 °C/s [17]. It is clear that the MA constituents in the fully annealed samples are larger than those in the normalized steel because the cooling rate of furnace is lower than the air and carbon has more time to be diffused in the austenite. The FESEM micrograph of the quenched in oil (OQ) and water (WQ) are shown in Fig. 2(c) and (d), respectively. Cooling rate more than 10 °C/s causes a lot of martensite to be formed [17]. As it is clear, the distance between the lath martensite was reduced by increasing the cooling rate since the driving force for the

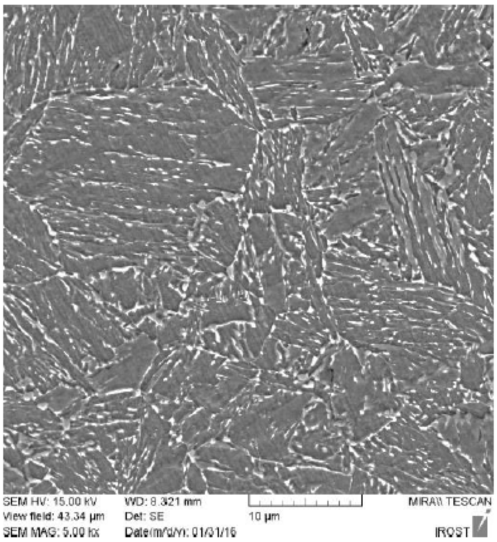

Fig. 1. FESEM micrograph of as-received steel.

formation of martensite increases by increasing the cooling rate and the density of martensite packages. The FESEM micrographs of aged samples are shown in Fig. 3(a) and (b). In general, ageing process caused changes in lath spacing structure, creation of copper precipitates in the matrix and formation of new phases of austenite and martensite [19]. The volume fraction of the lath structures was reduced by applying the ageing process (compare Fig. 2(c) and (d) with Fig. 3(a) and (b)). Moreover, the volume fractions of the lath structures of AR steel are more than WQ-A sample (compare Fig. 1 with 3(b)) because the Ac1 temperature of HSLA-100 steel is about 640 °C due to the presence of alloying elements. When the

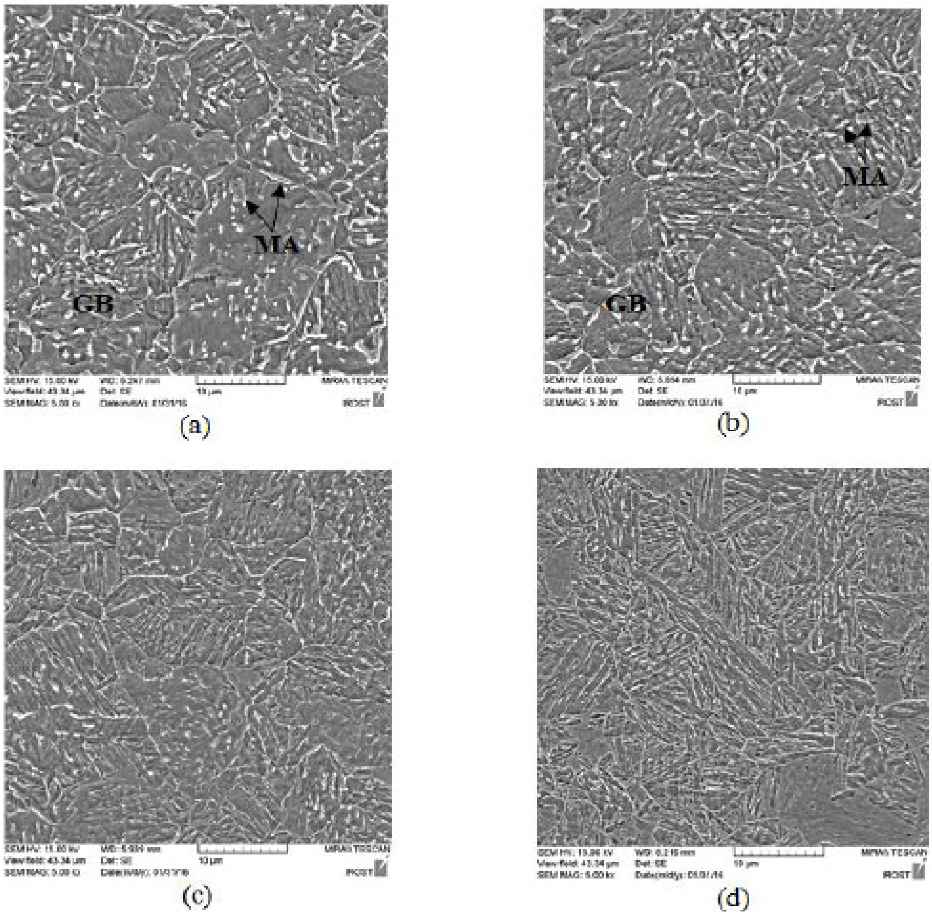

Fig. 2. FESEM micrograph of the steel quenched in: (a) furnace, (b) air, (c) oil and (d) water.

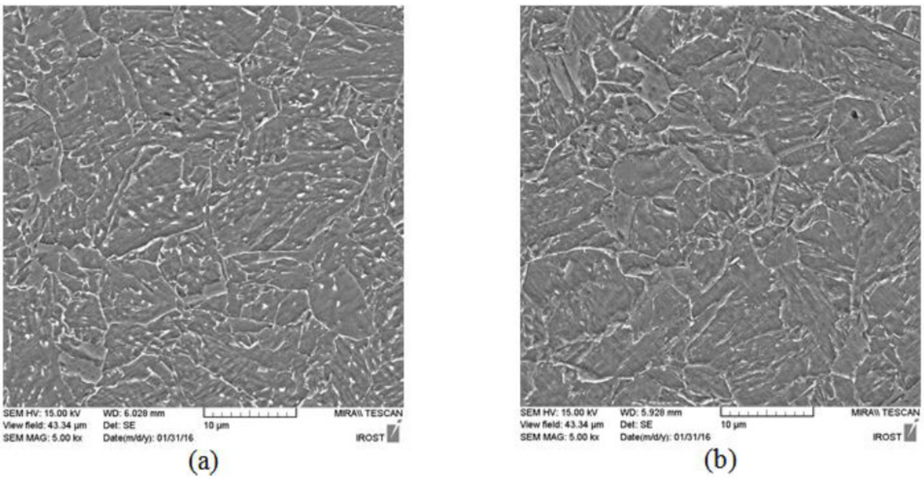

Fig. 3. FESEM micrograph of steel showing: (a) quenched in oil + aged and (b) quenched in water + aged.

steel was aged at temperatures over 640 °C, new austenite islands were formed and were transformed to martensite during cooling.

3. 2. Magnetic characterization

The magnetic hysteresis curves of as-received steel after different heat treatments are shown in Fig. 4, and the magnetic coercivity force of various conditions is shown in Fig. 5. The highest coercivity force was achieved in water quenched sample (WQ), which is related to high density of dislocations and lath structure in WQ steel. Both of these factors are barriers to the movement of magnetic domain walls, and increase the coercivity force [20]. Because the phases are the same in WQ and OQ specimens, the coercivity was measured approximately equal in both. For the same reason, the approximately equal coercivity force was obtained in fully annealed (FA) and normalized (N) steels. The presence of martensite-austenite (MA) constituents in fully annealed and normalized samples inhibited the movement of magnetic domain walls; and coercivity force was increased. The lowest coercivity force was obtained in WQ-A and OQ-A specimens. The increase in distance between the lath structures caused coercivity in aged steels to be reduced (Fig. 3). Additionally, the copper precipitates were not so great in ageing process at 600 °C. In other words, the coercivity is not affected if the width of the secondary phase is less than that of the magnetic domain walls [10]. The coercivity force of AR steel aged at 650 °C during production process is higher than that of WQ-A steel. The reasons for this are that the copper precipitates coarsen and the distance between lath structures of AR steel is reduced (Fig. 1). The variations of magnetic initial relative permeability with type of heat treatment are shown in Fig. 6. As known, the permeability was dropped in the OQ-A and WQ-A specimens because the copper precipitation (non-magnetic phase) was formed and dispersed in the field [21].

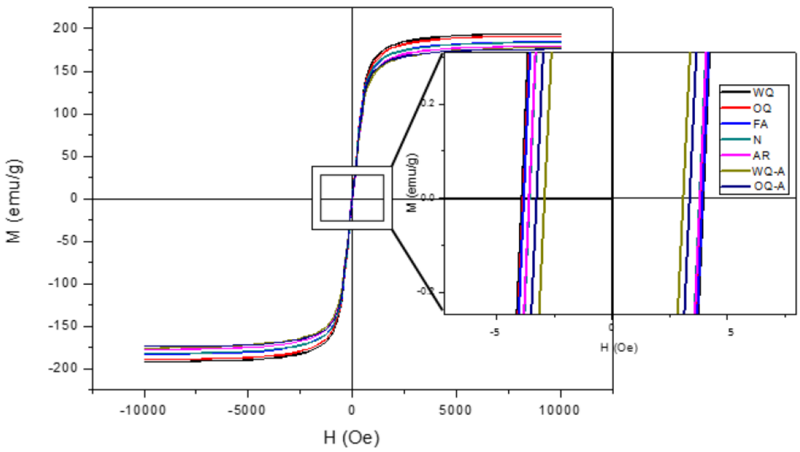

Fig. 4. Magnetization curves of HSLA-100 steel in: water-quenched (WQ), oil-quenched (OQ), full-annealing (FA), normalizing (N), as-received (AR), water-quenched+ aged (WQ-A) and oil-quenched+ aged (OQ-A).

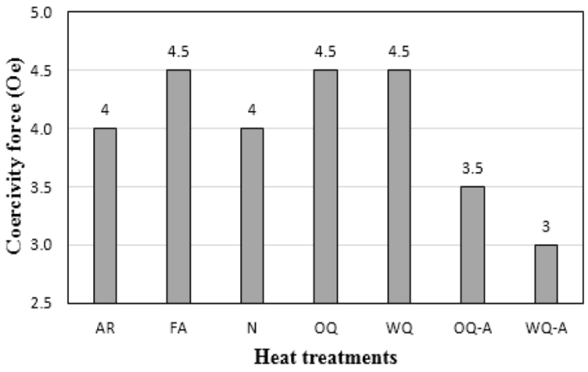

Fig. 5. Variation of magnetic coercivity force of HSLA-100 steel in: as-received (AR), fully-annealed (FA), normalized (N), oil-quenched (OQ), water-quenched (WQ), oil-quenched+ aged (OQ-A) and water-quenched+ aged (WQ-A).

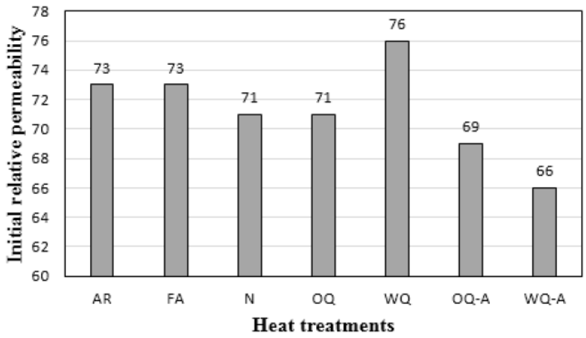

Fig. 6. Variation of initial relative permeability of HSLA-100 steel in: as-received (AR), fully-annealed (FA), normalized (N), oil-quenched (OQ), water-quenched (WQ), oil-quenched+ aged (OQ-A) and water-quenched+ aged (WQ-A).

Variations of real part of the magnetic susceptibility with Hd.c field are shown in Fig. 7. The susceptibility was increased linearly by increasing the applied field. In general, impurities and secondary phase (rich of copper precipitates) in field of steel are responsible for the pinning of the magnetic domain walls. These factors influenced the real part of the magnetic susceptibility. The magnetic domain walls will be in a position of stability with minimal energy under the external field if they are not of pinning factors. If there are pinning factors, the magnetic domain walls are pinned in various positions and external field leads to apply pressure on them that they move towards a more stable position. Although jump magnetic domain walls are a reversible process, the presence of an external field in a specific direction prevents the return of domain walls to their previous position [22, 23].

3. 3. Mechanical characterization

The hardness (VHN30), yield stress (YS), ultimate tensile strength (UTS), elongation percentage (%EL), reduction of area percentage (%RA) and Charpy V notch test of the number (CVN) at temperature -84 °C of the steel for various heat treatments are specified in Table 3.

The lowest VHN, YS and UTS were obtained in as-received (AR), fully annealed (FA) and normalized (N) specimens. While, as-received steel aged at 650 °C is found in industry, but YS and VHN are dropped, since the non-coherent and coarse copper precipitates were formed in AR steel aged at 650 °C. In these conditions, there is no accordance between crystals of field and copper precipitates and dislocations loop around the second phase (Orowan theory). In addition, copper precipitates coarsen and their volume fraction is reduced in the ageing at 650 °C. Increase in the distance of copper precipitates caused the movement of dislocations to be facilitated. The YS, UTS and VHN of normalized and fully-annealed steel have been much lower owing to the occurrence of the softer phase. Additionally, remains of the solution elements in the field were low because the cooling rate is slow in these specimens. The highest VHN, YS and UTS were obtained in WQ and OQ samples. The formation of lath martensite with a lot of high dislocations density and solution of elements caused an increase in the VHN and YS. Hardness value of WQ-A and OQ-A steel are the same as that of water quenched steel. The high hardness in aged samples at 600 °C was caused by the formation of coherent copper precipitates, which make dislocation motion difficult. The highest El and RA pct were measured in AR and aged steels. Ageing process at temperatures over 600 °C reduced dislocation density and coherent copper precipitation was removed; as a result, elongation increased. Reduction in El and RA pct of FA and N samples is due to the brittleness of martensite-austenite (MA) constituents. The MA constituents are more brittle and harder than the lath martensite formed in the WQ and OQ specimens.

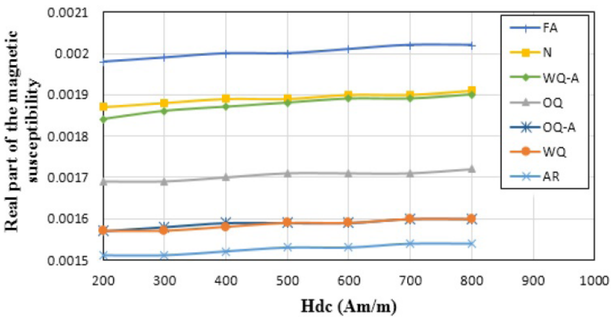

Fig. 7. Effective magnetic susceptibility as a function of H_{dc} in: fully-annealed (FA), normalized (N), water-quenched+ aged (WQ-A), oil-quenched (OQ), oil-quenched+ aged (OQ-A), water-quenched (WQ) and as-received (AR).

The highest impact energy was measured in as-received and aged steels because lath martensite and coarse copper precipitates prevented the propagation of cleavage cracks. Very low impact energy in fully annealed and normalized conditions were formed as a result of martensite-austenite constituents and granular bainite. The small size of martensite-austenite constituents in the normalized rather than fully annealed conditions increased the impact energy (Fig. 2(a) and (b)). Moreover, the granular bainite matrix with very low carbide and

Table 3. Mechanical properties in different conditions compared to the as-received steel.

Treatment	VHN30	YS (MPa)	UTS (MPa)	El (pct)	RA (pct)	CVN at -84 °C (J)
AR (as- received)	273	820	940	19	71	210
FA (full annealed)	270	775	880	14	56	18
N (normalized)	273	830	940	14	56	38
OQ (oil quenched)	305	950	1075	16	62	190
WQ (water quenched)	310	1050	1140	13	56	185
WQ-A (water quenched+ aged)	300	900	900	19	70	200
OQ-A (oil quenched+ aged)	290	795	850	18	70	200

nitride precipitates are suitable for propagation cracks. The high impact energy was achieved in water and oil quenched steels (approximately 190 J) because lath martensite propagation of crack was prevented.

The dimples in fracture surface of the impact tested AR, oil and water quenched steels are shown in Fig 8(a) to (c), respectively, and they confirmed the high impact energy obtained in these steels.

This theory is correct for aged steels (Fig 9(a) and (b)). Moreover, the presence of a cleavage structure in the fracture surface confirmed the low impact energy in fully annealed and normalized samples (Fig 10(a) and (b)).

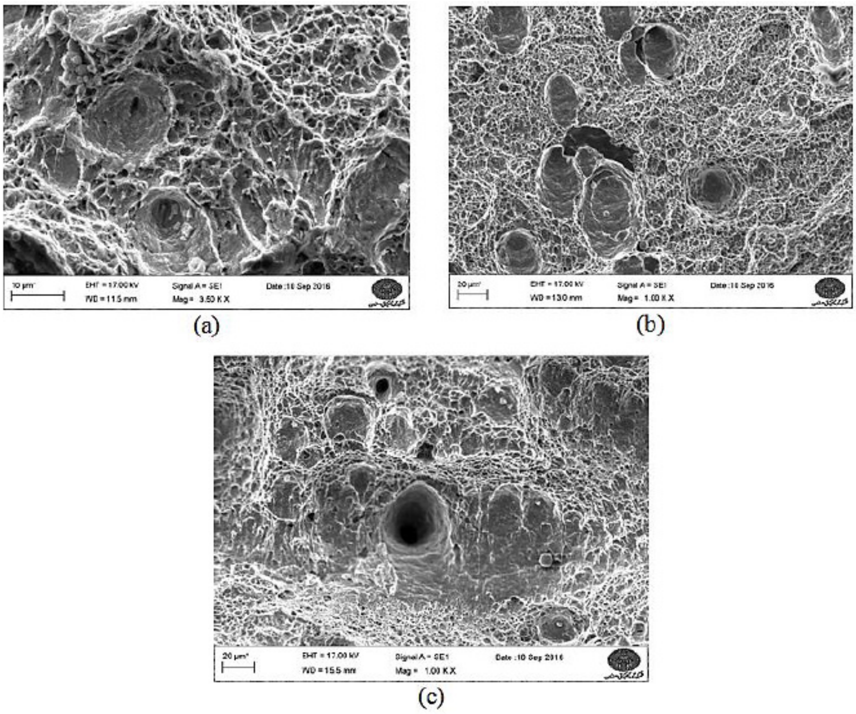

Fig. 8. SEM fractographs Charpy tested at -84 °C of the as-received (a), oil-quenched (b) and water-quenched (c).

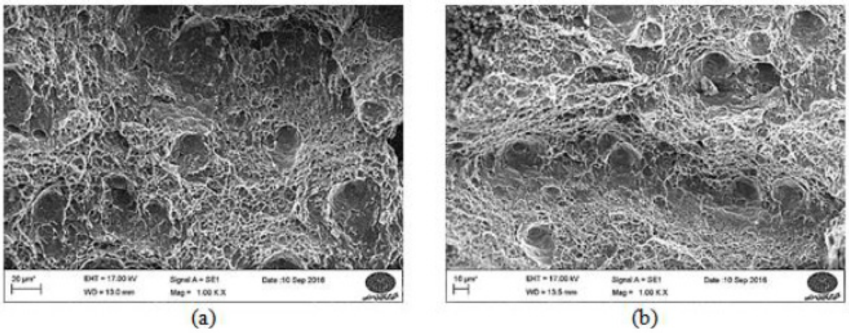

Fig. 9. SEM fractographs Charpy tested at -84 °C of the oil-quenched+ aged (a) water-quenched+ aged(b).

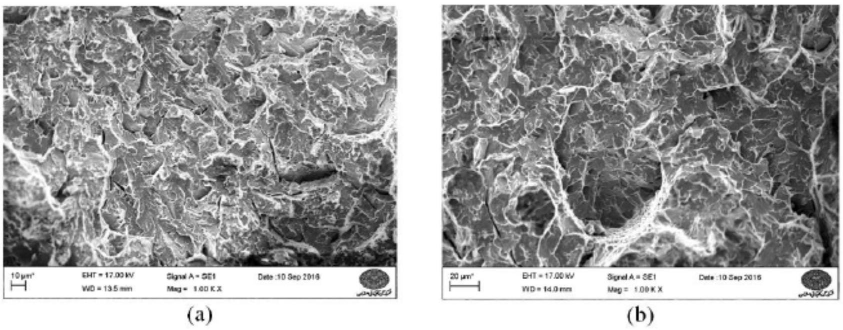

Fig. 10. SEM fractographs Charpy tested at -84 °C of the fully-annealed (a) and normalized (b).

4. Conclusions

A study has been carried out to check the effect of full annealing, normalizing and ageing processes at 600 °C on the microstructure, mechanical and magnetic properties of HSLA-100 steel. The following conclusions are drawn:

- Microstructures showed that the martensite phase was in the cooling rate of 0.5 to 150 °C/s.
- By ageing process at 600 °C, the volume fraction of the lath structure was reduced compared to as-received, oil and water quenched specimens.
- The results of magnetic coercivity force indicated that coarse copper precipitated and MA constituents hindered the domain wall motion and the observed behavior is effective in magnetic susceptibility. Moreover, initial relative permeability was reduced by fine copper particles precipitated and dispersed in a matrix.
- The highest hardness value achieved in aged steel was the same as that of quenched steel (300 and 310 VHN, respectively).
- After quenching steels, the highest YS and UTS were achieved at water-quenched steel with aged steel at 600 °C. Moreover, the highest elongation and reduction areas were obtained at as-received and aged steels (approximately 19 and 70%, respectively). In addition, the highest CVN energies were obtained at the as-received and aged steels (approximately 200 J).
- The best combination of strength, toughness and magnetic properties is obtained when the steel is aged, quenched in water and then aged at 600 °C.

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