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Study of the Structural Properties and Microscopic Hardness of a Carburized Stainless Steel Alloy AISI304

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Abstract- The results of the steel surface hardening (AISI304) by carburizing indicate that the time of carburizing had a significant effect on the thickness of the carbon layers and the carbon content. Also, carbonization and cooling processes by (air, water, oil) changed the microstructure of steel, which is related to the gradient of carbon diffusion on the surface and inside the material. The microstructure was divided into two regions; Surface area consisting of; Austenite, Perlite, Martensite, Pinite and Martensite, for carbonated and carbonated specimens cooled by (air, water, oil), respectively, and the core region consisting of austenite for all samples. The increase in the carbon content also led to an increase in the Vickers hardness, as it reached a maximum value (383.18 Hv) at a time (4 hours), then decreased with the carburizing time increase as a result of the increase in the amount of carbon, which led to the brittleness of the surface. It was also observed that the hardness changed with cooling method of the steel as a result of the change in the steel microstructure.

Keywords- Austenite Steel, Carbon Diffusion, Carbon Hardening, Heat Treatment, Steel Quenching, Vickers Hardness.

I. INTRODUCTION

The use of surface materials in many fields of engineering has increased tremendously in recent years. Surface hardening is in itself a dynamic operation, as a number of variables affect the efficiency of the process and the quality of the components.

In steels, surface hardening can be done by altering the surface composition of the part by diffusion heat treatment [1].

Austenitic stainless steel is common in the petrochemical, aerospace, and food processing industries for structural purposes. This is due to their excellent corrosion resistance and good mechanical properties, such as resistance, ductility and formability [2].

These steels however, have relatively limited area hardness and low wear resistance, because of the austinian structure, and scientists and specialists have therefore studied and developed the requirements of these alloys in many ways [3].

In order to enhance surface properties, diffusion and thermo chemical surface treatment techniques such as carburizing — inward diffusion of surface carbon, nitriding, cyanide or carbon dioxide, and diffusion coating have been used without impairing the corrosion resistance of the steel [4].

The high-carbon surface layer is imparted to steel in Pack Carburizing by heating it in contact with carbonaceous material such as Charcoal ($BaCO_3$). Salts in container weather stepwise at this temperature as the following reaction [5]:

$$BaCO_3 \leftrightarrow BaO + CO_2$$
$$CO_2 + C \leftrightarrow 2CO$$
$$2CO \leftrightarrow CO_2 + C_{atomic}$$

(1)

The effect of ($BaCO_3$) on carburization is the mechanism of acceleration. Carburization most rapidly occurs on the steel surface by decomposition of (Co) molecules.

The absorbed carbon atoms diffuse further down their gradient of chemical potential and create a profile of carbon concentration [6]. The reduction reaction occurs on the carbon releasing steel surface, which, is then diffused into the surface due to the high temperature [7].

Carburization is of interest to many sectors of industry because it is possible to enhance the resistance of the outer surface to corrosion and the external resistance of the material relative to the entire body, improves the mechanical properties of metals, and increases the resistance of the surface to abrasion and wear.

If the surface is subjected to external friction while maintaining the strength of the rest of the body, giving the substance an appealing external appearance and giving it an esthetic flair that is accomplished by covering the surface with metallic materials or by processes of coating[8][9].

Austenitic stainless steel carbureting is typically performed at high temperatures up to (1010 ° C), allowing quick precipitation on the carbured surface of chromium carbides [10] [11].

The hardness of the carbohydrates compounds is typically affected by the chemical composition of the stainless steel, the carbon profile of the carburized layer and the harden ability of the steel and the carbureted layer, the austenisation temperature and time until quenching, the strength and the cooling process[12][13].

After carburizing is carried out, the steel is immediately soaked at a low temperature by media like water, oil, or air. Water is the fastest cooling medium for the products. Oil has a lower cooling rate than water but is still commonly used because it has a higher viscosity and allows much more stable heat treatment.

Air is the lowest cooling rate cooling medium that contributes to lower thermal shock that is the source of thermal stress [14] [15].

The purpose of this method is to harden the steel by changing the structure from the large, shape-edged, coarse and irregular structure of the austenite structure to the marten site structure of the fine grain structure of hardened carbide [16].

II. EXPERIMENTAL

1. Materials and Methods:

The analysis was performed on stainless steel samples (AISI304). The samples were taken from long rods and milled to a diameter of (9 mm) and cut to a length of (3 mm) with a lathe machine and then smoothed with aluminum oxide paper, starting from grade (400) to grade (1200), for surface finishing and alcohol-cleaning, in order to remove oxide layers and external surface irregularities, to increase carbon uniformity.

The chemical composition of the alloy used is presented in Table (1):

Table 1. The stainless steel nominal composition (AISI 304) [17]

Element	Wt. %
C	0.07
Mn	2.00
P	0.045
S	0.03
Si	0.75
Ni	9.0
Cr	18.0
Fe	Reminder

2. Pack Carburization:

In pack carburization, a semi-closed cylindrical steel box with a length of (10cm) was used and a specimen was carried with it, then a mixture of carbon powder (charcoal) (70 wt. %) and barium salt ($BaCO_3$) (30 wt. %) was completely packed.

Clays blended with moderate water were used to close the steel box to prevent carbon oxide from leaking and to prevent the reintroduction of unwanted furnace gas into the steel box [18].

The carburizing process was then performed by placing this box at the center of a vacuumed furnace at temperature (1050C^{o}) for (2, 4, 6, and 8) hours.

3. Quenching and Tempering:

A series of thermal treatments were carried out during the carbonization process of the samples, which were divided into:

3.1 Group (A): Temperature was reduced to (800C^{o}) for (30 min) and then water-quenched, held for (2 minutes) stirring in water, followed by (1 h) temperature treatment at (200C^{o}), then cooled to room temperature.

3.2 Group (B): Temperature was reduced to (800C^{o}) for (30min) and hot oil (70C^{o}) was subsequently quenched and held for (2 minutes) stirring in oil, followed by (200C^{o}) air temperature treatment for (1h), then cooled to room temperature.

3.3 Group (C): The temperature was reduced to (800C^{o}) for (30min) and then cooled to room temperature in the air, followed by tempering for (1h) at (200C^{o}) and then cooled to room temperature.

4. Sample Surface Preparation for Photographic:

The treated samples were placed using a molding unit for ease of polishing, with the cut face facing down to the polishing side. These samples were surface polished by rotary grinders with (320, 400, 1000, 1200) aluminum grinding paper. Following this method, the specimens were picked for (15sec) in (2% HNO_3+ 98% ethanol) (Nital) solution, thoroughly rinsed in distilled water, then rinsed in acetone and then dried.

5. Micro hardness Test:

The surface hardness of the carburized specimens was measured using a micro-hardness tester (Karl Kolb) fitted with a Vickers Diamond Indenter with a load of (1500gm), with a holding time of (15 sec).

The micro hardness of the hard carbon layer to the center of the carburized specimen was measured at different locations on the carburized surface and also in the cross-section region.

The micro hardness of Vickers is given by the equation [19]:

$$HV = 1.8544 \frac{p}{d^2} \left[kg / mm^2 \right] \quad (2)$$

(2)

Where: P: is used for the load; d: is the average indentation of the diameter left by the pyramid.

III. RESULTS AND DISCUSSIONS

1. Weight and Thickness Measurements:

Weight gain data were determined by packing carburizing the samples together for each temperature treatment analyzed (1050C^{o}) for (2, 4, 6, 8hrs).

Table (1) displays the average weight gain of the specimen and the thickness of the coating as a function of time at (1050C^{o}) temperature:

Table 2. Average specimen weight gain and coating thickness for carburizing samples at different times.

Temp. (C^{o})	Carburizing time (hr)	Thickness of carbon layer M (μ)	weight gain of carbon (gm/cm^2)
1050	0	0	0
	2	36	0.0067
	4	53	0.0089
	6	65	0.0096
	8	74	0.0104

It is obvious that the rate of carbon absorption and the thickness of the carbon layer have increased with growing time [20].

This is consistent with the researcher Maziar Ramezani, et al., when researching carbon diffusion in (H13) steel during austenitization under various atmospheric conditions and period of treatment [21]. This is because the diffusion of carbon atoms on the surface of the alloy has intensified and the thickness of the carburized layer has increased [22].

In addition, the microstructure of fine grain (304AISI) also enhances carbon atomic motion into the material, as it gives more pathways to grain borders [23].

2. Microstructure Investigation:

After Pack carburizing in (2, 4, 6, 8 hr) at (1050Co) Carbon has strongly dissolved in (FCC) austenite, which subsequently influences the transformation of austenite into another surface structure, so that the

microstructure obtained is the retained austenite and the microstructure changes to austenite with respect to the depth of the surface [24].

As a result, the proportion of the retained austenite decreases the initiation from the surface with the determination of the core, as the proportion of carbon decreases, see fig (1).

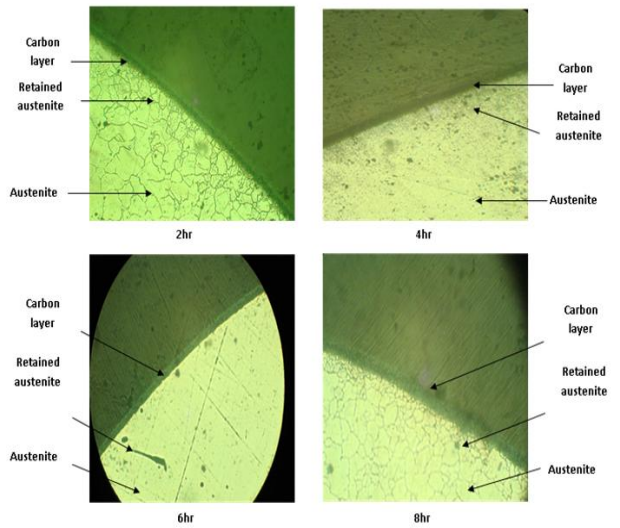

Fig 1. Microstructure of Carburizing specimens, 100X.

After carburization, the carbon content in the carburized layer is very high; causing brittleness and this phenomenon would reduce the hardness and thus decrease the strength.

In order to minimize the concentration of carbon on the surface of the sample and to achieve good ductility, heat treatment represented by a rapid cooling (quenching) process can be applied after carburization, in order to ensure the highest possible hardness of the centre of the components.

There are three forms of quench [25]:

- Quenching in air.
- Quenching in water.
- Quenching in oil.

During water quenching, the steel frameworks shift to a stronger final framework from the initially unhardened structure (austenite) (usually marten site). On the part surface, the martensitic structure begins to develop after the surface reaches a certain temperature and expands as the core cools into the part core. Carbon solubility in austenitic steels is slightly lower.

As a consequence, the carburizing of the material can lead to carbides forming in grain and at grain boundaries such as ($M_{23}C_6$, M_7C_3 and M_3C_2), where (M) stands for a mixture of (Fe) and (Cr) atoms see figure (2).

In other terms, as a result of the quenching, steel expands. This rise in part volumes (from austenite to marten site) is about 4 percent and leads to a substantial distortion and crack age and residual stress on the surfaces, which are harmful to the mechanical properties, resulting from the traditional quenching process.

Therefore, in order to alleviate the quenching stresses and to avoid the high degree of undesirable formation of carbides forming at the grain boundaries and residual tensile stresses, tempering of the carburized samples was required, these samples only need to be tempered in order to provide a homogeneous microstructure of the tempered marten site with minimum visible carbides [26][27].

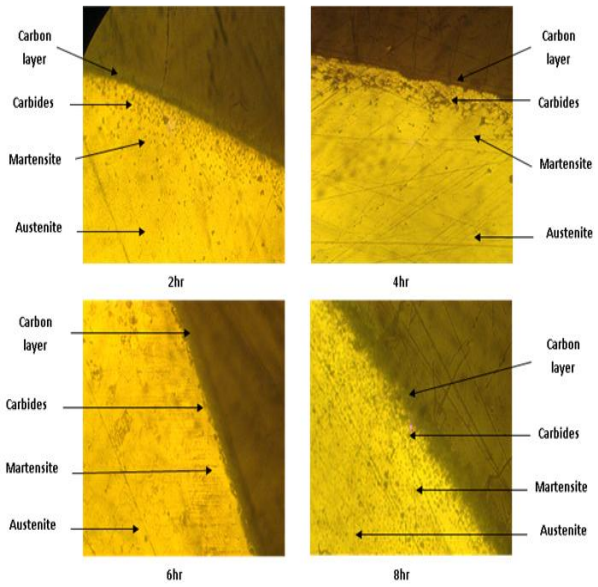

Fig 2. Microstructure of group (A) specimens, 100X.

Figure (3) is a micrograph of (304AISI) alloy after vacuum furnace carburizing and heat treatment at (1050C^o) during (30 min) and hot oil quenching at (85C^o) followed by vacuum furnace tempering at (200C^o) during (2hr). Due to the insufficiently high cooling rate, during the quenching process, bainite and marten site can form.

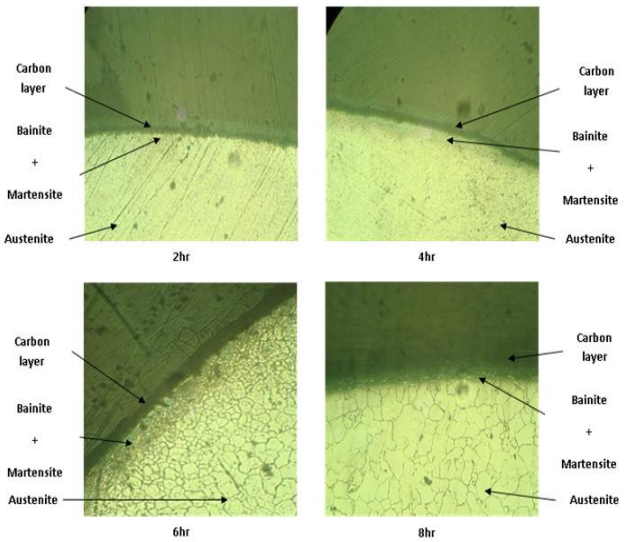

Fig 3. Microstructure of group (B) specimens, 100X.

This structure is the same as the marten site structure, but the hardness is smaller than the hardness of a finer marten site near the core observed [28].

On the other hand, we used this method in quenching because, compared to a slower quench in oil, quenching in water provides better part hardness, but the likelihood of part cracking or distortion is higher when quenching in water [29].

The micrographs of the samples quenched in the air and tempering are shown in figure (4). As a result of the diffusion between carbon and alloy elements, this treatment allows the matrix more time to saturate with carbon and alloying elements. The diffusion of carbon into the steel matrix is the diffusion of the interstitial, where the interstitial site location would be filled by the carbon.

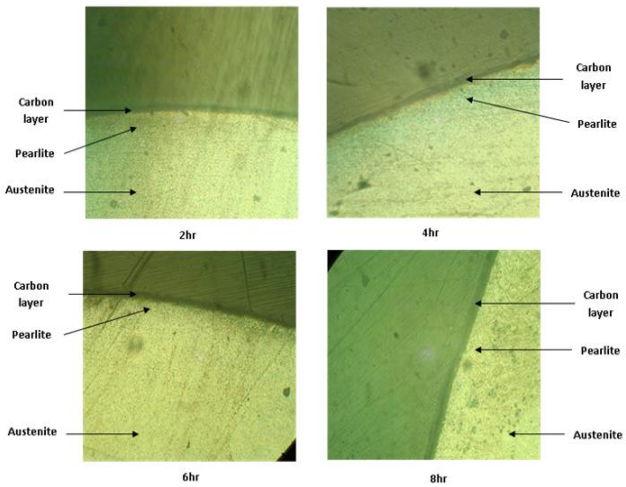

Fig 4. Microstructure of group (C) specimens, 100 X.

In addition, the diffused carbon may become a solid solution or precipitate with Fe / Cr. The state of the carburizing process depends on it [30]. It is obviously evident that the structural part was pearlite with no carbides.

3. Micro Hardness Results:

From the results shown in Figure (5), all samples that had carburized at (2, 4, 6, and 8) hour had a hardening effect on the surface. The hardness in the area (1.2mm-2mm) of all packed carburized samples ranged from (222.11HV) to (255.46 HV) outside the carburized layer.

It should be noted that the hardness area (0.05mm - 1.2mm) was comparatively greater than the core toughness, indicating a moderate carbon intake and a low carbon enhancement in the carburizing method [31].

The maximum hardness value was obtained at the carburized layer closest to the carburized surface area, where the maximum hardness is obtained at about (383.18HV) in samples of heat treated for (4) hours and (379.54HV) in samples of heat treated for (6) hours.

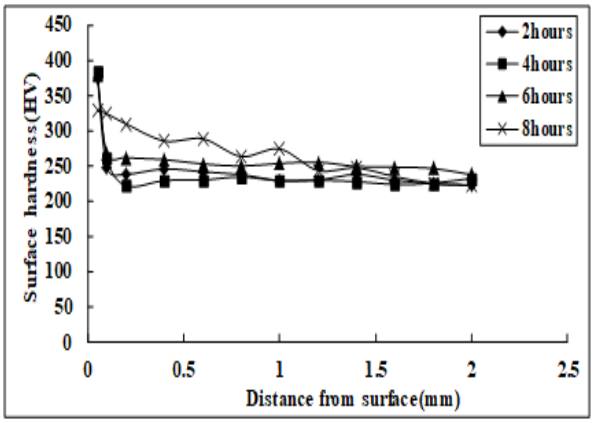

Fig 5. Hardness profile of samples of pack carburization heat treated at (1050C°) for 2, 4, 6 and 8 hours.

This increase can be explained as a moderate carbon concentration in the region (0.05 mm) on the surface, another explanation for increasing hardness may be due to the conversion of the austenite present in the case of retained austenite as shown in fig(1).

These carburization methods may also be attributed to high residual compressive stresses caused by the distortion of the interstitial carbon lattice by the

diffusion of carbon through the permeated surface [32].

On the other hand, the minimum hardness we get about (375HV) in heat samples treated for (2) hours due to a small carbon uptake content that could be due to the lowest heat treatment time. Another minimum hardness we can obtain when the samples are heat treated for (8) hours, which is equal to (329.22HV).

The sudden decrease in the hardness value can be due to an increase in the time of carbonization leading to an increase in the amount of carbon acquired as a result of the diffusion process, resulting in brittleness as a result of the high concentration of carbon on the surface in the area (0.05 mm). This is a phenomenon that will minimize hardness [33].

For certain samples that were heat treated and quenched in water and hot oil, the difference in hardness was studied. We can find that the cooling method has an effect on the material core hardness, as can be seen in figure (6) a and b.

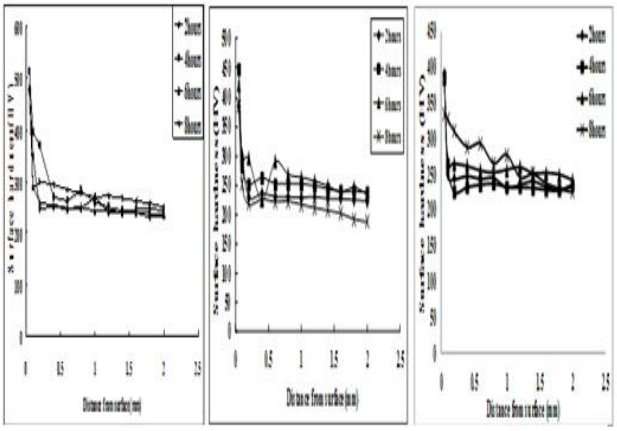

Fig 6. Hardness profile of; a-group (A), b-group (B), c- group (C) specimens.

Graphs show that water-cooled samples generally have higher hardness values (247.5 - 511.44HV) than hot oil-cooled samples (217.79-449.01HV).

Improving the hardness by quenching in water may be due to many factors, such as: the rapid cooling rate of the water, the conversion of the retained austenite present in the case of martensite, which is a much stronger structure, the presence of fine dispersion of small carbide particles forming on the boundary of the austenite grain as a result of combination the carbon atoms with chromium atoms. These carbides hindered the movement of dislocation [34] [35].

This increase in hardness in this way is not good due to the sudden quenching of much of the distortion and cracking in the samples and does not allow carbon to disperse to the sample rather than to quenching in hot oil.

Another notable difference is that the surface hardness in the region (1.4mm-1.6mm) of the sample that quenched in water for (2, 4, 6) hours of carburization was approximately the same (239HV) while the surface hardness in the same region of the sample that quenched in hot oil for (2, 4, 6) hours of carburization ranged from (227.5, 244.22, 249.23HV - 226.87, 240, 238.73HV) respectively.

It can be seen from the micrographs, the surface hardness in the region (0.05mm-2 mm) of the sample, that the quenching in water for (8) hour carburizing that varies from (473.05HV - 249.3HV) is higher than the surface hardness in the same region of the sample than the quenching in hot oil for (8) hour carburizing that varies from (384.68HV-187.7HV).

Figure (6c) shows the change in hardness values for all four heat treated specimens of group (C) from the carburized layer (surface) to the core. The surface hardness values (390.12HV – 333.45HV) have been shown to be extremely strong, and the hardness has decreased with increasing depth steadily (up to 1.8 mm).

With a hardness scale (233.66HV-225HV) in comparison to the surface the core was found slightly softer (or case). It should be noted that there is no substantial difference in hardness with respect to the four heat treated groups (C). Due to high residual compressive strain due to interstitial carbon glaze distortion, the very high surface hardness of these carburizing techniques can be achieved by diffusing carbon through the alloy surface[36][37].

IV. CONCLUSION

The carburizing process in (2, 4, 6, 8) hours at (1050C^{o}) showed different weights and thicknesses with a uniform and dense morphology of the carburized layer obtained from all carburized samples.

The lower cooling rate obtained by hot oil and air from quenched steels generally showed a stronger and finer stable microstructure composition as well as a limited percentage of defective material free of carbides and cracks compared to steel samples quenched in water, but the hot oil has a low hardness compared to water and air quenched samples.

Rapid cooling of steels by water, as well as lower carbon solubility, carbides will form in grain and at grain bounds, and this will lead to a significant amount of distortion and cracking and to the development of residual stress on the part of the surface of the specimens, which will lead to increased hardness.

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