Investigation and characterization of coating and carburizing AISI 1011 steel

Diyar Abdulrazzaq Jabbar

Assist. prof. Dr. Zeyad Doshan Kadhim

Materials Engineering Department, Al-Mustansiriyah University, Baghdad, Iraq

corresponding author’s e-mail: diyarabd19942017@gmail.com

Abstract. In this research work, the effect of coating layer thickness of nickel, which electrodeposited on low carbon steel AISI (1011), on mechanical properties has been studied. It also studied the mechanical properties of the carburized alloy at 950°C at the time of carburizing five hours. It was found that fatigue strength for this alloy increased as thickness of the nickel-coated layer increased. Also, the carburized specimen has a fatigue strength higher than the untreated metal. The metal that was treated with the carburizing process and nickel plating for 15 minutes has the best fatigue strength. The fatigue strength improvement against non-plated specimens was between 3.93% and 16.87%. Furthermore, hardness of metal increased for specimens treated with electrodeposition, and it became 219.7 Hv for metal coated with nickel at 15 minutes and had a coated layer of 20.42 µm. The hardness value became 431.65 Hv for carburized alloy treated with plating for 15 minutes. However, there was an improvement in the surface roughness due to electroplating.

Keywords: fatigue strength, hardness, low carbon steel, coating, surface roughness

1. Introduction

Steel is a very important industrial material, as it has many more applications than any other engineering material [1]. Steel with carbon content less than 0.25 is termed as low carbon steel, while that with carbon content between 0.25% and 0.65% is known as medium carbon steel. If the content of carbon is high and ranges from 0.65% to 1.5%, then the steel is termed as high carbon steel [2]. Low carbon steel has high ductility and moderate strength and is used for its extensive fabrication properties for structural purposes, in buildings, bridges, ships, and cars [3]. Fatigue is the permanent, localized, and progressive structural change that occurs in metals and alloys subjected to fluctuating or repeated strains at values of stresses less than the tensile strength of metal [4]. Fatigue strength is an important mechanical property. However, the failure of fatigue starts on the metal surface [5]. Fatigue failure has four different stages: crack initiation, crack growth, crack propagation, and final rupture [6]. Most surface treatments produce compressive stresses in the metal surface, which reduce the probability of crack initiation and its expansion at the interface between the surface and core, thus increasing resistance to fatigue [7]. Carburization is an important and widely used process for surface hardening. This process I performed with the addition of carbon to the surface of the metal (low carbon steel) at a temperature between 850 and 950°C [8]. The nickel electroplating can be used for three purposes: decorative, functional, and electroforming. However, the appearance and other properties of the electrodeposited nickel make this type of plating suitable for many applications by controlling the composition and the operating parameters of the plating solution [9].
2. Experimental Methods

A low carbon steel AISI (1011) has been used. The chemical analysis of this alloy was performed by the Thermo ARL 3460 optical emission spectrometer. The results can be summarized in the table below. This chemical analysis was carried out at 21 °C and moisture rate 44%.

| Elements | C%   | Si%  | Mn%  | P%     | S%     | Cr%   | Mo%   | Ni%   | Al%   | Cu%   | Fe%   |
|----------|------|------|------|--------|--------|-------|-------|-------|-------|-------|-------|
| Standard value | 0.08-0.13 | - | 0.6-0.9 | 0.04 (Max) | 0.05 (Max) | - | - | - | - | - | - |
| Actual value | 0.0912 | 0.185 | 0.667 | 0.0233 | 0.0144 | 0.0652 | 0.0028 | 0.0338 | 0.0075 | 0.0457 | Bal. |

Fatigue specimens were prepared according to DIN, as illustrated in Figure (1). To get suitable and accurate dimensions for the fatigue specimens according to the standard dimensions of (HSM20 rotating bending fatigue machine), specimens were manufactured using the conventional lathe machine (Harrison 600, M350, EW700). Stress relief process was carried out for all specimens at 200 °C for three hours by using an eclectic furnace.

Fatigue samples were treated by using the pack carburization process as shown in Figure (2). Samples packed were with carburizing compounds in a metal box, which was sealed with clay and then placed in an electric furnace, where it was heated to 950°C and held for five hours. However, samples were quenched in oil. Table (2) shows the carburizing components used in the carburizing process.

| Carburising component | charcoal | BaCO3 | CaCO3 | Na2CO3 |
|-----------------------|----------|-------|-------|--------|
| wt %                  | 80       | 10    | 9     | 1      |
Surface preparation of metals for electroplating was done in order to give good bonding of coating to the substrate and obtained finished parts. Surface preparation process consists of the following steps:

1- Alkaline cleaning: To remove fats from the outer surface. Sodium hydroxide is the best alkali in cleaners, so samples were immersed in hot solution of 75 gm/l NaOH and held for 5 minutes at 50°C.

2- Rinsing with water.

3-Acid cleaning: To immerse samples in dilute hydrochloric acid bath holding for two minutes at room temperature, in order to remove oxides and rust from the surface.

4- Rinsing in water.

After the surface preparation process for coating, samples were coated with nickel by electrodeposition. The coating with nickel was done for three groups of samples with different coating times. Group one, specimens of the base metal was subjected to electroless nickel plating for five minutes. while; the electroless nickel plating performed to the second group of specimens for ten minutes. However, group three consist of metal specimens subjected to electroless nickel plating for fifteen minutes. The nickel electroplating was done at 55 to 60 °C, current 7 A, voltage 3 volt, 4.5-5 pH, and the electroless nickel bath contained distilled water, 300 g/L of NiSO₄, 30 g/L of NaCL, 40 g/L of H3-BO3, and polishing materials. After the nickel electroplating, the specimens were immersed in sodium dichromate composite in order to fixing color and prevent rust. After finishing the plating process, the specimens were dried. Ten samples of the selected alloy were manufactured as fatigue test specimens for each test except one sample for the purpose of the tensile test, as shown in Table (2). Figure (3) shows number of specimens for each group that is used for fatigue test.
Table (3) Categorization of the fatigue specimens

| groups | conditions |
|--------|------------|
| A      | Ten untreated specimens |
| B      | Ten specimens subjected to nickel coating process for 5 minutes time |
| C      | Ten specimens subjected to nickel coating process for 10 minutes time |
| D      | Ten specimens subjected to nickel coating process for 15 minutes time |
| E      | Ten specimens subjected to carburizing process for five hour time at temperature 950°C |
| F      | Ten specimens subjected to (carburizing process for five hour time at temperature 950°C + nickel coating process for 15 minutes time) |

2.1 Fatigue Test

The fatigue test machine of type (HSM20) rotating bending fatigue machine is used to do all fatigue tests, with the constant and variable amplitudes, as illustrated in Figure (4). The samples are exposed to an applied load from the right side of the perpendicular to the axis of the samples, developing a bending moment. The surface of the samples is under tension and compression stresses when it rotates. Fatigue testing machine components are illustrated in Figure (4). Value of the load (P) is measured by Newton (N) and applied to a specimen for known value of stress (σ) is measured by (MPa) and can be obtained from applying relation below:

\[ \sigma = \frac{32 \times L \times P}{\pi \times d^3} \]

where
- P = force in Newton.
- L = arm of the force which is equal to (125) mm.
- d = diameter of the specimen in (mm).

The constant fatigue test was accomplished in the laboratory air (the relative humidity was 25_30%) at the room temperature on (HSM 20) rotary fatigue bending machine as illustrated in figure (4), with the stress ratio of R= -1. Cycle frequency was 50 Hz and rotating speed used is 3000 cycle/min.

Figure 4: Fatigue testing machine HSM20 (rotary bending)
3. Result and discussions

3.1. Microscopic examination results

From Figure (5), it can be noticed that the microstructure of low carbon steel AISI (1011) after being subjected to stress relief process at 200 ºC for three hours using an eclectic furnace consists of granules dark region (pearlite) and other light region (ferrite).

![Microstructure of low carbon steel AISI (1011)](image)

Figure 5: The microstructure of low carbon steel AISI (1011) magnification (X400) shows two phases (ferrite and pearlite)

3.2. Results of XRD test

XRD analysis was carried out on the tested samples, which are untreated (as received) and treated samples (nickel coating, carburizing), to examine the phases that exist in the material before and after surface treatment. Figure 6 illustrates XRD pattern for all the cases.
Figure 6: XRD pattern of a) Pure AISI 1011 steel; b) Steel coated with Ni at 5 min; c) Steel coated with Ni at 10 min; d) Steel coated with Ni at 15 min; e) Steel carburized; f) Steel carburized and coated with Ni at 15 min

In Table (4), the angles of diffraction ($2\theta$) and the intensities (I) of the X-ray beam for all cases are illustrated.

| Cases                                     | $2\theta$ (deg) | I     |
|-------------------------------------------|-----------------|-------|
| Pure AISI 1011 steel                      | 44.11, 81.84, 116.29 | 100, 18, 7 |
| Steel coated with Ni at 5 min             | 44.26, 51.54, 92.59 | 100, 16, 7 |
| Steel coated with Ni at 10 min            | 44.40, 51.65, 92.69 | 100, 11, 4 |
| Steel coated with Ni at 15 min            | 43.83, 51.14, 98  | 100, 10, 4 |
| Steel carburized                          | 44.22, 41.60, 34.91 | 100, 83, 5 |
| Steel carburized and coated with Ni at 15 min | 44.86, 52.17, 16.75 | 100, 20, 15 |
3.3. **SEM test results**

The coating thickness and coating solution temperature between (55-60º C) is influenced by many factors like (Temperature, plating time and current density). From table (5) it can be seen that the plating thickness increases with increasing time and this is due to increasing the rate of deposition as current density increased.

| groups | Plating thickness (μm) | Time (min) |
|--------|------------------------|------------|
| B      | 9.73                   | 5          |
| C      | 14.8                   | 10         |
| D      | 20.42                  | 15         |

**Figure 7**: Relationship between values of plating thickness and time of plating

From Figures (8-A,B,C), it can be seen thickness for coating layers of nickel at three times (5,10 and 15) minutes.

![SEM image of steel AISI (1011) coating with nickel for 5 min](image)

A: specimen of steel AISI (1011) coating with nickel for 5 min
Figure (8-A,B,C). The microstructure of low carbon steel AISI (1011), shows the coating layers thickness

3.4. Microhardness results

Vickers hardness method was performed for specimens, and it has been found that the hardness values increased for all surface treated specimens. The hardness values for specimens treated with nickel plating for (5, 10, 15 min) were higher than those of untreated and this is due to the formation of nickel phosphide precipitates [10]. Furthermore, hardness of the treated specimens by carburizing process (at 950 °C for 5hr) have higher values than plating specimens by nickel and those of untreated this is due to the formation of carbides, however, carbon diffusion (diffusivity) during the carburizing process increased with time as the relation:

\[ \text{Case depth} = k\sqrt{t} \]

where K is constant and t is time. So as time increase the diffusivity of carbon increased [11]. The higher carburizing temperature and the higher soaking time will result a large carbide layer and the harder case becomes, while core still tough.
Table (6) The Vickers hardness values for samples of low carbon steel (AISI 1011).

| Groups | Treatment                  | Vickers Hardness (HV) |
|--------|----------------------------|-----------------------|
| A      | Without treatment          | 169.65                |
| B      | 5 min. coating with Ni     | 183.43                |
| C      | 10 min. coating with Ni    | 201                   |
| D      | 15 min. coating with Ni    | 219.7                 |
| E      | Carburizing 5 hour         | 390                   |
| F      | Carburizing 5 hour + 15 min coating with Ni | 431.65 |

Figure 9: values of microhardness for all groups

3.5. Surface roughness results

The surface roughness decreased for samples that treated with nickel coating, As plating time increased, thickness of nickel layer increased and it will be 20.42 μm within 15 minutes time of plating which leads to surface finer roughness. While, after the carburization process the surface roughness of samples increased. So the surface roughness of carburised specimens became (1.98 μm) at carburising temperature 950°C and time of 5 hours.

Table (7) Roughness measurements for low carbon steel AISI (1011).

| Groups | Specimens conditions | Roughness (μm) |
|--------|----------------------|----------------|
| A      | un treated           | 0.92           |
| B      | Coated with Ni at 5 min | 0.61          |
| C      | Coated with Ni at 10 min | 0.39          |
| D      | Coated with Ni at 15 min | 0.19          |
| E      | Carburizing for (5h, 950°C) | 1.98          |
| F      | Carburizing for (5h, 950°C) + 15 min nickel coated | 1.30          |
Figure 10: values of surface roughness for all groups

3.6. Tensile test results

It can be seen from figure (11) the stress-strain diagram for low carbon steel AISI 1011. This test was carried out for this alloy after has subjected to stress relief process. It can be remarked, that the ultimate tensile strength was (550) MPa and the total percentage elongation became 30 mm.

Figure 11: Stress-Strain diagram for low carbon steel AISI 1011.

3.7. Rotating Bending Fatigue Tests

S-N curves for all groups were obtained from the fatigue test as shown in figure (12). It can be seen that the fatigue strength for low carbon steel AISI (1011) increased for specimens subjected to carburising process as compared with the same metal which don’t treated by this process. This improvement of fatigue limit was due to the compressive residual stresses and carbides formation at the surface of steel which may be stopping and blocking the crack. From S-N curves for specimens plating with nickel by electrodeposition process at room temperature, it can be seen that the effects of thickness of coating with nickel after coating process on the fatigue strength and value of fatigue limit
Furthermore, the characteristics of deposited film itself were examined. As the electroless nickel plating is performed on the metal, their fatigue strength rather increases, as compared with uncoated metal. The fatigue limit for this alloy coated with Ni at 5 minutes became (82.89) MPa. While the fatigue limit for this alloy coated with Ni at 15 minutes became (93.21) MPa. And for untreated alloy was (79.75) MPa. So it can be seen that fatigue strength improvement against non-plated specimens was between (3.93-16.87) %.

The fatigue limit for carburised alloy at 950°C for five hours was (98.12) MPa, while the fatigue limit for carburised alloy at 950°C for five hours and plating with nickel for time of plating 15 minutes became (109.30) MPa.

It can be remarked that the electroless deposited nickel film shows a good adhesion to the substrate. The fatigue damage of plated steels prevented by deposited film. Consequently, the nucleation of fatigue crack is delayed, and there is an improvement in fatigue strength.

In electroplating process, the micro-hardness of deposited film increases, beside that there is an structural changes. This improvement is due to the precipitation hardening of Ni₃P. Furthermore, the tensile residual stress in deposited film is reduced. So the improvement in fatigue limit may be due to compressive residual stresses and largest ductility/toughness exist in the electroless nickel plating which delayed the crack propagation.

Figure 12: S-N curves for all groups

The surface fractures could be recognize by using scanning electron microscope TESCAN, VEGA 3 LMU device. Figure (13) shows surface fracture. Which occurred in the specimens after sufficient cycles of the bending load.
Figure (13): Fatigue fracture surfaces for AISI 1011 steel, A: fatigue fracture, B: crack initiation, C: core of the fracture.

It can be seen the fracture is brittle, beach marks are often formed when the load is alternating during service or when the loading is intermittent. Beach marks always explain a fatigue failure, but unfortunately if there is no beach marks does not rule out fatigue failure [12].

4. CONCLUSIONS
1. Low carbon steel AISI 1011 electroplating by nickel had higher fatigue strength due compressive residual stresses and largest ductility/toughness exist in the electroless nickel coating that delayed the crack propagation. The maximum fatigue limit became 93.21 Mpa as the alloy plating with nickel at 15 mintes and thickness of coating layer is 20.42 µm, but the best fatigue limit was 109.30 Mpa as the metal subjected to carburizing and plating with nickel at 15 mintes.
2. Carburizing process promote the fatigue strength and the value of fatigue limit was 98.12 MPa as the specimens treated with carburizing temperature is 950˚C and time 5 hours.

3. Nickel coating showed increasing in hardness of the metal (steel the AISI 1011) as plating time increased, the maximum hardness value was (219.7) Hv at plating time 15 minutes. Furthermore the hardness value became (431.65) Hv at plating time 15 minutes and curburising temperature 950˚C with time 5 hours.

4. The thickness of the coating increased with the increasing coating time. The maximum thickness of coating was 20.42 µm at planting time of 15 min.

5. There is an improvement in surface roughness of samples with plating process, the surface roughness of the metal was 0.19 µm at plating time 15 minutes.

5. References

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