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Microstructure and Mechanical Properties of the 55CrMoV4 Steel Exposed to Boriding and Nitriding Treatments

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ABSTRACT

In this study boriding and nitriding treatments were carried out on 55CrMoV4 low alloyed steel. The thermochemical treatments were carried out in solid medium by the powder technique at 900°C for 4 hours for boriding treatment and at a temperature of 550°C for 12 hours for nitriding treatment. The phases analysis of the boride and nitrite layers formed on the surface was carried out by optical microscopy (OM), and X-ray diffraction (XRD). The results of the surface analysis show that the boride and nitride layers a presence of FeB, Fe_2B, CrN, Fe_3N and Fe_4N compounds. The thickness of boride layers and nitride layers was found to be 55 and 12 µm, respectively. Microhardness of boride and nitride layers are between 800 $HV_{0.2}$ and 1200 $HV_{0.2}$. Corrosion tests by immersion in a 1M HCl solution have shown the beneficial effect of boriding and nitriding treatments on treated steels. Increase in corrosion resistances was observed after nitriding and boriding treatment steel 55CrMoV4 was around 6 times.

1. INTRODUCTION

Low alloy steel is widely used for various industries such as spare parts, gasoline chemical industry, accessory parts in chemical industry and many other sectors. Low alloy steels are generally is used in these applications due to its corrosion resistance, which it is the reason why it is important to study it to better optimization use it. Surface modification by thermochemical affects material properties such as wear, fatigue, corrosion resistance. Thermochemical treatments (carburizing, nitriding, boriding...) are widely used for increase the surface hardness, the frictional wear, and the corrosion resistance. The diffusion of nitrogen, carbon and boron atoms modify the chemical composition of the surface leads to the formation of nitride, carbide and boride layers which are for their high hardness. The Boriding is a thermochemical surface hardening process in which diffused boron atoms, due to their small diameter, into the surface of steels to form boride layers. Boride layers may consist of either single (Fe_2B) layer [1] or (FeB + Fe_2B) double layer [2, 3]. The boriding treatment is carried out by the temperature range 900 K –1323 K for 1 h to 12 h, which can be applied to practically any ferrous material, as well as some nonferrous materials [4-6] and it can be carried out in solid medium in powders, liquid and gaseous medium [7]. It is used to improve the surface hardness, oxidation resistance and wear [8]. Nitriding is a thermochemical surface treatment process generally used s in which diffuses the nitrogen into the surface of a metal. The nitriding treatment can be carried out at temperature range between 500°C and 600°C for duration varying from a few hours to a few days. As regards the microstructure obtained after nitriding treatment, there are two different types: compound layer and diffusion zone. The composite layer formed on the surface consists mainly of different phases such as Fe_4N, CrN and Fe_3N [9], and its thickness of the nitride layer can vary from 10 to 50 µm. This layer obtained is responsible for improving tribological properties and corrosion resistance [10-12].

In this work, we study the effect of boriding and nitriding treatments on the Microstructure, the microhardness and corrosion resistance of treated steel 55CrMoV4 using optical microscopy (OM), microhardness tests and XRD. The corrosion resistance was evaluated of the layers developed obtained by boriding and nitriding under optimal conditions was monitored in a 1 M HCL of solution and data were obtained by electrochemical linear polarization techniques. The corrosion resistance of borided and nitrided samples was compared with untreated samples in the steel.

2. EXPERIMENTAL PROCEDURES

2.1 Material and thermochemical treatments

The test specimens were cut into cylinders with dimensions of 20mm Ø × 10mm. The chemical composition of 55CrMoV4 low alloy steel used for Boriding and Nitriding treatments, determined by spectrometric analysis is given in Table 1.

Table 1. Chemical composition of the steel 55CrMov4 to be coated (%)

Elements	C	Mn	Si	Cr	P	Mo	V
Mass %	0.55	0.81	0.274	1.00	0.015	0.454	0.035

Before boriding and nitriding treatments, all specimens are prepared by polishing the surface in sequence with (400, 600, 1200) grit wet SiC emery paper to eliminate any contamination and facilitate the diffusion process.

- The boriding treatment was realized in a mixture powder consisting of the following composition: 90% SiC as diluent, 5% B_4C as boron source, and 5% $NaBF_4$ as activator, at 900°C for 4 hours.
- The nitriding treatment was realized in a mixture powder rich in nitrogen, carbon, and boron at a temperature of 550°C for 12 hours.

After the treatments, the boride and the nitride steel parts were removed from the furnace then cooled in air air up to room temperature (25°C).

2.2 Characterization of boriding and nitriding

The microstructural characterization was carried out on cross sections of the samples were observed under an LEICA DMLM optical microscope (OM). X-ray diffraction (XRD) using Cu Ka, was applied for the identification of nitrides and borides phases formed in the coating layers. The surface hardness of the borided and nitrided layers obtained on steel 55CrMoV4, was measured at 5 different locations the average value was taken as the hardness at the same distance from the surface to the substrate. The five locations must be at the same depth of the surface and sufficiently spaced to avoid the influence of the imprints on each other and to allow. The first value of microhardness is measured on the surface of the formed layer. Then measurements are taken at a distance of 10 µm to a depth of 200 µm. For the rest, the measurements are taken at distances of 50 µm. The used durometer is a Mitutoyo MVK- with Vickers indenter and a load of 200 gf.

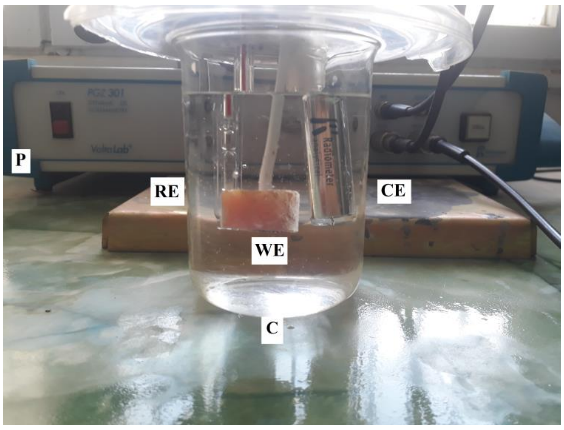

Figure 1. Mounting of the polarization test cell. WE: working electrode, RE: reference electrode, CE: counter electrode, C: electrochemical cell, P: potentiostat

2.3 Electrochemical corrosion

For corrosion testing, the electrochemical corrosion behavior of borided, nitrided and untreated 55CrMoV4 steel immersed in (1 M) HCl solution was studied with open circuit potential as a function of time, and Polarization resistance measurement. All tests were performed at room temperature at 25°C on a Radiometer model PGZ 301 potentiostat controlled by a PC and supported by Voltamaster 4.0 software for the experimental control for data acquisition (Figure 1). experiments set up consisted of an electrochemical cell comprising three electrodes. A working electrode and a saturated calomel reference electrode were used as reference electrode and counter-electrode. For the open circuit potential samples were immersed in an electrolyte solution for approximately 30 min to stabilize the open circuit potential in room temperature. The corrosion test was performed at a scanning rate of 1 mV/s.

3. RESULTS AND DISCUSSIONS

3.1 Characterization of nitride and boride layers

Figures 2 show cross-sections of the structures of the nitride layers obtained of steel 55CrMoV4 after nitriding treatment at 550°C during 12 hours. The obtained structural regions reveal a typical form of nitride: the first observed layer is the white layer or the composite layer and consists of iron nitride on the surface. they are mainly composed of iron nitrides ($Fe_{2-3}N$) and γ 'nitrides (Fe_4N) [13, 14]. This layer, estimated to be around 12 µm thick, is more or less homogeneous with uniform contrast. The second layer obtained by the nitriding bellow the compound layer is named diffusion layer. The cross-sections of the optical micrographs of the borided steel at 900°C for 4 hours is given in Figure 3. It shows the presence of two FeB and Fe_2B layers with a sawtooth morphology [15, 16]. The thickness of the boride layer on the surface is about 55 µm.

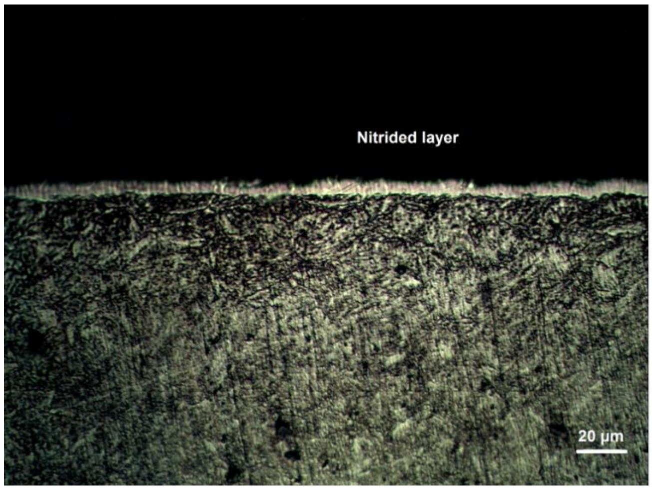

Figure 2. Microstructure after nitriding in (550°C, 12 hours): steel 55CrMoV4

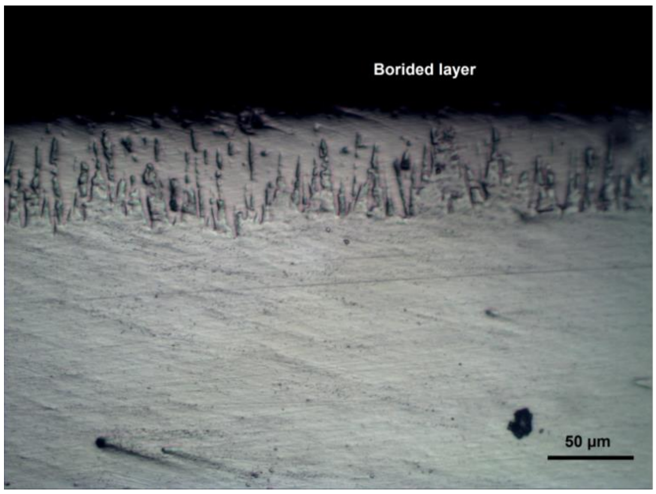

Figure 3. Microstructure after boriding in (900°C, 4 hours)0: Steel 55CrMoV4

3.2 XRD analysis

Figure 4 shows the X-ray diffraction spectra of steel 55CrMoV4, nitrided at 550°C for 12 hours. The obtained spectra indicate the existence of representative peaks of iron nitrides Fe_3N, Fe_4N and chromium nitride CrN. Despite the presence of the Fe_4N phase which is not desired in tribological applications, the layer formed is practically homogeneous and consistent across the entire treated surface, which in our opinion does not affect the improvement in resistance to corrosion. The two phases of nitrides Fe_3N, Fe_4N have also been found by Kulka et al. [17] and Panfil et al. [18].

The X-ray diffraction spectra of steel 55CrMoV4, borided at 900°C for 4 hours are shown in Figure 5. According to Figure 5, the boride layer obtained on the surface of the steel 55CrMoV4 mainly consists of the two phases of Fe_2B, FeB borides. Depending on the powder used (5% B_4C, 5% $NaBF_4$ and 90% SiC), and which is considered a low activity powder, a boride layer consisting of the single phase Fe_2B only was expected to be formed. The presence of FeB boride in addition to Fe_2B boride is justified by the high carbon content of the treated steel and the presence of chromium, which according to the literature [19, 20] activates the diffusion of boron atoms.

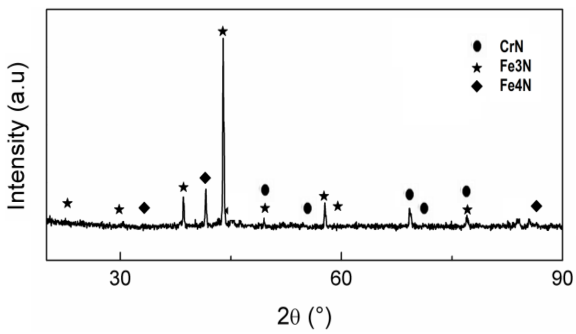

Figure 4. X-ray diffraction patterns of nitrided steel: 55CrMoV4

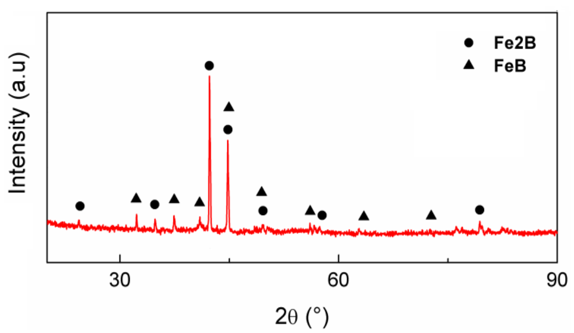

Figure 5. X-ray diffraction patterns of borided steel: 55CrMoV4

3.3 Microhardness profile

Figure 6 shows the micro-hardness profile of specimens of steel 55CrMoV4 with different treatements borided 900°C for 4 hours and nitrided at 550°C for 12 hours. it is observed that boriding treatment steel 55CrMoV4 obtained high values of microhardness of the surface then gradually decrease of towards the substrate. Microhardness of specimen borided is around 1200 $HV_{0.2}$, in the boride layer zone, while for the substrat, the hardness is around 300 $HV_{0.2}$. Diffusion of boron on the surface to form phases such as FeB and Fe_2B results in the increase in hardness. A high value of microhardness of the nitrided of around 800 $HV_{0.2}$, was obtained at the surface of steel, which is due to the forming of the layers Fe_3N, Fe_4N and CrN. The curve shows a comparison of hardness for both treatments that the sample treated by boriding has a higher hardness than nitriding treatment, irrespective of the type of steel treated.

3.4 Corrosion test results

Figure 7 shows the polarization curves of samples nitrided and borided as well as untreated sample 55CrMoV4 steel immersed in 1 M HCl. The forme of the three polarization curves is essentially very similar with an offset in the axes E and I. The corrosion potential values, I_{corr}, E_{corr} and Corrosion Rate were obtained from on the analysis of polarization curves and are shown in Table 2. The higher value of corrosion potential (E_{corr}) is observed for borided layer compared with nitrided layer. The more corrosion potential is on the positive side; the more the steel is considered corrosion resistant. According to these curves, we can easily see that the curves of borided samples correspond to the lowest current densities, followed by nitrided curves and finally those of the untreated samples. Corrosion measurements showed that the boriding and nitriding treatments playing an important role for the corrosion properties of the steel 55CrMoV4. borided and nitrided Samples show good resistance against corrosion. This may be due to presence of different phases (Fe_2B, FeB) (CrN, Fe_4N and Fe_3N) in the surface of the samples by the boriding and nitriding treatments. This allows us to say that boriding and nitriding treatments improve the corrosion resistance of studied steels, with a greater effect of boriding.

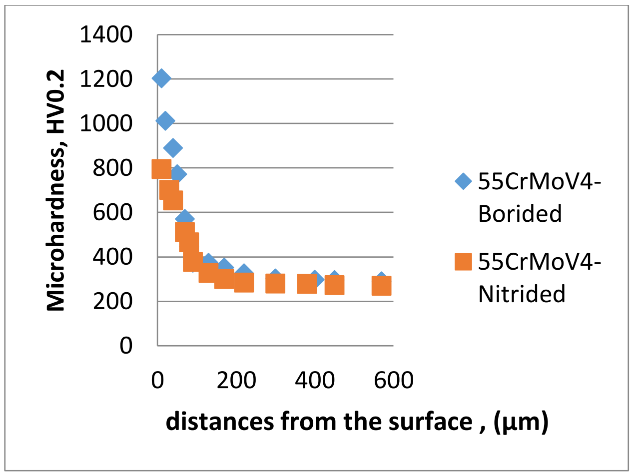

Figure 6. The variation of hardness depth in the borided and nitrided steel: 55CrMoV4

Table 2. Corrosion data as determined by Tafel extrapolation method for untreated, nitrided and borided, of three steels in HCl solutions

	Borided	Nitrided	Untreaded
I_{corr} (μA/cm^2)	8,627	70,73	242.4
corrosion rate (mm/Y)	0,10	0,827	2,835
E(I=0) (mV)	-440.5	-457.4	-482,7

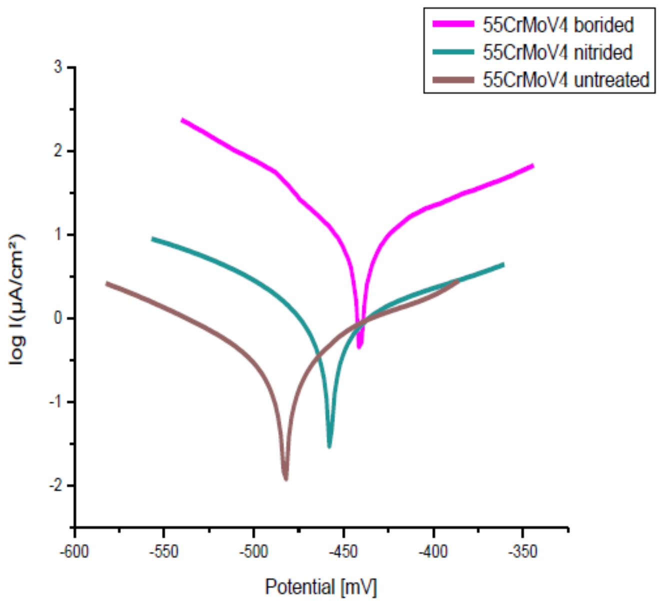

Figure 7. The Tafel curves for the untreated, borided, and nitrided on the steel in HCl solution

4. CONCLUSIONS

Finally, the following conclusions and remarks can be presented:

- Thermochemical Boriding and Nitriding treatments on treated steel lead to the formation of boride (Fe_2B and FeB) of and nitride (Fe_3N, Fe_4N and CrN) layers.
- The Microhardness tests indicate that boriding and nitriding treatments lead to an increase of the surface hardness compared with that of the matrix.
- The hardness surface of the borided 55CrMoV4 steel was around 1200 $HV_{0.2}$, while for the nitrided 55CrMoV4 steel is around 800 $HV_{0.2}$. On the other hand, hardness values the untreated steel were around 300 $HV_{0.2}$.
- The thickness on the boride layer was around 55µm and nitride layer was around 12 µm of the steel 55CrMoV4.
- The borided steel revealed higher corrosion resistance than the nitrided and untreated steel. Corrosion tests shown a beneficial effect of boriding and nitriding treatments on treated steel.

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