IRAQI JOURNAL OF APPLIED PHYSICS
Vol. 22, No. 3, July-September 2026, pp. 386-392

Effect of Dielectric Barrier Discharge Plasma on Surface Properties for Medium Carbon Steel

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This study investigates the effect of dielectric barrier discharge (DBD) plasma treatment on the surface modification of medium carbon steel (CK45). The process was carried out in ambient air at a voltage of 15 kV and frequency of 20 kHz for exposure times of 0–12 min. The influence of plasma duration on microstructure, surface hardness, wear, and corrosion resistance was systematically analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and electrochemical Tafel polarization. Results revealed the formation of a dense nitride–oxide layer mainly composed of Fe_4N and Fe_3N with minor Fe–O–N phases. The Vickers hardness increased from 240 Hv (untreated) to 330 Hv after 12 min of treatment, representing a 37.5 % improvement. The corrosion current density decreased from 2.4×10^{-3} A/cm^2 to 5.3×10^{-5} A/cm^2, while wear rate was significantly reduced. These enhancements are attributed to grain refinement, lattice strain, and the formation of a compact nitride barrier layer that suppresses anodic dissolution and surface wear. The results demonstrate that air-based DBD plasma is an eco-friendly, low-temperature, and energy-efficient alternative to conventional nitriding processes for improving the tribo-corrosion performance of medium carbon steels.

Keywords: Dielectric barrier discharge; Plasma; Carbon steel; Surface modification
Received: 1 September 2025; Revised: 4 December; Accepted: 11 December; Published: 1 July 2026

1. Introduction

Plasma is a quasi-neutral gas of charged and neutral particles that exhibits collective behavior, which incorporates charged particles (electrons, ions, and molecules) [1]. The plasma is classified according to its temperature to two main types: high temperature plasma or nuclear fusion plasma and low temperature plasma or so-called plasma discharge [2]. Low temperature plasma is divided into two major groups: thermal and non-thermal plasma [3].

Plasma-surface treatment is a particular and highly sophisticated methodology instead of a compound to control the surface features of materials, but without significantly affecting their bulk properties. The alteration in the microstructure and properties of the substrate can be induced by causing the reactive gases usually nitrogen, oxygen, or carbon to become chemically a part of the material's surface by placing them in a plasma environment. This method has been proven especially successful in increasing the hardness, wear resistance, and corrosion resistance of steels and other metals. Due to the high degree of control over the process based on the time-related parameters and the treatment temperature, it provides customizable surface changes, which will improve a material's performance in the name of the harsh industrial environment. The use of plasma-surface treatment, therefore, prevails in industries where high durability and resistance to wear and corrosion are essential as indicated by previous researches.

Bolotov et al. [4] conducted an experimental study on the influence of plasma treatment on the hardness of AISI 52100 and AISI 1020 steels. The research indicates that plasma treatment, utilizing gas combinations of nitrogen and hydrogen, significantly enhances surface hardness, evidenced by a hardness value of 585 HV under optimal circumstances and the effect of processing parameters, especially temperature and gas composition, on steel microstructure. The empirical data show that the surface layers enriched with nitrogen are needed to improve the mechanical characteristics of steel. These observations show that there is a possibility of local surface hardening, hence making plasma treatment particularly relevant in case of industrial applications requiring high hardness and corrosion resistance.

Subair at el. [5] investigate the impacts of the process of plasma treatment on the hardness of AISI 52100 and AISI 1020 steels. As they point out in their research, hardness after the treatment is done subsequently by the use of plasma treatment (of the nitrogen-hydrogen and argon-nitrogen gas collectors) considerably increases with a hardness gain in the surface area of up to 585 HV upon the initial value of 262 HV. The findings also underline that the hardness improvements in surface areas are also associated with localized differences in the surface that depend on the process parameters, which in this case were the composition of the gas mixtures.

Li at el. [6] investigated the wear properties and corrosion performance of AISI 420 martensitic stainless steel after active screen plasma treatment (ASPT), treatment of which was chosen as a convenient and straightforward method of increasing the corrosion resistance of this steel. The results indicated that ASPN had better hardness, wear resistance, and improved corrosion behavior; the best performance was when it was done at 480 °C. The nitride layers contained nitrogen-enriched phases and chromium nitrides, and improved wear and corrosion properties. But at a top plasma treatment temperature of 520 °C, the corrosion resistance score declined because of CrN precipitation and microcracking.

P. Ravi Kumar1 et al. [7] studied used AISI 304 stainless steel is a type of austenitic stainless steel that contains a high percentage of chromium and nickel. Plasma nitriding is carried out on AISI 304 at low temperatures 5500 °C for the time duration of 8 hours, 16 hours and 32 hours. It was noted that the sample treated to 32 hours. It has been improved the wear resistance when compared to the other treated samples. It is because of the hard layer comprising with chromium nitride and iron nitride formed on surface level. The phase transformations from austenite to expanded austenite were obtained.

There have been many studies on material modification using different types of plasma. The main objective of this work is to study the effect of DBD plasma on microhardness, macrostructure, wear and corrosion resistance of steel.

2. Experimental Part

The study utilized medium carbon steel cylinder samples with 16 mm diameter and 4 mm thickness. The material investigated in this study was steel CK45 with a chemical composition given in table (1). The chemical composition was measured using Spectro Max ASTME415-14.

Samples were prepared by grinding them with SiC emery papers starting from 240 rough grit, then 320, 400, 600, 800, 1000, and finally 1200 grit size, followed by 0.25 µm grain size alumina paste polishing. After polishing, the prepared samples were rinsed with acetone, washed, and dried.

After the samples' surface morphology prepared and deeply cleaned with acetone, the DBD (High Voltage Dielectric Barrier Discharge) system is used to generate cold plasma [8]. The capability of cold plasma consists of two electrodes made from stainless steel covered by Teflon and the quartz barrier between the electrodes. The system was energized using a high voltage AC power supply, the magnitude of applied voltage about 15 kV, frequency 30 kHz, current 112 mA, electrode-sample gap 10 mm, atmospheric conditions under normal atmospheric pressure and room temperature, it consisted mainly of high-voltage electrodes, the cold plasma was obtained by ionizing the ambient air and applied on the sample for different times of exposure 0, 4, 8, and 12 min. the DBD device used in this study illustrated in Fig. (1).

In the experimental tests, microhardness of the top surface and the zone of the plasma surface were measured by a Vickers microhardness tester with a 5.9N applied load, time of 15 s, cylindrical samples. After conducting this test, the best hardness value is choose and other tests are conducted on it. The microstructure of the treatment zone was characterized by scanning electron microscope (SEM). The phases formed due to plasma surface treatment were analyzed by X-ray diffractometer (XRD). The corrosion test sample were immersed in seawater (3.5 wt.% NaCl solution) at room temperature for an electrochemical corrosion test. Tafel polarization experiments were conducted using the CHI 604e and a standard electrochemical cell. The wear test (pin-on-disc test) speed of sliding 200 cycles/min, and disc 1100 HV hardness rating [9]. Each test lasted 20 s to 140 s and was conducted at room temperature and in normal weather conditions. A timer is programmed to function at 20s intervals. The device is activated, and the time is logged. Temporarily pause the gadget every 20s. Extract the sample and eliminate any corrosion residue. Precisely ascertain and document the weight. Reinstall the sample and recommence operation.

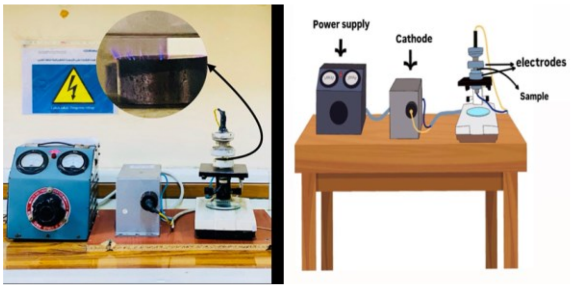


Fig. (1) DBD System

3. Results and discussion

Figure (2) provides the graph of the microhardness of CK45 steel against the surface plasma process time, whereby the microhardness shows an increment with respect to the surface plasma process time. The untreated sample had a value of 240, and the hardness value began to increase with increasing exposure time, reaching 330 after 12 minutes or the early nitrogen diffusion at very initial stages of surface plasma process, the nitrogen ions commence their diffusion into the steel surface. As it happens at this stage, the diffusion rate is relatively low and very small amount of nitrogen is presented in the steels. Causing formation of thin nitride layer during the insertion of the nitrogen atoms into the surface, the atoms start reacting with the iron and thereby form very minute iron nitrides (Fe_4N, Fe_3N, etc.). Such nitrides premature hardening of the surface. This formation of these phases raises the hardness on the surface significantly though the nitride layer is still thin. During this stage, there is minimal increment in regard to hardness because now the material is beginning to be hard due to the harder phases found [10]. However, since the surface plasma process layer is small compared to the surface plasma process layer and the nitrogen diffusion is in an early phase, the hardness increase is not dramatic as yet. A thermocouple positioned near the specimen indicated that the surface temperature remained below 180 °C during the 12-min DBD exposure. Although this is much lower than conventional nitriding temperatures, several plasma-specific mechanisms accelerate nitrogen incorporation energetic N_2^+ ions and metastable NO^+ species enhance adsorption and dissociation on the surface; ion bombardment and micro-arcing locally elevate the temperature within the top few microns, enabling short-range diffusion; and plasma-induced defects and lattice distortions increase diffusivity at low bulk temperatures.

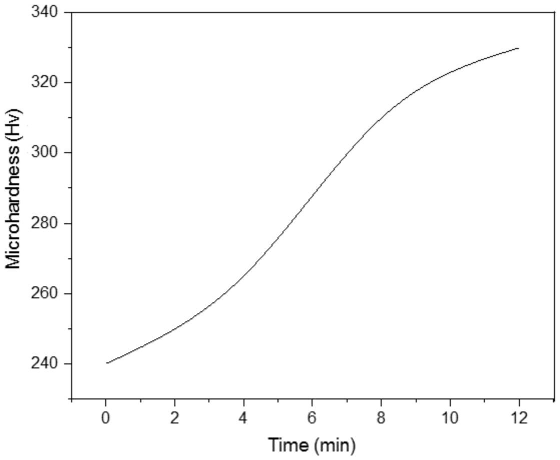


Fig. (2) The relation between microhardness of carbon steel and surface plasma process time

With the duration of surface plasma process, nitrogen ions enter more deeply into the surface, thus producing a thicker nitride layer. This is the decisive stage in which the nitrides develop further. The fraction of the volume of iron nitrides (Fe_4N and Fe_3N) rises. The process is performed at this stage as nitrogen atoms transform the steel matrix to result in a grain refining effect. An increase in surface hardness of the steel is achieved through fine nitrided grains. Also, a phase transformation of hardness of the material is formed, where the softer phases, such as ferrite, pearlite (exist in the untreated steel) get converted to the harder nitrogen-enriched ones. Such newly created nitrides are harder and add to whole body hardness. Due to the creation of the stable nitrided layer, hardness of the material is significantly increased. This nitrided layer is a mixture of Fe_4N and Fe_3N, which are highly hard and their wear resistance is more than that of original ferrite and pearlite structure of the original steel. The uniform nitride layer provides the best hardness of this range and makes the surface more resistant to wear and to fatigue [11].

Under the magnification of 4000 times, the SEM micrograph in Fig. (3a) shows the untreated CK45 steel, which is a relatively unsmooth and uniform. This particular steel, in its original state, was marked by irregularities in roughness and surface microscopic roughness, as some individuals may have seen. The fact that the material is in a rough form and has an ordinary microstructure, which may contain ferrite and pearlite in its creation, is what gives rise to these features. On non-protected surface, there is a possibility that scratches, microcracks, and gouges, dislocations, and/or strains may be present, some cases, the micro voids and roughness of the surface may allow for the oxidation or corrosion of the metal that lies beneath. It is also possible that this will result in rust or holes brought about by corrosion, both of which have the potential to mutilate the material to the point where it may weaken the substance's mechanical strength. Medium carbon steel, and will mostly be composed of ferrite (light area), which is a soft and ductile material, and pearlite (dark areas), which is a harder and more brittle material [12,13]. The exposed and unprocessed character of this steel, along with the fact that these grains have not been treated, causes it to rust rapidly.

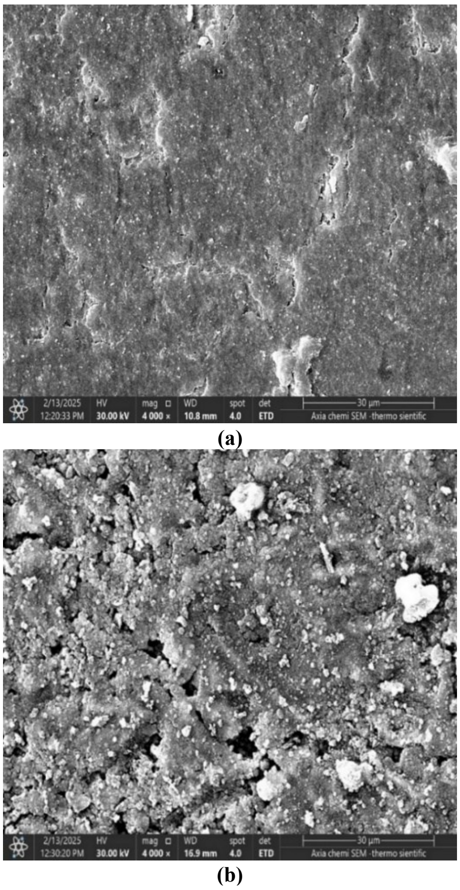


Fig. (3) CK45 SEM morphology (a) Untreated, (b) After 12 minutes plasma surface treatment process at 4000 magnifications

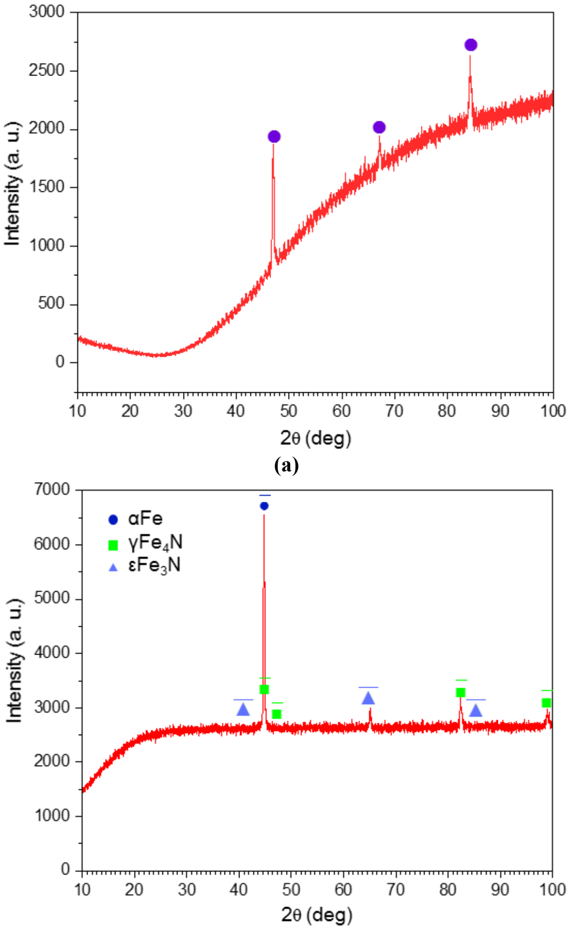
Figure (3b) demonstrates the result of CK45 steel plasma surface treatment over 12 min time period, the surface shape undergoes a significant transformation in the scanning electron micrograph. It is now possible to see individual nodules on the surface, which is rather rough in comparison to the state in which it was not treated [14]. Its roughness was confirmed by surface roughness testing, which reached 1.5 microns, compared to the untreated material whose surface roughness was 0.1 microns. The process involves the incorporation of nitrogen from the surrounding environments into steel, which leads to the formation of nitrided phases, such as iron nitrides depending on the length of time that the process is carried out and other circumstances. These morphological changes are the outcome of the plasma treatment process. During the process, the existence of nitride crystals and particles in this image is most likely the result of nitrogen onto the surface of the steel.

The impact of DBD plasma on the roughness and texture of steel is evident and contributes to the enhancement of its mechanical characteristics. The results, as mentioned above, demonstrate the importance of plasma as a surface treatment for enhancing steel performance in the industrial sector.

The provided XRD pattern in Fig. (4a) displays a diffraction pattern of CK45 steel in As-received conditions. In XRD, the peaks correspond to the positions where X-rays are diffracted by the crystal planes inside the material [15]. The X-axis represents the 2θ angle (diffraction angle), indicating the spacing between layers of the crystal lattice, while the Y-axis corresponds to the count or intensity of diffracted X-rays, reflecting the degree of X-ray radiation scattered by the crystal at a specific angle. The presence of pronounced and high-intensity peaks in the graph indicates well-defined crystalline structures. These peaks are typically associated with certain crystal planes, as observed in iron (Fe) and its many phases. The background signal comprises flaws in the sample, amorphous substances, or noise. The crystallization manifests in a cubic system, indicating a certain sort of symmetry within the cubic crystal system. The presence of specific diffraction peaks facilitates the confirmation of phase composition and ensures that the peaks correspond to the properties of ferrite, martensite, or other phases often found in carbon steels.

Figure (4b) has a distinct diffraction pattern, indicating its crystalline nature. These pronounced peaks signify prominent reflections of the crystal planes in the steel at specific 2θ points, such as 45.5°, 66.3°, and 84.1°. The two peaks correspond to the crystal lattice of iron (Fe), and the associated d-spacing validate the robust structure characteristic of carbon steel. The surface layer is usually subjected to the formation of tetra iron nitride (Fe_4N) that produces additional peaks in XRD. Phase transition occurs in steel and in the case of surface plasma process, becomes long, some extent of expanded austenite (gamma-N phase) can form thus modifying the pattern of diffusion. We see that the intensity of the peak was increased with after treatment, where the intensity increased. This indicated that the treatment makes the carbon steel much more crystalline.

Enlarged austenite some carbon steels especially those that contain high carbon levels, or some alloying elements can promote the formation of expanded austenite in the process of surface plasma process. These processes are of importance to the wear resistance and hardness of the material and as also to increase the surface corrosion resistance [16,17].



(a)

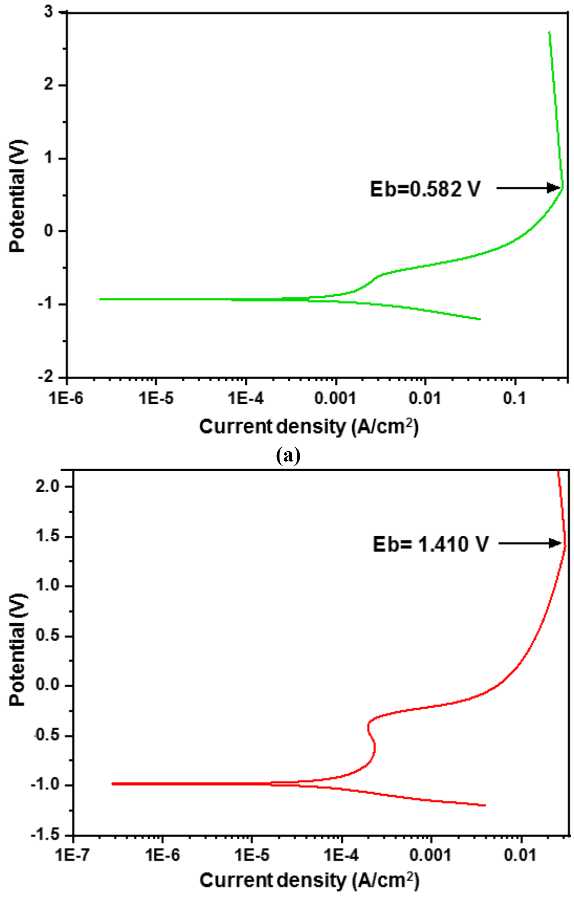
(b)

Fig. (4) XDR results (a) Untreated, (b) After 12 minutes surface plasma process

The Tafel polarization test curve shows obvious improvement in corrosion current reduction and consequently reduction in corrosion rate for untreated and DBD treated samples as shown in Fig. (5a) and table (2), corrosion behavior was assessed using the tafel extrapolation method supported by open circuit potential (OPC) [18]. The corrosion rate reduced from 28.37 mmpy for untreated sample to 1.612 mmpy for sample treated at 12 min., i.e., the corrosion rate reduced 17 times. The comparison of the two samples shows that surface plasmas treatment is essential to enhance the electrochemical stability in steel and resistance to corrosion [17]. Also, we obviously see the elevation in breakdown potential (E_b) where it reaches

1.433 V compared with 0.582 V for untreated sample, i.e., the passive layer formed by treated with DBD be harder to brake or betting than before treating.

The untreated CK45 steel surfaces used were smooth, yet rough, and contribute critically to process of corrosion. The inherent microstructure of CK45 ferrite and pearlite (composite of softer and harder phases) does not offer any protective capability against corrosion. Microcracks, scratches, and strain-induced marks (as a result of previous machining) are surface defects that can trap stress and thereby start localized corrosion (e.g., pitting or crevice corrosion) locally. These surface flaws offer easy accessibility to corrosive ions to penetrate the steel underneath thereby increasing the speed at which corrosion occurs [19].



(a)

(b)

Fig. (5) Tafel polarization test results (a) CK45 control, (d) CK45 after 12 minutes plasma DBD process

When the plasma treatment process occurs in 12 min, the potential of corrosion shifts in a more positive direction, as given in Fig. (5b). This is also among the key signs of superior resistance against corrosion. These E_{corr} values vary because the surface of the steel has become less reactive and more stabilized in the electrolyte. The (nitrided layer), which contains a considerable amount of iron nitrides (Fe_4N and Fe_3N), is regarded as a barrier to corrosion resistance. It is also not affected by the electrochemical reactions, which cause the material loss (oxidation) and passage of the electrolyte through the material. There is significant reduction in the phase corrosion (i_{corr}). The simultaneous decrease in i_{corr} will foretell direct decrease in the rate of corrosion.

The fact that the corrosion potential switched to positive (on the treatment sample) is sure evidence that the metal becomes more resistant to the electrochemical process when use plasma DBD, and the sample will resist corrosion. The lower corrosion current density of the nitrided sample proves that indeed the corrosion rate has been extremely slowed down by the effects of plasma a surface, further substantiating the fact that a plasma treatment process forms a hard, corrosion resistant surface. The OCP value of the untreated base metal was -0.509 V, while the value for the sample treated for 12 minutes shifted to -0.472 V. This shift toward less negative values indicates an improvement in the thermodynamic stability of the sample surface and the formation of a more protective passive layer before polarization occurs. It is well-known that the Tafel method may have limited accuracy in systems with strong passivation behavior due to narrow linear regions; however, combining stable OCP values with clearly defined linear regions in the Tafel curves allowed a reliable estimation of corrosion behavior trends. As for the corrosion potential (E_{corr}) of the sample treated for 12 minutes (-0.977 V), which was slightly more negative than that of the base metal (-0.927 V), this does not necessarily indicate a deterioration in corrosion resistance. This change can be attributed to differences in the surface electrochemical kinetics or in the composition of the surface layer resulting from the treatment. The formation of a stable and coherent passive layer contributes to a significant reduction in corrosion current density (I_{corr}), even if the potential becomes slightly more negative. Accordingly, the 17-fold decrease in the corrosion rate combined with the positive shift in OCP clearly confirms that the surface treatment effectively improved the material's corrosion resistance.

Table (3) XRD result for untreated sample

No.	h	k	l	d [A°]	2θ [°]	I [%]
1	0	1	1	1.99121	45.517	100.0
2	0	2	0	1.40800	66.335	13.6
3	1	2	1	1.14963	84.141	24.5

Improvement in the corrosion resistance of carbon steel treated with cold atmospheric air plasma can be explained by the formation of a compact and adherent layer of oxides and oxynitrides (Fe–O–N) on the metal surface. During plasma exposure in air, active species such as ions (N_2^+, O^+) and reactive radicals (NO, O, N) interact with the surface, leading to the incorporation of both oxygen and nitrogen into the steel's surface layer.

This results in the formation of a thin oxide-nitride layer that acts as an effective barrier against the penetration of chloride ions. This layer serves as a physical barrier, which reduces defect density and alters its semiconducting properties. These changes slow down the charge transfer kinetics at the metal/electrolyte interface, enhancing passivity and improving corrosion resistance. Although a positive shift in the corrosion potential (E_{corr}) indicates a general decrease in corrosion tendency, the stability of the passive layer is more accurately expressed by the breakdown potential (E_b). Measurement results showed an increase in the E_b value for the plasma-treated sample, indicating the formation of a more stable layer capable of resisting localized breakdown under anodic polarization. Thus, the improvement in corrosion resistance can be attributed to the synergistic effects of surface modification caused by the plasma, enhanced cohesion of the oxide/nitride layer, and changes in metallic behavior.

The graph shown in Fig. (6) illustrates the comparison of weight loss (in grams) over a timeline showing two CK45 specimens: the former refers to the untreated (as-received) specimen, and the latter specimen to the specimen that has undergone DBD plasma treatment. The results also show that the wear values have significantly improved after DBD treatment, assuring the usefulness of DBD as a form of surface modification.

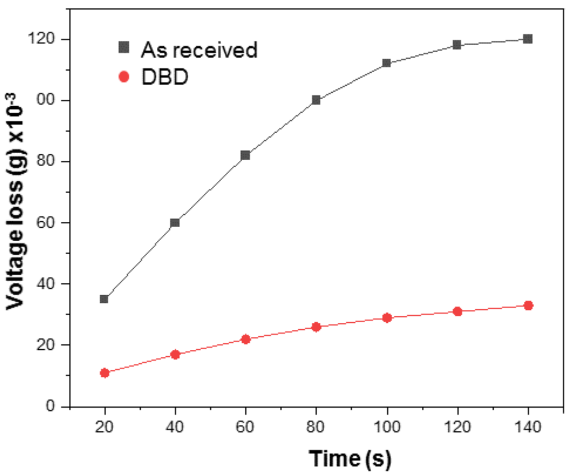


Fig. (6) Weight loss with time for CK45 control and CK45 after 12 minutes plasma DBD process

Weight loss increased with time for both samples, with the loss being less and slower for the plasma-treated sample. This constant higher performance can be explained by the fact that these phases are stable and at a harder phase (nitrided) than is the microstructure base (ferrite and pearlite). In the later phase the weight loss begins increasing, but the rate of increase is low compared with the rate in the untreated sample. This slower wear rate shows that the CK45 DBD-treated steel has the ability to retain its wear-resistant property even in an extended period of wear. The wear rates of the untreated sample was 0.85×10^{-3} g/m and the treated sample was 0.23×10^{-3} g/m. The lower the wear rate, the higher the material's wear resistance. The treated sample exhibited better wear resistance because it lost less material per unit time, resulting in greater hardness and reduced particle separation during friction. Therefore, the wear rate decreased significantly, approximately 73%. Microhardness also helps determine the wear resistance and durability of the material, which is crucial in industrial applications [20]. The significant improvement in corrosion resistance is primarily attributed to the formation of hardened phases, such as iron nitrates (ε-$Fe_{2-3}N$, γ'-Fe_4N), resulting from plasma treatment. These phases increase surface hardness, reduce plastic deformation, and act as barriers to crystal slip movement, thus mitigating the sticky and abrasive corrosion common in soft carbon steels.

However, the transition to a harder surface layer modifies the prevailing corrosion mechanisms. While untreated steel exhibits sticky and abrasive corrosion characterized by material transfer, pitting, and microcutting, the hardened nitride layer may be susceptible to brittle fracture, cracking, or delamination, especially under high loads or repeated friction. This balance between increased hardness and potential brittleness is a critical factor to consider, as it reduces conventional corrosion but may increase the likelihood of crack initiation and layer delamination.

In short, the improvement in corrosion resistance reflects both an increase in nitride layer stiffness and a change in the corrosion response, underscoring the importance of improving layer thickness, phase distribution, and adhesion to the substrate to achieve maximum performance.

4. Conclusion

In the study, it was shown that high-voltage dielectric barrier discharge (DBD) plasma treatment of promotes the corrosion resistance of medium carbon steel CK45 remarkably. This duration resulted in the formation of a smooth nitrided layer, which was composed of Fe_4N and Fe_3N and which offered the best hardness and wear resistance. The microhardness has increased and the corrosion current density values were very low which was a very large improvement considering that the corrosion rate was much reduced. Surface morphology indicated the generation of nitrided phases like Fe_4N and Fe_3N, which is behind the improvement of hardness, resistance to wear and corrosion. Surface roughness following plasma treatment were tremendous to demonstrate potential for applications in challenging industrial environments.

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Table (1) Chemical composition of medium carbon steel ck45

C%	Si%	Mn%	P%	S%	Cr%	Mo%	Ni%	Al%	Cu%	Fe%
0.425	0.221	0.723	0.014	0.011	0.024	<002	0.047	0.006	0.058	Bal

Table (2) Corrosion characteristics extract from tafel curve for untreated DBD treated samples

ITEM	$E_{corr.}$ (V)	$I_{corr.}$ (A/cm^2)	Corr. Rate (mmpy)	Bc (V/decade)	Ba (V/decade)	Eb (V)
Control	-0.927	2.447 × 10^{-3}	28.37	0.130	0.417	0.582
12min.	-0.977	5.355 × 10^{-5}	1.612	0.126	0.197	1.410