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STUDY OF THE MECHANICAL AND METALLOGRAPHIC PROPERTIES OF CARBON - ALUMINA COATED IRON PRODUCED BY IMPULSE PLASMA DEPOSITION

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ABSTRACT

Carbon - Alumina films were deposited on the surface of iron substrate at room temperature using impulse plasma deposition (IPD) method. The plasma beam was generated from a coaxial plasma gun. The pulsed plasma mainly consists of two kinds of ions: working gas ions and inner electrode ions. Acetylene gas (C_2H_2) was used as a source of carbon atoms. While the inner electrode was the source of the Aluminum present in the deposited Alumina phase. Coatings were produced on iron substrates by a number of plasma pulses (50 - 200) from 15.42 μF capacitor bank at 13.5 kV discharging voltage. The plasma coaxial gun parameters were determined. The microstructure of the deposited Carbon - Alumina films were observed using scanning electron microscope (SEM) while thesurface microhardness was measured using Vickers micro-hardness testing machine. The deposited film thickness was measured using electronic coating thickness tester. The surface microhardness increased by 65% at 200 plasma pulses from its value for untreated iron substrate. The film thickness increases with the number of plasma pulses with an average deposition rate of 30 nm/pulse.

KEY WORDS

IPD method, Carbon - Alumina film, Plasma Coaxial Gun.

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1. INTRODUCTION

Modifying mechanical, physical and chemical surface properties of different materials have become of a great industrial goal. Numerous treatment methods were used to achieve this goal. Coatings produced by impulse plasma deposition (IPD) method utilize the plasma generated and accelerated by the Lorentz force [1] in a coaxial plasma gun. This technique has the advantages of having good adhesion between the deposited film and the substrate, high deposition rate, high energy efficiency, room temperature operation and low structural investment [2]. In spite of the stated advantages changing the material of electrodes of the coaxial plasma gun, the working gases and operation parameters, different plasma composition could be obtained, thus films of different material can be produced. Implementation of carbon thin films using the stated technique have a wide variety of industrial applications due to their high hardness, excellent corrosion resistance, wide range of optical transparency and electrical properties [3]. In the past few years, carbon films were deposited as Diamond-like carbon (DLC) using high-density plasma [3]. Various techniques were used for depositing carbon films on different substrates with different operating conditions [4-7].

In this research work, an impulse plasma deposition technique is utilized to deposit Carbon - Alumina on iron substrate at room temperature. Acetylene gas (C_2H_2) was used as a source of carbon atoms. While the inner electrode was the source of the Aluminum present in the deposited Alumina phase. The plasma coaxial gun used for that purpose is a device capable of transforming electrical energy into kinetic energy in the form of direct plasma jet [8]. Bursts of plasma containing ions, atoms and electrons is produced and accelerated into vacuum chamber at a velocity of about 10^4 m/s to bombard the target [3,9]. These species of charged particles are deposited on the substrate surface and implanted in the bulk. The aim of the present work is to study some of the plasma coaxial gun parameters as well as mechanical and metallographic properties of the deposited layers.

2. EXPERMINTAL SETUP AND CONDITIONS OF OPERATION

Schematic diagram of the plasma coaxial gun system is shown in Fig.1, where as the construction of the plasma coaxial gun head is shown in Fig.2. Mainly the plasma gun head consists of two coaxial hollow cylindrical electrodes welded to the vacuum chamber. The inner electrode is 11 cm in length and 2 cm in diameter while the outer electrode is 10.5 cm in length and 4 cm in diameter. The two electrodes are isolated from each other by a Teflon disc. The discharge takes place between the inner and outer electrode. A clamping mechanism is designed to hold the iron substrate during deposition of the ejected particles. During deposition process the substrate holder is manually rotated every 10 pulses to achieve a more homogenous deposited film. While the coaxial tube is fixed in vertical position, the plasma sheath moves upwards [10].The dimensions of the vacuum chamber are choosen to be 13.5 cm in length and 8.6cm in diameter to secure the suitable vacuum level can be attained with the used vacuum pump. A metallic flange (ring flange) is attached to the end of the vacuum chamber using fixation bolts with sealing ring in between to prevent air/gas leakage into the vacuum chamber.

The energy driving the plasma processes is supplied from a 15.42 μF capacitor bank, via a spark gap switch, which prior to each plasma pulse was charged to 6 -13.5 kV (0.3 – 1.41 kJoule). A working gas consists of a mixture of acetylene (C_2H_2) and hydrogen (H_2) is fed to the inlet of the vacuum chamber with a ratio of (1/5). A rotary pump of Alcatel type model 2010 is connected to the outlet of the vacuum chamber. Its ultimate pressure reaches (1.52 mTorr) but the working gas pressure is about 100 mTorr. The pressure of the mixture is adjusted using a pressure gauge type VRC (vacuum research corporation of USA). A needle valve is used to regulate and control the flow of the working gases; also it secures the whole system from explosion due to escaping plasma spark to the gas tubes. The potential difference between the two electrodes is measured by a capacitive potential divider (locally designed and manufactured). Rogowisky coil is designed to be used in measuring the discharging current. Iron samples are used as a substrates for deposition of carbon film. Before bombardment, the samples are prepared by grinding using different grades of emery papers (180-800), then mechanically polished with 0.25 μm diamond suspension on a cloth pad, and finally washed by methanol. The coatings were deposited using 50-200 plasma impulses.

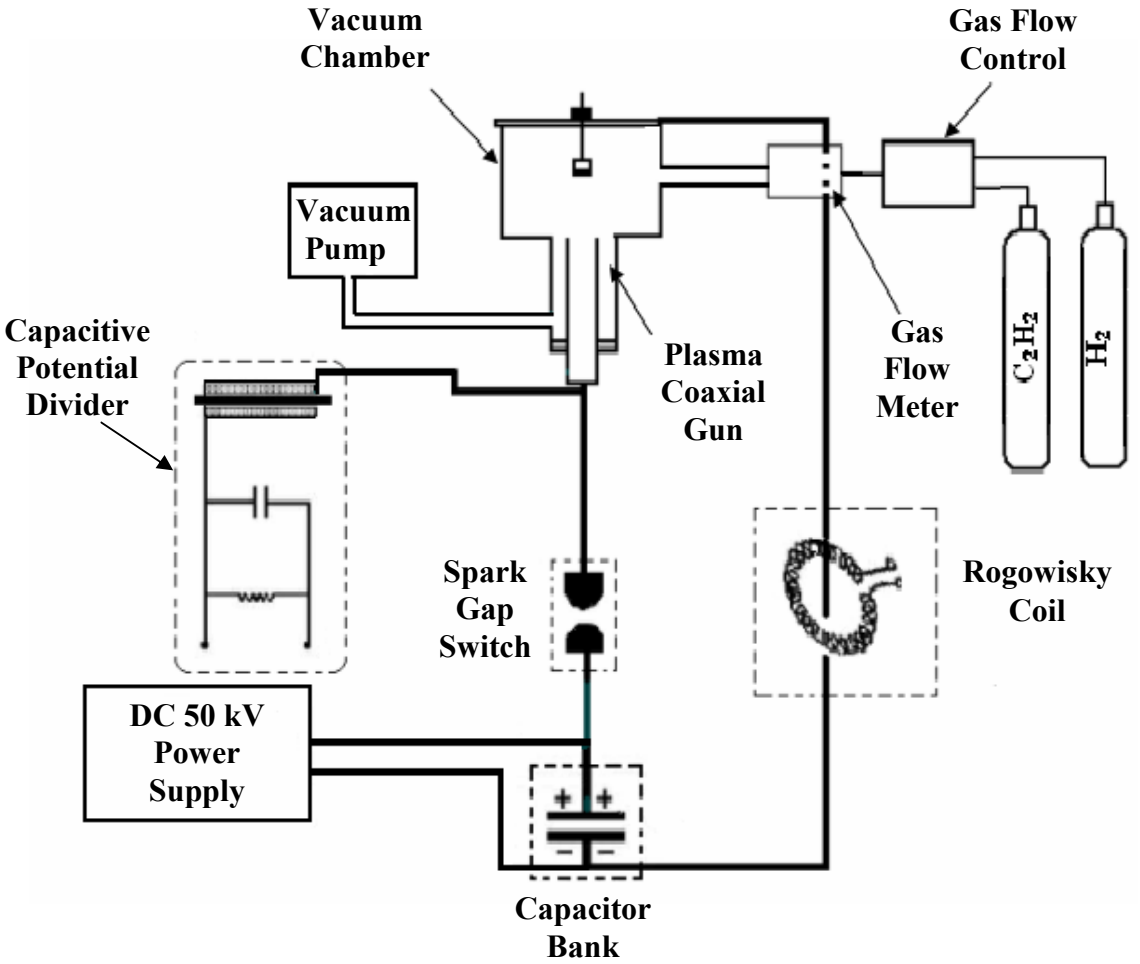

Fig.1. Plasma coaxial gun system

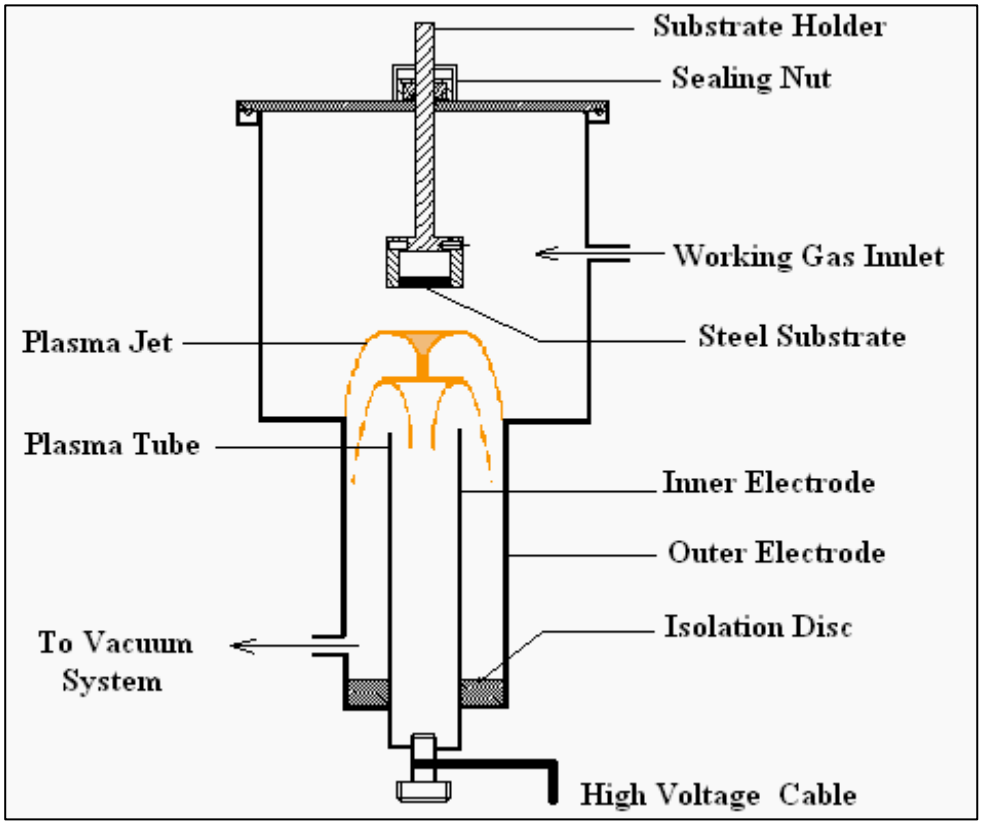

Fig.2. Plasma coaxial gun head

3. RESULTS AND DISCUSSION

I. Plasma coaxial gun parameters

Discharge current and voltage variation with time across the plasma coaxial gun are shown in Fig.3. The current sheath reached 15.9 kA after 17.2 μs from the beginning of the discharge. The discharge current is calculated from the equation [11]:

$$I_o = \frac{C_o V_o (1+f)}{} \qquad (1)$$

Where,

C_o is the capacity of the capacitor bank (15.42 μF).
V_o is the charging voltage (13.5 kV).
τ is the periodic time (average of several cycles taken to be 34 μsec).
f is the reversal ratio, given by :

$$f = \frac{V_2}{V_1} = \frac{1}{2}\left(\frac{V_3}{V_2} + \frac{V_2}{V_1}\right) \qquad (2)$$

The potentials V_1, V_2, and V_3 are the output peaks of the current appears on the oscilloscope as shown in Fig.3. For the stated discharge conditions above, the total

circuit inductance (L_t) and the total circuit resistance (R_t) were calculated as described in [12] to be1.898 μH and 32.17 mΩ, respectively.

Figure 4 shows that the plasma inductance had a peak value of 5.59 μH, 4.58 μH, 4.18 μH and 2.68 μH at discharge times of 2 μs, 16 μs, 34 μs and 50 μs respectively. These represent the times at which the plasma current sheath reached the gun muzzle at each half cycle of the discharge current [10]. From Fig.5 it has been observed that the plasma resistance has a minimum value of 0.1 mΩ, 0.07 mΩ, 0.04 mΩ and 0.01 mΩ at discharge times16 μs, 20 μs, 30 μs and 36 μs, respectively. These minimum values show the complete formation of the plasma sheath current at these times [12].

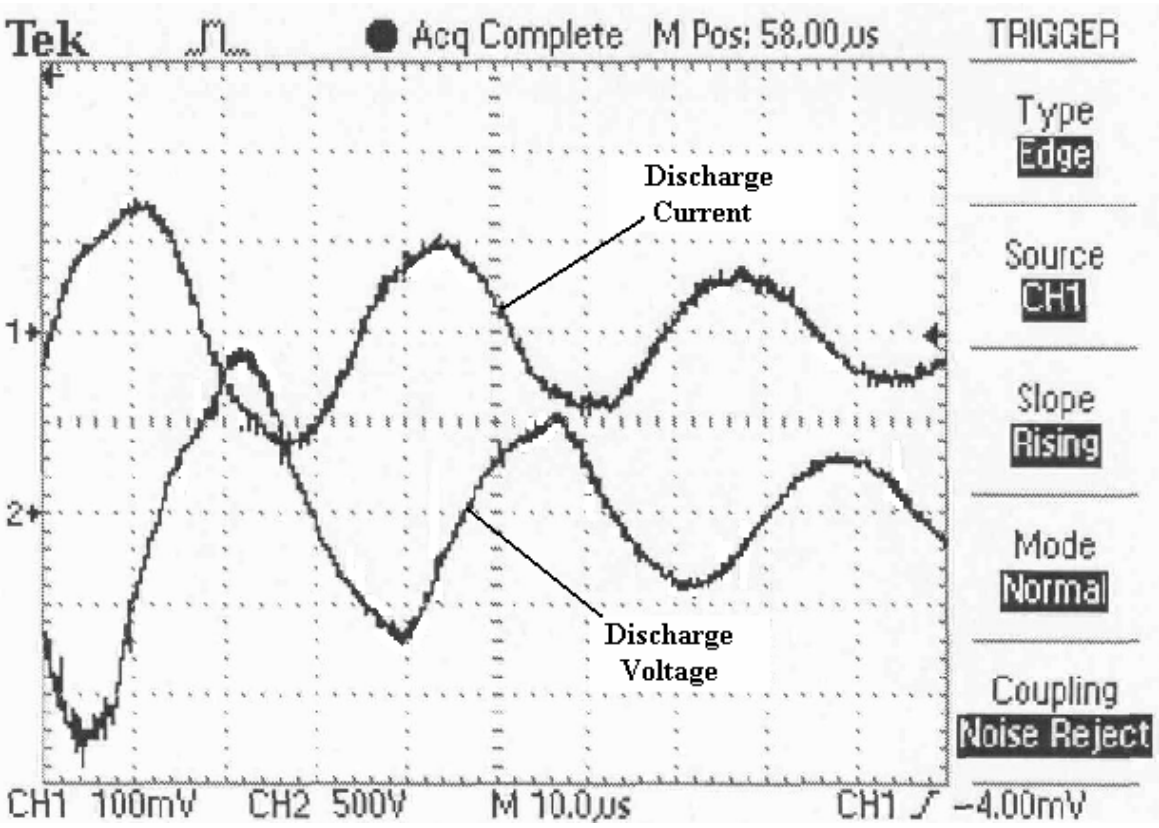

Fig.3. Current and voltage signals at 6 kV and 30 mTorr pressure.

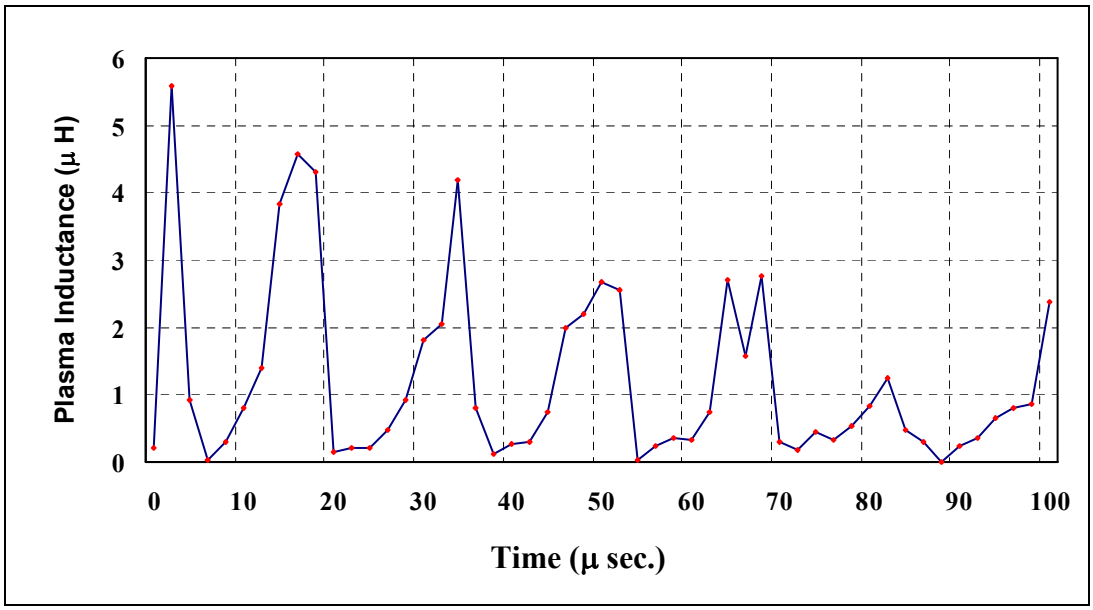

Fig.4. The plasma inductance versus discharge time at 6 kV charging voltage and 30 mTorr pressure.

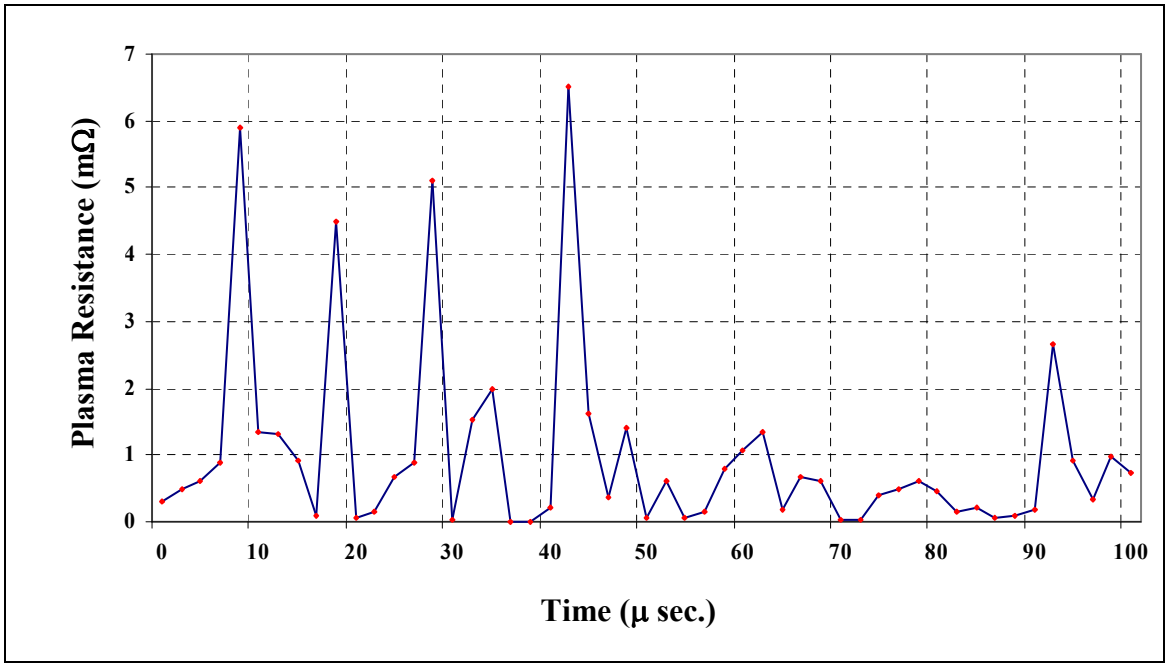

Fig.5. The plasma resistance versus discharge time at 6 kV charging voltage and 30 mTorr pressure.

II. Deposited thin film characterization

1- Influence of the number of pulses on the microstructure

The microstructure of the deposited layers was examined using Scanning Electron Microscope (SEM) type Remma 202. Three groups of the prepared specimens were subjected to the produced plasma jet. The plasma creating parameters are kept constant with a discharging voltage of 13.5 kV, and a constant flow of Acetylene (C_2H_2) and Hydrogen (H_2) mixture with a ratio of 1/5 is maintained.

Figure 6.a shows the microstructure of the first group of specimens exposed to 50 successive plasma pulses with the previously stated conditions. In this figure, particles of different sizes ranging from 0.25 μm to about 1.5μm can be observed . The arial density of these particles is low so that vast areas of dark background are clearly distinguished. Using the microchemical analysis capability attached to the SEM showed that the dark areas are very rich with carbon where as the particles are rich in Aluminum and Oxygen. Calculating the weight ratio of Aluminum and Oxygen and comparing with that obtained by the spot analysis showed that the particles are typically AL_2O_3 compound. This compound, classified as a ceramic material, is a very hard phase and characterized by its thermal (low conductivity) and chemical (anticorrosion) properties.

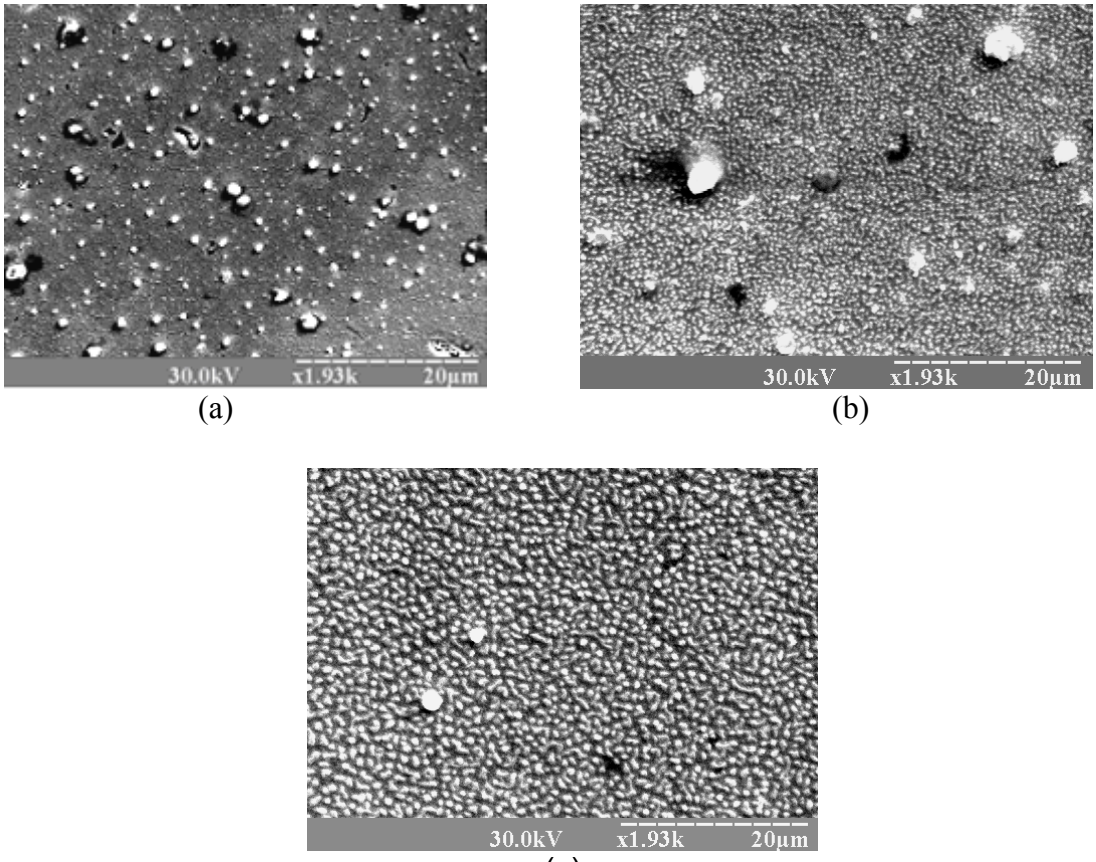

Fig.6. SEM micrograph of the deposited carbon film at 13.5 kV charging voltage and 100 mTorr pressure (a) 50 pulse.(b) 100 pulse.(c) 200 pulse.

These properties nominate the Alumina phase to be used as a deposited layer for enhancing the resistance against environmental, thermal and mechanical effects. Increasing the number of pulses to 100 pulse increases the number of these Alumina particles to cover most of these dark areas previously noticed in Fig. 6.a. Figure 6.b shows the microstructure of the substrate surface of the second group of specimens after receiving 100 pulse. This microstructure fits well with the mechanism of thin film formation [4,13], where surface migration of clusters followed by uncompleted coalescence and agglomeration. Increasing the number of pulses to reach 200 pulse with the same conditions changes the microstructure and the distribution of the deposited particles as shown in Fig. 6.c. In this microstructure the Alumina particles show more uniform distribution and clearly increased size, which may be attributed to the nature of the surface. Many references [1, 2, 7] stated that the main constituents of the deposited phase by plasma should be supplied by both the working gas and the inner electrode. Closer look for the inner electrode showed that the steel tube edge and surface are slightly eroded as a result of evaporation and ablation during discharge. Therefore, the Aluminum present in the deposited Alumina phase is a result of existing the Aluminum atoms normally present in the electrode material (6.52%).

2- Influence of the number of pulses on the microhardness

The relationship between the surface microhardness and the number of the plasma pulses is shown in Fig.7. The microhardness was measured using a Vickers hardness tester type ZWICK 3212. It was observed that when loading the surface with 2 N, 5 N and15 N respectively, there was no indentation observed on the surface due to increased hardness value of the deposited Alumina phase. Hence, 20 N load was choosed to be used to measure the surface microhardness. Fig.8 shows an indent of the microindentor on the deposited layer representing the effect of the second phase particles on the measured hardness value. The results of these measurements is not an indication for the hardness value of the deposited Carbon – Alumina layer, but both the deposited layer and the substrate bulk material together. Measured values of the treated substrates showed that the surface microhardness increased with the number of plasma pulses due to the excess number of Alumina clusters formed on thesubstrate surface. The surface microhardness increased by 65% for treated iron substrates at 200 plasma pulses from its value for untreated one.

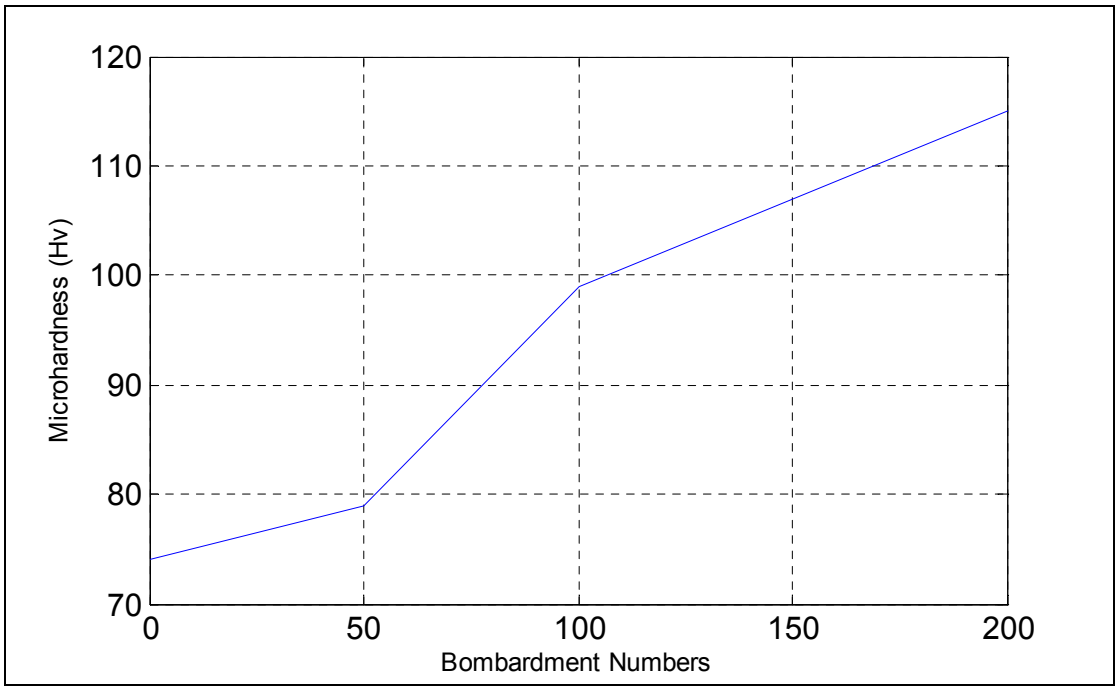

Fig.7. Surface microhardness of the deposited carbon films on the iron substrate as a function of plasma pulses at 13.5 kV charging voltage and 100mTorr pressure using 20 N load.

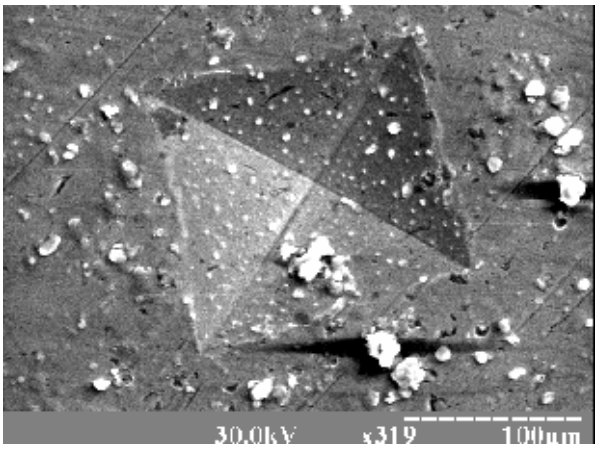

Fig.8. Indent of the microindentor on the deposited layer.

3- Influence of the number of pulses on the film thickness

The deposited film thicknesses were measured by electronic coating thickness tester type KETT model LZ-330. The thickness is one of the most important parameters. Many properties depend on it. Fig.9 shows that the film thickness increases linearly with the number of plasma pulses. This mainly due to the constant chamber gas pressure during the impulse plasma deposition process. Generally the chamber pressure has an effect on the amount of pulsed gases, which control the deposition rate [3].From this figure the average deposition rate is calculated to be 30 nm/pulse.

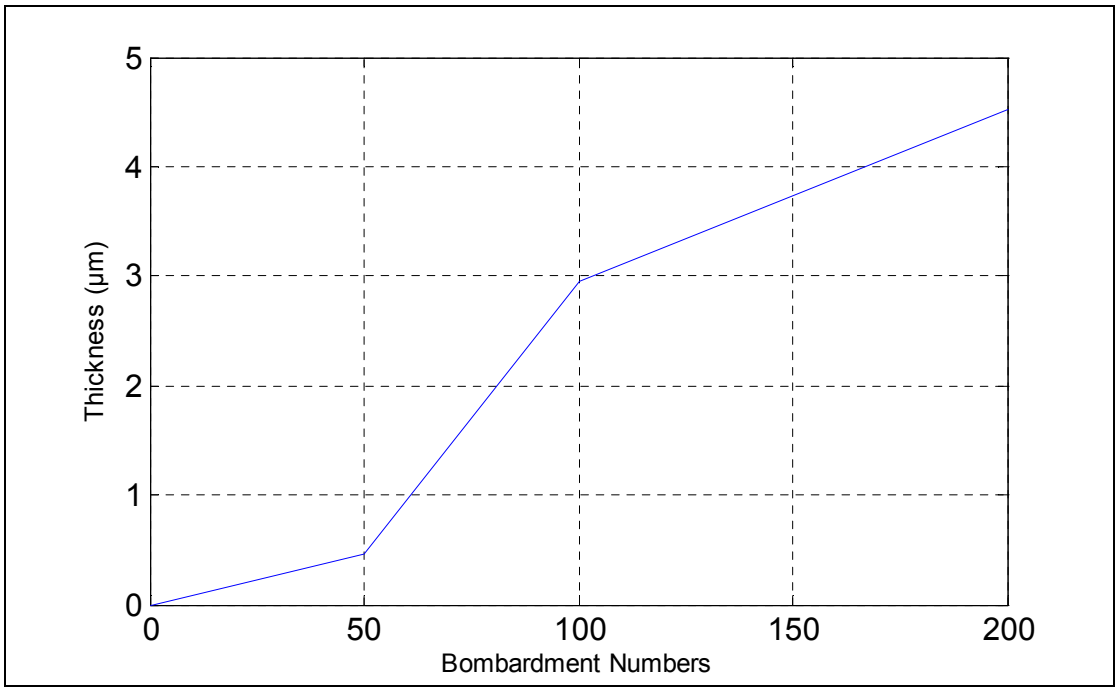

Fig.9. Relationship between the thickness of the deposited films and the number of plasma pulses at 13.5 kV charging voltage and 100mTorr pressure.

4. CONCLUSIONS

Deposition of Carbon - Alumina films on iron substrates were done using plasma coaxial gun. Discharge current and voltage were measured with first peak current of about 15.9 kA after 17.2 µs at 6 kV charging voltage. Plasma inductance had a peak value of 5.59 µH, 4.58 µH, 4.18 µH and 2.68 µH at discharge times of 2 µs, 16 µs, 34 µs and 50 µs respectively. These represent the times at which the plasma current sheath reached the gun muzzle at each half cycle of the discharge current. While plasma resistance had a minimum value of 0.1 m , 0.07 m , 0.04 m and 0.01 mΩ at discharge times16 µs, 20 µs, 30 µs and 36 µs, respectively. These minimum values show the complete formation of the plasma sheath current at these times. A more uniform distribution with increased size of deposited Alumina particles was

formed after 200 plasma pulses. The surface microhardness increased by 65% at 200 plasma pulses from its value for untreated iron substrate due to the excess number of Alumina particles in the deposited layer. The film thickness increases with the number of plasma pulses with an average deposition rate of30 nm/pulse.

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