

Effect of electro-thermal plasma on polymer materials (Teflon and polyethyleneterephthalate (PET))

F.Diab, G.M.El-Aragi*, G.M.El-Kashef

Plasma and Nuclear Fusion Department, AEA, Cairo, (EGYPT)

E-mail: elaragi@gmail.com

Abstract : Effects of capillary plasma discharge irradiation at different applied energy on polyethylene terephthalate (PET) polymer films have been investigated by Fourier transform infrared (FTIR) spectroscopy, and ultraviolet-visible (UV-VIS) spectroscopy. Moreover, Irradiation of polymers has become an important technique in producing special materials for electronic applications. Atmospheric pressure non-thermal plasmas are of great interest in polymer surface treatment due to their convenience, effectiveness and low cost. Moreover, irradiation of polymers has become an important technique in producing special materials for electronic applications. Surface of polyethylenetere-phthalate (PET) was modified by plasma produced from the capillary discharge. Some physical properties of PTFE samples were studied. In this paper the change in the surface free en-

ergy of (PTFE) samples which is induced by the capillary plasma discharge, is investigated. In addition, FTIR technique is applied for sample analysis, to study the change in the structure of the film as irradiated. The experimental analysis of the film hardness illustrate that the Vickers's micro hardness of the film increases upon plasma treatment. The morphology of the particulate and surface structure which generate by the plasma ablation is investigated. The surface morphology of the pristine and irradiated PET and PTFE samples are investigated as imaged by Scanning Electron Microscope (SEM).

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Keywords : Capillary discharge; Plasma ablation; Polymer; Spectroscopy; SEM.

INTRODUCTION

The capillary device, typically made out of Teflon and Artelon which are closed at one end by an electrode, the other end being open, with an annular electrode. The two electrodes are connected by a wire running through the capillary and by an external circuit. This circuit includes a pre-charged storage device, usually a capacitor bank, and a switch to close the circuit. When the switch is closed the wire rapidly heats and explodes, initiating a plasma discharge in the capillary

which then carries the current. The efflux of material from the open end is compensated for by material being ablated from the capillary wall. The area of plasma polymerization is now well recognized as an important part of material science. Several surface modification methods are employed to modify the polymer surfaces, such as chemical, thermal, mechanical and electrical treatments^[1]. The plasma discharge treatment of polymers has been gaining popularity as a surface modification technique with the aim to increase the polymer surface "attractivity" for applications in tissue engineering

and electronics^[2].

Another interesting property of radiation-modified polymers is the formation of conjugated double bonds between carbon atoms and increased electrical conductivity of the material which may support their colonization with living cells higher or adhesion of subsequently deposited metals^[3]. The plasma produced from the capillary in our work has a wide range of applications on some polymer samples; one of these samples is the polyethylene terephthalate (PET) film. Polyethylene terephthalate (PET) film is used in many technological fields for a wide variety of applications (packaging, decorative coatings, capacitors, magnetic tape, . . .) since it has some excellent bulk properties, such as very good barrier properties, crease resistance, solvent resistance, high melting point, resistance to fatigue, and high tenacity as either a film or a fiber. However, PET is sometimes an unsuitable material to use due to its low surface free energy, leading to poor wettability and poor adhesion^[4,5]. It is necessary to modify their surfaces to increase the surface free energy without change in their bulk properties for many commercial applications.

PET, an aliphatic-aromatic polymer composition and thermoplastic, shows a rather hydrophobic nature due to its rigid structure. Moreover, PET, having a chemical composition of C, H, and O, can be expected to be highly infrared active. It was, therefore, decided to carry out a careful infrared spectroscopy using a FTIR spectrometer to understand the molecular bonds. The chemical structure of a PET is:-

The two carbonyl functions together with the aromatic ring provide the structural rigidity of the macromolecule; little flexibility arises due to the presence of the ethylene group in the repeating unit.

For another polymer namely Polytetrafluoroethylene (PTFE), another specific experiments were carried out. Polytetrafluoroethylene (PTFE) is a semi-crystalline polymer having outstanding chemical, physical, electrical and mechanical properties, such as excellent resis-

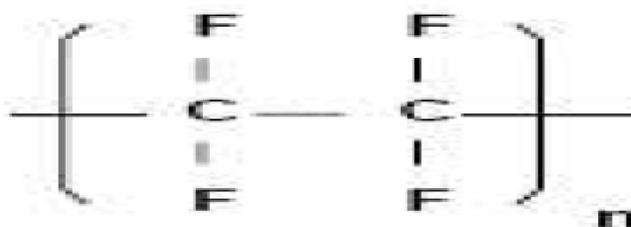
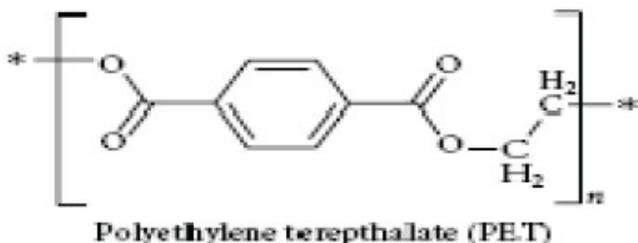
tance to chemical reagents, thermal stability in a wide temperature range, low dielectric constant, high electric resistance, very low surface free energy and friction coefficient. This self-lubricating property of PTFE is exploited in a wide range of applications from rubber ink stamps, ice-making machines, and ultrahigh vacuum bearings. Polymer films (PTFE) used in this study was irradiated by capillary plasma discharge at different energies. Moreover, Physical properties of pristine Polytetrafluoroethylen (PTFE) is: - white colour, transparent, density= 2.2 g/cm³, glass transition temperature (T_g) = 120 °C, melting temperature (T_m) = 327 °C Composition: [C2F4] n

A Scanning Electron Microscope (SEM), X-ray Florescence (XRF) was performed to examine the change of surface morphology for the pristine and irradiated polymer. The present study aims to improve the adhesion for PTFE by increasing the wettability and surface free energy. This can be done via inducing structural change with some surface modifications for the polymer through plasma irradiation.

METALLIZATION OF PTFE BY CAPILLARY PLASMA TREATMENT PROCESS

A crucial role in preparing metal layers on polymers is played by the interface properties between substrate and deposited metal. This attribute can benefit from ion beam treatment of the polymeric surface. Just by varying the treatment conditions, it is possible either to increase or to decrease the wettability of the given polymeric surface. In order to understand the changes made to the surfaces during treatment, we need analytical techniques that can accurately characterize the surface before and after modification. Metal deposition on polymers can be carried out by sputtering, vacuum deposition, and also by various electrochemical procedures^[6]. The structure of the metal layer is mainly influ-

Structure



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enced by nucleation processes^[7,8].

Metallized polymer films are widely used in industries ranging from food packaging to biosensors^[9]. Interesting studies have been made on various aspects of metal/polymer interface formation^[10]. Metallized polymers have been used in various technologies such as magnetic recording media, electro conductive films, computer technology, and flexible printed circuits^[11]. Different properties of metallized polymers are required depending on their applications. PTFE is a promising material for flexible printed circuits and capacitors due to good thermal and dielectric properties. Although PTFE has superior properties, their practical application has been hampered because of their poor adhesion due to their chemical inertness. In order to improve the adhesion between metals and polymers, many researchers have investigated various methods such as ion beam sputtering, wet chemical etching, plasma treatment^[12,13]. The effect plasma irradiation on the wettability and surface energy of PTFE polymer has been investigated. Upon irradiation, C-F and C-C bonds are broken and free radicals are produced, which interact with the ambient oxygen. In this way, oxygen are created, which increases the polymer surface polarity.

The irradiated as well as the un-irradiated PET and PTFE films were studied by different techniques.

1. Fourier Transform Infrared Spectroscopy (FTIR)
2. Scanning Electron Microscope (SEM)
3. X-ray Florescence (XRF)
4. Uv/Visible Absorption Spectroscopy

EXPERIMENTAL SET-UP

A Photograph of the experimental arrangement



Figure 1 : Photographic view of an electrothermal capillary discharge device

and its principle is shown in Figure 1. The system consists mainly of: the electrothermal source section (plasma gun), the capacitor bank, the air gap switch, the triggering system, the electrical power supply, transmission line, associated diagnostics, and safety procedures. Detailed description of each part will be given below.

The capacitor is connected to the cathode of the gun via a transmission line and a spark gap switch. The anode of the gun is inserted in the center of the capillary tube. The electrical energy of the capacitor is discharged through the gun upon closure of the switch. The plasma gun is typically operated in air and a high electrical current vaporizes an aluminum wire in a small tube and generates hot plasma by joule heating. Moreover, the plasma source is generated by the rapid discharge of 3.5 kJ of electrical energy into a Teflon (Lexan) capillary. This energy is stored in a 70 μF capacitor charged to a maximum of 10 kV. The capacitor was discharged with a wire load through a spark gap switch and inductance L . In the experiment, inductance L could be changed into eight values from 1.3 to 80 μH by an external coil. The capillary is (3mm-8mm) in diameter, (10 mm - 200 mm) long, and is open at one end only. The discharge is initiated with a thin copper or aluminum fuse wires (less than 1 mm), ablation and ionization of material from the capillary surface sustains the discharge. The peak current through the plasma is approximately 37 kA for a storage energy of 0.9 kJ, and the periodic discharge duration time is approximately (more or less 200 μs). The anode is made of copper and the cathode is made of aluminum. The aluminum wire is enclosed in a cylindrical tube, and has a good contact to two electrodes.

The discharge voltage across the capillary in this experiment was measured as a function of time using a high voltage probe (BK-PR-28A). The discharge current trace was measured using a Rogowski coil and the signal was integrated with respect to time to get the total current in the plasma circuit during the discharge. Both the capillary tube and the two flanges are installed on a holder such as the car which allows easy movement forward and back and also in which you can press on the flanges in order to insure proper installation.

RESULTS AND DISCUSSION

Current and voltage measurements of the capillary

In atmospheric pressure a clear signature of the wire explosion is evident (the current sharply drops and increases again shortly afterwards)^[14]. The peak in the current and voltage (resistance) is known as the “wire-

burst.” The wire fragmentation is accompanied by a small inflection in the current profile. Moreover, the discharge current trace shows a clear double peak structure. The first peak is due to the ignition wire explosion and the second peak represents the main discharge. The gun voltage and current for a 1.3 KJ discharge is shown in Figure 2 for charging voltage = 3.5 KV. The gun voltage is measured across the source section with a high voltage probe (BK-PR-28A). The current trace was measured using a Rogowski coil and the signal was integrated with respect to time to get the total current in the plasma circuit during the discharge. The gun voltage increases rapidly after the initiation of the discharge, typically reaching the maximum voltage within 10 μ s. The current increases much more slowly than the voltage. It takes about 22 μ s from the initiation of the discharge for the current to reach its maximum. It is apparent in the figure that the gun current lags the gun voltage; this indicates that the source section has an

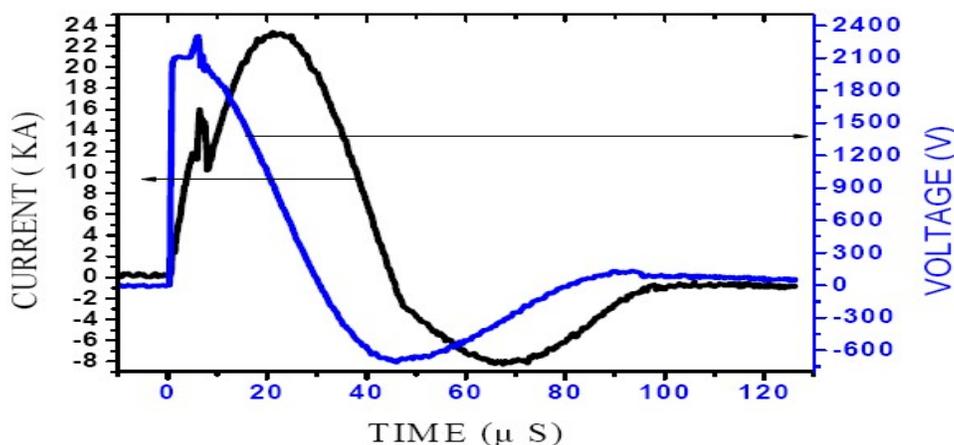


Figure 2 : Discharge current and voltage of the gun at charging voltage =3.5 KV.

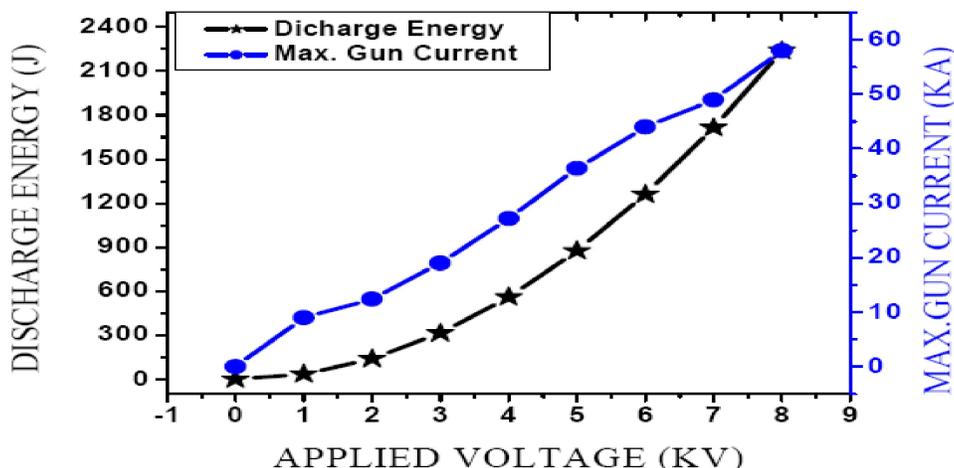


Figure 3 : Discharge energy and maximum gun current versus applied capacitor voltage.

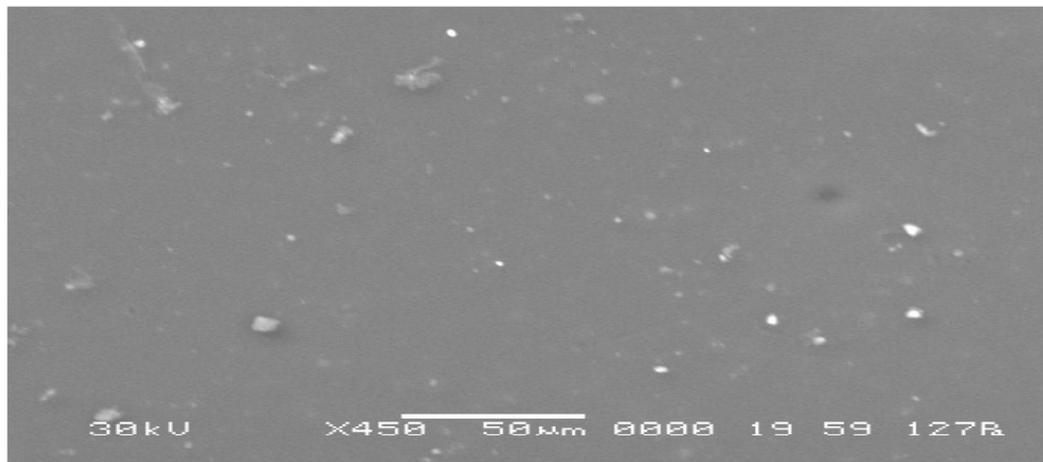
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Figure 4 : SEM photographs of a PET sample before exposed to the plasma

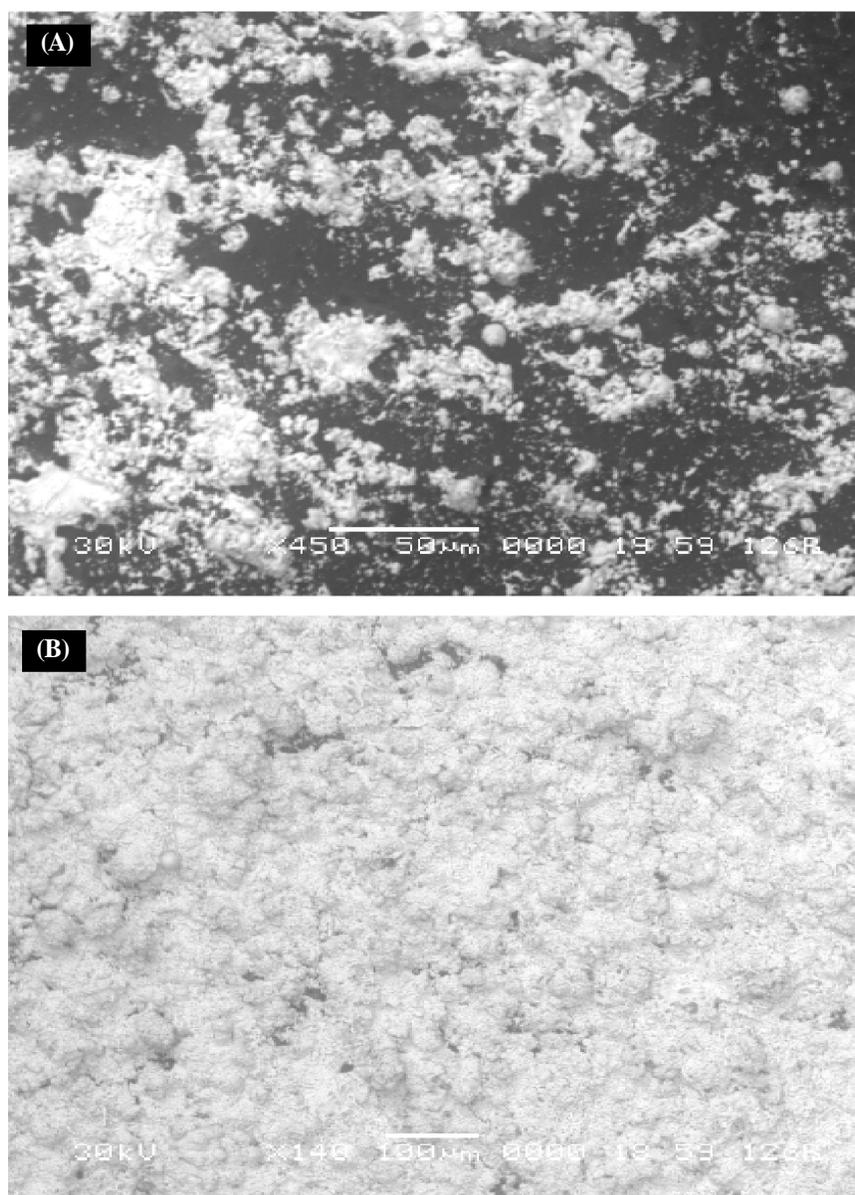


Figure 5 : SEM photographs of a PET sample after exposed to the plasma with energy of (A) 875 J and (B) 1715 J

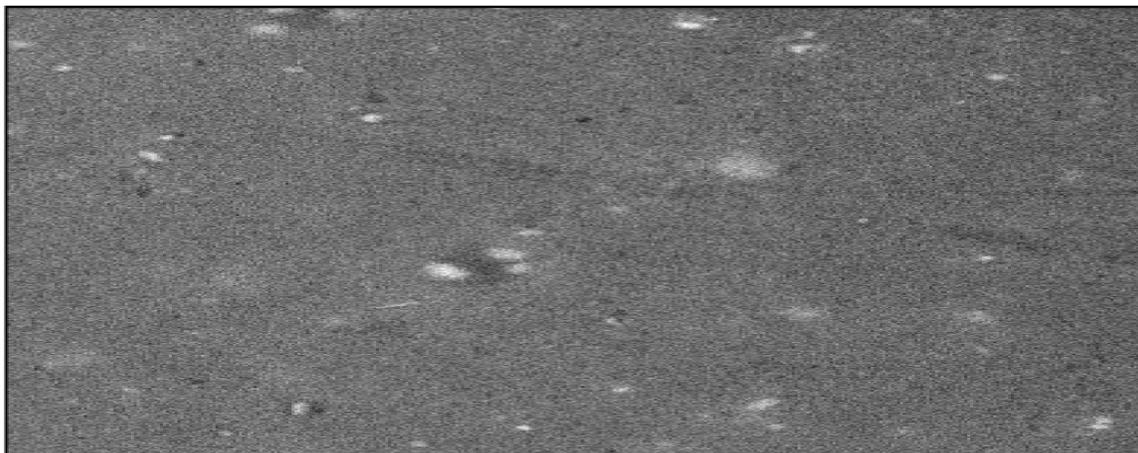


Figure 6 : SEM photographs of a PTFE sample before exposed to the plasma

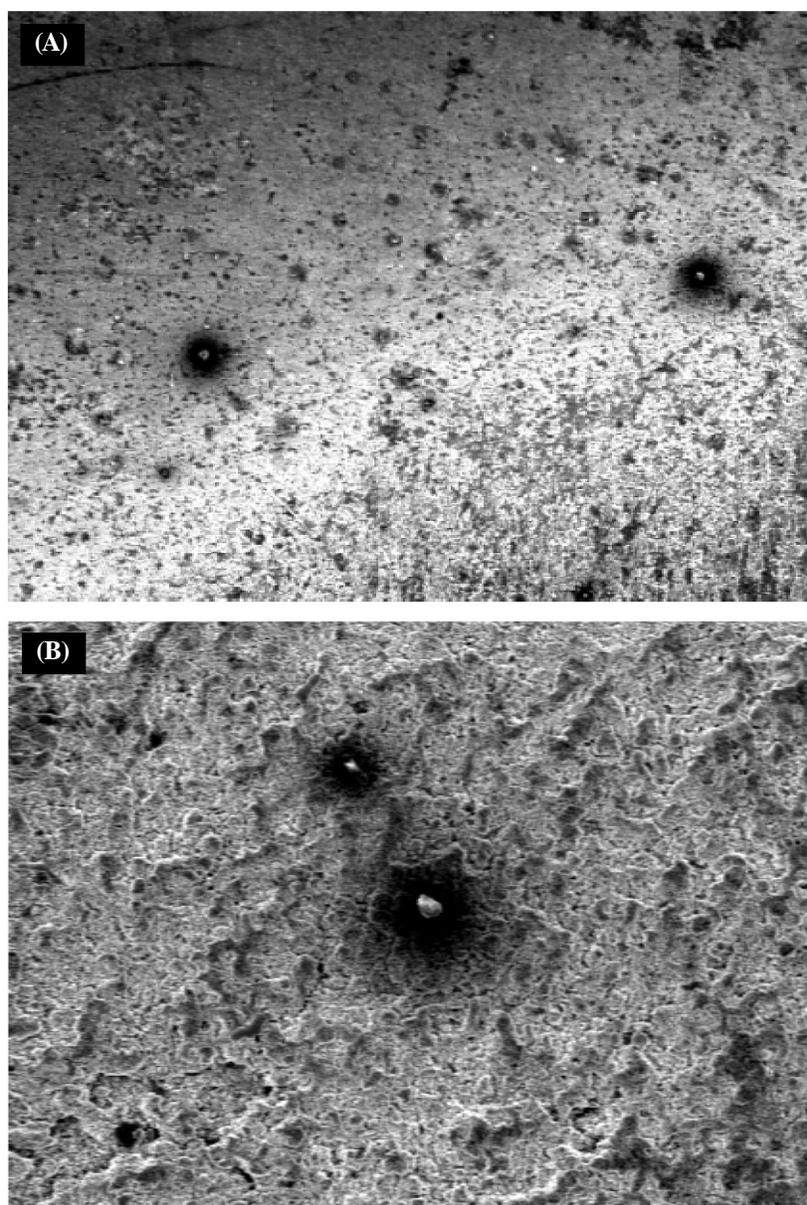


Figure 7 : SEM photographs of a PTFE sample after exposed to the plasma with energy of (A) 875 J and (B) 1715 J

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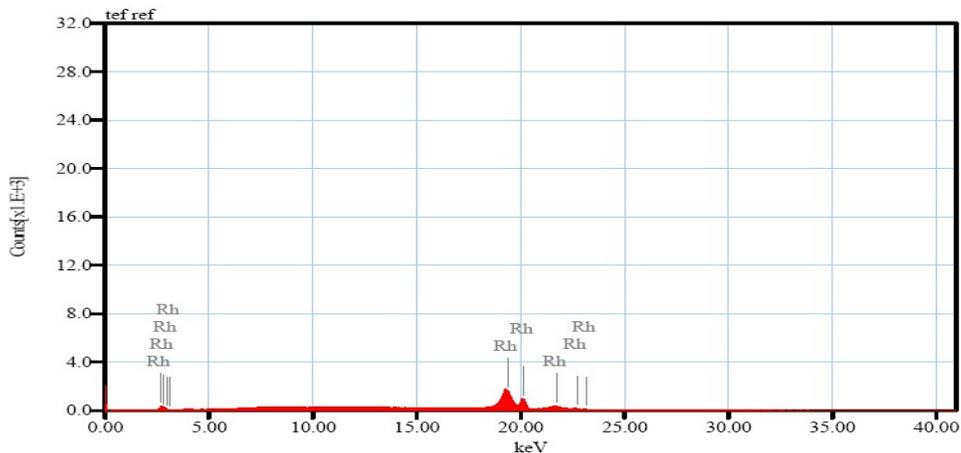


Figure 8 : Typical energy dispersive XRF spectrum of a pristine PTFE sample before exposed to the plasma

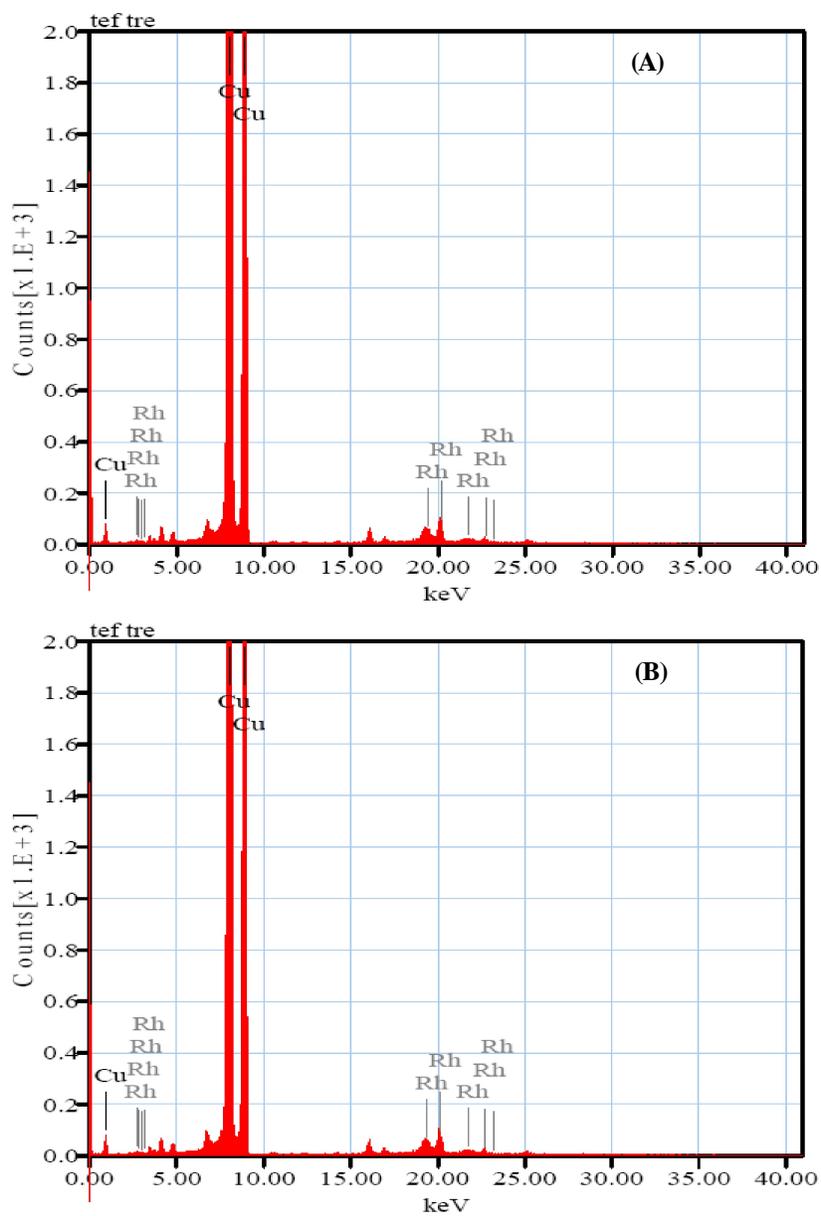


Figure 9 : Typical energy dispersive XRF spectrum of a pure Teflon sample

inductive component.

Discharge current and energy versus input capacitor voltage

Figure 3 illustrates the input energy and maximum gun current versus the applied voltage results of the electrothermal plasma gun. The applied voltage is the capacitor voltage before initiating the discharge. It can be seen from the figure that the peak of energy increases with increasing initial voltage and the peak of current increase also. The input energy in the above figure is calculated from the following relation:

$$E = \frac{1}{2} CV^2$$

Where E is the input energy, C is the capacitance, equal to 70 μ F of the electrothermal gun and V is the applied voltage.

SURFACE MORPHOLOGY OF PET POLYMER

The structure of the surface of PET with different treatment energy of capillary plasma discharge (875 J and 1715 J) was studied by SEM and shown in Figure 5. The surface of PET without capillary plasma discharge treatment is smooth (Figure 4). As the treatment energy increases the surface of the PET sample appear more change. The plasma treatment is a process in which the plasma and PET interacts. There are plenty of active particles in the atmosphere capillary plasma discharge, like carbon, carbon oxide, neutral particle, free radical, ultraviolet radiation and so on. Some of them can interact with the surface of PET to break the chemical bonds on the surface of PET.

SURFACE MORPHOLOGY OF PTFE POLYMER

In order to investigate the change in the surface morphology for the pristine and irradiated PTFE by capillary plasma discharge at different energies, SEM was performed. The pristine sample showed a very smooth surface, as shown in Figure 6^[15,16]. The morphology of the irradiated samples with capillary plasma is shown in Figures 7. The SEM photographs of the irradiated samples reveal that there is a change in the morphology

as the energies is increased. It can be seen from Figure 7 that numerous microfibers and small voids were formed on the surface of the irradiated PTFE with plasma energy of 1715 J.

Figure 7A shows the sample of PTFE after expose o the plasma with energy of 875 J, the SEM results illustrate a big spots on the surface of the sample. Figure 7B shows a lot of big spots of the copper materials deposited on the surface of the sample. It is pointed out that more main chains were broken by incident plasma, and heavy metal erosion occurred on the surface of the sample. SEM results have evidence that the irradiated samples have a higher degree of adhesion for metal deposition than the un-irradiated film and the surface being more homogeneous^[17].

As can be seen from Figures 6 and 7, the surface morphology of the pristine and irradiated PTFE illustrates a change in the surface color. The color of films changes gradually with started the plasma towards the surface of the sample, the change in the color of polymer could be attributed to the formation of hydrogenous carbon clusters and the density of such clusters increases with increasing the plasma energy.

X-RAY FLUORESCENCE (XRF) ANALYSIS

Figure 8 shows the XRF results of the pristine PTFE sample before exposed to the plasma and the major elements concentrations is the Rh and no other element appear. In general, especially in the case of thin film deposition, the structure and morphology as well as the stoichiometry of the film depend strongly on the energetic conditions at the surface^[14]. Here we present the study of the PTFE Sample as a thin film deposition by means of atmospheric pressure of electrothermal launcher Plasma. We use a capillary tube as a source of plasma with a maximum power 106 MW. The main purpose of these coatings is to provide materials with added resistance to wear, corrosion, and oxidation and may also have applications in electrical and electronic fields^[18].

As can be seen, Figure 9 (A) and (B) show a quite different of the surface of the sample and clearly showed the appearance of pure copper film deposited on the surface according to XRF analyses. It was found by

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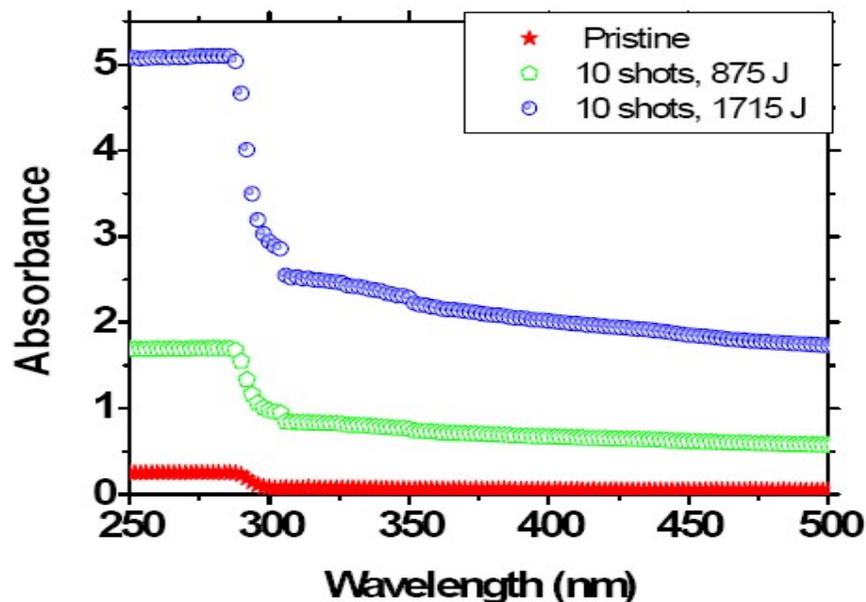


Figure 10 : UV-Vis. spectra of the PET before and after irradiated in atmospheric pressure at different exposure energy.

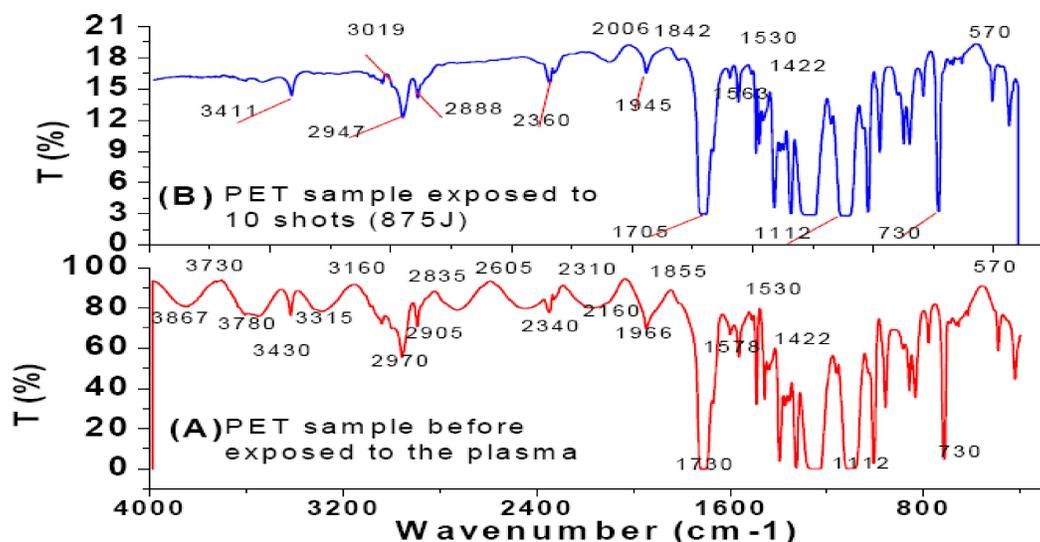


Figure 11 : FTIR spectrum of unirradiated (A) and irradiated (B) PET sample showing transmittance ($T\%$) as a function of wavenumber (cm^{-1}) for the range $4000\text{--}500\text{ cm}^{-1}$

analysis some other elements appear like copper and Rh peaks appears on the surface as shown in Figure 9. The operating voltage and the current of the X-ray tube were 30 kV and 0.6 mA, respectively. The measurement time for the determination of the main components of the sample inside the XRF system was 300 seconds. The sample exposed to the plasma for a 10 shots with energy of 875 J and the other PTFE sample exposed to the plasma for a 10 shots with energy of 1715 J. The appearance of copper peaks deposited on the surface of the two samples can be explained as the exploding of the copper wire and the anode which

made also from copper due to the energy supply. The XRF results of the PTFE sample after exposed to the plasma shows that the major elements concentrations is the copper which are (99 %). Other element such as Rh was observed with low concentrations which is (0.3851%) as shown in Figure 9.

ULTRAVIOLET-VISIBLE ABSORPTION (UV/VIS)

The absorption of light energy by polymeric materials^[19] in (UV/VIS) regions involves transition of elec-

tron in n , π and σ orbital from ground state to high energy states. (UV/VIS) spectroscopy has become an important tool to estimate the value of optical energy gap (E_g). Figure 10 shows the (UV/VIS) spectra of the PET irradiated to the capillary plasma at different energy. The results showed that the highest shift in absorption wavelength is in the range 295-320 nm. This indicates the carbonization of the PET under irradiation.

It is noticed that irradiation results in a shifting of the spectra towards longer wavelengths for all irradiated polymer samples. The absorbance spectrum shows how the absorbance of light depends upon the wavelength of the light. The spectrum itself is a plot of absorbance versus wavelength and is characterized by the wavelength (λ_{min}) at which the absorbance is the greatest. The value of λ_{min} is important for several reasons. This wavelength is characteristic of each compound and provides information on the electronic structure of the crystal or sample.

FOURIER TRANSFORM INFRARED (FTIR) SPECTROSCOPY

FTIR spectroscopy is one of the most powerful tools among the qualitative and quantitative methods of studying molecular bonding, structure and functional group analysis. The technique namely FTIR spectroscopy is used in this work to analyze two samples exposed to the plasma, these samples are polyethylene terephthalate (PET) and Polytetrafluoroethylene

(PTFE).

The peaks in FTIR spectra for PET give dual information^[20]. Firstly, every peak position is fundamental to molecular bonding structure or functional group existing in the infrared active material. So, any shift in the peak position in a spectrum directly refers to a change in bond strength or bond angle. Secondly, variation in intensity of particular peak in a spectrum correlates to the proportion of that functional group present in the material.

Infra-red spectroscopy is one of the most commonly used spectroscopic tools, which measures the vibration energy levels of molecules. FTIR spectroscopy is used to determine the extent of structural destruction in PET induced by ion beams. FTIR is an important tool to check the orientation of different segments in polymer. This technique applied to investigate the modifications induced in polymers by ion beam irradiation^[21].

Capillary Plasma Discharge of a solid target (polymer) causes significant changes in the structure and properties of the bombarded surface. These changes in the bombarded surface depend mainly on the energy, the material used and the type of irradiated ions, their kinetic energy and the density of ion flux.

Full FTIR spectrum (4000 to 500 cm^{-1}) for irradiated and unirradiated PET samples has been shown in Figure 11 (B) and (A) respectively. FTIR results for capillary plasma irradiated PET samples along with that for the unirradiated sample have been compiled into a composite graph (Figure 11) for easy comparison. In

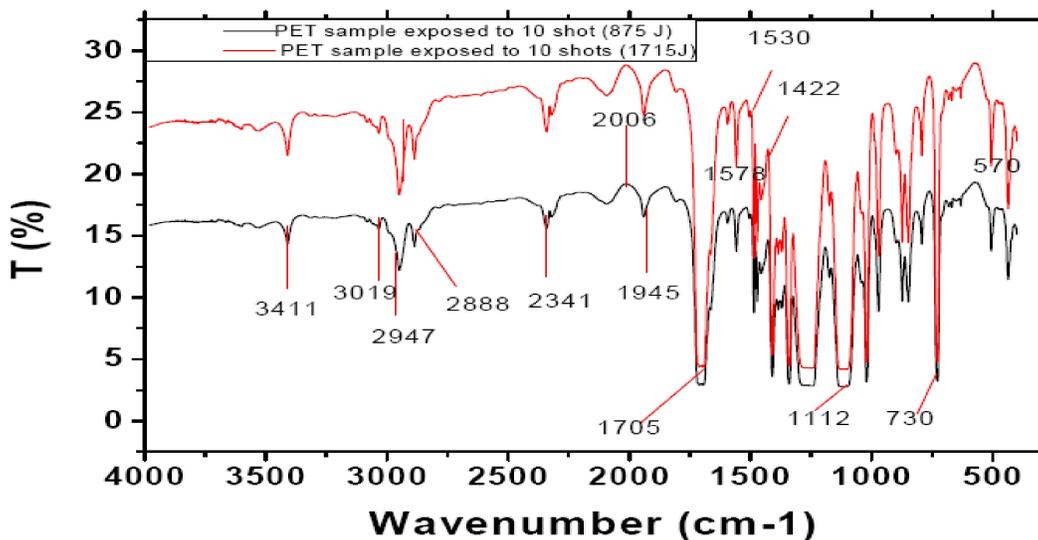


Figure 12 : FTIR spectrum of irradiated PET sample showing transmittance ($T\%$) as a function of wavenumber (cm^{-1}) for the range 4000-500 cm^{-1}

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this figure, the FTIR spectra of PET samples with successive capillary plasma irradiations are depicted in the region 4000 cm^{-1} to 500 cm^{-1} to focus on the main region of interest only. The usual practice of plotting transmission (in %), as a function of wave number (in cm^{-1}), has been followed.

From Figure 11(B) there are some peaks not appear after exposed to the plasma these peaks indicates to some bonds are broken in the particular bonding

structure. Moreover, there are peaks shifted towards the longer wave numbers that means the bonds become stronger than before irradiations. Also, the bond shifted towards the shorter wave number becomes weaker than before irradiations.

The dips in the transmission spectra refer to absorption maxima corresponding to characteristic molecules and groups^[19]. In polyethylene terephthalate (PET), there are molecular groups (involving carbon,

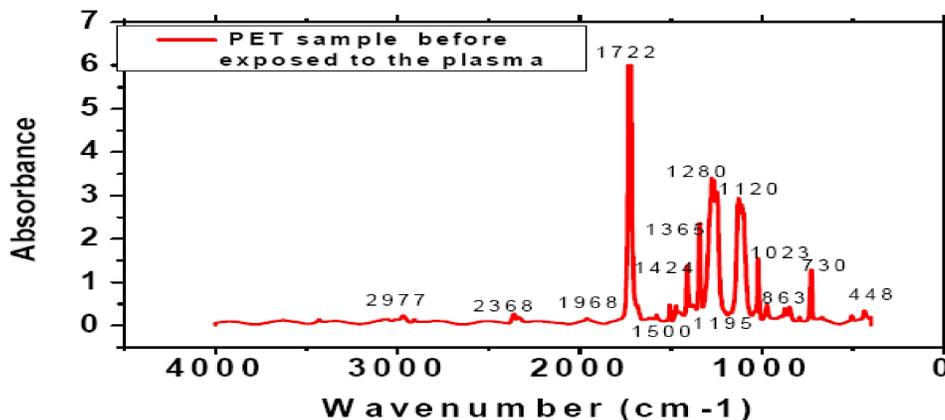


Figure 13 : FTIR spectra of PET sample before exposed to the plasma.

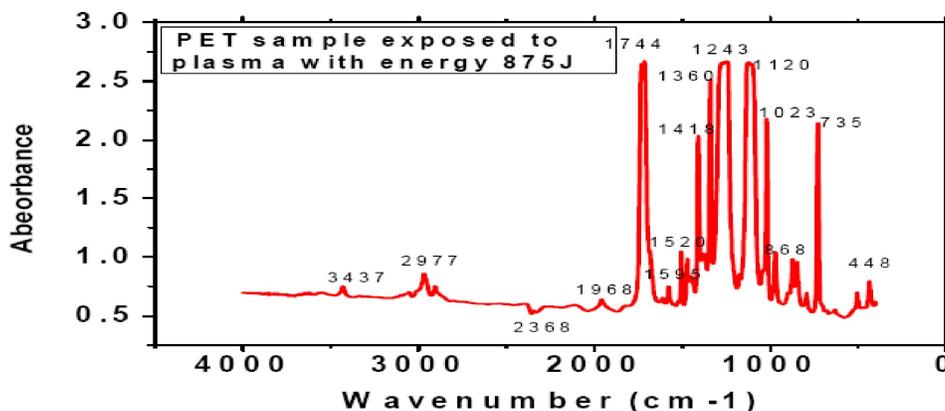


Figure 14 : FTIR spectra of PET sample after exposed to the plasma with energy of 875 J.

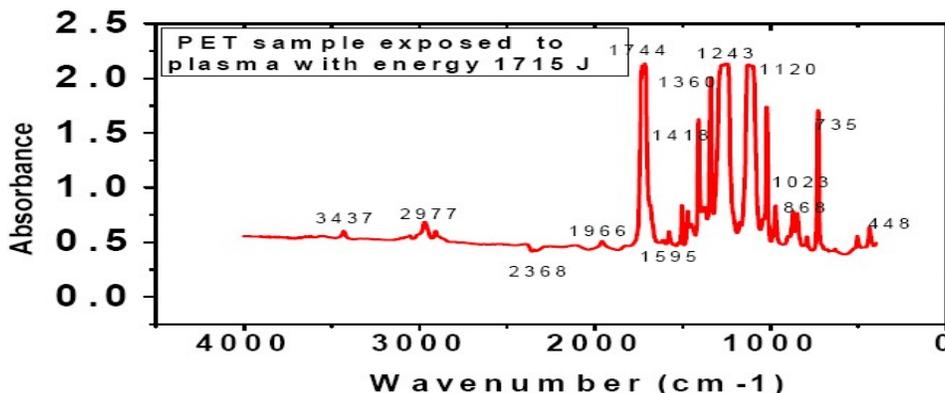


Figure 15 : FTIR spectra of PET sample after exposed to the plasma with energy of 1715J.

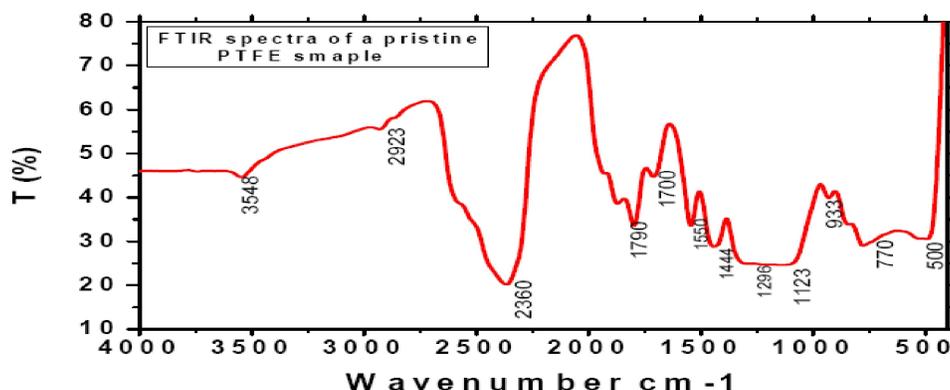


Figure 16 : FTIR spectra for the pristine PTFE sample.

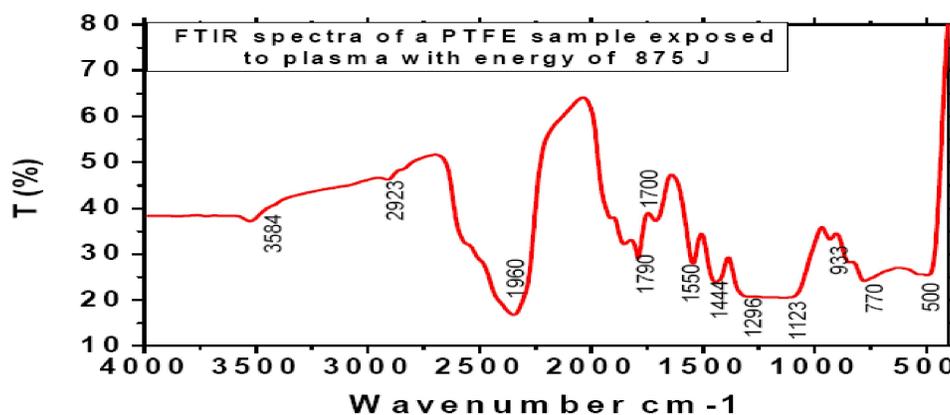


Figure 17 : FTIR spectra for the PTFE films exposed to plasma with energy of 875 J.

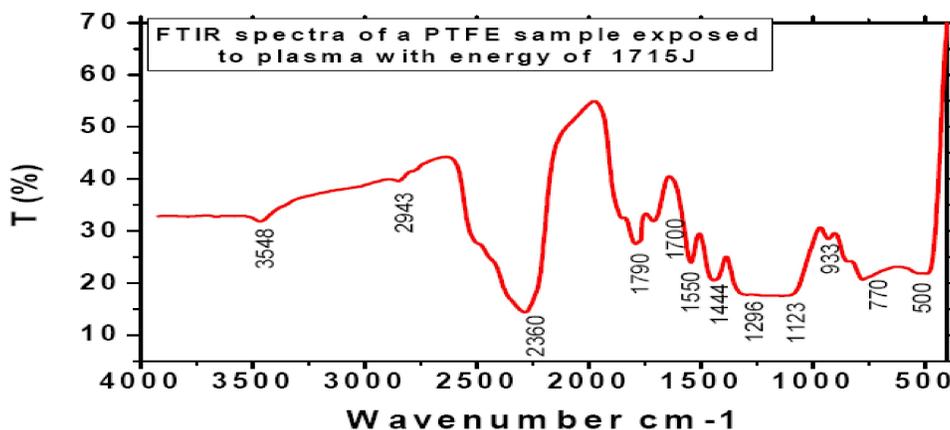


Figure 18 : FTIR spectra for the PTFE films exposed to plasma with energy of 1715 J.

hydrogen, and oxygen), which are highly infrared active and have characteristic absorption peaks.

So, any shift in peak position in a spectrum directly refers to a change in bond strength or bond angle. This means any change in the peak directly influence the bonding ($=\text{CO}$, $-\text{CHO}$, $-\text{OH}$, etc.) of the material. The influence either weakens or strengthens bonding corresponding to a functional group. Weakening and strengthening of a bond shifts the wave number of the corresponding absorption peak to lower and higher values, respectively.

Absence of a particular molecular bond relates to scission or break in the particular bonding structure^[19]. From Figure 12, for the PET pristine film, there are two wave numbers regions of interest: the first is between 3600 to 2500 cm^{-1} , where the C–H and O–H for stretching vibrations of different types of H-bonds, the second is between 1680–1430 cm^{-1} typical for vibration modes of the aromatic rings, for stretching vibrations of double bonds and for CH_2 bending vibrations^[22,23].

The band at 1578 cm^{-1} is assigned to the normal

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vibration (C-H stretching) of the phenyl ring. It is found to be stable for PET exposed to the plasma. The stability of aromatic group is due to its ability to delocalize the excited energies^[23]. The IR spectra of the irradiated PET samples by capillary plasma discharge show that this polymer was partially damaged by the plasma energy dissipated into the samples. This energy could be used for many processes, such as cross-linking of unsaturated adjacent radicals, amorphization of the crystalline fraction of the polymer, scission of the chains and oxidation^[22,25].

An interesting result is the absorbance of the 730 cm^{-1} vibration band, which corresponds to the bending (rocking) vibration of the CH_2 group of crystal phase^[23]. At lower energy fluence, there is no change in the absorbance band, followed by a partial decrease in the absorbance at higher energy fluence (1715 J) as shown in Figure 12. This might be ascribed to the possible transient re-crystallization phase upon according to the energy applied, which eventually get destroyed (partially amorphisation) at higher energy.

The absorbance spectrum shows how the absorbance of light depends upon the wavelength of the light. The spectrum itself is a plot of absorbance versus wavelength and is characterized by the wavelength (λ_{max}) at which the absorbance is the greatest. The value of λ_{max} is important for several reasons. This wavelength is characteristic of each compound and provides information on the electronic structure of the crystal or sample.

In order to better understand of the effect of the plasma on the surface of the polymer, it is important to study the inter actions between the plasma and polymer surface. FTIR spectra of three samples namely, PET sample before exposed to the plasma (Figure 13), PET sample exposed to the plasma with energy of 875 J (Figure 14), and PET sample exposed to the plasma with 1715 J (Figure 15).

It is found that the FTIR spectra of the PET sample before exposed to the capillary plasma do not indicate any significant difference in the surface chemical compositions. Absence of any distinctive difference in the FTIR spectra of the PET sample before and after exposed to the plasma can be attributed to the fact that the interaction of the plasma takes place at very thin surface layer, whereas sampling depth of FTIR technique is many times larger to detect structural alteration

after the plasma treatment^[26]

FTIR studies were carried out to determine the extent of structural changes (bond breaking) in PTFE polymer. Figure 16 illustrates the results obtained with FTIR spectroscopy for the sample before exposed to the plasma (pristine). The PTFE sample (thickness 0.5 μm) were exposed to the capillary plasma discharge at different energies (875 and 1715 J) was shown in Figures 17 and 18 respectively.

The spectra of the pristine PTFE reveal the characteristic bands at 500–770 cm^{-1} that are assigned to CF_2 rocking, bending and wagging vibrations^[27]. The nominated peaks are as following: the first at 1123 cm^{-1} that is due to $-\text{CF}_2-$ symmetrical stretching and the second at 1296 cm^{-1} that is due to $-\text{CF}_2-$ asymmetrical stretching. A strong band at 2387 cm^{-1} is a combination band of two modes associated with CF_2 backbone^[28].

On irradiation with capillary plasma discharge the bands that lie at the range 500–770 cm^{-1} remain unaffected by the irradiation. However, the intensity of the bands at 1123, 1296 and 2360 cm^{-1} decrease with the increasing energy of the plasma produced. These bands are seriously weakened in comparison with those of the pristine PTFE. This indicates that the polymer film experienced serious defluorination. As the plasma is increased the de-fluorination effect is aggravated. Therefore, it may be concluded that the plasma irradiation leads to C-C bond splitting and, at the same time, to liberation of CF_2 groups. The later together with other degradation products containing C and F were detected earlier during the PTFE irradiation^[29].

The FTIR spectrum of PTFE irradiated with ion beam fluence above 10^{18} ions/ cm^2 shows that the peak at 1790 cm^{-1} is gradually disappeared. In addition to the disappearance of a peak around 1790 cm^{-1} , there is gradual disappearance in the intensity of the band at 2360 cm^{-1} due to an increase in ion fluence as shown in Figures 17 and 18. This indicated that chain scission may take place at the carbonate site with probable destruction of the carbonyl group (C=O)^[29]. Hence, chain scission generation leads to the conclusion that the oxidation of a PTFE sample increases with increasing the irradiation dose^[29].

The main difference between PTFE and other common polymeric materials as exposed to ion beam ra-

diation is the high radiation sensitivity and extremely high ablation yield. The difference can be explained by different mechanisms of degradation, product creation and their different structures. The irradiation of PTFE preferentially breaks the weaker C-C bonds (290 kJ/mol) while much stronger C-F bonds (480 kJ/mol) remain untouched^[27,28]. This also confirms that the surface wettability and surface free energy will increase with increasing the ion beam fluence^[30].

CONCLUSION

The surface morphology of the pristine and irradiated PET and PTFE samples were observed by SEM. The pristine PET and PTFE Film are transparent and their surfaces are smooth. The color of films changes gradually with irradiation from colorless to yellowish to dark brown. The change in the color of polymer could be attributed to the copper deposition on the surface of the sample and formation of hydrogenous carbon because the discharge occurs in atmosphere.

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