Contents lists available at Article URL
KKU Journal of Basic and Applied Sciences
Journal homepage: jbas.kku.edu.sa

RF Plasma Treatment of Poly-Tetra-Fluoro-Ethylene (PTFE) Polymer Using Air and Argon gases

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Received: September 2018 / Revised: Ovtober 2018 / Accepted: December 2018 / Published: February 2019

Abstract: This study aims at identifying the properties of PTFE (poly-tetra-fluoroethylene) polymer surface after the application of RF plasma treatment using air and Argon gases. The morphology and roughness of the PTFE surface were observed by using the atomic force microscope (AFM). Treatment in Ar plasma causes changes in the roughness and morphology of the surface, but air plasma treatment does not cause changes in the surface. The study utilizes a scanning electron microscope (SEM) and X-Ray Diffraction (XRD) measurements for analyzing and characterizing the treated surface of the PTFE polymer. One of the findings of the study is that the plasma exposure time has the most significant effect on the changes in the surface of the polymer. Longer plasma treatment proves better PTEF by polymer exposure time, and RF plasma treatment in Ar gas causes significant morphological and chemical changes. Little Changes in the surface caused by air plasma treatment are observed using scanning electron microscopy (SEM). A significant change was observed in the X-ray diagram for both air an Argon plasma treated surface.

Keywords: RF-glow discharge, plasma surface treatment, PTFE (poly-tetra-fluoroethylene) polymer

1. INTRODUCTION

Solid surface films are usually treated by plasma for etching, crosslinking, surface activation (radical formation) and pre-deposition processes. Changes in the surface are not limited to plasma irradiation, for they can be induced by gases. Aging is a process that is characterized by significant chemical changes in the surface, the formation of new functional groups, branching, cross linking, and low molecular species formation [1–3].
Polymer surface treatment is used in conventional textiles and technical textiles. It is used for improving textile properties such as irritability, printability, printing, coating, shrinkage resistance, and usability [4].
This activation is induced by breaking the weak bonding of hydroxyl, carbonyl and carboxylic acid groups in the surface, and replacing them with stable groups yielding a positive influence on adhesion and hydrophilisation.
Teflon (PTFE -polytetrafluoroethylene) have special properties; therefore, it is used in many applications such as the electronic, medical, pharmaceutical and Biopharma industries [1]. Radio frequency plasma sources are used for the treatment of polymers to improve the adhesion and wettability of the PTFE surface [5, 6].

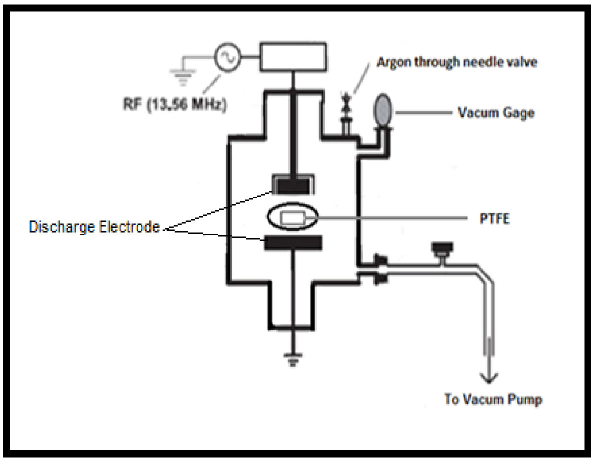

Figure 1. The experimental setup

The treatments of poly (tetrafluoroethylene) (PTFE) by O_2 plasma were thoroughly examined by Stefano Zanini et al. [7] by using different techniques, Fourier transforms infrared spectroscopy and X-ray photoelectron spectroscopy (XPS) to show the chemical

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modifications caused by plasma treatment. Although atomic force microscopy (AFM) analyses the surface topography of the samples, they require low-power plasma (up to 50 W). This study is aimed at treating and/or modifying the PTFE polymer surface using Air and Ar RF plasma source. It also aims to demonstrate the effect of some plasma parameters; namely, the treatment time, gas pressure, RF power on the polymer surface treatment.

2. Experimental Setup

The plasma discharge chamber is formed in a cylindrical vacuum chamber made of stainless-steel. Its diameter is 15 cm, and height is 25 cm. It also contains two parallel plates electrodes spacing of 6 cm and has a diameter of 5 cm. The two electrodes are covered with ceramic. The lower electrode is connected to the RF power supply 0-200W, and the frequency is 13.56 MH. The chamber is connected with a rotary pump down to base pressure of 10^{-4} Torr. High purity Argon gas and/or air is used to generate the plasma. The pressure of air or Ar gas varies from 1 to 10 Torr (figure 1). PTFE sheets have 127 µm thickness, and they are purchased from DuPont (USA). The PTFE polymer surface is shown by using scanning electron microscopy SEM (JSEM 7400F, Joel, Japan), atomic force microscopy AFM (CP-11 SPM, Veeco,USA) and X-ray diffraction XRD (SHIMADZU X-Ray Diffractometer 6000, 60KV,80mA, Cu-X-ray TUBE).

3. THE HARMONIC OSCILLATOR POTENTIAL

PTFE is one of the most inert polymer materials. Recently, it has been increasingly used in a variety of industries due to its desired physical and chemical properties. This study is aimed at the activation or modification of the PTFE surface.

3.1 SEM Observations

Figures 2(a) – (d) show the surface topography of the PTFE films using the SEM technique. Figure 2 (a) shows the surface of the control sample (untreated). It is possible to observe some granular unevenness of the surface.

Figure 2 (b-d) shows the surface of the sample treated by the Ar RF plasma of 100 W. The unevenness disappears in this surface image, and it becomes flatter or softer. It becomes more observable as the change in the surface increases with the increase of the time of treatment.

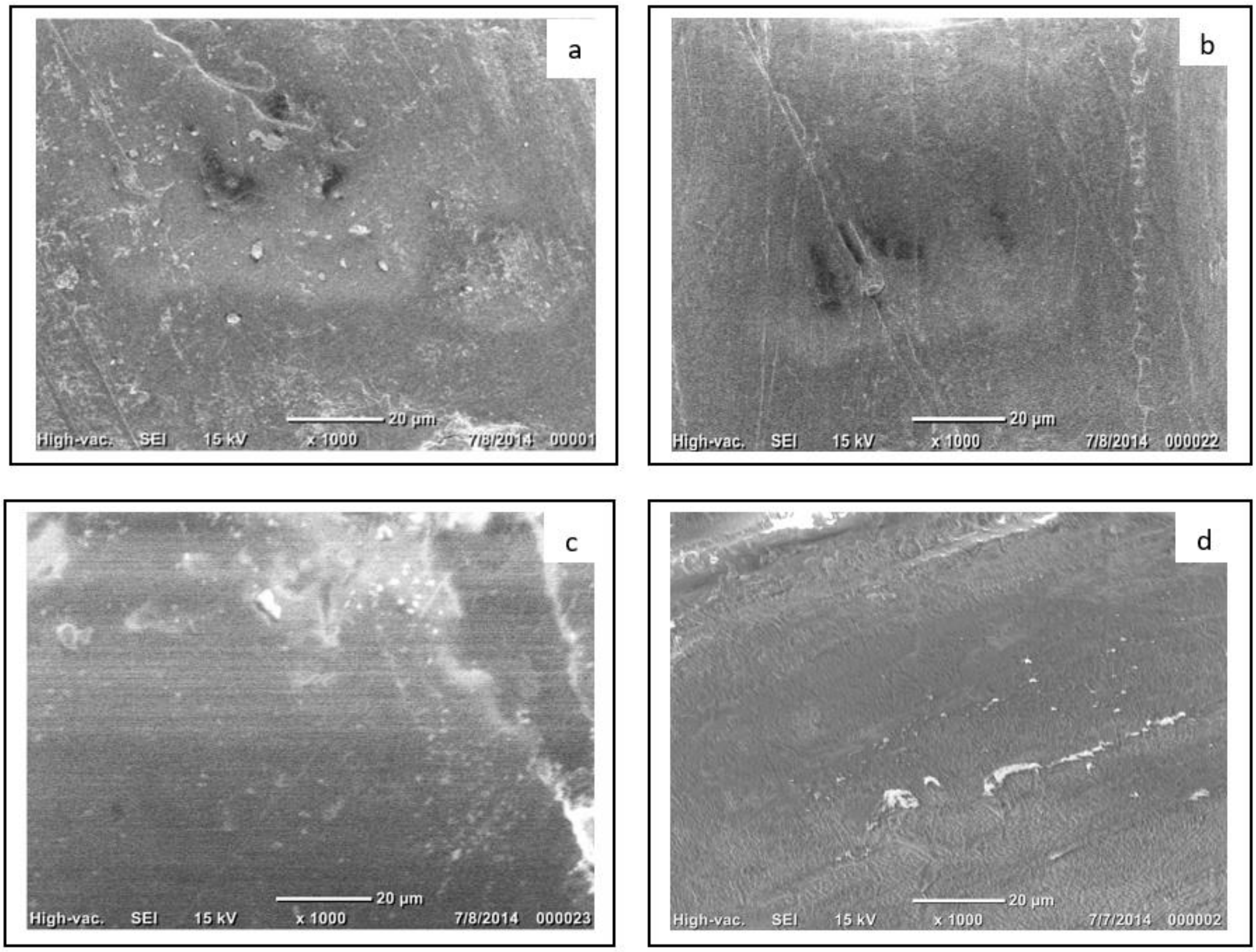

Figure 2(a-d). SEM images of the morphology of the PTFE surface treated by Ar RF plasma in 100 W (a) untreated sample (b,c,d) samples treated with Ar Plasma at 1, 5, 20 min and 1 torr gas pressure

The longer the treatment time, the more the changes.
Plasma treatment has little effect on the surface activation, i.e. the increase in the surface energy is because PTFE is a chemically inert polymer. The increase in the free energy of the surface is caused by the functional utilization of the polymer surface, with hydrophilic groups on it. After all subsequent treatments, there is a minor increase in the polar component. However, this change in the polar component is attributed to the chemically inert nature of PTFE. It is important to observe that the surface energy measurement shows that the increase in the polar component indicates the formation of covalent bonds [8].

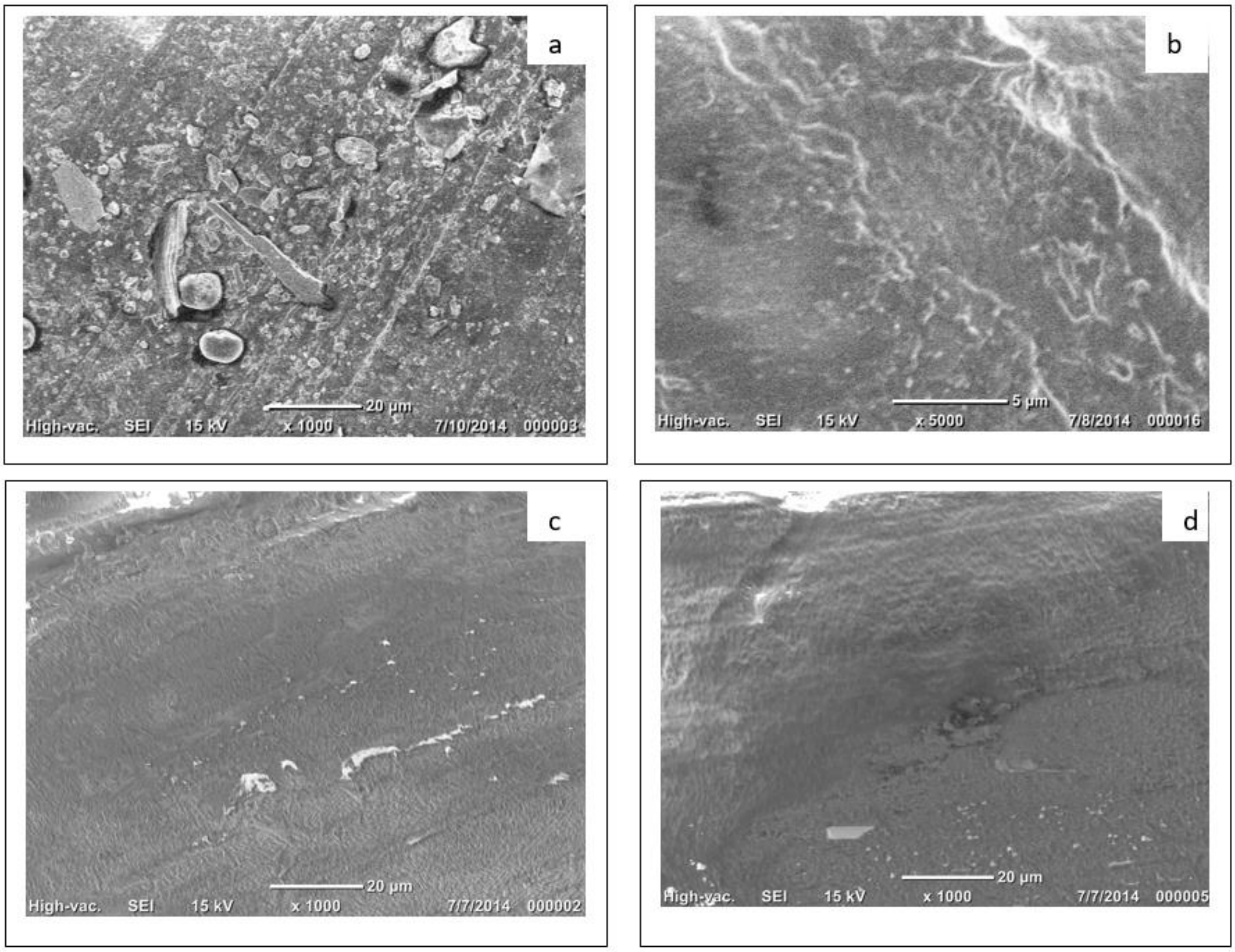

Figure.3 (a-d) SEM images of the morphology of the PTFE surface treated by air RF plasma in 100 W for 1, 5, 10,20 min and 1 torr gas pressure.

4. Atomic Force Microscopy (AFM)

The surface morphology of treated PTFE samples is measured by AFM in contact mode on the 5 × 5 μm2 areas. In figures. 4 (a)-(d), each AFM image is analyzed in terms of the average roughness of the surface. The data obtained show that the average roughness of the surface (RMS) increases when the time of treatment is increased. However, the untreated film and the film treated with argon plasma for 20 minutes have an average surface roughness of 6.5 nm and 18.5 nm, respectively. The roughness of the PTFE surfaces increases when the treatment time is increased; this is due to the bombardment of plasma species on the surface of PTFE films; therefore, it can support the adhesion improvement [11,12].

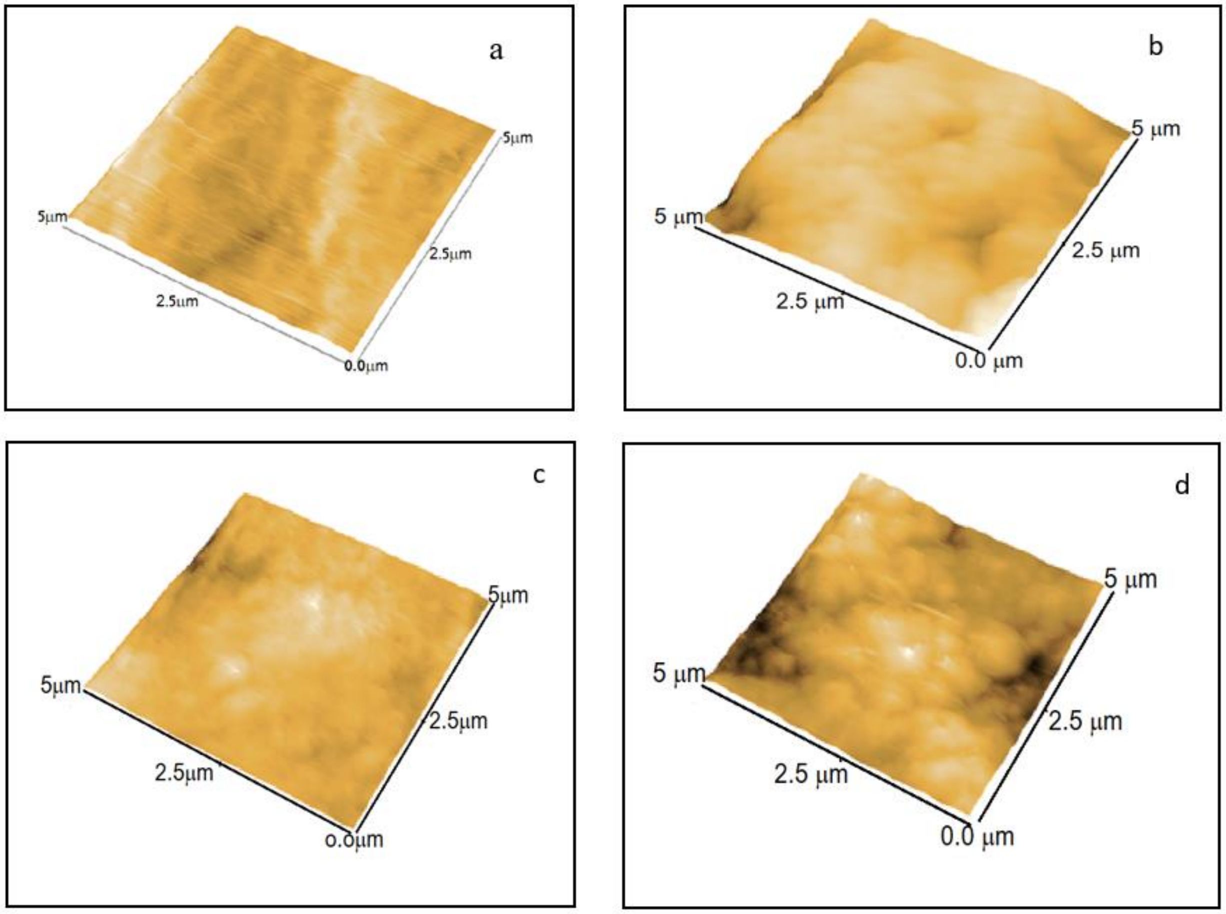

Figure 4. AFM images surface of PTFE film treatment with Ar Plasma at 100 watt power (a) untreated sample (b-d) treatment for 1, 5,20 min and 1 torr gas pressure

The increase in surface free energy refers to the improvement of adhesion on polymer surfaces. AFM results support this notion because they show that roughness increases.
Wilson et. al.(13) point out that the AFM of argon plasma treatment of PTFE results in a hummock relief, and the RMS roughness is equal to 28 nm.
However, figure 5 shows that reducing plasma treatment time results in a small change in the morphology of the polymer surface treated with air plasma.

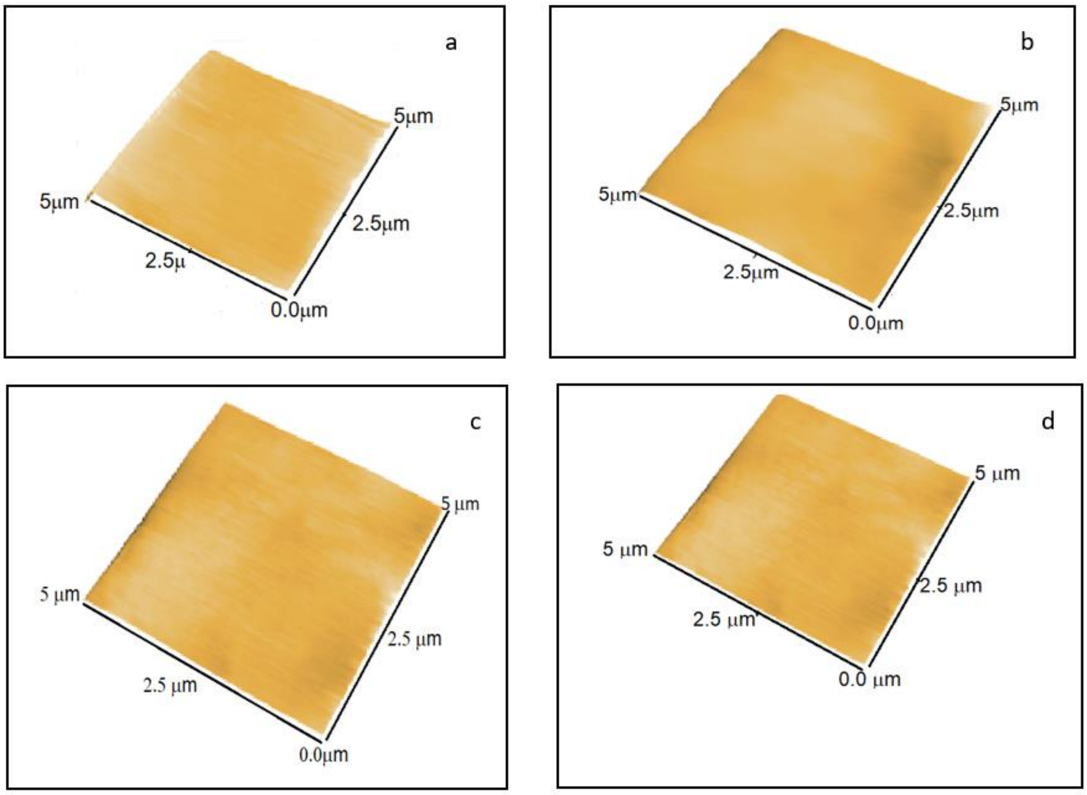

Figure.5 (a-d) AFM images surface of PTFE film treatment with air Plasma at 100 watt power for 1, 5, 10,20 min and 1 torr gas pressure.

Increasing the length of plasma treatment time results in oxidation of the surface, elimination of the surface contaminants, and amelioration of the wettability of the surface. However, long treatment time causes chemical etching incidence, increase in sample temperature, and the formation of nanostructures. Aging is the main disadvantage of plasma treatment aimed at the activation of polymers. Over time, the functional groups formed during plasma treatment lose their stability. As a result, the surface resorts to regain its untreated state. Thus, it loses its hydrophilic property spontaneously [9,10].

5. THE ASYMPTOTIC ITERATION METHOD DEVELOPMENT

XRD measurements are conducted on a Teflon sheet while changing the length of treatment time to obtain information

about the induced structural changes. Figure 6 (a-d) shows the obtained X-ray diffraction patterns in Teflon samples that result from changing the length of the treatment time, at RF plasma power = 100 watt using Agron gas plasma. These figures show that increasing the length of the plasma treatment time results in having higher peak intensity. It can also be noted that the XRD patterns are characterized by the appearance of two peaks; the dominant one is 49.72, and the small one is 17.48 degrees on 2 theta axes.

The small peak (at 17.48 in the 2 theta axes) is painted, and it increases by increasing the length of plasma treatment time. The more intense and pointed peak (at 49.72 on the 2 theta axes) indicates that the crystalline phase is the dominant phase after inducing the plasma treatment of the PTFE polymer.

Figure 7 shows the XRD patterns in air plasma that are characterized by the appearance of one peak of 18.3 degrees on the 2 theta axes. It also increases by increasing the length of plasma treatment time.

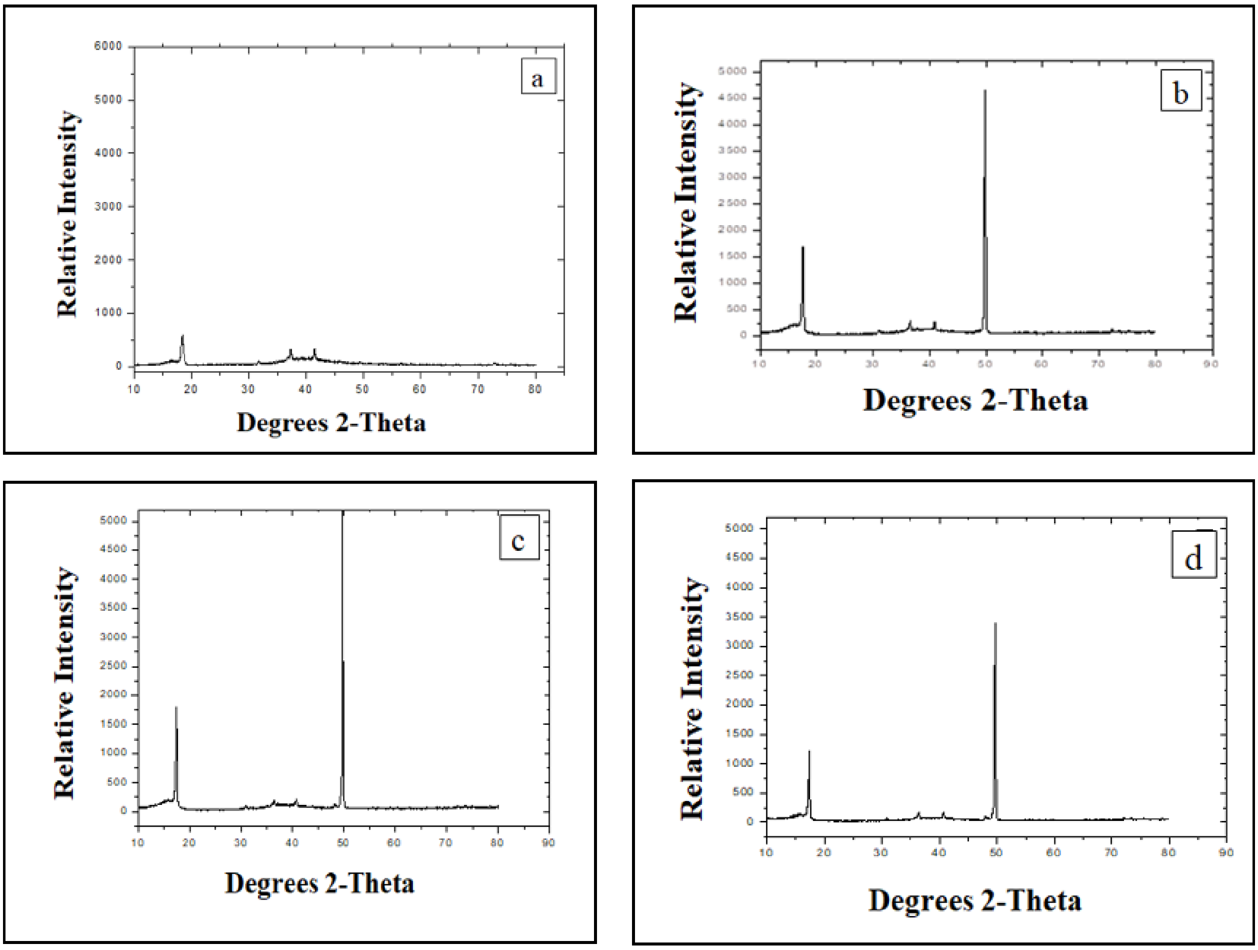

Figure 6 (a-d) X-ray diffraction patterns for PTFE film treatment with Ar gas plasma at100 watt power ((a) untreated sample (b-d) treatment for 1, 5,20 min and 1 torr gas pressure.

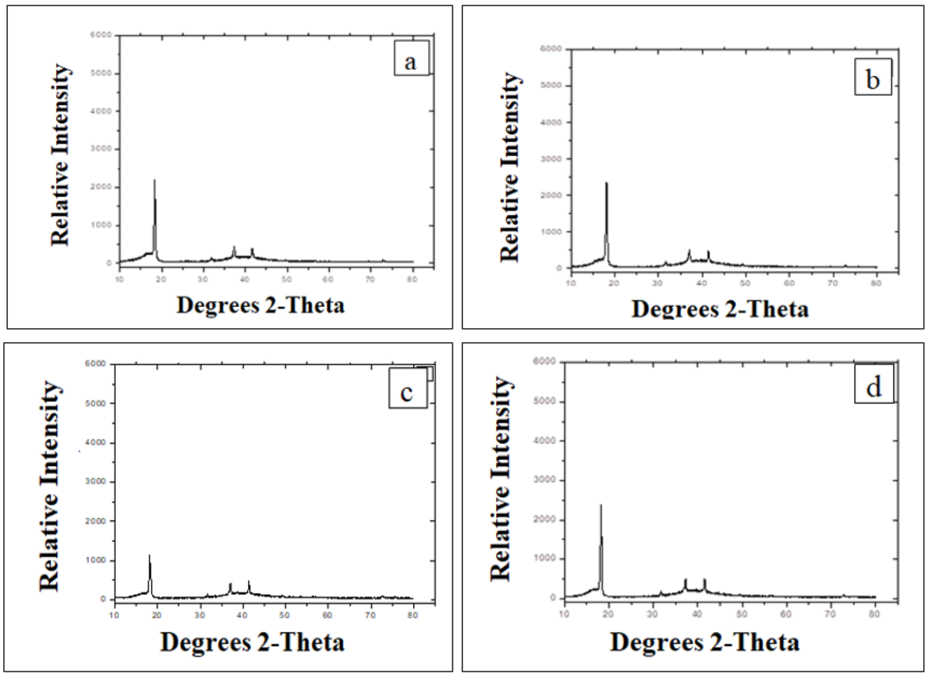

Figure.7 (a-d) X-ray diffraction patterns for PTFE film treatment with air plasma at100 watt power for 1, 5, 10,20 min and 1 torr gas pressure

7. CONCLUSION

The study tackles with the treatment of poly-tetrafluoroethylene –PTFE film surface using RF Argon and air plasma, where chemical modifications are induced. The modified surface is analyzed and measured by scanning electron microscopy (SEM), atomic force microscopy (AFM) and X-Ray Diffraction (XRD).

The SEM results show that the increase in the free energy of the surface is caused by the functional utilization of the polymer surface, with hydrophilic groups on it. A Little increase in the polar component indicates the formation of covalent bonds. Each AFM image is analyzed in terms of the average roughness of the surface. The data obtained show that the average roughness of the surface (RMS) increases with increased plasma exposure. .

The study shows that the XRD patterns are characterized by the appearance of two peaks of 17.48 and 49.72 degrees on 2 theta axes in Ar plasma, and one peak of 18.3 degrees on the 2 theta axes in air plasma. It can be concluded that increasing the length of treatment time results in higher peak intensity.

Plasma treatment proves to be an effective way for modifying and/or improving the properties of the PTFE polymer surface.

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